UNICAMP

Faculdade de Engenharia Mecânica
Universidade Estadual de Campinas

August 27, 2023

Review Report on Doctoral Thesis

Institution: Faculty of Mathematics and Physics – Charles University in Prague
Candidate: Dalibor Preisler
Supervisor: doc, PhDr. RNDr. Josef Stráský, PhD
Doctoral Thesis Title: Beta-Ti alloys for medical applications

This is a doctoral thesis related to the project, preparation, and characterization of β titanium alloys with potential applications as orthopedic biomaterials. In my opinion, the present study is quite extensive, involving the processing of a significant number of samples, which resulted in a large volume of relevant outcomes. Analyzing these results proved to be a challenging task. It is an excellent research effort, but some aspects may be reviewed, as I outline below.

1. Literature review – Ti alloys for medical applications
The literature review covers several crucial topics related to β titanium alloys. However, some of the addressed issues are not thoroughly examined in depth. Some suggestion to improve the text:
Pg. 5
It would be interesting to differentiate the martensite lines (Ms and Mf) between α’ and α” phases on the phase diagram shown in Figure 1.

Pg. 7
“The ω nanoparticles usually do not have a clear boundary with the surrounding β matrix, it is rather a continuous change of the level of collapse of the {111} planes. Their structure is hexagonal (P6/mmc), but not close-packed as in the case of α phase (P63/mmc).”
In terms of atomic packing factor, how do you compare β and ω phases?

Given the recent O' phase discovery, it would be interesting to incorporate a schematic diagram illustrating its formation from the β phase. For further reference, please consult the following papers:
Y. Zheng et al., Scripta Materialia 116 (2016) 49-52.
Y. Zheng et al., Scripta Materialia 116 (2016) 131-134.

Pg. 10
When dealing with precipitation strengthening, it would be interesting to describe the α phase precipitation mechanisms (classical and non-classical precipitations). For further reference, please consult the following papers:

S. Nag et al., Acta Materialia 57 (2009) 2136-2147.
S. Nag et al., Acta Materialia 60 (2012) 6247-6256.

Pg. 11
It would be interesting to discuss the effects of oxygen on the ωiso precipitation. Please consult the following paper:
M. Niinomi et al., Scripta Materialia 123 (2016) 144-148.

2. Aims of the thesis
When assessing the objectives of this study, it becomes clear that it not only meets the criteria for addressing a relevant scientific question but also showcases originality and international competitiveness.

3. Experimental methods
Despite containing relevant information about the experimental aspects of this study, this chapter is somewhat unclear. It departs from the conventional sequence by not following a logical order, such as starting with materials preparation or synthesis, followed by materials processing, material characterization, etc. Instead, it commences with descriptions of characterization methods. Consider reordering this chapter for better coherence.

Pg. 18
Unexpectedly, the text introduces layered samples. However, no prior information has been provided about layered materials. It is essential to include details about layered materials before delving into their preparation and characterization.

Pg. 23
Results (Figure 9) are not supposed to be presented in the Experimental Methods chapter.

In the Literature Review section, there is a discussion on the design methodologies for metastable β titanium alloys. However, when it comes to selecting the experimental compositions to be investigated, there is no clear application evident of the various methods described in this study.

4. Industrial processing of metastable β-Ti alloy for hip implant manufacturing
This chapter introduces relevant scientific findings that hold the potential for practical technological applications. Nonetheless, the way it is currently presented disrupts the sequential flow of the work. It might be more appropriate to incorporate this chapter as an appendix within the current document.

5. Metastable Ti alloys with lower β phase stability
This chapter showcases relevant scientific results. However, there remain certain questions that need to be addressed.

Pg. 39
How β transus temperature was estimated?

Pg. 43

All XRD investigations were carried out using transmission geometry and Mo radiation, rather than employing Bragg-Brentano geometry and Cu radiation. A more comprehensive discussion of this matter would undoubtedly offer valuable insights. How were the Ti alloy powders prepared?

Figure 26 illustrates XRD patterns corresponding to various compositions. Several of these patterns exhibit the athermal ω phase. Detecting the athermal ω phase using conventional Bragg-Brentano geometry is highly challenging. Additionally, Zr is expected to inhibit its precipitation. A discussion on this phase precipitation would add an interesting dimension to this work.

Furthermore, the XRD pattern of Ti-20Nb-7Zr-0.7O reveals pronounced athermal ω diffraction peaks, along with a set of weak peaks attributed to orthorhombic martensite. In certain alloys, a potential competition between these two phases could arise. Delving into a discussion about this competition would be a valuable addition.

Pg. 44
Precipitation of O' phase in Ti-26Nb-7Zr-6Ta-0.7O is a very interesting finding, demanding a more thorough and elaborate discussion.

Pg. 46
The Vickers hardness results should be compared with the XRD results presented in Figure 26, which would offer an in-depth analysis.

Pg. 47
The tensile curves depicted in Figure 29 exhibit a remarkably sharp elastic/plastic transition, attributed to the presence of oxygen. Additionally, the yield stress exceeding 1000 MPa is an interesting outcome. Have the tensile tests been replicated for verification?

In β Ti alloys, there is a general tendency for hardness and elastic modulus to exhibit parallel patterns, attributable to the phase within the microstructure. This holds true for the majority of the studied alloys, with the exception of the 17 wt.% Nb alloy. A more in-depth discussion of this observation would be interesting.

Pg. 51
Figure 31 illustrates a qualitative phase diagram corresponding to the investigated experimental alloys. This diagram was formulated using the experimental findings from distinct alloys incorporating varying compositions of Nb, Zr, Ta, and particularly, oxygen. I believe that this phase diagram constitutes a significant contribution of this study.

Pg. 53
An interesting comparison between the mechanical responses of the experimental alloys investigated in this study and findings from existing literature is shown in Figure 32. This illustration accentuates the significant contribution that this research makes to the field of titanium alloys utilized as load-bearing biomaterials.

6. High-throughput characterization of layered Ti-Nb based alloys

The study reported in this chapter is closely connected with the preceding one, although its content appears to stand somewhat independent from the overall thesis. Notably, the chapter begins by reiterating the experimental description. Also, Fe was added to the samples. The primary objective of the research discussed in this chapter revolves around producing and characterizing layered samples, which can be viewed as functionally graded materials. Please consider reorganizing this chapter to enhance its overall coherence.

Final Questions:

What is the expected effect of oxygen addition on α precipitation during aging?
What is the effect of O’ phase precipitation on mechanical behavior?

Final conclusion on this thesis:

In my opinion, this thesis not only meets the criteria for addressing a significant scientific inquiry but also shows originality and international competitiveness. The content of this study is highly relevant. It represents an exceptional research endeavor. All of my comments and questions should be regarded as suggestions aimed at enhancing the quality of the work. From my viewpoint, this thesis fulfills the requirements for obtaining a Doctoral degree and it is ready to be defended.