



Reviewer's report on a Ph.D. thesis

STUDIUM SLITIN TITANU S VYUŽITÍM NEUTRONOVÉ DIFRAKCE

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The thesis reports on experimental characterization and material properties of pure titanium (grade 2) processed by continuous ECAP (CONFORM-ECAP) and rotational swaging. While this topic is quite narrow from the material point of view, it reports on a quite broad range of experimental characterization techniques (from neutron-diffraction experiments to microhardness measurements) that enabled the candidate to achieve a quite complex knowledge of the material. The reported research is indubitably topical – the grain refinement and thermomechanical processing of Ti is nowadays an intensively studied field, and the results presented in the thesis bring a quite valuable addition to the current state-of-art.

The thesis is logically organized, consisting of three main parts: a theoretical introduction comprising mainly a literature survey (Introduction and Chapter 1), the description of the used experiments and material processing methods (Chapter 2), and the summary of the results and their discussion (Chapters 3 and 4). These three parts are complemented by a brief but comprehensive conclusion.

The first part is prepared with dignity and proves that the candidate is well familiar with the mechanical properties and deformation mechanism in Ti and Ti-based alloys, as well as with the main principles of grain refinement via severe plastic deformation and the ECAP method in particular. A short subsection is also devoted to the theoretical background for the analysis of residual stresses. This last subsection is a bit unfortunate, as it, compared with the rest of Chapter 1, gives very few literature references only. This subsection would serve better as a part of Chapter 2. Otherwise, the literature survey is clear and sufficiently comprehensive.

The second part brings an overview of the used characterization and processing methods, giving a brief theoretical introduction to each of them, and then a detailed description of the used experimental



arrangement. From this part I appreciate a lot the Section 2.4 (Material conditions and experimental parameters) that brings all experimental details necessary for presenting and discussing the results in the following chapters.

Chapter 4 summarizes the experimental results and discusses them with respect to the processing conditions and the deformation mechanisms of titanium. This chapter is the most valuable part of the thesis, bringing several novel findings and enabling a deep insight into the problem. For me, the most interesting was the observation of a clear correlation between Von Mises stress maps and microhardness results (Figure 4.32). Also, Figure 4.42 (and the discussion therearound) clearly demonstrates how the twinning is gradually suppressed by increasing the number of C-ECAP passes, which makes a perfect sense with respect to the microstructure evolution. This result also shows the power of in-situ ND measurements, enabling a clear detection of strains at which the twinning is triggered and at which a saturation appears.

Regarding the results reported in Chapter 4, I have the following three comments, which I would like the candidate to address during his defense:

1. For showing the heterogeneity of the residual strains, the shifts of the (10-11) peaks were evaluated. Why did you choose this particular peak? From elastic constants of pure Ti, one would expect the basal plane to have the most pronounced elastic strains. Was that because of the specific texture?
2. When evaluating the grains size distribution (Figures 4.6, 4.10. etc.), there appears to be an artificial linear trend for larger grains, arising probably because there was always just one grain of that size in the evaluated micrograph. Especially for “C1 Top” in Figure 4.6, the larger grains following this trend seem to be comparable in their total area with the refined grains. But there might be even larger grains than 30 μm^2 . Hence, is the claim that the average grain size was around 1 μm^2 justified?
3. The density of dislocations was evaluated just for C-ECAPed samples (Figure 4.24), but not for the material after rotary swaging. Do you think that the forging may lead to more a more uniform distribution of the dislocations as it is mainly the surface that is formed in shear?



To summarize, the thesis reports on a topical research and brings valuable results. The candidate already proved his ability to present his research at the international level, both in form of contributions at conferences and by paper publications (a paper covering partly the topic of the thesis and with the candidate being the first author was recently published in *Metals*). In addition to this the presented thesis proves his capability of independent and creative scientific work.

My only reservation regarding the thesis is concerned with its formal shortcomings. There are several issues in the figures, for example the vertical axes in the grain size graphs have incorrect units (should be (1) instead of (%)), or the colorbars with Figures 2.2 and 2.4 are flawed. The same holds for the grammar and misprints in the text, that would definitely require an additional proof-reading. These formal problems do not, however, lower the validity and importance of the obtained results, they just have a negative impact on my overall impression from the thesis. Despite of this, I find that **the thesis fulfills all legal requirements (§ 47 sect. 4) for Ph.D. dissertations, and I recommend the thesis to be advanced for a defense and the candidate to be awarded the degree.**

In Prague, July 1, 2021

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Hanuš Seiner