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Jury Member Report – Doctor of Philosophy thesis.

Name of Candidate: Konstantin Makarenko

PhD Program: Mathematics and Mechanics

Title of Thesis: Microstructural, mechanical, and thermal properties evaluation of functionally graded Fe-Cu structures after direct energy deposition

Supervisor: Associate Professor Igor Shishkovsky

Name of the Reviewer:

I confirm the absence of any conflict of interest (Alternatively, Reviewer can formulate a possible conflict)	**Date: 21-09-2023**

The purpose of this report is to obtain an independent review from the members of PhD defense Jury before the thesis defense. The members of PhD defense Jury are asked to submit signed copy of the report at least 30 days prior the thesis defense. The Reviewers are asked to bring a copy of the completed report to the thesis defense and to discuss the contents of each report with each other before the thesis defense.

If the reviewers have any queries about the thesis which they wish to raise in advance, please contact the Chair of the Jury.

Reviewer's Report

The research topic is relevant and of great practical interest. The author has conducted extensive research, which has been included in several scientific articles. Published articles and participation in conferences satisfy Skoltech's requirements for thesis defense. However, the dissertation is still in a preliminary stage and requires additional edits before submission. All images are created in different styles, and the graphs are poorly designed and difficult to interpret. Additionally, the author frequently utilizes data from instruments without replotting them. All methods relevance to the conducted research. Obtained results relevance. The results are published in international journal and satisfy the Skoltech requirements.

Below, you'll find some useful notes and comments:

I suggest revising the chapter layout as follows:

1. Begin by describing Functionally Graded Materials.

2. Discuss their applications.
3. Explain the methods for production FGMs. Present the disadvantages and advantages of certain methods.
4. Clarify the rationale for choosing Directed Energy Deposition (DED).
5. I propose including a section specifically dedicated to the features of printing using DED, not only in relation to FGMs.

Following this, introduce a dedicated "Methods" section where you describe all equipment, materials, sample preparation, etc. Avoid duplicating this information in each subsequent chapter.

Correct the keywords. Remove words related to equipment and methods such as: “digital image correlation (DIC), specific heat capacity, differential scanning calorimetry, transitional zone, cracking, InssTek)” Keywords should focus on properties rather than specific equipment and methods.

Add a comparison of the physical properties of steel and bronze, including their expansion coefficients, mechanical properties, lattice types and parameters, and chemical compositions.

Why two sections? 3.6 Interim conclusion и 3.5 Discussion ?

Replace "Table 3" with "Table 4" and include the composition of steel.

The description of the printer in section 4 appears unnecessary. The preparation and analysis methods should be relocated to the third section, "3.1 Materials, Methods, and Equipment."

«The results of SEM of specimens of group 7 proved» It is not clear why the seventh group of samples was chosen?

«see the results of the XRD analysis in Figure 9» it is EDX

Why was a different abbreviation is used?

C61800+SS and somewhere aluminum bronze? Same with C61800+SS (1:1 wt.%), C61800+SS (50 wt.% – 50 wt.%)

It would be a good idea to present sample porosity data if further claims are made that porosity affects mechanical performance.

Figure 8 shows an SEM image and states that these are dendrites. Why hasn't an EDX analysis been conducted to confirm this phase? In Figure 9, there are only spherical phase dropouts. In my opinion, the author often confuses the absence of material mixing with dendrites. Some inclusions are not dendrites. First, iron crystallizes, followed by copper, resulting in such

structures. Iron should begin to crystallize with an Fe-rich BCC phase at around 1300 ℃ and end with the formation of the Cu-rich FCC phase at approximately 1050 ℃.

Why haven't XRD patterns for clean materials been provided? How can we compare the results? Where is the Cu3Al phase? Why was Group 7 chosen?

«It could be ε-Cu, which could be responsible for the existence for a γ-Fe instead of α-Fe after solidification in this ternary system». As far as I understood this structure has the needle

structure. Please check Cu3Al.

Is black area in Figure 17 Cu? Did you analyse the phase composition or EDX composition of black part?

“For instance, size of dendritic elements depends on crystallization rate” Is it possible to estimate cooling rate and temperature gradient using the analytical solutions? Please explain the reason of this theoretical estimation.

In figure Figure 15 the pores have not spherical shapes. Can it be the problem of mechanical polishing? Also, desirable to make the EDX analysis. Subcellular analysis also desirable.

Move this part to section 3.
The materials and methods of making Fe-Cu structures were the same as it was discussed in the chapters 3 and 4. Specimens for tensile tests were prepared via electrical discharge machining using Mitsubishi-MV-1200R (Mitsubishi Electric Europe B.V., Ratingen, Germany). The resulting tensile test specimen shape was chosen according to [156-164]. Tensile tests were performed under the terms and conditions of ASTM E8/E8M-16a [116] at the rate of 2.7 mm/min using INSTRON 5969 dual column testing system and an INSTRON Bluehill® Universal materials testing software (Norwood, Massachusetts, United States), later the results were analyzed via Vic-3D DIC package……

“The specimens of groups 2-4 and 6 (from 0.114 to 0.145) showed the highest values of the strain-to-fracture” What does mean from 0.114 to 0.145?

Why are the results of mechanical tests of pure alloys not given?

It is also interesting to see the porosity results after ultrasound. Evaluate somehow the homogeneity of mixing, the size of the melt pool, etc., and present the comparative results in a table.

I didn’t see the specific heat capacity for aluminum bronze.

“Figure 32. CLTE of groups 1–8” – provide the temperature range. In which direction the measurements were done? The parameters should be difference for different direction. The same for Fig. 33.

At the beginning of Chapter 8, you can make a small goal of why this needed to be done. Now it looks like “And we also did this”, why for what?
Provisional Recommendation
☐ *I recommend that the candidate should defend the thesis by means of a formal thesis defense*
☒ *I recommend that the candidate should defend the thesis by means of a formal thesis defense only after appropriate changes would be introduced in candidate’s thesis according to the recommendations of the present report*
☐ *The thesis is not acceptable and I recommend that the candidate be exempt from the formal thesis defense*