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REVIEW

Doctoral dissertation

Mgr. **Augustine Nana Sekyi Appiah**

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

1. Basis for the review

The basis for the review is a letter from the Chair of the Discipline Council (RDIMa.512.33.2025), Prof. Adam Grajcar, PhD, Eng., dated July 8, 2025, informing me that the Discipline Council of Materials Engineering at the Silesian University of Technology appointed me as a reviewer of the doctoral dissertation of Augustine Nana Sekyi Appiah, MSc, in the discipline of "Materials Engineering." I received the documentation on July 15, 2025.

2. Assessment of the relevance and topicality of the research topic

The subject of the doctoral dissertation concerns the processes of manufacturing aluminum alloy products using the additive method, i.e., selective laser melting (PBF-LB/M), an area that has been the focus of scientific and industrial interest in recent years. The AlSi10Mg aluminum alloy selected by the doctoral student is one of the most commonly used casting alloys (silumins) in additive technologies. In its initial state, this alloy exhibits low plastic properties in relation to its strength properties. In his thesis, the doctoral student conducted research aimed at developing a technology for shaping the mechanical properties of the alloy by developing low-temperature heat treatment parameters, which, in combination with the use of plastic working processes with high deformation values (SPD), was to guarantee the achievement of strength parameters of the AlSi10Mg alloy that are impossible to obtain in conventional heat and plastic processing. Thus, the doctoral thesis responds to real application problems related to the production of structural elements with high strength and, at the same time, adequate plasticity from 4xxx group aluminum alloys. I conclude that the topic is relevant, given the dynamic development of additive technologies and the growing demand for lightweight, strong, and durable products for use in the aerospace and automotive industries. However, it is worth noting that the research topic undertaken by the doctoral student has been the subject of research and numerous studies in the world literature for many years, and therefore the originality of the approach to the objectives of the work should be assessed in the context of intensively developing competitive research activity in the area of shaping the properties of elements made of AlSi10Mg alloy in additive technologies.

In summary, the topic of the dissertation is relevant and well-grounded in contemporary trends in research on materials produced by additive methods.

3. General characteristics and structure of the dissertation

The doctoral dissertation by Mr. Augustine Nana Sekyi Appiah, M.Sc., entitled: ***"Deformation-Induced Size Effects on the Structure and Mechanical Properties of Heterogenous L-PBF Fabricated AlSi10Mg Alloys"*** was completed in the field of engineering and technical sciences in the discipline of materials engineering under the supervision of Supervisor dr. hab. inż. Marcin Adamiak, Prof. PŚ, and Assistant Supervisor dr. inż. Przemysław Snopiński. It was prepared as part of project no. 2021/43/D/ST8/01946 entitled "A new generation of heterogeneous nanostructured Al-Si alloys – research on structure, mechanical properties, and plastic deformation mechanisms" funded by the National Science Centre (NCN).

The thesis is written in English, consists of 207 pages and comprises 6 chapters, presenting 117 figures and 7 tables. The structure of the thesis corresponds to the classic layout – it is divided into a literature and an experimental part. The theoretical part, based on literature analysis (chapters 1–2, pp. 11–44), is preceded by a summary in English and Polish, a list of abbreviations, and a table of contents. In chapter 3, the doctoral student presented a research plan, discussed the methods of selecting and producing materials for research, as well as the conditions and methodology of experimental research. The subsequent parts of the thesis include a presentation of the research results (chapter 4), their analysis and discussion (chapter 5), and final conclusions (chapter 6). At the end of the thesis, there is an extensive bibliography, followed by a list of figures and tables. The bibliography includes 182 references, two of which are co-authored works involving the doctoral student. The vast majority of the cited works are from the last few years (2019–2025), while the older references are recognized theoretical classics, which confirms the timeliness and proper placement of the dissertation in the latest state of knowledge.

The work by Augustine Nana Sekyi Appiah, M.A., is coherent, the chapters form a logical whole, and the title of the dissertation, "***Deformation-Induced Size Effects on the Structure and Mechanical Properties of Heterogeneous L-PBF Fabricated AlSi10Mg Alloys***," fully reflects its content and scope.

4. Detailed description of the doctoral dissertation

Mr. Augustin Nana Sekyi Appiah included a literature review introducing theoretical issues related to the topic of the dissertation in the first two chapters of his work. In Chapter 1 ("1.0 Introduction"), the Author outlined the broad context of the research, pointed to the importance of weight reduction in transport, and the need to search for materials capable of combining high strength with plasticity. As a solution, He presented materials produced by the additive method – selective laser melting in a powder bed (PBF-LB/M), exhibiting a unique, hierarchical microstructure characterized by a fine, continuous network of eutectic silicon (Si) embedded in a ductile α-Al matrix. He then presented issues related to additive technologies, focusing on the PBF-LB/M process. He described its course, quality-determining parameters, as well as typical advantages and disadvantages. He paid particular attention to the microstructure of the AlSi10Mg alloy, pointing out the differences between cast and additively manufactured materials. He presented the characteristic cellular structure with a silicon-rich network at the cell boundaries, discussed the role of geometrically necessary dislocations, the mechanisms of their generation, accumulation, and annihilation, and the influence of heat treatment on the evolution of microstructure and mechanical properties. He devoted a separate section to melt pool boundaries as potential sites for damage initiation and stress concentration, as well as to grain refinement and dynamic recrystallization processes. He concluded with a discussion of intensive plastic deformation techniques used for further modification of the microstructure and an overview of the main strengthening mechanisms in Al-Si alloys.

The doctoral student unusually placed the main objectives of his work in subsection 2.12. In the following subsection 2.12.1, "*Research approach*," he presented the rationale for choosing the topic, and then in subsection 2.12.2, he formulated three specific research objectives. Usually, in the dissertations I evaluate, information about the thesis and research objectives is separated into a separate chapter, which promotes greater clarity and emphasizes the importance of this part of the work. In the case under analysis, the doctoral student, based on a detailed review of the literature, pointed to a significant research gap concerning the limited knowledge of the influence of low-temperature heat treatment and intensive microstructure shaping processes using high strain values (SPD) on the evolution of microstructure and the resulting mechanical properties of Al-Si alloys produced by additive manufacturing methods. These issues are of key importance for shaping the mechanical properties of AlSi10Mg alloys produced by additive manufacturing methods. The dissertation rightly emphasizes that understanding the synergistic interaction of LTA (low-temperature annealing) and SPD processes on the evolution of primary silicon crystals, eutectic silicon precipitates, and the dislocation network generated during deformation is the basis for designing new technological processes that improve both the strength and plasticity of the material.

Mr. Augustine Nana Sekyi Appiah formulated a research hypothesis according to which the use of the sequence "PBF-LB/M proces, then low-temperature heat treatment LTA and plastic working SPD (ECAP/TCAP)" sequence will enable the creation of a microstructure in the tested silumin that ensures high strength while maintaining relatively high plasticity. In order to verify the hypothesis, the doctoral student defined the scope of the research, which included:

a) the characteristics of powders and samples in the *"as-built"* state obtained by the PBF-LB/M method;

b) analysis of the effect of short-term annealing at 280°C for 9 minutes (LTA_280) and long-term annealing at 300°C for 30 minutes (LTA_300) on the morphology of primary silicon crystals and the hardness of the AlSi10Mg alloy;

c) application of ECAP and TCAP processes at different temperatures and numbers of passes, and assessment of their impact on the microstructure and mechanical properties of the AlSi10Mg alloy;

d) analysis of the effect of thermal-plastic treatment on the evolution of dislocation density and porosity;

e) testing of mechanical properties, including microhardness, compression tests, and LUR (loading–unloading–reloading) tests.

The doctoral student also pointed to the need to develop a research methodology and adapt existing analytical techniques (EBSD, TEM, XRD, CT) to the specific characteristics of the alloy under study. He emphasized the difficulties resulting from the heterogeneity of the primary silicon crystals, whose morphology reflects the direction of crystallization along the growth path, leading to the formation of a network structure and microstructural anisotropy typical of materials produced by the PBF-LB/M method. The planned research was aimed not only at confirming the possibility of developing technological parameters for shaping the properties of silumins, which are not available using classical thermo-plastic processing methods (which the PhD student describes as filling a knowledge gap), but also to supplement data on the influence of phase composition on the mechanical properties of the AlSi10Mg alloy produced using PBF-LB/M technology.

In the part of the thesis devoted to the research results, the doctoral student first made a conscious choice of the AlSi10Mg alloy from among the three analyzed (AlSi7Mg, AlSi10Mg, AlSi12Mg). He proved that the alloy with a composition close to eutectic is the most technologically suitable for the PBF-LB/M process, due to its favorable solidification conditions and the finest cellular structure. The analysis of porosity and hardness confirmed that AlSi10Mg is the optimal model material, which provided a solid basis for further research.

In the next stage, the doctoral student characterized the effects of low-temperature annealing (LTA). He showed that annealing at 280°C for 9 minutes causes only partial weakening of the silicon network, while annealing at 300°C for 30 minutes leads to its complete fragmentation and spheroidization. The results clearly prove that LTA_300 treatment is not a simple stress relief process, but a key stage in microstructure conditioning, preparing the material for further plastic working. The doctoral student linked the change in the silicon phase architecture with a marked increase in plasticity (from 35% to 47%), which he associated with a reduction in easy crack propagation paths.

Research into deformation mechanisms is also valuable. The doctoral student showed that a continuous silicon network generates intense strengthening induced by heterogeneous deformation through the formation of geometrically necessary dislocations (GNDs) and high back stress, which results in an increase in the yield strength but promotes premature cracking. After network fragmentation (LTA_300), the doctoral student observed the dominance of free dislocation accumulation (SSD) mechanisms, which weaken the effect of back stress while enabling more uniform plastic flow and increased deformability. He illustrated these phenomena with KAM analysis and dislocation density measurements, which gives the work an in-depth mechanistic dimension.

The main achievement of the dissertation is the research on the application of SPD processes. The doctoral student proved that the effectiveness of ECAP and TCAP is strongly dependent on the initial state of the material. In the case of as-built samples and samples after LTA_280, SPD processes led to brittleness due to secondary tearing of the Si network and the formation of local stress concentrators. In contrast, samples after LTA_300 treatment, in which the brittle phase was stabilized in the form of dispersed particles, underwent favorable grain refinement to an ultra-fine grain size (approx. 400 nm) during SPD, achieving an exceptional combination of strength (Re ≈ 403 MPa) and plasticity (εf ≈ 51%). Thus, the PhD student proposed a coherent technological and material path in which the LTA_300 + TCAP sequence allows the classic strength-plasticity compromise in AlSi10Mg alloys to be overcome.

Also noteworthy is the research on determining the critical temperature for the ECAP process. The doctoral student demonstrated that above 350°C, the process loses its effectiveness, as dynamic recrystallization and silicon particle growth lead to a degradation of mechanical properties reaching 70% compared to the initial state. This finding is of significant practical importance and provides clear technological guidelines for the use of SPD in additively manufactured alloys.

In conclusion, I find that the doctoral dissertation of Mr. Augustine N.S. Appiah's makes a significant contribution to research on materials produced by additive methods, focusing on the technological problem of shaping casting alloys in order to obtain a functional material with increased strength properties while maintaining relatively high plasticity. The research methodology used covered a wide range of microstructure characterization techniques (LOM, SEM, EBSD, TEM, XRD, CT) and mechanical property tests (microhardness, compression test, LUR tests). The results obtained allowed for the unambiguous documentation of the beneficial effects of low-temperature heat treatment and plastic working in the context of improving the mechanical properties of the AlSi10Mg alloy.
A strong point of the dissertation is the comprehensive characterization of the material, covering both the initial state (*as-built*) and subsequent stages of heat treatment and plastic working. In the research part, it is worth emphasizing the large amount of experimental work, reflected in the rich illustrative material in the form of microscopic images, EBSD maps, and hardness measurement results. I also positively assess the doctoral student's attempt at a quantitative analysis of dislocation density and consideration of the Bauschinger effect, which gives the work additional cognitive value.

Taking into account the analysis of the literature, knowledge of research methods, the manner of processing the results, and the conclusions formulated, it should be stated that the doctoral student demonstrated thorough substantive knowledge and the ability to critically evaluate the results obtained. Thus, He has demonstrated a level of knowledge and research competence fully commensurate with a doctoral dissertation, providing a solid foundation for further scientific consideration.

5. Substantive comments and questions for the Author

The doctoral dissertation submitted for review by Mr. Augustine Nana Sekyi Appiah is undoubtedly an original work with significant application potential, which considerably expands the current state of knowledge in the field under study. The research results obtained by the doctoral student, as well as the conclusions formulated on their basis, are based on a wide range of experiments, which testifies to the reliability of the approach and the large amount of research work. However, after reading the thesis, there are also points that require clarification and additional information from the Author. Please refer to the following comments:

1. The thesis uses SEM/EDS analysis of the chemical composition of powders. However, it should be emphasized that this technique is qualitative or semi-quantitative in nature and strongly depends on the location of the analysis. In particular, in the case of metallic powders, the sample size is of key importance – how many particles were tested and whether a statistical analysis of the results was performed. The lack of this information makes it impossible to assess the representativeness of the data obtained. SEM/EDS can be a useful tool for preliminary characterization, but spectrometric methods such as OES (*Optical Emission Spectroscopy*), XRF (*X-ray Fluorescence*) or ICP-OES/MS (*Inductively Coupled Plasma Optical Emission Spectroscopy*). Their use would allow for full verification of the declared chemical composition and would significantly increase the reliability of the experimental part
2. In my opinion, the results of the mechanical property tests are incomplete because they are limited to static compression tests and LUR (*loading–unloading–reloading*) tests. However, there are no results from static tensile tests, which are crucial for an unambiguous assessment of the structural properties of the alloy and would allow comparison with literature data. The results obtained in the compression test do not reflect the behavior of the material in service, especially in aerospace and automotive applications, where tensile loads and cyclic stresses predominate. Furthermore, the lack of fatigue testing or assessment of brittle fracture resistance makes the presented results incomplete and limits their applicability.
3. The presented selection of uniaxial compression levels (5%, 20%, and to failure) makes sense from a cognitive point of view, as it allows the evolution of the microstructure at different stages of deformation to be captured. However, in its current form, the description of the methodology is too general and leaves significant gaps that make it difficult to verify the correctness and comparability of the results. There is a lack of details regarding the conditions under which the compression tests were conducted—the Author does not specify whether the procedure was in accordance with the standard (e.g., ASTM E9), what the deformation rate was, or how the criterion of "destruction" was defined, which makes it impossible to compare the results with the literature. Furthermore, the method of recording strain (extensometer, DIC, piston displacement measurement) has not been specified, so it is not known whether the values obtained are a true reflection of the

deformation of the material or whether they are subject to errors resulting from the susceptibility of the test system.

4. Furthermore, the results of the static compression test are limited only to samples produced in the z-axis orientation, even though the Author himself emphasizes the significant anisotropy of properties in materials obtained by the PBF-LB/M method. In the absence of results from static tensile tests, fatigue tests, and analyses in other directions of orientation, it is premature to draw conclusions about the application potential of the material.
5. The failure criterion in compression tests has not been clearly defined. The tables provide values for "*strain at* failure," but there is no information as to whether they refer to the moment of shear fracture, to the observed change in the shape of the sample (so-called *barreling)* resulting from friction on the front contact surfaces, or to the limit of plastic deformation arbitrarily adopted as the condition for ending the test. This lack of precision limits the possibility of comparing the test results with data from the literature.
6. The analysis of microhardness measurement results is inconsistent because the doctoral student uses different loads (HV0.1 and HV0.3), which makes it difficult to compare the results. The work lacks consistent statistical analyses (e.g., standard deviations, number of samples), which means that the differences between the various treatment variants are not always entirely convincing. Furthermore, the use of HV microhardness measurements for materials with a soft matrix and very hard silicon precipitates or primary intermetallic phases is not appropriate, as the measurement results are highly dependent on the location of the indentation. Depending on whether the indentation hits the matrix or the precipitation, the hardness values obtained are characterized by a large spread, which limits the reliability of this method in the analyzed material. In this case, it would be more appropriate to use Brinell hardness measurements, as this method uses a much higher load and a spherical indenter, which allows the mechanical response of a larger volume of material to be averaged. This reduces the influence of local microstructural heterogeneity (soft matrix versus hard silicon precipitates and intermetallic phases) and makes the results more representative of the entire material.
7. Two variants of low-temperature annealing were used in the study: short-term at 280 °C for 9 minutes (LTA_280) and long-term at 300 °C for 30 minutes (LTA_300). Such a short heat treatment time, especially in the LTA_280 variant, raises serious doubts as to the possibility of modifying the microstructure throughout the entire sample volume and obtaining reliable and repeatable results. The lack of justification for the selection of these parameters is a weak point of the methodology and makes comparison with the literature difficult. It is therefore reasonable to ask whether, after 9 minutes of annealing, significant changes in the microstructure can actually be expected, especially in terms of fragmentation and spheroidization of the silicon network. Furthermore, the designation of a 30-minute annealing as *long-term* should be questioned. On what basis were these heat treatment conditions chosen?
8. The stability of the obtained alloy properties has not been verified, which significantly weakens some of the application conclusions. For materials intended for use in the

aerospace and automotive industries, it is crucial to maintain mechanical properties at elevated temperatures and after long-term use. However, the Author did not perform any aging tests or annealing tests that would allow the durability of the ultra-fine-grained microstructure obtained after TCAP to be assessed. This is a serious limitation, as the instability of the UFG microstructure is one of the main obstacles to its industrial implementation. The declarations about the potential application significance of the results have not been confirmed in this way. The Author repeatedly points to the possibility of using the sequence (AM + LTA + SPD) in the aerospace and automotive industries (e.g., Chapter 6 – *Conclusions)*, but did not present any tests under conditions similar to those in service. The lack of fatigue, corrosion, and high-temperature tests means that the results obtained are purely laboratory-based, and some of the conclusions should be considered overinterpretation.

9. Another weak point of the presentation of the results is the description of the SPD processes (ECAP and TCAP). The Author presents the course of the tests and the conditions of the process, i.e., the temperature, but the analysis of the impact of the process parameters on the material properties is not presented in a coherent manner. There is a lack of tabular summaries that would allow for a clear comparison of the effects of different temperatures and numbers of passes on the microstructure and strength. Furthermore, the observed deterioration of properties in the case of ECAP at high temperatures was attributed to recrystallization phenomena, but there are no microstructural results (e.g., EBSD showing grain growth) to support this conclusion. The discussion at this point is declarative in nature and not based on the results obtained.
10. In addition, substantive shortcomings in the method of analyzing the results should be pointed out. For example, the definition of *"back* stress" adopted by the Author in LUR studies is not entirely justified; the calculations were performed as the average value of the yield strength during unloading and reloading, while the definition accepted in the literature from the theory of kinematic strengthening is half the difference between the plasticity thresholds in opposite directions. The adopted calculation methodology deviates from the classical definition and may result in values burdened with systematic error. Similarly, the Author refers to the Bauschinger effect, even though no reversal of the load direction was applied, which makes the conclusions methodologically unjustified. In other places, there are incorrect formulations, such as the statement about "an increase in the yield strength at maximum deformation," where in fact it is an increase in yield stress. The dissertation also shows inconsistency in the use of terminology for testing the mechanical properties of materials. The Author uses the term "yield strength" even for large deformation values, where it would be correct to refer to *flow stress* (e.g., page 125). Such wording can be misleading in the interpretation of results.
11. The interpretation of the results of the Williamson–Hall method is simplified. The Author attributes the obtained dislocation density values to the SSD fraction, while in this method it is not possible to distinguish between SSD and GND. In addition, the analysis of KAM-based dislocation density results is mentioned in the text, but no numerical data is

presented to confirm the SSD and GND mechanisms referred to by the author in the discussion.

The above comments should be treated as a constructive contribution to further discussion with the doctoral student and as a starting point for possible additions, clarifications, or further elaboration of certain aspects of the dissertation. They do not in any way undermine the substantive value of the presented work, do not affect its essential content, nor do they change my positive assessment of the overall research effort and the results obtained. These comments are of a supporting nature and are intended to help the further scientific development of Mr. Augustine Nana Sekyi Appiah.

6. Final conclusion

To sum up my opinion I conclude that the doctoral dissertation submitted for review by **Mr. Augustine Nana Sekyi Appiah**, entitled *"Deformation-induced size effects on the structure and mechanical properties of Heterogenous L-PBF fabricated AlSi10Mg Alloys"* is a valuable study that makes a significant contribution to the development of knowledge on the relationship between additive manufacturing technologies, heat and plastic treatment processes, and the mechanical properties of Al-Si-Mg aluminum alloys. The results obtained are not only cognitive but also practical, as they indicate directions for further optimization of lightweight structural materials with a favorable combination of strength and plasticity.

The Author of the dissertation demonstrated a very good knowledge of the issues under study, the ability to select modern research methods and techniques, and the ability to independently and critically analyze and creatively interpret the results obtained. The work expands the current state of knowledge in the field of materials engineering and makes a notable contribution to the development of this discipline.

On this basis, I conclude that the doctoral dissertation meets the requirements for this type of work, as specified in Article 187 of the Act of July 20, 2018, Law on Higher Education and Science (i.e., Journal of Laws 2024, item 1571). I therefore request the High Council of the Scientific Discipline of Materials Engineering at the Silesian University of Technology to accept the dissertation, continue with the further stages of the doctoral procedure, and admit **Mr. Augustine Nana Sekyi Appiah** to the public defense.

Prof. G. Mrówka_Nowotnik

/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)