Review Report on PhD Thesis

Faculty: Central European Institute of Technology
Brno University of Technology in Brno

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Student: Ing. Štěpán Gamanov

Doctoral study program: Advanced Materials and Nanosciences

Supervisor: prof. RNDr. Antonín Dlouhý, CSc.

Reviewer: prof. RNDr. Miloš Janeček, CSc.

PhD thesis title: Plasticity of compositionally complex alloys characterized by advanced electron microscopy techniques

Topicality of doctoral thesis:

As compared to standard transmission electron microscopy (TEM) observations, conventional scanning electron microscopy (SEM) techniques, including electron back-scatter diffraction (EBSD), suffer from limited resolution. This particularly applies to the characterization of local misorientations associated with crystal lattice defects, e.g. dislocations or small coherent particles, which introduce angular lattice rotations below 1°. While the quantitative knowledge of these microstructural characteristics is of key importance to understand their impact on the macroscopic properties of materials, the standard EBSD measurements, which may provide statistically relevant information, cannot deliver the required data due to the mentioned angular resolution limit exceeding 1°. In his work, the author tackled these issues by developing and advancing EBSD data post-processing routines like Rotation Vector Baseline, Half Quadratic Filtering and Artificial Degradation of Spatial Resolution, which resulted in the improvement of the angular resolution by almost TWO ORDERS of magnitude. At the same time, the author addressed a question related to the optimum selection of the scanning step size with respect to the microstructural state of the material. All these steps led to the complex upgrade of the method, which is currently referred to as the high-resolution EBSD (HR-EBSD).

Meeting the goals set:

In the thesis, the applicant defined five primary and five secondary objectives addressing microstructural evolution during high temperature deformation of compositionally complex alloys and an advancement of

the conventional EBSD technique to the HR-EBSD level. In my opinion, ALL the objectives were fully achieved, yielding results of a high scientific standard and with apparent publication potential.

Problem solving and dissertation results:

In the thesis, the new HR-EBSD system has been applied to several long-standing material problems, e.g. the evolution of dislocation substructure during creep in High and Medium Entropy Alloys, the development of lattice rotations between gamma matrix and gamma' cuboidal particles subjected to high temperature loadings and quantitative assessment of grain boundary slides during creep, etc. In all these cases, the new HR-EBSD technique yielded unique microstructural data that provided a basis for a deeper understanding of how microstructural processes govern the macroscopic response of these advanced alloys to mechanical loadings. In the thesis, the author presents a rather unique combination of careful high-level creep testing, systematic microstructural characterization, and a numerical assessment of experimental data. All these aspects clearly demonstrate that this study provides an important contribution to the field of compositionally complex alloy research.

Importance for practice or development of the discipline:

After all years of conventional EBSD work, the results presented in the thesis clearly demonstrate that the limits of the technique can be extended far beyond the angular resolution of 1°, challenging the value of 0.01°. This has opened new horizons in the applicability of HR-EBSD systems, which can be currently employed for a microstructural characterization of numerous persisting problems in materials research and technology. I am deeply convinced that the HR-EBSD system will bring new insight into the microstructural mechanisms that govern deformation processes at different length scales.

Formal adjustment of the thesis and language level:

The thesis volume covers 146 pages, including 86 figures and 157 references to publications, all highly relevant. The volume is distributed meaningfully between a general introductory part, which, in Chapter 1, summarizes necessary basic knowledge and the state-of-the-art. Chapters 2-6 represent the main body of the thesis. Chapter 2 clearly formulates the objectives of the study, Chapter 3 describes the investigated materials, experimental methods, procedures used to elaborate experimental data, and numerical modelling approaches that helped to pin down processes governing the microstructural development. High-quality experimental data acquired in all three investigated areas are summarized and discussed in Chapter 4, and their relevance is underlined by a detailed and involved general discussion presented in Chapter 5. The overall conclusions are clearly formulated in the Chapter 6 which, according to my opinion, documents an outstanding standard of the research and results presented in the thesis.

Questions and comments:

1) Creep data acquired at 1073 K for the CoCrFeMnNi alloy and presented in Figure 36, exhibit the stress exponent of 3.1, which, according to Equation 12, may suggest that subgrains have been fully formed during the first 5% of the accumulated creep strain. However, the results of the careful HR-EBSD study shown in Figure 75a clearly suggest that almost no subgrains were observed at this deformation stage. What is the explanation for this discrepancy?
2) Are there any EDS data about the nature of the surface oxide layer shown e.g. in Figure 39b?
3) Since there are no values on the x-axis of the plot shown in Figure 53, what would be a practical (quantitative) suggestion for the HR-EBSD unexperienced user concerning the scanning step size which could be applied in the data acquisition?

Conclusion:

The thesis, its format and elaboration clearly document that the author is ready for his own independent scientific career. The results presented are of an outstanding scientific standard and considerably progress the field of microstructural development in compositionally complex alloys subjected to high temperature loadings. I do recommend the commission to accept the thesis and evaluate it as “Excellent” (summa/magna cum laude) and undertake further steps in the graduation procedure. Provided the defence progresses satisfactorily, I suggest to confer Mr. Stepan Gamanov the PhD degree with the mark “Excellent” (summa cum laude).

In Prague, 17 July, 2025

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prof. RNDr. Miloš Janeček, CSc.

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