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Thesis Changes Log

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PhD Program: Mathematics and Mechanics

Title of Thesis: Tailoring the functional properties of NiTi shape memory alloy by high-resolution laser powder bed fusion

Supervisor: Associate Professor Igor Shishkovsky

The thesis document includes the following changes in answer to the external review process.

Reply to comments of Jury Members

I'm grateful to my defense jury committee for their valuable comments and questions that allowed me to improve the thesis. The answers to all comments are following. Please note that in the answers, the numbering of figures, tables, sections and pages corresponds to the final version of the dissertation.

Professor Clément Fortin		
1	Comment	There are a number of grammatical and sentence structural problems in the thesis. Articles are sometimes missing or used improperly, such as: Page 40: The executive files were prepared Magics software (Materialise, Belgium) build processor.
	Answer	Thank you for this remark. The thesis was double-checked with appropriate corrections of the sentences' structure and general English grammar. The sentence (Page 37) was revised as follows: "The data files for Trumpf 1000 installation were prepared with Materialise Magics slicing software (Materialise, Belgium)."
2	Comment	Page 73: Thus, the heat conduction for investigated temperature range carried out by electrons
	Answer	The sentence (Page 81) was revised as follows: "Thus, heat transfer through free electrons is considered as the dominant mechanism for the studied temperature range."
3	Comment	Page 77: As far as common ratio is less than one for any physical powder…
	Answer	The sentence (Page 82) was revised as follows: "For any metallic powder, we assume that $f = \frac{\rho_{powder}}{\rho_{solid}} < 1$. Thus, the geometric series converges to an absolute value since the common ratio is less than 1."
4	Comment	Page 97: Results of thesis can be subdivided into four main bodies of knowledge. First part involves experimental study of single track based manufacturing via LPBF, second body include investigations of NiTi thermophysical properties, the

		third related to simulation of the NiTi melt pool during LPBF, and the fourth focused on the particular application of the technology for manufacturing of endodontic files.
	Answer	The first passage of conclusions (Page 106) was revised as follows: "In the thesis, the possibilities of manufacturing micro-objects from NiTi shape memory alloy via high-resolution Laser Powder Bed Fusion (LPBF) were studied. It was demonstrated that the functional properties in the as-built state are sensitive to the process conditions. The mechanisms of functional properties altering were studied by mesoscale simulation of the NiTi melt pool during the LPBF process. The results of the thesis can be divided into three main parts. The first part involves the experimental study of single track-based manufacturing of NiTi via LPBF. The second part includes the simulation of the melt pool during LPBF. The third part focuses on the particular application of the technology for manufacturing of NiTi endodontic files."
5	Comment	There are many cases like these. I suggest carrying out a very careful review of the text.
	Answer	Thank you for the recommendations. The text of thesis was carefully reviewed.

Associate Professor Ivan Sergeichev		
1	Comment	I would recommend adding an explanation of process parameters optimization for endodontic files in Chapter 5. It is obvious that this procedure was made on the basis of all previous chapters (2-4) devoted to experiments and calculations, but there is no generalization of this new knowledge.
	Answer	Thank you for the recommendation. A broad description of the process optimization was added in Section 5.1. Additionally, a process map in the laser power – scanning speed coordinates (Figure 46) was added for clarity. The diagram summarizes the results of Sections 2.4, 2.5.1, and 2.6 which include the experimental data on the thin wall thicknesses, mechanical tests, and temperatures of phase transformation, respectively.
2	Comment	According to the results of mechanical tests, more significant stress/strain characteristics can be determined. In particular, the values of superelastic recovery strains and recovery ratios. This would deepen the analysis of mechanical behavior.
	Answer	Thank you for the advice. Additional characteristics (recoverable strain, irrecoverable strain, recovery ratio) of the mechanical response were calculated and added to Table 5. A discussion on the change in the recovery ratio after consolidation of the material with different process conditions was added on Page 66.
3	Comment	Figure 41 depicts calculated temperature dependences of partial Gibbs free energy, activity coefficient, and activity for whole range of NiTi compositions. These diagrams can be presented as appendix as far as it is preliminary result before the actual calculation of evaporation dynamics.
	Answer	Thank you for the advice, the diagrams were transferred to Appendix A as they present preliminary results.

Associate Professor Alex Volinsky		
1	Comment	The change in the critical temperatures of the martensitic transformation should be illustrated as a function of the linear energy density.
	Answer	Thank you for the comment. An additional diagram with dependence of the start temperature of martensite transformation on the linear energy density was added in Figure 27 with corresponding discussion on Page 68.
2	Comment	The assessment of the change in the amount of B19′-martensite from the integral intensity of the X-ray lines should be carried out together with the assessment of the change in B2-austenite.
	Answer	The assessment of the change in the phase composition was performed both for the B2 and B19′ phases with an assumption of the two-phase composition of the consolidated material. Consequently, the histogram in Figure 29 represents the calculated result of the ratio between the integral intensity of B19′ peaks to all peaks taken into account. For clarity, an additional explanation of the diagram was added on Page 71: "It should be noted that all peaks (both austenitic and martensitic) from the X-ray diagram (Figure 28) were used for the calculation. The results of the volume fraction evaluation are presented with a histogram in Figure 29. The obtained values depict the ratio between the integral intensity of peaks corresponding to B19′ and all peaks taken into account."
3	Comment	Section 2.8 dedicated to demonstration of high-resolution LPBF 4D printing concept. A sample was printed with various transformation temperatures what was proved experimentally. However, Figures 28-29 can be corrected to better depicts the correlation of each part of the sample with its regime.
	Answer	Thank you for the comment. Figure 30 and Figure 31 were revised to better show the regimes of each part of the sample.
4	Comment	Is it possible to carry out mesoscale simulation discussed in Chapter 4 to achieve the geometry of Self-Adjusting Files that are referred in Chapter 5? Results can have practical application as far as it was shown that files have requirements of geometry and mechanical properties. Such approach also may have additional outcomes from the simulation of the melt pool during LPBF.
	Answer	Thank you for that valuable advice. Additional Section 5.4 was added in Chapter 5. The section is dedicated to the simulation of the layer-wise consolidation for SAF via high-resolution LPBF. Coordinates of generated vectors for laser movement were extracted directly from the data file created for the Trumpf TruPrint 1000 by slicing software. For the simulation, 250 layers from the upper part of the SAF were selected as they include a representative part of the whole file. The calculations had been running for 317 hours (~ 13 days), when the average time required for the simulation of a single layer was 76 minutes. The integrated geometry is shown in Figure 52 for layers 1250, 1350, and 1484.

Professor Igor Yadroitsev		
1	Comment	I recommend revising the list of abbreviations, and symbols as far as not all are involved in the present version.
	Answer	Thank you for recommendation. The text was carefully reviewed, and the list was supplemented with all missing abbreviations. The symbols used in the equations were also examined and added to the places where they were utilized.
2	Comment	In my opinion the introductive part can be expanded in terms of problems during laser fusion of intermetallic materials and existing applications of NiTi (including the concept of files for endodontics).
	Answer	Thank you for the advice. Chapter 1 related to the introduction part was expanded. Section 1.2.4 was added, which discusses existing works, problems, and trends related to LPBF manufacturing of micro-objects from different materials, including intermetallics. Laser cutting and laser powder bed fusion were compared in terms of manufacturing NiTi micro-devices such as stents or files. The concept of endodontic files was reviewed in Chapter 5 (Page 97).
3	Comment	There are no studies of the microstructure of the obtained NiTi samples. Knowledge about the features of the microstructure (grain/subgrain size) and crystallographic texture could refine the results obtained on the relationship between L-PBF regimes and functional properties.
	Answer	Thank you for that remark. An additional part dedicated to investigation of microstructure was added (Section 3.3.2): "All thin-walled samples demonstrate an elongated grain structure along the building direction (marked with BD and arrow). Observed texture and grain morphology is explained by the limited heat sink for the prescribed type of laser-based manufacturing. For the thin-walled part, heat sink has a single direction for heat transfer which governing the crystallization. The boundaries of solidified melt pools have different morphologies depending on the energy input. In the case of low LED (Figure 22a), the melt pool boundaries are straight and perpendicular to BD. Such morphology appeared during crystallization of a relatively small volume of liquid and short lifetime of the melt pool accompanied by low wetting condition. For the P100V850 regime (Figure 22b), melt pool shape corresponding to conventional conductive mode of melting. Finally, for high LED (Figure 22c) the morphology of the melt pool is represented with keyhole shape."

Assistant Professor Stanislav Evlashin		
1	Comment	The primary critique of the dissertation pertains to its structure. I suggest adding a section that describes methods for obtaining and examining samples.
	Answer	Thank you for the suggestion. Chapter 2 dedicated to materials and methodology was added. The chapter covers the methodology used in the present research. The chapter is divided onto three parts: 1) raw powder analysis and manufacturing of samples, 2) characterization methods applied onto obtained samples, and 3) physical model used for the numerical study of the LPBF process. First two parts are related to experimental work when the last corresponding to the description of simulation procedures.
2	Comment	I propose moving the description of calculations from the introduction to the methods section and Section 4 to the methods section as well.

	Answer	All equations utilized for calculations were transferred to Section 2.3 of Chapter 2. The description of methods used for the thermophysical properties measurements required for mesoscale simulations were moved to Section 2.2 of Chapter 2.
3	Comment	In Figure 16, please increase the size of the letters in "Inclined angle."
	Answer	Thanks for the recommendation, Figure 16 was revised correspondingly.
4	Comment	Optical microscopy (OM) of cross-sections for thin walls consolidated with the regimes listed in Table 4 is presented in Figure 16. It is necessary to include dimensions for the thin walls and specify the distance from the substrate where the sections were made.
	Answer	Thank you for the advice. Added information: All samples had a width of 5 mm and a height of 6 mm (Page 54). The samples were analyzed over the entire height with a panoramic tool of Thixomet software. Figure 16 shows a representative 2 mm long section from the central part of thin walls (Page 55). Additionally, Figure 15 was completely reworked to clearly show the selected geometry for printing of thin-walled samples.
5	Comment	Remove the numbering for section 6.
	Answer	The numbering was removed from the conclusions section.