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REVIEW OF THE THESIS

submitted by Augustine Nana Sekyi Appiah

entitled:

Deformation-Induced Size Effects on the Structure and Mechanical Properties of Heterogenous L-PBF Fabricated AlSi10Mg Alloys

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<u>**Importance of the topic and objectives of the thesis**</u>

Achieving an appropriate balance between strength and ductility is one of the fundamental challenges in materials science and engineering. Engineering materials are typically either ductile (readily undergoing plastic deformation) or mechanically strong (capable of sustaining high loads). An increase in strength is usually accompanied by a loss of ductility. The reason is that the phenomena behind strength, i.e. restriction in dislocation activity, is excluding the ductile behaviour, which requires dislocation motion.

The balance between strength and ductility is of critical importance from a practical perspective, as industry seeks structural materials that are not only easy to process and form but also sufficiently strong to withstand the required loads. Consequently, research groups worldwide are actively addressing this issue, either by designing new materials or by employing advanced thermomechanical treatments. Although the first strategies aimed at enhancing ductility while retaining high strength were reported approximately fifteen years ago, the challenge remains unsolved, and numerous research teams continue to propose novel concepts in pursuit of a solution.

The doctoral dissertation under review contributes to this ongoing discourse. Its principal objective, as articulated by the author, is to elucidate the fundamental deformation mechanisms governing the mechanical properties of heterogenous PBF-LM/M fabricated AlSi10Mg alloy, with the ultimate aim of developing a material that combines high strength with good ductility. In addition, the candidate formulated three specific research goals: (i) tailoring the microstructure of the additively manufactured material through low-temperature heat treatment, (ii) investigating the mechanisms of plastic deformation as a function of the resulting microstructure and deformation mode, and (iii) applying severe plastic deformation (SPD) techniques to obtain a material simultaneously exhibiting high strength and ductility.

The most important results and substantive assessment of the work

The experimental section of the dissertation is preceded by an extensive literature review addressing issues relevant to the subject of the doctoral research. It covers topics such as additive manufacturing technologies, with particular emphasis on the method employed in this work (Laser-Based Powder Bed Fusion), as well as the Al10SiMg alloy produced using this technique; dislocations and dislocation structures formed during plastic deformation; methods of severe plastic deformation; and strengthening mechanisms.

In my view, a doctoral dissertation literature review may omit textbook-level content taught during the early years of undergraduate education, such as the basics of dislocation theory or classical strengthening mechanisms. A literature review should not only summarize what is already known in the area of the dissertation (which is presented quite well here), but also highlight knowledge gaps or inconsistencies that the doctoral candidate intends to address or clarify (which is unfortunately lacking in this case). After reading the literature review, I had the impression that everything in the dissertation's subject area had already been thoroughly explored, and I began to wonder whether there was anything new that could still be added. The literature review discusses structures formed after 3D printing, how they are affected by heat treatment, and how the structure and properties evolve after ECAP processing. The only aspect that seemed novel was the treatment involving twist channel angular pressing (TCAP).

To tackle the stated research problem, the candidate designed an exceptionally comprehensive experimental program. At the technological level, it included 3D printing, low-temperature annealing, and plastic deformation (using compression, ECAP, and TCAP). On the characterization level, it employed a wide range of experimental techniques: optical microscopy, SEM (with EDS and EBSD), TEM, XRD, porosity measurements, hardness testing, and compression tests. I found the choice of compression testing somewhat surprising for evaluating strength and ductility—typically, in this type of study, tensile testing is used, since under tensile loading the problem of ensuring an adequate balance between strength and ductility is most evident.

The first step in the research plan was the selection of the alloy composition from three variants: Al7SiMg, Al10SiMg, and Al12SiMg. The choice of the intermediate composition was justified by the smallest variation in powder particle size, the lowest porosity, and the highest hardness of the alloy. This decision is consistent with the statements made in the Introduction, where the mechanical strength and ductility of the three alloys were analyzed in detail, and the 10% Si alloy was indicated as offering the most favourable balance between strength and ductility. However, this conclusion does not appear to be in agreement with the data presented in Table 1 (p. 13). Furthermore, the dissertation does not provide the precise chemical composition of the powders nor the bulk materials produced from them, nor any information regarding the source of the powders.

The next stage involved tailoring the microstructure of the as-built material (after 3D printing) by means of low-temperature heat treatment, with parameters chosen based on literature data. I particularly value the multiscale approach employed to describe microstructural changes during annealing. It was convincingly shown that the most important changes occur at the nanometre scale, specifically through the fragmentation of the Si nanoparticle network. Nevertheless, significant concerns arise with regard to the microstructural analyses performed at different stages of compression testing. First, the rationale for selecting deformation levels of 5% and 20% for detailed microstructural study is not made clear. Second, it initially seemed that these values referred to plastic strain; however, they in fact correspond to total strain, with 5% lying within the elastic regime. It is surprizing that at this strain level—rather than at 20%—detailed microstructural investigations were conducted, which even more surprisingly revealed pronounced changes at the micrometre grain scale, including a transformation from columnar to equiaxed morphology and a reduction in grain size. My question is how such extensive microstructural reconstruction could occur within the elastic range at room temperature.

For the compression-tested samples, crystallite size and dislocation density were determined using the Williamson–Hall method. I have 2 questions related to these results:

- Why were these parameters not determined for the undeformed sample? Without this reference point, it is impossible to assess whether the dislocation density increased as expected. All reported values fall within the same order of magnitude, whereas plastic deformation is generally associated with an increase in dislocation density by several orders of magnitude.
- Which microstructural features correspond to the "crystallites" identified from diffraction peak broadening?

The following section of the dissertation addresses microstructural modification via ECAP. Samples in three microstructural states were subjected to a single pass, carried out both at room temperature and at elevated temperatures—higher than those employed in the heat treatment. The justification for using such high processing temperatures is unclear, and require explanation. Moreover, the application of only a single pass corresponds to a very limited plastic strain applied. In conventional ECAP practice, multiple passes are required to achieve meaningful results. A particularly interesting and original aspect of the thesis was the use of a modified ECAP method with a twisted channel—TCAP. In my assessment, the entire section devoted to SPD methods (although the actual level of deformation applied barely qualifies as "severe") is the most valuable contribution of the dissertation. It offers a wealth of data on microstructural evolution and mechanical property development. I acknowledge the tremendous effort invested by the doctoral candidate, although it is likely that some parts of the experimental work were carried out with assistance from other team members.

Regrettably, due to vast number of data, the central point of the dissertation, namely achieving high ductility while maintaining high strength, becomes somewhat obscured. It is disappointing that the results were not consolidated, for example in the form of a summary plot presenting strength against ductility for all the samples investigated. Such an analysis would have been essential to demonstrate clearly whether, and which, thermo-mechanical treatments assured an improved combination of strength and ductility. Although one treatment (LTA_300 + 2TCAP) was highlighted as providing the best properties, this conclusion appears debatable. While this sample indeed exhibited very high ductility (50%), it also displayed one of the lowest yield strengths (approximately 400 MPa). It is therefore difficult to argue that the strength–ductility paradox was overcome. Taking the as-built material as a reference, in fact only one sample (LTA_280 + 2TCAP) demonstrated a modest improvement—around 10% higher ductility at comparable strength.

Editorial comments

The dissertation is organized in a classical structure, encompassing the introduction, literature review, research methodology, results, discussion, and conclusions. The text is written with clarity, the sample designations are consistent, and the graphs are presented in a legible manner. The work is further enhanced by high-quality microscopic images, with the EBSD maps and TEM micrographs deserving particular distinction. My only editorial remark concerns formatting: in some cases figure captions are placed on the following page rather than directly beneath the corresponding figures, and some tables are split across two pages. While this does not significantly diminish the quality of the work, it does somewhat hinder readability.

Concluding remarks

In my final assessment, I would like to emphasize that although I have pointed out certain weaknesses of the dissertation (as is the duty of a reviewer), I regard the work as scientifically valuable, addressing current research challenges and uncovering new scientific findings. The doctoral candidate has presented original solutions to the research problems (which is a statutory requirement), proposing a range of thermo-mechanical treatments to achieve the optimal combination of strength and ductility in the Al10SiMg alloy. He has also mastered a wide array of experimental techniques for materials characterization. The dissertation contains many valuable results which, in my opinion, could successfully be published in renouned journals in the field of materials engineering. Unfortunately, within the candidate's publication record directly related to the dissertation, I found only two papers, published in *Symmetry* and *Materials*—both MDPI journals.

Nevertheless, I can state with full conviction that the doctoral dissertation entitled *"Deformation-Induced Size Effects on the Structure and Mechanical Properties of Heterogeneous L-PBF Fabricated AlSi10Mg Alloys"* fulfils all statutory requirements for doctoral thesis, and that Mr. Augustine Nana Sekyi Appiah fully merits the award of the doctoral degree. I therefore recommend that the dissertation be admitted to public defence and to the subsequent stages of the doctoral degree evaluation process.

Prof. M. Lewandowska

/podpis odręczny/

*wyłączenie jawności w zakresie danych osobowych oraz ochrony prywatności osoby fizycznej na podstawie art. 5 ust. 2 ustawy z dnia 6 września 2001 r. o dostępie do informacji publicznej (tj. Dz.U. z 2016 r., poz. 1764)

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