



**Politechnika  
Śląska**

**Silesian University of Technology**  
**Department of Materials Technology**  
**Faculty of Materials Engineering and Industrial Digitalization**

# **DOCTORAL DISSERTATION**

**Dissertation Title:**

**Cu-Mg Based Alloys with Strength, Ductility, and High  
Conductivity Achieved by SPD Technique**

**Supervisor:**

**Prof. Kinga Rodak, Ph.D.**

**Ph.D. Candidate:**

**Alireza Kalhor, M.Sc. Eng.**

**2026, Katowice, Poland**

# Acknowledgements

First of all, I wish to express my deepest and most sincere gratitude to **Prof. Kinga Rodak** for her outstanding supervision, continuous support, and invaluable guidance throughout the entirety of my doctoral studies. Her expertise, thoughtful and constructive feedback, and unwavering commitment to academic excellence have significantly influenced both the direction and the quality of this research. I am truly grateful for her encouragement, patience, insightful guidance, and the confidence she consistently placed in my abilities, all of which have been instrumental in supporting my academic development and growth as an independent researcher. Working under her mentorship has been both a privilege and a profoundly enriching academic experience.

I would also like to extend my sincere appreciation to the **Faculty of Materials Engineering and Industrial Digitalization** at the **Silesian University of Technology** for their invaluable collaboration, technical support, and continuous encouragement throughout the course of this research. Their collective expertise, collegial atmosphere, and commitment to academic excellence fostered an intellectually stimulating and highly supportive environment that greatly facilitated the progress of this work. I am particularly grateful for their willingness to share knowledge, their readiness to provide assistance when needed, and the professional opportunities they offered, all of which contributed significantly to both the successful completion of this dissertation and my overall academic development.

Finally, I wish to express my deepest gratitude to **my beloved family and dear friends** for their unwavering support, understanding, and encouragement throughout this journey. Their patience, constant presence, and sincere belief in my abilities have been a continuous source of strength and motivation. I am profoundly thankful for their care, trust, and quiet sacrifices, which have supported me both personally and academically. Without their enduring support and companionship, reaching this milestone would not have been possible.

## Contents:

|                                                                               |    |
|-------------------------------------------------------------------------------|----|
| <b>1. Introduction</b>                                                        | 1  |
| <b>2. Literature Review</b>                                                   | 5  |
| 2.1. Copper and Copper Alloys                                                 | 5  |
| 2.1.1. Introduction to Copper and its Alloys                                  | 5  |
| 2.1.2. Cu-Mg Alloys                                                           | 9  |
| 2.1.3. Cu-Mg-Ca Alloys                                                        | 11 |
| 2.2. Strengthening Mechanisms                                                 | 12 |
| 2.2.1. Solid Solution Strengthening                                           | 12 |
| 2.2.2. Precipitation Hardening                                                | 13 |
| 2.2.3. Dispersion Strengthening                                               | 15 |
| 2.2.4. Deformation Strengthening                                              | 16 |
| 2.2.4.1 Conventional Deformation                                              | 16 |
| 2.2.4.2. Severe Plastic Deformation                                           | 18 |
| 2.2.4.2.1. Common SPD Techniques                                              | 18 |
| 2.2.4.2.2. MaxStrain Process                                                  | 20 |
| 2.2.4.2.3. KoBo Extrusion                                                     | 22 |
| 2.2.5. Grain Refinement                                                       | 23 |
| 2.3. Electrical Conductivity                                                  | 24 |
| 2.4. Review of Previous Research on Cu-Mg and Cu-Mg-Ca Alloys                 | 25 |
| 2.5. Manufacturing Technologies for Cu-Mg-based Alloys                        | 30 |
| 2.6. Summary of Literature Review                                             | 31 |
| <b>3. This Investigation</b>                                                  | 32 |
| <b>3.1. Approach 1: Ca Addition, Heat Treatment, and MaxStrain Processing</b> | 32 |
| 3.1.1. Methodology                                                            | 33 |
| 3.1.1.1. Materials Fabrication                                                | 33 |
| 3.1.1.2. Chemical Composition Analysis                                        | 33 |
| 3.1.1.3. Differential Scanning Calorimetry                                    | 34 |
| 3.1.1.4. MaxStrain Processing                                                 | 34 |
| 3.1.1.5. Microstructural Characterization                                     | 35 |
| 3.1.1.6. Mechanical Properties Assessment                                     | 35 |
| 3.1.1.7. Electrical Conductivity Evaluation                                   | 36 |
| 3.1.2. Results and Discussion                                                 | 37 |
| 3.1.2.1. Characterization of As-Cast Alloys                                   | 37 |

|                                                                                           |           |
|-------------------------------------------------------------------------------------------|-----------|
| 3.1.2.2. DSC Analysis.....                                                                | 40        |
| 3.1.2.3. Annealing Processes .....                                                        | 43        |
| 3.1.2.4 MaxStrain Processing .....                                                        | 45        |
| 3.1.2.5. Simulation of the MaxStrain Process.....                                         | 47        |
| 3.1.2.6. Microstructure of MaxStrain-Processed Samples.....                               | 51        |
| 3.1.2.7. Mechanical Properties.....                                                       | 61        |
| 3.1.2.8. Fractography .....                                                               | 68        |
| 3.1.2.9. Electrical Conductivity .....                                                    | 71        |
| 3.1.3. Conclusions of Approach 1 .....                                                    | 76        |
| <b>3.2. Approach 2: Combination of Different Deformation Methods .....</b>                | <b>78</b> |
| 3.2.1. Methodology .....                                                                  | 78        |
| 3.2.1.1. Material Fabrication.....                                                        | 78        |
| 3.2.1.2. Extrusion Processes .....                                                        | 78        |
| 3.2.1.3. MaxStrain Processing .....                                                       | 79        |
| 3.2.2. Results and Discussion.....                                                        | 80        |
| 3.2.2.1. Calculation of Equivalent Strain.....                                            | 80        |
| 3.2.2.1.1. Extrusion Processes .....                                                      | 80        |
| 3.2.2.1.2. MaxStrain Processing .....                                                     | 83        |
| 3.2.2.2. Post-Deformation Microstructures .....                                           | 85        |
| 3.2.2.3. Mechanical Properties.....                                                       | 89        |
| 3.2.2.4. Fractography .....                                                               | 92        |
| 3.2.2.5. Electrical conductivity .....                                                    | 93        |
| 3.2.3. Conclusions of Approach 2 .....                                                    | 96        |
| <b>3.3. Approach 3: Fabricating Density-Reduced Highly Conductive Cu/Mg Composite ...</b> | <b>97</b> |
| 3.3.1. Methodology .....                                                                  | 97        |
| 3.3.1.1. Composite Fabrication.....                                                       | 97        |
| 3.3.1.2. Transmission Electron Microscopy .....                                           | 99        |
| 3.3.1.3. X-Ray Computed Tomography .....                                                  | 100       |
| 3.3.1.4. Compression Test .....                                                           | 101       |
| 3.3.2. Results and Discussion.....                                                        | 101       |
| 3.3.2.1. Composite Structure .....                                                        | 101       |
| 3.3.2.2. XCT Scan Results.....                                                            | 104       |
| 3.3.2.3. Post-Extrusion Microstructures .....                                             | 106       |
| 3.3.2.3.1 EBSD Analysis of Cu Matrix.....                                                 | 106       |
| 3.3.2.3.2. TEM Analysis of Cu Matrix.....                                                 | 107       |

|                                                              |            |
|--------------------------------------------------------------|------------|
| 3.3.2.3.3. TEM Analysis of Mg Filaments .....                | 108        |
| 3.3.2.4. Hardness Test.....                                  | 109        |
| 3.3.2.5. Compression Test .....                              | 110        |
| 3.3.2.6. Microstructure of Cu Matrix after Compression ..... | 112        |
| 3.3.2.7. Electrical Conductivity .....                       | 113        |
| 3.3.3. Conclusions of Approach 3.....                        | 114        |
| <b>4. Discussion and Analysis of Research Findings.....</b>  | <b>115</b> |
| <b>5. Future Outlook and Opportunities .....</b>             | <b>121</b> |
| <b>6. Summary and Conclusions .....</b>                      | <b>123</b> |
| <b>References.....</b>                                       | <b>127</b> |
| List of Figures .....                                        | 148        |
| List of Tables .....                                         | 152        |
| <b>Abstract.....</b>                                         | <b>153</b> |
| <b>Streszczenie.....</b>                                     | <b>154</b> |
| Publications.....                                            | 155        |

## List of Technical Terms and Their Abbreviations

| Technical term used in this study         | Abbreviation |
|-------------------------------------------|--------------|
| Accumulative roll bonding                 | ARB          |
| Coefficient of thermal expansion          | CTE          |
| Constrained groove pressing               | CGP          |
| Continuous dynamic recrystallization      | CDRX         |
| Dense dislocation wall                    | DDW          |
| Differential scanning calorimetry         | DSC          |
| Dynamic recovery                          | DRV          |
| Dynamic recrystallization                 | DRX          |
| Electron backscatter diffraction          | EBSD         |
| Elongation to fracture                    | EL           |
| Energy-dispersive X-ray spectroscopy      | EDS          |
| Equal-channel angular pressing            | ECAP         |
| Face-centered cubic                       | FCC          |
| Grain orientation spread                  | GOS          |
| High-angle grain boundaries               | HAGB         |
| High-entropy alloy                        | HEA          |
| High-pressure torsion                     | HPT          |
| High-strength and high-conductivity       | HSHC         |
| International annealed copper standard    | IACS         |
| Inverse pole figure                       | IPF          |
| Kernel average misorientation             | KAM          |
| Low-angle grain boundary                  | LAGB         |
| Oxide dispersion strengthening            | ODS          |
| Room temperature                          | RT           |
| Scanning electron microscope              | SEM          |
| Scanning transmission electron microscope | STEM         |
| Severe plastic deformation                | SPD          |
| Short-range order                         | SRO          |
| Stacking fault energy                     | SFE          |
| Transmission electron microscopy          | TEM          |
| Ultimate tensile strength                 | UTS          |
| Ultrafine-grained                         | UFG          |
| Wire electrical discharge machine         | WEDM         |
| X-ray computed tomography                 | XCT          |
| Yield strength                            | YS           |

# 1. Introduction

Copper (Cu), which is one of the few elements naturally found in its metallic form, holds the distinction of being among the first metals ever used by early human societies [1]. Evidence of its utilization dates back to approximately the 9th millennium B.C., initially in the form of small objects such as beads and simple tools, with its applications gradually expanding in later periods to include household items, ritual objects, mirrors, statues, and containers [2]. Following the onset of industrialization, Cu and its alloys have become essential elements in a wide range of different sectors such as electronics, transportation, and aerospace, owing to their superior electrical [3–5] and thermal [6–8] conductivity, corrosion resistance [9–11], and desirable mechanical properties [12–14], attainable through precise alloy design and controlled processing conditions. Recently, the development of high-performance high-strength and high-conductivity (HSHC) Cu alloys, which can offer remarkable mechanical properties alongside desirable electrical conductivity, has attracted considerable attention [15–18]. HSHC Cu alloys are well-suited for specific applications, such as electric vehicles, 5G network equipment, electrical contacts, power transmission components, and high-performance electronic devices [19,20]. Figure 1, which shows the number of publications on Cu alloys with high strength and/or high conductivity since the early 21st century (retrieved from the Scopus database), clearly highlights the increasing research interest in both developing new high-performance Cu alloys and improving the properties of existing alloys through microstructure engineering.

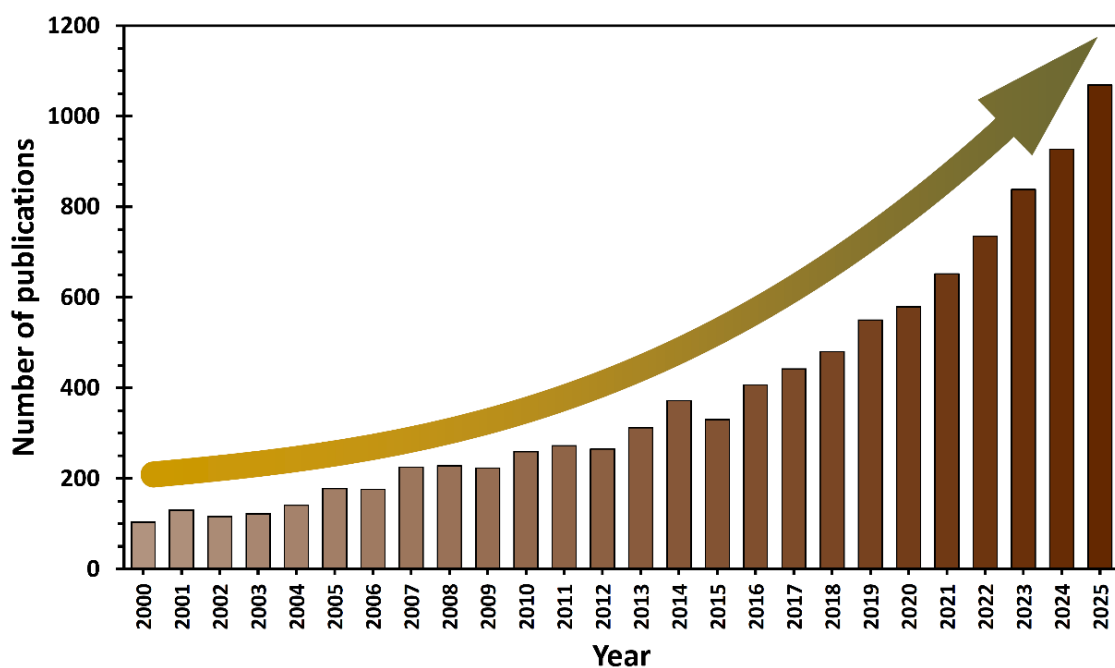


**Figure 1:** Number of publications on high-strength and/or high-conductivity Cu alloys from 2000 to 2025 (data retrieved from the Scopus database).

In general, the term HSHC refers to Cu alloys having tensile strengths 1.5–4 times greater than pure Cu (300–800 MPa) and electrical conductivities ranging from 50 to 95% of pure Cu [15]. Annealed pure Cu has an electrical conductivity of 58 MS/m, which corresponds to 100% on the international annealed copper standard (IACS) scale [21]. HSHC Cu alloys typically achieve their improved mechanical properties through a combination of strengthening methods such as solid solution strengthening [22], precipitation hardening [23], deformation strengthening [24], and grain refinement [25]. However, the simultaneous attainment of both high strength and favorable electrical conductivity still remains a significant challenge in the development of HSHC Cu alloys [26]. This limitation comes from the fact that crystal defects such as vacancies, solid solution atoms, dislocations, grain boundaries, and second-phase particles generated by strengthening mechanisms increase electron scattering and, as a result, the electrical resistivity of the alloy [27].

Among all grades of Cu alloys, Cu-Mg alloys, which are renowned for their exceptional mechanical properties, constitute a major category of HSHC Cu alloys and have been widely adopted across various industrial sectors, including in high-speed railway contact wires [28,29]. For instance, Zhu et al. [30] demonstrated the successful application of Cu-0.4Mg (wt.%) alloy as a contact wire material in the high-speed railway industry. Since Mg has a certain amount of solubility in Cu, as shown in the binary phase diagram of the Cu-Mg system in [Figure 2](#), the primary strengthening mechanism in Cu-Mg alloys is determined by the Mg content [3]. Specifically, alloys with lower levels of Mg achieve their strength merely through solid solution strengthening, while those with higher levels of Mg can benefit from both solid solution strengthening and second-phase hardening. As an example, the study of Gorsse et al. [31] can be mentioned, in which they fabricated Cu-Mg alloys with different Mg contents of 4.1, 8.1, and 23.1 (at.%) and reported that two-phase Cu-8.1Mg and Cu-23.1Mg alloys exhibited a higher yield strength (YS) compared to the single-phase Cu-4.1Mg solid solution.

The mechanical properties of Cu-Mg alloys can be further improved through deformation strengthening, especially severe plastic deformation (SPD) [32]. In this regard, the study conducted by Rodríguez-Calvillo et al. [33] can be mentioned as an example, in which they demonstrated that the YS and ultimate tensile strength (UTS) of Cu-0.2Mg (wt.%) and Cu-0.5Mg (wt.%) alloys were significantly increased by undergoing a modified equal-channel angular pressing (ECAP) process. In another study conducted by Ma et al. [34], it was observed that ECAP processing of Cu-0.43Mg (wt.%) alloy using route B<sub>C</sub> for up to 8 passes led to a

reduction in average grain size and an enhancement in both hardness and YS of the alloy by the number of ECAP passes. In a recent study, Bodyakova et al. [35] applied cold working by drawing, rolling, and ECAP to a Cu-0.25Mg (wt.%) alloy and observed that plastic deformation resulted in grain refinement through the formation of strain-induced low-angle grain boundaries (LAGBs,  $2^\circ \leq \theta < 15^\circ$ ) and their subsequent transformation into high-angle grain boundaries (HAGBs,  $15^\circ \leq \theta < 65^\circ$ ), which led to increasing the UTS of their samples.

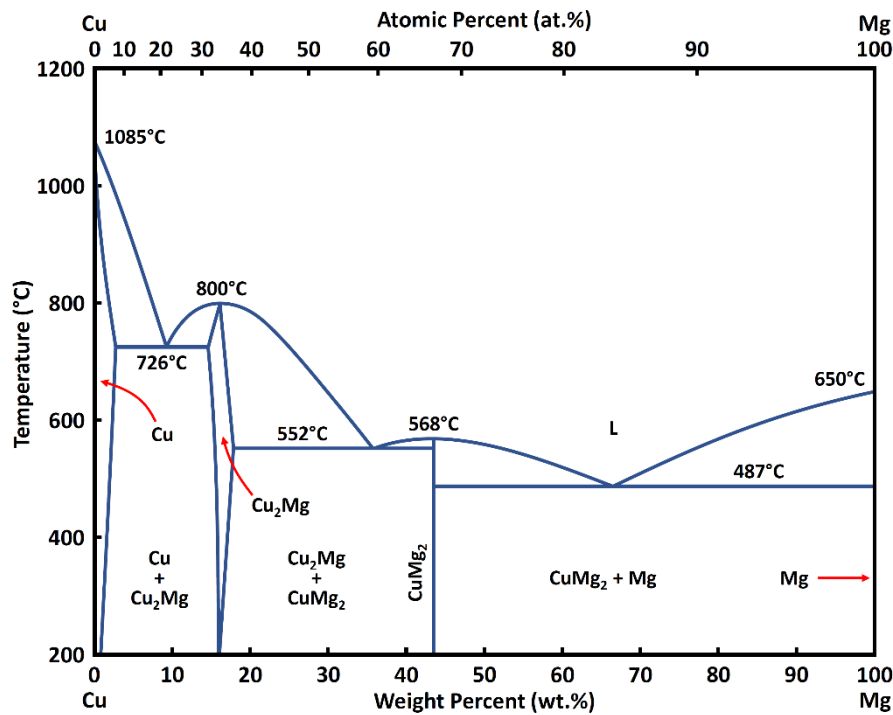


**Figure 2:** Binary phase diagram of Cu-Mg system (Adapted under the terms of CC BY-NC 4.0 license [36]. Copyright 2016, Springer Nature).

In addition to SPD processing, the incorporation of supplementary alloying elements such as Ca [37], Ce [38], Fe [39], and Y [38] is another viable approach for producing Cu-Mg-based alloys with enhanced mechanical properties. However, the key factor in selecting alloying elements is to choose those elements that do not significantly reduce the electrical conductivity. The influence of incorporating common alloying elements on the electrical conductivity of Cu is shown in Figure 3. It can be seen that Ca has a comparatively lower detrimental influence on the electrical conductivity of Cu when compared to other alloying elements such as Ni, Sn, Mn, Al, Co, Fe, and Si [37]. Moreover, as can be seen from the binary phase diagram of the Cu-Ca system shown in Figure 4 [3], the solid solubility of Ca in Cu is extremely limited, and even a small addition of Ca to Cu results in the formation of the  $\text{Cu}_5\text{Ca}$  intermetallic compound [40]. Hence, in small quantities, Ca addition can strengthen Cu-Mg-based alloys through second-phase hardening without significantly affecting the electrical conductivity of the alloy [37].

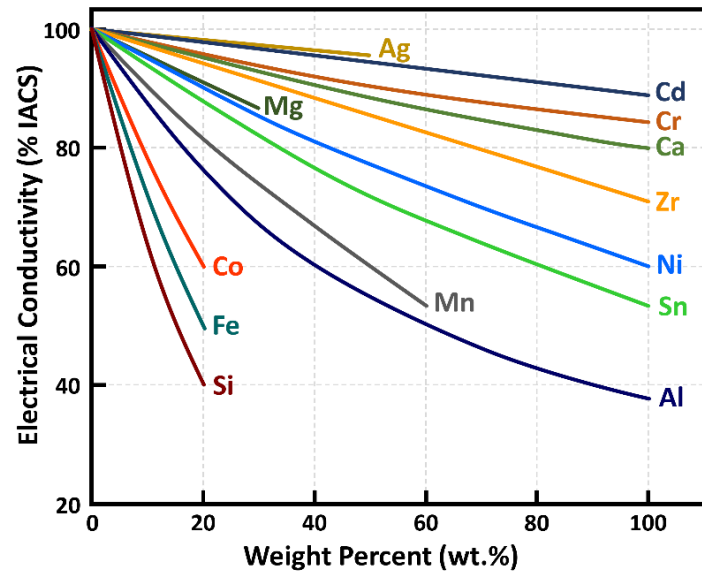


**Figure 3:** The influence of common alloying elements on the electrical conductivity of Cu (Adapted with permission [37]. Copyright 2017, Elsevier).

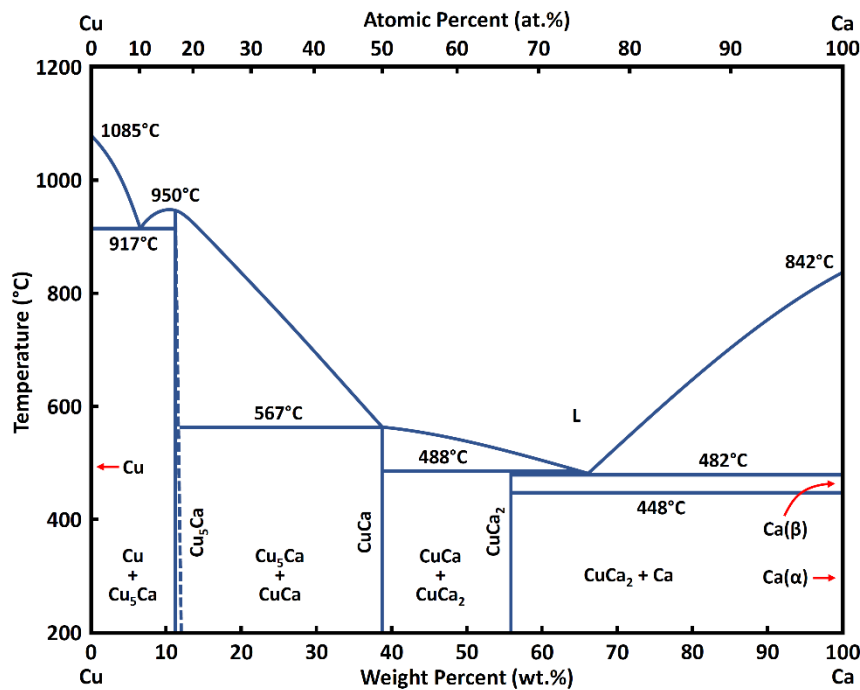


**Figure 4:** Binary phase diagram of Cu-Ca system (Adapted with permission [41]. Copyright 1984, Springer Nature).

Taken together, the above discussion clearly demonstrates that adding Ca to the chemical composition of Cu-Mg alloys, along with applying SPD processing, has the potential to produce a Cu-Mg-Ca alloy with even further enhanced mechanical properties compared to Cu-Mg alloys. There have also been some attempts in this direction. For example, Ma et al. [42] employed ECAP processing on Cu-0.43Mg (wt.%) and Cu-0.41Mg-0.06Ca (wt.%) alloys using route B<sub>C</sub> at room temperature (RT) and observed that the ternary Cu-Mg-Ca alloy

exhibited a higher hardness and higher YS across all ECAP passes compared to the binary Cu-Mg alloy.

Building on the findings of previous studies, the present work selected a Cu-0.7Mg (wt.%) alloy as the base material for investigation. The reason behind choosing 0.7 wt.% of Mg was that based on the Cu-Mg binary phase diagram shown in [Figure 2](#), this concentration falls below the minimum threshold of Mg required for the formation of the Cu<sub>2</sub>Mg intermetallic phase in Cu-Mg alloys [43] and, therefore, the base material would be a single-phase Cu-Mg solid solution. Additionally, 0.1 wt.% Ca was also added to the binary Cu-0.7Mg (wt.%) alloy to obtain a ternary alloy that simultaneously benefits from solid solution strengthening and second-phase hardening. This minimal Ca content was deliberately chosen to induce second-phase hardening while minimizing the adverse effect on electrical conductivity. Next, the binary and ternary alloys were subjected to various heat treatment routes combined with deformation through conventional methods, such as forward extrusion, as well as different SPD processes, including KoBo extrusion (extrusion with oscillatory reversible rotation of the die) and the MaxStrain module of the Gleeble thermomechanical simulator. The combined effects of these strengthening strategies on the microstructural evolution, mechanical properties, and electrical conductivity of the fabricated alloys were systematically investigated, with a detailed presentation and discussion of the results provided in Section 3.

## 2. Literature Review

### 2.1. Copper and Copper Alloys

#### 2.1.1. Introduction to Copper and its Alloys

Cu, a naturally occurring transition metal, has been a cornerstone of human civilization for millennia due to its exceptional physical and chemical properties [44,45]. It exists in the Earth's crust mainly as native metal or in sulfide (e.g., chalcopyrite, CuFeS<sub>2</sub>), oxide (e.g., cuprite, Cu<sub>2</sub>O; tenorite, CuO), or carbonate (e.g., malachite, Cu<sub>2</sub>CO<sub>3</sub>(OH)<sub>2</sub>; azurite, Cu<sub>3</sub>(CO<sub>3</sub>)<sub>2</sub>(OH)<sub>2</sub>) ores, and has been used since antiquity for tools, ornaments, and currency [46–48]. Cu alloys are so versatile because they can be alloyed with other metals, which helps improve both their strength and chemical resilience. From a mechanical properties perspective, pure Cu is relatively soft and ductile, primarily due to its face-centered cubic (FCC) crystal structure, which provides a high number of slip systems and facilitates extensive plastic deformation

before fracture [49–51]. A summary of the key physical and mechanical properties of pure Cu is presented in Table 1.

**Table 1:** Overview of key physical and mechanical properties of pure Cu ( $\geq 99.9\%$ ).

| Property                        | Value              | Unit                              | Reference |
|---------------------------------|--------------------|-----------------------------------|-----------|
| Atomic number                   | 29                 | —                                 | [52]      |
| Atomic weight                   | 63.546             | g/mol                             | [52]      |
| Crystal structure               | FCC                | —                                 | [52]      |
| Density                         | 8.95285            | g/cm <sup>3</sup>                 | [52]      |
| Melting point                   | 1084.88            | °C                                | [52]      |
| Boiling point                   | 2570               | °C                                | [53]      |
| Electrical conductivity         | $5.96 \times 10^7$ | S/m                               | [54]      |
| Thermal conductivity            | 398                | W/m·K                             | [55]      |
| Specific heat capacity          | 0.385              | J/g·K (RT)                        | [56]      |
| Modulus of elasticity (Young's) | 110–130            | GPa                               | [57]      |
| Poisson's ratio                 | 0.34               | —                                 | [58]      |
| Shear modulus                   | ~48                | GPa                               | [59]      |
| Yield strength (Annealed)       | ~33                | MPa                               | [60]      |
| Tensile strength (Annealed)     | 210–250            | MPa                               | [60]      |
| Elongation (Annealed)           | 40–50              | %                                 | [60]      |
| Hardness (Brinell, Annealed)    | ~35                | HB                                | [61]      |
| Magnetic properties             | Diamagnetic        | Relative permeability $\approx 1$ | [62]      |

Alloying Cu with elements such as Zn, Sn, and others can significantly enhance its mechanical strength, corrosion resistance, and other properties tailored to specific applications [63]. In this context, Cu alloys are typically classified into three main categories: brasses [64], bronzes [65], and other specialized alloys, including cupronickels, aluminum bronzes, Cu-Be alloys, etc., each alloy carefully engineered to fulfill the unique requirements of various specialized applications across different industries.

Brass, primarily composed of Cu with typically 5–40 wt.% Zn, is renowned for its good corrosion resistance in many environments (partly due to the formation of a protective oxide layer), good mechanical workability and machinability, moderate electrical and thermal conductivities, tunable strength, ductility, and golden color, as well as acoustic properties, making it a versatile and enduring choice for marine, electronic, coinage, decorative, architectural, musical, and precision engineering applications [66–70]. Bronze, primarily an alloy of Cu and typically 2–20 wt.% Sn, combines superior hardness, fatigue resistance, and

corrosion resistance (supported by a multiphase microstructure and protective oxide layer) with excellent wear, acoustic, and patina-forming properties, making it indispensable for applications ranging from bearings, gears, bushings, and marine fittings to sculpture, musical instruments, and modern engineering variants such as aluminum, phosphor, and silicon bronzes [71–76]. Cu-Ni alloys, which are known as Cupronickels, typically contain 10–30 wt.% Ni and are renowned for their excellent resistance to corrosion, especially in marine environments, which makes them ideal for seawater piping, coinage, and desalination plants [77,78]. Cu-Al alloys (aluminum bronze) composed of 5–12 wt.% Al, often with additions of Fe, Ni, or Mn, offer high strength, wear resistance, and corrosion resistance, particularly in marine hardware and valve components. They are occasionally used in specialized aerospace instruments [79,80]. Cu-Be alloys contain 0.5–3 wt.% Be and have the potential to develop exceptional strength after age-hardening while maintaining good electrical conductivity, which makes them useful in precision tools (however, Be handling requires safety precautions due to its toxicity) [81–83]. Cu-Si alloys (silicon bronze) with around 3 wt.% Si and trace amounts of Mn, Fe, or Zn, are valued for their strength, corrosion resistance, and weldability and are commonly used in sculptures, architecture, and shipbuilding [84,85].

The intentional addition of elements such as Ag, Cr, Fe, Mn, Te, and Ti can enhance the strength, hardness, thermal stability, corrosion resistance, and machinability of Cu alloys, enabling their use in specialized applications across electrical, automotive, aerospace, and marine engineering [86,87]. For instance, Cu-Ag alloys are highly valued because of their exceptional combination of high electrical and thermal conductivity and improved mechanical strength, which make them desirable for high-demand applications such as electrical contacts, power transmission components, and high-performance conductors [88–91]. Cu-Cr alloys, on the other hand, are heat-treatable and retain high strength over time, which qualifies them for demanding applications such as electrical switches and resistance welding electrodes, although their machinability may be lower than that of some other Cu alloy grades [92–94]. Cu-Fe alloys are another notable Cu alloy grade known for their high strength and good resistance to softening at elevated temperatures, while maintaining adequate electrical conductivity for practical use, which makes them suitable for electrical connectors, welding electrodes, and components subjected to thermal and mechanical stresses [95–97].

Another example is Cu-Mn alloys, valued for their high strength and relatively stable electrical resistivity. These alloys are used in applications such as precision resistors and other

components where a balance of mechanical strength and stable electrical properties is required, and their properties can be adjusted by varying the Mn content and applying appropriate processing [98,99]. Cu-Te alloys, on the other hand, are valued for their improved machinability due to the presence of Te, which acts as a machinability modifier, while retaining relatively good electrical conductivity [100–102]. Likewise, Cu-Ti alloys offer a valuable combination of high strength, relatively good corrosion resistance, and moderate electrical conductivity, with their performance characteristics significantly enhanced through aging and precipitation hardening treatments [103–105]. Another grade is Cu-Mg alloys, distinguished by their combination of strength, moderate electrical conductivity, and reasonable corrosion resistance, with small amounts of Mg enhancing their mechanical properties through solid solution strengthening, making them suitable for applications that require both durability and some electrical conductivity [3,4]. [Table 2](#) provides a concise overview of the primary compositions, key properties, and typical application areas of the Cu alloys discussed above.

**Table 2:** Summary of key composition, properties and primary applications of major Cu alloys.

| Alloy           | Primary Alloying Element (wt. %) | Key Properties                                                                                                                                                       | Typical Applications                                                        | Reference    |
|-----------------|----------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------|--------------|
| Brass           | 5–40% Zn                         | Excellent corrosion resistance, good electrical/thermal conductivity (25–50% IACS), high ductility (15–50% elongation), machinability (30–100% rating), golden color | Valves, gears, musical instruments, architecture, electronics, coinage      | [67,68,106]  |
| Bronze          | 2–20% Sn                         | High hardness, fatigue and wear resistance, corrosion-resistant, good machinability (20–40% rating)                                                                  | Bearings, bushings, gears, marine hardware, sculptures, industrial fittings | [72,73,75]   |
| Cupronickel     | 10–30% Ni                        | Excellent marine corrosion resistance, good strength (300–550 MPa UTS), stable in seawater                                                                           | Seawater piping, desalination plants, coinage, marine exchangers            | [77,78,107]  |
| Aluminum bronze | 5–12% Al                         | Very high strength (550–850 MPa UTS), excellent wear and corrosion resistance, heat-resistant                                                                        | Aerospace parts, marine hardware, heavy-duty valves, pump components        | [79,80,108]  |
| Cu-Be           | 0.5–3% Be                        | Very high strength after aging (1100–1400 MPa UTS), good conductivity (15–30% IACS), non-sparking, toxic during machining                                            | Precision tools, springs, connectors, aerospace components                  | [81,109,110] |
| Cu-Si           | ~3% Si                           | Good strength (350–600 MPa UTS), corrosion resistance, and weldability                                                                                               | Architecture, shipbuilding, sculpture, marine fasteners                     | [84,85,111]  |
| Cu-Ag           | 0.03–1% Ag                       | Superior electrical/thermal conductivity (>95% IACS), enhanced strength (300–500 MPa UTS)                                                                            | Power transmission, electrical contacts, high-performance conductors        | [88,112,113] |

|       |             |                                                                                                                  |                                                                                 |               |
|-------|-------------|------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------|---------------|
| Cu-Cr | 0.5–1.2% Cr | Heat-treatable, high strength retention (400–550 MPa UTS), wear resistance, limited machinability (~20% rating)  | Electrical switches, resistance welding electrodes, electrical hardware         | [92,93,114]   |
| Cu-Fe | 0.5–3% Fe   | High strength (400–600 MPa UTS), moderate conductivity (50–65% IACS), thermally stable, corrosion-resistant      | Electrical connectors, welding tips, high-stress mechanical parts               | [17,95,96]    |
| Cu-Mn | 0.5–2.5% Mn | High strength (350–600 MPa UTS), moderate conductivity (5–30% IACS), tunable via heat treatment                  | Connectors, electrical components, thermal-mechanical service parts             | [98,99,115]   |
| Cu-Te | 0.3–0.7% Te | Improved machinability (85–90% rating), good conductivity (90–95% IACS)                                          | Machined electrical parts, terminals, EDM electrodes                            | [100,101,116] |
| Cu-Ti | 1–4.5% Ti   | High strength (800–1100 MPa UTS), good corrosion resistance, moderate conductivity (10–20% IACS), age-hardenable | Electronic connectors, springs, electrical components                           | [103,104,117] |
| Cu-Mg | 0.2–1% Mg   | Balanced strength (400–700 MPa UTS) and conductivity (60–85% IACS), corrosion resistance, density-reduced        | Power transmission, electrical connectors, automotive and marine wiring systems | [3,4,118,119] |

Among all mentioned Cu alloy grades, Cu-Mg alloys have increasingly attracted attention across industries such as aerospace, automotive, railway transportation, and electronics due to their combination of desirable properties. Their versatility and balanced combination of electrical and mechanical characteristics make them particularly well suited for components exposed to mechanical stresses, temperature variations, and harsh operating conditions. These alloys can also be effectively processed to enhance mechanical strength while maintaining good electrical conductivity, ensuring both high performance and reliability [120]. Given these advantages, Cu-Mg alloys form the core focus of this PhD research, and a detailed explanation of their composition, processing, and performance is provided in the following sections.

### 2.1.2. Cu-Mg Alloys

Cu-Mg alloys generally contain Mg at concentrations ranging from less than 1 wt.% to over 10 wt.%, with the exact amount chosen to balance specific performance requirements, such as strength, corrosion resistance, or machinability, and to achieve the targeted physical and mechanical properties of the alloy [15]. Cu-Mg alloys containing 0.1–2 wt.% Mg provide a balanced combination of strength and electrical conductivity, while higher Mg contents promote the formation of the Cu<sub>2</sub>Mg intermetallic phase, which results in further enhancing the strength of the alloy but at the cost of reducing its electrical conductivity [3]. In fact, as the

Mg content in the alloy increases, its mechanical properties initially improve through solid solution strengthening and later through simultaneous solution strengthening and second-phase hardening, while electrical conductivity gradually declines [31]. Cu-Mg alloys are commonly produced using conventional casting techniques, with the method chosen based on part geometry, cooling rate, and production scale. Subsequent thermomechanical processing, including hot and/or cold working followed by annealing, is also typically employed to optimize strength and ductility [121–124]. Nevertheless, some challenges still remain in the way of the optimization of the properties of Cu-Mg alloy, such as achieving high strength without significantly reducing electrical conductivity, improving corrosion resistance (especially in higher-Mg alloys), and reducing processing costs through optimized heat treatments or emerging techniques such as additive manufacturing [125–129]. Table 3 provides a summary of the key characteristics of Cu-Mg alloys.

**Table 3:** Summary of the key characteristics of Cu-Mg alloys.

| Property / Feature      | Characteristics                                                                                                                               | Reference |
|-------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------|-----------|
| Density                 | Less than density of pure Cu due to Mg addition (8.80–8.92 g/cm <sup>3</sup> )                                                                | [130]     |
| Melting point           | Less than melting point of pure Cu depending on Mg content (1050–1080°C)                                                                      | [3,131]   |
| Electrical conductivity | Lower than electrical conductivity of pure Cu due to Mg alloying (60–85% IACS)                                                                | [4,32]    |
| Thermal conductivity    | Lower than thermal conductivity of pure Cu but still good for heat transfer applications (up to 340 W/m·K)                                    | [15,120]  |
| Applications            | Electrical and electronic connectors, switchgears, marine components, bearing materials, springs, and heat exchangers                         | [32,120]  |
| Advantages              | Stronger than pure Cu (400–700 MPa UTS), high corrosion resistance, good thermal and electrical properties                                    | [3–5]     |
| Limitations             | Reduced electrical conductivity compared to pure Cu, Mg increases susceptibility to oxidation at high temperatures (>450°C) during processing | [132,133] |

In the meantime, a highly effective approach to further enhance the mechanical properties of Cu-Mg alloys involves incorporating small traces of additional alloying elements to form ternary or quaternary systems [38,39]. Specifically, research has demonstrated that a small addition of Ca to the Cu-Mg system can notably improve mechanical strength while preserving electrical conductivity to a substantial degree [3,28,37,42]. This is achieved through the formation of finely dispersed Cu<sub>5</sub>Ca intermetallic phases, which strengthen the alloy matrix without substantially reducing electron mobility. Moreover, Ca additions also refine the grain structures and enhance thermal stability, which contribute to an improved performance under

elevated temperatures [5]. The ternary Cu-Mg-Ca system, with its promising balance of properties, is explored in the following section.

### **2.1.3. Cu-Mg-Ca Alloys**

Cu-Mg-Ca alloys represent a promising subclass of Cu-Mg alloys that are specifically designed to provide further enhanced mechanical properties and functional performance compared to binary Cu-Mg alloys [3,5]. Incorporating Ca into Cu-Mg alloys refines the microstructure through the formation of finely dispersed Cu<sub>5</sub>Ca intermetallic particles and improves mechanical strength and thermal stability without significantly compromising electrical conductivity, which makes this alloy well-suited for applications requiring high-performance electrical and thermal functionalities [42]. One key advantage of Ca addition to Cu-Mg alloys compared to alloying with other elements is that it has minimal effect on the electrical conductivity of the resulting ternary alloy (as illustrated in [Figure 3](#)), particularly when Ca content is precisely adjusted to avoid excessive intermetallic formation that can interfere with efficient conduction. However, excessive Ca addition may lead to the formation of coarse intermetallic phases, which can negatively affect ductility and processing characteristics.

Cu-Mg-Ca alloys respond well to thermomechanical processing and various heat treatments, which enable precise control of their microstructure and, consequently, their overall properties. In particular, these processes allow adjustment of the size, shape, and distribution of Cu<sub>5</sub>Ca intermetallic particles within the Cu matrix, as well as the crystallographic texture of the alloy, all of which play a critical role in determining mechanical and functional performance [28,37,42]. It has also been reported that the morphology of Cu<sub>5</sub>Ca intermetallic particles can be effectively modified through appropriate heat treatment procedures [5]. Specifically, applying a suitable annealing process can partially reduce the sharp edges of these particles and result in a more spheroidized morphology. This change is beneficial because it reduces local stress concentrations and promotes more uniform plastic deformation, thereby enhancing the ductility of the alloy [5]. Consequently, due to this combination of tunable microstructure and improved mechanical behavior, Cu-Mg-Ca alloys are particularly attractive for applications such as electrical contact wires, connectors, and high-performance conductors operating under elevated thermal and mechanical loads.

## 2.2. Strengthening Mechanisms

Following this introduction to the subject and a brief overview of all primary Cu alloys that are used in industry and their characteristics, the main strengthening mechanisms used to improve the mechanical properties of these alloys are discussed in the following sections. These mechanisms include solid solution strengthening, precipitation hardening, dispersion strengthening, deformation strengthening, and grain refinement. For each mechanism, the fundamental principles and their specific contributions to improving the mechanical properties of Cu alloys are explained. This discussion provides a clearer understanding of the strengthening methods applied in the fabrication of the HSHC Cu-Mg and Cu-Mg-Ca alloys in this investigation, which is discussed in Section 3.

### 2.2.1. Solid Solution Strengthening

Solid solution strengthening is a principal strengthening mechanism in which alloying atoms are incorporated into the crystal lattice of a base metal, creating lattice distortions that impede dislocation motion and thereby increase the YS and hardness of the material (often at the expense of ductility) [134,135]. The efficiency of solid solution strengthening is determined by several aspects, including solute concentration, atomic size mismatch, elastic modulus difference, and the nature of the solute-solvent interaction [136]. In general, solid solutions are classified into two main categories based on the relative atomic size of the solute element: substitutional and interstitial [137,138]. Both types can enhance the strength of a material, with the degree of strengthening typically increasing with solute content up to a certain limit, beyond which other phenomena (such as precipitation or embrittlement) may occur. This mechanism is extensively utilized in the majority of the Cu-based alloys to achieve an optimal balance between mechanical strength and ductility [137,139]. The increase in the athermal component of YS due to random solid solution strengthening can be approximated as:

$$\Delta\sigma \propto c^n \quad (1)$$

where  $c$  is the solute concentration and  $n$  typically ranges between 1/2 (Fleischer's model) and 2/3 (Labusch's model) depending on the strengthening model [140,141]. Among Cu alloys that form solid solutions, Cu-Ni alloys such as C70600 [142] and C71500 [143] are prominent examples. As illustrated in [Figure 5](#), Ni is completely soluble in Cu across the full composition range, resulting in significant solid solution strengthening [144]. Another important example is

brass, where Zn is substitutionally soluble in Cu within the  $\alpha$ -phase, with a maximum equilibrium solubility of approximately 30–35 at.% [145]. Aluminum bronzes are also worth mentioning, with Al soluble in Cu in the  $\alpha$ -phase up to roughly 9 wt.% under equilibrium conditions before secondary phases start to form [146]. Similarly, elements such as Si, Mn, Be, Sn, and Mg exhibit varying degrees of solubility in Cu. When present below their respective solubility limits, these elements form solid solutions, which can contribute to solid solution strengthening, with the magnitude depending on concentration and lattice interactions [147–149]. When their concentrations exceed the solubility limit, intermetallic phases are formed, which can promote second-phase hardening, as described in the following section. An example of the beneficial effect of solid solution strengthening can be seen in the study by Abe et al. [150], where they reported that incorporating 5.0 at.% In into Cu-In alloys significantly increased the YS and UTS from 130 MPa and 330 MPa, respectively, to 1280 MPa and 1340 MPa, primarily due to the pronounced atomic size mismatch of In and grain refinement induced by deformation twins.

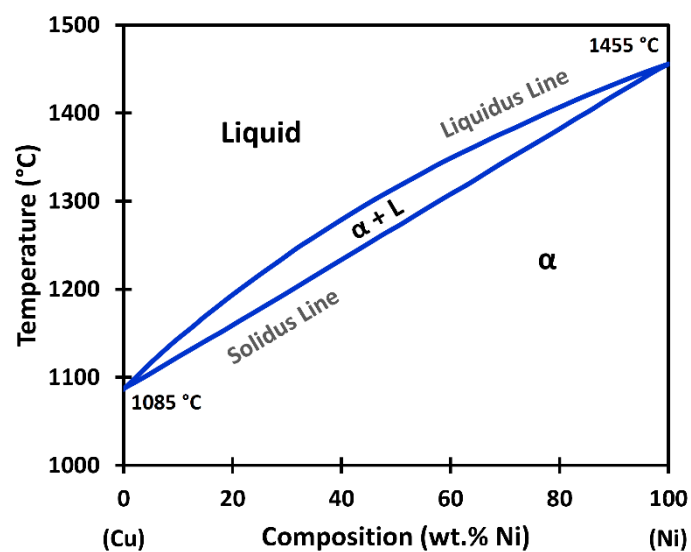


**Figure 5:** Binary phase diagrams of Cu-Ni system (Adapted with permission [144]. Copyright 2014, Elsevier).

### 2.2.2. Precipitation Hardening

Precipitation hardening, also known as age hardening, is another key strengthening mechanism used in a variety of Cu-based alloys to improve their mechanical properties by promoting the controlled formation of second-phase particles, typically intermetallic compounds [151–155]. Precipitation hardening generally involves three stages of solution treatment, quenching, and aging [156]. During solution treatment, the alloy is heated to a temperature at which the solute elements are fully dissolved in a single-phase solid solution.

Then, rapid quenching suppresses diffusion and creates a supersaturated solid solution at RT. Subsequent aging, either at RT (natural aging) or at an elevated temperature (artificial aging), enables controlled diffusion of solute atoms and results in the nucleation and growth of coherent or semi-coherent precipitates within the matrix. These second-phase particles impede dislocation motion and increase the strength of the alloy [156]. The size, distribution, and coherency of the precipitates critically influence the effectiveness of precipitation hardening, and uniformly dispersed and coherent particles provide the greatest resistance to dislocation motion, whereas coarse or incoherent precipitates are less effective [157,158]. The increase in yield strength due to precipitation hardening by non-shearable particles can be approximated by the Ashby-Orowan equation as:

$$\Delta\sigma_{Orowan} = M \frac{0.81\mu b}{(2\pi\sqrt{1-\nu})} \times \frac{\left(\ln\left(\frac{d_p}{b}\right)\right)}{(\lambda-d_p)} \quad (2)$$

$$\lambda = \frac{1}{2} d_p \sqrt{\frac{3\pi}{2f_v}} \quad (3)$$

where  $\mu$  is the shear modulus of the precipitated phase,  $b$  is the Burger vector of Cu matrix,  $\nu$  is the Poisson's ratio,  $d_p$  is the diameter of precipitates,  $\lambda$  is the space of precipitates, and  $f_v$  is the volume fraction of particles [159–161]. A clear example of the effective application of precipitation hardening to enhance the mechanical properties of Cu alloys is provided by the study of Markandeya et al. [162], who reported that precipitation hardening remarkably improved the performance of Cu-3Ti-1Cr and Cu-4Ti-1Cr (wt.%) alloys. Specifically, YS increased by 235% and 115%, UTS by 84% and 50%, and hardness rose from 118 to 277 HV10 and 224 to 326 HV10, respectively. These improvements were attributed to the formation of a metastable, ordered, and coherent  $\beta'$  (Cu<sub>4</sub>Ti) phase during peak aging at 450 °C. In a recent study, Dölling et al. [163] studied the hardness enhancement of low-alloy Cu-Sc alloys while maintaining electrical conductivity, examining the effects of aging (350–500 °C) and varying levels of prior cold deformation (25–75%). They detected that precipitation hardening was most effective below 450 °C, with cold working refining precipitates and significantly increasing peak hardness, particularly in Cu-0.3Sc (wt.%), which outperformed Cu-0.15Zr (wt.%). In addition, aging also improved electrical conductivity, and the favorable recrystallization behavior of Sc-containing alloys suggests their suitability for higher-temperature applications.

### 2.2.3. Dispersion Strengthening

Dispersion strengthening is another strengthening mechanism by which the mechanical properties of metals are enhanced through the uniform incorporation of fine, stable, non-soluble particles within the matrix [164–166]. In Cu alloys, oxide dispersion strengthening (ODS) is of particular significance [167–171]. Particularly, refractory oxides, such as yttrium oxide ( $\text{Y}_2\text{O}_3$ ) [171], aluminum oxide ( $\text{Al}_2\text{O}_3$ ) [172], or titanium oxide ( $\text{TiO}_2$ ) [173], are introduced into the Cu matrix, often via powder metallurgy and mechanical alloying. These oxides are thermodynamically stable and exhibit negligible solubility in Cu and retain their fine, uniform distribution even at elevated temperatures. The dispersed oxides impede dislocation motion through the Orowan bypass mechanism and, therefore, enhance the YS, high-temperature performance, and microstructural stability of the alloy [170,174]. Unlike precipitation hardening, dispersion hardening in Cu alloys does not rely on phase transformations or heat treatment but on the inherent stability and effective dispersion of oxide particles, which make it particularly valuable for applications which require both thermal and mechanical resilience. A simplified schematic illustration of dispersion strengthening is shown in [Figure 6](#).

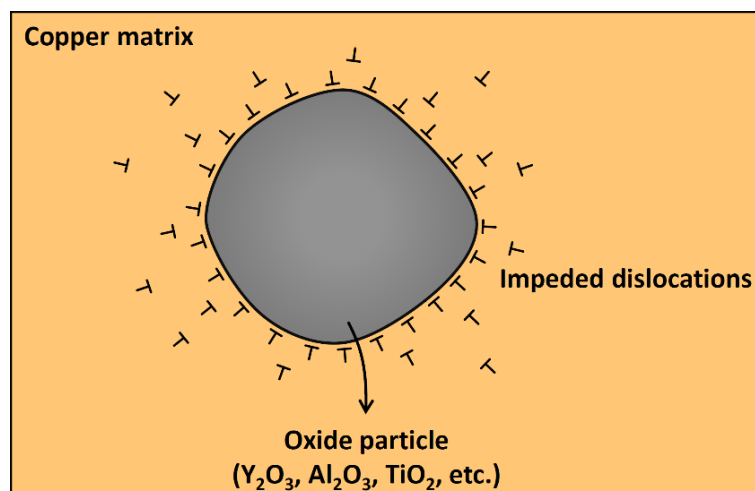


**Figure 6:** Simplified schematic illustration of dispersion strengthening.

Dispersion strengthening has been systematically investigated in multiple earlier works [175–177]. For example, Wang et al. [172] investigated ODS in a Cu alloy, where nanometer-sized  $\text{Al}_2\text{O}_3$  particles pinned dislocations and grain boundaries, refined sub-grains ( $\sim 600$  nm), and increased the YS to 442 MPa, while maintaining high strength during plastic deformation by stabilizing both grain size and dislocation density under severe strain. In another study, Sorkhe et al. [173] demonstrated that high-energy ball milling followed by conventional consolidation of Cu- $\text{TiO}_2$  composites, using 1–7 wt.%  $\text{TiO}_2$  and milling times up to 24 h,

refined Cu grains to nanoscale (<50 nm), significantly enhanced hardness (with a maximum of 105 HV achieved for Cu–5 wt.% TiO<sub>2</sub> milled for 12 h), and retained reasonable electrical conductivity up to ~3 wt.% TiO<sub>2</sub>, which made the alloys particularly suitable for wear-resistant welding electrodes. In a recent study, Gao et al. [178] prepared a Y<sub>2</sub>O<sub>3</sub> dispersion-strengthened Cu alloy via spray drying and observed that high-density Y<sub>2</sub>O<sub>3</sub> nanoparticles (~9 nm) were successfully incorporated into the Cu matrix, effectively refining grains and suppressing recrystallization. Moreover, the Y<sub>2</sub>O<sub>3</sub> nanoparticles significantly strengthened the Cu matrix by impeding dislocation and grain boundary motion, which resulted in a well-balanced combination of hardness (114 HV), UTS (326.5 MPa), elongation (8.5%), and electrical conductivity (85.3% IACS) in their fabricated Cu-2 wt.% Y<sub>2</sub>O<sub>3</sub> alloy.

#### **2.2.4. Deformation Strengthening**

Deformation strengthening, also referred to as strain hardening or work hardening, is another strengthening method in which the YS and hardness of metallic materials increase with the accumulation of plastic deformation [179–181]. In crystalline metallic systems, plastic deformation primarily occurs via the motion of dislocations (though other mechanisms such as twinning may contribute in specific cases) [182]. As deformation proceeds, dislocation density increases and leads to enhanced dislocation–dislocation interactions and the formation of tangled dislocation networks. These interactions impede further dislocation motion and result in an increase in the critical resolved shear stress required for continued plastic flow. The resulting microstructural state exhibits reduced ductility but significantly improved strength and hardness [182]. The extent of deformation strengthening depends on factors such as the amount of applied deformation, microstructure, stacking fault energy (SFE), and deformation temperature. At low to moderate temperatures, recovery processes are limited, which enables higher dislocation retention, whereas higher temperatures promote dynamic recovery (DRV) and dynamic recrystallization (DRX), reducing the strengthening effect [1]. The deformation process itself can be classified into two principal categories: conventional deformation and SPD processing, which are discussed separately in the following sections.

##### **2.2.4.1 Conventional Deformation**

Deformation strengthening via conventional deformation is a widely used metallurgical technique for enhancing the mechanical properties of metallic materials and can be applied

across a broad temperature range (from cryogenic to high temperatures) and strain rates [183]. In metallic materials, the primary mechanism for increasing YS and hardness through plastic deformation is the generation, multiplication, and interaction of dislocations within the crystal structure, although other mechanisms such as grain refinement, phase transformations, or twinning may also contribute depending on the material and deformation conditions [184–186]. It is well established that, during plastic deformation, dislocations are generated and glide along specific slip planes in response to the applied stress [187]. The accumulation and entanglement of these dislocations increases resistance to further dislocation motion, a phenomenon known as work hardening or strain hardening. The empirical relationship between true flow stress and true plastic strain during uniform plastic deformation in many metals is described by the Hollomon equation [188]:

$$\sigma = K \varepsilon^n \quad (4)$$

where  $\sigma$  is the true flow stress,  $\varepsilon$  is the true plastic strain,  $K$  is the strength coefficient, and  $n$  is the strain hardening exponent [189]. In addition to dislocation interactions, plastic deformation initially leads to the formation of subgrains bounded by LAGBs. These subgrains contribute to strengthening primarily through dislocation accumulation and subgrain boundary effects, rather than classical Hall-Petch-type grain boundary strengthening. With increasing strain and ongoing recovery or DRX, many LAGBs progressively rotate and transform into HAGBs, resulting in true grain refinement and activation of stronger Hall-Petch-type strengthening. The Hall-Petch relationship [190]:

$$\sigma_y = \sigma_0 + \frac{k}{\sqrt{d}} \quad (5)$$

where  $\sigma_y$  is the YS,  $\sigma_0$  is a material constant (intrinsic lattice friction stress),  $k$  is the Hall-Petch constant, and  $d$  is the grain size, describes how YS increases with decreasing grain size due to a higher density of HAGBs [191]. In some cases, cold deformation such as repetitive cold rolling passes induces a very high dislocation density in the material due to intense plastic strain. These dislocations interact with each other and with grain boundaries, creating internal stress fields that impede further dislocation motion. This strengthening effect is quantified by the Taylor relation [192]:

$$\sigma_d = \alpha M G b \sqrt{\rho} \quad (6)$$

where  $\sigma_d$  is the contribution to YS from dislocation strengthening,  $\alpha$  is a material-dependent constant (typically 0.2–0.5 for metals),  $M$  is the Taylor factor,  $G$  is the shear modulus,  $b$  is the Burgers vector, and  $\rho$  is the dislocation density [193].

For Cu-based alloys, as for many FCC metals, conventional deformation can influence other mechanical properties such as toughness [194–196] and fatigue resistance [197–199] depending on the deformation conditions and resulting microstructure. It should also be noted that, while moderate deformation can lead to a favorable balance between strength and ductility (particularly when combined with subsequent thermal treatments such as annealing), excessive cold working may reduce the ductility of metals [200]. To put an example, the study of Zhang et al. [201] can be mentioned, in which they fabricated a Cu-15Cr-0.2Ti (wt.%) alloy using a combination of casting, hot forging, and cold drawing, and investigated the influence of cold working degree and aging treatment on the ductility of this alloy. Their results revealed that the elongation to fracture decreases significantly with increasing drawing strain. However, aging for 1 h at 475 °C enhanced the ductility without compromising strength. In fact, such treatments allow controlled recovery and recrystallization, relieving internal stresses while preserving the work-hardened microstructure to optimize mechanical performance.

## **2.2.4.2. Severe Plastic Deformation**

### **2.2.4.2.1. Common SPD Techniques**

SPD processing is an advanced technique that enhances the mechanical properties of metallic materials by inducing extremely large plastic strains with limited changes to the overall dimensions of the workpiece, achieving high strength primarily through microstructural refinement and dislocation accumulation, although additional mechanisms may also contribute depending on the material and processing conditions [202,203]. Strengthening of metals during SPD processing is largely governed by grain refinement and dislocation accumulation, commonly described by the Hall-Petch relationship and Taylor hardening. However, additional mechanisms such as grain boundary-mediated plasticity, twinning, texture effects, and possible deviations from Hall-Petch behavior at very small grain sizes can also play significant roles depending on the material and processing conditions. Owing to the benefits of SPD processing over conventional deformation methods, a numerous of studies has been dedicated to the SPD

processing of pure Cu as well as a wide range of Cu alloys [204–220]. Among the various SPD methods, ECAP and high-pressure torsion (HPT) have been the most extensively studied for Cu and its alloys. As an example, the study of Mishra et al. [206] can be mentioned, in which they investigated the microstructure evolution and mechanical response of commercially pure Cu subjected to ECAP under different processing routes. They found that microstructural refinement depended on the processing route, with route B<sub>C</sub> proving to be the most effective. The deformation structure evolved from elongated dislocation cells to subgrains and finally to equiaxed grains with diameters of approximately 200–500 nm. In addition, the fraction of HAGBs increased with the number of passes. In another study, Khatibi et al. [207] investigated the influence of purity and process parameters on the fatigue behavior of ultrafine-grained (UFG, grain size < 1 μm) Cu produced by HPT. They found that the extremely fine microstructure of the HPT specimens and their enhanced tensile strength led to a considerable increase in fatigue life for HPT-processed samples, with the improvement attributed to the refined microstructure and the resulting high tensile strength.

Another important SPD method that is widely used on Cu alloys is accumulative roll bonding (ARB) [221]. This method is also capable of producing multi-laminated composites from Cu and other metals, such as Cu-Ag [222], Cu-Al [223], Cu-Ni [224], Cu-Fe [225], and Cu-Nb [226] systems. These multilayered composite structures can simultaneously harness the combined benefits of each constituent material [227–229]. As an example, the study by You et al. [222] can be mentioned, in which bulk Cu/Ag multilayered composites with high strength, excellent electrical conductivity, and outstanding thermal stability were fabricated through a process involving repeated cycles of cutting, cleaning, stacking, and roll bonding for up to 15 passes. In a very recent study, Safahi et al. [230] fabricated a Cu-W composite using the ARB method. High-purity Cu sheets were first laser-drilled to create periodic holes, which were then filled with W wires to form an ordered reinforcement pattern. The wire-reinforced sheets were stacked and subjected to two ARB cycles at RT. This process produced a well-bonded Cu-W interface with no detectable porosity. Their resulting composite exhibited a tensile strength of 328.5 MPa, an increase in hardness from 128.6 HV to 188.3 HV, and maintained a satisfactory level of electrical conductivity. In the ARB process, powders can also be applied between the sheets, which results in additional benefits that contribute to the improvement of mechanical properties [231–233]. A simplified schematic illustration of the ECAP, HPT, and ARB processes is presented in [Figure 7](#), while two particularly important SPD methods employed in

this study, MaxStrain processing using the Gleeble thermomechanical simulator and KoBo extrusion, are described in detail in the following sections.

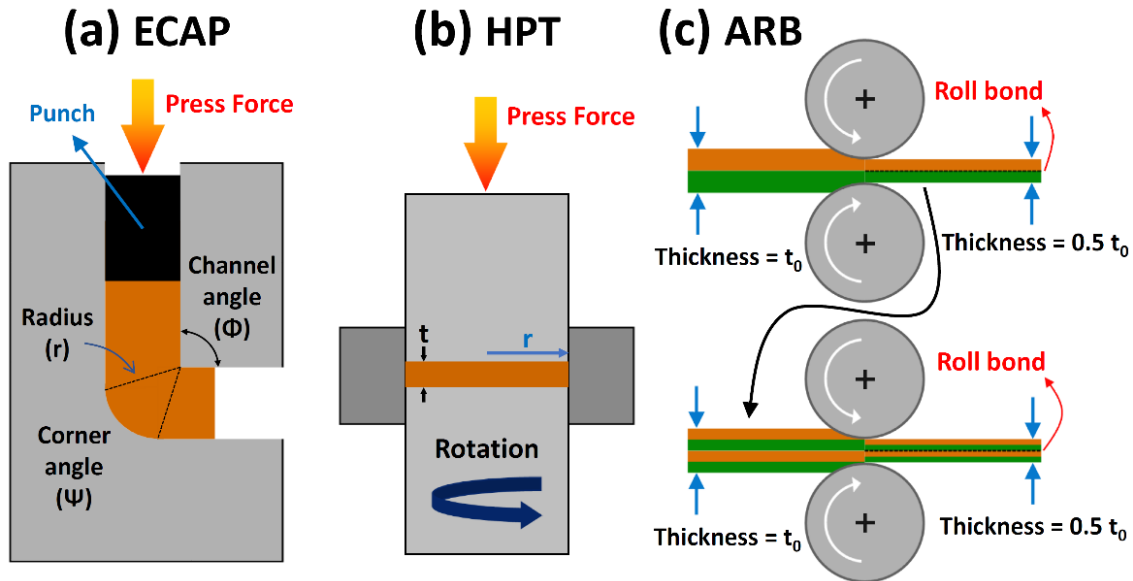


**Figure 7:** Simplified schematic illustration of (a) ECAP, (b) HPT, and (c) ARB processes.

#### 2.2.4.2.2 MaxStrain Process

MaxStrain processing, which is performed using the MaxStrain module of the Gleeble thermomechanical simulator, is a notable SPD technique that enables the systematic investigation of a wide range of deformation parameters and facilitates the study of microstructural evolution under diverse thermomechanical processing conditions [234–237]. In simple words, MaxStrain processing involves compressing the central region of a square cuboid sample with two opposing anvils with precise control over process variables such as force, strike, anvil speed, and temperature [3]. After the initial compression, the sample is rotated  $90^\circ$  along its longitudinal axis and compressed again in a perpendicular direction, and this process is repeated until the desired level of accumulated strain is achieved [238]. During deformation in MaxStrain processing, repeated multi-axial compression of the sample allows for the accumulation of the desired total equivalent plastic strain up to the ductility or geometric stability limit of the material. In a single compression operation, the magnitude of the equivalent plastic strain can range from minimal values up to values as high as 0.6, and the simulator can provide the implementation of arbitrary holding times for the sample both before and after each deformation.

Figure 8(a) shows the entire Gleeble thermomechanical simulator used for the experiments in the current PhD program, and Figure 8(b) shows the MaxStrain module within it with the

various components of the apparatus labeled. During the MaxStrain processing, the sample can be either constrained at its two side ends or left to freely expand during the deformation (unconstrained) [239]. Consequently, the material flows in the transverse direction when the sample is deformed under constrained conditions, whereas it predominantly flows in the longitudinal direction under unconstrained conditions. As a result, the length of the constrained sample remains approximately unchanged after the deformation, while the length of the unconstrained sample increases [239]. As an illustrative example, Figures 8(c) and 8(d) show the deformed samples in the constrained and unconstrained modes, respectively, following MaxStrain deformation.

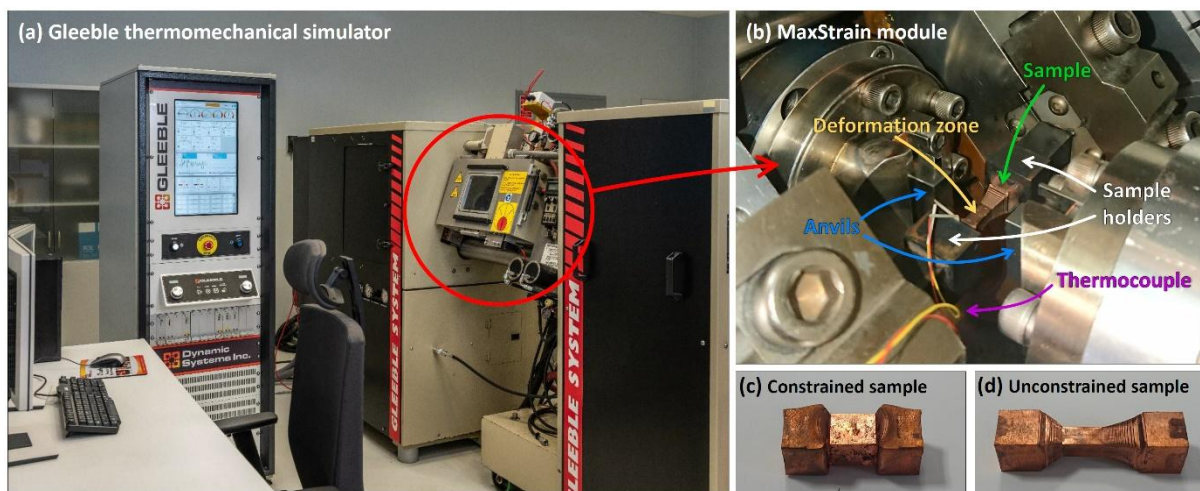


**Figure 8:** (a) The Gleeble thermomechanical simulator; (b) the MaxStrain module; MaxStrain-processed samples in (c) constrained and (d) unconstrained conditions.

Nevertheless, like all SPD processes, the MaxStrain process has some limitations that must be considered when interpreting results. For instance, the small sample size used in physical simulators may not fully represent bulk material behavior due to thermal gradients and grain-scale volume effects. Boundary effects, specifically interfacial friction between the sample and anvils, typically induce non-uniform strain distribution (barrelling). Furthermore, while high strain rates and temperatures are attainable, maintaining isothermal and homogeneous deformation under extreme conditions remains a challenge. Finally, MaxStrain utilizes discrete multi-axial loading that may not fully replicate the continuous shear components found in industrial processes like rolling or extrusion.

### 2.2.4.2.3 KoBo Extrusion

Among SPD methods with potential for large-scale industrial application, extrusion with oscillatory reversible rotation of the die, commonly referred to as KoBo extrusion, emerges as an efficient technique [240]. This process integrates the primary deformation associated with forward extrusion with an additional shear deformation induced by oscillatory die rotation at the end of the extrusion container [241–246]. As a result, the material undergoes plastic deformation through conventional forward extrusion combined with cyclically varying shear deformation arising from changes in the deformation path [247–249]. This method combines high extrusion ratios with reduced forming forces, facilitates the processing of hard-to-deform alloys at lower temperatures, and induces intensive microstructural refinement, which leads to superior mechanical and functional properties [250–252]. Figure 9 presents a simplified schematic illustration of the various components of the KoBo extrusion equipment. During the KoBo extrusion process, several process variables, such as the punch speed, shape and internal diameter of the container and the reversibly rotating die, oscillation frequency, and oscillation angle, affect the equivalent strain in the extruded component [245]. Any modification in these variables alters the equivalent strain inside the extruded billet, and consequently, affects the microstructure and properties of the final product [253]. For these reasons, the KoBo extrusion method can be regarded as an effective and reliable approach for practical applications, with considerable potential for further development and successful industrialization.

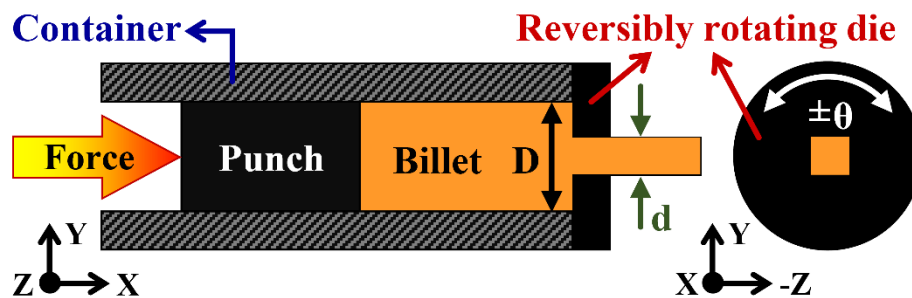


**Figure 9:** Simplified schematic illustration of various components of the KoBo extrusion equipment.

KoBo extrusion can also be applied to achieve effective consolidation of dispersed metal and alloy chips, enabling the production of long products under cold forming conditions [240,254,255]. During the process, the combination of high compressive stresses and cyclic shear deformation promotes fragmentation of surface oxides and enhances intimate contact between chip fragments. This facilitates bonding across their interfaces, resulting in a durable metallurgical connection and a consolidated structure that is typically homogeneous.

### 2.2.5. Grain Refinement

In most polycrystalline metallic materials under typical conditions, grain refinement is among the most effective strategies for enhancing mechanical properties (particularly YS) and is largely interpreted through the Hall-Petch relationship [256–258]. As noted earlier, the Hall-Petch principle explains that finer grains increase boundary barriers to dislocation motion and result in greater strength and hardness. In metallic materials, grain refinement can be achieved through conventional thermomechanical processing, which typically delivers grain sizes in the micrometer range, or through advanced SPD techniques, which can produce UFG or nanocrystalline (grain size < 100 nm) grain structure, depending on the material as well as the processing parameters [259].

Beyond strengthening, grain refinement can also influence other material properties, either beneficially or adversely. In most conventional polycrystalline metals, grain refinement generally improves high-cycle fatigue resistance by increasing strength and delaying crack initiation. In contrast, severely deformed UFG and nanocrystalline metals often show limited or even reduced fatigue resistance due to restricted cyclic plasticity [197]. Moreover, grain refinement typically does not enhance fracture toughness in ductile metallic alloys; in many systems it can even reduce it, as the associated strength increase often outweighs the potential benefits related to delayed crack initiation or modest crack-path deflection [260]. In certain cases, smaller grains may also improve corrosion behavior, depending on the material and environment [261]. For instance, Miyamoto et al. [262] reported that when the grain size is reduced in UFG Cu,  $\text{Cu}^{2+}$  ions dissolved at grain boundaries can easily flow in the close vicinity of the surface of grain interiors, interrupting the supply and reduction reaction of  $\text{O}_2$ . This process leads to a lower corrosion rate in UFG Cu.

Nonetheless, UFG or nanocrystalline grains may exhibit reduced thermal stability due to their high grain boundary energy and large boundary fraction, which can promote grain growth if grain boundary mobility is not effectively suppressed [263–265]. Additionally, fine grains may compromise ductility in some ductile metals [266]. Despite these trade-offs, grain refinement remains a reliable and widely applied method for tailoring the performance of metallic materials across diverse industrial applications.

## 2.3. Electrical Conductivity

Electrical conductivity ( $\sigma$ ) is a fundamental property of materials that quantifies their ability to conduct electric current. It is defined as the reciprocal of electrical resistivity ( $\rho$ ) [4]:

$$\sigma = \frac{1}{\rho} \quad (7)$$

where  $\rho$  is measured in ohm-meters ( $\Omega\cdot\text{m}$ ) and  $\sigma$  in Siemens per meter (S/m). Metals exhibit high electrical conductivity due to delocalized electrons in their conduction band that move freely under an electric field [267]. In metals, electrical resistivity typically rises with increasing temperature due to enhanced electron-phonon scattering [268]. Consequently, electrical conductivity decreases with increasing temperature, approximately following the inverse of a linear resistivity–temperature relationship at high temperatures [269]. At RT, pure Cu is the second most electrically conductive metal after Ag, with its high conductivity arising primarily from high electron mobility in a delocalized 4s-derived band crossing the Fermi level, and further influenced by hybridization with nearly filled 3d states that shape the Fermi surface and scattering behavior [270,271]. It is also noteworthy to mention that although Ag has slightly higher conductivity ( $\sigma = 63 \text{ MS/m}$  [272]), Cu is preferred in industry because it offers the best trade-off between conductivity, cost-effectiveness, and ease of fabrication [273].

Here, an important consideration regarding the solid solution strengthening of Cu alloys is the relationship between the concentration of solute atoms and the heating of the alloy during service. The heating of a conductor, such as a Cu wire, is primarily due to Joule heating, expressed as [274]:

$$Q = I^2 R t \quad (8)$$

where  $I$  is the electrical current,  $R$  the electrical resistance, and  $t$  the duration of current flow. According to this relationship, any increase in resistance results in greater heat generation for the same applied current [275]. In solid solution Cu alloys, solute atoms (such as Mg) in the Cu lattice scatter conduction electrons, which increases the electrical resistivity of the material. Consequently, as the concentration of such solute atoms increases (even within the solubility limit), the resistivity of the alloy can rise, which leads to slightly higher heating during service. This effect is particularly important in high-performance applications where excessive heat generation can negatively impact performance, reliability, or efficiency. Therefore, to

minimize heating while still benefiting from solid solution strengthening, it is generally preferable to use alloys that have the least detrimental effect on the electrical conductivity (Figure 3) and to apply the lowest effective concentration of solute atoms necessary to achieve the desired mechanical properties.

The simultaneous achievement of high strength and favorable electrical conductivity still represents a notable challenge in the development of Cu-based alloys [2]. This difficulty arises from the intrinsic interplay between mechanical strengthening mechanisms and electron transport [26]. Most strengthening approaches, such as solid solution alloying, grain refinement, work hardening, and precipitation, introduce crystal defects, such as vacancies, solute atoms, dislocations, grain boundaries, and second-phase particles into the crystal lattice, which act as scattering centers for conduction electrons [276,277]. The increased frequency of electron scattering events directly elevates the electrical resistivity of the alloy or, in other words, reduces its conductivity [278–280]. This trade-off is particularly critical in functional materials such as Cu alloys, where the simultaneous enhancement of mechanical properties and high electrical conductivity is essential for practical applications in areas such as power transmission, electronic devices, and advanced functional systems. Consequently, the design of next-generation Cu alloys requires strategies that dissociate mechanical strength from electron scattering.

## 2.4. Review of Previous Research on Cu-Mg and Cu-Mg-Ca Alloys

The fundamental principles discussed in the preceding sections pave the way to the present literature review, which systematically overviews the previous studies that were performed aiming at improving the mechanical properties of Cu-Mg and Cu-Mg-Ca alloys while minimizing the degradation of the electrical conductivity. This literature review provides the foundation for discussing the approaches and contributions of the present PhD work. Before proceeding with the discussion, it is worth noting that some studies have also investigated the synthesis and properties of bulk metallic glasses in the ternary Cu-Mg-Ca system [281–283].

As a representative example from previous studies on ameliorating the mechanical properties and electrical conductivity of Cu-Mg-Ca alloys, the study of Li et al. [37] can be mentioned in which they fabricated an economical Cu-0.29Mg-0.21Ca (wt.%) alloy by non-vacuum melting and casting, followed by thermomechanical processing. They reported that cold deformation significantly refined the initial grain structure into fine grains containing

abundant substructures, with an average size of approximately 400 nm. After homogenization at 780 °C for 1 h, subsequent cold rolling with an 80% reduction, and annealing at 200 °C for 1 h, the alloy exhibited outstanding comprehensive properties. Specifically, it achieved a UTS of 545 MPa, a YS of 526 MPa, an elongation of 3.6%, and an electrical conductivity of 71.79% IACS (Figure 10). The high electrical conductivity was attributed to the addition of Ca, which purified the Cu-Mg matrix by reacting with impurity elements while causing minimal electron scattering. The high strength was mainly ascribed to the combined effects of dispersion strengthening, solid solution strengthening, substructure strengthening, and dislocation strengthening, with substructure strengthening playing a dominant role.

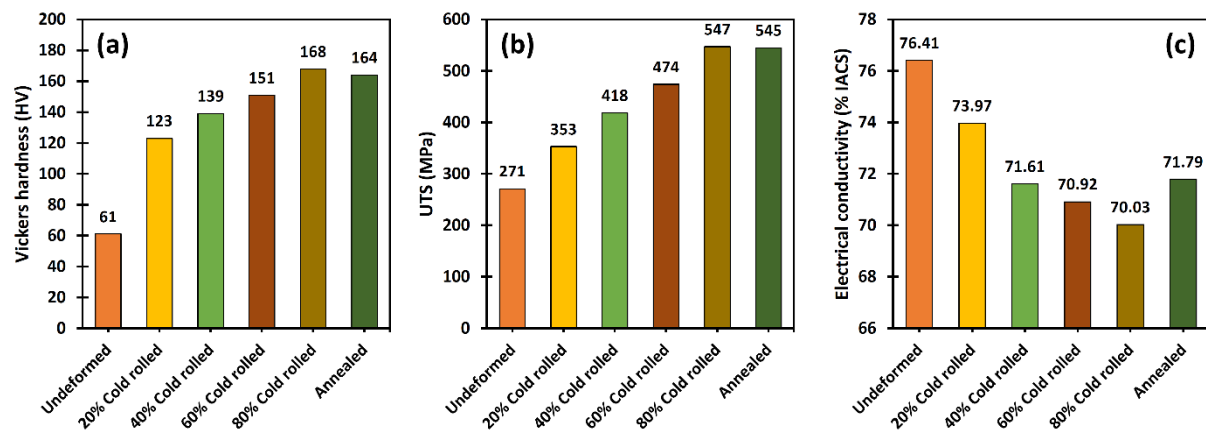


**Figure 10:** (a) Vickers hardness, (b) UTS, and (c) electrical conductivity of Cu-0.29Mg-0.21Ca (wt.%) alloy in different states (Adapted with permission [37]. Copyright 2017, Elsevier).

In another study, Ma et al. [42] fabricated Cu-0.43Mg and Cu-0.41Mg-0.06Ca (wt.%) alloys by melting and casting (in an air atmosphere), followed by ECAP processing at RT for up to 12 passes using route B<sub>C</sub>. They observed that the initially coarse grains were significantly refined after 12 ECAP passes, with the average grain size reduced to approximately 0.57 μm. At the same time, the coarse Cu<sub>5</sub>Ca phase was fragmented into particles with sizes of about 1–3 μm. As shown in Figure 11, after processing, the Cu-Mg-Ca alloy exhibited excellent comprehensive properties, including a YS of 619 MPa, an electrical conductivity of 67.86% IACS, and an elongation to fracture (EL) of 10.4%. When compared with a Cu-Mg alloy processed under the same conditions, the Cu-Mg-Ca alloy showed an increase in YS of 30 MPa and an improvement in electrical conductivity of 0.63% IACS, while maintaining nearly the same EL. These enhanced properties were attributed to the combined effects of ECAP processing and Ca addition. They also observed that Cu-Mg-Ca alloy possessed a higher hardness across almost all ECAP passes compared to the Cu-Mg alloy. They reported that the ECAP process contributed most significantly to the strength improvement, accounting for more

than 80% of the final YS after 12 passes. In addition, Ca addition promoted grain refinement, improved work-hardening behavior, and enhanced electrical conductivity.

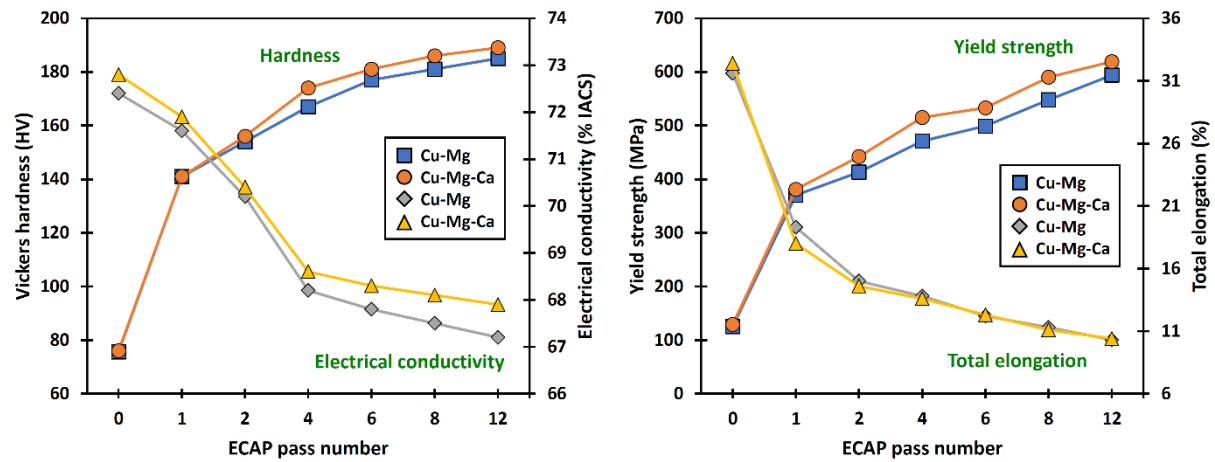


**Figure 11:** Hardness, electrical conductivity, YS, and EL for Cu-0.43Mg and Cu-0.41Mg-0.06Ca (wt.%) alloys as a function of ECAP passes (Adapted with permission [42]. Copyright 2019, Elsevier).

In a similar investigation, Ma et al. [28] subjected the same ternary Cu-0.41Mg-0.06Ca (wt.%) alloy to the ECAP process for up to 12 passes via route B<sub>C</sub> at RT and measured the hardness across the entire transverse planes. After a single ECAP pass, the hardness in the central region was found to be 9.4% higher than that in the bottom region (Figure 12). The microstructure in the central region consisted of elongated grains, whereas the bottom region exhibited slightly distorted grains. In addition, the crystallographic texture in the central region followed the ideal ECAP texture orientations, while the texture in the bottom region showed a clear deviation from any ideal ECAP texture orientation. With increasing numbers of ECAP passes, the differences in hardness and microstructure between the two regions gradually diminished, and the deviation of the crystallographic texture in the bottom region progressively decreased. After 8 ECAP passes, both regions exhibited identical hardness, similar UFG microstructures, and comparable crystallographic textures. The authors attributed the initial regional differences to non-uniform strain arising from friction between the sample and the die wall rather than from the corner gap, and the gradual weakening of these differences to the strain-hardening behavior of the alloy.

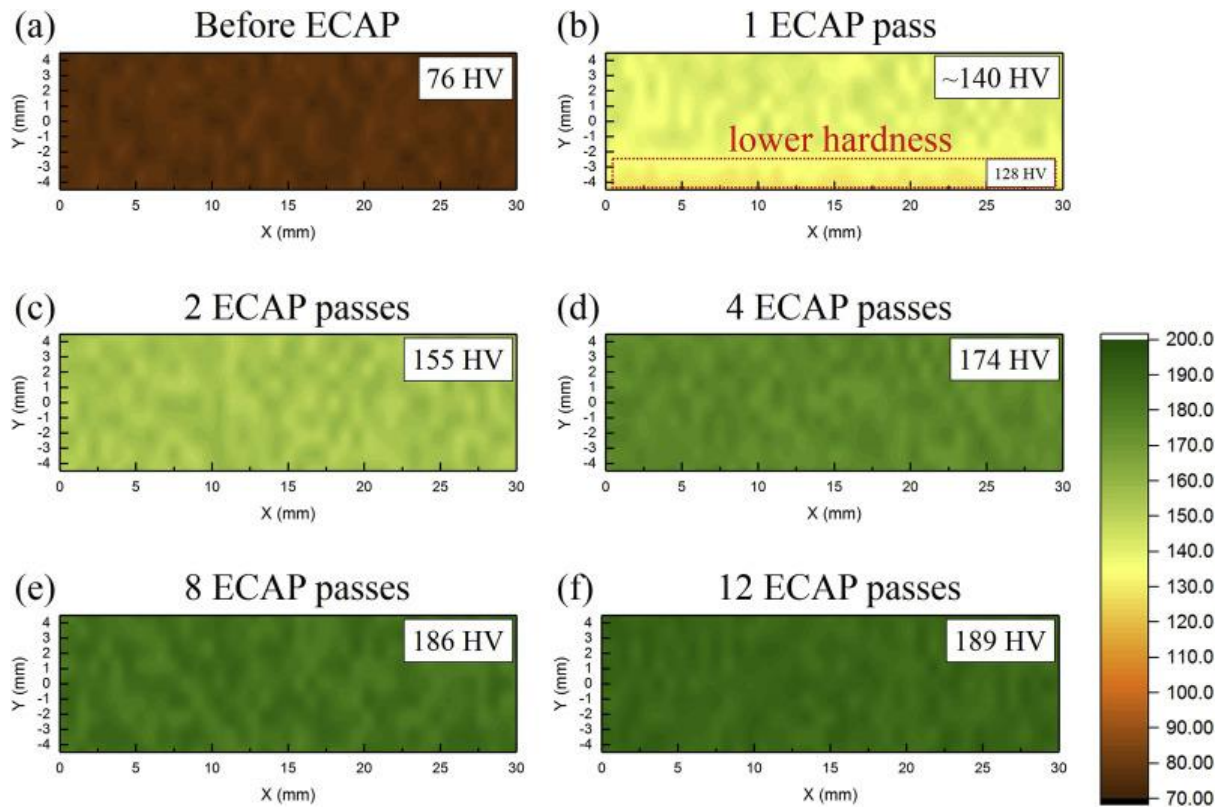


**Figure 12:** Color-coded contour maps illustrating the hardness distribution across the transverse planes of the Cu-0.41Mg-0.06Ca (wt.%) alloy: (a) prior to the ECAP process, and after (b) 1, (c) 2, (d) 4, (e) 8, and (f) 12 ECAP passes (Reproduced with permission [28]. Copyright 2020, Elsevier).

In another noteworthy study, Ma et al. [32] applied ECAP for up to 16 passes to a Cu-0.2Mg (wt.%) alloy at temperatures of 200 °C and 400 °C. Their results (Figure 13) showed that samples processed at 200 °C consistently exhibited higher microhardness than those deformed at 400 °C across all ECAP passes due to more obvious strain hardening and more efficient grain refinement at the lower temperature. In contrast, the higher processing temperature promoted faster grain recovery and recrystallization, which led to coarser grains and a lower hardness increment. However, ECAP conducted at 200 °C led to lower electrical conductivity compared to processing at 400 °C, caused by the higher density of grain boundaries (including LAGBs) and increased dislocation multiplication that occurred during deformation at the lower temperature, which enhanced electron scattering and thereby increased electrical resistivity, whereas greater DRV occurred at 400 °C.

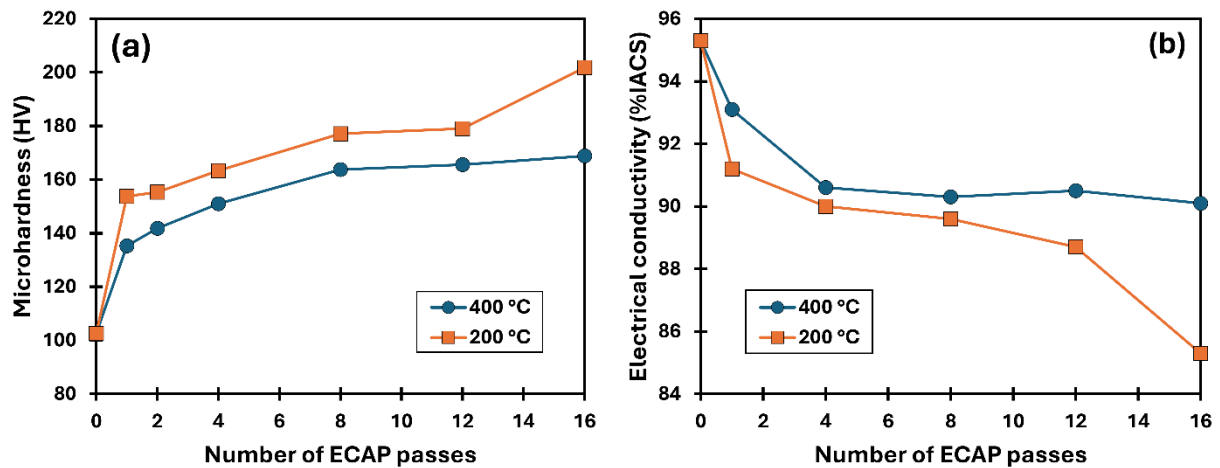


**Figure 13:** Variation in (a) microhardness and (b) electrical conductivity of a Cu-0.2Mg (wt.%) alloy at 200 °C and 400 °C vs. the number of ECAP passes (Adapted under the terms of CC BY 4.0 license [32]. Copyright 2014, MDPI).

Regarding the influence of Mg content, the study by Rodríguez-Calvillo et al. [33] provides a useful example. They investigated the microstructural evolution and strengthening behavior of two Cu-Mg alloys containing 0.2 and 0.5 wt.% Mg, processed by ECAP at RT for up to 16 passes following route BC. Significant grain refinement was observed after the second ECAP pass and continued with increasing passes. After 16 passes, average grain sizes of approximately 0.31  $\mu\text{m}$  and 0.24  $\mu\text{m}$  were achieved for the Cu-0.2Mg and Cu-0.5Mg alloys, respectively, mainly as a result of continuous DRX (CDRX). The recrystallized fraction remained below 30%, and the resulting recrystallized grain sizes were about 0.22  $\mu\text{m}$  for Cu-0.2Mg and 0.18  $\mu\text{m}$  for Cu-0.5Mg. ECAP processing also led to a marked improvement in mechanical properties. After 16 passes, the UTS reached about 570 MPa for the Cu-0.2Mg alloy and 750 MPa for the Cu-0.5Mg alloy, and the Cu-0.5Mg alloy consistently exhibited better tensile properties compared to the Cu-0.2Mg alloy across all ECAP passes. The strengthening was mainly attributed to grain and subgrain refinement and solid solution strengthening, with the subgrain structure providing the dominant contribution, while deformation was primarily accommodated by dislocation glide rather than twinning.

Regarding Cu-Mg alloys with high Mg content, a recent study by Strzpek et al. [124] provides valuable insight. In their work, cast rods of Cu-Mg alloys containing 2, 2.4, 2.8, 3, 3.2, 3.6, and 4 wt.% Mg were produced using a continuous casting process. The authors reported that solution treatment at 700 °C for 10 h led to the dissolution of Mg-rich phases in alloys containing up to 3 wt.% Mg. In contrast, alloys with higher Mg contents retained Mg-rich phases, which fragmented during subsequent cold upsetting when the materials were in the cold-drawn condition. This behavior suggests limited deformability in two-phase Cu-Mg

alloys, even after prior solution treatment. Furthermore, after solution treatment, all studied Cu-Mg alloys could undergo cold upsetting to a strain of 50% without the formation of cracks. Based on these results, the authors concluded that Cu-Mg alloys can be successfully subjected to cold deformation after solution treatment or, alternatively, to hot deformation in the as-cast state. In a comparable study, Strzpek et al. [120] reported that the above-mentioned Cu-Mg alloys in the as-cast state exhibited mechanical properties comparable to or higher than those of commercial brass or bronze alloys, while also having lower density and significantly higher electrical conductivity. At the end, it should be noted that several other comparable studies have also investigated Cu-Mg and Cu-Mg-Ca alloys; however, to maintain conciseness, only the most important examples are included in this section.

## 2.5. Manufacturing Technologies for Cu-Mg-based Alloys

As mentioned earlier, Cu-Mg alloys are widely used in industry due to their combination of good mechanical properties, reasonable electrical conductivity, and corrosion resistance [284]. Alloys with up to 0.7 wt.% Mg are commonly produced industrially, while higher Mg contents (such as 2.8–3.2 wt.%) are largely experimental and studied to assess their mechanical behavior and fatigue strength [285,286]. In this regard, Strzpek et al. [286] have mentioned that the manufacturing technology used for wire drawing has a critical influence on the final properties of Cu-2.8Mg and Cu-3.2Mg (wt.%) wires. In their study, wires with 2.8–3.2 wt.% Mg were produced from 14 mm cast rods down to a final diameter of 3 mm using two different processes: a laboratory chain drawbench with a constant low drawing speed of approximately 0.25 m/s and an industrial drum-type drawing machine with significantly higher drawing speeds reaching up to 2.5 m/s. Both technologies successfully produced 3 mm wires regardless of Mg concentration. However, the manufacturing method strongly affected surface quality, defect formation, tensile strength, and fatigue performance of the fabricated alloys. Cu-2.8Mg wires showed smooth surfaces with both laboratory and industrial drawing processes. In contrast, Cu-3.2Mg wires were highly sensitive to the manufacturing technology. Industrially drawn Cu-3.2Mg wires developed severe delamination and brittle fractures during unwinding due to high drawing speeds and direct coiling, while laboratory-drawn ones showed only minor surficial damage but remained straight. Cu-2.8Mg wires achieved higher UTS than Cu-3.2Mg wires, with industrially drawn Cu-3.2Mg exhibiting the lowest values due to its more developed two-phase microstructure and process-induced defects. Fatigue testing was possible only on Cu-2.8Mg wires. Laboratory-drawn Cu-2.8Mg wires showed superior fatigue strength, especially

above 100 MPa; below 55 MPa, industrially drawn wires performed better. Laboratory fractures typically initiated centrally with parallel beach marks, whereas industrial ones showed surface-initiated cracks and larger residual fracture zones due to residual stresses from high-speed drawing and coiling. Overall, they noted that the manufacturing technology, specifically drawing speed, strain rate, coiling method, and equipment type, plays a decisive role in determining the formation of surface and internal defects, tensile strength, and fatigue resistance of Cu-Mg wires. They concluded that proper control of these technological parameters is essential to achieve the required structural integrity and reliable performance under cyclic loads.

## 2.6. Summary of Literature Review

In the preceding sections, it was demonstrated that Cu has played a pivotal role in technological development due to its favorable combination of strength, ductility, and excellent electrical and thermal conductivity; however, its relatively low strength can be significantly enhanced through alloying. Cu alloys, including brasses and bronzes, as well as specialized systems such as Cu-Ni, Cu-Al, and Cu-Be, provide a wide range of mechanical and functional properties for diverse engineering applications. Particular attention was given to Cu-Mg alloys, which offer a desirable balance between strength and electrical conductivity, especially at Mg contents above 0.1 wt.%, where strengthening results primarily from solid solution effects with the formation of Cu<sub>2</sub>Mg precipitates becoming relevant at higher Mg contents. Nevertheless, increasing Mg content can lead to reduced conductivity due to enhanced electron scattering. Incorporation of Ca in Cu-Mg-Ca alloys further improves strength and thermal stability through the formation of Cu<sub>5</sub>Ca second-phase particles, while maintaining relatively high conductivity. It was also highlighted that the mechanical properties of Cu alloys can be tailored through established strengthening mechanisms, including solid solution strengthening, precipitation strengthening, second-phase hardening, and grain refinement, particularly when combined with SPD techniques that promote UFG microstructures. Overall, previous studies indicate that careful control of composition and processing routes enables Cu-Mg-based alloys to achieve a favorable balance between strength, ductility, and conductivity, although simultaneously maximizing these properties remains a key challenge.

Building upon the identified research gap and the scientific relevance of this topic, and following a comprehensive review of the existing literature, three complementary and systematically designed approaches were adopted to fulfill the objectives of this PhD program.

Two approaches were specifically developed to address limitations in current knowledge and to provide new insights into the design of HSHC binary Cu-Mg and ternary Cu-Mg-Ca alloys. Particular emphasis is placed on achieving an optimized balance between strength and ductility without significantly compromising electrical conductivity. By integrating alloy design with advanced processing strategies, and guided by the principles of physical metallurgy and microstructural engineering, this work aimed to contribute to a deeper understanding of the underlying structure–property relationships in these systems. The third approach aimed at the fabrication of a high-performance Cu/Mg composite through careful control of processing parameters and composite architecture. Each approach is presented in detail in the subsequent sections, together with a comprehensive discussion and interpretation of the corresponding results and outcomes.

### **3. This Investigation**

#### **3.1. Approach 1: Ca Addition, Heat Treatment, and MaxStrain Processing**

This approach focused on enhancing the mechanical properties of a solid solution Cu-0.7Mg (wt.%) alloy through the addition of a precisely controlled amount of Ca, the application of industrially relevant heat treatments of relatively short duration, and SPD processing using the MaxStrain module of the Gleeble thermomechanical simulator. The primary objective of this approach was to systematically investigate the synergistic effects of Ca addition, heat treatment, and SPD processing on microstructural evolution and the resulting mechanical properties and electrical conductivity. Therefore, particular attention was given to identifying processing conditions that enable an optimal balance between strength, ductility, and electrical conductivity. In order to provide a clear overview of the experimental design and sequence of processing steps, the overall workflow adopted in this approach is schematically illustrated in the flowchart presented in [Figure 14](#). The following sections present a comprehensive description of the experimental procedures, followed by a detailed analysis and discussion of the corresponding results and their implications.

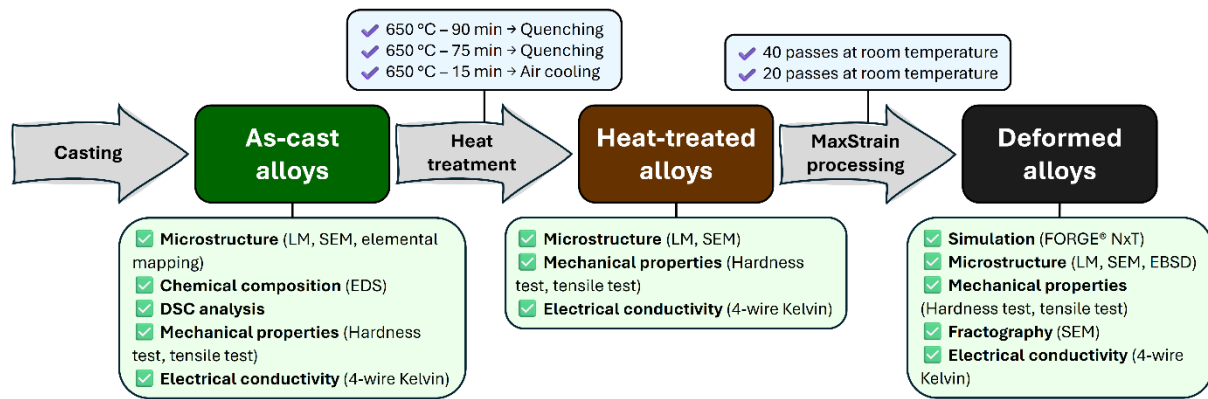


**Figure 14:** Schematic representation of the overall workflow employed in Approach 1, presented in a simplified flowchart format.

### 3.1.1. Methodology

#### 3.1.1.1. Materials Fabrication

The investigated alloys were produced by melting high-purity Cu, Mg, and Ca elements (99.99 wt.%) in a graphite crucible coated with boron nitride (BN) using an induction furnace. To prevent oxidation during casting and subsequent heat treatments, a continuous flow of argon gas was maintained throughout all steps. The melts were cast into preheated cylindrical steel molds with an internal diameter of 43 mm and a length of 200 mm, followed by air cooling to RT. The as-cast ingots were then machined to a diameter of 39.5 mm to remove surface defects resulting from casting. Subsequently, differential scanning calorimetry (DSC) was performed on the as-cast alloys, and based on the obtained results, heat treatments were applied (discussed in detail in their relevant sections). Finally, in accordance with the requirements of the MaxStrain process, specimens with dimensions of  $10 \times 10 \times 27.2 \text{ mm}^3$  were cut from the longitudinal direction of the billets.

#### 3.1.1.2. Chemical Composition Analysis

After casting, the concentrations of the constituent elements in the alloys were determined using energy-dispersive X-ray spectroscopy (EDS). The quantitative EDS results for the investigated alloys are summarized in [Table 4](#). In order to ensure the accuracy and reliability of the compositional measurements, all analyses were performed using the PROZA correction method, which compensates for matrix effects related to atomic number, X-ray absorption, and fluorescence. In addition, the EDS system was calibrated and validated using certified reference standards with high purity, including Cu and Mg (both 99.999 wt.%) standards obtained from Astimex Standards Ltd. (catalog no. METM25-44), as well as a Ca reference

derived from CaSO<sub>4</sub> (catalog no. MINM25-53). The combined use of matrix corrections and calibration with certified reference standards significantly minimized systematic errors in quantitative EDS analysis and improved measurement precision, consistency, and the reliability and reproducibility of the compositional data for the studied alloys.

**Table 4:** Elemental quantification by EDS in as-cast binary and ternary alloys.

| Element              | Concentration (wt.%) |             |             |            |
|----------------------|----------------------|-------------|-------------|------------|
|                      | Cu                   | Mg          | Ca          | Impurities |
| <b>Binary Alloy</b>  | 99.33 ± 0.05         | 0.67 ± 0.05 | —           | < 0.01     |
| <b>Ternary Alloy</b> | 99.17 ± 0.05         | 0.71 ± 0.06 | 0.12 ± 0.04 | < 0.01     |

### 3.1.1.3. Differential Scanning Calorimetry

Given the novelty of the developed alloys and the lack of available data on their thermal behavior and phase transformation characteristics during heating and cooling, DSC analysis was employed to systematically characterize their thermal transition behavior. This technique enables the identification of characteristic thermal events, such as phase transformations and melting reactions, and provides insight into the stability and transformation behavior of the alloys over the investigated temperature range [287]. For this purpose, rectangular specimens with dimensions of  $2 \times 2 \times 8 \text{ mm}^3$  were machined from each alloy and subjected to DSC analysis to determine their melting and critical phase transformation temperatures. The measurements were performed using a Setaram Setsys 16/18 DSC instrument, in which each specimen and an empty reference crucible were placed inside a thermoelectric disc furnace. The samples were heated linearly to 1200 °C at a rate of 10 °C/min under a constant argon atmosphere and subsequently cooled to RT at the same rate. Throughout the thermal cycles, temperature and heat flow signals were continuously recorded and analyzed using Setsoft software. The resulting DSC thermograms were then used to identify characteristic thermal events and determine the critical transition temperatures of the investigated alloys.

### 3.1.1.4. MaxStrain Processing

In this approach, the samples were subjected to either 20 or 40 passes of deformation in an unconstrained condition, with molybdenum disulfide (MoS<sub>2</sub>) used as a lubricant at the two side ends of the sample holders to enable sample elongation in the longitudinal direction with less friction. A thermocouple was also inserted at the side ends of the samples to constantly record their temperature throughout the deformation process. However, the average temperature

recorded during the deformation process was approximately 32.5 °C, with the highest temperature roughly reaching 37 °C. In fact, the high thermal conductivity of the alloys caused rapid transfer of the heat generated by each deformation pass to the sample holders and the rest of the apparatus and prevented heat accumulation and the subsequent temperature increase within the samples. Therefore, considering the fact that the melting point of pure Cu is about 1085 °C [288], the small temperature variations observed during MaxStrain processing are negligible under the specific experimental conditions employed in this study and are not expected to influence the deformation behavior of the samples.

#### **3.1.1.5. Microstructural Characterization**

Microstructural characterization was performed on specimens cut using a wire electrical discharge machining (WEDM) system equipped with a 0.3 mm brass wire, which ensured high dimensional accuracy while minimizing temperature rise and mechanically induced deformation as well as residual stresses at the cut surfaces. Samples prepared for optical microscopy were subjected to standard metallographic preparation, including successive mechanical grinding up to 2000-grit SiC paper, followed by polishing with 3 µm and 1 µm alumina suspensions. The polished surfaces were subsequently chemically etched using a solution containing 5 g FeCl<sub>3</sub>, 3 ml HCl, and 97 ml ethanol for 5–10 s to reveal grain structure and intermetallic phases. Detailed compositional analysis and fractographic examination were carried out using a Hitachi S-3400N scanning electron microscope (SEM) operated at an accelerating voltage of 15 keV, which enabled high-quality imaging and compositional contrast. Crystallographic characterization was conducted by electron backscatter diffraction (EBSD) using a JEOL JSM-7100F SEM equipped with an EDAX EBSD detector and operated at an accelerating voltage of 20 kV. EBSD data acquisition and subsequent analysis were performed using OIM TSL software. To improve the quality and reliability of the EBSD datasets, post-processing procedures included a single iteration of grain dilation clean-up and confidence index standardization. These steps reduced the presence of misindexed points and improved the reliability and interpretability of crystallographic orientation mapping and grain boundary determination.

#### **3.1.1.6. Mechanical Properties Assessment**

The mechanical properties of the investigated alloys were characterized through hardness measurements and quasi-static uniaxial tensile testing. Hardness was evaluated using the

Vickers method under a load of 1 kg (HV1) with a dwell time of 10 s, employing a Struers Duramin A300 hardness tester. Multiple indentations were performed for each condition to obtain representative hardness values. Due to the limited size of the severely deformed region in the MaxStrain-processed samples, tensile testing was carried out using miniature specimens with geometries shown in Figure 15(a). These specimens were precisely cut from the central region of the deformation zone, in the transverse direction perpendicular to the pressing direction after the 20th (and 40th) pass (Figure 15(b)), to obtain a representative assessment of the deformed material. Tensile tests were conducted at RT using an MST QTest/10 universal testing machine at an initial strain rate of  $10^{-3} \text{ s}^{-1}$ . For each condition, three tensile tests were performed to ensure repeatability and reliability of the measured mechanical properties.

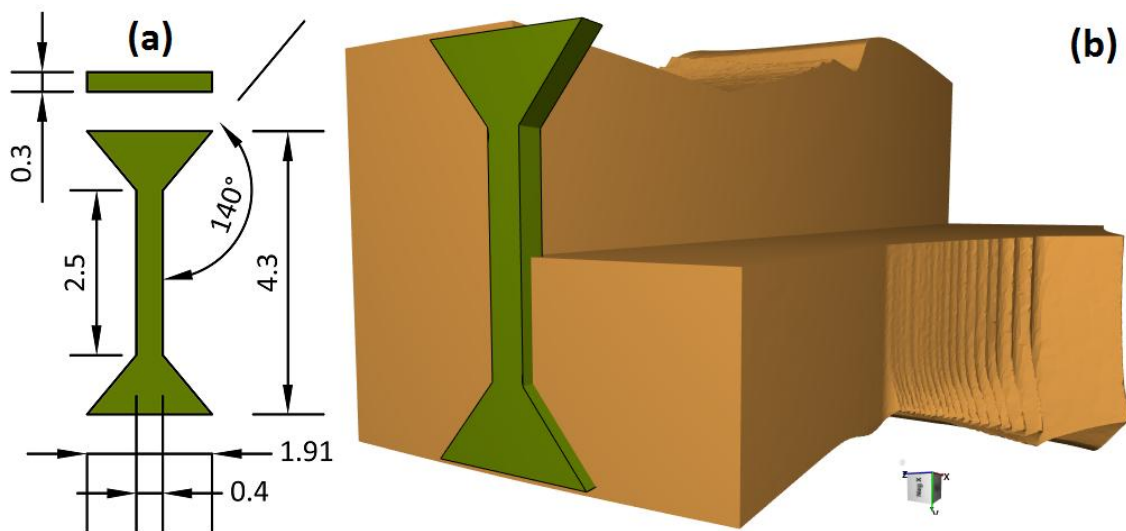


**Figure 15:** (a) Dimensions and (b) spatial location of minisamples used for performing the tensile test.

### 3.1.1.7. Electrical Conductivity Evaluation

The electrical conductivity of the samples was determined using the 4-wire Kelvin resistance measurement technique, which minimizes the influence of contact and lead resistances and is therefore well suited for low-resistance materials. Each specimen, with a cross-sectional area of approximately  $2 \text{ mm}^2$  and a gauge length of 4 mm, was cut along the same orientation as the tensile test minisamples to ensure consistent characterization of the material. During the tests, a controlled electrical current was supplied through two outer probes, and the resulting voltage drop was measured independently using two inner probes, which enabled the accurate calculation of the electrical resistivity of the alloys [289]. In addition, the current was supplied by a Fluke 5700A calibrator, and the voltage response was recorded using a Keithley 2182A nanovoltmeter, providing high measurement sensitivity and stability.

Preliminary measurements indicated a slight temperature rise at applied currents above 1 A due to Joule heating, as well as a comparable contribution from thermal electromotive forces at currents below 200 mA. Based on these observations, a test current of 750 mA was selected to balance signal resolution and thermal stability. All measurements were performed at RT, and each specimen was measured four times to improve data reliability and assess measurement repeatability. A simplified schematic of the 4-wire Kelvin resistance measurement setup is shown in Figure 16.

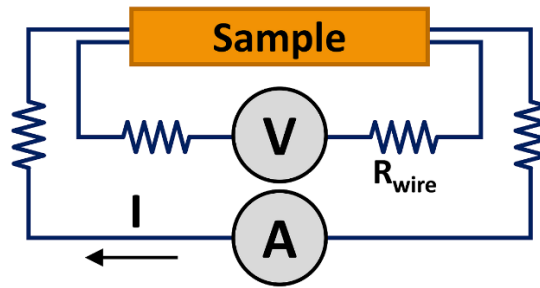


**Figure 16:** Simplified illustration of the 4-wire Kelvin resistance measurement setup.

### 3.1.2. Results and Discussion

#### 3.1.2.1. Characterization of As-Cast Alloys

Figure 17(a) presents the as-cast macrostructure of the binary Cu-Mg alloy, which exhibits a well-defined columnar grain structure characteristic of directional solidification. The microstructure consists of a homogeneous single-phase Cu-Mg solid solution, with an average grain thickness of  $927 \pm 129 \mu\text{m}$ . Such columnar grains typically form under strong thermal gradients, where grain growth is oriented along the heat flow direction [290]. In contrast, Figure 17(b) shows that the addition of Ca to the ternary Cu-Mg-Ca alloy leads to a significant change in grain structure. The as-cast macrostructure shifts from the columnar morphology observed in the binary alloy to a predominantly equiaxed grain structure, with an average grain diameter of  $883 \pm 93 \mu\text{m}$ . This transition indicates that Ca promotes the formation of new nucleation sites during solidification and reduces the dominance of directional grain growth. As a result, Ca enhances heterogeneous nucleation and/or restricts grain growth, leading to a more refined and equiaxed structure, suggesting that Ca acts as an effective grain refiner in this alloy system. A similar observation was also made by Guo et al. [29], in which they found that adding up to 0.33 wt.% Cr to a Cu-0.16Mg (wt.%) alloy noticeably refined the as-cast grain structure.

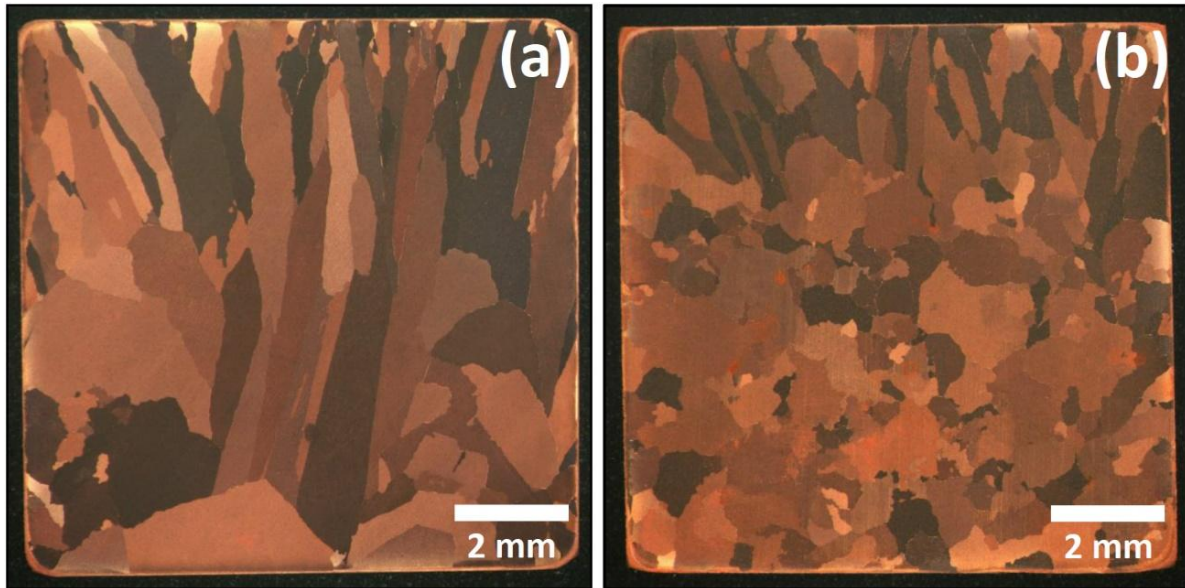


**Figure 17:** Macrostructures of as-cast (a) binary and (b) ternary alloys.

For better clarity, [Figure 18](#) has been included to show the microstructure of the ternary Cu-Mg-Ca alloy at higher magnification, revealing the presence of primary intermetallic particles formed during solidification with an average diameter of  $7.1 \pm 1.8 \mu\text{m}$ . These intermetallic particles are distributed throughout the microstructure, occurring both along grain boundaries and within grain interiors. This distribution indicates that the particles formed during solidification are not restricted to a single microstructural region (such as grain boundary zones) but instead appear in multiple characteristic locations within the grains, suggesting a broadly dispersed second phase in the as-cast condition.

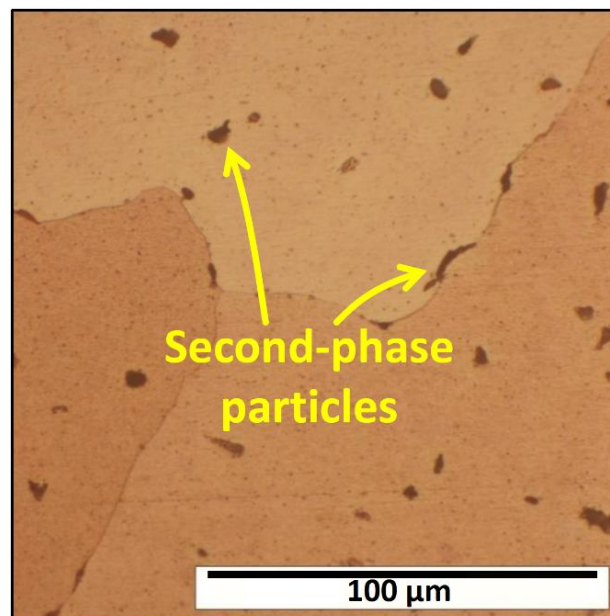


**Figure 18:** Optical microscopy image of the microstructure of the ternary alloy.

Based on the binary Cu-Ca phase diagram shown in Figure 4, the observed second-phase particles were expected to correspond to the  $\text{Cu}_5\text{Ca}$  intermetallic compound. However, to verify this assumption, detailed SEM investigations were carried out, and the corresponding elemental mapping results are presented in Figures 19(b) to 19(d). In addition, EDS analyses were performed on regions 1 and 2 indicated in Figure 19(a), with the quantitative results shown in Figure 20. The elemental mapping results in Figure 19 clearly reveal that Cu (Figure 19(b)) and Mg (Figure 19(c)) are homogeneously distributed across the entire microstructure, whereas Ca (Figure 19(d)) is predominantly concentrated within the shown second-phase particle. This distinct elemental segregation strongly supports the identification of these particles as  $\text{Cu}_5\text{Ca}$  intermetallic phases.

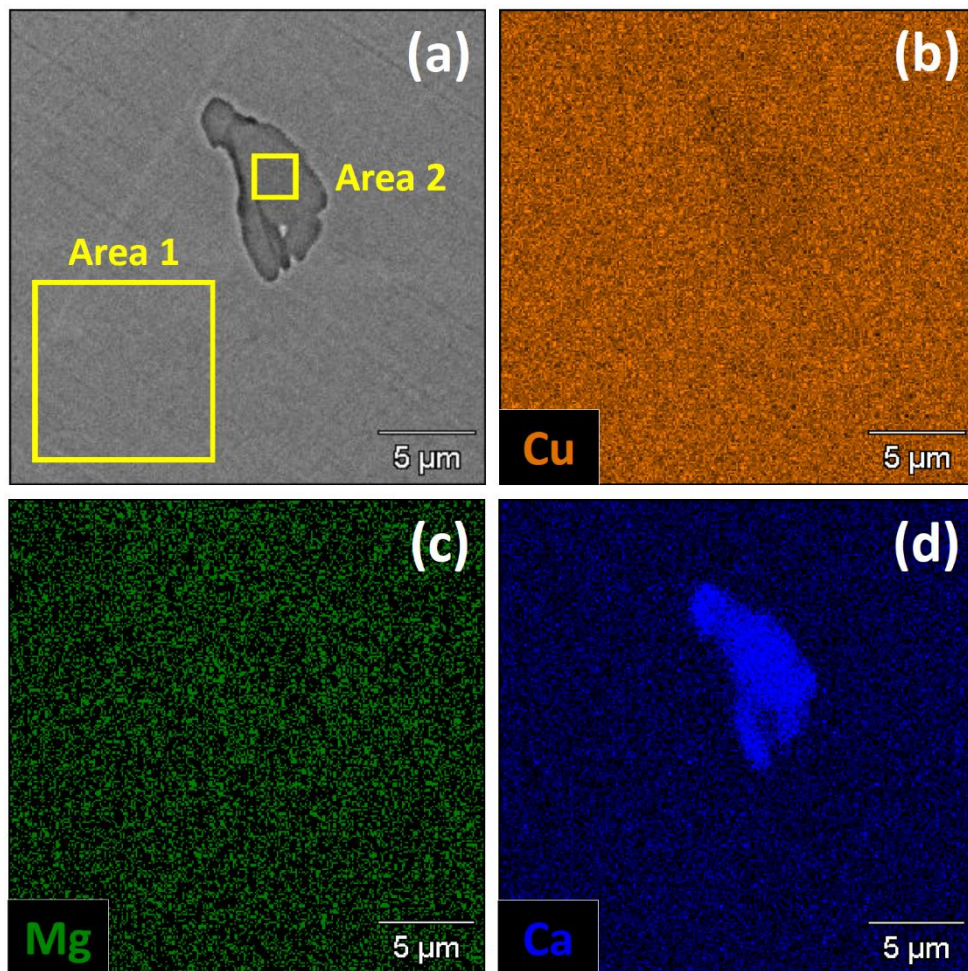


**Figure 19:** (a) SEM image of the ternary alloy acquired in backscattered electron (BSE) mode, and the corresponding elemental mapping images for (b) Cu, (c) Mg, and (d) Ca elements.

Figure 20(a) shows the EDS spectrum obtained from area 1 marked in Figure 19(a), confirming that the matrix phase consists of a solid solution of Mg in Cu. This observation is consistent with the nominal chemical composition of the alloy and with the Cu-Mg binary

phase diagram presented in Figure 2. In contrast, the EDS spectrum of area 2, shown in Figure 20(b), reveals that the second-phase particles are primarily composed of Cu and Ca, with a measured Cu/Ca atomic ratio of 4.18. This ratio closely approaches the ideal stoichiometric value of 5 for the  $\text{Cu}_5\text{Ca}$  intermetallic compound. Accordingly, the intermetallic particles observed in the ternary alloy can be confidently identified as the  $\text{Cu}_5\text{Ca}$  phase. It should be noted that the small amount of Mg detected in the EDS spectrum of area 2 can be attributed to electron beam penetration beyond the  $\text{Cu}_5\text{Ca}$  particles into the underlying Cu-Mg matrix, resulting in the detection of trace Mg contributions.

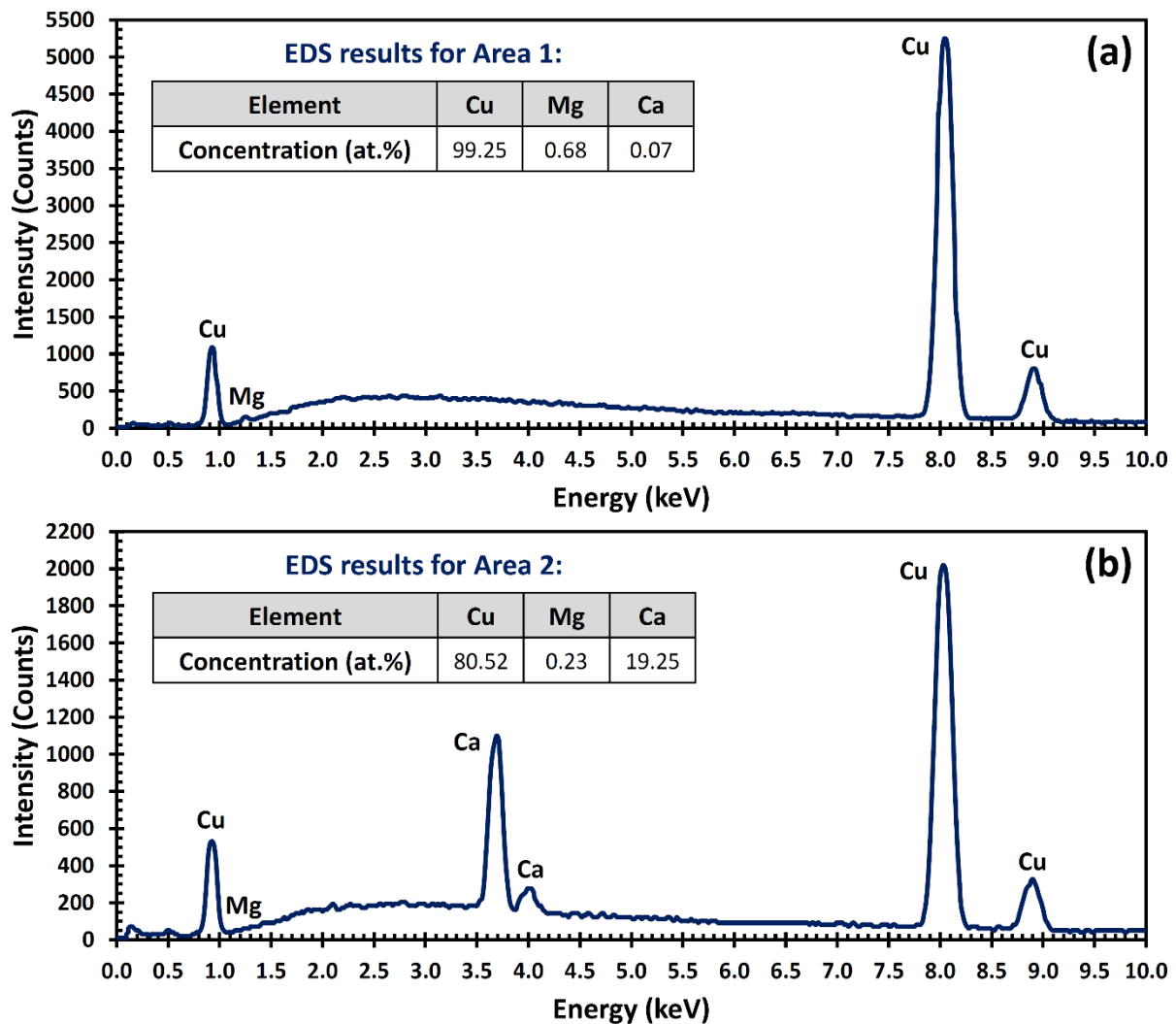


**Figure 20:** EDS analysis results for (a) area 1 and (b) area 2 marked in Figure 19(a).

### 3.1.2.2. DSC Analysis

As mentioned earlier, DSC analysis is often performed to investigate precipitation processes in metallic alloys as they are subjected to heating and cooling cycles [291]. In general, endothermic peaks observed during heating are commonly associated with precipitate

dissolution and melting reactions, whereas exothermic peaks may correspond to precipitation or solidification processes. During cooling, exothermic peaks typically indicate heat release associated with solidification and the formation of secondary phases [292]. [Figure 21\(a\)](#) displays the DSC thermogram of the binary alloy for the heating mode, in which no visible peaks were observed during heating except for one endothermic peak at 1094 °C, which corresponds to the melting of the alloy. Likewise, in its cooling mode, there is only one distinct exothermic peak at 1053 °C that represents the solidification of the alloy ([Figure 21\(b\)](#)). These observations are consistent with the microstructural analysis, which revealed that in the binary alloy, all Mg atoms were completely dissolved in the Cu matrix without the formation of the Cu<sub>2</sub>Mg intermetallic phase. It must also be noted that the variation in peak temperatures in heating and cooling modes is due to the superheating and supercooling, which occurred as a result of the constant rate of temperature change during the DSC analysis [293].

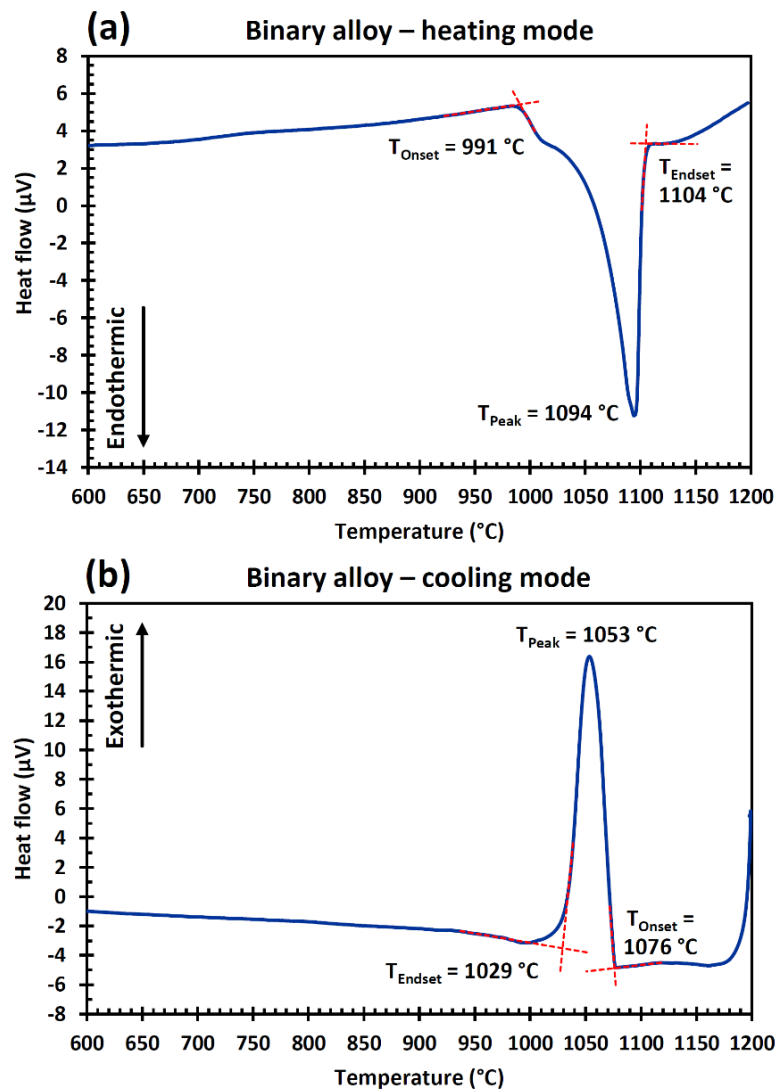


**Figure 21:** DSC thermograms for the binary alloy in (a) heating and (b) cooling modes.

Meanwhile, as shown in Figure 22(a), during heating of the ternary alloy, a small endothermic peak was observed at 856 °C prior to the main melting peak at 1078 °C, which indicates the occurrence of a distinct endothermic transformation, likely associated with phase dissolution or a secondary reaction. Similarly, the DSC cooling curve of the ternary alloy (Figure 22(b)) revealed an additional exothermic peak at 799 °C following the primary solidification peak at 1044 °C, which suggests the presence of an exothermic transformation, possibly related to the formation of secondary phases or precipitation. Taking into account these observations, an annealing temperature of 650 °C was selected prior to SPD processing to avoid the onset of such thermal transformations and to ensure microstructural stability during subsequent processing.

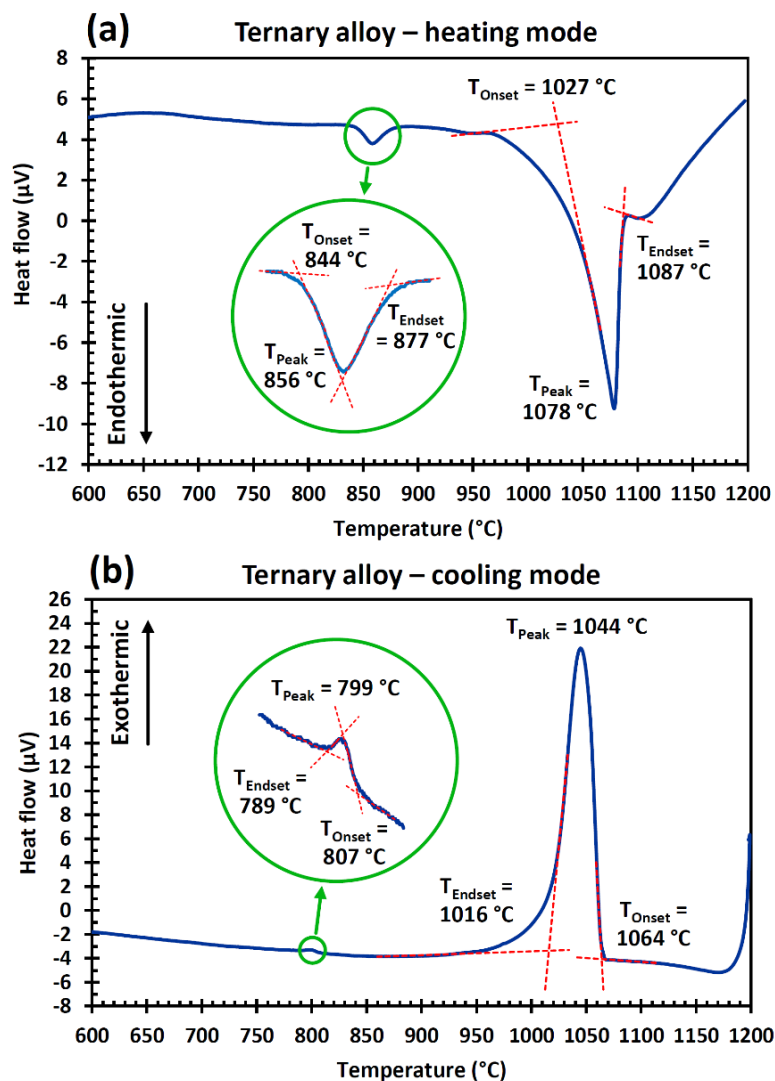


**Figure 22:** DSC thermograms for the ternary alloy in (a) heating and (b) cooling modes.

### 3.1.2.3. Annealing Processes

Based on the results obtained from the DSC analysis and microstructural observations, the annealing treatments illustrated in Figure 23 were carried out prior to subsequent MaxStrain processing. In summary:

- i. Annealing at 650 °C for 90 min followed by water quenching (applied to both binary and ternary alloys), followed by 40 passes of MaxStrain processing;
- ii. Annealing at 650 °C for 75 min followed by water quenching (applied only to the ternary alloy), followed by 20 passes of MaxStrain processing;
- iii. Annealing at 650 °C for 15 min followed by air cooling (applied only to the ternary alloy), followed by 20 passes of MaxStrain processing.

These annealing routes were designed to homogenize the as-cast microstructure of both binary and ternary alloys and to promote a more uniform distribution of Mg atoms within the matrix at the atomic scale and reduce chemical segregation and microstructural heterogeneity. Moreover, the annealing durations were intentionally kept relatively short to maintain industrial applicability and to minimize excessive grain growth.

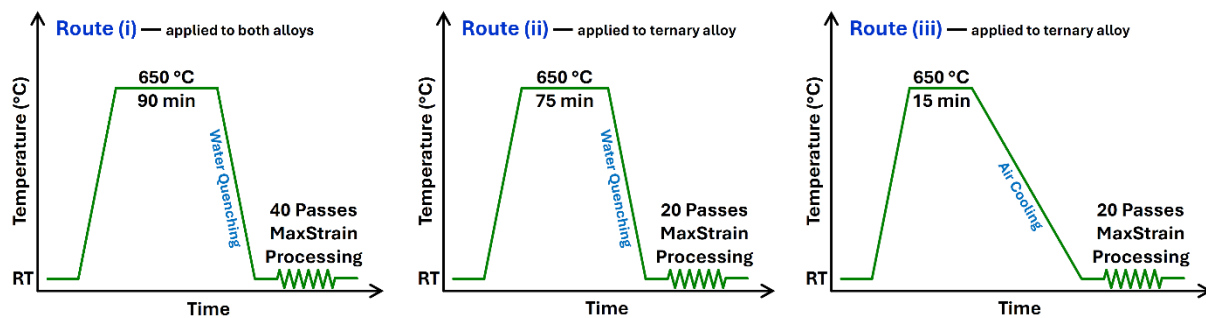


**Figure 23:** Thermomechanical processing routes used in Approach 1.

In annealing routes (i) and (ii), quenching was considered to introduce residual thermal stresses into the alloys through rapid cooling, particularly in the ternary alloy where the difference in the coefficient of thermal expansion (CTE) between the Cu<sub>5</sub>Ca intermetallic particles and the Cu-Mg matrix are capable to generate internal stress fields during rapid cooling and contribute to a quench-strengthening effect [294,295]. It is well established that Cu possesses a FCC crystal structure, with lattice parameters of  $a = b = c = 3.615 \text{ \AA}$  and  $\alpha = \beta = \gamma = 90^\circ$  [296,297], as illustrated in Figure 24(a). Given the relatively low Mg content in both alloys (0.7 wt.%), it can be assumed that Mg atoms are predominantly dissolved in the matrix, forming a substitutional solid solution by replacing a small fraction of Cu atoms within the FCC lattice. As a result, the dominant crystal structure of the matrix remains FCC (although

the presence of Mg in solid solution may induce a slight lattice distortion). In contrast, the  $\text{Cu}_5\text{Ca}$  intermetallic phase exhibits a totally different crystallographic structure. As shown in Figure 24(b), the  $\text{Cu}_5\text{Ca}$  unit cell is characterized by lattice parameters of  $a = b = 5.074 \text{ \AA}$  and  $c = 4.074 \text{ \AA}$ , with  $\alpha = \beta = 90^\circ$  and  $\gamma = 120^\circ$ , which is consistent with a hexagonal crystal system [298]. This mismatch in crystal structures between the single-phase Cu-Mg solid solution matrix and the  $\text{Cu}_5\text{Ca}$  intermetallic particles leads to differences in their physical properties, including their CTE. Therefore, in the case of the ternary alloys, heating to  $650^\circ\text{C}$  caused both the Cu-Mg matrix and the  $\text{Cu}_5\text{Ca}$  particles to undergo thermal expansion. However, due to their different CTE values, the expansion was not uniform, which resulted in an increase in the density of dislocations in the Cu-Mg matrix adjacent to the  $\text{Cu}_5\text{Ca}$  particles at the onset of the annealing process. As the annealing continued, the microstructure gradually approached a new stable equilibrium by the end of the soaking period at  $650^\circ\text{C}$ . However, upon quenching the alloy to RT, a non-equilibrium thermal contraction occurred. Both the Cu-Mg matrix and the  $\text{Cu}_5\text{Ca}$  particles experienced rapid shrinkage, generating a new population of dislocations around the  $\text{Cu}_5\text{Ca}$  particles within the matrix to maintain structural cohesion. This thermally induced increase in dislocation density was more pronounced in the ternary alloys compared to the binary alloys, which also provided a higher density of other defects, which is expected to result in a higher degree of grain refinement during subsequent MaxStrain processing.

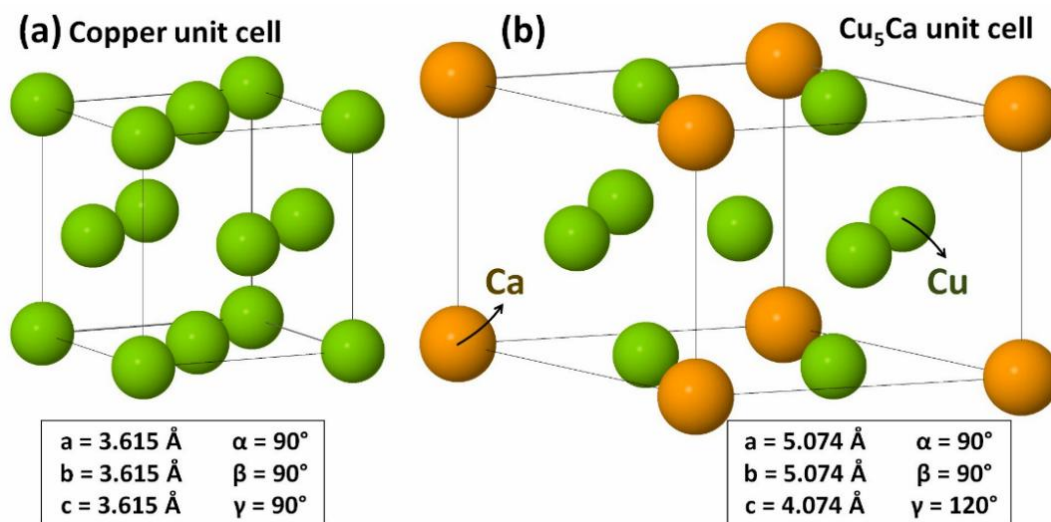


**Figure 24:** Schematic illustrations of the crystal structures of (a) Cu [297], and (b)  $\text{Cu}_5\text{Ca}$  [298] unit cells.

In addition, the annealing routes (i) and (ii) were intended to promote partial spheroidization of the  $\text{Cu}_5\text{Ca}$  particles in the ternary alloy, which was expected to result in improving ductility by reducing stress concentration sites during subsequent deformation. Interestingly, a noticeable change in particle morphology was observed in the ternary alloys following the

mentioned heat treatments, wherein a portion of the irregular  $\text{Cu}_5\text{Ca}$  particles present in the as-cast condition transformed from sharp-edged to more spheroidized shapes. Illustrative examples of this morphological evolution are shown in Figures 25 and 26, which present the irregular and spheroidized  $\text{Cu}_5\text{Ca}$  particles in the ternary alloy before and after the heat treatment process using the routes (i) and (ii), respectively.

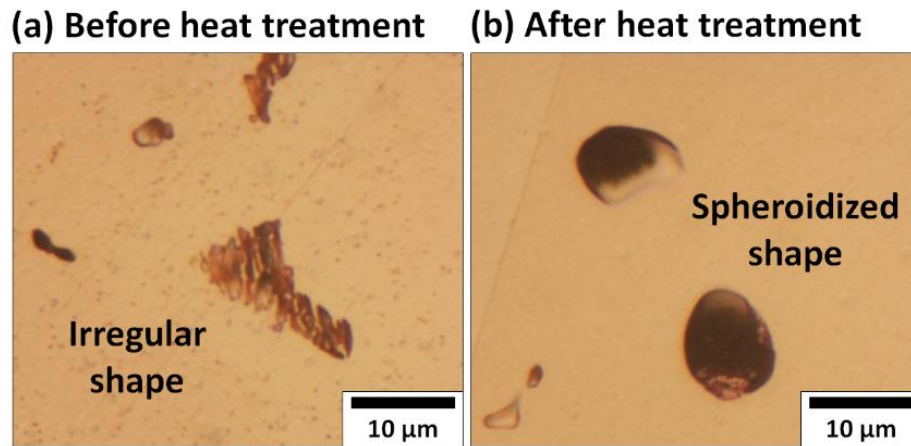


**Figure 25:** Optical microscopy microstructures of the ternary alloy (a) before and (b) after application of heat treatment process route (i).

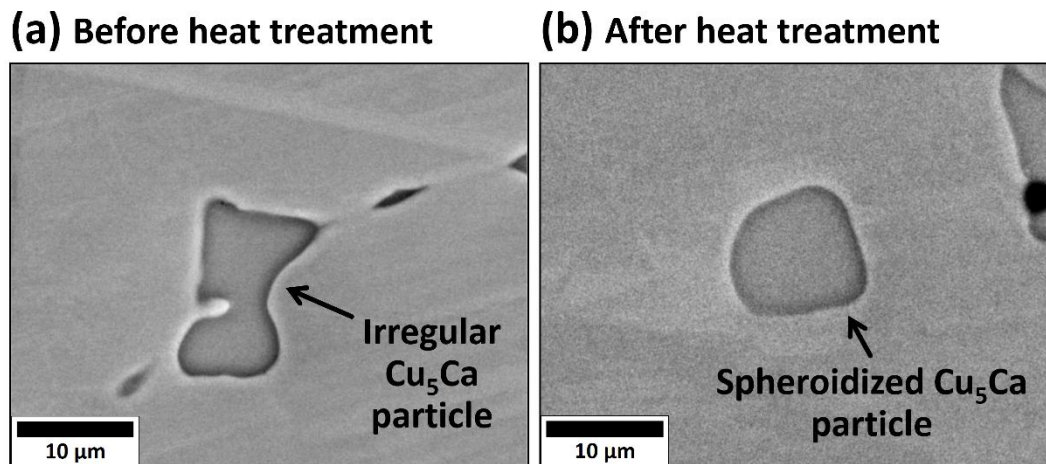


**Figure 26:** SEM-BSE microstructures of the ternary alloy (a) before and (b) after application of heat treatment process route (ii).

### 3.1.2.4 MaxStrain Processing

Figure 27 illustrates the strike pattern as a function of operation time during the MaxStrain process in route (i), highlighting a systematic deformation response characterized by alternating compression levels between odd and even passes. In simple words, the sample exhibited a slight increase in transverse thickness following the initial compression. This can be attributed to the plastic flow and constrained deformation, which results in a barreling effect.

This transverse expansion increased the effective distance between the anvils during the subsequent compression pass and, therefore, reduced the imposed anvil movement compared to the preceding pass. This alternating deformation behavior was consistently observed throughout the entire processing sequence, which indicates stable material flow conditions and a repeatable deformation pattern under the applied MaxStrain processing parameters. The presence of the barreling effect made it impractical to accurately calculate the percentage of compression applied in each deformation pass. As a result, a simulation of the deformation process was considered the most reliable approach for determining the equivalent strain distribution within different regions of the deformation zone. Accordingly, the entire deformation sequence was simulated, as described in detail in the following section. Based on the data obtained from the Gleeble thermomechanical simulator, the positions of the anvils relative to the center of the sample were recorded as 2.22 mm and 2.47 mm at the end of the 39th and 40th deformation passes, respectively. This corresponds to nominal final sample thicknesses of 4.44 mm and 4.94 mm at the end of the 40th pass, assuming ideal deformation without considering the barreling effect during the final pressing step. However, the experimentally measured average thicknesses at the center of the deformation zone after completion of the entire deformation process were  $4.83 \pm 0.06$  mm and  $4.97 \pm 0.05$  mm. The discrepancy between the nominal and measured values can be attributed to the combined effects of transverse barreling and elastic springback.

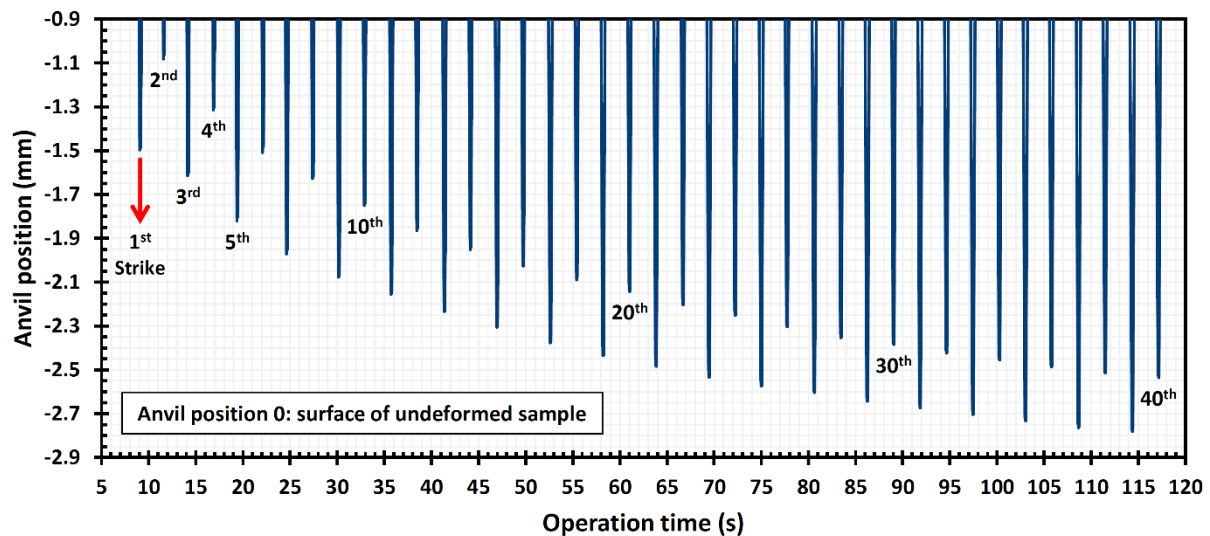


**Figure 27:** Strike pattern used for performing the MaxStrain process in route (i).

Likewise, the strike patterns of routes (ii) and (iii) are presented in [Figure 28](#). The experimentally measured average thicknesses at the center of the deformation zone after

completion of the full deformation process were  $5.58 \pm 0.14$  mm and  $5.73 \pm 0.07$  mm, respectively, at the end of the 20th pass.

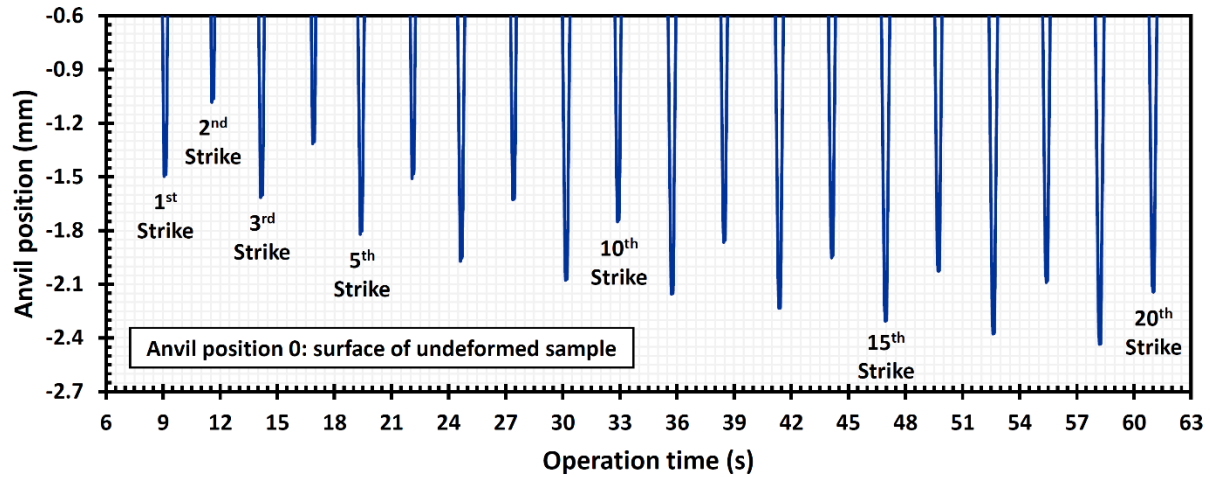


**Figure 28:** Strike pattern used for performing the MaxStrain process in routes (ii) and (iii).

### 3.1.2.5. Simulation of the MaxStrain Process

As discussed in the previous section, the occurrence of barreling during each deformation pass made it impractical to determine the equivalent strain for individual passes, as well as the total accumulated strain over the entire deformation process. Therefore, the entire deformation process was simulated using FORGE® NxT software (version 3.2). To ensure close agreement between the simulation and the experimental conditions, the final anvil height for each deformation pass was derived from experimental measurements and used to define the corresponding anvil displacement. In the simulation, the anvils were consistently initialized at the same position, 8 mm above the surface of the undeformed specimen, to ensure uniform starting conditions. The final anvil position at the end of each pass was then adjusted based on experimental data obtained from the Gleeble thermomechanical simulator to allow the simulation to accurately reflect the imposed deformation in each stage. Figures 29(a) to 29(f) display the simulation results for 1/2 of the sample after 40 passes of MaxStrain processing, with transverse cutting planes inserted at 1 mm intervals starting from the center of the deformation zone to allow for the observation of the distribution of equivalent strain inside the deformation zone. As expected, the equivalent strain distribution was non-uniform throughout the sample, with higher strain concentrations observed at both the center and the corners of the deformation zone. The results further indicated that, as the cutting plane moved away from the center of the deformation zone, the location of maximum equivalent strain gradually shifted from the center toward the corners of the transverse plane.

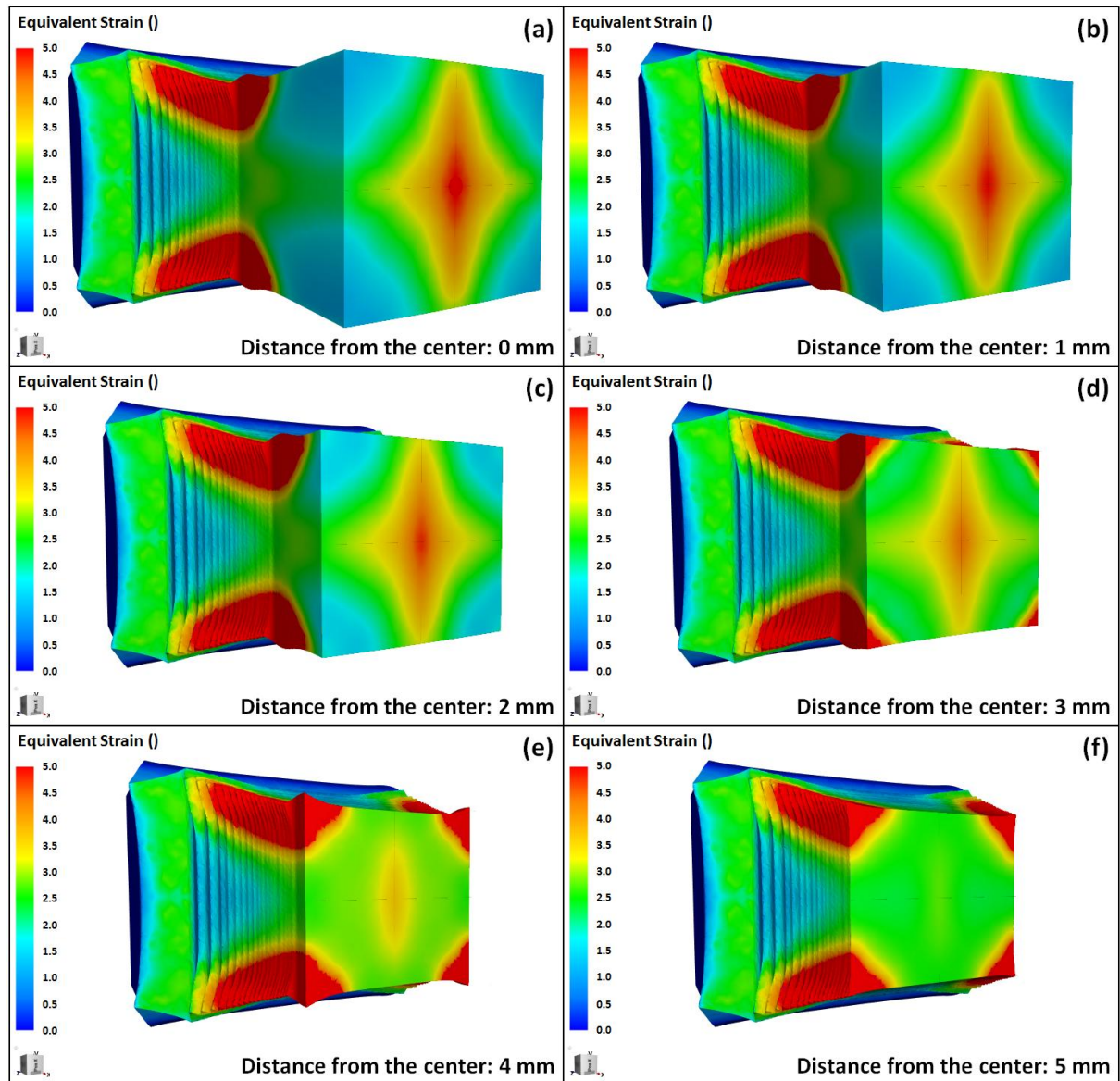


**Figure 29:** Distribution of equivalent strain after 40 passes of MaxStrain processing in transverse planes located at distances of (a) 0 mm, (b) 1 mm, (c) 2 mm, (d) 3 mm, (e) 4 mm, and (f) 5 mm from the center of the deformation zone.

In addition, analysis of the equivalent strain distribution after 40 passes of MaxStrain processing, as illustrated in [Figures 30\(a\) and 30\(b\)](#) for the central longitudinal and central transverse planes, revealed a maximum equivalent strain of 5.0 at the center of the deformation zone, indicating the region of most intense deformation. In contrast, the minimum equivalent strain values, approximately 2.6 and 1.2 in the central longitudinal and transverse planes, respectively, were observed at the corners of these planes, reflecting comparatively lower levels of deformation toward the outer regions.

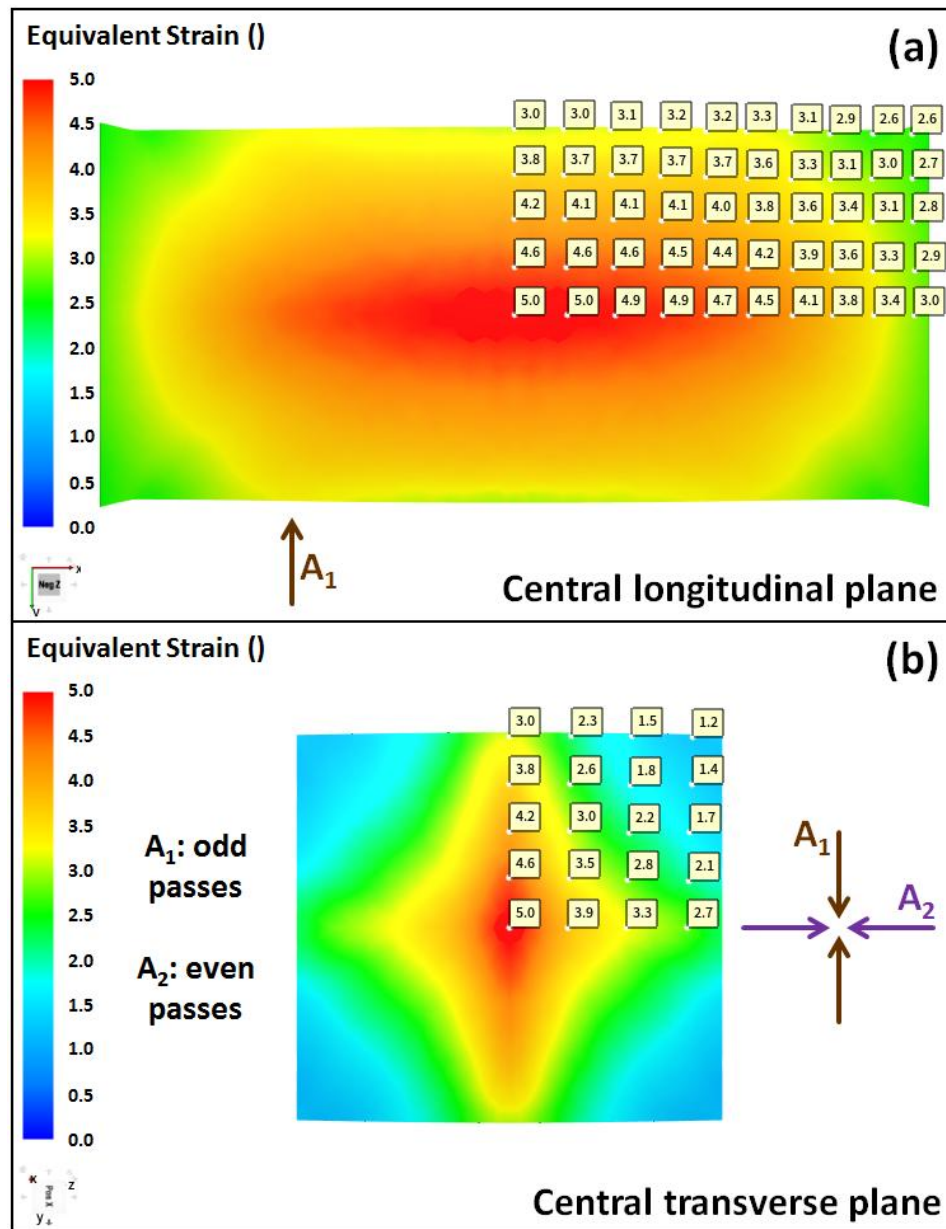


**Figure 30:** Calculated values for the equivalent strain in (a) central longitudinal and (b) central transverse planes after 40 passes of MaxStrain processing.

Likewise, [Figures 31\(a\) to 31\(c\)](#) present the simulation results for the primary halves of the specimens subjected to 20 passes of MaxStrain processing. Cross-sectional planes taken along the principal directions at the specimen center are included to clearly illustrate the internal distribution of equivalent strain and to enable direct comparison of strain variations along different orientations.

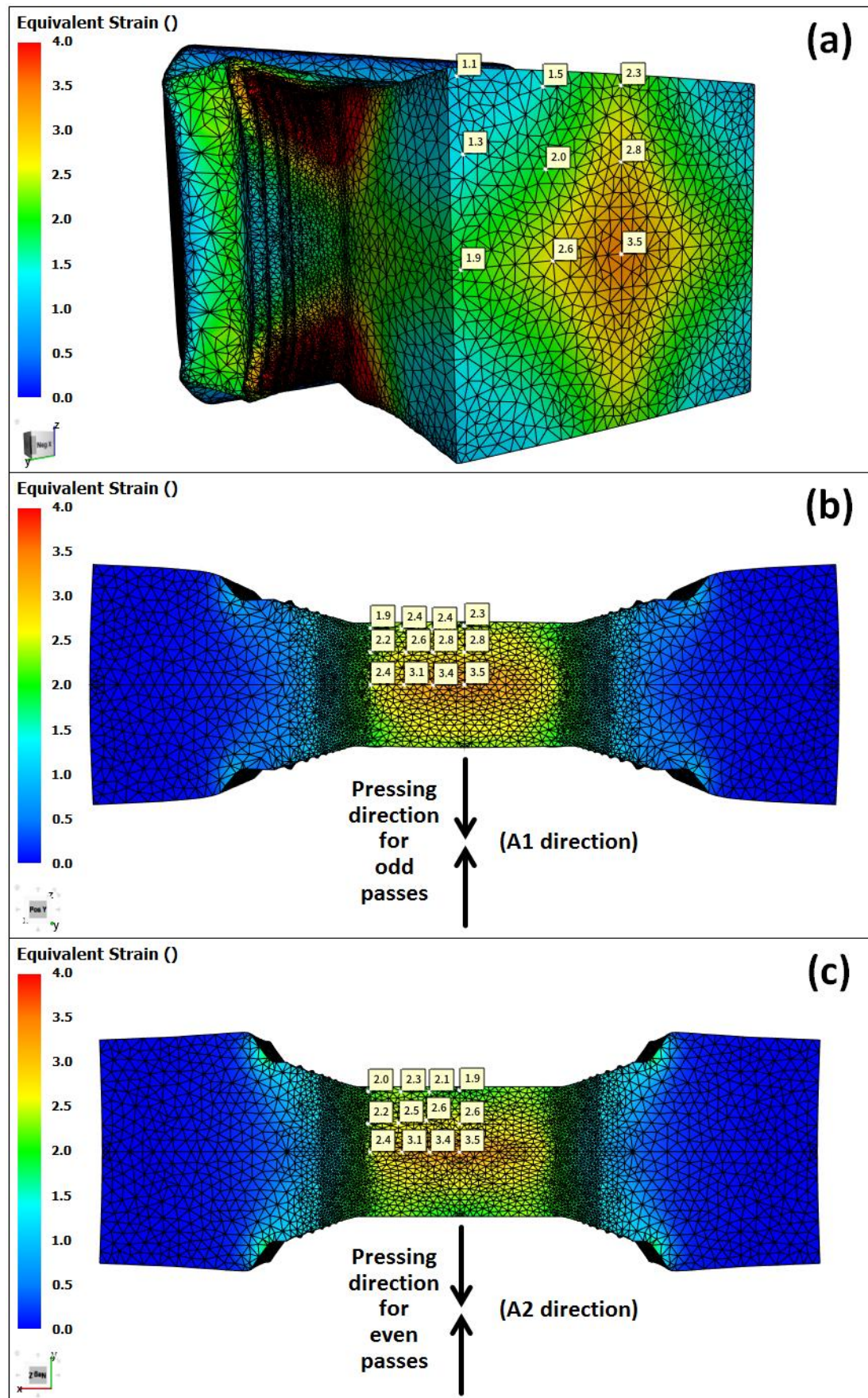


**Figure 31:** Simulation results illustrating the distribution of equivalent strain after 20 passes of MaxStrain processing in (a) central transverse, (b) central longitudinal (X-Z), and (c) central longitudinal (X-Y) planes.

For this processing condition (20 passes, routes (ii) and (iii)), the maximum equivalent strain, approximately 3.5, was observed at the center of the central transverse plane (Figure 31(a)), which identifies this region as the location of the most intense plastic deformation. The strain magnitude gradually decreased with increasing distance from the center toward the edges and corners of the plane, which reflect the heterogeneous nature of the deformation imposed during processing. A similar trend is evident in the major longitudinal planes shown in Figures 31(b) and 31(c), where the equivalent strain reaches its highest value at the central region of plastic deformation, again with a maximum of about 3.5, and progressively decreases along the principal directions away from the center. These results indicate that the most pronounced strain-hardening effect is expected to occur in the central region of plastic deformation, where the equivalent strain is highest and deformation is most concentrated. Accordingly, all microstructural analyses were focused on this region in order to capture the areas most significantly affected by MaxStrain processing and to ensure a representative assessment of the material's response under the most severe deformation conditions. For ease of reference, Table 5 summarizes the processing history and lists the abbreviated names assigned to each sample, which are used throughout this work.

**Table 5:** Summary of processing history and abbreviated sample names for Approach 1.

| Samples' names | Processing history                                     |
|----------------|--------------------------------------------------------|
| BC             | Binary alloy, as-cast                                  |
| TC             | Ternary alloy, as-cast                                 |
| BC-M40         | BC processed by MaxStrain (40 passes, RT)              |
| TC-M40         | TC processed by MaxStrain (40 passes, RT)              |
| BC-90Q         | BC annealed at 650 °C for 90 min, water-quenched to RT |
| TC-90Q         | TC annealed at 650 °C for 90 min, water-quenched to RT |
| BC-90Q-M40     | BC-90Q processed by MaxStrain (40 passes, RT)          |
| TC-90Q-M40     | TC-90Q processed by MaxStrain (40 passes, RT)          |
| TC-75Q         | TC annealed at 650 °C for 75 min, water-quenched to RT |
| TC-15A         | TC annealed at 650 °C for 15 min, air-cooled to RT     |
| TC-75Q-M20     | TC-75Q processed by MaxStrain (20 passes, RT)          |
| TC-15A-M20     | TC-15A processed by MaxStrain (20 passes, RT)          |

### 3.1.2.6. Microstructure of MaxStrain-Processed Samples

Figures 32(a) to 32(d) illustrate the inverse pole figure (IPF) maps acquired from the center of the central transverse plane at the deformation zone of the samples subjected to 40 passes of MaxStrain processing. This plane is oriented perpendicular to the A<sub>2</sub> pressing direction shown

in [Figure 30\(b\)](#). In these maps, HAGBs are marked by black lines to clearly distinguish grain boundaries with misorientation angles greater than  $15^\circ$ . Due to the inherent difficulties in reliably indexing intermetallic particles using EBSD, the IPF maps for the TC-M40 and TC-90Q-M40 samples were intentionally obtained exclusively from the Cu-Mg matrix phase. To compensate for this limitation, SEM investigation was employed to separately analyze and characterize the  $\text{Cu}_5\text{Ca}$  intermetallic particles present in these samples. Based on the quantitative data obtained from the EBSD analysis, the average grain sizes were measured to be  $5.6\text{ }\mu\text{m}$  for the BC-M40 sample and  $4.0\text{ }\mu\text{m}$  for the BC-90Q-M40 sample. Interestingly, the TC-M40 and TC-90Q-M40 samples exhibited significantly finer microstructures, with average grain sizes of  $1.8\text{ }\mu\text{m}$  and  $1.5\text{ }\mu\text{m}$ , respectively.

The first notable observation from the IPF mapping results is that the MaxStrain-processed ternary samples exhibited a smaller average grain size compared with the corresponding binary samples. This grain refinement can be primarily attributed to the presence of  $\text{Cu}_5\text{Ca}$  intermetallic particles in the microstructure of the ternary alloys, acting through two main mechanisms. First, as illustrated in [Figure 17](#), the TC sample already displayed a finer initial grain structure compared to the BC sample. Second, these particles served as effective obstacles to dislocation motion during deformation, which promoted dislocation accumulation and resulted in a higher dislocation density. Since CDRX is associated with the rearrangement of deformation-induced dislocations into subgrain boundaries and the progressive increase in their misorientation toward HAGBs, the higher dislocation density in the ternary samples increased the stored deformation energy and provided a stronger driving force for progressive grain subdivision and CDRX [299,300]. However, because the deformation was carried out at RT, limited atomic mobility and restricted grain boundary migration constrained the extent of CDRX, and complete microstructural transformation through CDRX was not achieved under the applied processing conditions. In other words, only partial CDRX occurred during MaxStrain processing within the processing conditions used in this approach. This behavior is clearly reflected in the IPF maps of the processed samples, which reveal a heterogeneous microstructure consisting of a mixture of coarse deformed grains and fine grains generated through progressive grain subdivision. The coexistence of these grain populations provides strong evidence for the occurrence of partial CDRX induced by MaxStrain processing under the applied deformation conditions.

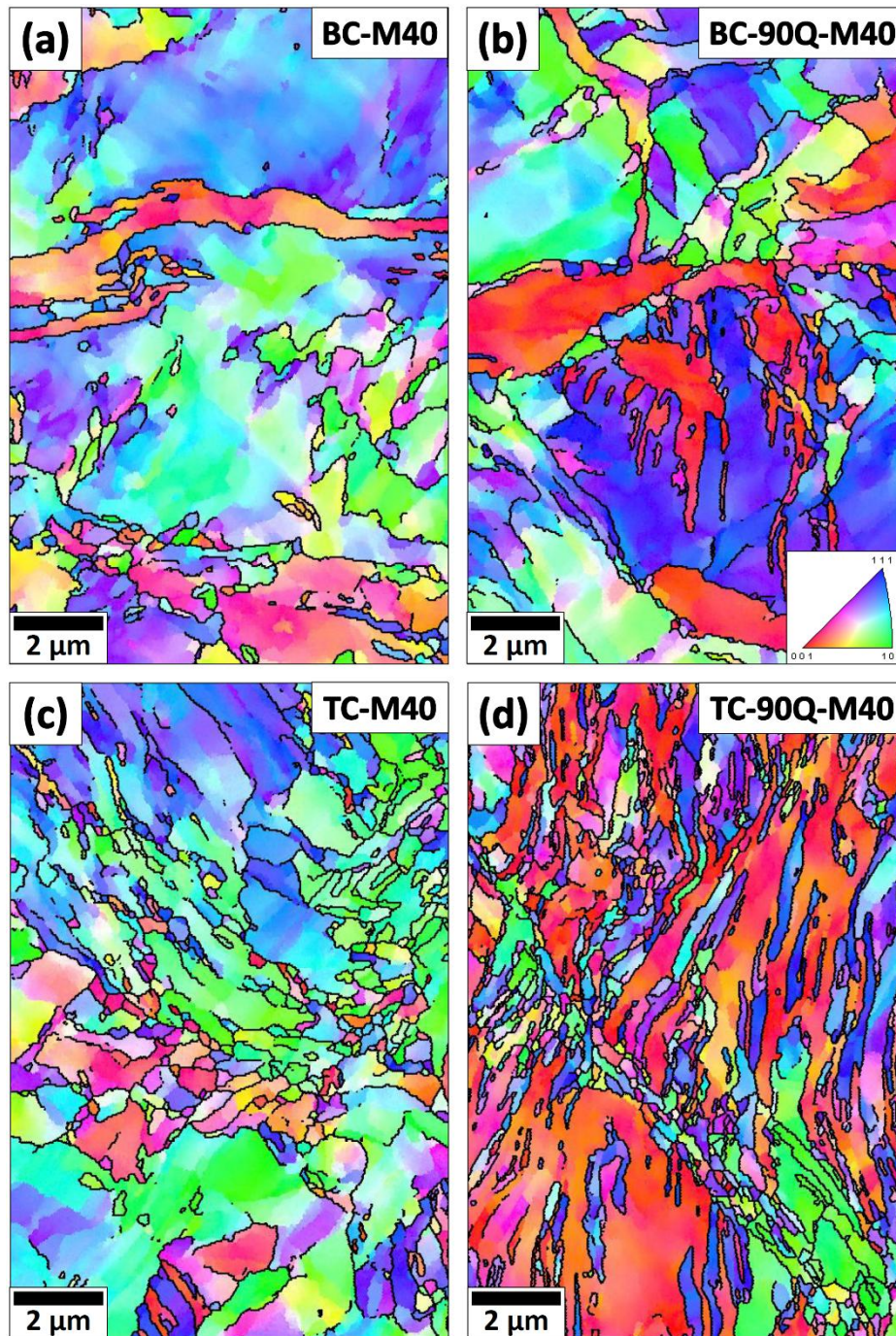


**Figure 32:** IPF maps of (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, and (d) TC-90Q-M40 samples.

In order to evaluate the fraction of CDRXed grains among samples, grain orientation spread (GOS) maps were created using EBSD data. GOS represents the average variation in orientation between the average grain orientation and all measurements within a single grain [301]. Generally, a higher GOS value indicates a more pronounced lattice distortion and a higher dislocation density [302]. Therefore, using a critical GOS value allows for the differentiation of CDRXed grains from deformed grains and the calculation of the volume fraction of CDRXed grains [303]. In alignment with previous studies [304–306], the minimum

threshold of  $GOS < 2^\circ$  was considered to distinguish CDRXed grains from deformed grains. Figures 33(a) to 33(d) show the GOS maps corresponding to Figures 32(a) to 32(d), with grains having GOS values less than  $2^\circ$  represented in red, grains having GOS values equal to or greater than  $2^\circ$  shown in blue, and LAGBs outlined by black lines.

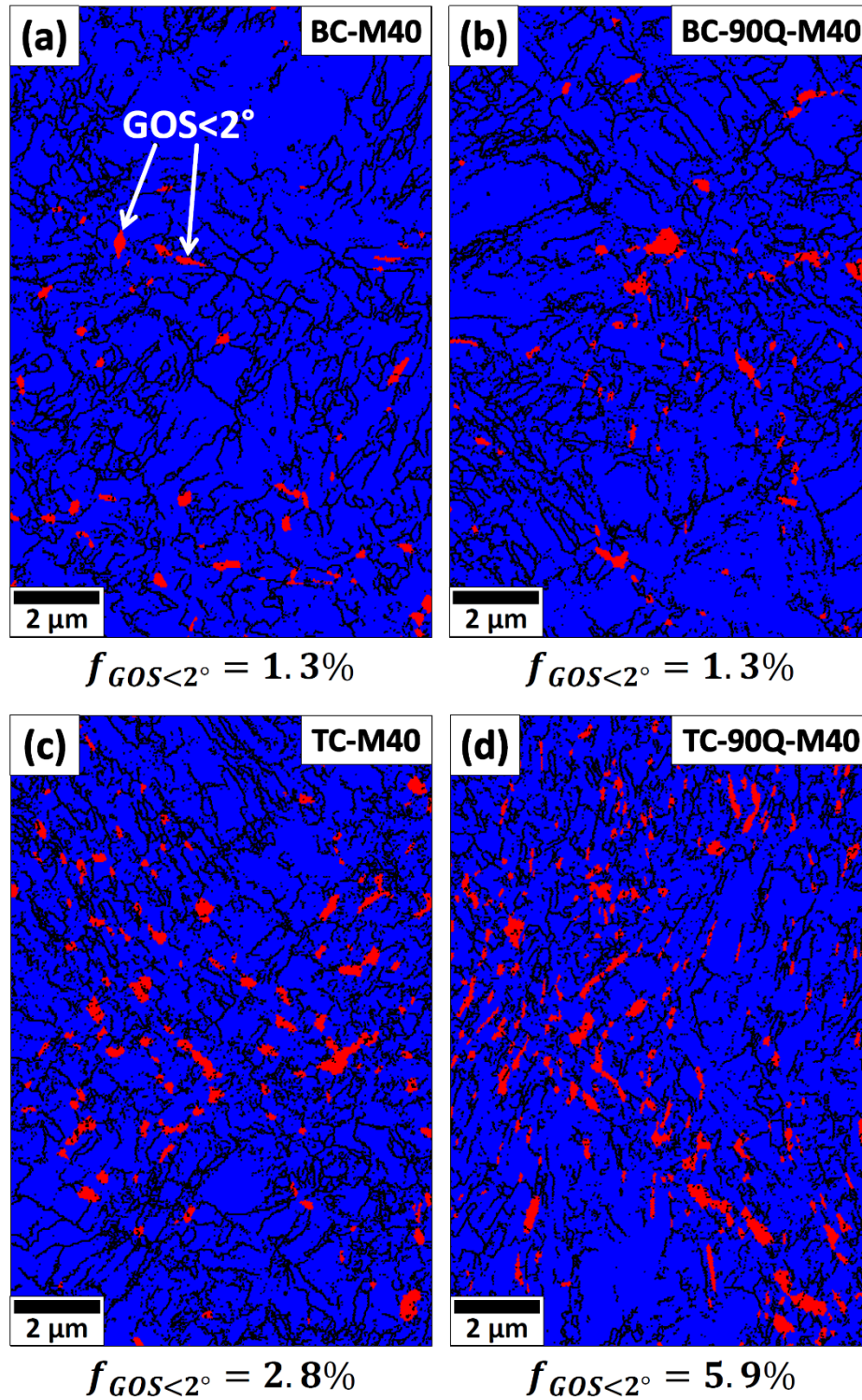


**Figure 33:** GOS maps of (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, and (d) TC-90Q-M40 samples (grains having a GOS value less than  $2^\circ$  are shown in red).

Within these GOS maps, regions displayed in red can be classified as CDRXed grains, whereas blue regions correspond to deformed grains with higher internal misorientation. However, it is important to emphasize that, within the framework of the present study, the use of GOS maps alone is not sufficient for a comprehensive evaluation of grain refinement. This limitation arises because the samples were subjected to 40 passes of repetitive deformation, during which grains that recrystallized at earlier stages were subsequently deformed in later passes. As a result, some of the early CDRXed grains accumulated additional distortion and therefore appear in the GOS maps with values exceeding the  $2^\circ$  threshold, which led to their classification as deformed grains. Despite this limitation, the fraction of grains exhibiting GOS values lower than  $2^\circ$  remains a meaningful and useful parameter for comparing the relative extent of CDRX among the different samples. A higher proportion of low-GOS grains was observed in the ternary samples compared with the binary samples. This observation is consistent with the earlier discussion and further supports the beneficial role of  $\text{Cu}_5\text{Ca}$  intermetallic particles in enhancing dislocation accumulation and facilitating CDRX in the ternary alloys during MaxStrain processing.

The EBSD results also showed that the grain sizes in BC-90Q-M40 and TC-90Q-M40 samples were comparatively smaller than those in BC-M40 and TC-M40 samples. This difference can be attributed to the higher dislocation density within the grains of BC-90Q and TC-90Q samples, as well as the residual thermal stresses introduced during quenching, which facilitated the grain refinement process through subsequent MaxStrain deformation. In fact, the higher dislocation density in BC-90Q and TC-90Q samples enabled more pronounced grain refinement during MaxStrain processing by facilitating the formation of dislocation cell structures, which subsequently transformed into LAGBs and further evolved into HAGBs [307]. The GOS results also showed that the TC-90Q-M40 sample had a greater proportion of grains with a GOS value less than  $2^\circ$  compared to the TC-M40 sample, which is consistent with the above explanation of the beneficial influence of the prior heat treatment process on grain refinement during MaxStrain processing.

Figure 34 displays the results of the SEM analysis conducted on  $\text{Cu}_5\text{Ca}$  intermetallic particles within the TC-M40 and TC-90Q-M40 samples. Remarkably, despite undergoing the deformation process, the  $\text{Cu}_5\text{Ca}$  particles retained their structural integrity. However, upon closer inspection, it was observed that some cracks had formed within these particles. Figures 34(b) and 34(d) show the bird's-eye view of the  $\text{Cu}_5\text{Ca}$  particles in the TC-M40 and TC-90Q-

M40 samples, clearly showing the presence of distinct cracks within these particles, which are identifiable by variations in depth.

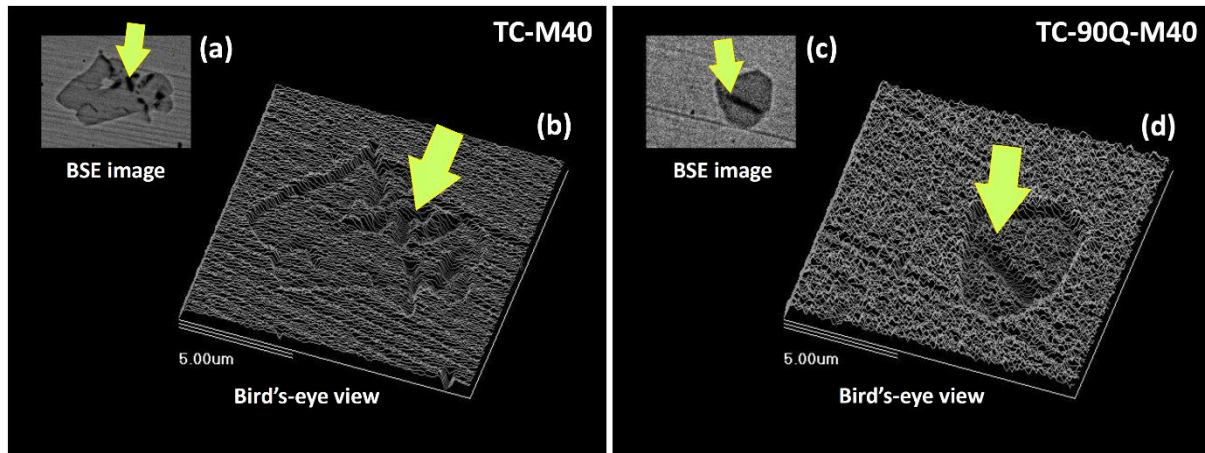


**Figure 34:** (a) SEM and (b) bird's-eye view images TC-M40 sample; (c) SEM and (d) bird's-eye view images TC-90Q-M40 sample.

Figure 35 illustrates the distribution of misorientation angles for the samples subjected to 40 passes of MaxStrain processing. A comparative analysis shows that the TC-M40 and TC-90Q-M40 samples contain a higher fraction of HAGBs than the BC-M40 and BC-90Q-M40 samples. Since HAGBs are commonly associated with more advanced stages of grain refinement and recrystallization, this indicates a more pronounced microstructure refinement in the ternary alloys. This observation is consistent with the earlier discussion, which attributed the enhanced grain refinement in the TC-M40 and TC-90Q-M40 samples to the presence of  $\text{Cu}_5\text{Ca}$  intermetallic particles. These particles acted as effective obstacles to dislocation motion and promoted dislocation accumulation and rearrangement, which in turn facilitated the formation of new grain boundaries. In contrast, the BC-M40 and BC-90Q-M40 samples, being essentially single-phase, lack such second-phase particles and therefore exhibited comparatively less efficient grain subdivision. The higher fraction of HAGBs in the ternary alloys thus reflects more extensive grain subdivision and the formation of newly recrystallized grains during deformation. On the other hand, both the BC-90Q-M40 and TC-90Q-M40 samples exhibit a greater HAGB fraction than their non-heat-treated counterparts (BC-M40 and TC-M40). This suggests that the prior heat treatment contributed to a more favorable initial microstructure, which enhanced the effectiveness of subsequent MaxStrain processing. These results highlight the combined influence of alloy composition and pre-processing heat treatment on the development of refined microstructures, emphasizing their role in promoting the formation of HAGBs and, consequently, improved structural homogeneity.

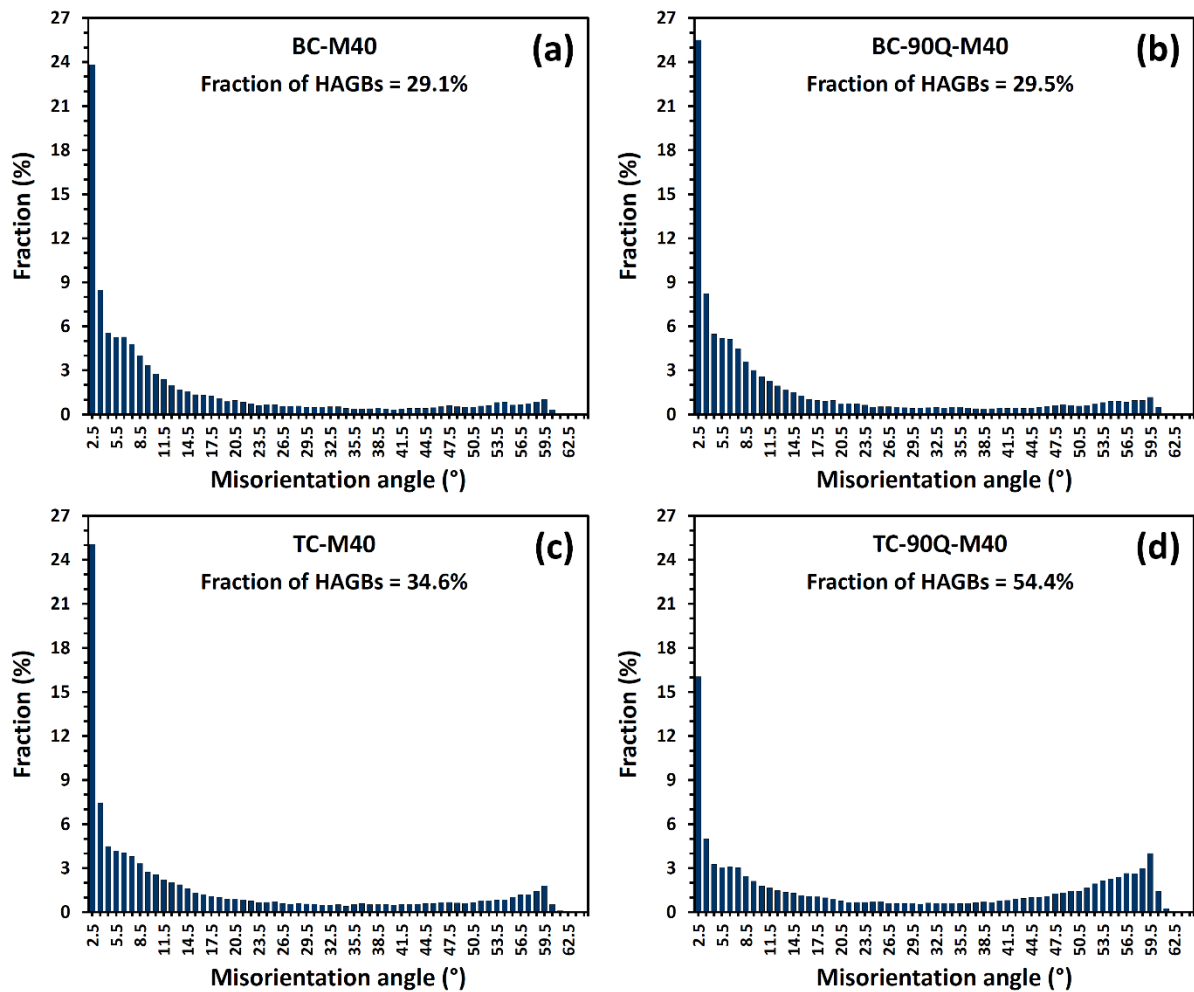


**Figure 35:** Distribution of misorientation angles (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, and (d) TC-90Q-M40 samples.

Now, building on the comparative results obtained for the binary and ternary alloys, the focus shifts to examining the influence of different processing conditions within the ternary alloy system. Given that the ternary alloy demonstrates a more pronounced microstructural response to thermomechanical processing, it has been deliberately selected for this comparative investigation. Figures 36(a) and 36(b) present the IPF maps obtained from EBSD data for the central transverse plane of the plastic deformation region in the TC-15A-M20 and TC-75Q-M20 specimens, respectively. To maintain consistency with previous analyses and facilitate a direct crystallographic comparison, the longitudinal direction is oriented perpendicular to the A2 direction (the pressing direction during even passes), and HAGBs are denoted by black lines. Given the limitations of EBSD in resolving second-phase particles, indexing was restricted to the Cu-Mg matrix; consequently, SEM analysis was utilized to characterize the morphology and distribution of Cu<sub>5</sub>Ca intermetallic particles. The results demonstrate significant grain refinement with increasing deformation: the average grain size decreased from

2.2  $\mu\text{m}$  in the TC-15A-M20 specimen to 1.6  $\mu\text{m}$  in the TC-75Q-M20 specimen. In particular, the EBSD results demonstrated that annealing at 650  $^{\circ}\text{C}$  for 75 min followed by quenching in agitated water prior to MaxStrain processing (route (ii)) resulted in a greater degree of grain refinement in the deformed specimen than annealing at the same temperature for 15 min followed by air cooling before MaxStrain processing (route (iii)). The IPF maps also reveal a heterogeneous microstructure comprising coarse elongated grains and fine equiaxed grains in both samples, which suggests the occurrence of partial CDRX during MaxStrain processing. However, as the deformation was performed at RT (which is also below the temperature typically required for DRX in Cu alloys [308,309]), limited atomic mobility and restricted grain boundary migration constrained the extent of CDRX and prevented complete microstructural transformation under the applied processing conditions.

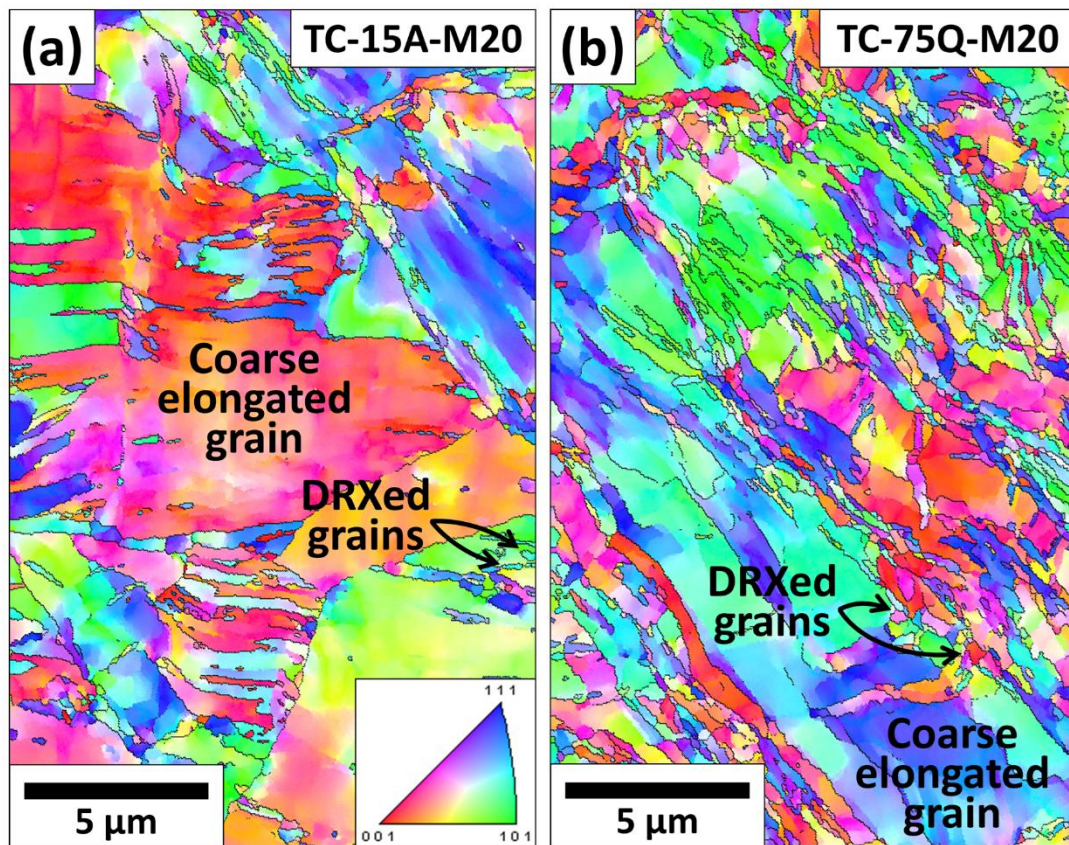


**Figure 36:** IPF maps of the TC-15A-M20 and TC-75Q-M20 samples.

The more pronounced grain refinement observed in the TC-75Q-M20 specimen relative to the TC-15A-M20 specimen is further evidenced by the GOS maps obtained from the EBSD data. Consistent with the methodology adopted in the previous analyses, a critical GOS threshold of  $2^{\circ}$  was used to differentiate recrystallized grains from deformed grains. [Figures 37\(a\)](#) and [37\(b\)](#) show the GOS maps of the TC-15A-M20 and TC-75Q-M20 specimens,

respectively, corresponding to the same regions presented in Figures 36(a) and 36(b). In these maps, grains with GOS values below  $2^\circ$  are highlighted in red, whereas grains with GOS values equal to or greater than  $2^\circ$  are outlined in blue, and LAGBs are marked by black lines. Accordingly, the red regions represent recrystallized grains, while the blue regions correspond to deformed grains. A comparison of the two specimens indicates that the TC-75Q-M20 sample contained a higher fraction of grains with GOS values below  $2^\circ$  than the TC-15A-M20 specimen. This finding suggests that annealing for 75 min followed by quenching was more effective in promoting the occurrence of CDRX during the subsequent MaxStrain deformation than annealing for 15 min followed by air cooling. It should, however, be emphasized that GOS measurements alone were not sufficient for a comprehensive assessment of grain refinement in the present study. Owing to the 20 cycles of repetitive deformation, grains that had recrystallized during the earlier stages of processing may have undergone subsequent deformation, leading to GOS values greater than  $2^\circ$ . Nevertheless, the fraction of grains with GOS values below  $2^\circ$  remained a reliable parameter for the comparative evaluation of the extent of recrystallization between the specimens.

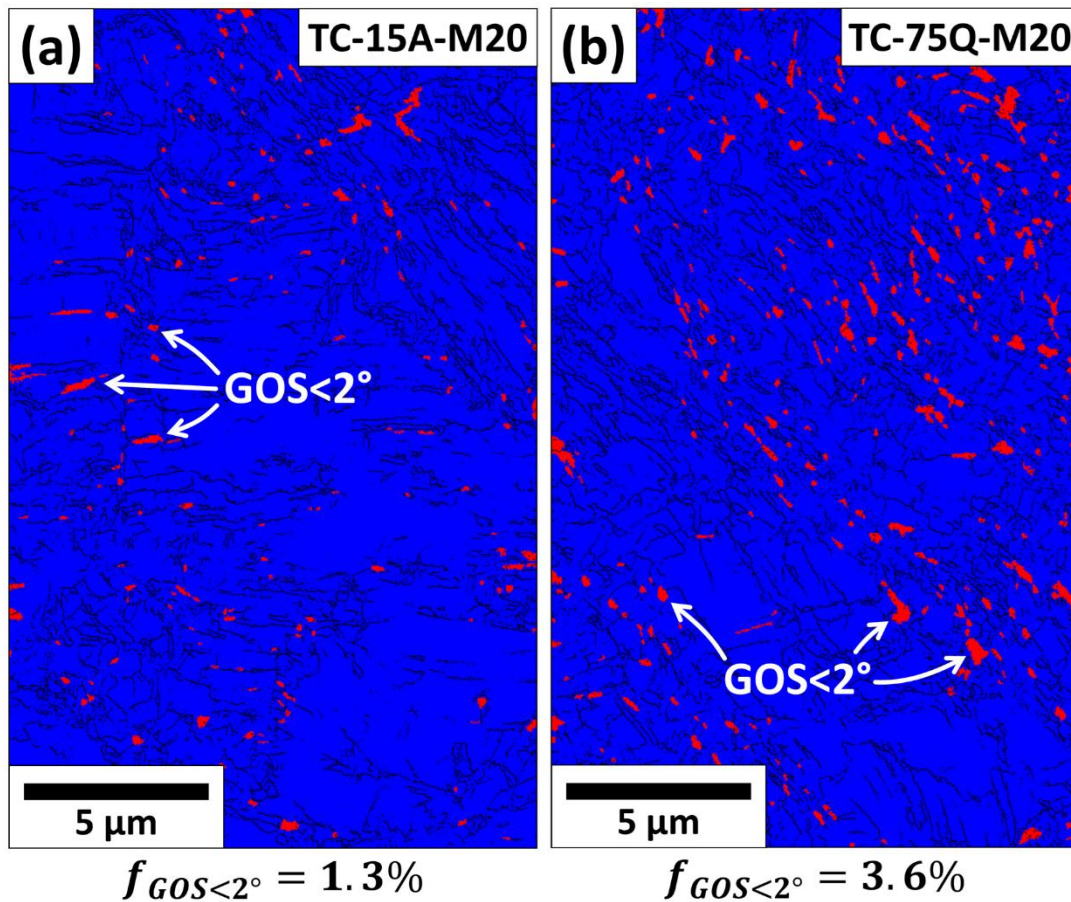


Figure 37: GOS maps of the TC-15A-M20 and TC-75Q-M20 samples.

Figure 38 presents the distribution of misorientation angles measured in the TC-15A-M20 and TC-75Q-M20 samples. As anticipated, comparison of these distributions reveals a higher fraction of HAGBs in the TC-75Q-M20 specimen than in the TC-15A-M20 specimen. This finding is consistent with the earlier discussion and indicates that annealing for 75 min followed by quenching in agitated water promotes more pronounced grain refinement after MaxStrain processing, compared with annealing for 15 min followed by air cooling. Furthermore, this trend is in full agreement with the observations reported for samples subjected to 40 passes of MaxStrain processing, which confirm the reproducibility of the effect across different deformation conditions.

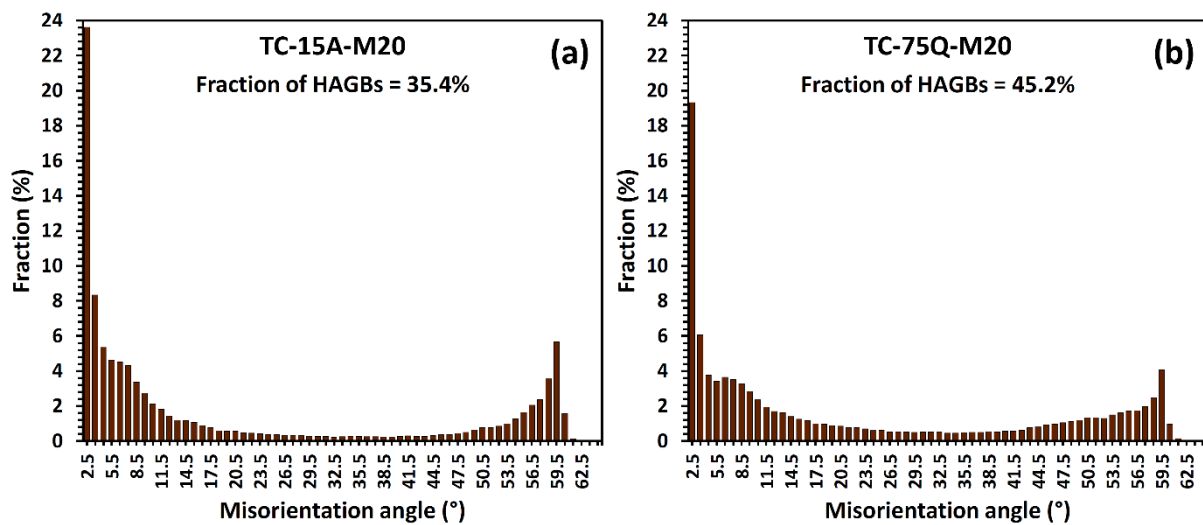


**Figure 38:** Distribution pattern of misorientation angles (a) TC-15A-M20, and (b) TC-75Q-M20 specimens.

As expected, it was also observed that the  $\text{Cu}_5\text{Ca}$  particles remained largely undeformed after 20 passes of MaxStrain deformation, which highlights their mechanical stability under the performed deformation conditions. However, some of these particles developed cracks as a result of the deformation, as indicated by the arrows in Figures 39(a) and 39(b) for the  $\text{Cu}_5\text{Ca}$  particles in the TC-15A-M20 and TC-75Q-M20 specimens, respectively.

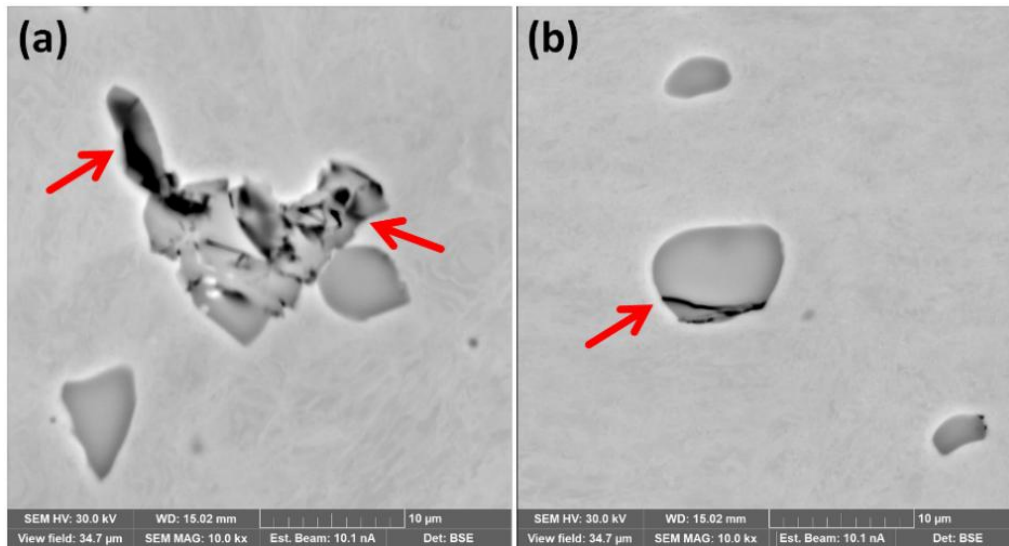


**Figure 39:** SEM-BSE images of  $\text{Cu}_5\text{Ca}$  intermetallic particles in (a) TC-15A-M20, and (b) TC-75Q-M20 specimens.

### 3.1.2.7. Mechanical Properties

The hardness test results revealed that the BC and BC-90Q samples exhibited average hardness values of  $60 \pm 2$  HV and  $69 \pm 3$  HV, respectively. Similarly, the TC and TC-90Q samples demonstrated average hardness values of  $80 \pm 4$  HV and  $82 \pm 8$  HV, respectively. These results indicate that the TC sample achieved an average hardness approximately 33% higher than that of the BC sample. This notable increase can be attributed to the presence of uniformly dispersed  $\text{Cu}_5\text{Ca}$  intermetallic particles within the microstructure of the TC sample. Owing to their inherently higher hardness compared with the Cu-Mg solid solution matrix, these intermetallic particles effectively impede plastic deformation and thereby enhance the overall hardness of the alloy. In addition, the results show that the heat treatment process led to a further increase in average hardness for both alloy systems. Specifically, the binary alloy exhibited an increase of approximately 15%, while the ternary alloy showed a more modest increase of about 2%. The relatively smaller hardness improvement in the ternary alloy may be associated with the presence of pre-existing strengthening phases prior to heat treatment. The increased hardness observed in the heat-treated samples can be primarily attributed to a higher dislocation density generated during quenching, as well as the development of residual thermal stresses within the material, which, together, contributed to enhanced hardness. Likewise, the TC-15A and TC-75Q specimens exhibited mean hardness values of  $78 \pm 4$  HV and  $81 \pm 4$  HV, respectively, which indicate that annealing at 650 °C for 75 min followed by quenching in agitated water resulted in an ~4% increase in mean hardness compared with annealing for 15 min followed by air cooling.

For the MaxStrain-processed samples, it was not practical to determine a single representative average hardness value due to the pronounced strain inhomogeneity observed across the samples, as illustrated in [Figures 29 to 31](#). To account for this variation, hardness measurements were systematically performed along the primary A1 and A2 directions marked in [Figures 30 and 31](#). The indentations were made in the central transverse plane of the samples at regular intervals of 0.45 mm to allow for a detailed assessment of the spatial variation in hardness. The hardness values obtained from these measurements are presented in [Figure 40](#). It can be seen that the hardness distribution in all MaxStrain-processed samples generally resembled the equivalent strain distribution predicted by the simulations, irrespective of the number of passes or the alloy composition. This agreement suggests that the simulated model reasonably captured the spatial variation of plastic deformation during processing. Specifically, the highest hardness values were consistently observed at the center of the deformation zone, whereas comparatively lower values were recorded in the intermediate, edge, and corner regions of the samples. This behavior can be primarily attributed to the higher equivalent strain experienced in the central region, which promoted a more pronounced strain-hardening effect. In fact, the intensified plastic deformation in the center of the deformation zone increased dislocation density and enhanced dislocation interactions, restricted their mobility, and contributed to higher local hardness. In contrast, the lower strain levels in the middle, edge, and corner regions resulted in less pronounced microstructural refinement and, consequently, comparatively lower hardness.

Another noteworthy observation that warrants explanation is that in the ternary alloy samples the presence of  $\text{Cu}_5\text{Ca}$  intermetallic particles locally influenced the hardness distribution due to the inherent microstructural heterogeneity of the material. Since these intermetallic particles act as obstacles to dislocation motion and contribute to strengthening through particle-matrix interactions (such as load transfer and constrained plastic deformation), they affected the local mechanical response during indentation. As a result, indentations that interacted with or included these intermetallic phases exhibited locally elevated hardness values compared to the surrounding matrix. This effect can be explicitly seen in [Figure 40\(d\)](#) in hardness measurements in the A2 direction.

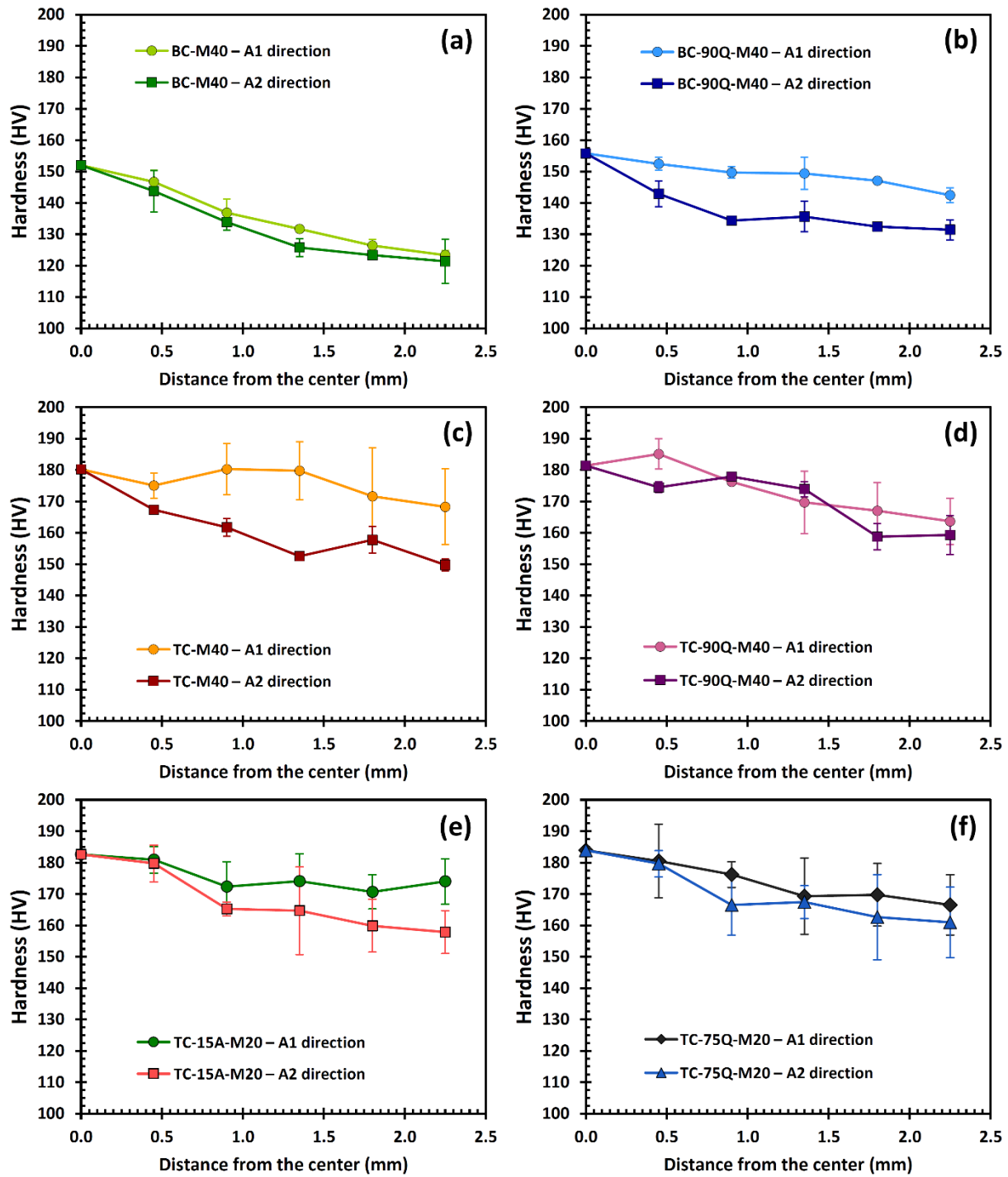


**Figure 40:** Hardness measurement results for (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, (d) TC-90Q-M40, (e) TC-15A-M20, and (f) TC-75Q-M20 samples.

The tensile test results for samples subjected to 40 passes of MaxStrain processing are presented in Figure 41. It must be mentioned that due to the small dimensions of the miniaturized specimens used in these tests, the use of an extensometer for direct displacement measurement was not feasible. Instead, the lengths of the fractured specimen segments (later illustrated in Figure 43) were measured individually and then summed to obtain the total

change in length ( $\Delta L$ ). This value was subsequently divided by the initial gauge length (2.5 mm) to determine the standard engineering strain ( $\Delta L/L_0$ ), which is reported as the EL value for each sample.

An initial assessment of the tensile test data indicates that the TC sample exhibited YS and UTS values that were both approximately 13% higher than those of the BC sample. However, this increase in strength was accompanied by a reduction in ductility, with the EL of the TC sample being about 15% lower. A similar trend was observed for the heat-treated samples, where the TC-90Q sample demonstrated an 11% increase in YS and a 15% increase in UTS compared to the BC-90Q sample, while its EL was reduced by approximately 13%. The observed enhancement in strength for the ternary alloy samples can be primarily attributed to the presence of insoluble  $\text{Cu}_5\text{Ca}$  intermetallic particles within the microstructure. These particles remained stable and largely undistorted during deformation and served as effective barriers to dislocation motion. By interrupting the continuous glide of dislocations within the Cu-Mg matrix, the intermetallic particles increased the resistance to plastic deformation. As a result, a higher applied stress was required to deform the ternary alloys, which led to the observed increase in strength relative to the corresponding binary samples. Chen et al. [310] also observed a comparable effect in an as-cast Cu-0.32La (wt.%) alloy. In their TEM analysis of the tensile specimens, they identified a large amount of dislocation lines surrounding the intermetallic  $\text{Cu}_6\text{La}$  particles, along with substantial dislocation pile-ups in the matrix. They explicitly reported that these  $\text{Cu}_6\text{La}$  intermetallic particles effectively inhibited the motion of dislocations, acting through second-phase (dispersion) strengthening.

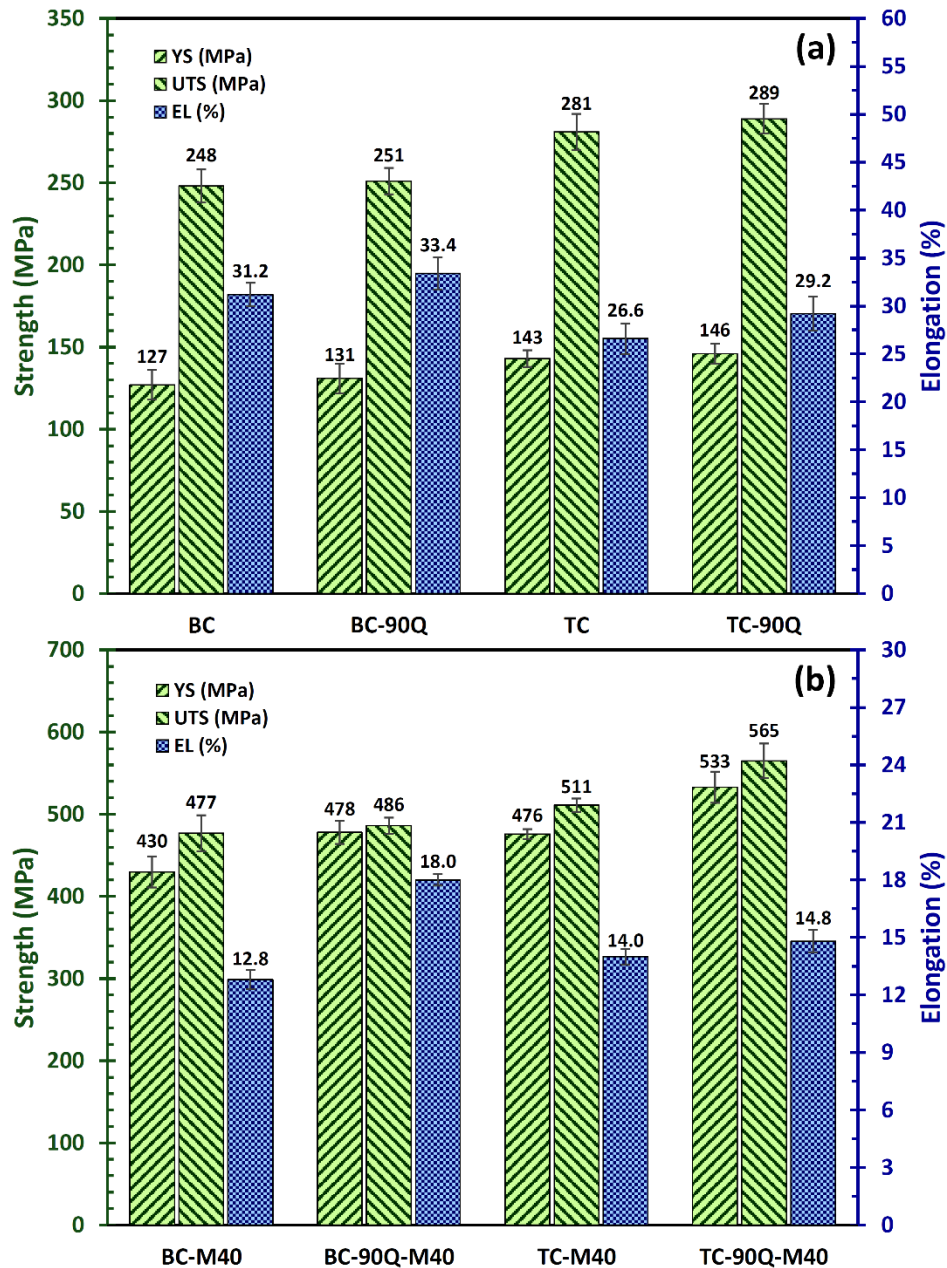


**Figure 41:** Tensile test data obtained for samples subjected to 40 passes of MaxStrain processing and their corresponding initial counterparts.

Tensile test results also show that the heat treatment process also slightly enhanced the YS, UTS, and EL of both binary and ternary alloys, with a more pronounced effect observed in the ternary alloy. The increased YS and UTS were the result of the presence of residual thermal stresses and the higher dislocation density within the alloys, which were induced by the annealing and quenching process. Specifically, in the case of the TC-90Q sample, attention should also be given to the difference in the CTE values of the Cu-Mg matrix and Cu<sub>5</sub>Ca intermetallic particles. Upon heating the TC sample up to 650 °C, both the Cu-Mg matrix and Cu<sub>5</sub>Ca particles experienced volume expansion. Due to the difference in CTE values of the

matrix and Cu<sub>5</sub>Ca particles, the dislocation density increased in the vicinity of Cu<sub>5</sub>Ca particles at the beginning of the annealing process, and after 90 min, it reached a new equilibrium state. However, upon quenching the alloy to RT, in which both the Cu-Mg matrix and Cu<sub>5</sub>Ca particles underwent thermal shrinkage, a non-equilibrium volume change occurred, which resulted in the generation of new dislocations in the vicinity of Cu<sub>5</sub>Ca particles in the TC-90Q sample to maintain the integrity of the alloy. This higher dislocation density in the TC-90Q sample resulted in its better mechanical properties compared to the TC sample.

Notably, the increase in EL of the BC-90Q sample attained by the heat treatment process can be attributed to the homogenization of the as-cast microstructure that took place through the annealing treatment, which resulted in a more homogeneous distribution of Mg atoms inside the Cu-Mg matrix and improved the ductility. In the case of the TC-90Q sample, the increase in EL could also be attributed to the partial spheroidization of Cu<sub>5</sub>Ca particles that was shown in [Figure 25](#). Although spheroidization remained partial due to the relatively short annealing time, it still provided satisfactory results by reducing stress concentration sites at the edges of these particles during deformation. To be more specific, the sharp edges of the Cu<sub>5</sub>Ca particles in the TC sample served as stress concentration points during deformation, promoting crack formation and reducing the ductility of the material. However, this issue was solved in the TC-90Q sample via the annealing process, which partially transferred the irregular-shaped Cu<sub>5</sub>Ca particles into more spherical-shaped ones, and resulted in a more uniform stress distribution around these particles during the tensile test, which showed its effect on the better EL of the material.

After MaxStrain processing, the YS of the BC, BC-90Q, TC, and TC-90Q samples increased significantly by 239%, 265%, 233%, and 265%, respectively. Similarly, the UTS exhibited substantial improvements of 92%, 94%, 82%, and 96% for the same samples. This behavior is characteristic of deformation-induced strengthening in metallic materials and has been extensively documented in both pure Cu as well as Cu-based alloys [311–314]. During the MaxStrain process, repeated deformation generated a high density of dislocations within the severely deformed grains, which led to pronounced dislocation hardening. In addition, the process promoted grain refinement through the occurrence of partial CDRX, which increased the fraction of HAGBs. The higher grain boundary density effectively impeded dislocation motion and enhanced resistance to plastic deformation and, consequently, contributed to the overall strengthening of the alloys. However, MaxStrain processing also leads to a reduction in EL, with decreases of 59%, 46%, 47%, and 49% observed in the BC-M40, BC-90Q-M40,

TC-M40, and TC-90Q-M40 samples compared to their pre-MaxStrain counterparts, respectively.

The tensile test results for the samples subjected to 20 passes of MaxStrain processing, together with their initial counterparts, are presented in [Figure 42](#). For the initial condition, the TC-75Q specimen exhibited a 5% higher YS, a 4% higher UTS, and a 6% greater EL compared with the TC-15A specimen. The improvements in strength are attributed to the higher dislocation density and the presence of residual thermal stresses in the TC-75Q material relative to TC-15A. In addition, the slightly higher ductility of TC-75Q is associated with its more homogeneous microstructure and the reduced morphological irregularity of the Cu<sub>5</sub>Ca particles. In particular, the irregularly shaped Cu<sub>5</sub>Ca particles in TC-15A acted as stress concentration sites during deformation and promoted earlier crack initiation. In contrast, the partially spheroidized particles in TC-75Q facilitated a more uniform stress distribution under tensile loading and resulted in a modest increase in EL. Same as previous observations, a pronounced enhancement in tensile properties was observed for both materials following MaxStrain processing. Compared with their initial states, the TC-15A-M20 and TC-75Q-M20 specimens exhibited increases in YS of 229% and 232% and in UTS of 86% and 91%, respectively.

Nevertheless, these improvements were achieved at the cost of reductions in elongation of 48% for TC-15A-M20 and 44% for TC-75Q-M20. The substantial strengthening can be linked to the processing-induced microstructural evolution. During MaxStrain processing, severe cyclic deformation introduced a high density of dislocations, particularly in regions adjacent to Cu<sub>5</sub>Ca intermetallic particles. Concurrently, CDRX promoted grain refinement and transformed the initially coarse grains into a finer structure and increased the fraction of HAGBs. The combined effects of dislocation accumulation and grain boundary strengthening effectively impeded dislocation motion and, consequently, led to a significant increase in strength.

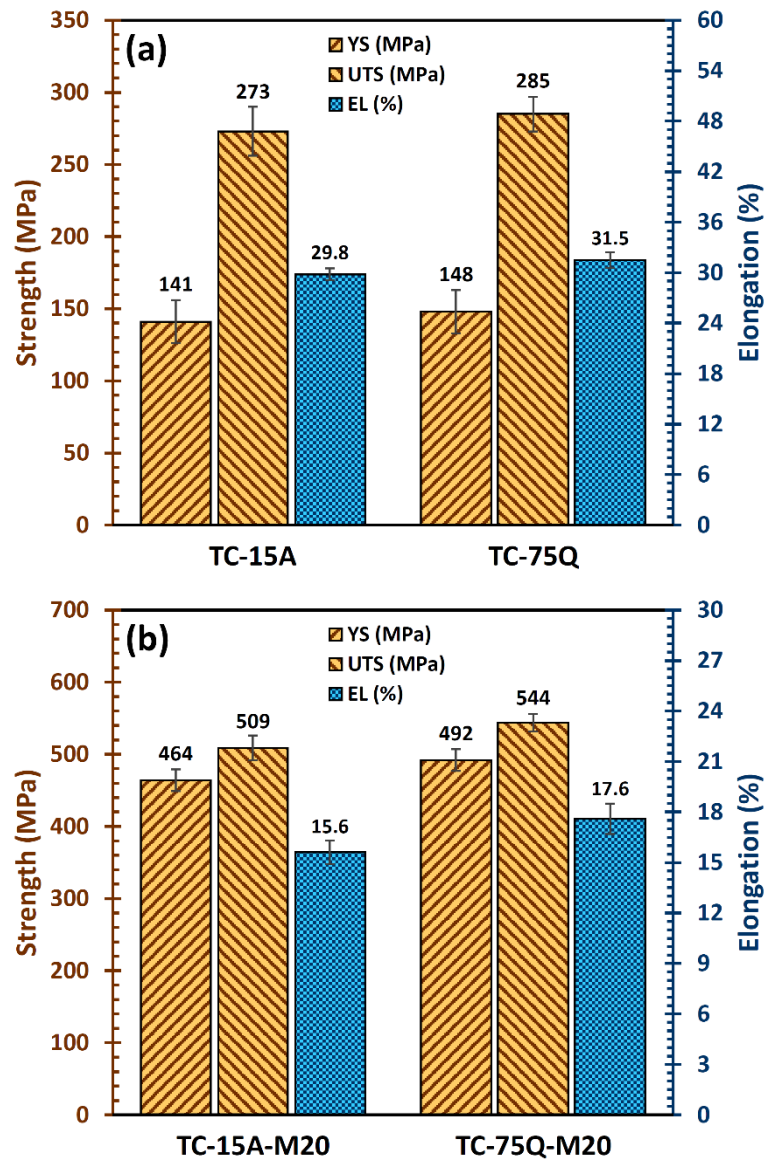


**Figure 42:** Tensile test data obtained for samples subjected to 20 passes of MaxStrain processing and their corresponding initial counterparts.

### 3.1.2.8. Fractography

Figures 43(a) and 43(e) respectively show the broken pieces of the BC-M40 sample and its corresponding fracture surface. This sample exhibited a moderate level of EL of 12.8%, which is a typical EL value observed in SPD processed Cu alloys [15]. Figures 43(b) and 43(f) respectively show the broken pieces and the fracture surface of the BC-90Q-M40 sample. Notably, the BC-90Q-M40 sample exhibited a better EL value of 18.0%, which can be attributed to the homogenization of the initial as-cast microstructure, achieved through the annealing process. The presence of larger and deeper dimples on the fracture surface of the BC-90Q-M40 sample relative to the BC-M40 sample indicates its enhanced elongation compared to the BC-M40 sample [315].

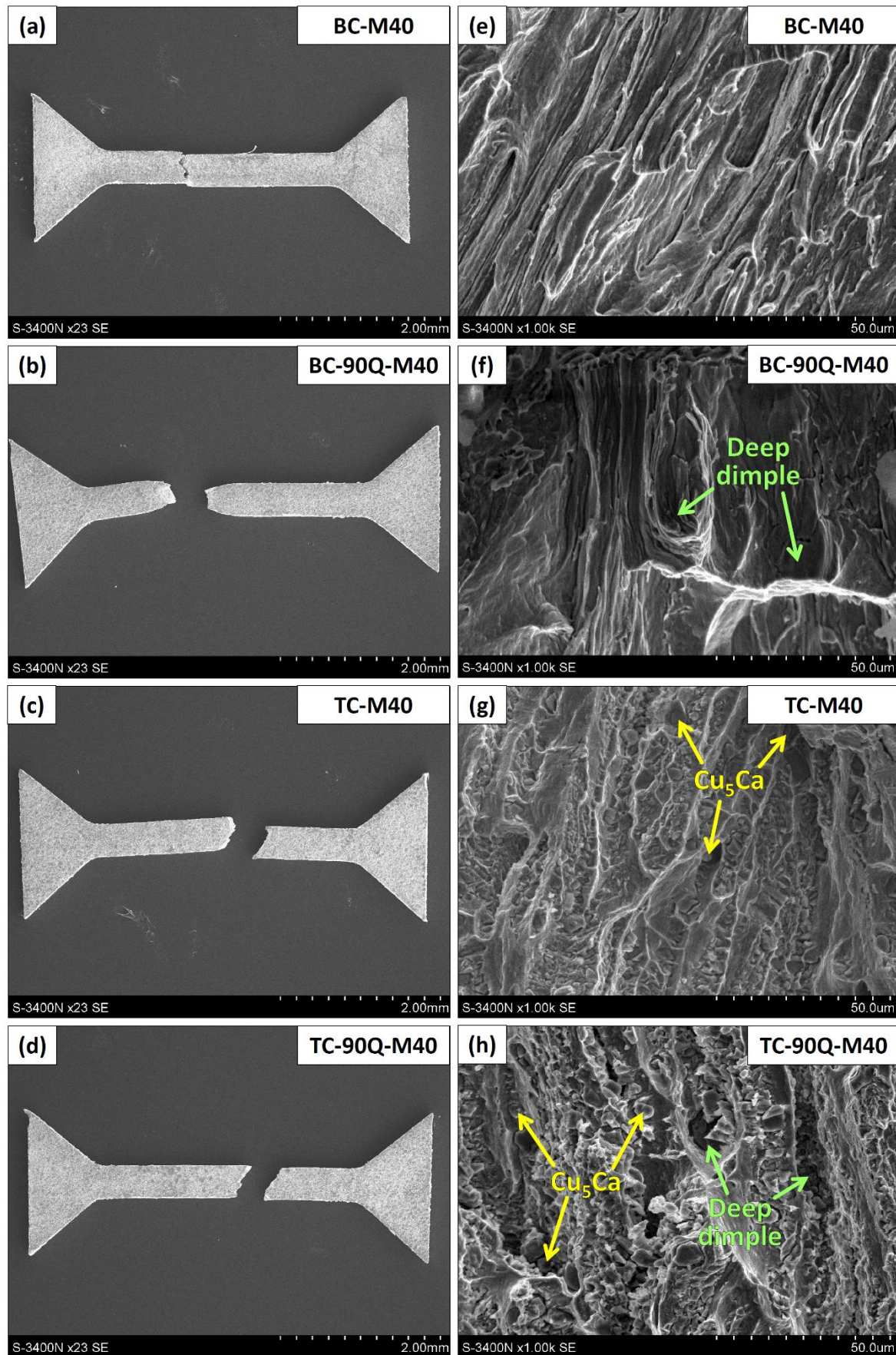


**Figure 43:** Broken pieces of (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, and (d) TC-90Q-M40 samples; fracture surfaces of (e) BC-M40, (f) BC-90Q-M40, (g) TC-M40, and (h) TC-90Q-M40 samples.

Likewise, both TC-M40 and TC-90Q-M40 samples displayed intermediate values of 14.0% and 14.8%, respectively, which are comparable to previously reported EL values in SPD processed Cu-Mg-Ca alloys [42]. Similar to the binary samples, the presence of some deeper dimples on the fracture surface of the TC-90Q-M40 sample indicates its better elongation compared to the TC-M40 sample. Here, an important observation is the presence of nearly undeformed Cu<sub>5</sub>Ca intermetallic particles within dimples in both the TC-M40 and TC-90Q-M40 samples, as evident in [Figures 43\(g\) and 43\(h\)](#). This observation aligns with the SEM findings regarding the Cu<sub>5</sub>Ca intermetallic particles ([Figure 39](#)), which showed that these particles remained undeformed after undergoing the MaxStrain processing, but some cracks were formed within them.

The broken fragments and the fracture surface of the TC-15A-M20 specimen are respectively shown in [Figures 44\(a\) and 44\(c\)](#). This specimen demonstrated a moderate level of elongation to failure of 15.6%, which is in the usual range of the typical elongation value recorded for SPD processed Cu-Mg alloys [33,42,316]. Likewise, [Figures 44\(b\) and 44\(d\)](#) respectively present the broken pieces together with the fracture surface of the TC-75Q-M20 specimen, which exhibited a slightly better elongation value of 17.6%, which is attributable to the relatively better homogenization of the initial as-cast microstructure accomplished through a longer annealing process compared to the TC-15A-M20 specimen. The heterogeneous dimple morphology observed on the fracture surfaces of both specimens is consistent with their microstructural heterogeneity and is characteristic of a ductile fracture mode governed by microvoid nucleation, growth, and coalescence. Larger dimples are often associated with regions containing coarser microstructural features and more pronounced localized plastic deformation, whereas smaller dimples are typically observed in finer, recrystallized regions that deform more uniformly. Furthermore, nearly undeformed but partially fragmented Cu<sub>5</sub>Ca intermetallic particles are present within dimples across the fracture surfaces. These particles likely served as preferential sites for void nucleation during deformation, either through particle fracture or particle-matrix decohesion and, consequently, contributed to the development of the characteristic dimpled fracture morphology.

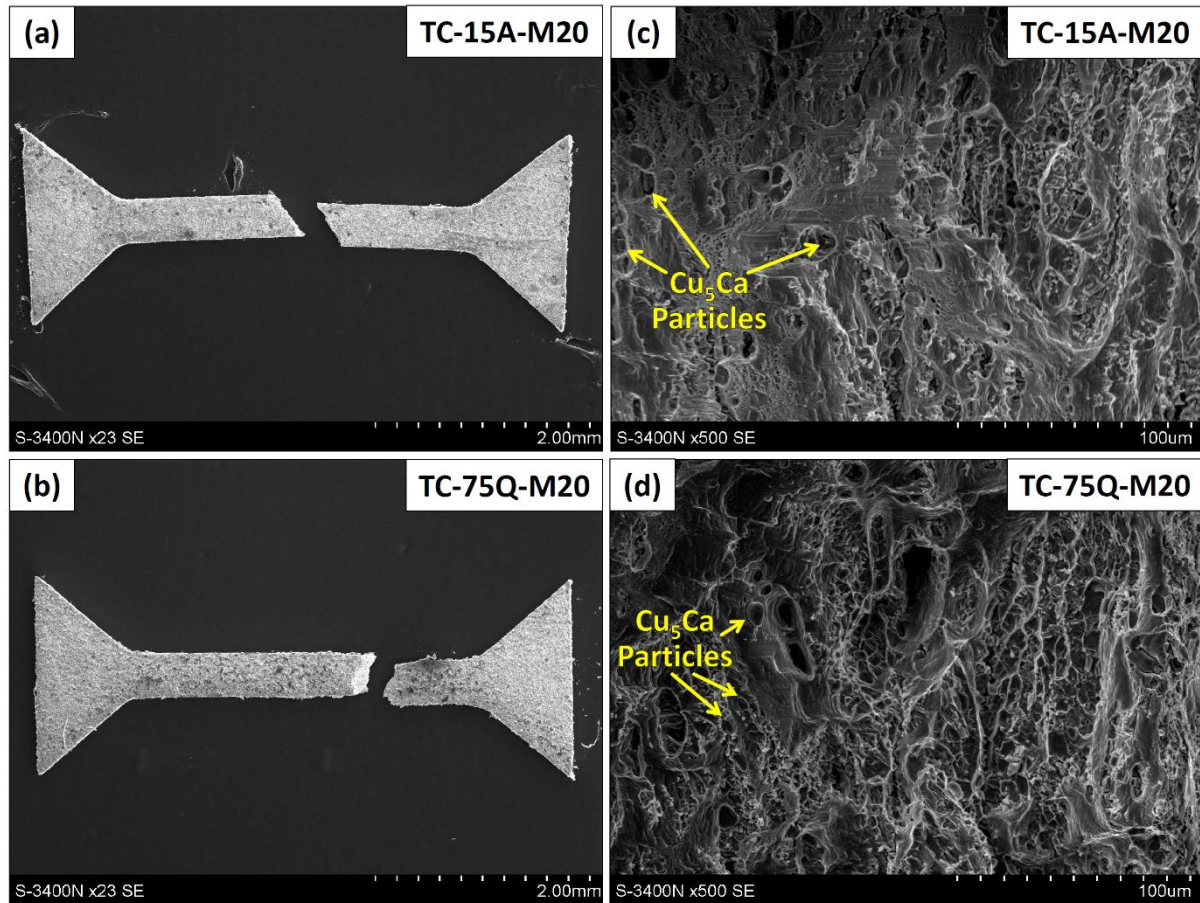


**Figure 44:** The fractured pieces of (a) TC-15A-M20, and (b) TC-75Q-M20 specimens; fracture surface of (c) TC-15A-M20, and (d) TC-75Q-M20 specimens.

### 3.1.2.9. Electrical Conductivity

The electrical conductivity results for samples that were exposed to 40 passes of MaxStrain process are presented in Figure 45. The results show that the BC sample exhibited a conductivity of  $39.1 \pm 2.4$  MS/m (67.4% IACS), which decreased to  $37.5 \pm 2.7$  MS/m (64.7% IACS) after heat treatment. In contrast, the TC sample demonstrated an electrical conductivity of  $33.9 \pm 2.3$  MS/m (58.4% IACS), which remained unchanged after heat treatment ( $33.9 \pm 2.4$  MS/m), suggesting a stable electrical response to the heat treatment. The fact that all investigated alloys displayed significantly lower electrical conductivity than pure Cu comes from the detrimental influence of Mg-induced solid solution strengthening on electron transport due to increased lattice distortion and electron scattering. On the other hand, a clear distinction can be observed between the electrical conductivity values in binary and ternary alloys. The TC and TC-90Q samples exhibited electrical conductivities approximately 13% and 10% lower, respectively, than those of the BC and BC-90Q samples, which clearly highlights the adverse effect of Ca addition on the electrical conductivity. In fact, this reduction can be

attributed to the presence of Cu<sub>5</sub>Ca intermetallic particles, which act as additional electron-scattering centers and disrupt the continuity of the conductive Cu-Mg matrix. Consequently, the combined effects of solute atoms and intermetallic phases impeded the free movement of electrons and led to a further decrease in electrical conductivity in the ternary samples compared to the binary samples. The approximately 4% reduction in electrical conductivity observed for the BC sample after heat treatment can be primarily attributed to the higher dislocation density in the BC-90Q sample compared with the BC condition. In contrast, for the ternary alloy, the effect of heat treatment on electrical conductivity was relatively minor when compared with the pronounced detrimental influence of Cu<sub>5</sub>Ca intermetallic particles. This finding indicates that, in Cu-Mg-Ca alloys, microstructural features associated with intermetallic phases play a more dominant role in governing electrical conductivity than heat-treatment-induced changes in defect density. Consequently, annealing these alloys at temperatures below the dissolution threshold of the dissolution of intermetallic phases, followed by quenching, may represent an effective approach to improving mechanical properties while largely preserving electrical conductivity.

After MaxStrain processing, the electrical conductivity values were measured as  $37.6 \pm 2.6$  MS/m (64.8% IACS) for the BC-M40 sample,  $34.5 \pm 2.4$  MS/m (59.5% IACS) for the BC-90Q-M40 sample,  $33.5 \pm 2.1$  MS/m (57.8% IACS) for the TC-M40 sample, and  $33.2 \pm 2.2$  MS/m (57.2% IACS) for the TC-90Q-M40 sample. These values represent reductions of approximately 4%, 8%, 1%, and 2%, respectively, relative to their corresponding initial conditions. The observed decrease in electrical conductivity can be primarily attributed to microstructural changes induced by the MaxStrain process. In particular, MaxStrain processing led to a higher fraction of HAGBs and introduced a substantial density of lattice defects, including dislocations and point defects. These microstructural features acted as effective electron-scattering centers and impeded electron transport within the Cu-Mg matrix. As a result, the increased scattering of conducting electrons led to a measurable reduction in electrical conductivity [317]. However, it can also be seen that this reduction in electrical conductivity was considerably less pronounced than the detrimental effect associated with Mg-induced solid solution strengthening. Moreover, the conductivity decrease resulting from MaxStrain processing was smaller in the ternary alloys than in the binary alloys, which indicate that the presence of Cu<sub>5</sub>Ca intermetallic particles in the ternary systems exerts a more significant adverse influence on electrical conductivity than the MaxStrain processing itself.

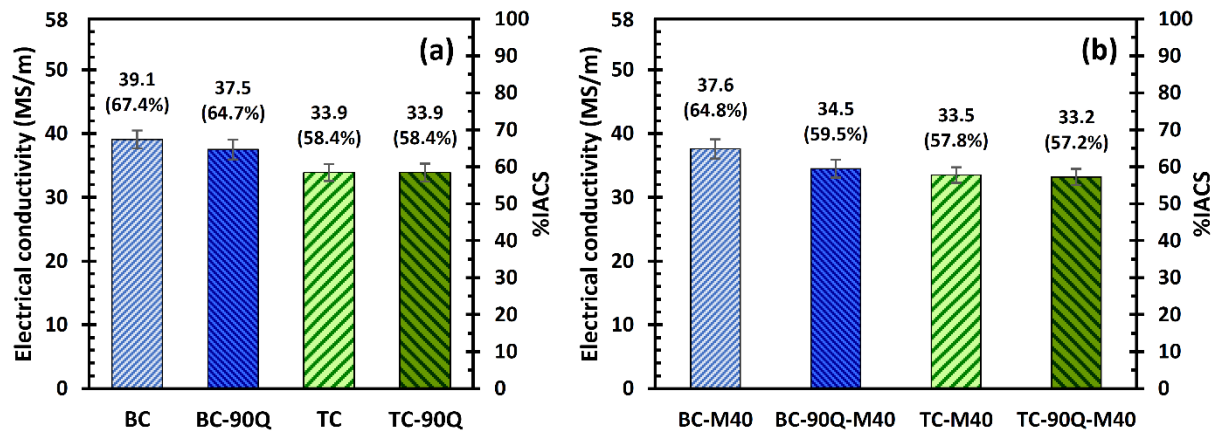


**Figure 45:** Electrical conductivity values for the binary and ternary samples (a) before and (b) after the exposure to 40 passes of MaxStrain processing.

Regarding the samples exposed to 20 passes of MaxStrain processing, according to the values recorded as the electrical conductivity of the specimens represented in Figure 46, the TC-15A specimen possessed a conductivity of  $33.9 \pm 2.6$  MS/m (58.4% IACS), the same as the value for the TC-75Q specimen ( $33.9 \pm 2.1$  MS/m). As discussed above, the fact that the conductivities of both the TC-15A and TC-75Q specimens are 41.6% lower compared to annealed pure Cu (58 MS/m [21]) is the result of the Mg-induced solid solution as well as the presence of  $\text{Cu}_5\text{Ca}$  intermetallic particles in their microstructure. Moreover, the identical electrical conductivity values for the TC-15A and TC-75Q specimens indicate that in this case, the electrical conductivity was independent of both annealing duration and cooling rate. The results also show that the application of MaxStrain processing has led to an approximately 1% reduction in the conductivities of the specimens, as evidenced by the conductivity value of the TC-15A-M20 specimen measured  $33.6 \pm 1.9$  MS/m (57.9% IACS), and the TC-75Q-M20 specimen recorded  $33.5 \pm 2.2$  MS/m (57.8% IACS). Same as the discussion above, this can be attributed to the introduction of a higher percentage of HAGBs and crystallographic defects, including point defects and dislocations, within both the TC-15A-M20 and TC-75Q-M20 specimens during the MaxStrain processing, which contributed to a more pronounced electron scattering within these specimens and their lower electrical conductivity [318].

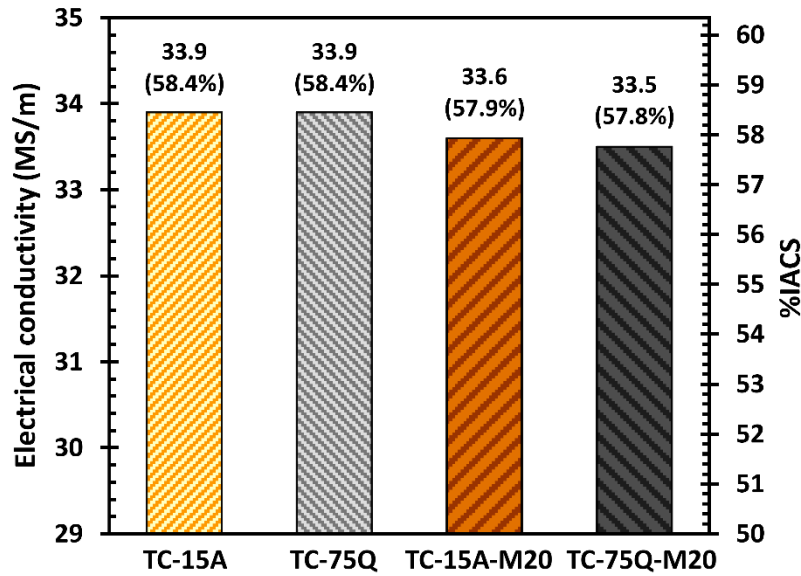


**Figure 46:** Measured electrical conductivity values of the samples prior to and following exposure to 20 passes of MaxStrain processing.

Figure 47 presents the UTS vs. electrical conductivity values for the samples fabricated and investigated in this approach. The chart also indicates the minimum threshold values of UTS and electrical conductivity, as proposed by Yang et al. [15] required for the classification of HSHC Cu alloys. Based on the obtained results, although the initial samples do not satisfy these criteria, all MaxStrain-processed samples (both 20-pass and 40-pass conditions) exceed the proposed thresholds and can therefore be considered to meet the criteria for HSHC Cu alloys. This outcome indicates the effectiveness of MaxStrain processing in enhancing the mechanical strength of the alloys while maintaining electrical conductivity within the specified limits. Moreover, the combined improvement in strength and conductivity suggests the potential of MaxStrain processing as a viable approach for developing Cu-based alloys with a favorable balance of mechanical and functional properties.

Nevertheless, it is important to emphasize that, while MaxStrain processing represents a powerful research tool for systematically investigating the influence of a wide range of processing parameters on the microstructure, mechanical behavior, and physical properties of metallic materials, its current application is largely limited to experimental studies and research purposes rather than industrial implementation. In laboratory settings, this technique is particularly valuable because it allows precise control over deformation conditions and enables detailed investigation of how processing history affects material response. More specifically, previous studies have used MaxStrain processing across a wide range of metals and alloys to better understand key underlying mechanisms such as grain refinement, recrystallization

behavior, dislocation generation and evolution, and defect interactions [319–327]. These mechanisms play a key role in determining the final properties of metallic materials. As a result, the method enables researchers to establish fundamental structure-property relationships that are often difficult to isolate or study using conventional metal forming techniques.

However, from an industrial perspective, the practical implementation of MaxStrain processing has not been noticeably reported. In fact, its inherently time-intensive nature, discontinuous processing sequence, and limitations in sample size introduce significant challenges for large-scale production, where high throughput, process reproducibility, and scalability are critical requirements. These constraints make direct industrial translation difficult under current conditions. Consequently, it can be concluded that while the MaxStrain process offers significant advantages for scientific exploration and alloy development at the laboratory scale, bridging the gap between laboratory research and industrial application will likely require the development of adapted or hybrid processing strategies. Such approaches would need to preserve the beneficial microstructural effects observed in experimental studies while simultaneously meeting the efficiency, cost, and scale demands of industrial manufacturing.

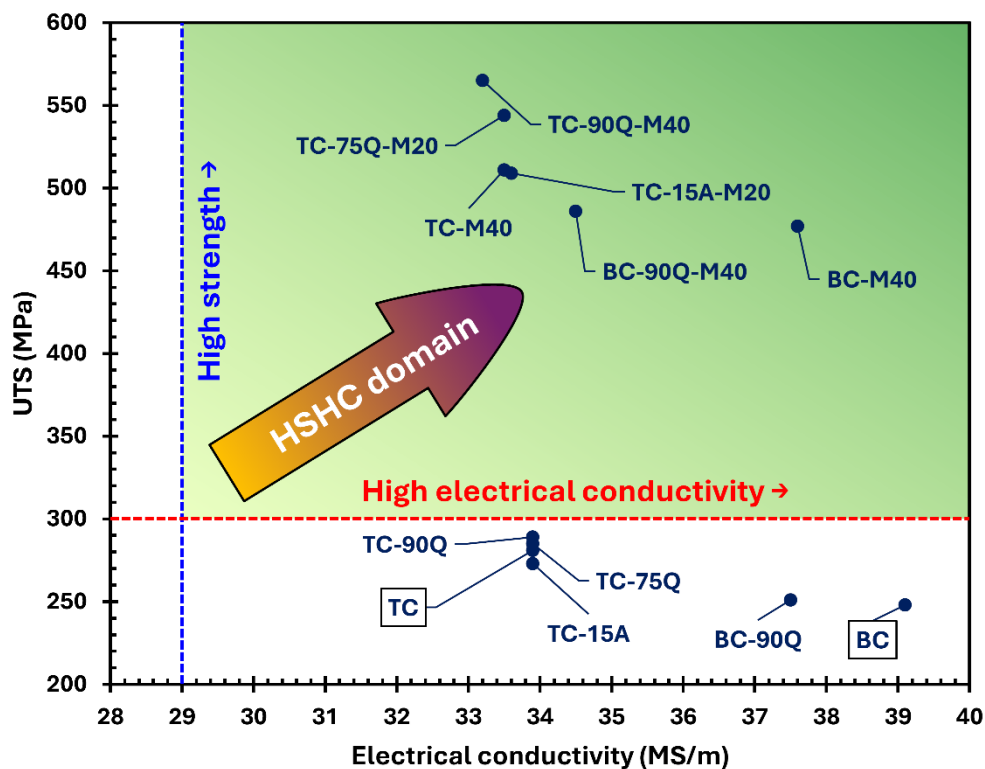


**Figure 47:** UTS versus electrical conductivity values for the investigated samples in Approach 1.

### 3.1.3. Conclusions of Approach 1

In summary, this approach investigated the effects of Ca addition, heat treatment, and SPD processing using the MaxStrain module of the Gleeble thermomechanical simulator on the microstructural features, mechanical properties, and electrical conductivity of Cu-0.7Mg (wt.%) alloy. Based on the obtained results, the following conclusions can be drawn:

- 1.1. The addition of 0.1 wt.% Ca to the Cu-0.7Mg (wt.%) solid solution led to the formation of  $\text{Cu}_5\text{Ca}$  intermetallic particles, which were uniformly distributed both along the grain boundaries as well as inside the grains. The presence of these particles promoted a transformation of the as-cast columnar macrostructure into a predominantly equiaxed grain structure.
- 1.2. The TC sample exhibited a 33% increase in hardness, a 13% increase in YS, and a 13% increase in UTS compared to the BC sample, which was attributed to the strengthening effect of the  $\text{Cu}_5\text{Ca}$  intermetallic particles present within its microstructure. However, it possessed a 15% reduced EL and a 13% decreased electrical conductivity relative to the BC sample, which highlights the trade-off between enhanced mechanical strength and diminished ductility and conductivity.
- 1.3. A heat treatment consisting of annealing at 650 °C for 90 min followed by water quenching resulted in a slight increase in both YS and UTS for both BC and TC samples, which was attributed to the combined effects of thermally induced residual stresses and an increased dislocation density within their resulting microstructures. In addition, the EL value of both samples showed a modest improvement, mainly due to the homogenization of the as-cast microstructure and, in the case of the TC sample, partial spheroidization of the  $\text{Cu}_5\text{Ca}$  intermetallic particles. However, this heat treatment led to a 4% decrease in the electrical conductivity of the BC sample, which indicates the trade-off between mechanical strengthening and electrical performance.
- 1.4. Annealing at 650 °C for 75 min followed by quenching in agitated water promoted a more homogeneous distribution of Mg atoms within the single-phase Cu-Mg matrix, induced partial spheroidization of  $\text{Cu}_5\text{Ca}$  intermetallic particles, and generated residual thermal stresses. In comparison with the sample annealed at 650 °C for 15 min and then air-cooled, these combined effects resulted in increases of approximately 4% in hardness, 5% in YS, 4% in UTS, and 6% in elongation.
- 1.5. The simulation results revealed a non-uniform distribution of equivalent strain inside the samples after MaxStrain processing. This strain gradient directly influenced the hardness

profile, with the maximum hardness achieved at the center, where the equivalent strain was highest, and gradually decreasing toward the edges.

- 1.6. Following MaxStrain processing, the ternary samples exhibited finer grain structures with a higher fraction of HAGBs compared to the binary samples. This refinement was attributed to the presence of Cu<sub>5</sub>Ca intermetallic particles within their microstructure, which effectively promoted CDRX and consequently increased the proportion of CDRXed grains. Furthermore, in both binary and ternary alloys subjected to 40 passes of MaxStrain processing, a smaller grain size was achieved in their heat-treated states compared to their as-cast condition, which was ascribed to the presence of residual thermal stresses as well as a higher dislocation density in the heat-treated samples, which facilitated further grain subdivision during MaxStrain processing.
- 1.7. 20 passes of MaxStrain processing also led to a substantial enhancement in YS, with the TC-15A-M20 and TC-75Q-M20 specimens exhibiting increases of 229% and 232%, respectively, relative to their non-deformed counterparts (TC-15A and TC-75Q). UTS was also remarkably improved, increasing by 86% for TC-15A-M20 and 91% for TC-75Q-M20. However, these strength gains were accompanied by a reduction in ductility, as evidenced by decreases in elongation of 48% for TC-15A-M20 and 44% for TC-75Q-M20.
- 1.8. The electrical conductivity of the ternary samples was consistently lower than that of their binary counterparts in all processing conditions. Furthermore, the MaxStrain-processed samples exhibited a slight reduction in electrical conductivity relative to their initial conditions, which was attributed to the increased fraction of HAGBs and the elevated density of defects. Nevertheless, all MaxStrain-processed samples, whether subjected to either 20 or 40 deformation passes, retained conductivity values exceeding the minimum threshold required for classification as HSHC Cu alloys.

## 3.2. Approach 2: Combination of Different Deformation Methods

By adopting a different strategy from the previous approach, which involved the incorporation of intermetallic particles into the microstructure, the present approach focused on enhancing the mechanical properties of a single-phase Cu-Mg solid solution through the integration of conventional deformation techniques, such as forward extrusion, with SPD methods including KoBo extrusion and MaxStrain processing. Therefore, no additional alloying elements were introduced into the Cu-Mg system to ensure that any observed changes in properties can be attributed exclusively to the applied deformation processes. A detailed account of the experimental procedures, along with the corresponding results and discussion, is presented in the following sections. To clearly outline the experimental design and the sequence of processing steps, the overall workflow employed in this approach is schematically presented in the flowchart shown in [Figure 48](#).

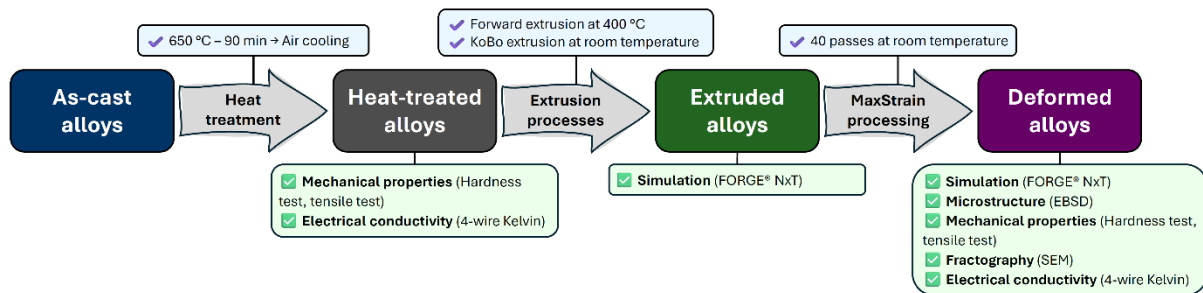


**Figure 48:** Simplified flowchart illustrating the overall workflow adopted in Approach 2.

### 3.2.1. Methodology

#### 3.2.1.1. Material Fabrication

The Cu-Mg alloy was fabricated according to the procedure described in Section 3.1.1.1. After casting, the as-cast ingots were subjected to homogenization annealing at 650 °C for 90 min to reduce chemical and microstructural heterogeneities formed during solidification and were then air-cooled to RT. Billets with a length of 100 mm were subsequently cut from the ingots along the longitudinal direction and were then machined to a diameter of 39.5 mm to remove residual surface defects, such as oxide layers and casting irregularities, and to ensure compliance with the dimensional requirements for the subsequent extrusion process.

#### 3.2.1.2. Extrusion Processes

Taking into account the fact that the billets initially expanded to completely fill the extrusion container before the onset of the extrusion process, the cross-sectional area was reduced from

an initial circular cross section with a diameter of 40 mm ( $\sim 1257 \text{ mm}^2$ ) to a final square cross section with a side length of 10 mm ( $100 \text{ mm}^2$ ). Therefore, the nominal extrusion ratio ( $R = A_0/A_e$ ) for both forward extrusion and KoBo extrusion was  $\sim 12.6$  based on the configuration used for this study. Owing to the oscillatory motion of the reversibly rotating die, which reduces the required extrusion force [328], the KoBo extrusion could be successfully performed at RT. The punch speed was set to 0.5 mm/s, and the reversibly oscillating die was ordered to rotate at a frequency of 6 Hz with an oscillation amplitude of  $\pm 8^\circ$ . Extrusion was carried out using a hydraulic press with a maximum capacity of 1.5 MN. To minimize friction between the billets and the extrusion container, MoS<sub>2</sub> was applied as a lubricant. For the forward extrusion process, the billets were preheated to 400 °C to enhance material flow and facilitate deformation during extrusion.

### 3.2.1.3. MaxStrain Processing

Following the performed preparation and extrusion steps, samples in the annealed, forward-extruded, and KoBo-extruded conditions were each subjected to 40 passes of MaxStrain processing at room temperature. The processing was carried out under the same conditions described in Section 3.1.2.4, following the identical pattern illustrated in Figure 27. Table 6 summarizes the processing history and lists the abbreviated names assigned to each sample, which are used throughout the remainder of this work.

**Table 6:** Processing history and abbreviated sample names for specimens in Approach 2.

| Sample name     | Processing history                                                                     |
|-----------------|----------------------------------------------------------------------------------------|
| <b>Annealed</b> | As-cast alloy → Annealed at 650 °C for 90 min → Air cooled to RT                       |
| <b>AM</b>       | Annealed sample → MaxStrain processed for 40 passes at RT                              |
| <b>FM</b>       | Annealed sample → Forward extruded at 400 °C → MaxStrain processed for 40 passes at RT |
| <b>KM</b>       | Annealed sample → KoBo extruded at RT → MaxStrain processed for 40 passes at RT        |

All methodological procedures, including the simulation parameters, SEM analysis, mechanical properties assessment, and electrical conductivity evaluation, were conducted in accordance with the methods described in Section 3.1.1. As previously noted, multiple repeated measurements were performed for all experimental tests to improve statistical reliability and to minimize experimental uncertainty.

## 3.2.2. Results and Discussion

### 3.2.2.1. Calculation of Equivalent Strain

#### 3.2.2.1.1. Extrusion Processes

The simulation results for the forward extrusion and KoBo extrusion processes are presented in [Figures 49](#) and [50](#), respectively. As shown in [Figures 49\(a\)](#) and [50\(a\)](#), the distribution of equivalent strain within the initial segments of the extruded billets is non-uniform in both processes. This indicates that, at the early stage of deformation, the material flow had not yet stabilized, which led to local variations in strain. Due to this inhomogeneity and the associated transient deformation effects, these regions were excluded from further analysis, and only the steady-state portions of the extruded billets were considered for the rest of investigations. It was also evident that, despite the use of the same extrusion ratio in both processes, different levels and distributions of equivalent strain developed within the extruded billets. This difference was attributed to the distinct deformation mechanisms inherent to forward extrusion and KoBo extrusion, as well as differences in their processing temperatures, which influenced material flow behavior and strain accumulation during deformation. [Figures 49\(b\)](#) and [50\(b\)](#), which correspond to the steady-state regions of the extruded billets, showed that the maximum equivalent strain in the forward-extruded sample occurred at the edges and corners, where frictional constraints and material flow resistance are typically the highest. In contrast, for the KoBo-extruded billet, the highest strain was mainly concentrated at the corners, although elevated strain levels were also observed near the outer regions. Notably, the equivalent strain values at the edges and corners of the KoBo-extruded billet were higher than those observed in the forward-extruded counterpart. This behavior was explained by the additional plastic deformation introduced by the oscillatory motion of the reversibly rotating die in the KoBo extrusion process. This motion generated a superimposed torsional (shear) component on the primary compressive deformation, which enhanced material flow, promoted more intense plastic straining, and ultimately led to greater strain accumulation in the processed material.

In addition, the simulation results indicate that the equivalent strain at the center of the forward-extruded billet reached a value of 2.3, which is approximately 10% higher than the corresponding value of 2.1 obtained at the center of the KoBo-extruded billet. This difference can be attributed to the combined effects of deformation temperature and the distinct deformation characteristics of the two processes. As noted, all simulations were performed under conditions closely replicating the actual deformation processes, including the respective processing temperatures and associated material flow conditions. Specifically, the relatively

higher deformation temperature used in the forward extrusion process (400 °C) reduced the flow stress of the material and, as a consequence, facilitated plastic deformation, which promoted more effective strain accumulation in the central region of the billet. In contrast, the lower effective deformation temperature associated with the KoBo extrusion process, together with its different deformation mode, resulted in comparatively lower strain development at the billet center. As a result, a higher equivalent strain was obtained in the central region of the forward-extruded billet compared with that of the KoBo-extruded billet.

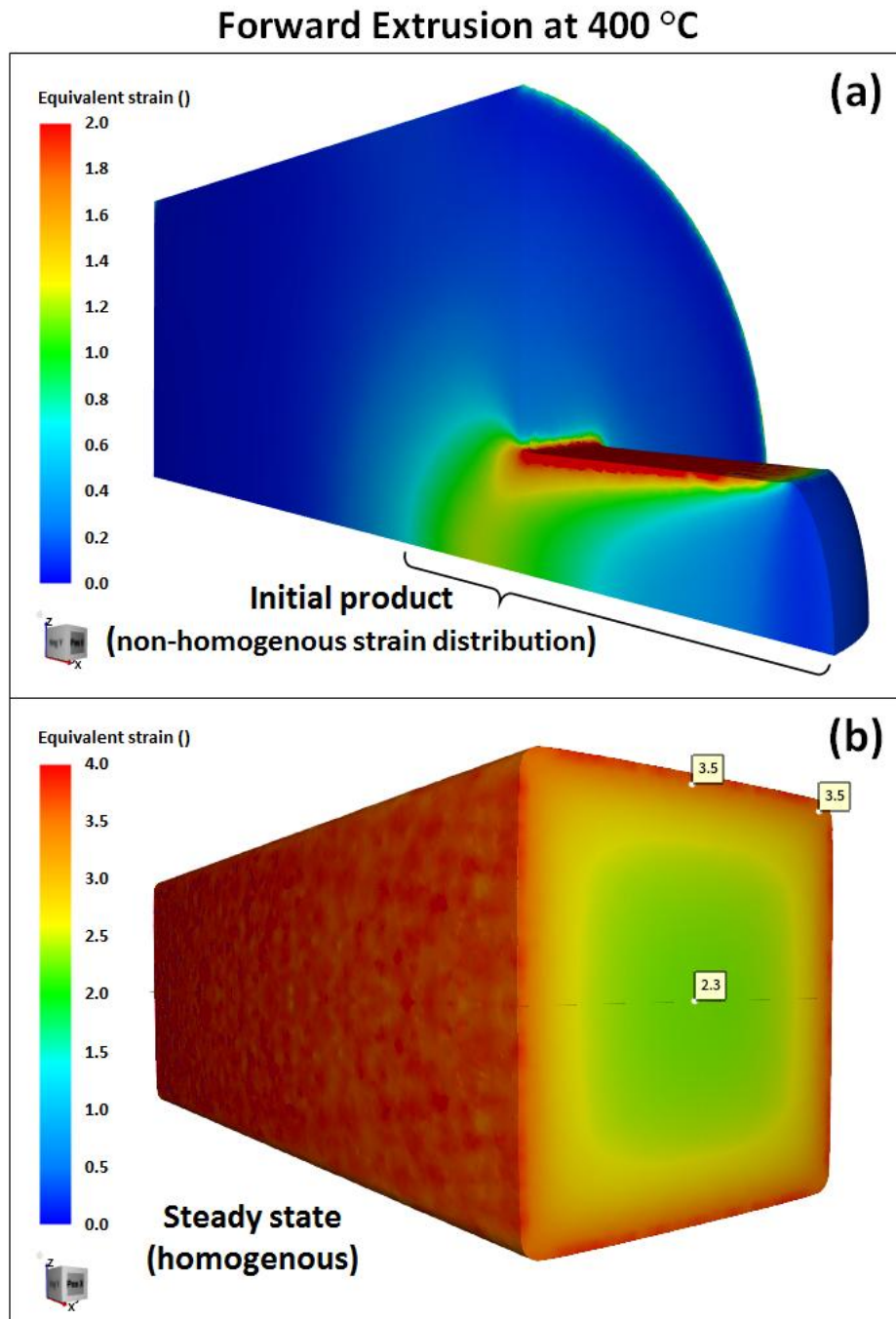


**Figure 49:** Distribution of equivalent strain in (a) the initial product and (b) the steady-state region of the forward-extruded billet.

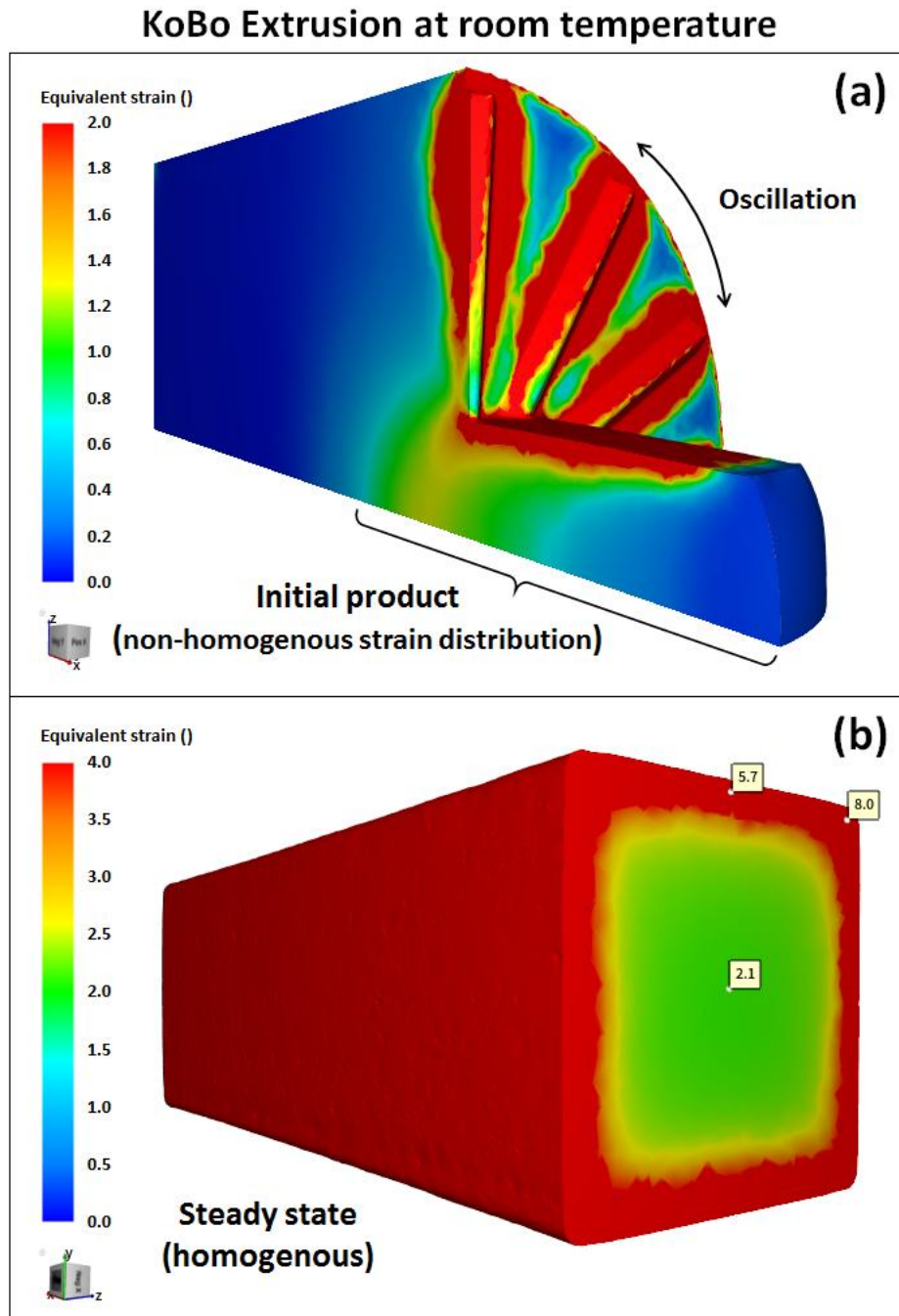


**Figure 50:** Distribution of equivalent strain in (a) the initial product and (b) the steady-state region of the KoBo-extruded billet.

These simulations represent the first stage of the overall modeling procedure. The steady-state regions of the extruded billets shown in [Figures 49\(b\)](#) and [50\(b\)](#) were extracted and used as input data for the simulation of the MaxStrain process, and the results are subsequently discussed.

### 3.2.2.1.2. MaxStrain Processing

Figures 51, 52, and 53, respectively, present the simulation results for one half of the AM, FM, and KM samples. In each case, a central transverse cutting plane was introduced through the middle of the deformation zone perpendicular to the longitudinal direction of the samples to enable a detailed examination of the equivalent strain distribution within the core region. The simulation results demonstrated that the initial non-uniformity in equivalent strain distribution within the extruded billets resulted in a fundamentally different equivalent strain gradient following the MaxStrain processing. For the AM sample, a maximum equivalent strain of 5.0 was attained at the center of the deformation zone, while the minimum equivalent strain, with a value of 1.6, was observed near the corner of the central transverse plane, as shown in Figure 51. Furthermore, it is also important to note that the nature of the MaxStrain process, which imposes a high amount of plastic deformation sequentially in two mutually perpendicular directions, led to variations in the calculated equivalent strain along adjacent edges of the samples. This behavior reflects the complex strain path and non-uniform deformation imposed during MaxStrain processing, which contributed to the development of localized strain gradients across the cross section.

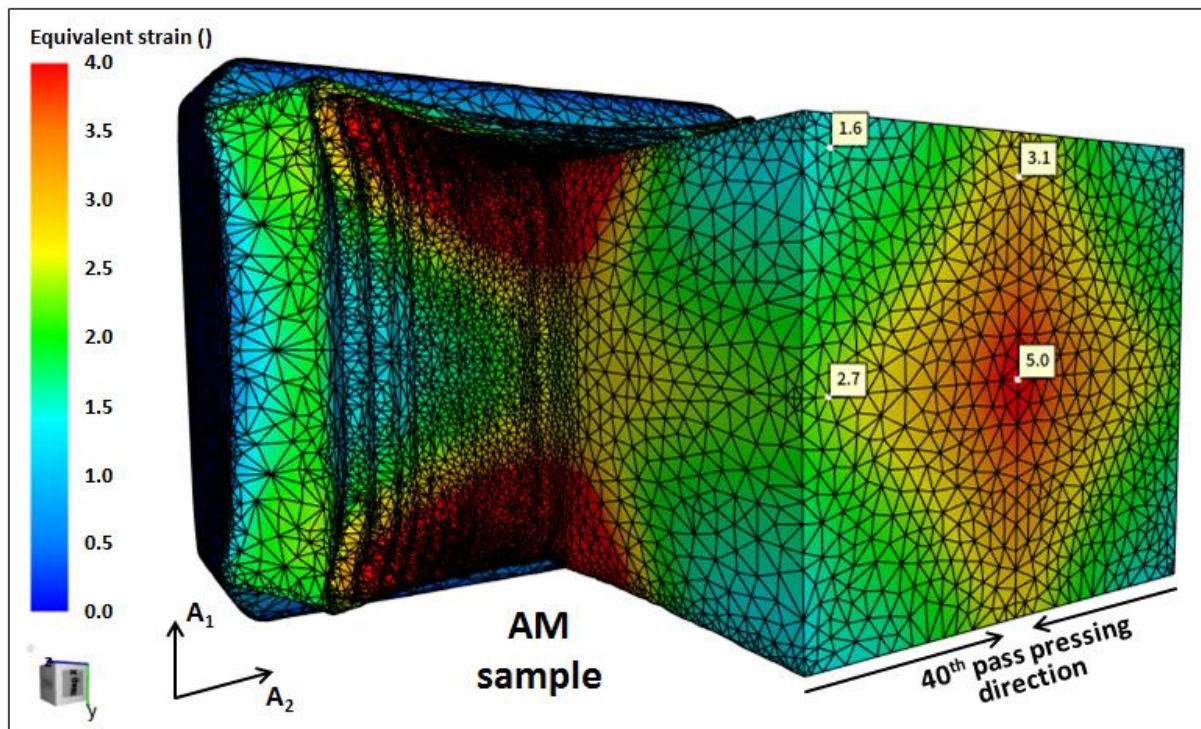


**Figure 51:** Distribution of equivalent strain in the central transverse cutting plane of the AM sample.

The distribution of equivalent strain within the central transverse plane of the FM sample exhibited a pattern similar to that observed in the AM sample. As illustrated in Figure 52, the

maximum equivalent strain was 7.5 at the center of the plane, intermediate values of 6.6 and 6.2 were observed along the edges, and the minimum value of 4.9 occurred at the corner. This distribution indicated a pronounced strain concentration in the central region, accompanied by a gradual reduction toward the edges and corners, which reflects the influence of the imposed deformation path during processing.

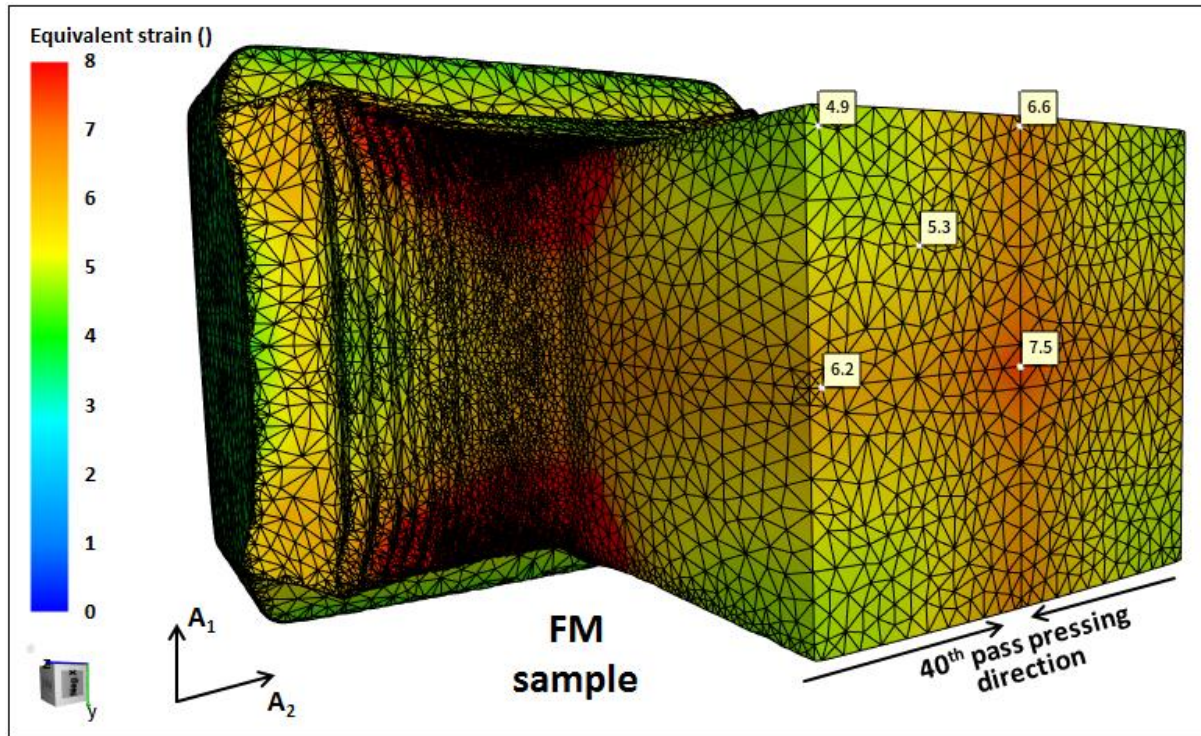


**Figure 52:** Distribution of equivalent strain in the central transverse cutting plane of the FM sample.

In the meantime, the KM sample exhibited a distinctly different distribution of equivalent strain compared to the FM and AM samples. As shown in [Figure 50](#), in the initial KoBo-extruded billet, the regions near the corners and edges already displayed relatively high equivalent strain, which further increased via MaxStrain processing. As a result, the central transverse plane of the KM sample reached a maximum equivalent strain of 9.4 at the corner, while the minimum equivalent strain of 5.6 was observed at a location approximately midway between the center and the corner, as shown in [Figure 53](#). The equivalent strain at the center of the plane was calculated to be 7.8, and the strains along the edges were 8.0 and 9.0, respectively. This pronounced heterogeneity in strain emphasizes the strong influence of the initial deformation state on the final strain distribution. These results highlight the critical role of processing history in determining the ultimate equivalent strain achieved in the samples. Specifically, the different initial strain distributions in the forward-extruded billets ([Figure 49](#)) and KoBo-extruded billets ([Figure 50](#)) led to fundamentally distinct strain patterns following

MaxStrain processing. In other words, the FM sample (Figure 52) and the KM sample (Figure 53) display significantly different equivalent strain distributions, because the initial distribution of equivalent strain strongly governs the evolution of strain during subsequent deformation.

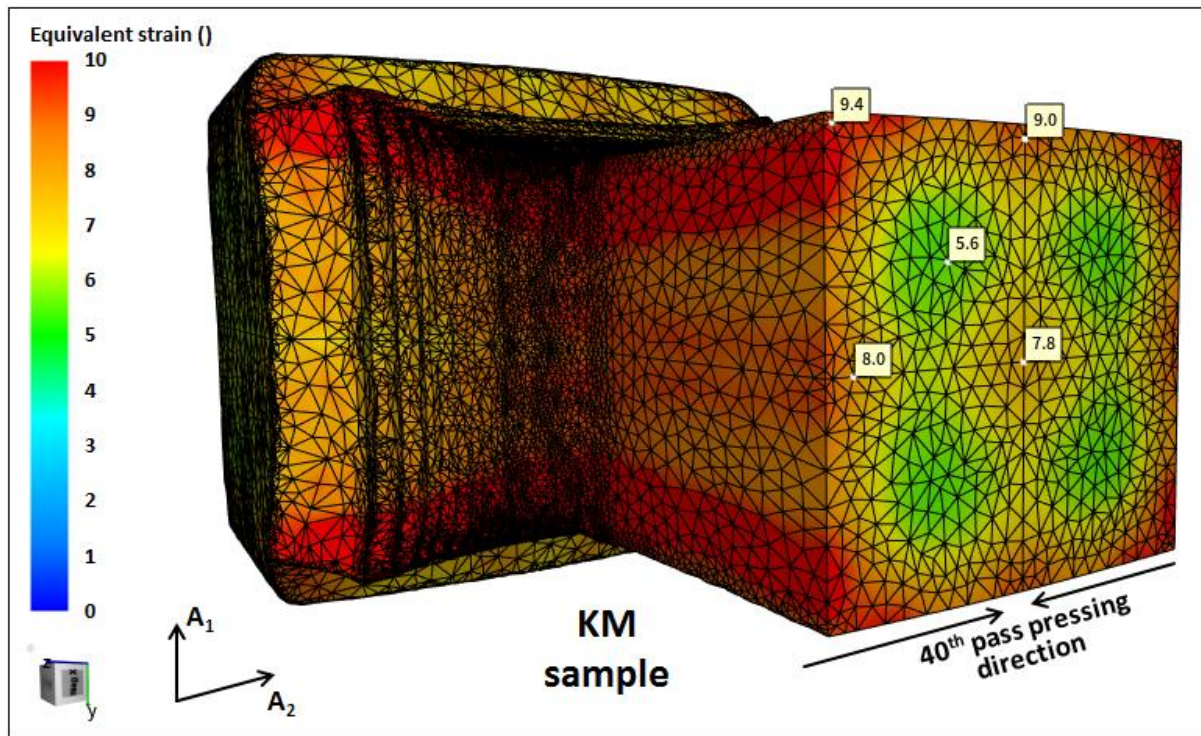


**Figure 53:** Distribution of equivalent strain in the central transverse cutting plane of the KM sample.

### 3.2.2.2. Post-Deformation Microstructures

The IPF maps obtained from the center of the central transverse plane within the deformation zone of the MaxStrain-processed samples are presented in Figure 54. The image length direction is perpendicular to the pressing direction of the 40th pass, and HAGBs are highlighted by black lines within the images. Based on quantitative EBSD analysis, the average grain size at the center of the central transverse plane was determined to be approximately 5.8  $\mu\text{m}$  for the AM sample, 1.4  $\mu\text{m}$  for the FM sample, and 2.0  $\mu\text{m}$  for the KM sample. During deformation in both extrusion routes and during MaxStrain processing, CDRX is suggested to be the dominant mechanism governing grain refinement. For the AM sample (Figure 54(a)), the microstructure exhibits a heterogeneous grain morphology characterized by the coexistence of coarse and elongated grains as well as fine and equiaxed grains, which is indicative of partial CDRX. In fact, since MaxStrain processing was performed at RT, limited atomic mobility and restricted grain boundary migration constrained the extent of CDRX and prevented complete microstructural transformation under the applied processing conditions.

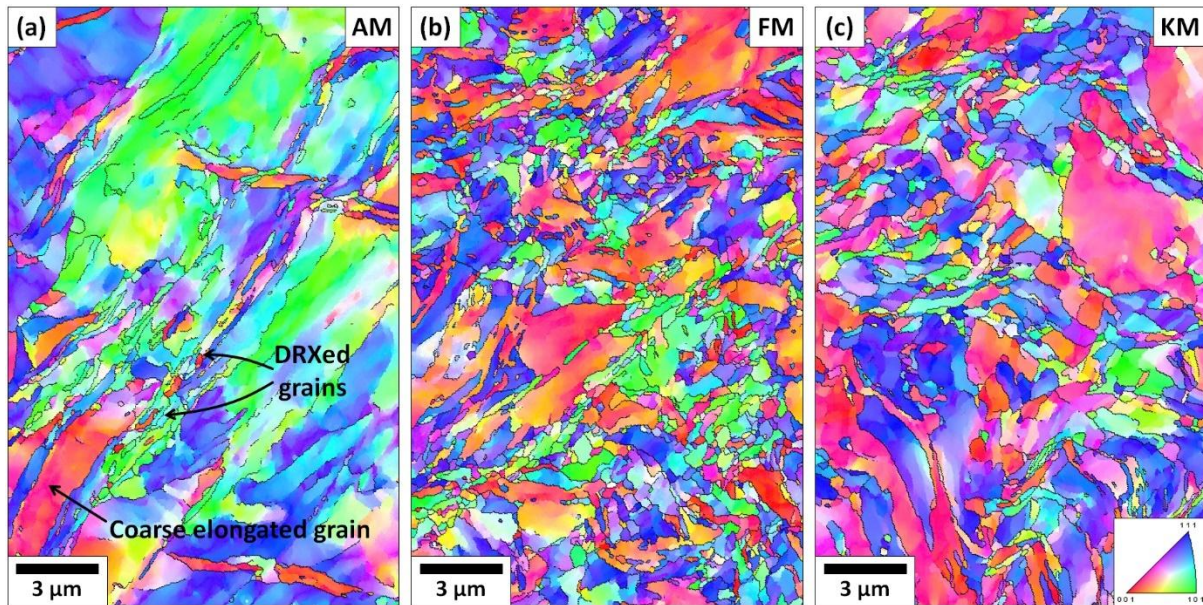


**Figure 54:** IPF maps showing the microstructures of (a) AM, (b) FM, and (c) KM samples.

In the case of the FM sample, since the forward extrusion was carried out at 400 °C (which corresponds to approximately 0.5  $T_m$  of the alloy) prior to the MaxStrain processing, this relatively high pre-deformation temperature provided sufficient thermal activation to facilitate a greater extent of partial DRX during the forward extrusion itself, which provided a more favorable initial microstructure for subsequent MaxStrain deformation at RT, resulting in a higher final extent of CDRX. As a result, a higher fraction of fine equiaxed grains can be observed in the FM sample compared to the AM sample, as clearly shown in [Figure 54\(b\)](#). This microstructural refinement is further reflected in the EBSD quantitative analysis, which revealed that the average grain size of the FM sample is approximately 76% smaller than that of the AM sample.

In the meantime, although the calculated equivalent strain at the center of the central transverse plane of the KM sample was about 4% higher than that of the FM sample, EBSD results indicated that the KM sample possessed an average grain size approximately 43% larger than that of the FM sample, as seen in [Figures 54\(b\)](#) and [54\(c\)](#). The presence of coarser grains in the KM sample, despite its slightly higher imposed strain, suggests a more pronounced influence of deformation temperature relative to strain magnitude in governing the extent of CDRX and grain refinement. Meanwhile, compared with the AM sample, the KM sample still demonstrated significant grain refinement, with an average grain size reduction of approximately 66%. The kernel average misorientation (KAM) maps for the AM, FM, and KM samples are presented in [Figure 55](#). The AM sample, which experienced a limited extent of CDRX, is characterized by a higher fraction of LAGBs, which is indicative of a deformation-

dominated microstructure with a high density of subgrain boundaries. In contrast, the FM and KM samples exhibited lower LAGB fractions, which is consistent with a higher extent of CDRX, where progressive subgrain rotation and increasing misorientation lead to the transformation of LAGBs into HAGBs.

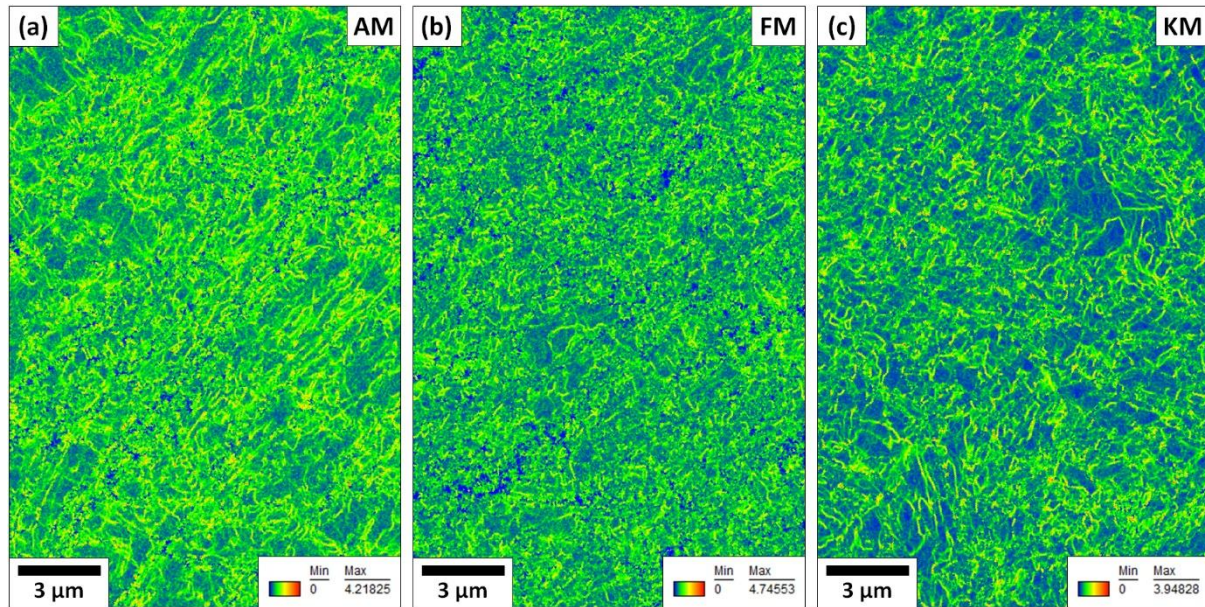


**Figure 55:** KAM maps of (a) AM, (b) FM, and (c) KM samples.

Figure 56 presents the misorientation angle distributions of the AM, FM, and KM samples. It can be seen that the FM and KM samples show a higher fraction of HAGBs compared with the AM sample, which is consistent with the trends observed in the IPF and KAM analyses. This increase in HAGBs can be attributed to the development of finer grain structures, as grain refinement generally promotes the formation of a larger number of grain boundaries with higher misorientation angles due to progressive subgrain rotation and boundary evolution. In addition, the higher HAGB fractions in the FM and KM samples suggest a greater extent of recrystallization compared with the AM sample. This interpretation aligns well with the IPF maps, which indicated a lower proportion of recrystallized grains in the AM condition compared to the FM and KM samples. Overall, the misorientation angle distributions provide strong support for the microstructural changes observed in the IPF and KAM results and indicate that a more advanced recrystallization process occurred in the FM and KM samples, which contributed to their refined grain structures.

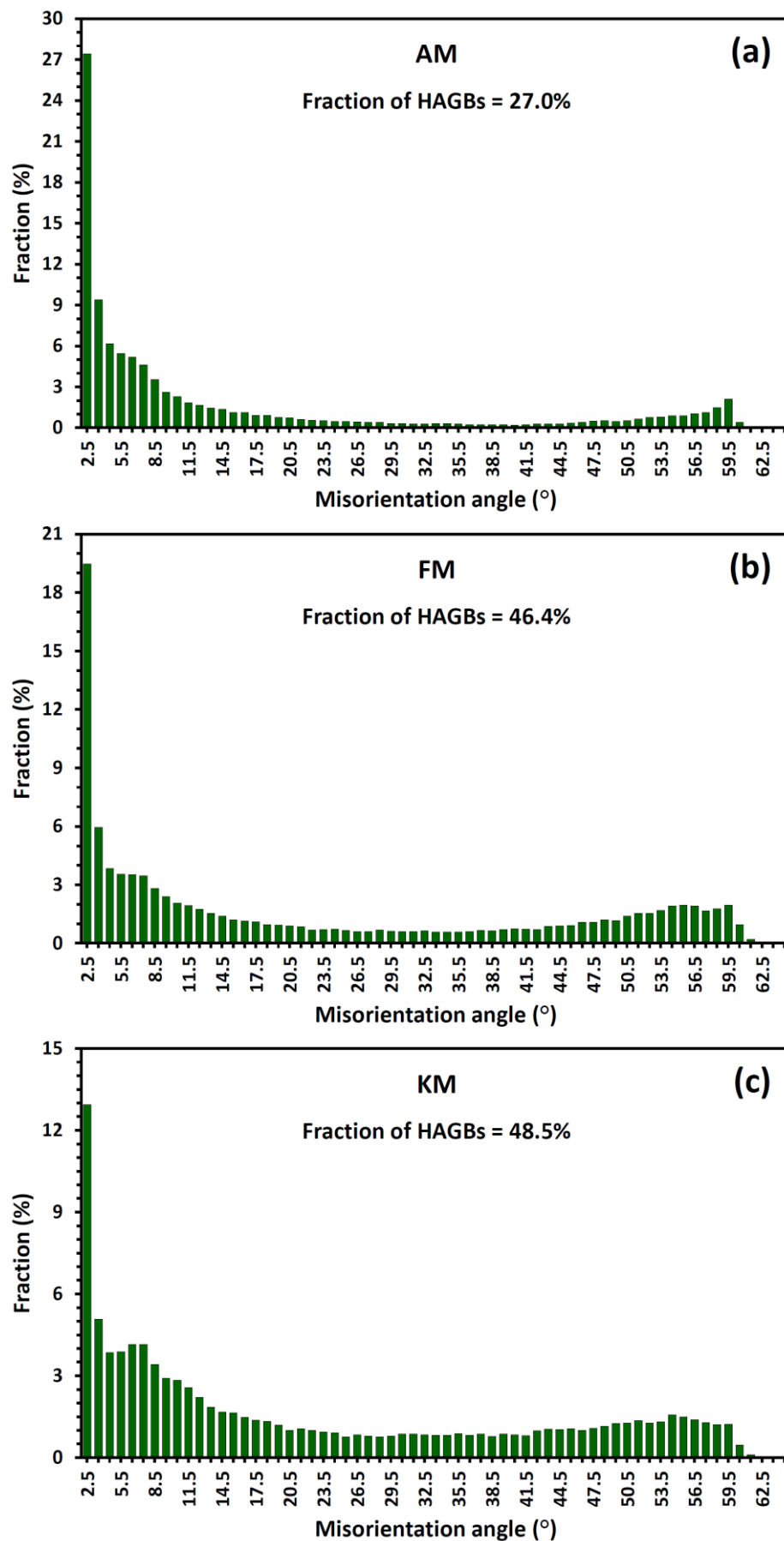


**Figure 56:** Distribution of misorientation angles in (a) AM, (b) FM, and (c) KM samples.

### 3.2.2.3. Mechanical Properties

The hardness test results demonstrated that the annealed alloy exhibited a relatively uniform average hardness of  $59 \pm 2$  HV, which reflects its homogeneous microstructural condition after annealing. For the MaxStrain-processed samples, a single representative average hardness value was not appropriate due to significant strain inhomogeneity; therefore, hardness was evaluated through a series of indentations performed at regular 0.45 mm intervals along the A1 and A2 directions within the central transverse plane, as shown in [Figures 50 to 53](#), to capture the spatial variation in hardness. As expected, the observed hardness distribution within the MaxStrain-processed samples closely followed the equivalent strain distribution predicted by the simulation, which indicates a strong correlation between local strain accumulation and hardness.

For the AM sample, the highest hardness was measured at the central location, with a value of 138.3 HV, which was higher than those recorded in the middle and edge regions ([Figure 57\(a\)](#)). Similar to the preceding discussion in Section 3.1.2.7, this variation in hardness can be attributed to the higher equivalent strain concentrated at the center of the sample, which results in a more pronounced strain-hardening response in this region. Similarly, the FM sample exhibited its maximum hardness at the center of the central transverse plane, reaching 163.7 HV ([Figure 57\(b\)](#)). From this central position, the hardness values showed a gradual and consistent decrease toward the edge regions, which indicates a clear hardness gradient across the section. In the case of the KM sample, the maximum hardness was also observed at the center, with a value of 159.7 HV. However, unlike the AM and FM samples, the edge region of the central transverse plane in the KM sample displayed relatively higher hardness than the middle regions located between the center and the edge ([Figure 57\(c\)](#)). The KM sample also demonstrated improved hardness uniformity across its cross section compared to the FM and AM samples, which reflect a more homogeneous strain-hardening distribution within the center of the deformation zone within this sample. In a comparable study, Mou et al. [329] observed a similar correlation between equivalent strain and hardness. In their study, simulation results indicated that the surface region of H62 brass subjected to multi-cycle constrained groove pressing (CGP) exhibited a higher equivalent strain than the interior, which led to higher surface hardness compared to the interior.

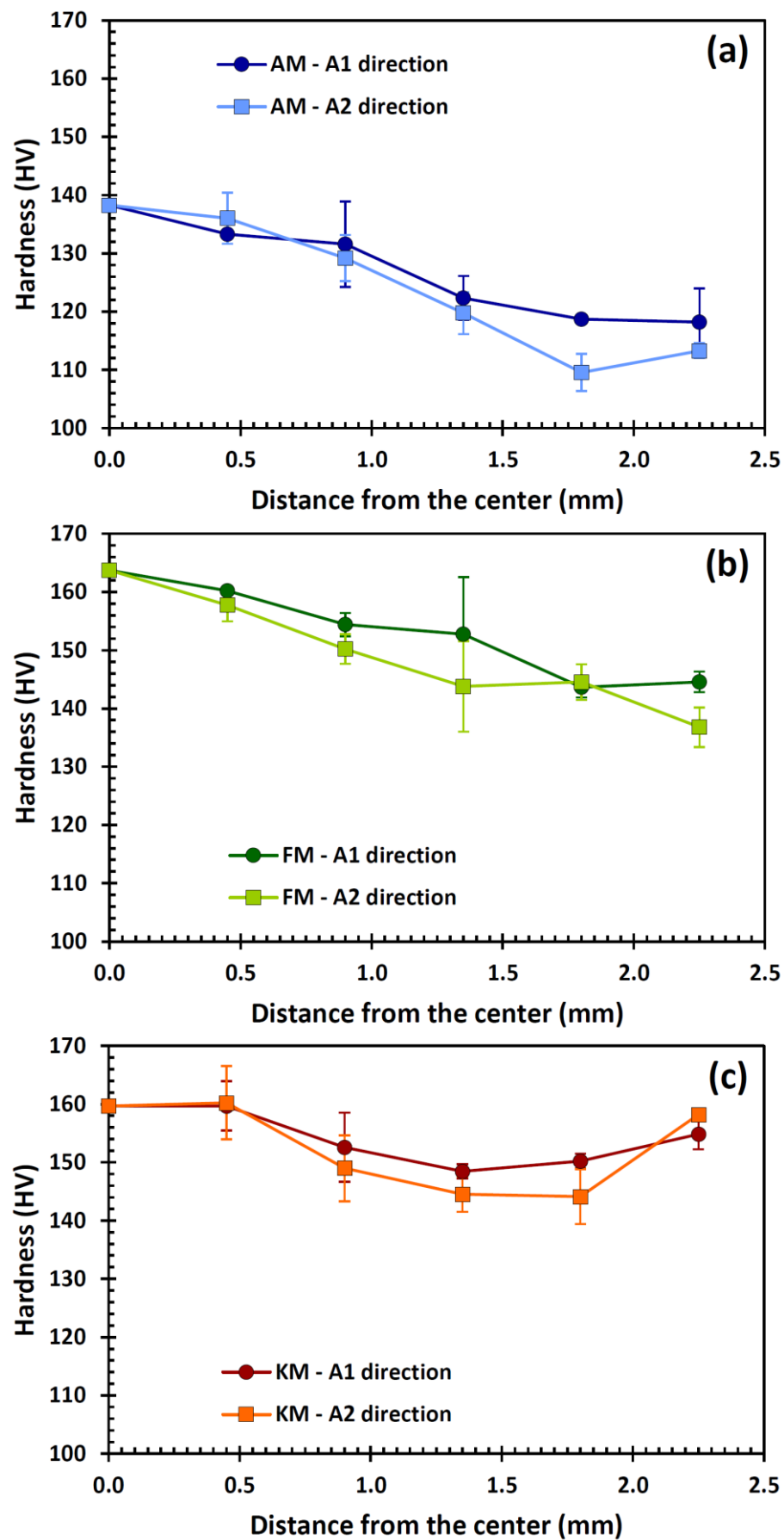


**Figure 57:** The hardness profiles for (a) AM, (b) FM, and (c) KM samples.

The tensile test results obtained for the investigated samples are presented in Figure 58. Due to the small dimensions of the minisamples used in the tensile tests, it was not feasible to employ an extensometer for direct displacement measurement during loading. Therefore, following the procedure described in Section 3.1.2.7, the EL value for each sample was determined by individually measuring the lengths of the fractured tensile specimens (Figure 59) to ensure reliable and consistent results. The annealed alloy exhibited a YS of  $119 \pm 11$  MPa, an UTS of  $232 \pm 15$  MPa, and an EL of  $33.5 \pm 1.6$ , which are similar to the values reported in the literature for non-deformed Cu-Mg alloys [30,33].

The tensile test results for the AM sample demonstrated that MaxStrain processing resulted in a substantial enhancement of the mechanical strength of the annealed alloy. Specifically, the YS and UTS increased by approximately 251% and 95%, respectively, compared to the annealed condition. The pronounced improvement in YS can be primarily attributed to the significant grain refinement achieved during processing. According to the Hall-Petch relationship [330], a reduction in grain size increases the resistance to dislocation motion and leads to a higher YS. In addition to grain refinement, the increased dislocation density introduced during MaxStrain processing likely also contributed to the observed strengthening. Likewise, the increase in UTS can be attributed to the combined effects of grain refinement, strain hardening, and the elevated dislocation density introduced during processing, which enhance the resistance to plastic deformation. However, the EL of the AM sample was reduced by approximately 32% compared to that of the annealed alloy. This reduction in ductility is associated with the increased density of crystal defects, such as dislocations and subgrain boundaries, which limit dislocation mobility and reduce the material's ability to accommodate uniform plastic deformation [331]. This is also consistent with the observation that the AM sample, which exhibited the lowest fraction of recrystallized grains, showed the lowest elongation. In contrast, the FM and KM samples, which exhibited a higher fraction of recrystallized grains and correspondingly lower dislocation densities, showed slightly higher elongation values.

In general, the tensile test results demonstrated that performing an extrusion step on the annealed alloy prior to MaxStrain processing, whether by forward extrusion or KoBo extrusion, led to a significant additional improvement in the mechanical properties. Specifically, the FM sample exhibited YS and UTS values that were 288% and 121% higher, respectively, than those of the annealed alloy. The KM sample showed an even greater enhancement, with YS and UTS values increased by 360% and 169%, respectively, relative to the annealed condition.

This pronounced strengthening can be attributed to the combined effects of extrusion and subsequent MaxStrain deformation. In particular, the prior extrusion step is suggested to have promoted a higher extent of recrystallization during MaxStrain processing and resulted in a finer grain structure (as shown in Figure 54). Moreover, the pre-deformation imposed by extrusion likely increased the dislocation density and, consequently, enhanced the strain-hardening response during MaxStrain processing. The combined contribution of increased recrystallization, refined grain size, and strain hardening accounts for the superior YS and UTS observed in the FM and KM samples and demonstrates the effectiveness of combining extrusion with MaxStrain deformation in strengthening Cu-Mg alloys.

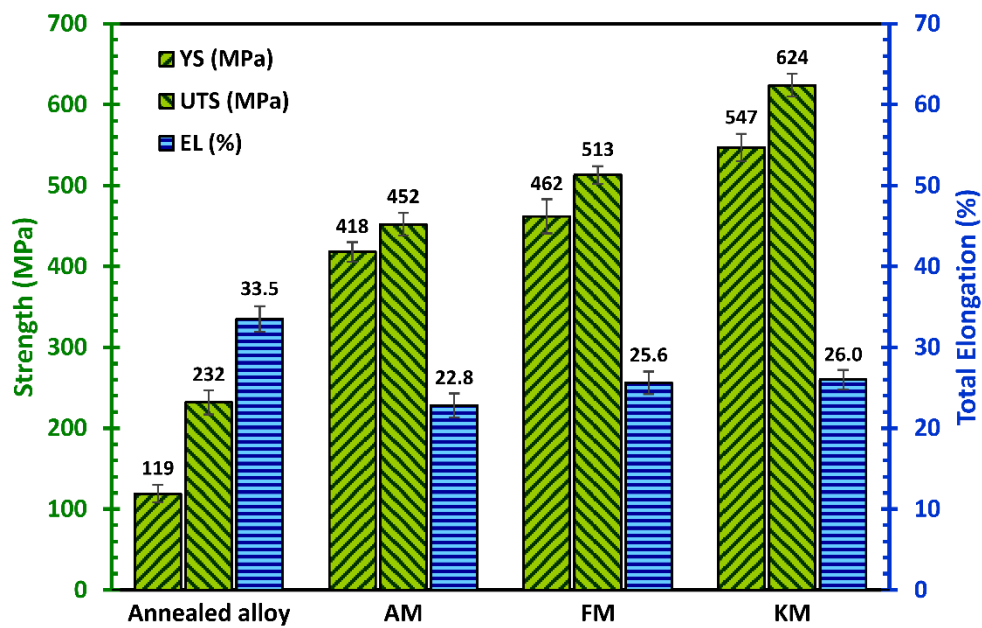


Figure 58: Data obtained from the tensile test results for all investigated samples in this approach.

### 3.2.2.4. Fractography

The fractured specimens and the corresponding fracture surface of the AM sample are presented in Figures 59(a) and 59(d), respectively. The fracture surface exhibits typical ductile fracture features, characterized predominantly by the presence of dimples formed through microvoid nucleation, growth, and coalescence, typical for metallic materials [332]. Similarly, the fracture surfaces of the FM and KM samples, shown in Figures 59(e) and 59(f), also display ductile fracture behavior with well-defined dimple structures. Notably, all samples exhibited fracture surfaces with a heterogeneous dimple morphology. In these surfaces, larger dimples were associated with coarse-grained regions, whereas smaller dimples corresponded to fine-grained zones. Consistent with the EBSD results presented in Figure 54, the FM and KM samples, characterized by a higher fraction of fine recrystallized grains, exhibited a greater

proportion of fine-sized dimples on their fracture surfaces. This observation further supports the strong correlation between grain refinement, microstructural homogeneity, and ductile fracture behavior.

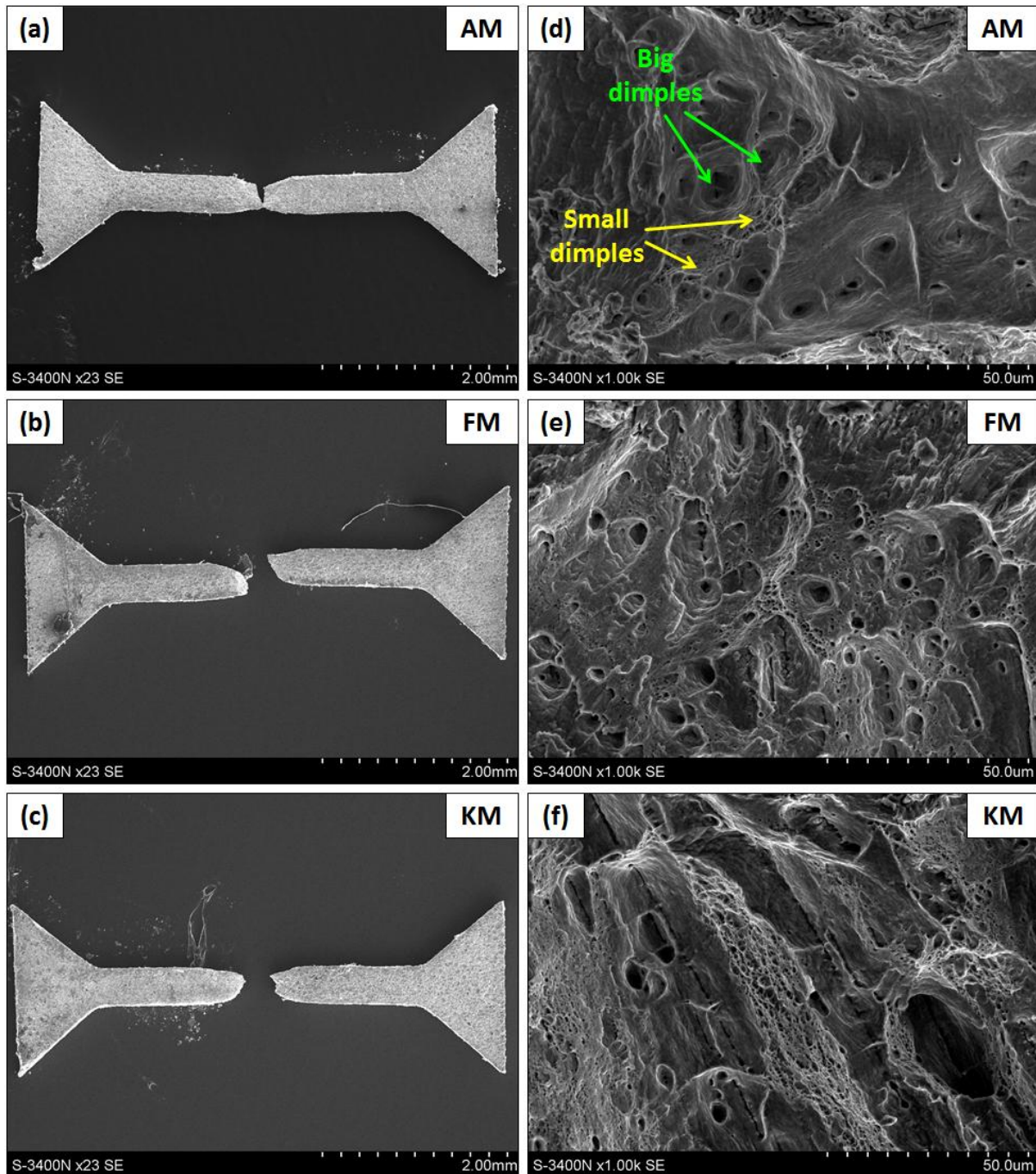


**Figure 59:** Broken pieces and the corresponding fracture surfaces of (a,d) AM, (b,e) FM, (c,f) KM samples.

### 3.2.2.5. Electrical conductivity

The measured electrical conductivity values of the investigated samples are presented in Figure 60. It can be seen that the annealed alloy exhibited an electrical conductivity of  $39.2 \pm$

2.5 MS/m, corresponding to 67.6% IACS, which is 32.4% lower than that of pure Cu. This reduction reflects the effect of Mg addition in solid solution, as solute atoms act as scattering centers for conduction electrons and thereby decrease electrical conductivity. Following MaxStrain processing, the electrical conductivity further decreased to  $37.9 \pm 2.3$  MS/m (65.3% IACS) for the AM sample. This additional reduction can be attributed to the increased density of defects, such as dislocations and grain boundaries, introduced during severe plastic deformation, which enhance electron scattering [35,333].

The FM and KM samples exhibited even lower electrical conductivity compared to the AM sample. In particular, the FM sample showed an electrical conductivity of  $35.6 \pm 3.0$  MS/m (61.4% IACS), while the KM sample exhibited a further reduced value of  $32.0 \pm 3.0$  MS/m (55.2% IACS). Relative to the annealed alloy, the electrical conductivity decreased by approximately 3%, 9%, and 18% for the AM, FM, and KM samples, respectively. These results indicate the negative effect of deformation-induced microstructural refinement on electrical conductivity. The progressive reduction in conductivity can be attributed to the increasing density of crystal defects introduced during processing. In particular, the higher fraction of recrystallized grains in the FM and KM samples resulted in a greater proportion of HAGBs (as evidenced in [Figure 56](#)). Since grain boundaries act as scattering sites for conduction electrons [334], their increased density likely contributed to the lower electrical conductivity in the FM and KM samples compared to the AM sample.

Nevertheless, it should be mentioned that the adverse effect of deformation-induced microstructural refinement on electrical conductivity was less pronounced than the effect associated with Mg in solid solution. Despite the additional electron scattering introduced by deformation-related defects, solid solution effects remained the dominant factor governing the overall reduction in electrical conductivity. Notably, the KM sample, which experienced the highest equivalent strain during the deformation processes, exhibited the lowest electrical conductivity among all investigated conditions. In contrast, the AM sample, characterized by the lowest equivalent strain, showed the highest electrical conductivity among the processed samples. This trend further indicates a correlation between accumulated strain, defect density, and electrical conductivity in the studied alloy system.

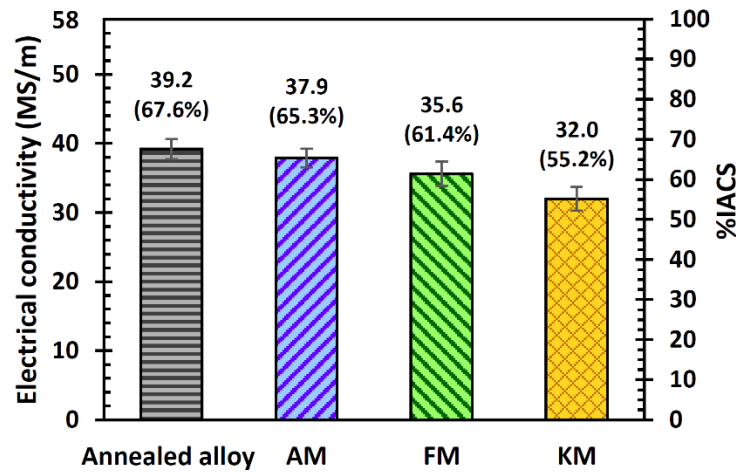


**Figure 60:** Measured electrical conductivity values for the investigated samples in this approach.

In conclusion, as illustrated in [Figure 61](#), which presents the relationship between UTS and electrical conductivity for all samples produced via Approaches 1 and 2, it can be seen that all MaxStrain-processed samples consistently exhibit electrical conductivities exceeding 50% IACS and UTS values above 300 MPa. Therefore, based on the definition proposed by Yang et al. [15], all of the MaxStrain processed samples in these approaches can be classified as HSHC Cu alloys regardless of their processing history.

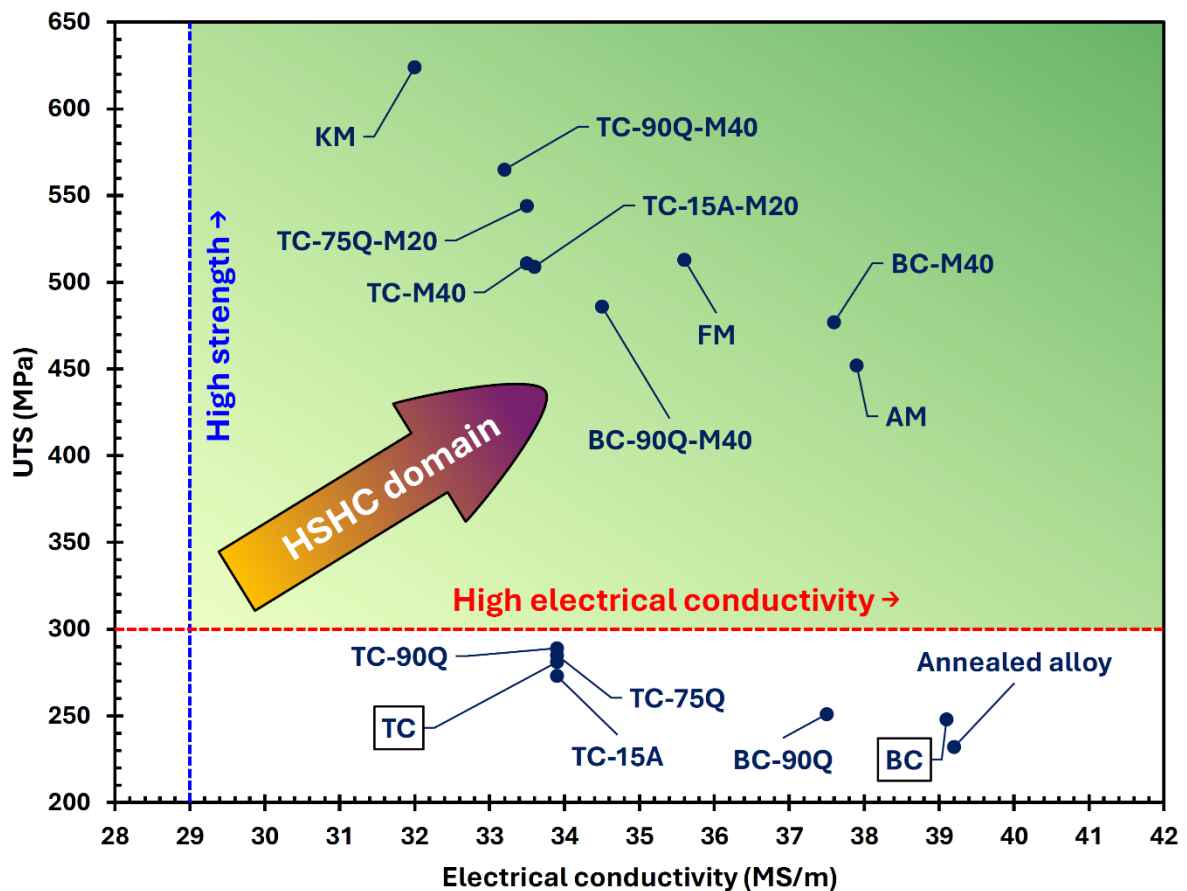


**Figure 61:** UTS versus electrical conductivity values for all samples studied in Approaches 1 and 2.

### 3.2.3. Conclusions of Approach 2

In summary, the effects of forward extrusion at 400 °C and KoBo extrusion at RT, followed by additional deformation via the MaxStrain module of the Gleeble thermomechanical simulator, on the microstructural features, mechanical properties, and electrical conductivity of a Cu-0.7Mg (wt.%) alloy were investigated in this approach. Based on the obtained results, the following conclusions can be drawn:

- 2.1. The deformation simulations demonstrated that the processing history played a critical role in determining the magnitude and spatial distribution of equivalent strain within the deformed samples. The results revealed that the initially distinct equivalent strain distributions introduced by forward extrusion and KoBo extrusion led to completely different equivalent strain patterns after MaxStrain processing.
- 2.2. Following MaxStrain processing, the initially coarse-grained microstructure of the annealed alloy was transformed into a heterogeneous grain structure comprising both coarse elongated grains and fine equiaxed grains, resulting from the occurrence of partial CDRX. The fraction of recrystallized grains was noticeably higher in the FM and KM samples, which had undergone forward extrusion and KoBo extrusion, respectively, prior to MaxStrain processing. Furthermore, EBSD analysis revealed that the FM and KM samples exhibited a higher fraction of HAGBs compared to the AM sample, which was subjected solely to MaxStrain processing. This higher fraction of HAGBs was attributed to the greater extent of recrystallization present in the FM and KM samples.
- 2.3. According to the tensile test results, MaxStrain processing led to significant improvements in the mechanical strength of the annealed alloy, with the YS and UTS increasing by 251% and 95%, respectively. In comparison, the FM and KM samples exhibited even greater strengthening, with YS enhancements of 288% and 360%, respectively, and corresponding increases in UTS of 121% and 169% relative to the initial annealed condition. These improvements were primarily attributed to the combined effects of grain refinement associated with enhanced recrystallization, increased HAGB fraction, and deformation-induced strengthening mechanisms, including the accumulation of dislocations during SPD processing.
- 2.4. The annealed alloy exhibited an electrical conductivity of  $39.2 \pm 2.5$  MS/m (67.6% IACS), which decreased to  $37.9 \pm 2.3$  MS/m following MaxStrain processing (AM sample). Furthermore, the FM sample, characterized by a lower equivalent strain than the KM sample, exhibited an electrical conductivity of  $35.6 \pm 3.0$  MS/m. In contrast, the KM

sample, which experienced the highest accumulated equivalent strain, showed a further reduction in conductivity to  $32.0 \pm 3.0$  MS/m. Yet, all MaxStrain-processed samples satisfied the criteria for classification as HSHC Cu alloys.

### **3.3. Approach 3: Fabricating Density-Reduced Highly Conductive Cu/Mg Composite**

In this approach, instead of fabricating Cu-Mg or Cu-Mg-Ca alloys, a density-reduced Cu/Mg composite structure, composed of a pure Cu matrix with continuous pure Mg filaments embedded within it, was produced via KoBo extrusion. This design was intended to allow the pure Cu matrix to retain its inherently high electrical conductivity, as it remained free of any solute elements that could otherwise increase electron scattering and hinder free electron transport. On the other hand, the embedded Mg filaments act as a density-reducing second phase. While the incorporation of softer Mg inherently lowers the yield strength of the whole composite structure compared to solid work-hardened Cu, this configuration was designed to balance structural integrity with a significant reduction in overall density.

#### **3.3.1. Methodology**

##### **3.3.1.1. Composite Fabrication**

The matrix of the composite structure was fabricated from as-cast pure Cu ingots, prepared according to the procedure described in Section 3.1.1.1. These ingots were machined into cylindrical billets with a diameter of 39.5 mm to serve as the primary matrix material. Subsequently, seven evenly spaced bores, each with a diameter of 5.5 mm, were drilled along the longitudinal direction of the cylinder to accommodate the structural constituent Mg rods, as illustrated in [Figure 62](#). The number, size, and arrangement of these bores were intentionally selected to promote an effective, uniform, and symmetric spatial distribution of Mg filaments and to facilitate symmetrical load transfer between the matrix and the structural constituents. The Mg filaments were produced by casting pure Mg, followed by machining to an initial diameter of 9.5 mm. They were then extruded to achieve the final diameter of 5 mm required for insertion into the Cu matrix. This reduction in diameter via forward extrusion ensured dimensional compatibility with the pre-machined bores and also contributed to a more refined microstructure of the Mg phase. Prior to assembly, the filaments were carefully cleaned to remove residual surface contaminants and oxide layers. The cleaning procedure involved

sequential degreasing with acetone, mechanical brushing using a steel brush, rinsing with isopropanol, and finally drying by blowing compressed air. This cleaning procedure was performed to promote contamination-free interfacial bonding between the Cu matrix and Mg filaments during subsequent processing. The cleaned filaments were then inserted into the prepared bores in the Cu matrix. The assembled structure (Cu matrix containing embedded Mg filaments) was then heated to 200 °C for 5 min, inserted into the extrusion chamber, and subsequently subjected to KoBo extrusion. [Figure 63](#) presents an image of the Mg filaments embedded within the Cu matrix prior to the cleaning process.

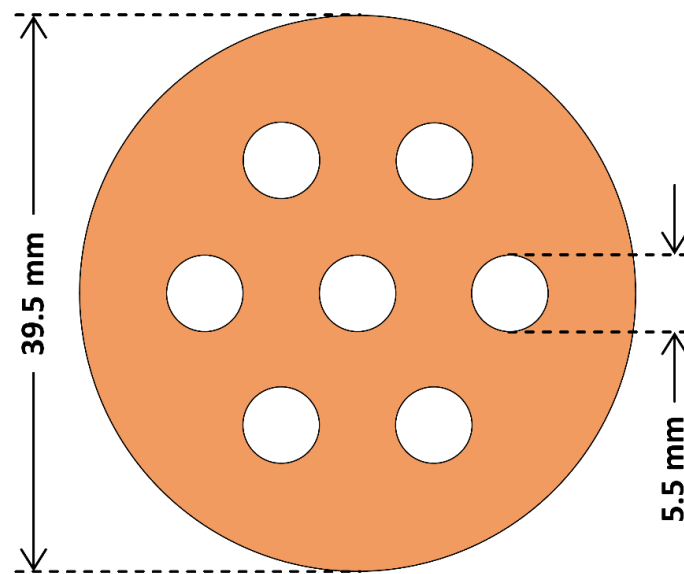


**Figure 62:** Schematic illustration of the transverse section of pure Cu matrix prior to the KoBo extrusion.

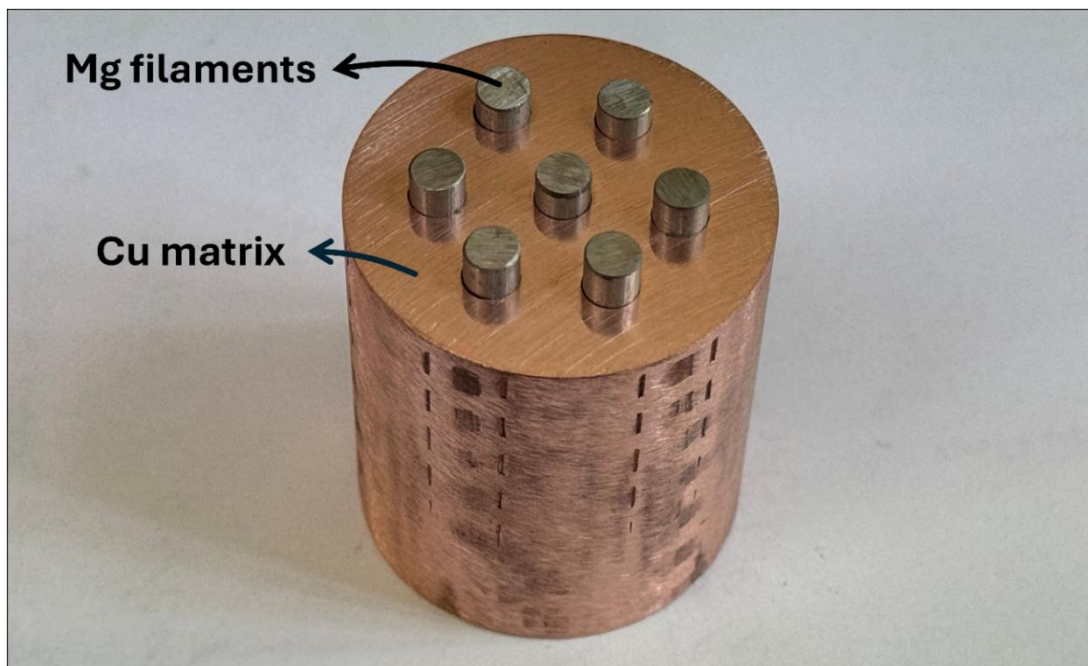


**Figure 63:** Photograph of Mg filaments embedded within a Cu matrix prior to the KoBo extrusion.

The extrusion process was performed with a constant punch speed of 0.5 mm/s. The reversibly oscillating die was operated at a frequency of 6 Hz with an oscillation amplitude of  $\pm 8^\circ$ , using a hydraulic press with a load capacity of 1.5 MN. To minimize friction and ensure smooth material flow during processing, MoS<sub>2</sub> was carefully applied as a lubricant to the outer surface of the Cu matrix prior to extrusion. The diameter of the reversibly rotating die at the outlet of the extrusion system was 8 mm. Based on these parameters, the nominal extrusion ratio ( $R = A_0/A_e$ ) for this condition was theoretically calculated to be 25, assuming that the initial structure first expanded to completely fill the extrusion container before the onset of material extrusion [245]. Hardness test, SEM analysis, and electrical conductivity measurements were performed in accordance with the procedures described in Section 3.1.1. Same as all extrusion processes in which the initial extrudate is not homogeneous, the product in this case also exhibited non-uniformity at the early stage of the extrusion due to the fact that the system did not yet reach steady-state conditions. More specifically, the early stage of extrusion produced material composed predominantly of Cu, and after a certain period, Mg filaments began to enter the deformation zone, and the process gradually transitioned to steady-state with the extruded billet exhibiting a symmetric composite structure.

### **3.3.1.2. Transmission Electron Microscopy**

Transmission electron microscopy (TEM) investigations were performed using a HITACHI H-800 microscope operated at an accelerating voltage of 200 kV, which provides sufficient beam energy for high-resolution imaging and microstructural characterization. Where applicable, scanning transmission electron microscopy (STEM) observations were conducted using the STEM mode available on the instrument. Thin foils suitable for TEM and STEM analyses were prepared using a conventional multi-step procedure to obtain electron-transparent regions. Initially, bulk samples were sectioned and mechanically ground from an initial thickness of approximately 1 mm down to  $\sim 100 \mu\text{m}$  in order to reduce the material thickness to a level suitable for subsequent fine thinning. Disc-shaped specimens were then prepared and subjected to electrochemical polishing using a Struers TenuPol system, which selectively removes material to produce thin areas with minimal mechanical deformation. To further enhance specimen quality, a final ion polishing step was carried out using a GATAN DuoMill. This step helped eliminate residual surface damage and improved surface uniformity, which is critical for reliable TEM imaging and analysis. The ion milling conditions were carefully controlled to limit the introduction of artifacts.

### 3.3.1.3. X-ray Computed Tomography

Generally speaking, X-ray computed tomography (XCT) is a nondestructive 3D imaging technique that scans the internal structure of an object from a series of X-ray projections acquired at different angles [335]. In the context of metallic materials, XCT operates on the principle of differential X-ray attenuation, which depends on material density, atomic number, and thickness [336]. During a scan, the specimen is rotated between an X-ray source and a detector, and the recorded projection data are reconstructed into volumetric datasets using numerical algorithms [337]. The resulting voxel-based representation enables visualization and quantitative analysis of internal features without sectioning or altering the material. In metallic materials research and industrial inspection, CT is extensively used to characterize internal defects and structural features such as porosity, cracks, inclusions, lack of fusion, and dimensional deviations [338–340]. It is particularly valuable for evaluating metal matrix composites [341], additively manufactured metals [342], and welded components [343], where internal integrity is critical to performance. In fact, the XCT method provides unique three-dimensional insight into the internal state of metallic components and support quality control, failure analysis, and materials development with a level of detail unattainable by conventional surface-based inspection methods [342].

The X-ray computed tomography test was performed to obtain three-dimensional images of Mg filaments embedded within the Cu matrix. The measurements were carried out using a Phoenix V|tome|x S X-ray computed tomography system (Waygate Technologies, formerly GE Sensing and Inspection Technologies / Phoenix X-ray, Wunstorf, Germany), which was equipped with an X-ray source, a precision rotation stage, and a flat-panel detector [344]. During the test, a cone beam of polychromatic X-radiation was generated by accelerating electrons emitted from a tungsten filament towards a tungsten target. The sample was mounted on the rotation stage and rotated during image acquisition while a series of two-dimensional radiographic projections were collected by the detector. In order to achieve sufficient X-ray penetration through the Cu/Mg composite while maintaining adequate contrast between the constituent phases, the accelerating voltage and tube current were carefully selected. The tube voltage primarily controlled the penetration of the X-ray beam, whereas the tube current governed the photon flux and image brightness. The test was performed at an accelerating voltage of 200 kV and a tube current of 350  $\mu$ A. Three-dimensional visualization and segmentation of the Mg filaments from the Cu matrix, based on grayscale contrast, were subsequently performed using the Drishti software package (Ajay Limaye, ANU Vizlab).

Multiple renderings were generated from different viewing angles in order to illustrate the spatial distribution and morphology of the Mg filaments within the Cu matrix.

#### **3.3.1.4. Compression Test**

A series of compression tests were carried out at RT using a Zwick universal testing machine at a constant crosshead speed of 2 mm/min. The compression specimens were prepared in accordance with the ASTM E9 standard, with a length-to-diameter (L/D) ratio of 2 [345]. During testing, compressive force and crosshead displacement were continuously recorded. These data were subsequently converted into engineering stress-strain curves by dividing the applied force by the initial cross-sectional area of the specimen and the displacement by the initial specimen height.

### **3.3.2. Results and Discussion**

#### **3.3.2.1. Composite Structure**

Figure 64 presents an SEM image obtained from the transverse cross section of the extruded rod, which shows the transverse macrostructure of the fabricated composite. The first observation is that the initially symmetrically positioned rods retained their symmetrical arrangement after undergoing the KoBo extrusion. This behavior arises from the symmetry of all elements with respect to the center, which was ensured by the deliberate design featuring one central Mg filament and six others placed at 60° intervals. Moreover, each of these six bores was positioned midway between the central bore and the outer edge to ensure the highest possible degree of geometric symmetry. Consequently, this design resulted in a symmetric material flow during the KoBo extrusion and, as a result, preserved the relative positions of the Mg filaments in the final extruded rod. In this regard, a similar observation was reported by Volkov et al. [346], who observed that their Cu/Mg composite preserved a highly symmetric arrangement after extrusion and subsequent drawing. In our case, the next observation is that, following the KoBo extrusion, the Mg filaments had an average diameter of  $\sim 1.15 \pm 0.12$  mm, and their initially round shape deviated toward a more serrated morphology. However, as shown in Figure 65, which presents a bird's-eye SEM view of the central Mg filament, no observable gaps were present at the Cu/Mg interface. This observation was further verified at multiple cross sections, all of which consistently confirmed the absence of any interfacial gaps.

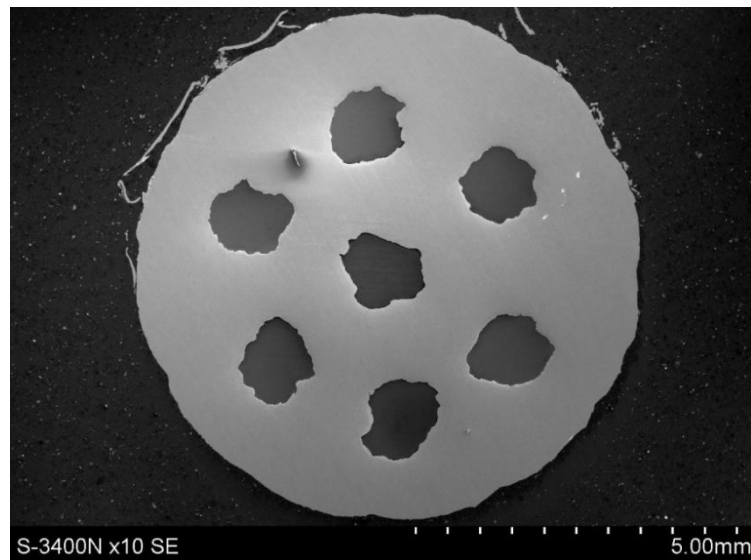


**Figure 64:** SEM image of the macrostructure of the fabricated Cu/Mg composite.

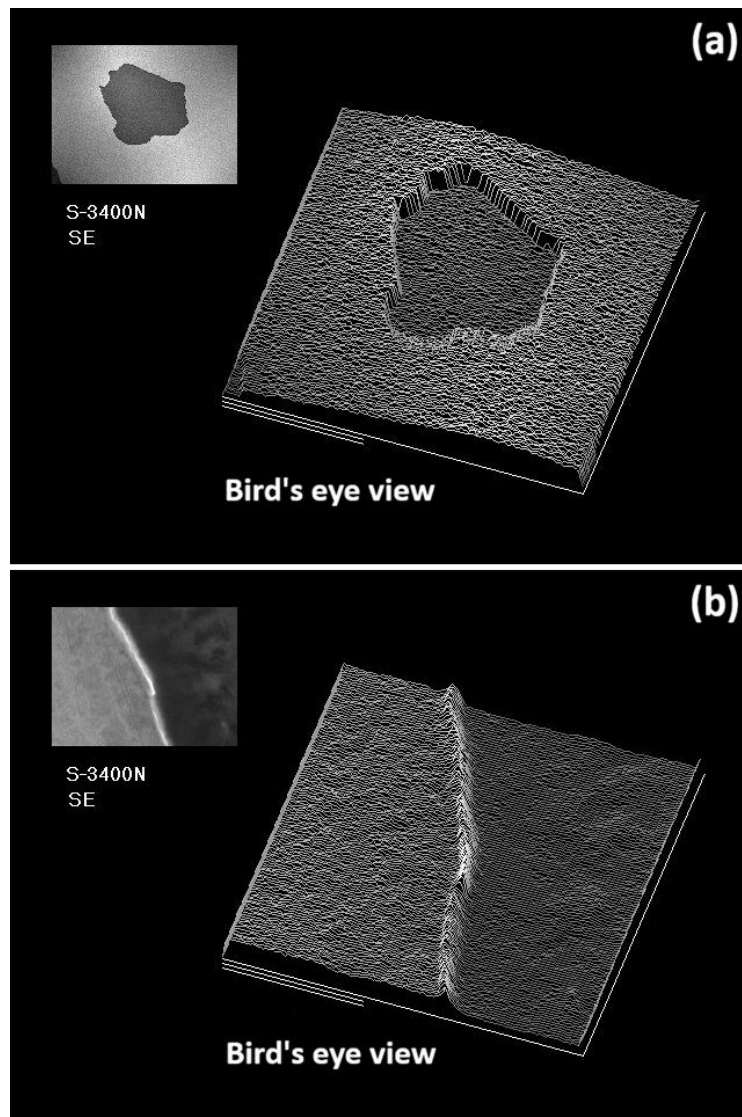


**Figure 65:** SEM bird's eye view of the central Mg filament within the Cu matrix in the fabricated Cu/Mg composite in (a) low magnification and (b) high magnification.

To characterize the bonding between the Cu matrix and Mg filaments, an SEM line scan analysis of the elemental distribution was performed across the transverse interface. The results, presented in Figure 66, show a relatively sharp compositional boundary between the Cu matrix and the Mg filament, without evidence of a reliable interdiffusion zone at the resolution of SEM-EDS. In fact, despite the initial deformation temperature being set to 200 °C and the actual temperature likely exceeding this value due to heat generated during the KoBo extrusion process, the deformation temperature at the deformation zone remained insufficient to promote a noticeable solid-state diffusion between Cu and Mg over the processing time. As a result, the bonding between the two phases is interpreted to be predominantly mechanical in nature rather than long-range atomic-scale mixing. In general, diffusion bonding in metallic systems requires a combination of sufficiently high temperature, sustained exposure time, and applied pressure to promote atomic mobility and intermixing at the interface [347–349]. However, in the present case, these conditions were not met to a degree sufficient to produce measurable diffusion. Consequently, the observed Cu-Mg interface is consistent with a mechanically bonded composite structure rather than a diffusion-bonded system. One final point that requires clarification is that, since pure Mg is significantly softer than pure Cu, mechanical grinding and polishing during sample preparation (which caused local pull-out or smearing of each phase) resulted in attaching both removed elements mostly to the Mg phase. This effect is clearly visible through the Cu particles observable in Figure 66(a) on the Mg region and also via the more fluctuated elemental distribution in Figure 66(b) within the Mg region. Conversely, a much lower amount of detached elements is observed on the Cu side due to its higher hardness, which is reflected by the relatively stable elemental distribution and minimal fluctuations detected within this region.

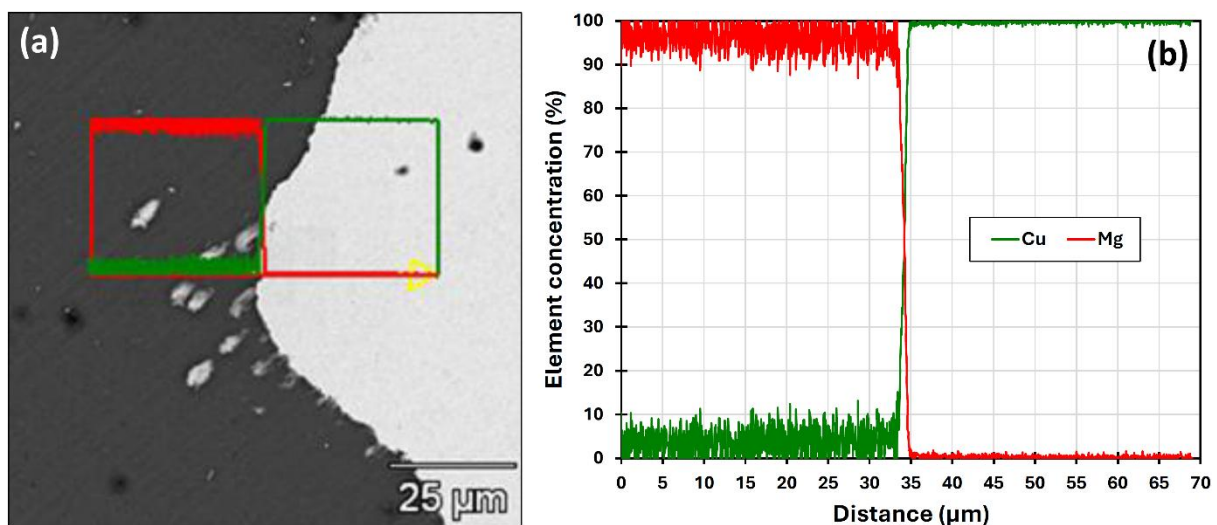


Figure 66: (a) SEM image and (b) line scan analysis result across the Cu/Mg interface.

### 3.3.2.2. XCT Scan Results

In order to provide a comprehensive overview of the internal structure of the extruded rod, representative images obtained from various cross-sections and viewing orientations in the XCT analysis are presented in Figure 67. Figure 67(a) shows the top view of the extruded rod, which is identical to the SEM image of the transverse cross-section shown in Figure 64. Figures 67(b) and 67(c) present the corresponding side views of the Y-Z and X-Z cutting planes shown in Figure 67(a), respectively. These orthogonal views provide complementary perspectives of the internal configurations and clearly demonstrate that the Mg filaments are uniformly distributed and continuously aligned along the longitudinal (extrusion) direction within the Cu matrix. In addition, the 3D images shown in Figures 67(d) and 67(e) provide an integrated visualization of the internal filament architecture and allow for a more intuitive understanding of their spatial arrangement. They also further confirm the structural integrity and continuity of the Mg filaments throughout the Cu matrix, with no observable interruptions, fractures, or discontinuities along their longitudinal extrusion direction. Another important observation from Figure 67 is that the continuous and straight Mg rods after extrusion confirm highly stable KoBo processing conditions, as any deviation from the initially set parameters would have disturbed their alignment and symmetry.

At this stage, it is essential to emphasize one of the key advantages of the fabricated composite structure compared to conventional Cu-Mg alloys, which is its reduced density. This lower density contributes directly to improved weight efficiency, which is particularly advantageous for applications such as transportation systems and aerospace components, where minimizing mass is critical and even a modest reduction in density can lead to meaningful improvements in overall performance and energy efficiency. Prior to extrusion, the theoretical density of the composite was calculated by considering the respective volume fractions and densities of the Cu matrix and the embedded Mg filaments. Based on this Rule of Mixtures approach, a theoretical density of  $8.13 \text{ g/cm}^3$  was expected for this composite structure, which corresponds to an approximate 9.3% reduction relative to the density of pure Cu ( $\sim 8.96 \text{ g/cm}^3$ ). This value was calculated assuming an idealized condition in which a uniform and fully developed filament distribution could be achieved via KoBo extrusion. However, following KoBo extrusion, the density of the composite was experimentally measured to be approximately  $7.85 \text{ g/cm}^3$  in the steady-state region of the extruded rod. This value corresponds to a reduction of  $\sim 12.4\%$  relative to pure Cu and indicates an even greater weight-saving effect than initially predicted.

The lower experimentally measured density of the fabricated composite, compared with its calculated theoretical value, can simply be attributed to non-steady-state conditions during the initial stage of the extrusion process. As previously mentioned, the initial portion of the extruded rod consisted predominantly of Cu, as the Mg filaments had not yet been effectively incorporated into the extruded rod. However, with continued extrusion, a steady-state condition was progressively established, and the Mg filaments became uniformly distributed and continuously aligned within the Cu matrix. Consequently, the actual Mg-to-Cu volume fraction in the steady-state region was actually higher than the value assumed in the initial theoretical calculation. This increased relative content of the lower-density Mg phase led to a further reduction in the overall composite density and accounted for the experimentally measured density value being lower than the theoretical calculation.

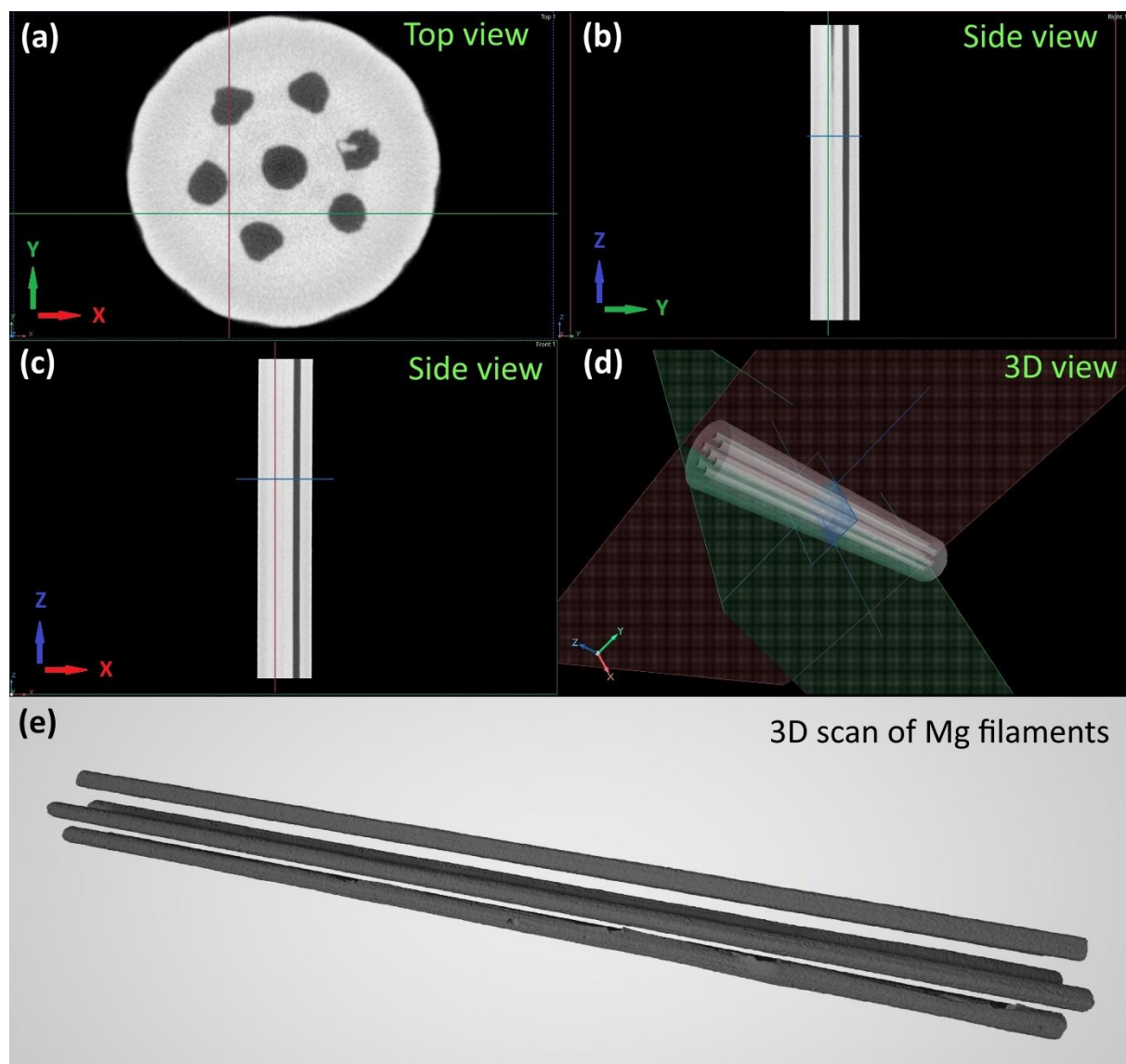


**Figure 67:** XCT analysis results in (a) top view, (b,c) side view, and (d) 3D view; (e) 3D scan of Mg filaments.

### 3.3.2.3. Post-Extrusion Microstructures

#### 3.3.2.3.1 EBSD Analysis of Cu Matrix

Figure 68(a) shows the IPF map obtained from EBSD analysis of the Cu matrix after KoBo extrusion, which reveals a predominantly equiaxed grain structure with an average grain size of  $11.2 \pm 3.1 \mu\text{m}$ . Considering the very large imposed deformation during KoBo extrusion (nominal extrusion ratio of 25) along with the starting temperature of the deformation process being approximately 200 °C, the formation of such a fine equiaxed microstructure suggests that significant recovery and recrystallization processes occurred during deformation and shortly after deformation while the extruded rod remained at an elevated temperature. Figure 68(b) presents the corresponding GOS map, from which it can be observed that the majority of grains exhibit GOS values below 2° (red). Such low GOS values are generally associated with recrystallized or strongly recovered grains characterized by minimal internal orientation gradients and low intragranular misorientation [350]. Therefore, the predominance of low-GOS grains indicates that a large fraction of the grains attained a low stored deformation state indicative of extensive recovery and recrystallization processes during and after KoBo extrusion, before the sample completely cooled down to RT. As mentioned above, it is important to consider that such intense plastic deformation itself also generates heat, and thus the actual deformation temperature was likely above 200 °C.

This interpretation is further supported by Figure 68(c), which shows the corresponding KAM map. The KAM distribution indicates that most of the analyzed area exhibits values well below 5°, with many regions approaching zero. Such low KAM values indicate a relatively low degree of local lattice distortion and a reduced density of geometrically necessary dislocations, which is characteristic of strongly recovered and recrystallized microstructures [351]. Hence, the overall EBSD results are consistent with extensive deformation-induced recovery accompanied by recrystallization in the Cu matrix during and shortly after KoBo extrusion. Figure 68(d) shows the corresponding (001) pole figure and indicates a shear-modified texture with a peak intensity of 6.837, where the {100} crystallographic planes are tilted by approximately 45° relative to the extrusion direction. Such texture evolution is characteristic of the non-uniform metal flow and intense shear deformation generated during KoBo extrusion. The coexistence of a distinct deformation-related texture together with low KAM and GOS values is consistent with extensive recovery and recrystallization occurring during deformation and/or shortly after extrusion while the material remained at an elevated temperature.

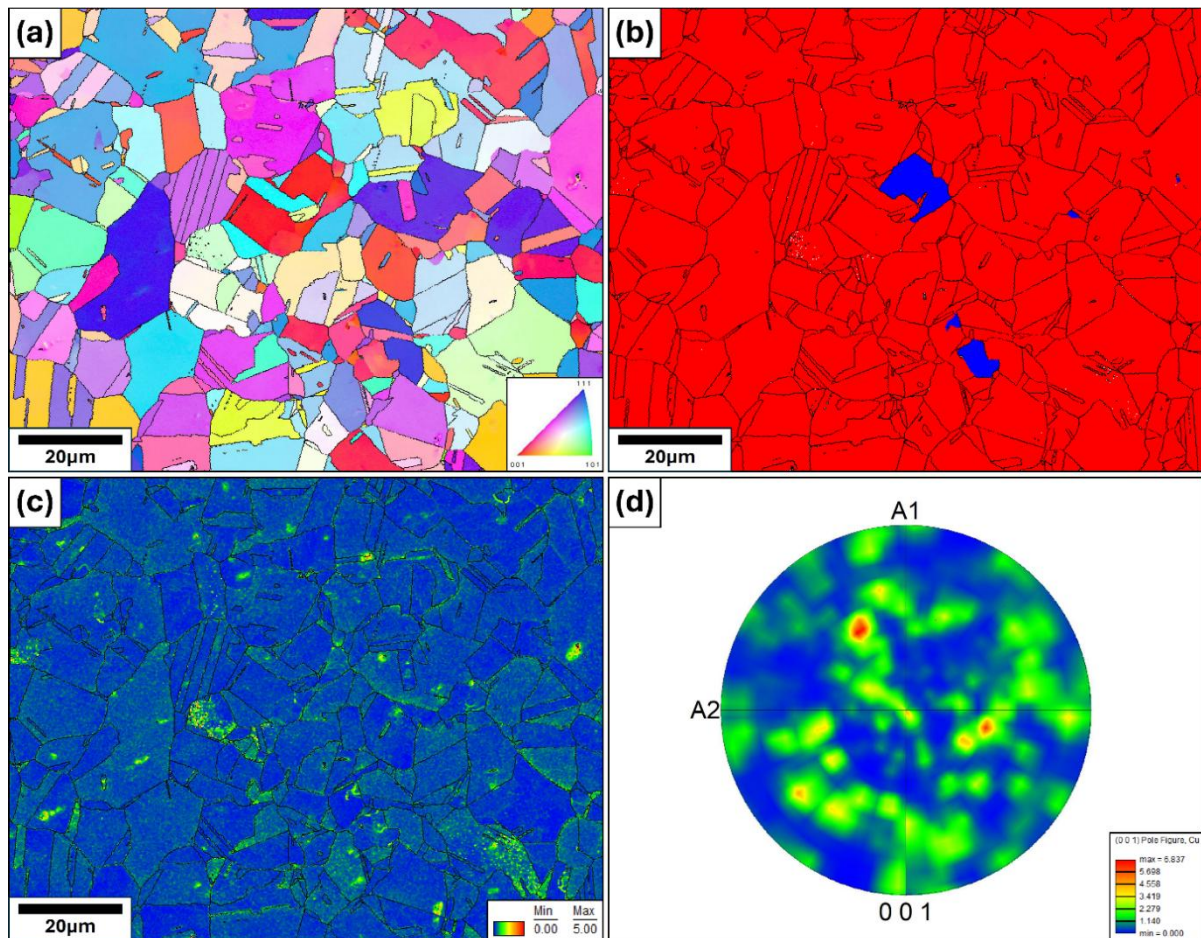


**Figure 68:** (a) IPF, (b) GOS (grain with GOS < 2° are shown in red), (c) and KAM maps, and (d) pole figure of the Cu matrix.

### 3.3.2.3.2. TEM Analysis of Cu Matrix

For high-resolution characterization of the Cu matrix microstructure, TEM analysis was performed to further investigate the substructure developed under the thermomechanical conditions of KoBo extrusion. Figure 69 presents TEM images of the Cu matrix in two different regions located between the central Mg filament and the surrounding Mg filaments. These images, which were intentionally taken from highly deformed regions, reveal a microstructure characterized by deformation-induced dislocation cell structures, subgrain formation, and heterogeneous dislocation distributions. The dark, irregular perimeter surrounding the brighter cell interior is attributed to regions of high dislocation density, corresponding to dense dislocation walls (DDWs), while the cell interiors exhibit a comparatively lower dislocation density. Such cellular structures have been widely reported in FCC metals such as Cu, which is generally classified as a medium-SFE (reported SFE values for pure Cu vary depending on the measurement method, but values in the range of ~40–45 mJ/m<sup>2</sup> at RT are commonly reported and widely accepted [352–355]), as well as in Cu-based alloys subjected to different

SPD processing routes [356–360]. The observed dislocation cell structures indicate substantial dislocation rearrangement during deformation, which is consistent with dynamic recovery processes in Cu. These rearrangements promote the formation of metastable cellular and subgrain structures characterized by heterogeneous dislocation distributions.

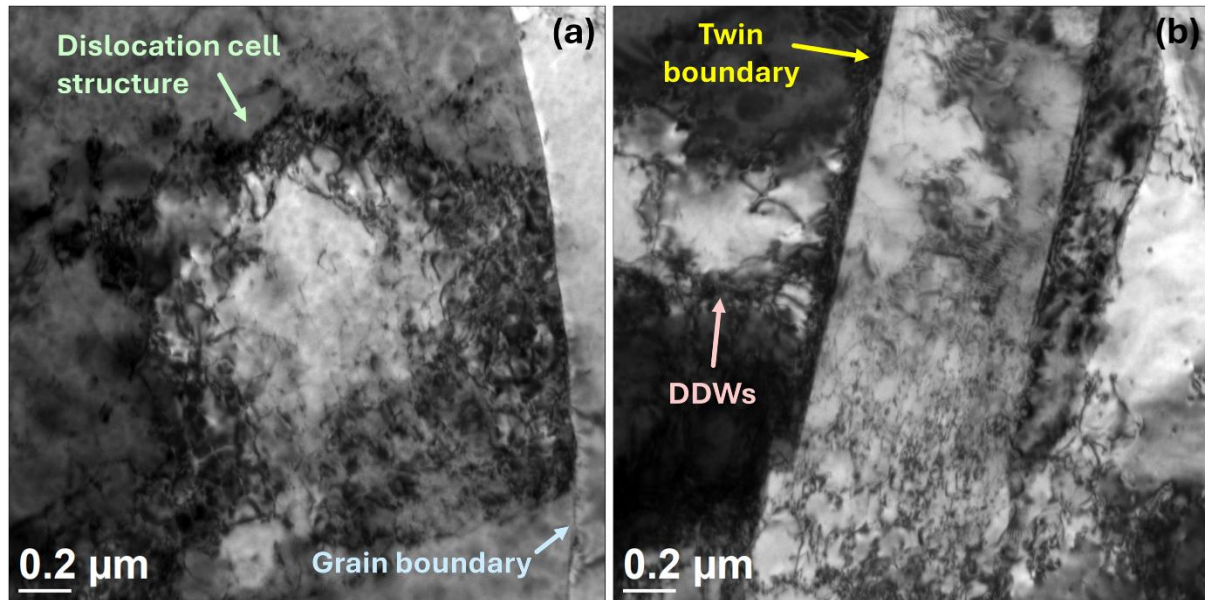


**Figure 69:** TEM images of the Cu matrix in the fabricated composite.

### 3.3.2.3.3. TEM Analysis of Mg Filaments

Figure 70 presents STEM images of the central region of the central Mg filament and another neighboring Mg filament. In both microstructures, individual Mg grains with an average diameter of  $354 \pm 119$  nm are distinguishable. This UFG microstructure with its well-distinguishable grain boundaries is attributed to the cumulative grain refinement induced by the initial forward extrusion of the Mg rods and their subsequent KoBo extrusion inside the Cu matrix. This observation is consistent with previously reported grain structures in pure Mg subjected to SPD processing [361–363]. As an example, Wei et al. [364] reported an average grain size of approximately 273 nm in high-purity Mg (99.9%) after HPT processing at RT under a pressure of 1 GPa for 10 revolutions, with disk specifications of 10 mm diameter and ~2 mm thickness.

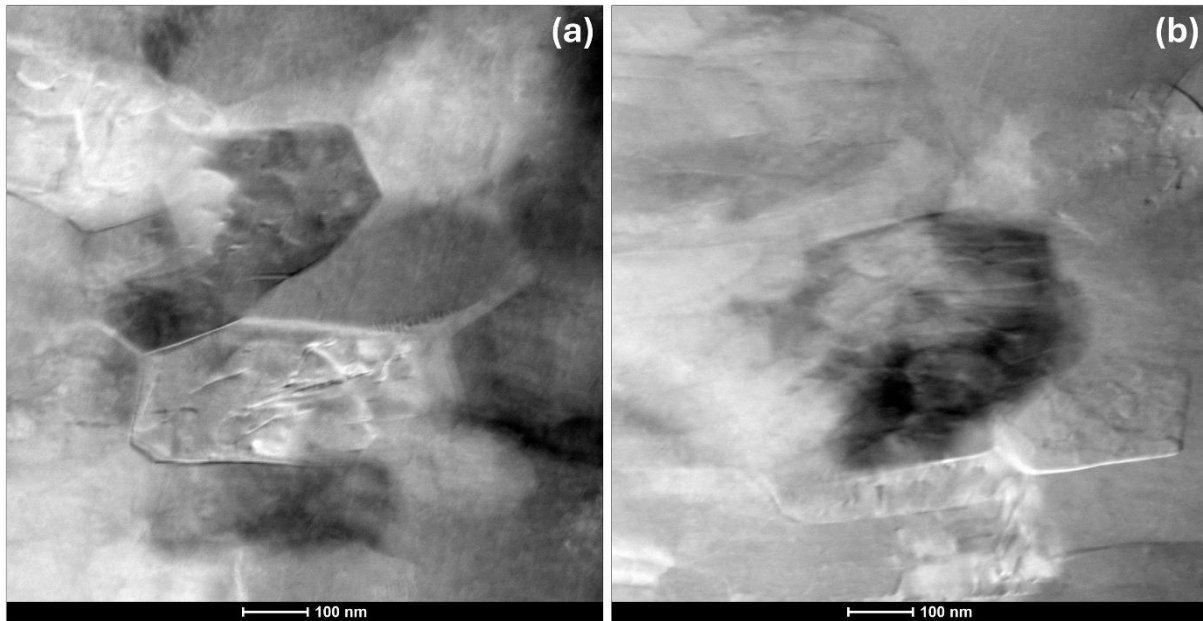


**Figure 70:** STEM images of the (a) central and (b) outer Mg filaments within the fabricated composite structure.

#### 3.3.2.4. Hardness Test

As represented in [Figure 71](#), the average hardness of the initial Cu matrix was measured as  $47.1 \pm 6.3$  HV, and the average hardness of the initial Mg filaments was measured as  $34.5 \pm 3.8$  HV, which align well with previously reported hardness values for annealed pure Cu [365,366] and extruded pure Mg [367,368]. Following KoBo extrusion, hardness measurements were performed at multiple locations within both the Cu matrix and the Mg filaments to improve statistical reliability. The overall average hardness values increased to  $67.5 \pm 1.8$  HV for the Cu matrix and  $38.2 \pm 3.6$  HV for the Mg filaments. In addition, no noticeable difference was observed between the hardness of the central Mg filament and that of the surrounding filaments, which is consistent with the similar microstructural characteristics observed between these regions ([Figure 70](#)). According to the obtained results, the average hardness of the Cu matrix increased by approximately 43% following KoBo extrusion, and the Mg filaments exhibited an average hardness approximately 11% higher than that of the initial Mg filaments. These hardness enhancements can be attributed to the combined effects of grain refinement, residual dislocation strengthening, and deformation-induced substructures formed during KoBo extrusion within both the Cu matrix and Mg filaments.

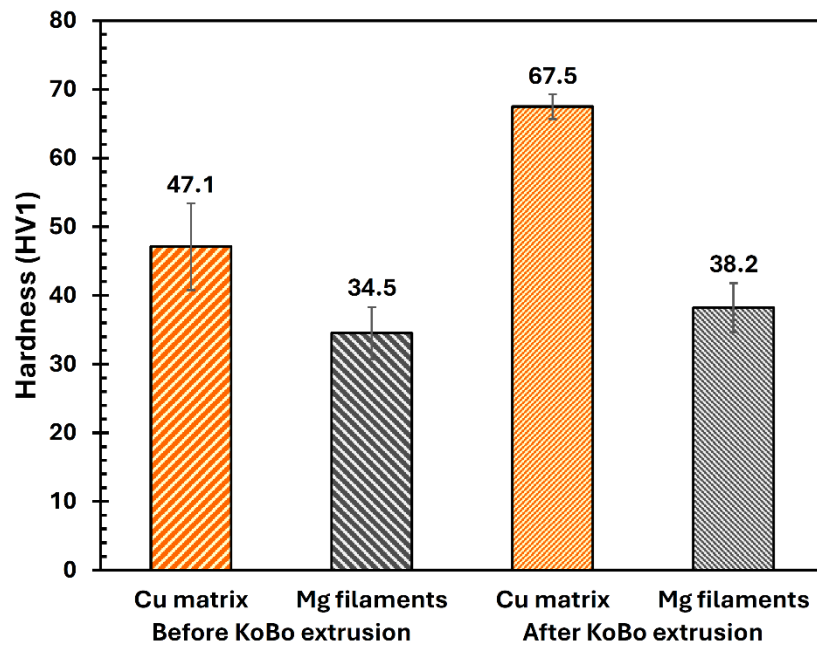


**Figure 71:** Hardness measurement results for the Cu matrix and Mg filaments before and after KoBo extrusion.

### 3.3.2.5. Compression Test

Due to the structural design of the fabricated composite, standard tensile testing according to ASTM requirements was not suitable for obtaining reliable results. Specifically, ASTM E8/E8M tensile testing of metallic materials defines standardized specimen geometries with a reduced and uniform gauge section to ensure controlled stress conditions during testing [369]. In the present case, machining the composite to meet these requirements would have required reducing the cross-sectional area of the Cu matrix and reshaping the material into a standard dog-bone geometry. Such a reduction would alter the original geometry of the composite by increasing the relative cross-sectional contribution of the Mg filaments compared to the Cu matrix. This change would modify the load-sharing behavior between the constituent phases and, consequently, lead to tensile test results that would not be representative of the actual mechanical behavior of the fabricated composite structure. Therefore, to avoid introducing this geometric and mechanical inaccuracy, tensile testing was not performed; instead, compression testing was carried out as a more suitable method for evaluating the mechanical response of the composite without altering its cross-sectional configuration.

Figure 72 presents the results obtained from compression tests for two different compression levels of 61% and 79%. In both conditions, the compressive YS of the composite structure was determined to be approximately  $119 \pm 12$  MPa using the 0.2% offset method. However, this value is much lower than previously reported compressive YS values for SPD-processed pure

Cu [370]. For instance, Yu et al. [371] reported a compressive YS of 221 MPa for extruded pure Cu. This difference, however, can be attributed to the presence of Mg filaments, which exhibit a significantly lower compressive yield strength than the Cu matrix. For example, Li et al. [372] reported a compressive YS of ~83 MPa for ECAP-processed pure Mg. Under compressive uniaxial loading, the Mg phase yields at earlier stages of deformation and contributes significantly to the initial nonlinearity of the macroscopic stress-strain response, while the Cu matrix provides increasing load-bearing capability as deformation progresses. Accordingly, the YS of the composite reflects the combined deformation behavior of both constituents, with a strong influence from the early plastic deformation of the relatively softer Mg filaments.

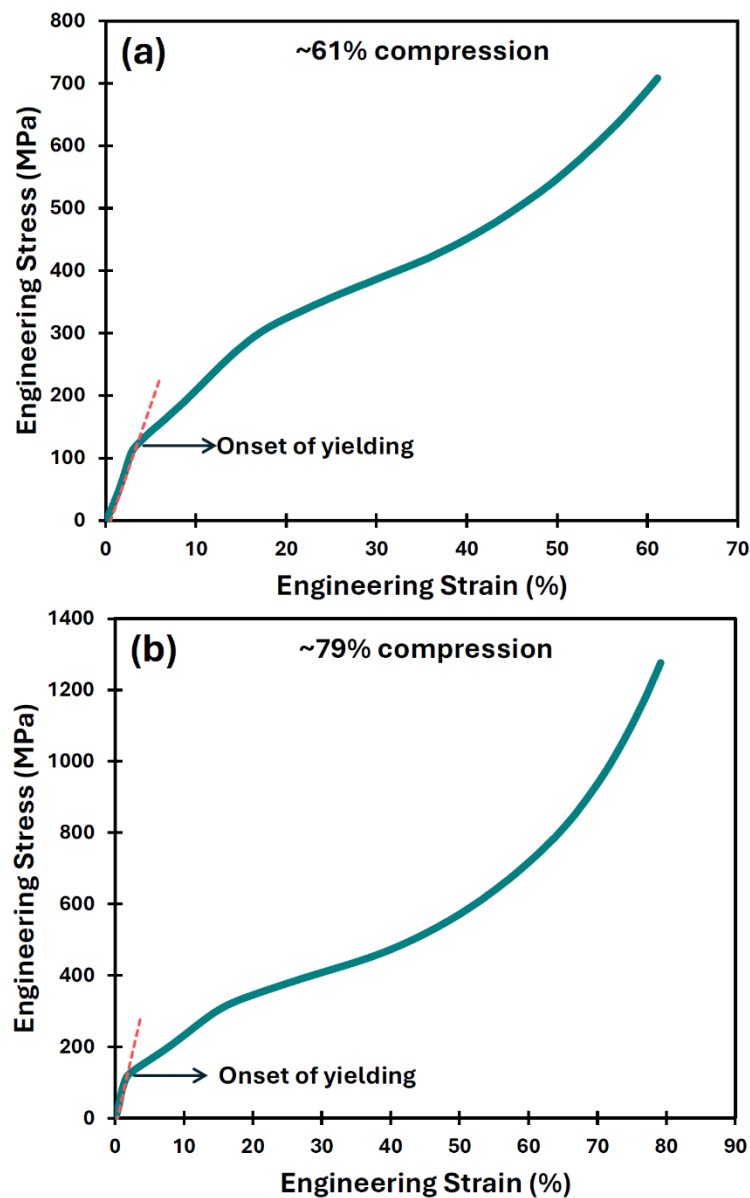


**Figure 72:** Compressive mechanical response of the fabricated Cu/Mg composite at deformation levels of (a) 61% and (b) 79% compressions.

### 3.3.2.6. Microstructure of Cu Matrix after Compression

Figure 73 presents the EBSD results for the Cu matrix following the compression tests described above. Similar to Figure 68, grains with a GOS value below  $2^\circ$  are highlighted in red. The IPF maps reveal that the initially equiaxed grain structure obtained through KoBo extrusion has evolved into a heavily deformed and heterogeneous microstructure, comprising both large deformed grains as well as some smaller grains that are consistent with deformation-induced grain subdivision and possible CDRX along with deformation-induced subgrains.

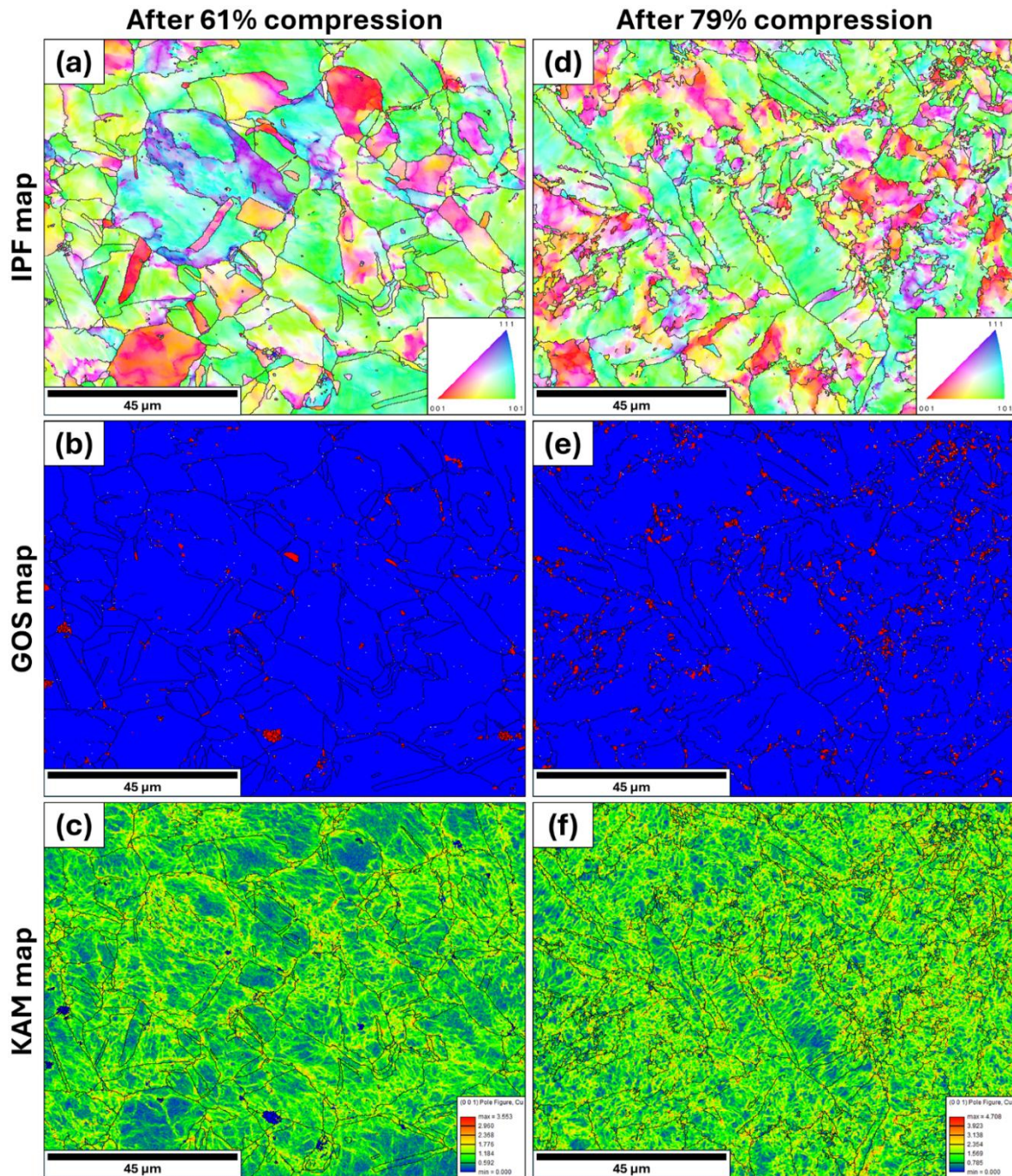


**Figure 73:** (a,d) IPF, (b,e) GOS, and (c,f) KAM maps of the Cu matrix following compressive deformation to strain levels of 61% and 79%, respectively.

A higher fraction of grains exhibiting GOS values below  $2^\circ$  was observed after 79% compression compared to 61% compression. Since low GOS values are generally associated with recrystallized or strongly recovered grains that possess lower internal misorientation, the increase in the fraction of such grains suggests enhanced recovery and rearrangement of deformation-induced defects, with possible contributions from recrystallization-related processes at higher strain levels [373]. In this case, the fraction increased from 1.2% after 61% compression to 3.5% after 79% compression. This trend suggests that the higher deformation level promoted progressive subgrain subdivision and enhanced recovery, with possible contributions from CDRX-related mechanisms. The KAM maps further support these observations by showing a higher average KAM value after 79% compression compared to 61% compression. Because KAM is related to local lattice curvature and geometrically necessary dislocation density, the increase in KAM indicates a higher level of localized lattice distortion and accumulated strain within the microstructure [374]. This suggests that, although recovery and recrystallization activity became more pronounced at the higher compression level, substantial plastic deformation was still retained in most parts of the material. Consequently, the microstructure after 79% compression remained heterogeneous, consisting of regions associated with recovery/recrystallization alongside areas with elevated dislocation density and residual deformation.

### **3.3.2.7. Electrical Conductivity**

Consistent with previous approaches, the 4-wire Kelvin resistance measurement method was used to evaluate the electrical conductivity of the fabricated composite structure. The composite exhibited an electrical conductivity of  $52.6 \pm 1.2$  MS/m, which indicates a high level of electrical conductivity comparable to highly conductive engineering materials. This result is consistent with the expected conductivity behavior of the composite, as the continuous Cu matrix provided the dominant pathways for electron transport. The reduction in electrical conductivity compared with annealed pure Cu can be attributed to the presence of Mg filaments and deformation-induced microstructural defects within the Cu matrix, such as increased dislocation density, residual stresses, and other lattice imperfections, as well as the presence of Cu/Mg interfaces, which can act as additional electron scattering sites and consequently increase electrical resistivity.

### 3.3.3. Conclusions of Approach 3

In this approach, a density-reduced Cu/Mg composite structure composed of a pure Cu matrix with pure Mg filaments embedded within it was fabricated using the KoBo extrusion method. This configuration was specifically designed to combine the excellent electrical conductivity of pure Cu with the low density of the Mg filaments. Based on the obtained results, the following conclusions can be drawn:

- 3.1. The overall configuration and symmetrical arrangement of the initial composite structure were preserved after KoBo extrusion. In the steady-state region of the extruded rod, the distribution of Mg filaments closely resembled the initial arrangement. However, due to the higher proportion of Cu in the initial segment of the extruded rod, the Mg-to-Cu ratio in the steady-state region exceeded the theoretically calculated value. As a result, the experimentally measured density of the extruded rod ( $7.85 \text{ g/cm}^3$ ) was  $\sim 3.4\%$  lower than the corresponding theoretical density ( $8.13 \text{ g/cm}^3$ ).
- 3.2. SEM line scan analysis across the Cu/Mg interface revealed a sharp compositional boundary with no detectable interdiffusion within the detection capability of SEM/EDS, which indicated that the bonding formed during KoBo extrusion was predominantly mechanical in nature rather than resulting from diffusion-driven processes.
- 3.3. The 3D XCT analysis demonstrated that the Mg filaments in the steady-state region of the extruded rod were continuously aligned and uniformly distributed along the extrusion direction within the Cu matrix. Various cross-sectional views and 3D reconstructions further confirmed the structural integrity of the filaments throughout the composite, with no evidence of fragmentation, discontinuity, fracture, or other structural interruptions.
- 3.4. EBSD analysis of the Cu matrix after KoBo extrusion revealed a fine equiaxed grain structure with low GOS and KAM values, together with a shear-modified texture, which collectively suggested that warm KoBo extrusion was accompanied by substantial dynamic recovery and recovery/recrystallization-related microstructural evolution during and shortly after deformation.
- 3.5. TEM/STEM analysis revealed that KoBo extrusion produced a heterogeneous substructure in the Cu matrix, composed of dislocation cells and subgrains indicative of significant recovery, while the Mg filaments developed a UFG microstructure ( $\sim 354 \text{ nm}$ ), consistent with SPD-induced grain refinement typical of SPD-processed pure Mg.
- 3.6. KoBo extrusion significantly increased the hardness of both constituents in the Cu/Mg composite, with the Cu matrix showing a  $\sim 43\%$  increase and the Mg filaments a  $\sim 11\%$

increase, which were attributed to grain refinement, residual dislocation strengthening, and deformation-induced substructures developed through KoBo extrusion.

3.7. Compression testing revealed that the Cu/Mg composite exhibited a reduced initial YS of  $\sim 119 \pm 12$  MPa, consistent with early plastic yielding of the relatively softer Mg filaments. Consequently, the onset of plastic deformation reflected the combined mechanical response of both constituent phases rather than the Cu matrix alone.

3.8. The 4-wire Kelvin electrical resistance measurements showed that the composite structure exhibited an electrical conductivity of  $52.6 \pm 1.2$  MS/m, where the continuous Cu matrix provided the primary conduction pathways. The reduction in conductivity compared with annealed pure Cu was attributed to deformation-induced microstructural features, including increased dislocation density, lattice imperfections, and Cu/Mg interfaces, which increased electron scattering and, consequently, resulted in a slight decrease in the overall electrical conductivity.

## 4. Discussion and Analysis of Research Findings

As outlined in the preceding sections, the primary objective of this PhD research was to develop HSHC Cu-Mg-based alloys that simultaneously exhibit desirable mechanical properties and satisfactory electrical conductivity through the combined application of appropriate alloying strategies, controlled heat treatment routes, and deformation strengthening via SPD processing. Achieving this objective required a systematic approach to both material design and processing methodology, with careful selection and optimization of each stage to ensure a balanced combination of strength, ductility, and electrical conductivity. To attain this goal, three main approaches were employed, each based on tailoring the microstructure through thermomechanical processing. The results and insights gained from these approaches have been discussed in detail in the previous sections. In this section, the focus is shifted to a systematic analysis and interpretation of the integrated research findings, with particular emphasis on highlighting the relationships between processing, microstructure, and the resulting mechanical properties and electrical conductivity of the developed alloys.

First of all, it was found that the addition of 0.7 wt.% Mg to Cu (which is below the solubility limit of Mg in Cu [43]) resulted in a favorable improvement in mechanical properties with an acceptable reduction in the electrical conductivity. In fact, since the Mg content remained within the solubility limit, the Cu-0.7Mg (wt.%) alloy retained a single-phase solid solution microstructure, which resulted in its enhanced mechanical properties while also preserving its

electrical conductivity within an acceptable range. This combination of increased mechanical strength and an acceptable decrease in electrical conductivity makes this composition an attractive option for being used in applications that require a balanced combination of mechanical and electrical performance. However, it is essential to note that the optimal Mg content ultimately depends on the specific performance requirements of the intended application. As mentioned earlier, the addition of Mg improves mechanical properties at the expense of electrical conductivity due to enhanced electron scattering. Accordingly, for applications in which mechanical strength and load-bearing capability are the dominant design criteria, a relatively higher Mg content may be justified despite a moderate decrease in conductivity. In contrast, for applications where efficient electrical current transmission and minimal resistive losses are critical, lower Mg additions are more appropriate to preserve the conductivity. Therefore, the selection of Mg content should be guided by a careful assessment of the required balance between mechanical performance and electrical efficiency.

The next important aspect observed in this study is the effectiveness of second-phase hardening as a strengthening mechanism when the alloying element is added in very low amounts. In this study, Ca was found to be an excellent alloying element for this purpose. It was demonstrated that the addition of only 0.1 wt.% Ca to a single-phase Cu-0.7Mg (wt.%) alloy led to the formation of the Cu<sub>5</sub>Ca intermetallic phase, which effectively impeded dislocation motion during deformation and thereby contributed to enhanced work hardening and promoted microstructural refinement during processing, including an increased fraction of HAGBs associated with dynamic recrystallization processes. Consequently, an increase in the overall strength of the alloy was achieved. Notably, this strengthening effect was obtained without a significant reduction in electrical conductivity, as the primary contribution to the decrease in conductivity originated from Mg-induced solid solution scattering rather than from the sparsely distributed Cu<sub>5</sub>Ca second-phase particles. Therefore, the controlled addition of Ca can be nominated as an effective strategy for enhancing the mechanical properties of Cu alloys through second-phase hardening while largely preserving their functional electrical performance.

Different heat treatment routes were also applied to both binary Cu-0.7Mg (wt.%) and ternary Cu-0.7Mg-0.1Ca (wt.%) alloys in order to evaluate their influence on microstructural evolution and the resulting changes in mechanical properties. Particularly for the ternary alloy, the results indicated that short-term annealing at 650 °C for 90 min promoted partial spheroidization of the Cu<sub>5</sub>Ca intermetallic particles. More specifically, a fraction of the initially

irregular and angular particles underwent morphological rounding, while the remainder retained their original angular morphology. This microstructural modification led to a slight improvement in ductility, as demonstrated by an increase in the measured EL values obtained from tensile testing of the heat-treated samples. This improvement was attributed to the reduced sharpness of particle corners and edges following heat treatment. In general, it is widely accepted that the sharp-edged intermetallic particles in metallic materials act as local stress concentration sites during plastic deformation and contribute to localized stress intensification and strain incompatibility between the particles and the surrounding matrix [375–378]. Under such conditions, these sites may facilitate crack initiation and subsequent propagation and potentially contribute to premature failure under loading [379,380]. By reducing the severity of these local stress concentrations at the microscopic level, the applied heat treatment improved the ability of the ternary alloy to accommodate plastic deformation prior to fracture. This behavior is a well-established feature of spheroidization annealing and has been widely reported in various Cu-based alloy systems [381–383].

The applied heat treatments, consisting of annealing at 650 °C for 90 min or 75 min followed by water quenching, produced an increased dislocation density in the vicinity of the Cu<sub>5</sub>Ca intermetallic particles. This was attributed to the mismatch in CTE values between the Cu-Mg solid solution matrix and the Cu<sub>5</sub>Ca intermetallic phase. During annealing at 650 °C, both phases expand according to their respective CTE values. Upon subsequent rapid cooling (water quenching), the differing contraction rates generated thermal mismatch strains. The unconstrained thermal strain in each phase is given by [384]:

$$\varepsilon_{th} = \alpha \Delta T \quad (9)$$

where  $\alpha$  is the linear coefficient of thermal expansion and  $\Delta T$  is the temperature change. Owing to the CTE mismatch, differential contraction led to strain incompatibility at the particle-matrix interfaces [385]. The relatively rigid Cu<sub>5</sub>Ca particles constrained the surrounding ductile Cu-Mg matrix and resulted in the development of internal residual stresses to maintain interfacial compatibility. When these thermally induced stresses exceeded the local yield strength of the matrix, plastic deformation occurred via the generation and multiplication of dislocations (primarily geometrically necessary dislocations). Stress concentrations at the interfaces promoted the accumulation of these dislocations around the Cu<sub>5</sub>Ca particles and produced a locally elevated dislocation density after quenching.

Regarding the influence of processing history, SPD processing was found to be highly effective in introducing extremely large plastic strains into the alloys, generating a very high dislocation density and consequently a substantial stored strain energy within the microstructure. The stored energy associated with dislocations can be approximated as [386]:

$$E \approx \frac{1}{2} G b^2 \rho \quad (10)$$

where  $G$  is the shear modulus,  $b$  is the Burgers vector, and  $\rho$  is the dislocation density. The dramatic increase in  $\rho$  during SPD processing provides a significant thermodynamic driving force that, despite limited atomic mobility at RT, can promote CDRX [387]. Under these conditions, dislocations rearrange into cell structures and subgrains, and progressive lattice rotation increases the misorientation across subgrain boundaries. As LAGBs gradually evolve into HAGBs, new strain-free or low-strain grains are formed through a continuous boundary transformation process rather than classical nucleation-and-growth recrystallization [388]. However, in the case of this study, due to limited grain boundary mobility at RT, together with solute drag effects arising from Mg atoms in solid solution and Zener pinning exerted by Cu<sub>5</sub>Ca intermetallic particles, boundary migration was significantly restricted. As a consequence, recrystallization remained partial rather than complete.

Regarding the MaxStrain processing, the results also demonstrated that, under the applied deformation conditions, while 40 passes resulted in slightly superior performance, 20 passes were already sufficient to produce a microstructure and mechanical strength characteristic of HSHC Cu alloys. This behavior can be explained by the SPD-induced progressive microstructural refinement. As mentioned earlier, with an increasing number of passes, the accumulated strain led to intense dislocation multiplication and interaction, which promoted the formation of dislocation cells and subgrain boundaries that gradually evolved into HAGBs through CDRX, although some regions still contain LAGBs. Consequently, the grain size was significantly reduced, and the strength increased primarily through grain boundary strengthening, as described by the Hall-Petch relationship (Equation (5)), together with contributions from dislocation strengthening. After 20 passes, the microstructure had already undergone substantial refinement, which resulted in mechanical properties consistent with those required for HSHC Cu alloys under the applied conditions. Increasing the number of passes to 40 led to further grain refinement, a higher fraction of HAGBs, and a more homogeneous strain distribution throughout the material and resulted in an additional increase in strength. Consequently, while 40 passes provided slightly superior mechanical properties

compared to 20 passes, the improvement remained incremental, indicating that the material approached a saturation-like microstructural regime typical of severely deformed Cu-based alloys, where further strengthening becomes progressively limited.

The next important point to consider is that combining MaxStrain processing with additional deformation techniques, such as KoBo extrusion or forward extrusion at 400 °C, resulted in further refinement of the grain structure of the samples. This effect arose because the sequential application of these processes increased the overall equivalent strain experienced by the samples, which in turn increased the dislocation density and stored energy within their microstructure. The higher stored energy provided a stronger driving force for the occurrence of CDRX and facilitated the formation and evolution of new grains. As a result, the alloys subjected to the combined deformation developed a finer and more uniform microstructure, which contributed to improved mechanical properties. Importantly, this enhanced microstructural refinement was achieved without significantly compromising electrical conductivity, which enabled the processed samples to maintain the balance of properties required for classification as HSHC Cu alloys.

In addition to the processing parameters discussed above, it is important to consider fabrication cost when designing and producing HSHC Cu alloys, as economic efficiency plays an important role in their practical and industrial implementation. In general, the selection of alloying elements can have a noticeable impact on the overall production cost, particularly for large-scale manufacturing [389–391]. This is mainly due to the significant variation in the market prices of different alloying elements. In other words, even minimal changes in the chemical composition can lead to appreciable differences in total raw material cost. Among the elements shown in [Figure 3](#), Mg and Ca are relatively low-cost options compared with several of the other elements listed, especially when compared with precious metals such as Ag. From a cost-efficiency perspective, this makes Mg and Ca particularly attractive for scalable alloy design. In contrast, while the incorporation of high-cost elements into Cu alloys can bring a better set of mechanical properties and electrical conductivity, it can substantially increase material expenses and may therefore limit their economic competitiveness and industrial applicability, especially in applications requiring bulk production. Therefore, cost-effectiveness should be regarded as a key design criterion in the material selection process, particularly for mass production, where even small differences in the prices of alloying elements can translate into significant variations in overall manufacturing costs. Therefore, the use of Mg and Ca as alloying additions to Cu is advantageous not only because of the

advantages that they provide for mechanical properties enhancement but also because they enable a more cost-efficient alloy design strategy. As discussed above, when added in appropriate amounts, Mg and Ca can contribute to improved mechanical strength while generally maintaining acceptable electrical conductivity, therefore, offering a favorable balance between performance and cost. Consequently, their incorporation not only supports the development of HSHC Cu alloys with desirable mechanical and functional properties but also significantly enhances cost-effectiveness, which is a critical factor for large-scale fabrication and industrial deployment.

Approach 3 demonstrated the fabrication of a density-reduced Cu/Mg composite structure through KoBo extrusion, using pure Cu as the matrix and continuous pure Mg filaments as structural constituents, with the aim of combining the excellent electrical conductivity of pure Cu with the low density of pure Mg. This composite structure was specifically designed because it separated the functional roles of the constituent phases: Cu primarily carried electrical current and Mg acted as a density-reducing structural constituent. Following extrusion, the Mg filaments remained symmetrically distributed, continuous, and well aligned along the extrusion direction, as confirmed by SEM and XCT analyses. This indicated that the severe shear deformation associated with KoBo processing did not disrupt filament integrity. In addition, SEM-EDS line scans showed a sharp Cu/Mg interface with no measurable interdiffusion, which indicated that the bonding between Cu and Mg was predominantly mechanical rather than diffusion-driven. From a materials engineering perspective, this is important because limiting Cu-Mg interdiffusion suppresses the formation of brittle intermetallic phases and minimizes solute-related electron scattering and, consequently, helps preserve the electrical (and also thermal) conductivity.

The fabricated composite structure exhibited a measured density of  $7.85 \text{ g/cm}^3$ , which, together with its high electrical conductivity and mechanical performance, resulted in improved specific electrical conductivity and specific strength. These are two parameters that are especially important for advanced applications in aerospace systems, electrified transportation, and lightweight electrical conductors [392]. EBSD and TEM analyses revealed that the Cu matrix underwent extensive deformation-induced recovery accompanied by recrystallization-related microstructural evolution during KoBo extrusion (which was started at  $200 \text{ }^\circ\text{C}$ ) and resulted in the formation of a fine equiaxed grain structure together with dislocation cell substructures and shear-modified texture development. This combination of recovery and retained deformation substructure is particularly beneficial because it enables a balance

between conductivity and mechanical strength: recovery can contribute to reduced resistivity by lowering dislocation density, while grain refinement and residual dislocation structures maintain strengthening. Meanwhile, the Mg filaments developed a UFG structure ( $\sim 354$  nm), which indicated that the imposed plastic deformation was sufficient to activate substantial grain refinement even within the HCP Mg phase, which normally exhibits more limited slip activity at RT compared with FCC Cu.

Compression testing results highlighted the heterogeneous mechanical response of the composite, where the Mg filaments yielded earlier than the Cu matrix because of their lower strength. Together, such early plastic deformation in composite materials may contribute to improved strain accommodation and could be beneficial for energy absorption and damage tolerance by promoting local stress redistribution during deformation. Post-compression EBSD results suggested that continued deformation was accompanied by dislocation accumulation, progressive subgrain subdivision, and possible CDRX-related microstructural evolution, containing regions consistent with recovery/recrystallization alongside heavily deformed areas. The composite structure maintained a high electrical conductivity of  $52.6 \pm 1.2$  MS/m despite severe deformation, which supports the well-established concept that retaining a continuous pure Cu network is generally more favorable for preserving electrical conductivity than strengthening approaches based on solid-solution alloying, where solute atoms introduce additional electron scattering. Overall, this approach demonstrated that KoBo extrusion is not only capable of producing structurally stable Cu/Mg composites with reduced density and excellent electrical conductivity but also offers a promising route for tailoring other fundamental properties through microstructural and phase distribution control.

## 5. Future Outlook and Opportunities

During this PhD program, our research extended beyond its primary objectives, and we obtained valuable insights into how alloying elements and processing conditions influence the microstructure as well as the mechanical and physical properties of Cu-Mg alloys and other metallic systems. Based on our findings, the following areas are proposed as promising research directions for future investigations.

At present, high-entropy alloys (HEAs) have emerged as a major focus in physical metallurgy owing to their multi-principal-element design and their potential to achieve a combination of strength, ductility, and thermal stability [393]. In this context, Cu-based multi-

component alloys represent a promising direction, as introducing concentrated alloying elements (e.g., Zn, Sn, Al, Ni, and others) into Cu can (under suitable conditions) lead to compositionally complex or solid-solution microstructures in which short-range order (SRO) may be tuned depending on composition and processing conditions. Such controlled SRO in Cu-based systems may contribute to dislocation pinning and strengthening effects, which make these materials an interesting subject for future research.

Another highly promising research direction is the application of laser-based additive manufacturing to fabricate functionally graded Cu-based alloys in which the alloying content varies spatially, ranging from zero (or low concentrations) to optimize electrical conductivity to higher concentrations aimed at enhancing mechanical strength. By controlling the spatial distribution of alloying elements, this approach could enable a gradual transition of properties within a single component and potentially mitigate the traditional trade-off between strength and electrical conductivity. Here, laser-based additive manufacturing offers the precision and flexibility required to realize such compositional gradients in suitable Cu-based systems, as demonstrated in related multi-material and compositionally graded alloys [394], which make it a powerful tool for designing location-specific performance in advanced engineering applications.

Finally, but importantly, the application of emerging computational and data-driven approaches such as artificial intelligence, machine learning algorithms, materials informatics, and big data analytics to the design and optimization of Cu-Mg alloys aimed at improving the balance between mechanical properties and electrical conductivity represents a promising and increasingly viable research direction. By integrating experimental and computational datasets, these methods can help capture complex composition-microstructure-property relationships and support the identification of non-intuitive alloying strategies and processing routes within the limits of available data and underlying physical constraints [395]. In particular, such approaches show strong potential for supporting the quantitative prediction and optimization of precipitation behavior (e.g., Cu<sub>2</sub>Mg and Cu<sub>5</sub>Ca), including their size, morphology, and composition, especially when combined with established thermodynamic and kinetic modeling frameworks. Furthermore, data-driven methodologies can accelerate alloy and process development by guiding the selection of promising alloying contents and heat-treatment parameters that contribute to improved trade-offs among strength, ductility, electrical and thermal conductivity, manufacturability, and production cost, thereby substantially reducing (although not fully eliminating) the need for extensive experimental trial-and-error.

## 6. Summary and Conclusions

The effects of Ca addition, heat treatment, conventional deformation, and SPD processing using various methods on the microstructural evolution, mechanical properties, and electrical conductivity of Cu-Mg-based alloys were systematically investigated within the scope of this PhD program. A density-reduced Cu/Mg composite structure was also designed and fabricated. In summary, the research was conducted through three approaches: **Approach 1:** Ca alloying combined with heat treatment and MaxStrain processing of the Cu-0.7Mg (wt.%) alloy; **Approach 2:** application of different extrusion routes to the Cu-0.7Mg (wt.%) alloy followed by MaxStrain processing; and **Approach 3:** fabrication of a density-reduced Cu/Mg composite structure by KoBo extrusion. Together, the obtained results provided a comprehensive understanding of the relationships between alloy composition, processing history, microstructural features, mechanical properties, and electrical conductivity in HSHC Cu-Mg-based alloys. Based on the obtained results, the following conclusions can be drawn:

- (1) The addition of 0.1 wt.% Ca to the binary Cu-0.7Mg (wt.%) alloy altered the solidification behavior, and led to the transformation of the as-cast microstructure from predominantly columnar grains in the binary alloy to a finer and more equiaxed grain morphology in the ternary alloy. This refinement was attributed to the formation of Cu<sub>5</sub>Ca intermetallic particles in the ternary alloy, which effectively acted as preferable sites for grain nucleation during solidification.
- (2) Cu<sub>5</sub>Ca particles were uniformly distributed along the grain boundaries and also inside the grains of the ternary alloy. Together with the refined grain structure, they contributed to the enhanced mechanical strength, as evidenced by increased hardness, YS, and UTS of the ternary alloy compared to the binary alloy. However, these improvements were accompanied by reductions in ductility and electrical conductivity, which reflect the inherent trade-off between mechanical strengthening and electrical conductivity.
- (3) Heat treatment played a critical role in controlling the microstructural evolution and mechanical properties of the investigated alloys. Annealing at 650 °C for either 90 min or 75 min, followed by water quenching, promoted a more uniform distribution of Mg atoms, partial spheroidization of the Cu<sub>5</sub>Ca intermetallic particles, the generation of residual thermal stresses, and an increase in dislocation density. These combined microstructural changes led to improved mechanical properties while causing only a slight reduction in the electrical conductivity (only for binary alloy).

- (4) The obtained results demonstrated that the annealing duration and cooling conditions applied after annealing influenced the final microstructure and mechanical properties. Longer annealing durations of 90 min and 75 min promoted greater microstructural homogenization and a higher degree of partial spheroidization of the Cu<sub>5</sub>Ca intermetallic particles. Subsequent water quenching preserved these microstructural characteristics and resulted in simultaneous improvements in strength and ductility.
- (5) In general, SPD processing using the MaxStrain module of the Gleeble thermomechanical simulator proved to be an effective approach for disrupting the as-cast microstructure, promoting grain refinement, and enhancing the mechanical properties of both binary and ternary alloys. The observed strengthening was attributed to the combined effects of microstructural refinement, increased dislocation density, and the progressive transformation of LAGBs into HAGBs during CDRX, which together contributed to improved mechanical properties.
- (6) Simulations of the plastic deformation processes revealed that the accumulated strain distribution during extrusion and subsequent MaxStrain processing was highly heterogeneous, which resulted in spatial variations in hardness across the MaxStrain-processed samples. Hardness measurements demonstrated that regions experiencing higher equivalent strains exhibited increased hardness values, which highlights the strong correlation between local strain accumulation and hardness variation.
- (7) CDRX was identified as one of the key mechanisms contributing to microstructural refinement during SPD processing. The presence of Cu<sub>5</sub>Ca particles in the ternary alloys, combined with prior deformation processes, enhanced the fraction of recrystallized grains, promoted the development of HAGBs, and contributed to the formation of finer microstructures.
- (8) The processing history prior to MaxStrain deformation had a decisive influence on the subsequent strain accumulation behavior and resulting mechanical properties. Forward extrusion at 400 °C and KoBo extrusion at RT generated distinct initial strain states, which influenced the equivalent strain distributions and recrystallization responses developed during subsequent MaxStrain processing.
- (9) The combination of extrusion and subsequent MaxStrain deformation was demonstrated to be an effective processing route for achieving enhanced mechanical strength in Cu-Mg alloys. Samples subjected to prior forward extrusion or KoBo extrusion, followed by MaxStrain processing, exhibited superior mechanical properties compared with those

- processed only by MaxStrain, which shows the important role of cumulative deformation in improving the mechanical performance of HSHC Cu-Mg alloys.
- (10) Among the investigated processing routes, KoBo extrusion at RT, followed by 40 passes of MaxStrain processing at RT, resulted in the most pronounced strengthening effect. This improvement was attributed to the higher accumulated equivalent strain, enhanced grain refinement, increased fraction of HAGBs, and greater contribution of deformation-induced strengthening mechanisms.
  - (11) The reduction in electrical conductivity observed after SPD processing was primarily associated with increased electron scattering arising from the high dislocation density and the increased fraction of HAGBs developed during deformation. Nevertheless, this decrease in electrical conductivity associated with deformation-induced strengthening remained relatively low compared with the Mg-induced conductivity loss.
  - (12) Despite the significant strengthening achieved through SPD processing, all fabricated Cu-Mg alloys subjected to final MaxStrain processing retained electrical conductivity values above the minimum requirements for classification as HSHC Cu alloys. This demonstrated the effectiveness of the applied thermomechanical processing routes in achieving a favorable combination of high mechanical strength and good electrical conductivity.
  - (13) KoBo extrusion enabled the successful fabrication of a Cu/Mg composite structure consisting of a continuous pure Cu matrix containing embedded pure Mg filaments, while the Mg filaments preserved their initial symmetric arrangement along the extrusion direction. XCT analysis confirmed that the Mg filaments remained continuous without fracture or fragmentation after KoBo extrusion, which demonstrates the effectiveness of this processing route in preserving filament integrity during deformation.
  - (14) The fabricated Cu/Mg composite structure exhibited a lower density than pure Cu, and the continuous Cu matrix contributed to maintaining high electrical conductivity of the whole structure by providing continuous conductive pathways. Moreover, interface analysis indicated that the bonding between Cu and Mg was predominantly mechanical in nature, with no detectable interdiffusion layer observed after KoBo extrusion within the resolution limits of SEM-EDS.
  - (15) Warm KoBo extrusion induced significant microstructural refinement and evolution in both the Cu matrix and Mg filaments of the fabricated composite. The Cu matrix developed a fine equiaxed grain structure characterized by relatively low intragranular misorientation and reduced local lattice distortion, which indicated the occurrence of

- recovery-related processes and the formation of stable deformation substructures. Meanwhile, Mg filaments exhibited a UFG microstructure typical of SPD-processed Mg.
- (16) The fabricated Cu/Mg composite exhibited a heterogeneous mechanical response during compression, characterized by preferential early plastic deformation of the softer Mg filaments and progressive load transfer to the Cu matrix as deformation proceeded. This behavior highlighted the important roles of interfacial load transfer and phase-specific deformation mechanisms in governing the mechanical performance of the composite.
- (17) Electrical conductivity measurements of the Cu/Mg composite demonstrated that, although microstructural features introduced during SPD processing slightly reduced conductivity compared with pure Cu, the high electrical conductivity of the Cu matrix was largely retained. Considering the reduced density of the fabricated composite structure together with its high electrical conductivity, this design appeared promising for the development of density-reduced highly conductive components.

In conclusion, this work showed that advanced SPD-assisted processing strategies, particularly KoBo extrusion and MaxStrain processing, can provide useful approaches for developing high-performance Cu-Mg-based alloys and composites. By carefully controlling alloy composition, heat treatment conditions, and deformation routes, an improved balance between mechanical strength, ductility, and electrical conductivity was achieved. The obtained results provided valuable information about the relationships between processing history, microstructural evolution, and properties in Cu-Mg-based systems and highlighted the importance of proper alloy design and thermomechanical processing for mitigating the conventional strength-conductivity trade-off in Cu alloys. It can be concluded that the combination of alloying, heat treatment, and SPD processing offers a promising strategy for tailoring the performance of Cu-based alloys and can open opportunities for the development of weight-optimized, multifunctional alloys and composites for advanced electrical, electronic, and structural applications.

## References

- [1] J.-S. Liu, W.-X. Yu, D.-Y. Chen, S.-W. Wang, H.-W. Song, S.-H. Zhang, A Review of Studies on the Influence of Rare-Earth Elements on the Microstructures and Properties of Copper and Copper Alloys and Relevant Applications, *Metals* 15 (2025) 536. <https://doi.org/10.3390/met15050536>.
- [2] Q. Mao, Y. Liu, Y. Zhao, A review on copper alloys with high strength and high electrical conductivity, *J. Alloys Compd.* 990 (2024) 174456. <https://doi.org/10.1016/j.jallcom.2024.174456>.
- [3] A. Kalhor, K. Rodak, M. Tkocz, H. Myalska-Głowacka, I. Schindler, Ł. Poloczek, K. Radwański, H. Mirzadeh, M. Grzenik, K. Kubiczek, M. Kampik, Tailoring the microstructure, mechanical properties, and electrical conductivity of Cu–0.7Mg alloy via Ca addition, heat treatment, and severe plastic deformation, *Arch. Civ. Mech. Eng.* 24 (2024) 71. <https://doi.org/10.1007/s43452-024-00890-0>.
- [4] A. Kalhor, K. Rodak, M. Tkocz, B. Chmiela, I. Schindler, Ł. Poloczek, K. Radwański, H. Mirzadeh, M. Kampik, Influence of processing history on microstructure, mechanical properties, and electrical conductivity of Cu–0.7Mg alloy, *Trans. Nonferrous Met. Soc. China* 35 (2025) 1197–1211. [https://doi.org/10.1016/S1003-6326\(24\)66743-9](https://doi.org/10.1016/S1003-6326(24)66743-9).
- [5] A. Kalhor, K. Rodak, H. Myalska-Głowacka, B. Chmiela, K. Kuglarz, I. Schindler, K. Radwański, M. Kampik, High-strength and high-conductivity Cu-0.7Mg-0.1Ca alloy fabricated via heat treatment and severe plastic deformation, *J. Min. Metall. Sect. B Metall.* 61 (2025) 43–58. <https://doi.org/10.2298/JMMB241017004K>.
- [6] M. Xia, L. Lai, M. Fu, Y. Wu, S. Chen, G. Liu, Electrodeposition of antibacterial, anti-corrosion copper nanopillars arrays with heat conductive properties for marine environments, *J. Mater. Sci. Technol.* 232 (2025) 89–102. <https://doi.org/10.1016/j.jmst.2024.12.087>.
- [7] Y. Aghayar, P. Moazzen, L.A.I. Kestens, M. Mohammadi, Tailoring the microstructure, physical, and mechanical properties of pure copper using various additive manufacturing techniques, *J. Alloys Compd.* 1010 (2025) 178332. <https://doi.org/10.1016/j.jallcom.2024.178332>.
- [8] H. Xie, J. Jiang, H. Sun, Z. Li, J. Liu, J. Wang, Z. Mo, J. Shen, Tailoring cryogenic thermal conductivity in EuTiO<sub>3</sub>-based magnetic refrigeration materials, *J. Rare Earths* 43 (2025) 997–1002. <https://doi.org/10.1016/j.jre.2024.03.005>.
- [9] Y. Zhang, X. Zhang, L. Zuo, Microstructure and corrosion behaviour of metal copper parts formed by laser shock, *Corros. Sci.* 237 (2024) 112325. <https://doi.org/10.1016/j.corsci.2024.112325>.
- [10] S.-Y. Park, N.-Y. Yee, M. Baek, H.-S. Kim, K.-A. Lee, Microstructure heterogeneity, mechanical properties and thermal conductivity of pure copper fabricated by metal material extrusion additive manufacturing process, *Mater. Sci. Eng. A* 927 (2025) 147939. <https://doi.org/10.1016/j.msea.2025.147939>.
- [11] Y. Wei, G. Chen, Z. Xiao, Y. Zhang, Y. Zhou, X. Liu, W. Li, J. Xu, Microstructures and mechanical properties of pure copper manufactured by high-strength laser powder bed fusion, *Opt. Laser Technol.* 182 (2025) 112134. <https://doi.org/10.1016/j.optlastec.2024.112134>.
- [12] C. Li, H. Wei, M. Peng, Z. Li, Study on the high-strength and high-conductivity modification mechanisms of C7035 copper alloy induced by La and Sc doping, *Vacuum* 229 (2024) 113590. <https://doi.org/10.1016/j.vacuum.2024.113590>.
- [13] Y. Zhu, Y. Cao, L. Peng, Q. Yu, Z. Yang, Z. Li, J. Wu, H. Xie, Design of a novel Cu-Cr-X alloy with high strength and high electrical conductivity based on mechanical learning, *Mater. Des.* 250 (2025) 113599. <https://doi.org/10.1016/j.matdes.2025.113599>.
- [14] B.-Y. Chen, D.-P. Tran, K.-P. Lee, Y.-H. Chen, C. Chen, Temperature-dependent mechanical properties and fracture behaviors of copper foils with different microstructures, *Mater. Charact.* 224 (2025) 115051. <https://doi.org/10.1016/j.matchar.2025.115051>.
- [15] H. Yang, Z. Ma, C. Lei, L. Meng, Y. Fang, J. Liu, H. Wang, High strength and high conductivity Cu alloys: A review, *Sci. China Technol. Sci.* 63 (2020) 2505–2517. <https://doi.org/10.1007/s11431-020-1633-8>.
- [16] Y. Liu, J. Zhang, R. Niu, M. Bayat, Y. Zhou, Y. Yin, Q. Tan, S. Liu, J.H. Hattel, M. Li, X. Huang, J. Cairney, Y.-S. Chen, M. Easton, C. Hutchinson, M.-X. Zhang, Manufacturing of high strength and high conductivity copper with laser powder bed fusion, *Nat. Commun.* 15 (2024) 1283. <https://doi.org/10.1038/s41467-024-45732-y>.
- [17] Y. Wu, W. Zhang, Y. Li, F. Yang, H. Liu, J. Zou, J. Jiang, F. Fang, A. Ma, An overview of microstructure regulation treatment of Cu-Fe alloys to improve strength, conductivity, and electromagnetic shielding, *J. Alloys Compd.* 1002 (2024) 175425. <https://doi.org/10.1016/j.jallcom.2024.175425>.

- [18] B. Xu, Q. Wang, K. Wang, W. Wang, H. Zhang, B. Gao, J. Sun, F. Wang, Achieving ultra-high strength in copper-titanium alloys through multistage thermomechanical processing induced strengthening, *Mater. Sci. Eng. A* 955 (2026) 149865. <https://doi.org/10.1016/j.msea.2026.149865>.
- [19] S. Tang, M. Zhou, Y. Zhang, D. Xu, Z. Zhang, X. Zheng, D. Li, X. Li, B. Tian, Y. Jia, Y. Liu, A.A. Volinsky, E.S. Marchenko, Improved microstructure, mechanical properties and electrical conductivity of the Cu–Ni–Sn–Ti–Cr alloy due to Ce micro-addition, *Mater. Sci. Eng. A* 871 (2023) 144910. <https://doi.org/10.1016/j.msea.2023.144910>.
- [20] X. Li, R. Tang, H. Huang, X. Ma, W. Qiu, L. Zheng, G. Chao, Y. Wang, S. Gong, Microstructure and properties of high strength, high conductivity Cu-2.5Fe-0.3Cr-0.3Mg-0.2Si-0.1Ca-0.1Zr alloy treated by multi-stage thermomechanical treatment, *Mater. Charact.* 207 (2024) 113601. <https://doi.org/10.1016/j.matchar.2023.113601>.
- [21] X. Tang, X. Chen, F. Sun, P. Liu, H. Zhou, S. Fu, The current state of CuCrZr and CuCrNb alloys manufactured by additive manufacturing: A review, *Mater. Des.* 224 (2022) 111419. <https://doi.org/10.1016/j.matdes.2022.111419>.
- [22] K. Maki, Y. Ito, H. Matsunaga, H. Mori, Solid-solution copper alloys with high strength and high electrical conductivity, *Scr. Mater.* 68 (2013) 777–780. <https://doi.org/10.1016/j.scriptamat.2012.12.027>.
- [23] Y. Jiang, X. Zhang, P. Cai, P. Li, F. Cao, F. Gao, S. Liang, Precipitation behavior and microstructural evolution during thermo-mechanical processing of precipitation hardened Cu-Hf based alloys, *Acta Mater.* 245 (2023) 118659. <https://doi.org/10.1016/j.actamat.2022.118659>.
- [24] C. Wang, H. Fu, H. Zhang, X. He, J. Xie, Simultaneous enhancement of mechanical and electrical properties of Cu–Ni–Si alloys via thermo-mechanical process, *Mater. Sci. Eng. A* 838 (2022) 142815. <https://doi.org/10.1016/j.msea.2022.142815>.
- [25] K.X. Wei, Z.Q. Chu, W. Wei, Q.B. Du, I.V. Alexandrov, J. Hu, Effect of Deep Cryogenic Treatment on Microstructure and Properties of Pure Copper Processed by Equal Channel Angular Pressing, *Adv. Eng. Mater.* 21 (2019) 1801372. <https://doi.org/10.1002/adem.201801372>.
- [26] K. Yang, Y. Wang, M. Guo, H. Wang, Y. Mo, X. Dong, H. Lou, Recent development of advanced precipitation-strengthened Cu alloys with high strength and conductivity: A review, *Prog. Mater. Sci.* 138 (2023) 101141. <https://doi.org/10.1016/j.pmatsci.2023.101141>.
- [27] Z. Lai, K. Peng, B. Gao, Y. Mai, X. Jie, Achieving high strength and high electrical conductivity in Cu–Cr–Zr alloy by modifying shear banding during rolling, *J. Alloys Compd.* 958 (2023) 170402. <https://doi.org/10.1016/j.jallcom.2023.170402>.
- [28] M. Ma, Z. Li, W. Qiu, Z. Xiao, Z. Zhao, Y. Jiang, Z. Xia, H. Huang, Development of homogeneity in a Cu–Mg–Ca alloy processed by equal channel angular pressing, *J. Alloys Compd.* 820 (2020) 153112. <https://doi.org/10.1016/j.jallcom.2019.153112>.
- [29] J. Guo, H. Wan, H. Xu, H. Yan, Y. Shao, Z. Hu, C. Zhao, Q. Hu, Effects of different Cr contents on the microstructure, mechanical and electrical properties of Cu–Mg–Cr alloy, *J. Mater. Res. Technol.* 40 (2026) 3741–3757. <https://doi.org/10.1016/j.jmrt.2026.01.087>.
- [30] C. Zhu, A. Ma, J. Jiang, X. Li, D. Song, D. Yang, Y. Yuan, J. Chen, Effect of ECAP combined cold working on mechanical properties and electrical conductivity of Conform-produced Cu–Mg alloys, *J. Alloys Compd.* 582 (2014) 135–140. <https://doi.org/10.1016/j.jallcom.2013.08.007>.
- [31] S. Gorsse, B. Ouyard, M. Gouné, A. Poulon-Quintin, Microstructural design of new high conductivity – high strength Cu-based alloy, *J. Alloys Compd.* 633 (2015) 42–47. <https://doi.org/10.1016/j.jallcom.2015.01.234>.
- [32] A. Ma, C. Zhu, J. Chen, J. Jiang, D. Song, S. Ni, Q. He, Grain Refinement and High-Performance of Equal-Channel Angular Pressed Cu–Mg Alloy for Electrical Contact Wire, *Metals* 4 (2014) 586–596. <https://doi.org/10.3390/met4040586>.
- [33] P. Rodríguez-Calvillo, N. Ferrer, J.M. Cabrera, Analysis of microstructure and strengthening in CuMg alloys deformed by equal channel angular pressing, *J. Alloys Compd.* 626 (2015) 340–348. <https://doi.org/10.1016/j.jallcom.2014.12.043>.
- [34] M. Ma, X. Zhang, Z. Li, Z. Xiao, H. Jiang, Z. Xia, H. Huang, Effect of Equal Channel Angular Pressing on Microstructure and Mechanical Properties of a Cu–Mg Alloy, *Crystals* 10 (2020) 426. <https://doi.org/10.3390/cryst10060426>.
- [35] A. Bodyakova, M. Tkachev, A. Pilipenko, A. Belyakov, R. Kaibyshev, Effect of deformation methods on microstructure, texture, and properties of a Cu–Mg alloy, *Mater. Sci. Eng. A* 876 (2023) 145126. <https://doi.org/10.1016/j.msea.2023.145126>.

- [36] Y. Chen, J. Xie, W. Cao, Research on friction stir welded joints of Copper to Magnesium alloy, in: Proc. Int. Symp. Mech. Eng. Mater. Sci., Atlantis Press, Jeju Island, South Korea, 2016. <https://doi.org/10.2991/ismems-16.2016.53>.
- [37] Y. Li, Z. Xiao, Z. Li, Z. Zhou, Z. Yang, Q. Lei, Microstructure and properties of a novel Cu-Mg-Ca alloy with high strength and high electrical conductivity, *J. Alloys Compd.* 723 (2017) 1162–1170. <https://doi.org/10.1016/j.jallcom.2017.06.155>.
- [38] B. Wang, Y. Zhang, B. Tian, V. Yakubov, J. An, A.A. Volinsky, Y. Liu, K. Song, L. Li, M. Fu, Effects of Ce and Y addition on microstructure evolution and precipitation of Cu-Mg alloy hot deformation, *J. Alloys Compd.* 781 (2019) 118–130. <https://doi.org/10.1016/j.jallcom.2018.12.022>.
- [39] B. Wang, Y. Zhang, B. Tian, J. An, A.A. Volinsky, H. Sun, Y. Liu, K. Song, Effects of Ce addition on the Cu-Mg-Fe alloy hot deformation behavior, *Vacuum* 155 (2018) 594–603. <https://doi.org/10.1016/j.vacuum.2018.06.006>.
- [40] D. Risold, B. Hallstedt, L.J. Gauckler, H.L. Lukas, S.G. Fries, Thermodynamic optimization of the Ca-Cu and Sr-Cu systems, *Calphad* 20 (1996) 151–160. [https://doi.org/10.1016/S0364-5916\(96\)00022-3](https://doi.org/10.1016/S0364-5916(96)00022-3).
- [41] D.J. Chakrabarti, D.E. Laughlin, The Ca–Cu (Calcium-Copper) system, *Bull. Alloy Phase Diagr.* 5 (1984) 570–576. <https://doi.org/10.1007/BF02868318>.
- [42] M. Ma, Z. Li, W. Qiu, Z. Xiao, Z. Zhao, Y. Jiang, Microstructure and properties of Cu–Mg–Ca alloy processed by equal channel angular pressing, *J. Alloys Compd.* 788 (2019) 50–60. <https://doi.org/10.1016/j.jallcom.2019.01.335>.
- [43] M. Mezbahul-Islam, A.O. Mostafa, M. Medraj, Essential Magnesium Alloys Binary Phase Diagrams and Their Thermochemical Data, *J. Mater.* 2014 (2014) 1–33. <https://doi.org/10.1155/2014/704283>.
- [44] D.M. Stefanescu, A Succinct History of Metalcasting Knowledge, *Int. J. Met.* 17 (2023) 2373–2388. <https://doi.org/10.1007/s40962-023-00971-5>.
- [45] H. Sicius, Copper Group: Elements of the First Subgroup, in: *Handb. Chem. Elem.*, Springer Berlin Heidelberg, Berlin, Heidelberg, 2024: pp. 795–836. [https://doi.org/10.1007/978-3-662-68921-9\\_16](https://doi.org/10.1007/978-3-662-68921-9_16).
- [46] C.B. Tabein, I. Park, T. Phengsaart, S. Jeon, M. Villacorte-Tabein, D. Alonzo, K. Yoo, M. Ito, N. Hiroyoshi, Copper and critical metals production from porphyry ores and E-wastes: A review of resource availability, processing/recycling challenges, socio-environmental aspects, and sustainability issues, *Resour. Conserv. Recycl.* 170 (2021) 105610. <https://doi.org/10.1016/j.resconrec.2021.105610>.
- [47] K.J. Nyembwe, E. Fosso-Kankeu, F. Waanders, M. Mkandawire, B.B. Mamba, Formation of Phase Transition During the Dissolution of Silicate and Carbonate Chalcopyrite in Acidic Ferric Sulfate, *Trans. Indian Inst. Met.* 75 (2022) 1767–1779. <https://doi.org/10.1007/s12666-022-02546-0>.
- [48] A. Ciurej, M. Struska, A. Wolska, M. Szczerba, J. Olszak, Copper-Bearing Mineralisation in the Upper Devonian Limestones: A Case Study from the Historical Teresa Adit in the Świętokrzyskie Mountains, Poland, *Minerals* 13 (2022) 54. <https://doi.org/10.3390/min13010054>.
- [49] I. Dinaharan, T. Albert, Effect of reinforcement type on microstructural evolution and wear performance of copper matrix composites via powder metallurgy, *Mater. Today Commun.* 34 (2023) 105250. <https://doi.org/10.1016/j.mtcomm.2022.105250>.
- [50] K. Franczak, M. Sadzikowski, P. Kwaśniewski, G. Kiesiewicz, W. Ścieżor, S. Kordaszewski, Research on Alloying Elements' Influence on CuETP-Grade Copper's Mechanical and Electrical Properties, *Materials* 17 (2024) 3020. <https://doi.org/10.3390/ma17123020>.
- [51] Z. Gao, T. Wang, Y. Zhao, H. Ding, Q. Huang, Mechanical Properties Optimization and Microstructure Analysis of Pure Copper Heterostructured Laminates via Rolling, *Chin. J. Mech. Eng.* 37 (2024) 126. <https://doi.org/10.1186/s10033-024-01102-9>.
- [52] K.J.A. Kundig, J.G. Cowie, Copper and Copper Alloys, in: M. Kutz (Ed.), *Mech. Eng. Handb.*, 1st ed., Wiley, 2005: pp. 117–220. <https://doi.org/10.1002/0471777447.ch4>.
- [53] J.W. Arblaster, Thermodynamic Properties of Copper, *J. Phase Equilibria Diffus.* 36 (2015) 422–444. <https://doi.org/10.1007/s11669-015-0399-x>.
- [54] S. Mehvari, Y. Sanchez-Vicente, S. González, K. Lafdi, Conductivity Behaviour under Pressure of Copper Micro-Additive/Polyurethane Composites (Experiment and Modelling), *Polymers* 14 (2022) 1287. <https://doi.org/10.3390/polym14071287>.
- [55] S.D. Jadhav, L.R. Goossens, Y. Kinds, B.V. Hooreweder, K. Vanmeensel, Laser-based powder bed fusion additive manufacturing of pure copper, *Addit. Manuf.* 42 (2021) 101990. <https://doi.org/10.1016/j.addma.2021.101990>.
- [56] Y. Chen, C. Zeng, H. Ding, S. Emanet, P.R. Gradl, D.L. Ellis, S. Guo, Thermophysical properties of additively manufactured (AM) GRCop-42 and GRCop-84, *Mater. Today Commun.* 36 (2023) 106665. <https://doi.org/10.1016/j.mtcomm.2023.106665>.

- [57] Y. Zhang, D. Choudhuri, T.W. Scharf, S. Descartes, R.R. Chromik, Tribologically induced nanolaminate in a cold-sprayed WC-reinforced Cu matrix composite: a key to high wear resistance, *Mater. Des.* 182 (2019) 108009. <https://doi.org/10.1016/j.matdes.2019.108009>.
- [58] S. Qu, Z. You, R. Gu, Y. Jiang, J.T. Wang, Fracture toughness anisotropy of ultrafine-grained pure copper processed by equal channel angular pressing, *Mater. Sci. Eng. A* 782 (2020) 139260. <https://doi.org/10.1016/j.msea.2020.139260>.
- [59] J. Escobar, J. Silverstein, F. Ishrak, L. Li, A. Soulam, S. Li, A. Yu, S. Mathaudhu, A. Ortiz, C. Koch, A. Devaraj, M. Efe, B. Gwalani, Microstructural evolution in shear-punch tests: A comparative study of pure Cu and Cu-Cr alloy, *Mater. Sci. Eng. A* 886 (2023) 145715. <https://doi.org/10.1016/j.msea.2023.145715>.
- [60] M. Afifeh, S.J. Hosseinipour, R. Jamaati, Effect of post-annealing on the microstructure and mechanical properties of nanostructured copper, *Mater. Sci. Eng. A* 802 (2021) 140666. <https://doi.org/10.1016/j.msea.2020.140666>.
- [61] S.M. Uddin, T. Mahmud, C. Wolf, C. Glanz, I. Kolaric, C. Volkmer, H. Höller, U. Wienecke, S. Roth, H.-J. Fecht, Effect of size and shape of metal particles to improve hardness and electrical properties of carbon nanotube reinforced copper and copper alloy composites, *Compos. Sci. Technol.* 70 (2010) 2253–2257. <https://doi.org/10.1016/j.compscitech.2010.07.012>.
- [62] L. Gao, Z. Shi, D. Li, Y. Yang, G. Zhang, A. McLean, K. Chattopadhyay, Dimensionless Analysis and Mathematical Modeling of Electromagnetic Levitation (EML) of Metals, *Metall. Mater. Trans. B* 47 (2016) 67–75. <https://doi.org/10.1007/s11663-015-0457-0>.
- [63] A. Klassert, L. Tikana, Copper and copper–nickel alloys – an overview, in: *Corros. Behav. Prot. Copp. Alum. Alloys Seawater*, Elsevier, 2007: pp. 47–61. <https://doi.org/10.1533/9781845693084.2.47>.
- [64] X.T. Fang, G.Z. He, C. Zheng, X.L. Ma, D. Kaoumi, Y.S. Li, Y.T. Zhu, Effect of heterostructure and hetero-deformation induced hardening on the strength and ductility of brass, *Acta Mater.* 186 (2020) 644–655. <https://doi.org/10.1016/j.actamat.2020.01.037>.
- [65] S. Scudino, C. Unterdörfer, K.G. Prashanth, H. Attar, N. Ellendt, V. Uhlenwinkel, J. Eckert, Additive manufacturing of Cu–10Sn bronze, *Mater. Lett.* 156 (2015) 202–204. <https://doi.org/10.1016/j.matlet.2015.05.076>.
- [66] G.A. Pantazopoulos, A.I. Toulfatzis, Fracture Modes and Mechanical Characteristics of Machinable Brass Rods, *Metallogr. Microstruct. Anal.* 1 (2012) 106–114. <https://doi.org/10.1007/s13632-012-0019-7>.
- [67] Y. Yen, P. Tsai, Y. Fang, B. Chen, C. Lee, Interfacial reactions of Pb-free solders with Au/Pd/Ni/brass multilayer substrates, *J. Alloys Compd.* 517 (2012) 111–117. <https://doi.org/10.1016/j.jallcom.2011.12.046>.
- [68] Z. Liang, K. Jiang, T. Zhang, S. Lin, Corrosion behavior of brass from the Western Zhou Dynasty in an archeological-corrosive medium, *J. Alloys Compd.* 865 (2021) 158579. <https://doi.org/10.1016/j.jallcom.2020.158579>.
- [69] S. Berndorf, A. Markelov, S. Guk, M. Mandel, L. Krüger, U. Prah, Development of a Dezincification-Free Alloy System for the Manufacturing of Brass Instruments, *Metals* 14 (2024) 800. <https://doi.org/10.3390/met14070800>.
- [70] S. Jasper, R. Subash, K. Muthuneelakandan, D. Vijayakumar, S. Jhansi Ida, The Mechanical Properties of Brass Alloys: A Review, in: *ICMechD 2024*, MDPI, 2025: p. 11. <https://doi.org/10.3390/engproc2025093011>.
- [71] P. De Barros Damgaard, R. Martiniano, J. Kamm, J.V. Moreno-Mayar, G. Kroonen, M. Peyrot, G. Barjamovic, S. Rasmussen, C. Zacho, N. Baimukhanov, V. Zaibert, V. Merz, A. Biddanda, I. Merz, V. Loman, V. Evdokimov, E. Usmanova, B. Hemphill, A. Seguin-Orlando, F.E. Yediyar, I. Ullah, K.-G. Sjögren, K.H. Iversen, J. Choin, C. De La Fuente, M. Ilardo, H. Schroeder, V. Moiseyev, A. Gromov, A. Polyakov, S. Omura, S.Y. Senyurt, H. Ahmad, C. McKenzie, A. Margaryan, A. Hameed, A. Samad, N. Gul, M.H. Khokhar, O.I. Goriunova, V.I. Bazaliiskii, J. Novembre, A.W. Weber, L. Orlando, M.E. Allentoft, R. Nielsen, K. Kristiansen, M. Sikora, A.K. Outram, R. Durbin, E. Willerslev, The first horse herders and the impact of early Bronze Age steppe expansions into Asia, *Science* 360 (2018) eaar7711. <https://doi.org/10.1126/science.aar7711>.
- [72] T. Chang, G. Herting, S. Goidanich, J.M. Sánchez Amaya, M.A. Arenas, N. Le Bozec, Y. Jin, C. Leygraf, I. Odnevall Wallinder, The role of Sn on the long-term atmospheric corrosion of binary Cu-Sn bronze alloys in architecture, *Corros. Sci.* 149 (2019) 54–67. <https://doi.org/10.1016/j.corsci.2019.01.002>.
- [73] M. Karthik, J. Abhinav, K.V. Shankar, Morphological and Mechanical Behaviour of Cu–Sn Alloys—A review, *Met. Mater. Int.* 27 (2021) 1915–1946. <https://doi.org/10.1007/s12540-020-00899-z>.

- [74] Su. Venkatesan, J. Jerald, P. Asokan, R. Harichandran, A study on the mechanical and corrosion behaviour of bronze – TiB<sub>2</sub> metal matrix composites, *Mater. Today Proc.* 39 (2021) 1426–1433. <https://doi.org/10.1016/j.matpr.2020.05.116>.
- [75] K. Manu, J. Jezierski, M.R.S. Ganesh, K.V. Shankar, S.A. Narayanan, Titanium in Cast Cu-Sn Alloys—A Review, *Materials* 14 (2021) 4587. <https://doi.org/10.3390/ma14164587>.
- [76] H. Li, Z. Zhang, H. Guo, C. Ren, C. Liu, L. Xiang, Recent Progress on Corrosion Behavior, Mechanism, and Protection Strategies of Bronze Artefacts, *Heritage* 8 (2025) 340. <https://doi.org/10.3390/heritage8080340>.
- [77] Y. Zhao, L. Peng, H. Xie, W. Zhang, S. Huang, Z. Yang, Z. Li, X. Mi, Enhancing the erosion–corrosion resistance of cupronickel alloy through grain boundary engineering, *Corros. Sci.* 219 (2023) 111228. <https://doi.org/10.1016/j.corsci.2023.111228>.
- [78] X. Liu, H. Tong, H. Zhang, J. Zhou, X. Li, J. Hou, Corrosion behavior of 70/30 cupronickel in electrolytic seawater antifouling environment, *Mater. Today Commun.* 41 (2024) 110471. <https://doi.org/10.1016/j.mtcomm.2024.110471>.
- [79] A. Ezanno, C. Doudard, S. Calloch, T. Millot, J.-L. Heuzé, Fast characterization of high-cycle fatigue properties of a cast copper–aluminum alloy by self-heating measurements under cyclic loadings, *Procedia Eng.* 2 (2010) 967–976. <https://doi.org/10.1016/j.proeng.2010.03.105>.
- [80] B. Wu, L. Fang, X. Chen, Z. Zou, X. Yu, G. Chen, Fabricating Aluminum Bronze Rotating Band for Large-Caliber Projectiles by High Velocity Arc Spraying, *J. Therm. Spray Technol.* 23 (2014) 447–455. <https://doi.org/10.1007/s11666-013-0010-0>.
- [81] X. Huang, G. Xie, X. Liu, H. Fu, L. Shao, Z. Hao, The influence of precipitation transformation on Young's modulus and strengthening mechanism of a Cu–Be binary alloy, *Mater. Sci. Eng. A* 772 (2020) 138592. <https://doi.org/10.1016/j.msea.2019.138592>.
- [82] X. Bu, Q. Lei, X. Zhang, C. Chen, L. Tian, Y. Luo, F. Liu, Effect of heat treatment on the microstructure and properties of a Cu–Ni–Si alloy manufactured by laser powder bed fusion, *Mater. Sci. Eng. A* 953 (2026) 149775. <https://doi.org/10.1016/j.msea.2026.149775>.
- [83] Z. Liu, H. Wu, W. Liang, X. Wang, Y. Xu, D. Chen, P. Yao, Experimental investigation and machine learning prediction of stress relaxation resistance in C17200 alloy under aging treatment, *J. Alloys Compd.* 1057 (2026) 186702. <https://doi.org/10.1016/j.jallcom.2026.186702>.
- [84] C. Chiavari, A. Colledan, A. Frignani, G. Brunoro, Corrosion evaluation of traditional and new bronzes for artistic castings, *Mater. Chem. Phys.* 95 (2006) 252–259. <https://doi.org/10.1016/j.matchemphys.2005.06.034>.
- [85] G. Masi, C. Josse, J. Esvan, C. Chiavari, E. Bernardi, C. Martini, M.C. Bignozzi, C. Monticelli, F. Zanotto, A. Balbo, E. Svara Fabjan, T. Kosec, L. Robbiola, Evaluation of the protectiveness of an organosilane coating on patinated Cu–Si–Mn bronze for contemporary art, *Prog. Org. Coat.* 127 (2019) 286–299. <https://doi.org/10.1016/j.porgcoat.2018.11.027>.
- [86] A. Vahedi Nemani, M. Ghaffari, K. Sabet Bokati, N. Valizade, E. Afshari, A. Nasiri, Advancements in Additive Manufacturing for Copper-Based Alloys and Composites: A Comprehensive Review, *J. Manuf. Mater. Process.* 8 (2024) 54. <https://doi.org/10.3390/jmmp8020054>.
- [87] W. Zhang, L. Huang, X. Mi, H. Xie, X. Feng, J.H. Ahn, Researches for higher electrical conductivity copper-based materials, *cMat* 1 (2024) e13. <https://doi.org/10.1002/cmt2.13>.
- [88] X. Zhu, Z. Xiao, J. An, H. Jiang, Y. Jiang, Z. Li, Microstructure and properties of Cu–Ag alloy prepared by continuously directional solidification, *J. Alloys Compd.* 883 (2021) 160769. <https://doi.org/10.1016/j.jallcom.2021.160769>.
- [89] T. Zhong, L. Huang, Q. Lei, S. Zhou, T. Mao, H. Hu, X. Mo, H. Shao, H. Wang, X. Meng, Effect of Cr Addition and Different Processes on the Microstructure and Properties of Cu–Ag Alloys, *Adv. Eng. Mater.* 27 (2025) 2501215. <https://doi.org/10.1002/adem.202501215>.
- [90] Y. Niu, Z. Zhao, R. Zhang, X. Hu, K. Zhou, X. Kong, Y. Wang, Y. Fu, Effect of rapid annealing on microstructure and properties of Cu–4 wt.% Ag alloy wire, *J. Mater. Res. Technol.* 41 (2026) 4231–4241. <https://doi.org/10.1016/j.jmrt.2026.01.238>.
- [91] C. Yang, B. Wen, H. Shi, X. Wang, Z. Wang, Y. Luo, K. Xiao, J. Wang, X. Fan, N. Song, Y. Tan, Y. Tang, Electrodeposited HEDP-based ultrafine-grained Cu Ag alloy foils with ultra-high strength and high conductivity, *Mater. Charact.* 233 (2026) 116093. <https://doi.org/10.1016/j.matchar.2026.116093>.
- [92] S. Xu, H. Fu, Y. Wang, J. Xie, Effect of Ag addition on the microstructure and mechanical properties of Cu–Cr alloy, *Mater. Sci. Eng. A* 726 (2018) 208–214. <https://doi.org/10.1016/j.msea.2018.04.077>.

- [93] Y. Sun, L. Peng, G. Huang, H. Xie, X. Mi, X. Liu, Effects of Mg addition on the microstructure and softening resistance of Cu–Cr alloys, *Mater. Sci. Eng. A* 776 (2020) 139009. <https://doi.org/10.1016/j.msea.2020.139009>.
- [94] Z. Liu, Y. Ma, Q. Lei, T. Wei, X. Mo, C. Bai, T. Mao, Effect of microelements on the microstructure evolution and properties of a Cu–3Cr alloy with high–strength and high–conductivity, *Mater. Today Commun.* 49 (2025) 114352. <https://doi.org/10.1016/j.mtcomm.2025.114352>.
- [95] S. Liu, J. Jie, Z. Guo, S. Yue, T. Li, A comprehensive investigation on microstructure and magnetic properties of immiscible Cu–Fe alloys with variation of Fe content, *Mater. Chem. Phys.* 238 (2019) 121909. <https://doi.org/10.1016/j.matchemphys.2019.121909>.
- [96] S. Huang, B. Lin, W. Niu, G. Wang, J. Li, Study on the microstructure evolution and mechanical properties of spray-forming Cu–Fe alloys with varying Fe compositions, *J. Alloys Compd.* 1013 (2025) 178452. <https://doi.org/10.1016/j.jallcom.2025.178452>.
- [97] Q. Yu, X. Niu, W. Qiu, P. Jian, Y. Liu, S. Sun, S. Gong, Z. Li, Effect of thermo-mechanical treatment on the microstructure and properties of Cu–6.5Fe–0.3Mg–0.1Si alloy wire, *J. Alloys Compd.* 1053 (2026) 186334. <https://doi.org/10.1016/j.jallcom.2026.186334>.
- [98] N. Karakaya, E. Aldirmaz, Research on change of phase transformation temperatures and electrical resistance triggered by heat treatment of alloy from Cu–Mn system, *Appl. Phys. A* 122 (2016) 542. <https://doi.org/10.1007/s00339-016-0079-7>.
- [99] C. Zhang, X. Xiao, R. Zhou, W. Gao, Q. Li, J. He, Effect of Mn/P atomic ratio on the microstructure and properties of Cu–Mn–P alloy, *J. Alloys Compd.* 1010 (2025) 177705. <https://doi.org/10.1016/j.jallcom.2024.177705>.
- [100] Q. Fu, B. Li, M. Gao, Y. Fu, R. Yu, C. Wang, R. Guan, Quantitative mechanisms behind the high strength and electrical conductivity of Cu–Te alloy manufactured by continuous extrusion, *J. Mater. Sci. Technol.* 121 (2022) 9–18. <https://doi.org/10.1016/j.jmst.2021.12.046>.
- [101] Q. Fu, B. Li, H. Fu, M. Gao, C. Wang, Y. Fu, R. Guan, Dislocation behavior and strengthening mechanisms of Cu–Te alloy drawn at room and cryogenic temperatures, *J. Mater. Res. Technol.* 26 (2023) 6419–6432. <https://doi.org/10.1016/j.jmrt.2023.08.253>.
- [102] H. Zhi, H. Zhao, S. Wang, F. Kong, H. Song, Tailoring Multiscale Microstructures in Cu–xTe–Cr Alloys via Aging: Correlating Te Content With Mechanical and Electrical Properties, *Adv. Eng. Mater.* (2026) e202502603. <https://doi.org/10.1002/adem.202502603>.
- [103] W.A. Soffa, D.E. Laughlin, High-strength age hardening copper–titanium alloys: redivivus, *Prog. Mater. Sci.* 49 (2004) 347–366. [https://doi.org/10.1016/S0079-6425\(03\)00029-X](https://doi.org/10.1016/S0079-6425(03)00029-X).
- [104] L. Huang, Z. Cui, X. Meng, X. Zhang, X. Zhang, X. Song, N. Tang, Z. Xiao, Q. Lei, Z. Li, Effects of microelements on the microstructure evolution and properties of ultrahigh strength Cu–Ti alloys, *Mater. Sci. Eng. A* 823 (2021) 141581. <https://doi.org/10.1016/j.msea.2021.141581>.
- [105] Y. Zhu, L. Peng, M. Guo, G. Huang, K. Wang, J. Wu, D. Liu, H. Xie, Overcoming the trade-off among strength, ductility, and electrical conductivity in an ultra-high strength Cu–3Ti–0.2Fe alloy through regulating precipitates and nanostructures, *J. Mater. Sci. Technol.* 260 (2026) 253–267. <https://doi.org/10.1016/j.jmst.2025.10.010>.
- [106] G. Pantazopoulos, A review of defects and failures in brass rods and related components, *Pract. Fail. Anal.* 3 (2003) 14–22. <https://doi.org/10.1007/BF02715925>.
- [107] M.S. Parvizi, A. Aladjem, J.E. Castle, Behaviour of 90–10 cupronickel in sea water, *Int. Mater. Rev.* 33 (1988) 169–200. <https://doi.org/10.1179/imr.1988.33.1.169>.
- [108] Y. Li, T.L. Ngai, W. Xia, Mechanical, friction and wear behaviors of a novel high-strength wear-resisting aluminum bronze, *Wear* 197 (1996) 130–136. [https://doi.org/10.1016/0043-1648\(95\)06890-2](https://doi.org/10.1016/0043-1648(95)06890-2).
- [109] X. Guoliang, W. Qiangsong, M. Xujun, X. Baiqing, P. Lijun, The precipitation behavior and strengthening of a Cu–2.0wt% Be alloy, *Mater. Sci. Eng. A* 558 (2012) 326–330. <https://doi.org/10.1016/j.msea.2012.08.007>.
- [110] K. Bhole, V. Sonavane, Parameter Based Method for Three Arm Spiral Shaped Flexural Bearing, *Mater. Today Proc.* 5 (2018) 19380–19390. <https://doi.org/10.1016/j.matpr.2018.06.298>.
- [111] C. Ensslen, O. Kraft, R. Monig, J. Xu, G.-P. Zhang, R. Schneider, Mechanical annealing of Cu–Si nanowires during high-cycle fatigue, *MRS Commun.* 4 (2014) 83–87. <https://doi.org/10.1557/mrc.2014.18>.
- [112] G. Bao, Y. Xu, L. Huang, X. Lu, L. Zhang, Y. Fang, L. Meng, J. Liu, Strengthening Effect of Ag Precipitates in Cu–Ag Alloys: A Quantitative Approach, *Mater. Res. Lett.* 4 (2016) 37–42. <https://doi.org/10.1080/21663831.2015.1091795>.

- [113] L. Mosesso, S. Macis, A. D'Arco, A. Marcelli, A. Notargiacomo, M. Pea, B. Spataro, V. Stagno, S. Lupi, Characterization of CuAg Alloys with Low Ag Concentrations, *Materials* 17 (2024) 1823. <https://doi.org/10.3390/ma17081823>.
- [114] K.X. Wei, W. Wei, F. Wang, Q.B. Du, I.V. Alexandrov, J. Hu, Microstructure, mechanical properties and electrical conductivity of industrial Cu–0.5%Cr alloy processed by severe plastic deformation, *Mater. Sci. Eng. A* 528 (2011) 1478–1484. <https://doi.org/10.1016/j.msea.2010.10.059>.
- [115] M. Liu, J.X. Yang, Y.D. Wang, X.G. Dong, P. Xue, L.H. Wu, F.C. Liu, D.R. Ni, B.L. Xiao, Z.Y. Ma, Fabrication of ultrafine-grained Cu–Mn alloy with uniform microstructure and high mechanical properties, *Mater. Charact.* 205 (2023) 113270. <https://doi.org/10.1016/j.matchar.2023.113270>.
- [116] Z. Shen, Z. Lin, P. Shi, G. Tang, T. Zheng, C. Liu, Y. Guo, Y. Zhong, Enhanced strength, ductility and electrical conductivity of Cu–Te alloys via dynamic recrystallization and precipitation, *Mater. Sci. Eng. A* 820 (2021) 141548. <https://doi.org/10.1016/j.msea.2021.141548>.
- [117] X. Wang, Y. Ding, X. Jiang, X. Meng, Z. Xiao, Effects of Fe content on properties and microstructure of Cu–Ti alloys during aging, *J. Mater. Res. Technol.* 27 (2023) 5518–5532. <https://doi.org/10.1016/j.jmrt.2023.10.211>.
- [118] Y. Tong, S. Li, D. Zhang, L. Li, Y. Zheng, High strength and high electrical conductivity CuMg alloy prepared by cryorolling, *Trans. Nonferrous Met. Soc. China* 29 (2019) 595–600. [https://doi.org/10.1016/S1003-6326\(19\)64968-X](https://doi.org/10.1016/S1003-6326(19)64968-X).
- [119] T. Ozdal, B. Nalcaci, O. Akyuz, H. Goker, A.A. Kaya, Cu–Mg dilute alloys- Assessment of strengthening, internal friction, and electrical conductivity, *Mater. Chem. Phys.* 319 (2024) 129327. <https://doi.org/10.1016/j.matchemphys.2024.129327>.
- [120] P. Strzpek, M. Zasadzińska, A. Mamala, M. Walkowicz, P. Osuch, The Effect of the Percentage of Mg on the Properties of Cu–Mg Alloys Manufactured by Horizontal Continuous Casting Process, *JOM* 76 (2024) 3153–3160. <https://doi.org/10.1007/s11837-024-06517-2>.
- [121] R. Chaim, J. Pelleg, M. Talianker, TEM observation of a Cu–Mg age-hardenable alloy, *J. Mater. Sci.* 22 (1987) 1609–1612. <https://doi.org/10.1007/BF01132380>.
- [122] K.I. Nishikawa, S. Semboshi, T.J. Konno, Transmission Electron Microscopy Observations on Cu–Mg Alloy Systems, *Solid State Phenom.* 127 (2007) 103–108. <https://doi.org/10.4028/www.scientific.net/SSP.127.103>.
- [123] X. Guo, Y. Zhou, K. Song, X. Wang, S. Liang, Microstructure and properties of Cu–Mg alloy treated by internal oxidation, *Mater. Sci. Technol.* 34 (2018) 648–653. <https://doi.org/10.1080/02670836.2017.1410354>.
- [124] P. Strzpek, M. Zasadzińska, P. Noga, T. Skrzekut, Evolution of Properties of High-Strength and High-Mg-Content CuMg Alloys After Being Subjected to Single Operation 50% Deformation in Hot and Cold Upsetting Tests, *Materials* 17 (2024) 5467. <https://doi.org/10.3390/ma17225467>.
- [125] G. Li, J. Zhang, Z. Huang, Y. Wang, J. Xu, S. Wang, S. Yin, C. Zhang, Simultaneous enhancement of strength and conductivity in LPBF-fabricated CuCrZr alloys via trace LaB<sub>6</sub> additions, *J. Alloys Compd.* 1045 (2025) 184702. <https://doi.org/10.1016/j.jallcom.2025.184702>.
- [126] Q. Yan, Y. Li, Y. Wei, Z. Shi, S. Chen, J. Zhu, W. Jia, S. Liu, Superior strength-ductility-conductivity synergy of GRCop-42 alloy manufactured via electron beam powder bed fusion, *J. Mater. Res. Technol.* 40 (2026) 1202–1212. <https://doi.org/10.1016/j.jmrt.2025.12.068>.
- [127] Y. Luo, Z. Zhang, W. Guan, P. Wang, S. Zhang, L. Zeng, Z. Xue, H. Zhou, J. Liu, Substrate-regulated solidification enables high strength-conductivity synergy in selective laser melted CuCrZr alloy, *J. Manuf. Process.* 157 (2026) 1290–1307. <https://doi.org/10.1016/j.jmapro.2025.12.046>.
- [128] S. You, B. Lin, J. Xue, G. Hou, A. Li, X. Wang, D. Jiang, A multi-scale understanding of interlayer sintering mechanism of pure copper fabricated by extrusion-based additive manufacturing, *Powder Technol.* 469 (2026) 121899. <https://doi.org/10.1016/j.powtec.2025.121899>.
- [129] T. Fiedler, E. Zadow, J. Caspar, Laser powder-bed fusion of CuCr1Zr: parameter study, properties and microstructure, *Prog. Addit. Manuf.* 11 (2026) 2765–2780. <https://doi.org/10.1007/s40964-025-01494-7>.
- [130] P. Strzpek, M. Zasadzińska, Pure Alloy Additive or Preliminary Alloy: A Comparative Study on Obtaining High-Strength Copper Magnesium Alloys Designed for Electrical Power Systems, *Energies* 15 (2022) 2093. <https://doi.org/10.3390/en15062093>.
- [131] A.A. Nayeb-Hashemi, J.B. Clark, The Cu–Mg (Copper–Magnesium) system, *Bull. Alloy Phase Diagr.* 5 (1984) 36–43. <https://doi.org/10.1007/BF02868722>.
- [132] R. Torres, A. Cruz, G. Plascencia, High-temperature oxidation resistance of Cu–0.65 wt-% Mg alloy, *Can. Metall. Q.* 63 (2024) 483–487. <https://doi.org/10.1080/00084433.2023.2205768>.

- [133] Q. Tan, A. Atrens, N. Mo, M.-X. Zhang, Oxidation of magnesium alloys at elevated temperatures in air: A review, *Corros. Sci.* 112 (2016) 734–759. <https://doi.org/10.1016/j.corsci.2016.06.018>.
- [134] S. Laube, A. Kauffmann, F. Ruebeling, J. Freudenberger, M. Heilmaier, C. Greiner, Solid solution strengthening and deformation behavior of single-phase Cu-base alloys under tribological load, *Acta Mater.* 185 (2020) 300–308. <https://doi.org/10.1016/j.actamat.2019.12.005>.
- [135] B. Sankar, C. Vinay, J. Vishnu, K.V. Shankar, G.P. Gokul Krishna, V. Govind, A.J. Jayakrishna, Focused Review on Cu–Ni–Sn Spinodal Alloys: From Casting to Additive Manufacturing, *Met. Mater. Int.* 29 (2023) 1203–1228. <https://doi.org/10.1007/s12540-022-01305-6>.
- [136] S.B. Kadambi, V.D. Divya, U. Ramamurty, Evaluation of Solid-Solution Hardening in Several Binary Alloy Systems Using Diffusion Couples Combined with Nanoindentation, *Metall. Mater. Trans. A* 48 (2017) 4574–4582. <https://doi.org/10.1007/s11661-017-4250-3>.
- [137] W.D. Callister, D.G. Rethwisch, *Materials science and engineering: an introduction*, 8th ed, John Wiley & Sons, Hoboken, NJ, 2010.
- [138] M. Joele, W.R. Matizamhuka, A Review on the High Temperature Strengthening Mechanisms of High Entropy Superalloys (HESA), *Materials* 14 (2021) 5835. <https://doi.org/10.3390/ma14195835>.
- [139] E.W. Collings, H.L. Gegel, Physical Principles of Solid Solution Strengthening in Alloys, in: E.W. Collings, H.L. Gegel (Eds.), *Phys. Solid Solut. Strength.*, Springer New York, Boston, MA, 1975: pp. 147–182. [https://doi.org/10.1007/978-1-4684-0757-0\\_6](https://doi.org/10.1007/978-1-4684-0757-0_6).
- [140] C. Varvenne, G.P.M. Leyson, M. Ghazisaeidi, W.A. Curtin, Solute strengthening in random alloys, *Acta Mater.* 124 (2017) 660–683. <https://doi.org/10.1016/j.actamat.2016.09.046>.
- [141] R. Henle, S. Kött, N. Jost, G. Nandi, J. Dölling, A. Zilly, U. Prah, Investigation of the Solid Solution Hardening Mechanism of Low-Alloyed Copper–Scandium Alloys, *Metals* 14 (2024) 831. <https://doi.org/10.3390/met14070831>.
- [142] H. Mehdikhani, A. Mostafapour, B. Binesh, Mechanical properties and microstructure of the C70600 copper-nickel alloy and C46500 brass joint using brazing technique, *J. Adv. Join. Process.* 11 (2025) 100294. <https://doi.org/10.1016/j.jajp.2025.100294>.
- [143] X. Gao, H. Wu, M. Liu, Y. Zhang, F. Gao, H. Sun, Processing Map of C71500 Copper-nickel Alloy and Application in Production Practice, *J. Wuhan Univ. Technol.-Mater Sci Ed* 35 (2020) 1104–1115. <https://doi.org/10.1007/s11595-020-2361-y>.
- [144] Y. Zhang, T.T. Zuo, Z. Tang, M.C. Gao, K.A. Dahmen, P.K. Liaw, Z.P. Lu, Microstructures and properties of high-entropy alloys, *Prog. Mater. Sci.* 61 (2014) 1–93. <https://doi.org/10.1016/j.pmatsci.2013.10.001>.
- [145] K. Liu, W. Ding, X. Tao, H. Chen, Y. Ouyang, Y. Du, Diffusional behaviors and mechanical properties of Cu–Zn system, *J. Alloys Compd.* 812 (2020) 152141. <https://doi.org/10.1016/j.jallcom.2019.152141>.
- [146] E. Huerta, A.I. Oliva, F. Avilés, J. González-Hernández, J.E. Corona, Elastic Modulus Determination of Al–Cu Film Alloys Prepared by Thermal Diffusion, *J. Nanomater.* 2012 (2012) 895131. <https://doi.org/10.1155/2012/895131>.
- [147] L. Zhang, Y. Li, R. Zhou, X. Wang, Q. Wang, L. Xie, Z. Li, B. Xu, First-Principles Study of the Effect of Sn Content on the Structural, Elastic, and Electronic Properties of Cu–Sn Alloys, *Crystals* 13 (2023) 1532. <https://doi.org/10.3390/cryst13111532>.
- [148] K. Yamaguchi, T. Ishigaki, Y. Inoue, S. Arisawa, H. Matsunoshita, Y. Ito, H. Mori, K. Suehiro, K. Maki, K. Nagata, M. Demura, Comprehensive elemental screening of solid-solution copper alloys, *Sci. Technol. Adv. Mater. Methods* 3 (2023) 2250704. <https://doi.org/10.1080/27660400.2023.2250704>.
- [149] F. Li, W. Liu, C. Ding, S. Wang, X. Meng, The Microstructures and Properties of Cu–Ni–Co–Si Alloys: A Critical Review, *Metals* 15 (2025) 564. <https://doi.org/10.3390/met15050564>.
- [150] Y. Abe, S. Semboshi, N. Masahashi, S.H. Lim, E.-A. Choi, S.Z. Han, Mechanical Strength and Electrical Conductivity of Cu–In Solid Solution Alloy Wires, *Metall. Mater. Trans. A* 54 (2023) 928–938. <https://doi.org/10.1007/s11661-022-06938-1>.
- [151] D.-P. Lu, J. Wang, W.-J. Zeng, Y. Liu, L. Lu, B.-D. Sun, Study on high-strength and high-conductivity Cu–Fe–P alloys, *Mater. Sci. Eng. A* 421 (2006) 254–259. <https://doi.org/10.1016/j.msea.2006.01.068>.
- [152] C. Xia, W. Zhang, Z. Kang, Y. Jia, Y. Wu, R. Zhang, G. Xu, M. Wang, High strength and high electrical conductivity Cu–Cr system alloys manufactured by hot rolling–quenching process and thermomechanical treatments, *Mater. Sci. Eng. A* 538 (2012) 295–301. <https://doi.org/10.1016/j.msea.2012.01.047>.
- [153] A. Cui, X. Wang, Y. Cui, Multiscale modelling of precipitation hardening: a review, *J. Mater. Sci. Mater. Theory* 8 (2024) 13. <https://doi.org/10.1186/s41313-024-00066-6>.
- [154] Y. Jia, L. Li, Y. Pang, K. Ren, Y. Li, C. Zhang, J. Yi, Z. Li, Enhanced thermostability via dislocation-assisted heterogeneous co-precipitation in Cu–Cr–Hf alloy, *J. Mater. Sci. Technol.* 244 (2026) 88–101. <https://doi.org/10.1016/j.jmst.2025.04.040>.

- [155] Y. Zhang, Z. Liu, Y. Luo, S. Xiao, F. Liu, C. Guo, Q. Lei, High conductivity and high strength Cu-Ni-Co-Si alloys with hierarchical nanoscale and microscale precipitates, *J. Alloys Compd.* 1054 (2026) 186321. <https://doi.org/10.1016/j.jallcom.2026.186321>.
- [156] J.W. Martin, *Precipitation hardening*, 2nd ed, Butterworth-Heinemann, Oxford ; Boston, 1998.
- [157] Q. Wang, Z. Li, S. Pang, X. Li, C. Dong, P.K. Liaw, Coherent Precipitation and Strengthening in Compositionally Complex Alloys: A Review, *Entropy* 20 (2018) 878. <https://doi.org/10.3390/e20110878>.
- [158] X. Wu, W. Pang, T. Li, J. Sun, X. Liu, Achieving superior high-temperature strength–ductility synergy in a Cu–Ag–Zr alloy via trace Ce doping induced atomic substitution and precipitate refinement, *Acta Mater.* 308 (2026) 122011. <https://doi.org/10.1016/j.actamat.2026.122011>.
- [159] H.M. Nam, H. Lee, H. Kim, J.G. Lee, J. Kim, Precipitation strengthening of Cu–Ni–Si-based alloys: Experimental and computational insights, *J. Mater. Res. Technol.* 27 (2023) 5372–5379. <https://doi.org/10.1016/j.jmrt.2023.11.019>.
- [160] B. Shi, D. Hong, W. Wang, L. Su, C. Wang, L. Xiao, T. Ma, J. Zhou, A Novel Precipitation-Strengthened Conductive Copper Alloy: Cu–Hf–Si Alloy, *Adv. Eng. Mater.* 25 (2023) 2300944. <https://doi.org/10.1002/adem.202300944>.
- [161] Z. Niu, R. Ma, P. Wang, Z. Hao, Y. Shu, J. He, Precipitate sequence, strengthening mechanism and properties of Cu-0.9Be-1.0Ni alloys prepared by powder metallurgy, *Mater. Des.* 262 (2026) 115481. <https://doi.org/10.1016/j.matdes.2026.115481>.
- [162] R. Markandeya, S. Nagarjuna, D.S. Sarma, Precipitation hardening of Cu–Ti–Cr alloys, *Mater. Sci. Eng. A* 371 (2004) 291–305. <https://doi.org/10.1016/j.msea.2003.12.002>.
- [163] J. Dölling, R. Henle, U. Prah, A. Zilly, G. Nandi, Copper-Based Alloys with Optimized Hardness and High Conductivity: Research on Precipitation Hardening of Low-Alloyed Binary CuSc Alloys, *Metals* 12 (2022) 902. <https://doi.org/10.3390/met12060902>.
- [164] D.W. Lee, B.K. Kim, Nanostructured Cu–Al<sub>2</sub>O<sub>3</sub> composite produced by thermochemical process for electrode application, *Mater. Lett.* 58 (2004) 378–383. [https://doi.org/10.1016/S0167-577X\(03\)00505-6](https://doi.org/10.1016/S0167-577X(03)00505-6).
- [165] Y. Qin, Y. Tian, Y. Peng, L. Luo, X. Zan, Q. Xu, Y. Wu, Research status and development trend of preparation technology of ceramic particle dispersion strengthened copper-matrix composites, *J. Alloys Compd.* 848 (2020) 156475. <https://doi.org/10.1016/j.jallcom.2020.156475>.
- [166] X. Wang, X. Pan, P. Liu, Z. Xiao, T. Zhou, C. Gan, J. Wang, Improvement of Microstructure and Mechanical Properties of a Hot-Extruded Cu–Al<sub>2</sub>O<sub>3</sub> Alloy After Thermomechanical Treatment, *Materials* 18 (2025) 1606. <https://doi.org/10.3390/ma18071606>.
- [167] X. Zhao, L. Guo, L. Zhang, T. Jia, C. Chen, J. Hao, H. Shao, Z. Guo, J. Luo, J. Sun, Influence of nano-Al<sub>2</sub>O<sub>3</sub>-reinforced oxide-dispersion-strengthened Cu on the mechanical and tribological properties of Cu-based composites, *Int. J. Miner. Metall. Mater.* 23 (2016) 1444–1451. <https://doi.org/10.1007/s12613-016-1368-z>.
- [168] Y. Liu, S. Kondo, H. Yu, K. Yabuuchi, R. Kasada, Statistical Approach for Understanding the Effect of Specimen Size on the Yield Stress and Its Scattering in Mechanically-Alloyed Cu and ODS-Cu Obtained by Micro-Pillar Compression Test, *Mater. Trans.* 61 (2020) 955–962. <https://doi.org/10.2320/matertrans.MT-MBW2019005>.
- [169] M.B. Wilms, S.-K. Rittinghaus, Laser Additive Manufacturing of Oxide Dispersion-Strengthened Copper–Chromium–Niobium Alloys, *J. Manuf. Mater. Process.* 6 (2022) 102. <https://doi.org/10.3390/jmmp6050102>.
- [170] S. Qu, L. Wang, S. Zhang, C. Yang, H.Y. Chia, G. Wu, Z. Hu, J. Ding, W. Yan, Y. Zhang, C.H. Chan, W. Chen, Y. Lu, X. Song, Oxide-dispersion-enabled laser additive manufacturing of high-resolution copper, *Nat. Commun.* 16 (2025) 3234. <https://doi.org/10.1038/s41467-025-58373-6>.
- [171] B. Liu, C. Yuan, W. Zhao, X. Zhang, L. Zeng, Achieving the strength-conductivity synergy of Y<sub>2</sub>O<sub>3</sub> diffusion reinforced Cu matrix composites based on supercritical liquid system reduction, *Ceram. Int.* 51 (2025) 8116–8128. <https://doi.org/10.1016/j.ceramint.2024.12.247>.
- [172] S. Wang, G. Xie, J. Yang, F. Liu, X. Liu, Analysis of microstructure evolution and deformation mechanism of nano-oxides Al<sub>2</sub>O<sub>3</sub> dispersion strengthened copper alloy during compression at room temperature, *J. Alloys Compd.* 949 (2023) 169837. <https://doi.org/10.1016/j.jallcom.2023.169837>.
- [173] Y.A. Sorkhe, H. Aghajani, A. Taghizadeh Tabrizi, Mechanical alloying and sintering of nanostructured TiO<sub>2</sub> reinforced copper composite and its characterization, *Mater. Des.* 58 (2014) 168–174. <https://doi.org/10.1016/j.matdes.2014.01.040>.

- [174] D.P. Zhang, J. Chen, H.J. Li, L.F. Lu, Z.H. Chen, K.X. Wei, Softening and Strengthening Mechanism Induced by Cold Work in a Cu-Al<sub>2</sub>O<sub>3</sub> Composite with 0.1 wt.% Al Content, *Strength Mater.* 56 (2024) 764–773. <https://doi.org/10.1007/s11223-024-00689-7>.
- [175] L. Han, J. Wang, Y. Chen, Y. Huang, Y. Liu, Z. Wang, Fabrication and mechanical properties of WC nanoparticle dispersion-strengthened copper, *Mater. Sci. Eng. A* 817 (2021) 141274. <https://doi.org/10.1016/j.msea.2021.141274>.
- [176] Y. Yu, Y. Liu, Y. Teng, S. Feng, X. Wang, G. Chen, J. Yang, W. Zhang, Effect of Composite Plastic Deformation Technologies on the Microstructure Evolution and Properties of Cu-0.3wt.%Al<sub>2</sub>O<sub>3</sub> Dispersion-Strengthened Copper Alloy, *J. Mater. Eng. Perform.* 34 (2025) 19302–19311. <https://doi.org/10.1007/s11665-024-10561-z>.
- [177] Y. Li, C. Chen, W. Sun, M. Qi, J. Yan, C. Zhang, H. Liu, Y. Ren, X. Liu, Synergistic enhancement of strength-ductility in ODS copper alloy by exploring heterogeneous structure, *Mater. Sci. Eng. A* 931 (2025) 148194. <https://doi.org/10.1016/j.msea.2025.148194>.
- [178] H. Gao, F. Wu, P. Zhang, X. Zhang, X. Zhang, H. Xie, G. Sun, M. Li, Microstructure and mechanical property of Y<sub>2</sub>O<sub>3</sub> dispersion-strengthened copper alloy for fusion reactor prepared by spray drying, *Mater. Today Commun.* 46 (2025) 112549. <https://doi.org/10.1016/j.mtcomm.2025.112549>.
- [179] H. Liu, B. Gao, Y. Yang, M. Xu, X. Li, C. Li, H. Pan, J. Yang, H. Zhou, X. Zhu, Y. Zhu, Strain hardening behavior and microstructure evolution of gradient-structured Cu-Al alloys with low stack fault energy, *J. Mater. Res. Technol.* 19 (2022) 220–229. <https://doi.org/10.1016/j.jmrt.2022.05.027>.
- [180] X. Hu, H. Guan, Z. Chen, X. He, M. Wang, S. Qu, Effect of ultrasonic-assisted surface plastic deformation on the microstructures and tensile properties of discrete laser hardening treated Cr–Ni–Mo alloyed steel, *Mater. Sci. Eng. A* 862 (2023) 144495. <https://doi.org/10.1016/j.msea.2022.144495>.
- [181] H.R. Lin, Z.J. Zhang, Z.F. Zhang, Revealing and predicting the mechanical and strain hardening behaviors of Cu-Al alloys based on microscopic mechanism, *J. Alloys Compd.* 1003 (2024) 175604. <https://doi.org/10.1016/j.jallcom.2024.175604>.
- [182] E.C. Aifantis, The physics of plastic deformation, *Int. J. Plast.* 3 (1987) 211–247. [https://doi.org/10.1016/0749-6419\(87\)90021-0](https://doi.org/10.1016/0749-6419(87)90021-0).
- [183] W. Zhang, Y. Yi, S. Huang, H. He, F. Dong, Effects of Deformation at High, Medium, and Cryogenic Temperatures on the Microstructures and Mechanical Properties of Al-Zn-Mg-Cu Alloys, *Materials* 15 (2022) 6955. <https://doi.org/10.3390/ma15196955>.
- [184] A.F. Padilha, R.L. Plaut, P.R. Rios, Annealing of Cold-worked Austenitic Stainless Steels., *ISIJ Int.* 43 (2003) 135–143. <https://doi.org/10.2355/isijinternational.43.135>.
- [185] S.N. Naik, S.M. Walley, The Hall–Petch and inverse Hall–Petch relations and the hardness of nanocrystalline metals, *J. Mater. Sci.* 55 (2020) 2661–2681. <https://doi.org/10.1007/s10853-019-04160-w>.
- [186] P. Lisiecka-Graca, K. Bzowski, J. Majta, K. Muszka, A dislocation density-based model for the work hardening and softening behaviors upon stress reversal, *Arch. Civ. Mech. Eng.* 21 (2021) 84. <https://doi.org/10.1007/s43452-021-00239-x>.
- [187] P. Li, S.X. Li, Z.G. Wang, Z.F. Zhang, Fundamental factors on formation mechanism of dislocation arrangements in cyclically deformed fcc single crystals, *Prog. Mater. Sci.* 56 (2011) 328–377. <https://doi.org/10.1016/j.pmatsci.2010.12.001>.
- [188] O. Bouaziz, H. Zurob, M. Huang, Driving Force and Logic of Development of Advanced High Strength Steels for Automotive Applications, *Steel Res. Int.* 84 (2013) 937–947. <https://doi.org/10.1002/srin.201200288>.
- [189] M. Selin, Comparing Three Equations Used for Modeling the Tensile Flow Behavior of Compacted Graphite Cast Irons at Elevated Temperatures, *Metall. Mater. Trans. A* 41 (2010) 2805–2815. <https://doi.org/10.1007/s11661-010-0354-8>.
- [190] C.S. Pande, K.P. Cooper, Nanomechanics of Hall–Petch relationship in nanocrystalline materials, *Prog. Mater. Sci.* 54 (2009) 689–706. <https://doi.org/10.1016/j.pmatsci.2009.03.008>.
- [191] J. Li, K. Yamanaka, Y. Zhang, T. Furuhashi, G. Cao, J. Hu, A. Chiba, Reaching unconventionally large Hall–Petch coefficients in face-centered cubic high-entropy alloys, *Mater. Res. Lett.* 12 (2024) 399–407. <https://doi.org/10.1080/21663831.2024.2337211>.
- [192] I.P. Semenova, V.V. Polyakova, G.S. Dyakonov, A.V. Polyakov, Ultrafine-Grained Titanium-Based Alloys: Structure and Service Properties for Engineering Applications, *Adv. Eng. Mater.* 22 (2020) 1900651. <https://doi.org/10.1002/adem.201900651>.

- [193] J.J. Sidor, J.G. Bátorfi, Vickers indentation-based assessment of dislocation density and substructure evolution in aluminum alloys, *Mater. Charact.* 221 (2025) 114764. <https://doi.org/10.1016/j.matchar.2025.114764>.
- [194] H.S. Liu, B. Zhang, G.P. Zhang, Enhanced toughness and fatigue strength of cold roll bonded Cu/Cu laminated composites with mechanical contrast, *Scr. Mater.* 65 (2011) 891–894. <https://doi.org/10.1016/j.scriptamat.2011.08.001>.
- [195] S. Xie, J.J. Kruzic, Cold rolling improves the fracture toughness of a Zr-based bulk metallic glass, *J. Alloys Compd.* 694 (2017) 1109–1120. <https://doi.org/10.1016/j.jallcom.2016.10.134>.
- [196] K. Kim, M. Park, J. Jang, H.C. Kim, H.-S. Moon, D.-H. Lim, J.B. Jeon, S.-H. Kwon, H. Kim, B.J. Kim, Improvement of Strength and Impact Toughness for Cold-Worked Austenitic Stainless Steels Using a Surface-Cracking Technique, *Metals* 8 (2018) 932. <https://doi.org/10.3390/met8110932>.
- [197] R. Liu, Y. Tian, Z. Zhang, X. An, P. Zhang, Z. Zhang, Exceptional high fatigue strength in Cu-15at.%Al alloy with moderate grain size, *Sci. Rep.* 6 (2016) 27433. <https://doi.org/10.1038/srep27433>.
- [198] M. Liu, X.H. An, B.B. Wang, L.H. Wu, P. Xue, F.C. Liu, D.R. Ni, B.L. Xiao, Z.Y. Ma, Enhanced fatigue properties of large-scale ultrafine-grained copper fabricated by friction stir additive manufacturing and subsequent cold rolling, *Int. J. Fatigue* 183 (2024) 108253. <https://doi.org/10.1016/j.ijfatigue.2024.108253>.
- [199] E. Jiménez-Ruiz, R. Lostado-Lorza, C. Berlanga-Labari, A Comprehensive Review of Fatigue Strength in Pure Copper Metals (DHP, OF, ETP), *Metals* 14 (2024) 464. <https://doi.org/10.3390/met14040464>.
- [200] D. Xu, X. Wan, J. Yu, G. Xu, G. Li, Effect of Cold Deformation on Microstructures and Mechanical Properties of Austenitic Stainless Steel, *Metals* 8 (2018) 522. <https://doi.org/10.3390/met8070522>.
- [201] D.L. Zhang, K. Mihara, E. Takakura, H.G. Suzuki, Effect of the amount of cold working and ageing on the ductility of a Cu–15%Cr–0.2%Ti in-situ composite, *Mater. Sci. Eng. A* 266 (1999) 99–108. [https://doi.org/10.1016/S0921-5093\(99\)00028-3](https://doi.org/10.1016/S0921-5093(99)00028-3).
- [202] K. Edalati, Z. Horita, Recent Research Trends in Severe Plastic Deformation of Metallic and Non-Metallic Materials, *Mater. Trans.* 66 (2025) 450–461. <https://doi.org/10.2320/matertrans.MT-M2024189>.
- [203] K. Edalati, A. Bachmaier, V.A. Beloshenko, Y. Beygelzimer, V.D. Blank, W.J. Botta, K. Bryła, J. Čížek, S. Divinski, N.A. Enikeev, Y. Estrin, G. Faraji, R.B. Figueiredo, M. Fuji, T. Furuta, T. Grosdidier, J. Gubicza, A. Hohenwarter, Z. Horita, J. Huot, Y. Ikoma, M. Janeček, M. Kawasaki, P. Král, S. Kuramoto, T.G. Langdon, D.R. Leiva, V.I. Levitas, A. Mazilkin, M. Mito, H. Miyamoto, T. Nishizaki, R. Pippan, V.V. Popov, E.N. Popova, G. Purcek, O. Renk, Á. Révész, X. Sauvage, V. Sklenicka, W. Skrotzki, B.B. Straumal, S. Suwas, L.S. Toth, N. Tsuji, R.Z. Valiev, G. Wilde, M.J. Zehetbauer, X. Zhu, Nanomaterials by severe plastic deformation: review of historical developments and recent advances, *Mater. Res. Lett.* 10 (2022) 163–256. <https://doi.org/10.1080/21663831.2022.2029779>.
- [204] K. Neishi, T. Uchida, A. Yamauchi, K. Nakamura, Z. Horita, T.G. Langdon, Low-temperature superplasticity in a Cu–Zn–Sn alloy processed by severe plastic deformation, *Mater. Sci. Eng. A* 307 (2001) 23–28. [https://doi.org/10.1016/S0921-5093\(00\)01970-5](https://doi.org/10.1016/S0921-5093(00)01970-5).
- [205] X. Sauvage, F. Wetscher, P. Pareige, Mechanical alloying of Cu and Fe induced by severe plastic deformation of a Cu–Fe composite, *Acta Mater.* 53 (2005) 2127–2135. <https://doi.org/10.1016/j.actamat.2005.01.024>.
- [206] A. Mishra, B. Kad, F. Gregori, M. Meyers, Microstructural evolution in copper subjected to severe plastic deformation: Experiments and analysis, *Acta Mater.* 55 (2007) 13–28. <https://doi.org/10.1016/j.actamat.2006.07.008>.
- [207] G. Khatibi, J. Horky, B. Weiss, M.J. Zehetbauer, High cycle fatigue behaviour of copper deformed by high pressure torsion, *Int. J. Fatigue* 32 (2010) 269–278. <https://doi.org/10.1016/j.ijfatigue.2009.06.017>.
- [208] J. Gubicza, S.V. Dobatkin, E. Khosravi, A.A. Kuznetsov, J.L. Lábár, Microstructural stability of Cu processed by different routes of severe plastic deformation, *Mater. Sci. Eng. A* 528 (2011) 1828–1832. <https://doi.org/10.1016/j.msea.2010.11.026>.
- [209] Y.Z. Tian, S.D. Wu, Z.F. Zhang, R.B. Figueiredo, N. Gao, T.G. Langdon, Comparison of microstructures and mechanical properties of a Cu–Ag alloy processed using different severe plastic deformation modes, *Mater. Sci. Eng. A* 528 (2011) 4331–4336. <https://doi.org/10.1016/j.msea.2011.01.057>.
- [210] K. Abib, J.A.M. Balanos, B. Alili, D. Bradai, On the microstructure and texture of Cu–Cr–Zr alloy after severe plastic deformation by ECAP, *Mater. Charact.* 112 (2016) 252–258. <https://doi.org/10.1016/j.matchar.2015.12.026>.
- [211] D. Lu, J. Jiang, L. Lu, X. Liao, K.M. Nesterov, R.K. Islamgaliev, R.Z. Valiev, K. Liu, Hardness, Electrical Conductivity and Thermal Stability of Externally Oxidized Cu–Al<sub>2</sub>O<sub>3</sub> Composite Processed by SPD, *J. Mater. Eng. Perform.* 26 (2017) 2110–2117. <https://doi.org/10.1007/s11665-017-2660-4>.

- [212] M.Y. Alawadhi, S. Sabbaghianrad, Y. Huang, Terence.G. Langdon, Direct influence of recovery behaviour on mechanical properties in oxygen-free copper processed using different SPD techniques: HPT and ECAP, *J. Mater. Res. Technol.* 6 (2017) 369–377. <https://doi.org/10.1016/j.jmrt.2017.05.005>.
- [213] T. Kunimine, M. Watanabe, A Comparative Study of Hardness in Nanostructured Cu–Zn, Cu–Si and Cu–Ni Solid-Solution Alloys Processed by Severe Plastic Deformation, *Mater. Trans.* 60 (2019) 1484–1488. <https://doi.org/10.2320/matertrans.MF201944>.
- [214] G. Kapoor, T. Kvackaj, A. Heczal, J. Bidulská, R. Kočíško, Z. Fogarassy, D. Simcak, J. Gubicza, The Influence of Severe Plastic Deformation and Subsequent Annealing on the Microstructure and Hardness of a Cu–Cr–Zr Alloy, *Materials* 13 (2020) 2241. <https://doi.org/10.3390/ma13102241>.
- [215] P.H.F. Oliveira, D.C.C. Magalhães, L.F.K. Unti, O.M. Cintho, A.M. Kliauga, V.L. Sordi, Tailoring the microstructure of a Cu-0.7Cr-0.07Zr alloy submitted to ECAP at cryogenic temperature for improved thermal stability, *Mater. Character.* 190 (2022) 112045. <https://doi.org/10.1016/j.matchar.2022.112045>.
- [216] T. Zargar, F. Sadeghi, T. Mousavi, S. Planat, S. Ögüt, Y. Huang, T.G. Langdon, Characterization of Cu-Nb-Cu heterostructure fabricated by high-pressure torsion, *J. Alloys Compd.* 1029 (2025) 180732. <https://doi.org/10.1016/j.jallcom.2025.180732>.
- [217] P. Thibakarmoorthy, A. Thounaojam, A. Sahadevan, S. Vivekanandan, M.S. Krishnalal, S. Dhasan, Multi-pass friction stir processing of pure copper: A pathway to superior strength and corrosion resistance, *Mater. Lett.* 396 (2025) 138752. <https://doi.org/10.1016/j.matlet.2025.138752>.
- [218] P.C.A. Flausino, N.A.N. Da Silva, B.C. De Almeida, T.M. Manhabosco, E.C.S. Corrêa, M.T.P. Aguilar, P.R. Cetlin, The Structural Refinement of Commercial-Purity Copper Processed by Equal Channel Angular Pressing with Low Strain Amplitude, *Adv. Eng. Mater.* 27 (2025) 2501058. <https://doi.org/10.1002/adem.202501058>.
- [219] P.C.A. Flausino, N.A.N.D. Silva, L.M. Reis, T.M. Manhabosco, M.T.P. Aguilar, P.R. Cetlin, Microstructural and mechanical behavior evolution in copper under ECAP+MDF strain paths, *Mater. Sci. Eng. A* 949 (2026) 149395. <https://doi.org/10.1016/j.msea.2025.149395>.
- [220] S.O. Rogachev, R.V. Sundeev, V.A. Troshina, N.Yu. Tabachkova, I.N. Pashkov, Features of hardening behavior of Cu–20Ni–20Mn (wt%) alloy during a combination of aging treatment and severe plastic deformation process, *Mater. Lett.* 406 (2026) 139841. <https://doi.org/10.1016/j.matlet.2025.139841>.
- [221] S.M. Ghalehbandi, M. Malaki, M. Gupta, Accumulative Roll Bonding—A Review, *Appl. Sci.* 9 (2019) 3627. <https://doi.org/10.3390/app9173627>.
- [222] C. You, W. Xie, S. Miao, T. Liang, L. Zeng, X. Zhang, H. Wang, High strength, high electrical conductivity and thermally stable bulk Cu/Ag nanolayered composites prepared by cross accumulative roll bonding, *Mater. Des.* 200 (2021) 109455. <https://doi.org/10.1016/j.matdes.2021.109455>.
- [223] M.R. Toroghinejad, R. Jamaati, J. Dutkiewicz, J.A. Szpunar, Investigation of nanostructured aluminum/copper composite produced by accumulative roll bonding and folding process, *Mater. Des.* 51 (2013) 274–279. <https://doi.org/10.1016/j.matdes.2013.04.002>.
- [224] Y. Sun, Y. Chen, N. Tsuji, S. Guan, Microstructural evolution and mechanical properties of nanostructured Cu/Ni multilayer fabricated by accumulative roll bonding, *J. Alloys Compd.* 819 (2020) 152956. <https://doi.org/10.1016/j.jallcom.2019.152956>.
- [225] A. Ran, A. Afshar, S. Mehmet, Y. Kemal, Obtaining ultrafine-grained structure in copper/steel/copper multi-laminated composite by different accumulative roll bonding methods, *Vacuum* 236 (2025) 114155. <https://doi.org/10.1016/j.vacuum.2025.114155>.
- [226] J.S. Carpenter, S.C. Vogel, J.E. LeDonne, D.L. Hammon, I.J. Beyerlein, N.A. Mara, Bulk texture evolution of Cu–Nb nanolamellar composites during accumulative roll bonding, *Acta Mater.* 60 (2012) 1576–1586. <https://doi.org/10.1016/j.actamat.2011.11.045>.
- [227] H. Mansouri, B. Eghbali, M. Afrand, Producing multi-layer composite of stainless steel/aluminum/copper by accumulative roll bonding (ARB) process, *J. Manuf. Process.* 46 (2019) 298–303. <https://doi.org/10.1016/j.jmapro.2019.08.025>.
- [228] A.H. Eslami, S.M.H. Seyedkashi, M.H. Farshidi, Study of the effects of heat treatment on structural and mechanical properties of Cu/Ni/Al layered composite produced by accumulated roll bonding, *J. Alloys Compd.* 1049 (2025) 185418. <https://doi.org/10.1016/j.jallcom.2025.185418>.
- [229] M.M. Mahdavian, H.R. Jafarian, A.R. Khodabandeh, Sh. Mirdamadi, Innovative Al/Cu/Sn/Ni multi-layer composites: Tailoring mechanical properties and wear behavior through accumulative roll bonding (ARB) and post heat treatment, *J. Alloys Compd.* 1045 (2025) 184593. <https://doi.org/10.1016/j.jallcom.2025.184593>.
- [230] H. Safahi, M.E. Imani, G. Faraji, A new approach for processing fibrous W Cu composite via ARB process, *Mater. Lett.* 405 (2026) 139772. <https://doi.org/10.1016/j.matlet.2025.139772>.

- [231] A.R. Eivani, A. Shojaei, N. Park, H.R. Jafarian, Fabrication of Cu-CuG nanocomposites with enhanced mechanical strength and reduced electrical resistivity, *J. Mater. Res. Technol.* 11 (2021) 650–666. <https://doi.org/10.1016/j.jmrt.2021.01.045>.
- [232] H.N. Omran, A.R. Eivani, M. Farbakhti, H.R. Jafarian, Tribological properties of copper-graphene (CuG) composite fabricated by accumulative roll bonding, *J. Mater. Res. Technol.* 25 (2023) 4650–4657. <https://doi.org/10.1016/j.jmrt.2023.06.261>.
- [233] D. Zhang, L. Zhang, Y. Zhang, X. Guo, J. Du, E. Wang, Microstructural characteristics and properties of dual-heterogeneous layered Cu/Cu-Ag-HEA composites, *Mater. Sci. Eng. A* 946 (2025) 149108. <https://doi.org/10.1016/j.msea.2025.149108>.
- [234] P. Berczki, V. Szombathelyi, G. Krállics, Determination of flow curve at large cyclic plastic strain by multiaxial forging on MaxStrain System, *Int. J. Mech. Sci.* 84 (2014) 182–188. <https://doi.org/10.1016/j.ijmecsci.2014.04.025>.
- [235] J. Dutkiewicz, P. Bobrowski, S. Rusz, O. Hilser, T.A. Tański, W. Borek, M. Łagoda, P. Ostachowski, P. Pałka, G. Boczekal, D. Kuc, T. Mikuszewski, Effect of Various SPD Techniques on Structure and Superplastic Deformation of Two Phase MgLiAl Alloy, *Met. Mater. Int.* 24 (2018) 1077–1089. <https://doi.org/10.1007/s12540-018-0118-3>.
- [236] V. Komarov, I. Khmelevskaya, R. Karelin, S. Prokoshkin, M. Zaripova, M. Isaenkova, G. Korpala, R. Kawalla, Effect of biaxial cyclic severe deformation on structure and properties of Ti-Ni alloys, *J. Alloys Compd.* 797 (2019) 842–848. <https://doi.org/10.1016/j.jallcom.2019.05.127>.
- [237] M. Wojtaszek, G. Korpala, T. Śleboda, K. Zyguła, U. Prah, Hot Processing of Powder Metallurgy and Wrought Ti-6Al-4V Alloy with Large Total Deformation: Physical Modeling and Verification by Rolling, *Metall. Mater. Trans. A* 51 (2020) 5790–5805. <https://doi.org/10.1007/s11661-020-05942-7>.
- [238] D.S. Svyetlichnyy, J. Majta, R. Kuziak, K. Muszka, Experimental and modelling study of the grain refinement of Fe-30wt%Ni-Nb austenite model alloy subjected to severe plastic deformation process, *Arch. Civ. Mech. Eng.* 21 (2021) 20. <https://doi.org/10.1007/s43452-021-00178-7>.
- [239] H. Petryk, S. Stupkiewicz, R. Kuziak, Grain refinement and strain hardening in IF steel during multi-axis compression: Experiment and modelling, *J. Mater. Process. Technol.* 204 (2008) 255–263. <https://doi.org/10.1016/j.jmatprotec.2007.11.068>.
- [240] D. Leśniak, J. Zasadziński, W. Libura, Z. Gronostajski, R. Śliwa, B. Leszczyńska-Madej, M. Kaszuba, A. Woźnicki, B. Płonka, P. Widomski, J. Madura, Latest advances in extrusion processes of light metals, *Arch. Civ. Mech. Eng.* 24 (2024) 184. <https://doi.org/10.1007/s43452-024-00988-5>.
- [241] A. Korbel, W. Bochniak, Lüders deformation and superplastic flow of metals extruded by KOB method, *Philos. Mag.* 93 (2013) 1883–1913. <https://doi.org/10.1080/14786435.2013.765977>.
- [242] D. Andrzejewski, J. Jakubowicz, J. Borowski, Structure and properties of 7075 aluminum alloy products obtained with the KOB method, *Arch. Civ. Mech. Eng.* 16 (2016) 217–223. <https://doi.org/10.1016/j.acme.2015.10.005>.
- [243] M. Wójcik, A. Skrzat, The Coupled Eulerian-Lagrangian Analysis of the KOB Extrusion Process, *Adv. Sci. Technol. Res. J.* 15 (2021) 197–208. <https://doi.org/10.12913/22998624/131663>.
- [244] P. Długosz, W. Bochniak, P. Ostachowski, R. Molak, M. Duarte Guigou, M. Hebda, The Influence of Conventional or KOB Extrusion Process on the Properties of AZ91 (MgAl9Zn1) Alloy, *Materials* 14 (2021) 6543. <https://doi.org/10.3390/ma14216543>.
- [245] A. Kalhor, K. Rodak, M. Tkocz, H. Myalska-Głowacka, B. Chmiela, M. Wątroba, S. Boczekal, G. Junak, Microstructure, mechanical properties, and corrosion behavior of a biodegradable Zn–1.7Mg–1Ca alloy processed by KoBo extrusion, *Mater. Sci. Eng. A* 887 (2023) 145771. <https://doi.org/10.1016/j.msea.2023.145771>.
- [246] P. Snopiński, K. Matus, Characterisation of Microstructure and Special Grain Boundaries in LPBF AlSi10Mg Alloy Subjected to the KoBo Extrusion Process, *Symmetry* 15 (2023) 1634. <https://doi.org/10.3390/sym15091634>.
- [247] A. Korbel, W. Bochniak, Refinement and control of the metal structure elements by plastic deformation, *Scr. Mater.* 51 (2004) 755–759. <https://doi.org/10.1016/j.scriptamat.2004.06.020>.
- [248] P. Ostachowski, W. Bochniak, M. Łagoda, S. Ziółkiewicz, Strength properties and structure of CuCrZr alloy subjected to low-temperature KOB extrusion and heat treatment, *Int. J. Adv. Manuf. Technol.* 105 (2019) 5023–5044. <https://doi.org/10.1007/s00170-019-04602-4>.
- [249] L. Qian, Z. Cui, C. Sun, S. Geng, Z. Sun, Investigation of deformation compatibility and power consumption during KOB extrusion of bimetallic composite tube, *Int. J. Adv. Manuf. Technol.* 118 (2022) 3477–3486. <https://doi.org/10.1007/s00170-021-08608-9>.

- [250] P. Koprowski, M. Bieda, S. Boczkal, A. Jarzębska, P. Ostachowski, J. Kawałko, T. Czeppe, W. Maziarz, M. Łagoda, K. Sztwiertnia, AA6013 aluminium alloy deformed by forward-backward rotating die (KoBo): Microstructure and mechanical properties control by changing the die oscillation frequency, *J. Mater. Process. Technol.* 253 (2018) 34–42. <https://doi.org/10.1016/j.jmatprotec.2017.10.043>.
- [251] W. Bochniak, P. Ostachowski, A. Korbel, M. Łagoda, Potential of the KOBO extrusion process for nonferrous metals in the form of solids and chips, *Int. J. Adv. Manuf. Technol.* 127 (2023) 733–750. <https://doi.org/10.1007/s00170-023-11596-7>.
- [252] A. Korbel, L. Błaż, W. Bochniak, M. Pawlyta, P. Ostachowski, M. Łagoda, Nano-Dimensional Elements in the Structure of Zinc Subjected to KOBO Extrusion, *Metallogr. Microstruct. Anal.* 12 (2023) 427–432. <https://doi.org/10.1007/s13632-023-00945-3>.
- [253] P. Ostachowski, A. Paliborek, W. Bochniak, M. Łagoda, Mechanical Characteristics and Structure of Highly Deformed Zinc, *J. Mater. Eng. Perform.* 31 (2022) 3638–3660. <https://doi.org/10.1007/s11665-021-06520-7>.
- [254] W. Bochniak, A. Korbel, P. Ostachowski, M. Łagoda, Plastic flow of metals under cyclic change of deformation path conditions, *Arch. Civ. Mech. Eng.* 18 (2018) 679–686. <https://doi.org/10.1016/j.acme.2017.11.004>.
- [255] B. Pawłowska, R.E. Śliwa, M. Zwolak, Possibilities to Obtain Products from 2024 and 7075 Chips in the Process of Consolidation by KoBo Extrusion, *Arch. Metall. Mater.* (2019) 1213–1221. <https://doi.org/10.24425/amm.2019.130083>.
- [256] Z.C. Cordero, B.E. Knight, C.A. Schuh, Six decades of the Hall–Petch effect – a survey of grain-size strengthening studies on pure metals, *Int. Mater. Rev.* 61 (2016) 495–512. <https://doi.org/10.1080/09506608.2016.1191808>.
- [257] Y. Gu, R. Huang, Y. Hao, Review on Grain Refinement of Metallic Materials to Regulate Cellular Behavior, *Metals* 12 (2022) 829. <https://doi.org/10.3390/met12050829>.
- [258] S. Dangwal, K. Edalati, R.Z. Valiev, T.G. Langdon, Breaks in the Hall–Petch Relationship after Severe Plastic Deformation of Magnesium, Aluminum, Copper, and Iron, *Crystals* 13 (2023) 413. <https://doi.org/10.3390/cryst13030413>.
- [259] Z. Li, K. Chen, X. Chen, Y. Zhu, M. Chen, Y. Wang, J. Shen, J. Shi, Z. Wang, In-Situ Fabrication, Microstructure and Mechanical Performance of Nano Iron-Rich Precipitate Reinforced Cu and Cu Alloys, *Metals* 12 (2022) 1453. <https://doi.org/10.3390/met12091453>.
- [260] N. Liang, Y. Zhao, J. Wang, Y. Zhu, Effect of grain structure on Charpy impact behavior of copper, *Sci. Rep.* 7 (2017) 44783. <https://doi.org/10.1038/srep44783>.
- [261] Y. Zhao, I.C. Cheng, M.E. Kassner, A.M. Hodge, The effect of nanotwins on the corrosion behavior of copper, *Acta Mater.* 67 (2014) 181–188. <https://doi.org/10.1016/j.actamat.2013.12.030>.
- [262] H. Miyamoto, K. Harada, T. Mimaki, A. Vinogradov, S. Hashimoto, Corrosion of ultra-fine grained copper fabricated by equal-channel angular pressing, *Corros. Sci.* 50 (2008) 1215–1220. <https://doi.org/10.1016/j.corsci.2008.01.024>.
- [263] N. Liang, Y. Zhao, Y. Li, T. Topping, Y. Zhu, R.Z. Valiev, E.J. Lavernia, Influence of microstructure on thermal stability of ultrafine-grained Cu processed by equal channel angular pressing, *J. Mater. Sci.* 53 (2018) 13173–13185. <https://doi.org/10.1007/s10853-018-2548-1>.
- [264] J. Ding, Y. Zhang, T. Niu, Z. Shang, S. Xue, B. Yang, J. Li, H. Wang, X. Zhang, Thermal Stability of Nanocrystalline Gradient Inconel 718 Alloy, *Crystals* 11 (2021) 53. <https://doi.org/10.3390/cryst11010053>.
- [265] W.S. Cunningham, S.T.J. Mascarenhas, J.S. Riano, W. Wang, S. Hwang, K. Hattar, A.M. Hodge, J.R. Trelewicz, Unraveling Thermodynamic and Kinetic Contributions to the Stability of Doped Nanocrystalline Alloys using Nanometallic Multilayers, *Adv. Mater.* 34 (2022) 2200354. <https://doi.org/10.1002/adma.202200354>.
- [266] E. Ma, T. Zhu, Towards strength–ductility synergy through the design of heterogeneous nanostructures in metals, *Mater. Today* 20 (2017) 323–331. <https://doi.org/10.1016/j.mattod.2017.02.003>.
- [267] S. Zhong, X. Zhang, A. Liu, B. Zhang, Electrical and Thermal Conductivity of Graphene/Copper Composites and Their Applications in High-Efficiency Current-Carrying Conductors: A Review, *Adv. Eng. Mater.* 27 (2025) 2401950. <https://doi.org/10.1002/adem.202401950>.
- [268] B. Sundqvist, Resistivity saturation in crystalline metals: Semi-classical theory versus experiment, *J. Phys. Chem. Solids* 165 (2022) 110686. <https://doi.org/10.1016/j.jpcs.2022.110686>.
- [269] K.F. Lunn, D. Apelian, Thermal and Electrical Conductivity of Aluminum Alloys: Fundamentals, structure-property relationships, and pathways to enhance conductivity, *Mater. Sci. Eng. A* 924 (2025) 147766. <https://doi.org/10.1016/j.msea.2024.147766>.

- [270] M.R. Akbarpour, F. Gazani, H. Mousa Mirabad, I. Khezri, A. Moeini, N. Sohrabi, H.S. Kim, Recent advances in processing, and mechanical, thermal and electrical properties of Cu-SiC metal matrix composites prepared by powder metallurgy, *Prog. Mater. Sci.* 140 (2023) 101191. <https://doi.org/10.1016/j.pmatsci.2023.101191>.
- [271] J. Dai, G. Liu, The Mechanism of High Electrical Conductivity in Copper–Chromium Alloy, *Metals* 15 (2025) 735. <https://doi.org/10.3390/met15070735>.
- [272] C.E. Owens, G.H. McKinley, A.J. Hart, Manufacturing of high-conductivity carbon nanotube fibers and extensible coils by immersed extrusion, *Mater. Today* 75 (2024) 97–111. <https://doi.org/10.1016/j.mattod.2024.04.008>.
- [273] T. Tanaka, A. Yabuki, Fabrication of chitosan nanofiber composite copper thin film with high electrical conductivity using copper formate complex ink, *Mater. Sci. Eng. B* 324 (2026) 118960. <https://doi.org/10.1016/j.mseb.2025.118960>.
- [274] S.W. Wang, L.D. Chen, Y.S. Kang, M. Niino, T. Hirai, Effect of plasma activated sintering (PAS) parameters on densification of copper powder, *Mater. Res. Bull.* 35 (2000) 619–628. [https://doi.org/10.1016/S0025-5408\(00\)00246-4](https://doi.org/10.1016/S0025-5408(00)00246-4).
- [275] S.-G. Hwang, M.-Y. Lee, B.-S. Ko, Numerical Analysis on Cooling Performances for Connectors Using Immersion Cooling in Ultra-Fast Chargers for Electric Vehicles, *Symmetry* 17 (2025) 624. <https://doi.org/10.3390/sym17040624>.
- [276] T.G. Sousa, I.A.D.B. Moura, F.D.C. Garcia Filho, S.N. Monteiro, L.P. Brandão, Combining severe plastic deformation and precipitation to enhance mechanical strength and electrical conductivity of Cu–0.65Cr–0.08Zr alloy, *J. Mater. Res. Technol.* 9 (2020) 5953–5961. <https://doi.org/10.1016/j.jmrt.2020.03.124>.
- [277] J.E. González-Hernández, J.M. Cubero-Sesin, Electrical Conductivity of Ultrafine-Grained Cu and Al Alloys: Attaining the Best Compromise with Mechanical Properties, *Mater. Trans.* 64 (2023) 1754–1768. <https://doi.org/10.2320/matertrans.MT-MF2022046>.
- [278] J.H. Moon, E. Jeong, S. Kim, T. Kim, E. Oh, K. Lee, H. Han, Y.K. Kim, Materials Quest for Advanced Interconnect Metallization in Integrated Circuits, *Adv. Sci.* 10 (2023) 2207321. <https://doi.org/10.1002/adv.202207321>.
- [279] V. Palenskis, New Insight into Electric Force in Metal and the Quadratic Electrical Resistivity Law of Metals at Low Temperatures, *Metals* 14 (2024) 526. <https://doi.org/10.3390/met14050526>.
- [280] T. Kim, D. Lee, Unveiling key descriptors for electrical resistivity of alloys using high-throughput experiments and explainable AI, *Npj Comput. Mater.* 11 (2025) 300. <https://doi.org/10.1038/s41524-025-01799-9>.
- [281] K.J. Laws, K.F. Shamlaye, B. Gun, M. Ferry, Synthesis of copper-based bulk metallic glasses in the ternary Cu–Mg–Ca system, *J. Alloys Compd.* 486 (2009) L27–L29. <https://doi.org/10.1016/j.jallcom.2009.06.205>.
- [282] K.J. Laws, K.F. Shamlaye, K. Wong, B. Gun, M. Ferry, Prediction of Glass-Forming Compositions in Metallic Systems: Copper-Based Bulk Metallic Glasses in the Cu–Mg–Ca System, *Metall. Mater. Trans. A* 41 (2010) 1699–1705. <https://doi.org/10.1007/s11661-010-0274-7>.
- [283] W. Ben Mbarek, E. Pineda, L. Escoda, J.J. Suñol, M. Khitouni, Dealloying of Cu–Mg–Ca Alloys, *Metals* 8 (2018) 919. <https://doi.org/10.3390/met8110919>.
- [284] P. Strzpek, M. Zasadzińska, P. Noga, M. Walkowicz, P. Osuch, Immersion Corrosion Resistance of Binary High-Strength Cu–Mg Alloys, *J. Mater. Eng. Perform.* 34 (2025) 30555–30565. <https://doi.org/10.1007/s11665-025-12402-z>.
- [285] P. Strzpek, M. Zasadzińska, Prospective cold metal working and analysis of deformation susceptibility of CuMg alloys with high magnesium content, *Sci. Rep.* 14 (2024) 6447. <https://doi.org/10.1038/s41598-024-57083-1>.
- [286] P. Strzpek, A. Nowak, M. Zasadzińska, Various Manufacturing Technologies and Their Influence on the Fatigue Strength of Cu–Mg Wires, *Metals* 15 (2025) 216. <https://doi.org/10.3390/met15020216>.
- [287] N. Gao, M.J. Starink, T.G. Langdon, Using differential scanning calorimetry as an analytical tool for ultrafine grained metals processed by severe plastic deformation, *Mater. Sci. Technol.* 25 (2009) 687–698. <https://doi.org/10.1179/174328409X408901>.
- [288] J. Zyguntowicz, P. Wiecińska, M. Wachowski, M. Kurek, W. Kaszuwara, Thermogravimetric analysis coupled with mass spectrometry of ceramic–metal ternary composites Al<sub>2</sub>O<sub>3</sub>–Cu–Mo, *Measurement* 217 (2023) 113049. <https://doi.org/10.1016/j.measurement.2023.113049>.
- [289] P. Mandal, S.C. Mondal, Investigation of Electro-Thermal property for Cu–MWCNT composite coating on anodized 6061 aluminium alloy, *Appl. Surf. Sci.* 454 (2018) 138–147. <https://doi.org/10.1016/j.apsusc.2018.05.130>.

- [290] Y. Wang, H.-Y. Huang, J.-X. Xie, Texture evolution and flow stress of columnar-grained polycrystalline copper during intense plastic deformation process at room temperature, *Mater. Sci. Eng. A* 530 (2011) 418–425. <https://doi.org/10.1016/j.msea.2011.09.105>.
- [291] R. Henle, J. Dölling, U. Prah, G. Nandi, A. Zilly, DSC Analysis of the Effect of Cold Deformation on the Precipitation Kinetics of a Binary Cu-Sc Alloy, *Materials* 16 (2023) 3462. <https://doi.org/10.3390/ma16093462>.
- [292] B. Milkereit, L. Burgschat, R.H. Kemsies, A. Springer, C. Schick, O. Kessler, In situ differential scanning calorimetry analysis of dissolution and precipitation kinetics in Mg–Y–RE alloy WE43, *J. Magnes. Alloys* 7 (2019) 1–14. <https://doi.org/10.1016/j.jma.2019.01.002>.
- [293] Ł. Rakoczy, M. Grudzień-Rakoczy, R. Cygan, B. Rutkowski, T. Kargul, T. Dudziak, E. Rząd, O. Milkovič, A. Zielińska-Lipiec, Characterization of the as-cast microstructure and selected properties of the X-40 Co-based superalloy produced via lost-wax casting, *Arch. Civ. Mech. Eng.* 22 (2022) 143. <https://doi.org/10.1007/s43452-022-00466-w>.
- [294] K. Lin, S.D. Pang, The influence of thermal residual stresses and thermal generated dislocation on the mechanical response of particulate-reinforced metal matrix nanocomposites, *Compos. Part B Eng.* 83 (2015) 105–116. <https://doi.org/10.1016/j.compositesb.2015.08.008>.
- [295] Y.N. Hou, K.M. Yang, J. Song, H. Wang, Y. Liu, T.X. Fan, A crystal plasticity model for metal matrix composites considering thermal mismatch stress induced dislocations and twins, *Sci. Rep.* 11 (2021) 16053. <https://doi.org/10.1038/s41598-021-95439-z>.
- [296] H. Lin, C. Lu, C. Liu, C. Chen, D. Chen, J.-C. Kuo, K.N. Tu, Microstructure control of unidirectional growth of  $\eta$ -Cu<sub>6</sub>Sn<sub>5</sub> in microbumps on  $\langle 1\ 1\ 1 \rangle$  oriented and nanotwinned Cu, *Acta Mater.* 61 (2013) 4910–4919. <https://doi.org/10.1016/j.actamat.2013.04.056>.
- [297] A.R. Alian, Y. Ju, S.A. Meguid, Comprehensive atomistic modeling of copper nanowires-based surface connectors, *Mater. Des.* 175 (2019) 107812. <https://doi.org/10.1016/j.matdes.2019.107812>.
- [298] G. Bruzzone, The binary systems calcium-copper, strontium-copper and barium-copper, *J. Common Met.* 25 (1971) 361–366. [https://doi.org/10.1016/0022-5088\(71\)90178-0](https://doi.org/10.1016/0022-5088(71)90178-0).
- [299] S. Gourdet, F. Montheillet, A model of continuous dynamic recrystallization, *Acta Mater.* 51 (2003) 2685–2699. [https://doi.org/10.1016/S1359-6454\(03\)00078-8](https://doi.org/10.1016/S1359-6454(03)00078-8).
- [300] D. Xu, M. Zhou, Y. Zhang, S. Tang, Z. Zhang, Y. Liu, B. Tian, X. Li, Y. Jia, A.A. Volinsky, D. Li, Q. Liu, Microstructure and hot deformation behavior of the Cu–Sn–Ni–Zn–Ti(–Y) alloy, *Mater. Charact.* 196 (2023) 112559. <https://doi.org/10.1016/j.matchar.2022.112559>.
- [301] D. Field, L. Bradford, M. Nowell, T. Lillo, The role of annealing twins during recrystallization of Cu, *Acta Mater.* 55 (2007) 4233–4241. <https://doi.org/10.1016/j.actamat.2007.03.021>.
- [302] H. Wang, D. Yin, M.-C. Zhao, Y. Tian, A. Atrens, The hot deformation characteristics and the associated microstructural evolution of an Al–Cu–Li–Zn alloy studied by constitutive equations and processing maps, *J. Mater. Res. Technol.* 27 (2023) 401–418. <https://doi.org/10.1016/j.jmrt.2023.09.222>.
- [303] H. Zhang, K. Zhang, H. Zhou, Z. Lu, C. Zhao, X. Yang, Effect of strain rate on microstructure evolution of a nickel-based superalloy during hot deformation, *Mater. Des.* 80 (2015) 51–62. <https://doi.org/10.1016/j.matdes.2015.05.004>.
- [304] L. Lapeire, J. Sidor, P. Verleysen, K. Verbeken, I. De Graeve, H. Terry, L.A.I. Kestens, Texture comparison between room temperature rolled and cryogenically rolled pure copper, *Acta Mater.* 95 (2015) 224–235. <https://doi.org/10.1016/j.actamat.2015.05.035>.
- [305] Y. Ban, Y. Zhang, B. Tian, K. Song, M. Zhou, X. Zhang, Y. Jia, X. Li, Y. Geng, Y. Liu, A.A. Volinsky, EBSD analysis of hot deformation behavior of Cu–Ni–Co–Si–Cr alloy, *Mater. Charact.* 169 (2020) 110656. <https://doi.org/10.1016/j.matchar.2020.110656>.
- [306] Q. Guo, X. Jiang, L. Li, S. Li, Z. Xiao, Y. Sun, Z. Li, Hot workability, microstructural evolution, and dual path DRX mechanism of the 70Cu–30Ni alloy, *J. Mater. Res. Technol.* 38 (2025) 5301–5317. <https://doi.org/10.1016/j.jmrt.2025.08.282>.
- [307] J.A. Muñoz, T. Khelfa, D. Gheorghe, O.F. Higuera, P. Rodriguez, J.M. Cabrera, Microstructure Characterization of Metallic Materials Processed by Equal Channel Angular Pressing (ECAP): An Electron Backscatter Diffraction (EBSD) Analysis, *Mater. Trans.* 64 (2023) 1791–1805. <https://doi.org/10.2320/matertrans.MT-MF2022042>.
- [308] Y. Zhang, P. Liu, B. Tian, Y. Liu, R. Li, Q. Xu, Hot deformation behavior and processing map of Cu–Ni–Si–P alloy, *Trans. Nonferrous Met. Soc. China* 23 (2013) 2341–2347. [https://doi.org/10.1016/S1003-6326\(13\)62739-9](https://doi.org/10.1016/S1003-6326(13)62739-9).

- [309] Y. Liu, H. Huang, J. Xie, Effect of compression direction on the dynamic recrystallization behavior of continuous columnar-grained CuNi10Fe1Mn alloy, *Int. J. Miner. Metall. Mater.* 22 (2015) 851–859. <https://doi.org/10.1007/s12613-015-1142-7>.
- [310] Y. Chen, M. Cheng, H. Song, S. Zhang, J. Liu, Y. Zhu, Effects of lanthanum addition on microstructure and mechanical properties of as-cast pure copper, *J. Rare Earths* 32 (2014) 1056–1063. [https://doi.org/10.1016/S1002-0721\(14\)60183-6](https://doi.org/10.1016/S1002-0721(14)60183-6).
- [311] M. Lipińska, L. Olejnik, M. Lewandowska, The influence of an ECAP-based deformation process on the microstructure and properties of electrolytic tough pitch copper, *J. Mater. Sci.* 53 (2018) 3862–3875. <https://doi.org/10.1007/s10853-017-1814-y>.
- [312] S. Atefi, M.H. Parsa, D. Ahmadkhaniha, C. Zanella, H.R. Jafarian, A study on microstructure development and mechanical properties of pure copper subjected to severe plastic deformation by the ECAP-Conform process, *J. Mater. Res. Technol.* 21 (2022) 1614–1629. <https://doi.org/10.1016/j.jmrt.2022.09.103>.
- [313] F. Samadpour, A. Siah Sarani, G. Faraji, Microstructure and mechanical properties of pure copper processed via hydrostatic cyclic expansion extrusion, *J. Mech. Sci. Technol.* 39 (2025) 6739–6745. <https://doi.org/10.1007/s12206-025-1020-9>.
- [314] Y. Zheng, J. Niu, Y. Lu, S. Xu, Z. Li, H. Zhao, P. La, High strength and high conductivity pure copper prepared by ECAP-Conform combined with cryogenic rolling and recrystallization annealing, *J. Mater. Res. Technol.* 36 (2025) 3192–3204. <https://doi.org/10.1016/j.jmrt.2025.04.020>.
- [315] X. Sun, M. Ebrahimi, S. Attarilar, The decisive impact of microstructure on the machinability of pure copper, *Arch. Civ. Mech. Eng.* 21 (2021) 136. <https://doi.org/10.1007/s43452-021-00290-8>.
- [316] G. Yang, Z. Li, Y. Yuan, Q. Lei, Microstructure, mechanical properties and electrical conductivity of Cu–0.3Mg–0.05Ce alloy processed by equal channel angular pressing and subsequent annealing, *J. Alloys Compd.* 640 (2015) 347–354. <https://doi.org/10.1016/j.jallcom.2015.03.218>.
- [317] O.F. Higuera-Cobos, J.M. Cabrera, Mechanical, microstructural and electrical evolution of commercially pure copper processed by equal channel angular extrusion, *Mater. Sci. Eng. A* 571 (2013) 103–114. <https://doi.org/10.1016/j.msea.2013.01.076>.
- [318] K. Zhou, Y. Zhao, H. Dong, Q. Mao, S. Jin, M. Feng, R. Zhang, S. Li, J. Liu, Fractal structure and nano-precipitates break comprehensive performance limits of CuCrZr alloys, *Nano Today* 56 (2024) 102234. <https://doi.org/10.1016/j.nantod.2024.102234>.
- [319] R. Kuziak, W. Zalecki, S. Węglarczyk, M. Pietrzyk, New Possibilities of Achieving Ultrafine Grained Microstructure in Metals and Alloys Employing MaxStrain Technology, *Solid State Phenom.* 101–102 (2005) 43–48. <https://doi.org/10.4028/www.scientific.net/SSP.101-102.43>.
- [320] J. Bystrzycki, A. Fraczkiewicz, R. Łyszkowski, M. Mondon, Z. Pakieła, Microstructure and tensile behavior of Fe–16Al-based alloy after severe plastic deformation, *Intermetallics* 18 (2010) 1338–1343. <https://doi.org/10.1016/j.intermet.2010.01.014>.
- [321] P. Macioł, W. Zalecki, R. Kuziak, Results of experimental investigations of tool steel during forming in semi-solid state, *Int. J. Mater. Form.* 3 (2010) 759–762. <https://doi.org/10.1007/s12289-010-0881-2>.
- [322] V. Komarov, I. Khmelevskaya, G. Korpala, R. Kawalla, S. Prokoshkin, Metal Forming Aspects of Cyclic Severe Plastic Deformation of Ti-Ni Shape Memory Alloys Using MaxStrain Device, *Key Eng. Mater.* 746 (2017) 214–218. <https://doi.org/10.4028/www.scientific.net/KEM.746.214>.
- [323] P. Snopiński, T. Tański, W. Borek, Surface Hardening of AlMg<sub>5</sub>Si<sub>2</sub>Mn Alloy through Multi-Axis Compression Using Max Strain, *Solid State Phenom.* 308 (2020) 171–180. <https://doi.org/10.4028/www.scientific.net/SSP.308.171>.
- [324] K. Muszka, D. Zych, P. Lisiecka-Graca, L. Madej, J. Majta, Experimental and Molecular Dynamic Study of Grain Refinement and Dislocation Substructure Evolution in HSLA and IF Steels after Severe Plastic Deformation, *Metals* 10 (2020) 1122. <https://doi.org/10.3390/met10091122>.
- [325] J.B. Renkó, G. Kralics, Comparison of physical and virtual simulation of multi-axial forging processes on EN-AW 6082 aluminium alloy, *Mater. Today Proc.* 45 (2021) 4100–4104. <https://doi.org/10.1016/j.matpr.2020.11.329>.
- [326] A.S. Borisov, A.A. Naumov, A.Yu. Borisova, O.G. Zotov, V.N. Tsemenko, Effect of Severe Plastic Deformation on the Formation of the Nonferrous Metal Alloy Microstructure, *Steel Transl.* 53 (2023) 830–836. <https://doi.org/10.3103/S0967091223100054>.
- [327] P. Dhondapure, S. Nayak, S. Dourandish, M. Jahazi, Influence of deformation path on microstructure evolution during multi-step deformation of a high strength steel: experiments and FE analysis, *Sci. Rep.* 16 (2025) 321. <https://doi.org/10.1038/s41598-025-29713-9>.
- [328] T. Balawender, M. Zwolak, Ł. Bąk, Experimental Analysis of Mechanical Characteristics of KOB0 Extrusion Method, *Arch. Metall. Mater.* (2020) 615–619. <https://doi.org/10.24425/amm.2020.132800>.

- [329] X. Mou, K. Peng, J. Zeng, L.L. Shaw, K.-W. Qian, The influence of the equivalent strain on the microstructure and hardness of H62 brass subjected to multi-cycle constrained groove pressing, *J. Mater. Process. Technol.* 211 (2011) 590–596. <https://doi.org/10.1016/j.jmatprotec.2010.11.013>.
- [330] L. Jiang, H. Fu, H. Zhang, J. Xie, Physical mechanism interpretation of polycrystalline metals' yield strength via a data-driven method: A novel Hall–Petch relationship, *Acta Mater.* 231 (2022) 117868. <https://doi.org/10.1016/j.actamat.2022.117868>.
- [331] T. Guo, D. Qian, D. Huang, K. Li, Y. Gao, Y. Ding, Deformation microstructure and properties control of Cu-0.6Cr alloy in cryo-equal channel angular pressing, *J. Cent. South Univ.* 30 (2023) 2094–2106. <https://doi.org/10.1007/s11771-023-5369-6>.
- [332] W. Wciślik, S. Lipiec, Void-Induced Ductile Fracture of Metals: Experimental Observations, *Materials* 15 (2022) 6473. <https://doi.org/10.3390/ma15186473>.
- [333] L. Yuan, F. Liu, C. Wu, C. Lou, Improving mechanical properties and electrical conductivity of Al-Cu-Mg matrix composites by GNPs and sc additions, *Sci. Rep.* 15 (2025) 2418. <https://doi.org/10.1038/s41598-025-86744-y>.
- [334] Y. Tu, X. Liu, W. Wang, W. Zhang, Q. Feng, Grain boundary evolution and effect on electrical conductivity of Cu–Ti alloys prepared by accumulative roll bonding-diffusion alloying process, *Mater. Charact.* 201 (2023) 112958. <https://doi.org/10.1016/j.matchar.2023.112958>.
- [335] P.J. Withers, C. Bouman, S. Carmignato, V. Cnudde, D. Grimaldi, C.K. Hagen, E. Maire, M. Manley, A. Du Plessis, S.R. Stock, X-ray computed tomography, *Nat. Rev. Methods Primer* 1 (2021) 18. <https://doi.org/10.1038/s43586-021-00015-4>.
- [336] K. Ostrowska, J. Śladek, P. Wołkanowski, I. Dominik, D. Owczarek, M. Nykiel, K. Tomczyk, M. Stoliński, A New Methodology for Selecting CT Scanning Parameters Depending on the Density of Materials, *Materials* 17 (2024) 6172. <https://doi.org/10.3390/ma17246172>.
- [337] D. Gallardo, L.-C. Díaz, F. Zanini, J.A. Albajez, S. Carmignato, J.A. Yagüe-Fabra, On the effect of material density in dimensional evaluations by X-ray computed tomography of metal-polymer multi-material parts, *CIRP J. Manuf. Sci. Technol.* 54 (2024) 1–13. <https://doi.org/10.1016/j.cirpj.2024.08.003>.
- [338] I. Holgado, N. Ortega, J.A. Yagüe-Fabra, S. Plaza, H. Villarraga-Gómez, Metrological evaluation and classification of porosity in metal additive manufacturing using X-ray computed tomography, *Mater. Des.* 254 (2025) 114057. <https://doi.org/10.1016/j.matdes.2025.114057>.
- [339] J. Wilbig, A.E. Wilson-Heid, L. Bernard, J. Baptista, A.-F. Obaton, Comparison of Porosity Analysis Based on X-Ray Computed Tomography on Metal Parts Produced by Additive Manufacturing, *Appl. Sci.* 15 (2025) 9876. <https://doi.org/10.3390/app15189876>.
- [340] E. Avoledo, M. Petruzzi, M. Pelegatti, A. Tognan, F. De Bona, M. Pressacco, R. Toninato, E. Salvati, Defect analysis by computed tomography in metallic materials: Optimisation, uncertainty quantification and classification, *Precis. Eng.* 97 (2026) 235–248. <https://doi.org/10.1016/j.precisioneng.2025.09.008>.
- [341] M. Korobenkova, M. Lebedev, V. Promakhov, A. Narikovich, X-ray Tomographic Method to Study the Internal Structure of a TiNi–TiB<sub>2</sub> Metal Matrix Composite Obtained by Direct Laser Deposition, *Materials* 16 (2023) 1353. <https://doi.org/10.3390/ma16041353>.
- [342] X. Sun, L. Huang, B.G. Xiao, Q. Zhang, J.Q. Li, Y.H. Ding, Q.H. Fang, W. He, H.M. Xie, X-ray computed tomography in metal additive manufacturing: A review on prevention, diagnostic, and prediction of failure, *Thin-Walled Struct.* 207 (2025) 112736. <https://doi.org/10.1016/j.tws.2024.112736>.
- [343] M. Inês Silva, E. Malitckii, T.G. Santos, P. Vilaça, Review of conventional and advanced non-destructive testing techniques for detection and characterization of small-scale defects, *Prog. Mater. Sci.* 138 (2023) 101155. <https://doi.org/10.1016/j.pmatsci.2023.101155>.
- [344] M. Machalski, P. Duda, The influence of burrow-generated pseudobreccia on the preservation of fossil concentrations, *Sci. Rep.* 14 (2024) 18893. <https://doi.org/10.1038/s41598-024-69846-x>.
- [345] V. Shivam, S. Kar, G.K. Bansal, A.K. Chandan, B.K. Sahoo, G.K. Mandal, N.K. Mukhopadhyay, V.C. Srivastava, A novel Fe-rich non-equiatomic medium-entropy alloy with superior mechanical properties, *J. Alloys Compd.* 952 (2023) 170029. <https://doi.org/10.1016/j.jallcom.2023.170029>.
- [346] A.Y. Volkov, B.D. Antonov, E.I. Patrakov, E.G. Volkova, D.A. Komkova, A.A. Kalonov, A.V. Glukhov, Abnormally high strength and low electrical resistivity of the deformed Cu/Mg-composite with a big number of Mg-filaments, *Mater. Des.* 185 (2020) 108276. <https://doi.org/10.1016/j.matdes.2019.108276>.
- [347] A. AlHaza, N. Haneklaus, Diffusion Bonding and Transient Liquid Phase (TLP) Bonding of Type 304 and 316 Austenitic Stainless Steel—A Review of Similar and Dissimilar Material Joints, *Metals* 10 (2020) 613. <https://doi.org/10.3390/met10050613>.

- [348] A. AlHazaa, N. Haneklaus, Z. Almutairi, Impulse Pressure-Assisted Diffusion Bonding (IPADB): Review and Outlook, *Metals* 11 (2021) 323. <https://doi.org/10.3390/met11020323>.
- [349] M. Khedr, A. Hamada, A. Järvenpää, S. Elkatatny, W. Abd-Elaziem, Review on the Solid-State Welding of Steels: Diffusion Bonding and Friction Stir Welding Processes, *Metals* 13 (2022) 54. <https://doi.org/10.3390/met13010054>.
- [350] G. Xin, M. Zhou, K. Jing, H. Hu, Z. Li, Y. Zhang, C. Tian, Y. Sun, B. Tian, X. Li, A.A. Volinsky, J. Zou, Hot deformation behavior and microstructure evolution of the Cu-1.5Ti-(0.5Fe) alloys, *J. Mater. Res. Technol.* 30 (2024) 4961–4972. <https://doi.org/10.1016/j.jmrt.2024.04.208>.
- [351] A. Kozłowska, K. Radwański, A. Grajcar, Identification of Structural Constituents in Advanced Multiphase High-Strength Steels Using Electron Back-Scattered Diffraction, *Symmetry* 16 (2024) 1630. <https://doi.org/10.3390/sym16121630>.
- [352] N.M. Heckman, L. Velasco, A.M. Hodge, Influence of Twin Thickness and Grain Size on the Tensile Behavior of Fully Nanotwinned CuAl Alloys, *Adv. Eng. Mater.* 18 (2016) 918–922. <https://doi.org/10.1002/adem.201500496>.
- [353] J.X. Yan, Z.J. Zhang, H. Yu, K.Q. Li, Q.M. Hu, J.B. Yang, Z.F. Zhang, Effects of pressure on the generalized stacking fault energy and twinning propensity of face-centered cubic metals, *J. Alloys Compd.* 866 (2021) 158869. <https://doi.org/10.1016/j.jallcom.2021.158869>.
- [354] Y.A. Sun, Z.P. Luo, X.Y. Li, K. Lu, Effects of stacking fault energy on deformation induced grain boundary relaxation in nanograined Cu alloys, *Acta Mater.* 239 (2022) 118256. <https://doi.org/10.1016/j.actamat.2022.118256>.
- [355] M.T. Islam, S.R. Broderick, A Data-Driven Framework for Accelerated Modeling of Stacking Fault Energy from Density of States Spectra, *Crystals* 15 (2025) 390. <https://doi.org/10.3390/cryst15050390>.
- [356] W. Junfeng, C. Jinshui, G. Chengjun, Z. Jianbo, X. Xiangpeng, Y. Bin, Effect of heat treatment on low cycle fatigue properties of Cu–Cr–Zr alloy, *Mater. Charact.* 158 (2019) 109940. <https://doi.org/10.1016/j.matchar.2019.109940>.
- [357] F. Zhou, Y. Zhang, L. Lu, K. Song, H. Gao, Effects of a thermal-ultrasonic stress relaxation process on the residual stress, mechanical properties and microstructures of C19400 copper alloy strips, *Mater. Sci. Eng. A* 841 (2022) 143014. <https://doi.org/10.1016/j.msea.2022.143014>.
- [358] D. Wan, A.B. Hagen, L.M. Viespoli, A. Johanson, F. Berto, A. Alvaro, In-situ tensile and fatigue behavior of electrical grade Cu alloy for subsea cables, *Mater. Sci. Eng. A* 835 (2022) 142654. <https://doi.org/10.1016/j.msea.2022.142654>.
- [359] J. Yang, K. Bu, Y. Zhou, K. Song, T. Huang, X. Peng, H. Liu, Y. Du, Microstructure, residual stress, and mechanical properties evolution of a Cu–Fe–P alloy under different conditions, *J. Mater. Res. Technol.* 24 (2023) 7896–7909. <https://doi.org/10.1016/j.jmrt.2023.05.015>.
- [360] X. Yu, Y. Song, C. Wang, K. Gu, L. Zheng, W. Qiu, B. Liu, S. Gong, Z. Li, Effect of Mg content on the microstructure and properties of high strength, high conductivity Cu–Fe–Cr–Si–Mg alloy, *Mater. Sci. Eng. A* 883 (2023) 145510. <https://doi.org/10.1016/j.msea.2023.145510>.
- [361] A. Malik, U. Masood Chaudry, K. Hamad, T.-S. Jun, Microstructure Features and Superplasticity of Extruded, Rolled and SPD-Processed Magnesium Alloys: A Short Review, *Metals* 11 (2021) 1766. <https://doi.org/10.3390/met11111766>.
- [362] M.P. Medeiros, D.R. Lopes, M. Kawasaki, T.G. Langdon, R.B. Figueiredo, An Overview on the Effect of Severe Plastic Deformation on the Performance of Magnesium for Biomedical Applications, *Materials* 16 (2023) 2401. <https://doi.org/10.3390/ma16062401>.
- [363] S.A. Paranjape, T.G. Nikhil, S. Suwas, L.S. Toth, S.V. Kailas, Comparison of microstructure and texture in pure magnesium sheets produced by hot rolling and a new SPD process; the high-pressure compressive reciprocating shear, *Mater. Charact.* 225 (2025) 115209. <https://doi.org/10.1016/j.matchar.2025.115209>.
- [364] K. Wei, R. Hu, D. Yin, L. Xiao, S. Pang, Y. Cao, H. Zhou, Y. Zhao, Y. Zhu, Grain size effect on tensile properties and slip systems of pure magnesium, *Acta Mater.* 206 (2021) 116604. <https://doi.org/10.1016/j.actamat.2020.116604>.
- [365] M. Zhang, L. Liu, S. Liang, J. Li, Evolution in Microstructures and Mechanical Properties of Pure Copper Subjected to Severe Plastic Deformation, *Met. Mater. Int.* 26 (2020) 1585–1595. <https://doi.org/10.1007/s12540-019-00395-z>.
- [366] A. Thomas, G. Fribourg, J.-J. Blandin, P. Lhuissier, R. Dendievel, G. Martin, Effect of the build orientation on mechanical and electrical properties of pure Cu fabricated by E-PBF, *Addit. Manuf.* 48 (2021) 102393. <https://doi.org/10.1016/j.addma.2021.102393>.

- [367] H. Somekawa, T. Mukai, Effect of grain refinement on fracture toughness in extruded pure magnesium, *Scr. Mater.* 53 (2005) 1059–1064. <https://doi.org/10.1016/j.scriptamat.2005.07.001>.
- [368] H. Yamagishi, M. Fukuhara, Degradation behavior of moduli in extruded pure magnesium during low- to giga-scale cyclic tension fatigue, *Acta Mater.* 60 (2012) 4759–4767. <https://doi.org/10.1016/j.actamat.2012.05.027>.
- [369] E28 Committee, Test Methods for Tension Testing of Metallic Materials, (n.d.). [https://doi.org/10.1520/E0008\\_E0008M-25](https://doi.org/10.1520/E0008_E0008M-25).
- [370] M. Asano, M. Yuasa, H. Miyamoto, T. Tanaka, C. Erdogan, T. Yalçinkaya, Potential of High Compressive Ductility of Ultrafine Grained Copper Fabricated by Severe Plastic Deformation, *Metals* 10 (2020) 1503. <https://doi.org/10.3390/met10111503>.
- [371] H. Yu, C. Zhang, C. Liu, Z.X. He, W. Yu, H.Y. Ma, B.A. Jiang, S.H. Park, F.X. Yin, Microstructural evolution and mechanical properties of pure copper by caliber rolling, *Mater. Sci. Eng. A* 947 (2025) 149156. <https://doi.org/10.1016/j.msea.2025.149156>.
- [372] J. Li, W. Xu, X. Wu, H. Ding, K. Xia, Effects of grain size on compressive behaviour in ultrafine grained pure Mg processed by equal channel angular pressing at room temperature, *Mater. Sci. Eng. A* 528 (2011) 5993–5998. <https://doi.org/10.1016/j.msea.2011.04.045>.
- [373] T. Yeo, N. Shigematsu, T. Katori, Dynamically recrystallized grains identified via the application of Gaussian mixture model to EBSD data, *J. Struct. Geol.* 167 (2023) 104800. <https://doi.org/10.1016/j.jsg.2023.104800>.
- [374] S.-S. Rui, Q.-N. Han, X. Wang, S. Li, X. Ma, Y. Su, Z. Cai, D. Du, H.-J. Shi, Correlations between two EBSD-based metrics Kernel Average Misorientation and Image Quality on indicating dislocations of near-failure low alloy steels induced by tensile and cyclic deformations, *Mater. Today Commun.* 27 (2021) 102445. <https://doi.org/10.1016/j.mtcomm.2021.102445>.
- [375] N. Moulin, D. Jeulin, H. Klöcker, Stress concentrations in non-convex elastic particles embedded in a ductile matrix, *Int. J. Eng. Sci.* 47 (2009) 170–191. <https://doi.org/10.1016/j.ijengsci.2008.08.012>.
- [376] Z. Li, N. Limodin, A. Tandjaoui, P. Quaegebeur, J.-F. Witz, D. Balloy, Influence of Fe content on the damage mechanism in A319 aluminum alloy: Tensile tests and digital image correlation, *Eng. Fract. Mech.* 183 (2017) 94–108. <https://doi.org/10.1016/j.engfracmech.2017.05.006>.
- [377] J. Mathew, G. Remy, M.A. Williams, F. Tang, P. Srirangam, Effect of Fe Intermetallics on Microstructure and Properties of Al-7Si Alloys, *JOM* 71 (2019) 4362–4369. <https://doi.org/10.1007/s11837-019-03444-5>.
- [378] D. Xu, C. Zhu, C. Xu, K. Chen, Microstructures and Tensile Fracture Behavior of 2219 Wrought Al–Cu Alloys with Different Impurity of Fe, *Metals* 11 (2021) 174. <https://doi.org/10.3390/met11010174>.
- [379] W. Wciślik, R. Pała, Some Microstructural Aspects of Ductile Fracture of Metals, *Materials* 14 (2021) 4321. <https://doi.org/10.3390/ma14154321>.
- [380] J. Chen, M. Magdy, T. Cao, Y. Hu, C. Cheng, J. Zhao, Analysis of stress state and crack initiation time around hard-brittle particles in nickel-based alloys by interface model, *Mater. Today Commun.* 38 (2024) 108462. <https://doi.org/10.1016/j.mtcomm.2024.108462>.
- [381] C.M. Abreu, I. Feijoo, C. Pérez, Effect of the Annealing Treatment on the Microstructure and Corrosion Resistance of a Cu-Al-Ni Alloy, *Metals* 12 (2022) 1473. <https://doi.org/10.3390/met12091473>.
- [382] S. Li, Y. Huang, L. Cai, H. Peng, J. Yan, Y. Wen, Simultaneously improving memory effect and mechanical properties in Cu-based alloys by  $\alpha$  phase spheroidization and Fe alloying: A CuAlMnFe as an example, *Mater. Sci. Eng. A* 881 (2023) 145396. <https://doi.org/10.1016/j.msea.2023.145396>.
- [383] H. Jiang, Z. Yang, L. Peng, H. Guo, H. Xie, D. Liu, Y. Cao, The thermal instability mechanism and annealed behavior of ultra-high strength Cu-Ni-Sn alloy, *J. Alloys Compd.* 1013 (2025) 178508. <https://doi.org/10.1016/j.jallcom.2025.178508>.
- [384] M. Gouge, P. Michaleris, E. Denlinger, J. Irwin, The Finite Element Method for the Thermo-Mechanical Modeling of Additive Manufacturing Processes, in: *Thermo-Mech. Model. Addit. Manuf.*, Elsevier, 2018: pp. 19–38. <https://doi.org/10.1016/B978-0-12-811820-7.00003-3>.
- [385] O. Matvienko, O. Daneyko, T. Kovalevskaya, A. Khrustalyov, I. Zhukov, A. Vorozhtsov, Investigation of Stresses Induced Due to the Mismatch of the Coefficients of Thermal Expansion of the Matrix and the Strengthening Particle in Aluminum-Based Composites, *Metals* 11 (2021) 279. <https://doi.org/10.3390/met11020279>.
- [386] D. Irmer, C. Yildirim, M. Sennour, V.A. Esin, C. Moussa, Effect of second-phase precipitates on deformation microstructure in AA2024 (Al–Cu–Mg): dislocation substructures and stored energy, *J. Mater. Sci.* 59 (2024) 18978–19002. <https://doi.org/10.1007/s10853-024-10205-6>.

- [387] M. Bacca, D.R. Hayhurst, R.M. McMeeking, Continuous dynamic recrystallization during severe plastic deformation, *Mech. Mater.* 90 (2015) 148–156. <https://doi.org/10.1016/j.mechmat.2015.05.008>.
- [388] T. Sakai, A. Belyakov, R. Kaibyshev, H. Miura, J.J. Jonas, Dynamic and post-dynamic recrystallization under hot, cold and severe plastic deformation conditions, *Prog. Mater. Sci.* 60 (2014) 130–207. <https://doi.org/10.1016/j.pmatsci.2013.09.002>.
- [389] A. Gramlich, C. Helbig, M. Schmidt, W. Hagedorn, A comprehensive design approach to increase the performance of steels under minimal costs and environmental impacts, *Sustain. Mater. Technol.* 41 (2024) e01040. <https://doi.org/10.1016/j.susmat.2024.e01040>.
- [390] E. Olorundaisi, P.A. Olubambi, The prospect and limitation of high entropy alloy as 4th industrial material, *Mater. Today Sustain.* 31 (2025) 101163. <https://doi.org/10.1016/j.mtsust.2025.101163>.
- [391] R. Zhu, X. Long, F. Zhang, Z. Yang, Y. Li, Y. Yang, Y. Wang, Optimization of alloying element types and contents in high strength and high toughness bainitic steels, *J. Mater. Res. Technol.* 34 (2025) 2728–2739. <https://doi.org/10.1016/j.jmrt.2024.12.229>.
- [392] F. Czerwinski, Aluminum alloys for electrical engineering: a review, *J. Mater. Sci.* 59 (2024) 14847–14892. <https://doi.org/10.1007/s10853-024-09890-0>.
- [393] E.P. George, D. Raabe, R.O. Ritchie, High-entropy alloys, *Nat. Rev. Mater.* 4 (2019) 515–534. <https://doi.org/10.1038/s41578-019-0121-4>.
- [394] L. Yan, Y. Chen, F. Liou, Additive manufacturing of functionally graded metallic materials using laser metal deposition, *Addit. Manuf.* 31 (2020) 100901. <https://doi.org/10.1016/j.addma.2019.100901>.
- [395] L. Li, G. Liu, H. Yu, Z. Chen, H. Kang, Z. Chen, E. Guo, J. Jie, T. Wang, A machine learning strategy to achieve strength-conductivity-ductility synergy of high-performance Cu-Ni-Co-Si alloys via rolling and aging process, *J. Mater. Sci. Technol.* 267 (2026) 184–197. <https://doi.org/10.1016/j.jmst.2025.12.040>.

## List of Figures

|                                                                                                                                                                                                                                                                                                           |    |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| <b>Figure 1:</b> Number of publications on high-strength and/or high-conductivity Cu alloys from 2000 to 2025 (data retrieved from the Scopus database).....                                                                                                                                              | 1  |
| <b>Figure 2:</b> Binary phase diagram of Cu-Mg system (redrawn from [41] with permission from Springer) .....                                                                                                                                                                                             | 3  |
| <b>Figure 3:</b> The influence of common alloying elements on the electrical conductivity of Cu (redrawn from [37] with permission from Elsevier).....                                                                                                                                                    | 4  |
| <b>Figure 4:</b> Binary phase diagram of Cu-Ca system (redrawn from [36]).....                                                                                                                                                                                                                            | 4  |
| <b>Figure 5:</b> Binary phase diagrams of Cu-Ni system (redrawn from [144] with permission from Elsevier).....                                                                                                                                                                                            | 13 |
| <b>Figure 6:</b> Simplified schematic illustration of dispersion strengthening .....                                                                                                                                                                                                                      | 15 |
| <b>Figure 7:</b> Simplified schematic illustration of (a) ECAP, (b) HPT, and (c) ARB processes..                                                                                                                                                                                                          | 20 |
| <b>Figure 8:</b> (a) The Gleeble thermomechanical simulator; (b) the MaxStrain module; MaxStrain-processed samples in (c) constrained and (d) unconstrained conditions .....                                                                                                                              | 21 |
| <b>Figure 9:</b> Simplified schematic illustration of various components of the KoBo extrusion equipment.....                                                                                                                                                                                             | 22 |
| <b>Figure 10:</b> (a) Vickers hardness, (b) UTS, and (c) electrical conductivity of Cu-0.29Mg-0.21Ca (wt.%) alloy in different states (redrawn from [37] with permission from Elsevier) .....                                                                                                             | 26 |
| <b>Figure 11:</b> Hardness, electrical conductivity, YS, and EL for Cu-0.43Mg and Cu-0.41Mg-0.06Ca (wt.%) alloys as a function of ECAP passes (redrawn from [42] with permission from Elsevier) .....                                                                                                     | 27 |
| <b>Figure 12:</b> Color-coded contour maps illustrating the hardness distribution across the transverse planes of the Cu-0.41Mg-0.06Ca (wt.%) alloy: (a) prior to the ECAP process, and after (b) 1, (c) 2, (d) 4, (e) 8, and (f) 12 ECAP passes (reprinted from [28] with permission from Elsevier)..... | 28 |
| <b>Figure 13:</b> Variation in (a) microhardness and (b) electrical conductivity of a Cu-0.2Mg (wt.%) alloy at 200 °C and 400 °C vs. the number of ECAP passes (redrawn from [32]).....                                                                                                                   | 29 |
| <b>Figure 14:</b> Schematic representation of the overall workflow employed in Approach 1, presented in a simplified flowchart format .....                                                                                                                                                               | 33 |
| <b>Figure 15:</b> (a) Dimensions and (b) spatial location of minisamples used for performing the tensile test.....                                                                                                                                                                                        | 36 |
| <b>Figure 16:</b> Simplified illustration of the 4-wire Kelvin resistance measurement setup.....                                                                                                                                                                                                          | 37 |
| <b>Figure 17:</b> Macrostructures of as-cast (a) binary and (b) ternary alloys.....                                                                                                                                                                                                                       | 38 |
| <b>Figure 18:</b> Optical microscopy image of the microstructure of the ternary alloy.....                                                                                                                                                                                                                | 38 |

|                                                                                                                                                                                                                                                       |    |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| <b>Figure 19:</b> (a) SEM image of the ternary alloy acquired in backscattered electron (BSE) mode, and the corresponding elemental mapping images for (b) Cu, (c) Mg, and (d) Ca elements ..                                                         | 39 |
| <b>Figure 20:</b> EDS analysis results for (a) area 1 and (b) area 2 marked in Figure 19(a) .....                                                                                                                                                     | 40 |
| <b>Figure 21:</b> DSC thermograms for the binary alloy in (a) heating and (b) cooling modes .....                                                                                                                                                     | 41 |
| <b>Figure 22:</b> DSC thermograms for the ternary alloy in (a) heating and (b) cooling modes .....                                                                                                                                                    | 42 |
| <b>Figure 23:</b> Thermomechanical processing routes used in Approach 1 .....                                                                                                                                                                         | 43 |
| <b>Figure 24:</b> Schematic illustrations of the crystal structures of (a) Cu [297], and (b) Cu <sub>5</sub> Ca [298] unit cells .....                                                                                                                | 44 |
| <b>Figure 25:</b> Optical microscopy microstructures of the ternary alloy (a) before and (b) after application of heat treatment process route (i) .....                                                                                              | 45 |
| <b>Figure 26:</b> SEM-BSE microstructures of the ternary alloy (a) before and (b) after application of heat treatment process route (ii) .....                                                                                                        | 45 |
| <b>Figure 27:</b> Strike pattern used for performing the MaxStrain process in route (i) .....                                                                                                                                                         | 46 |
| <b>Figure 28:</b> Strike pattern used for performing the MaxStrain process in routes (ii) and (iii) ..                                                                                                                                                | 47 |
| <b>Figure 29:</b> Distribution of equivalent strain after 40 passes of MaxStrain processing in transverse planes located at distances of (a) 0 mm, (b) 1 mm, (c) 2 mm, (d) 3 mm, (e) 4 mm, and (f) 5 mm from the center of the deformation zone ..... | 48 |
| <b>Figure 30:</b> Calculated values for the equivalent strain in (a) central longitudinal and (b) central transverse planes after 40 passes of MaxStrain processing .....                                                                             | 49 |
| <b>Figure 31:</b> Simulation results illustrating the distribution of equivalent strain after 20 passes of MaxStrain processing in (a) central transverse, (b) central longitudinal (X–Z), and (c) central longitudinal (X–Y) planes .....            | 50 |
| <b>Figure 32:</b> IPF maps of (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, and (d) TC-90Q-M40 samples .....                                                                                                                                                | 53 |
| <b>Figure 33:</b> GOS maps of (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, and (d) TC-90Q-M40 samples (grains having a GOS value less than 2° are shown in red) .....                                                                                      | 54 |
| <b>Figure 34:</b> (a) SEM and (b) bird's-eye view images TC-M40 sample; (c) SEM and (d) bird's-eye view images TC-90Q-M40 sample .....                                                                                                                | 56 |
| <b>Figure 35:</b> Distribution of misorientation angles (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, and (d) TC-90Q-M40 samples .....                                                                                                                      | 57 |
| <b>Figure 36:</b> IPF maps of the TC-15A-M20 and TC-75Q-M20 samples .....                                                                                                                                                                             | 58 |
| <b>Figure 37:</b> GOS maps of the TC-15A-M20 and TC-75Q-M20 samples .....                                                                                                                                                                             | 59 |
| <b>Figure 38:</b> Distribution pattern of misorientation angles (a) TC-15A-M20, and (b) TC-75Q-M20 specimens .....                                                                                                                                    | 60 |
| <b>Figure 39:</b> SEM-BSE images of Cu <sub>5</sub> Ca intermetallic particles in (a) TC-15A-M20, and (b) TC-75Q-M20 specimens .....                                                                                                                  | 61 |

|                                                                                                                                                                                                     |    |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| <b>Figure 40:</b> Hardness measurement results for (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, (d) TC-90Q-M40, (e) TC-15A-M20, and (f) TC-75Q-M20 samples.....                                          | 63 |
| <b>Figure 41:</b> Tensile test data obtained for samples subjected to 40 passes of MaxStrain processing and their corresponding initial counterparts .....                                          | 65 |
| <b>Figure 42:</b> Tensile test data obtained for samples subjected to 20 passes of MaxStrain processing and their corresponding initial counterparts .....                                          | 68 |
| <b>Figure 43:</b> Broken pieces of (a) BC-M40, (b) BC-90Q-M40, (c) TC-M40, and (d) TC-90Q-M40 samples; fracture surfaces of (e) BC-M40, (f) BC-90Q-M40, (g) TC-M40, and (h) TC-90Q-M40 samples..... | 69 |
| <b>Figure 44:</b> The fractured pieces of (a) TC-15A-M20, and (b) TC-75Q-M20 specimens; fracture surface of (c) TC-15A-M20, and (d) TC-75Q-M20 specimens.....                                       | 71 |
| <b>Figure 45:</b> Electrical conductivity values for the binary and ternary samples (a) before and (b) after the exposure to 40 passes of MaxStrain processing .....                                | 73 |
| <b>Figure 46:</b> Measured electrical conductivity values of the samples prior to and following exposure to 20 passes of MaxStrain processing .....                                                 | 74 |
| <b>Figure 47:</b> UTS versus electrical conductivity values for the investigated samples in Approach 1.....                                                                                         | 75 |
| <b>Figure 48:</b> Simplified flowchart illustrating the overall workflow adopted in Approach 2 ...                                                                                                  | 78 |
| <b>Figure 49:</b> Distribution of equivalent strain in (a) the initial product and (b) the steady-state region of the forward-extruded billet.....                                                  | 81 |
| <b>Figure 50:</b> Distribution of equivalent strain in (a) the initial product and (b) the steady-state region of the KoBo-extruded billet.....                                                     | 82 |
| <b>Figure 51:</b> Distribution of equivalent strain in the central transverse cutting plane of the AM sample .....                                                                                  | 83 |
| <b>Figure 52:</b> Distribution of equivalent strain in the central transverse cutting plane of the FM sample .....                                                                                  | 84 |
| <b>Figure 53:</b> Distribution of equivalent strain in the central transverse cutting plane of the KM sample .....                                                                                  | 85 |
| <b>Figure 54:</b> IPF maps showing the microstructures of (a) AM, (b) FM, and (c) KM samples                                                                                                        | 86 |
| <b>Figure 55:</b> KAM maps of (a) AM, (b) FM, and (c) KM samples.....                                                                                                                               | 87 |
| <b>Figure 56:</b> Distribution of misorientation angles in (a) AM, (b) FM, and (c) KM samples ...                                                                                                   | 88 |
| <b>Figure 57:</b> The hardness profiles for (a) AM, (b) FM, and (c) KM samples.....                                                                                                                 | 90 |
| <b>Figure 58:</b> Data obtained from the tensile test results for all investigated samples in this approach.....                                                                                    | 92 |
| <b>Figure 59:</b> Broken pieces and the corresponding fracture surfaces of (a,d) AM, (b,e) FM, (c,f) KM samples .....                                                                               | 93 |

|                                                                                                                                                                                   |     |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| <b>Figure 60:</b> Measured electrical conductivity values for the investigated samples in this approach.....                                                                      | 95  |
| <b>Figure 61:</b> UTS versus electrical conductivity values for all samples studied in Approaches 1 and 2.....                                                                    | 95  |
| <b>Figure 62:</b> Schematic illustration of the transverse section of pure Cu matrix prior to the KoBo extrusion.....                                                             | 98  |
| <b>Figure 63:</b> Photograph of Mg filaments embedded within a Cu matrix prior to the KoBo extrusion.....                                                                         | 98  |
| <b>Figure 64:</b> SEM image of the macrostructure of the fabricated Cu/Mg composite.....                                                                                          | 102 |
| <b>Figure 65:</b> SEM bird's eye view of the central Mg filament within the Cu matrix in the fabricated Cu/Mg composite in (a) low magnification and (b) high magnification ..... | 102 |
| <b>Figure 66:</b> (a) SEM image and (b) line scan analysis result across the Cu/Mg interface.....                                                                                 | 103 |
| <b>Figure 67:</b> XCT analysis results in (a) top view, (b,c) side view, and (d) 3D view; (e) 3D scan of Mg filaments.....                                                        | 105 |
| <b>Figure 68:</b> (a) IPF, (b) GOS (grain with GOS < 2° are shown in red), (c) and KAM maps, and (d) pole figure of the Cu matrix .....                                           | 107 |
| <b>Figure 69:</b> TEM images of the Cu matrix in the fabricated composite .....                                                                                                   | 108 |
| <b>Figure 70:</b> STEM images of the (a) central and (b) outer Mg filaments within the fabricated composite structure .....                                                       | 109 |
| <b>Figure 71:</b> Hardness measurement results for the Cu matrix and Mg filaments before and after KoBo extrusion .....                                                           | 110 |
| <b>Figure 72:</b> Compressive mechanical response of the fabricated Cu/Mg composite at deformation levels of (a) 61% and (b) 79% compressions .....                               | 111 |
| <b>Figure 73:</b> (a,d) IPF, (b,e) GOS, and (c,f) KAM maps of the Cu matrix following compressive deformation to strain levels of 61% and 79%, respectively.....                  | 112 |

## List of Tables

|                                                                                                      |     |
|------------------------------------------------------------------------------------------------------|-----|
| <b>Table 1:</b> Overview of key physical and mechanical properties of pure Cu ( $\geq 99.9\%$ )..... | 6   |
| <b>Table 2:</b> Summary of key composition, properties and primary applications of Cu alloys..       | 8–9 |
| <b>Table 3:</b> Summary of the key characteristics of Cu-Mg alloys.....                              | 10  |
| <b>Table 4:</b> Elemental quantification by EDS in as-cast binary and ternary alloys.....            | 34  |
| <b>Table 5:</b> Summary of processing history and abbreviated sample names for Approach 1 .....      | 51  |
| <b>Table 6:</b> Processing history and abbreviated sample names for specimens in Approach 2....      | 79  |

## ABSTRACT

This PhD work systematically investigated the effects of Ca addition, heat treatment, extrusion-based processing, and severe plastic deformation (SPD) on the microstructure, mechanical properties, and electrical conductivity of Cu-0.7Mg (wt.%) alloy, as well as the development of a density-reduced Cu/Mg composite structure via KoBo extrusion. The addition of 0.1 wt.% Ca to the Cu-0.7Mg (wt.%) alloy promoted the formation of Cu<sub>5</sub>Ca intermetallic particles, which were uniformly distributed inside grains as well as along grain boundaries, while also transforming the as-cast columnar microstructure of the binary alloy into an equiaxed structure in the ternary alloy. Consequently, the ternary alloy exhibited a 33% increase in hardness, a 13% increase in yield strength (YS), and a 13% increase in ultimate tensile strength (UTS) compared with the binary alloy, accompanied by a 15% reduction in total elongation (EL) and a 13% decrease in electrical conductivity. Subsequent heat treatment consisting of annealing at 650 °C for 90 min followed by water quenching induced partial spheroidization of the Cu<sub>5</sub>Ca particles in the ternary alloy and also enhanced the YS, UTS, and EL of both alloys. SPD processing using the MaxStrain module of the Gleeble thermomechanical simulator resulted in pronounced grain refinement and an increased fraction of high-angle grain boundaries (HAGBs), particularly in the ternary alloy, where the intermetallic particles effectively impeded dislocation motion during deformation. Sequential processing routes involving either forward extrusion at 400 °C or KoBo extrusion at room temperature prior to MaxStrain deformation produced substantially finer grain structures, reducing grain size by 76% and 66%, respectively, compared with MaxStrain processing alone. In the binary alloy, MaxStrain deformation, with or without prior extrusion, increased YS by 251–360% and UTS by 95–169% relative to the annealed condition due to the combined effects of grain refinement and deformation strengthening. Although SPD processing caused minor reductions in electrical conductivity, all processed alloys maintained a favorable balance between strength and conductivity and remained within the range characteristic of high-strength and high-conductivity (HSHC) Cu alloys. In addition, a density-reduced Cu/Mg composite structure (with a density of 7.85 g/cm<sup>3</sup>) composed of a pure Cu matrix reinforced with continuous pure Mg filaments was successfully fabricated using KoBo extrusion. Microstructural analyses confirmed strong mechanical interfacial bonding without detectable interdiffusion, continuous and defect-free Mg filament distribution, dynamic recovery and recrystallization within the Cu matrix, and the formation of an ultrafine-grained (UFG) Mg microstructure with an average grain size of ~354 nm. These microstructural features contributed to hardness increases of approximately 43% in the Cu matrix and 11% in the Mg filaments. Mechanical properties evaluation revealed an early YS value of  $\sim 119 \pm 12$  MPa for the composite structure, which was associated with the onset of plastic deformation of the relatively softer Mg filaments within the Cu matrix. Despite deformation-induced defects that slightly increased electron scattering, the composite maintained a high electrical conductivity of  $52.6 \pm 1.2$  MS/m due to the continuous Cu conduction network. Overall, the results demonstrated that the controlled integration of alloying elements, heat treatment, extrusion-based processing, and SPD constitutes an effective strategy for the development of advanced HSHC Cu-Mg alloys and density-reduced Cu/Mg composite structures with a desirable combination of mechanical properties and electrical conductivity.

**Keywords:** High-strength and high-conductivity copper alloys; Solid solution strengthening; Second-phase hardening; Heat treatment; Microstructure engineering; MaxStrain process; Gleeble thermomechanical simulator; KoBo extrusion; Simulation; Mechanical properties; Electrical conductivity, Composite materials.

## STRESZCZENIE

W niniejszej rozprawie doktorskiej przeprowadzono systematyczne badania wpływu dodatku Ca, obróbki cieplnej, procesów przeróbki plastycznej opartych na wyciskaniu oraz intensywnego odkształcenia plastycznego (SPD) na mikrostrukturę, właściwości mechaniczne i przewodność elektryczną stopu Cu-0,7% mas. Mg, a także opracowanie kompozytowej struktury Cu/Mg o obniżonej gęstości metodą wyciskania KoBo. Dodatek 0,1% mas. Ca do stopu Cu-0,7Mg (% mas.) sprzyjał powstawaniu cząstek międzymetalicznych Cu<sub>5</sub>Ca, równomiernie rozmieszczonych zarówno wewnątrz ziaren, jak i na granicach ziaren, a także powodował transformację kolumnowej mikrostruktury odlewniczej stopu binarnego w strukturę równoosiową w stopie trójskładnikowym. W konsekwencji stop trójskładnikowy wykazał wzrost twardości o 33%, wzrost granicy plastyczności (YS) o 13% oraz wzrost wytrzymałości na rozciąganie (UTS) o 13% w porównaniu ze stopem binarnym, przy jednoczesnym obniżeniu całkowitego wydłużenia (EL) o 15% oraz przewodności elektrycznej o 13%. Następnie zastosowana obróbka cieplna, obejmująca wyżarzanie w temperaturze 650 °C przez 90 min z następującym po nim chłodzeniem w wodzie, wywołała częściową sferoidyzację cząstek Cu<sub>5</sub>Ca w stopie trójskładnikowym oraz poprawę wartości YS, UTS i EL dla obu stopów. Obróbka SPD z wykorzystaniem modułu MaxStrain symulatora termomechanicznego Gleeble doprowadziła do wyraźnego rozdrobnienia ziaren oraz zwiększenia udziału granic ziaren wielokątowych (HAGBs), szczególnie w stopie trójskładnikowym, gdzie cząstki międzymetaliczne skutecznie hamowały ruch dyslokacji podczas odkształcenia. Zastosowane sekwencje przetwarzania obejmujące wyciskanie współbieżne w temperaturze 400 °C lub wyciskanie KoBo w temperaturze pokojowej przed odkształceniem MaxStrain doprowadziły do uzyskania znacznie drobniejszej struktury, zmniejszając średnią wielkość ziaren odpowiednio o 76% i 66% w porównaniu z samą obróbką MaxStrain. W przypadku stopu binarnego odkształcenie MaxStrain, zarówno z wcześniejszym wyciskaniem, jak i bez niego, zwiększało YS o 251–360% oraz UTS o 95–169% w porównaniu ze stanem wyżarzonym, na skutek połączonego efektu rozdrobnienia ziaren i umocnienia odkształceniowego. Pomimo niewielkiego obniżenia przewodności elektrycznej spowodowanego obróbką SPD, wszystkie przetworzone stopy zachowały korzystną równowagę pomiędzy wytrzymałością a przewodnością oraz mieściły się w zakresie charakterystycznym dla stopów Cu o wysokiej wytrzymałości i wysokiej przewodności (HSHC). Ponadto, metodą wyciskania KoBo z powodzeniem wytworzono kompozytową strukturę Cu/Mg o obniżonej gęstości (7,85 g/cm<sup>3</sup>), składającą się z osnowy czystej Cu wzmocnionej ciągłymi włóknami czystego Mg. Analiza mikrostruktury potwierdziła trwałe mechaniczne połączenie międzyfazowe bez widocznej interdyfuzji, ciągle na całej długości i wolne od defektów rozmieszczenie filamentów Mg, dynamiczne odzyskiwanie i rekrytalizację osnowy Cu oraz powstanie ultradroboziarnistej (UFG) mikrostruktury Mg o średniej wielkości ziarna wynoszącej około 354 nm. Cechy mikrostrukturalne przyczyniły się do wzrostu twardości o około 43% w osnowie Cu oraz o 11% w filamentach Mg. Charakterystyka właściwości mechanicznych wykazała wczesną wartość YS na poziomie  $\sim 119 \pm 12$  MPa dla struktury kompozytowej, odpowiadającą początkowi odkształcenia plastycznego stosunkowo bardziej miękkich filamentów Mg w osnowie Cu. Pomimo obecności defektów indukowanych odkształceniem, które nieznacznie zwiększały rozpraszanie elektronów, kompozyt zachował wysoką przewodność elektryczną na poziomie  $52,6 \pm 1,2$  MS/m, dzięki zachowaniu ciągłej struktury Cu jako osnowy. Podsumowując, uzyskane wyniki wskazują, że kontrolowane zastosowanie dodatków stopowych, obróbki cieplnej, procesów przeróbki plastycznej opartych na wyciskaniu oraz SPD stanowi skuteczną strategię opracowywania zaawansowanych stopów Cu-Mg typu HSHC oraz kompozytowych struktur Cu/Mg o obniżonej gęstości, charakteryzujących się pożądanym połączeniem właściwości mechanicznych i przewodności elektrycznej.

**Słowa Kluczowe:** Stopy miedzi o wysokiej wytrzymałości i wysokiej przewodności; Umocnienie roztworowe; Umocnienie przez fazę drugą; Obróbka cieplna; Inżynieria mikrostruktury; Proces MaxStrain; Symulator termomechaniczny Gleeble; Wyciskanie KoBo; Symulacja; Właściwości mechaniczne; Przewodność elektryczna; Materiały kompozytowe.

## Publications

- [1] **A. Kalhor**, K. Rodak, H. Myalska-Głowacka, B. Chmiela, K. Kuglarz, I. Schindler, K. Radwański, M. Kampik, *High-Strength and High-Conductivity Cu-0.7Mg-0.1Ca Alloy Fabricated via Heat Treatment and Severe Plastic Deformation*, *Journal of Mining and Metallurgy, Section B: Metallurgy* **61** (2025) 43–58, DOI: <https://doi.org/10.2298/JMMB241017004K>.
- [2] **A. Kalhor**, K. Rodak, M. Tkocz, B. Chmiela, I. Schindler, Ł. Poloczek, K. Radwański, H. Mirzadeh, M. Kampik, *Influence of Processing History on Microstructure, Mechanical Properties, and Electrical Conductivity of Cu-0.7Mg Alloy*, *Transactions of Nonferrous Metals Society of China* **35** (2025) 1197–1211, DOI: [https://doi.org/10.1016/S1003-6326\(24\)66743-9](https://doi.org/10.1016/S1003-6326(24)66743-9).
- [3] **A. Kalhor**, K. Rodak, M. Tkocz, H. Myalska-Głowacka, I. Schindler, Ł. Poloczek, K. Radwański, H. Mirzadeh, M. Grzenik, K. Kubiczek, M. Kampik, *Tailoring the Microstructure, Mechanical Properties, and Electrical Conductivity of Cu-0.7Mg Alloy via Ca Addition, Heat Treatment, and Severe Plastic Deformation*, *Archives of Civil and Mechanical Engineering* **24** (2024) 71, DOI: <https://doi.org/10.1007/s43452-024-00890-0>.