FACULTY
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Reviewer's report on the dissertation entitled "Diffraction study of deformation process for selected group of grains and different phases in polycrystalline materials" by Przemysław Andrzej KOT prepared to meet of the requirements of the PhD. degree at AGH University of Science and Technology, Krakow

Prague, August 12, 2022

Despite the rapid development of materials science in the last several decades, there are many open problems that still wait on comprehensive explanation. Answering these issues is of key importance from both a scientific and technological point of view. The study of deformation processes in lightweight alloys (Al, Mg) is one of these problems.
The PhD thesis of Przemysław Andrzej Kot is focused on the methodology of stress measurement using the time-of-flight neutron diffraction technique in lightweight alloys and composites.
The topic of the thesis is up to date, since the investigated phenomena are not fully understood yet. In contrast to modern trends, when the analytical, fact-based thinking is replaced by weird computer calculations, the author has chosen a scientific approach and provided a fundamental experimental work. Simply said, his work is the victory of the mind over the computer power.

The author builds his arguments in eight well-crafted chapters. After a short introduction dealing with the reasoning of the research topic selection, in Chapters 2 and 3 the author exhaustively describes various aspects of the elastic and plastic behavior of metallic materials and the corresponding theoretical models for their characterization.
Chapter 4 summarizes the details on the investigated materials and familiarizes the reader with the experimental methods and data processing used. The applied experimental methods are well chosen; they reflect the current demand of the scientific community for characterization of stress development in materials. In addition to scientific qualities, this chapter also has a high educational value.
It can be concluded that the background, existing work in the area of the topic of the thesis, investigation methods, results to date, and discussion are presented consistently with the results obtained in the thesis. The state-of-the-art research in the field of the stress measurement by diffraction methods is covered comprehensively. To these chapters I have the following question:

- It is not clear from the text what was the criterion for stopping the deformation for diffraction measurement. Were the points chosen at previously specified force or elongation values?

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In Chapter 5, the author describes the stress evolution in the Al/SiC particle reinforced metal matrix composite (MMC) during heat treatment and mechanical loading. The novelty of this work is ensured with careful analysis of the diffraction data combined with implementation of the crystalline group method (CGM). This approach provides new insights into the physics underlying the microstructural evolution of particle-reinforced MMCs. I find particularly valuable that Przemysław extended the original elasto-plastic self-consistent (EPSC) model with terms taking into account thermo-mechanical effects. The determination of phase stresses also belongs to the significant achievements of the work. I would like to ask the following questions:

- In Fig. 5.15, above 550 MPa, a significant relaxation of the stress values for SiC can be observed, which is not predicted by the model. What is the physical background for this behavior?
- Did you compare, how the magnitude of the load transfer from the Al matrix to the SiC particles agrees with the shear-lag model of Cox [Cox, H.L. The Elasticity and Strength of paper and other fibrous materials (1952) British Journal of Applied Physics, 3 (3), pp. 72-79], which says that the amount of load transferred to the particles is equal to $\Delta\sigma_{LT} = \sigma_m 0.5f$, where σ_m is the yield stress in the matrix and f is the volume fraction of the particles?

Chapter 6 contains the crucial part of the thesis, namely the diffraction study of grain stresses and activation of twin- and slip systems in AZ31 magnesium alloys. Przemysław has chosen a combination of experimental and modeling techniques. As I have already written in the introduction, his approach first experimentally determines the critical resolved shear stresses (CRSS) values of the particular slip- or twin systems, and only *after* this step performing the EPSC modeling is the only correct way, how to get reasonable results. Unfortunately, the literature is full of works where the fitting parameters are plucked out of the air.
The work nicely present, how the activation of the deformation mechanisms depends on the orientation with respect to the loading direction. I find valuable that the author confirms that the threshold assumption works better for the description of the twinning evolution.
I am interested in the opinion of the author on the following issues:

- The CRSS value for the tensile twinning system determined by the author (~50 MPa) is significantly higher than that in the literature (5-10 MPa – Chapuis, Driver Acta Mater., 2013, Kelly, Hosford Trans AIME, 1968 – both for pure Mg single crystals). Of course, part of the increase can be ascribed to the hardening effect of the solutes and grain boundaries. Is it possible that your experimental results overestimate the twinning CRSS for the reason that a relatively large volume has to be twinned for producing detectable change in

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diffraction pattern? The acoustic emission measurements detect first signals from twinning also in the range of 5-10 MPa, almost independently on the magnesium alloys studied.

- Did you take into account the effect of the detwinning caused by relaxation during the acquisition of the diffraction pattern? Could you present force-time deformation curves, which show the extent of the relaxation?
- Do you have any assumption of the relative population of the grain orientations in your sample? In particular, the ratio of the A-B grains would be interesting.
- Could you present, how the evolution of RSS for A grains looks like for the RD compression experiments? The reason for this request is that I personally think that the disagreement between the experiment and the model for A grains is given by the fact that in A grains usually several twin variants nucleates at the same time (J. Čapek et al., Acta Mater. 2017). Their interaction significantly influences the evolution of internal stress. This effect is not included in continuous nor in the threshold assumption. Therefore, I think that RSS-based explanation for different evolution of twinning in A- and B-grains, respectively, written on page 141 is not fully correct.
- Is it possible that the discrepancy between the estimation of continuous and threshold models and the experimental data on the twin volume is given by the autocatalytic manner of twinning? In strongly textured AZ31 alloy, the twinning develops in bands (Hazeli et al., Scripta Mater. 2013), where twins in a grain triggers twinning in neighboring grains.

The most important findings of the thesis are listed at the end of all chapters. According to my opinion, all the conclusions are new scientific results, which significantly extend current knowledge in this field.

The results are well disseminated in six peer-reviewed publications (it is a very impressive record) and are well received in the scientific community. I particularly find very valuable Przemysław's contribution to papers in Materials & Design and Materials Science and Engineering A, which belong to the leading journals in his research field.

The manuscript is well organized and focused, with a high standard of presentation in good English and carefully selected illustrations. The thesis undoubtedly demonstrates that the candidate is highly capable of conducting independent scientific research in either the academy or industry.

In summary, the dissertation by Przemysław Andrzej Kot is a highly appropriate and intellectually demanding work with well-defined aims identified within a well

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understood conceptual framework based on an extensive understanding of the literature. Methodology and data collection are thorough, comprehensive, and innovative in many experimental aspects. High-quality results, insightful interpretations, and detailed discussion exhibit excellent higher-level cognitive skills. The analysis is rigorous, leading to sound conclusions.

In view of this, the dissertation deserves the grade ***with honors*** without even a shade of a reservation, and I do recommend to the committee that the thesis be accepted for a further promotion and defense.

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