UNIVERSITY OF ŽILINA
Faculty of Mechanical Engineering

Department of Materials Engineering

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Reviewer report on the doctoral thesis

Name of the thesis: Microstructural investigations of novel high entropy alloys

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Reviewer: prof. Ing. František Nový, Ph.D.

The doctoral thesis presents results of experimental investigations of high entropy alloys (HEAs) with bcc structure. Several HEAs consisting of refractory metals Hf, Nb, Ta, Ti, V, Zr were prepared by arc melting. The microstructure and phase composition of HEA samples with various compositions was characterized. As a next step the five element HEA with composition HfNbTaTiZr was subjected to severe plastic deformation employing high pressure torsion processing technique. Application of severe plastic deformation resulted in a strong grain refinement and formation of ultra-fine grained structure. The development of the microstructure during high pressure torsion processing was studied and is described in details in the thesis. The ultra-fine grained HfNbTaTiZr alloy exhibits improved mechanical properties, in particular the high yield strength. This result proved that application of severe plastic deformation is suitable method for improvement of mechanical properties of HEAs. Finally lattice distortions in HEAs were investigated using positron annihilation spectroscopy. This can be regarded as a novel approach for study of the lattice distortion phenomenon which seems to play the key role in HEAs.

The topic of the thesis is highly relevant due to the used experimental material, the implementation of the methodology and the achieved results.

Author of the thesis uses adequately available technologies and methods to determine the observed parameters, and thus it can be concluded that the used methodology was chosen appropriately in view of the objectives of the work.

The methodology of the experiments is described sufficiently and the individual methods are described clearly. From a professional point of view, the thesis is very well conceived and its experimental part contains original results.

The doctoral student fully fulfilled the set goals of the thesis.

The doctoral thesis is well and carefully written and provides a good description of results of obtained by various experimental techniques. The thesis has a logical structure and is divided into 6 main chapters providing (1) introduction to HEA materials; (2) description of characterization methods; (3) description of sample preparation; (4) microstructure characterization of prepared HEAs; (5) results of grain refinement of HfNbTaTiZr alloy by severe plastic deformation; (6) investigations of lattice distortions in HEAs. Finally, the main results are summarized in conclusions. The results of the thesis have already been published in 7 papers in impacted journals included as attachments to the thesis. In addition, Tomáš Vlasák is a co-author of 8 papers on different topics.

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Although the thesis was written carefully it still contains some minor mistakes or misprints, e.g.:

- on page 20: "... depth in the order of tens of hundreds microns" the correct form should be "... depth in the order of tens or hundreds microns";
- on page 21: "This photon serve the purpose of start signal..." it probably means "This photon serves as start signal...";
- on page 46: "low mobilitiy" it should read "low mobility";
- on page 52: "segregation of V at ground boundaries" it should be "segregation of V at grain boundaries.

I have the following questions to the thesis:

1. HfNbTaTiZr alloy annealed at 1000^{o}C and slowly cooled contains precipitates at grain boundaries shown in Fig. 4.11. It is written in the corresponding text that these precipitates are particles of Hf, Ti, Zr rich hcp phase. How we know it?
   Moreover, if particles rich in Hf, Ti, Zr were formed than there should be also some regions depleted in Hf, Ti, Zr. Are there such regions?

2. What is the meaning of the curve plotted by blue solid line in Figure 5.4 on page 56?

3. Fig. 6.2. shows that the bulk positron lifetime increases with the misfit parameter *d*. The data were obtained for HEAs with the bcc structure. Can one expect similar dependence also for HEAs with the fcc structure, e.g. for 3-d element HEA based on the Cantor alloy?

In summary, Tomáš Vlasák has clearly shown his research abilities in the field of solid state physics and capability of independent scientific work. His doctoral thesis provides original and valuable results and demonstrates creative thinking and skilful performance. I recommend his thesis for acceptance and Tomáš Vlasák to get a Ph.D. degree after the successful defence.

Žilina, December 19th, 2022

prof. Ing. František Nový, Ph.D.

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