Direct Energy Deposition of Cu-Fe System Functionally Graded Structures

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Abstract. The paper demonstrates the results of microstructure, microhardness and elasticity analysis of the functionally graded (FG) specimens with multilayer structure created of stainless steel and aluminium bronze powder materials via direct energy deposition (DED) laser technology. Increase of microhardness (up to 266 HV) and Young’s modulus of elasticity (up to 43.2 GPa) along with growth of the dendritic crystals in the gradient structures are observed. The results of numerical simulation demonstrate stress distribution in FG Cu-Fe system structure with a sharp interface. The results of the research can be used for 3D-printing of the aerospace industry details created from two kinds of material with rather different thermomechanical properties.

1. Introduction. Theoretical part, scholarly importance and problem statement
A problem of functionally graded materials (FGM) synthesis (via inter alia methods of rapid prototyping) was first time investigated near the end of the XX – beginning of the XXI centuries [1–3]. Classification of FGM by factor of volume (thin FGM and bulk FGM) [4] is the most common and obvious among others. The first group – thin FGM – are combinations of several layers, segments or multimaterial coatings deposited on a flat or curved surface; FGM of the second group are complete tridimensional gradient parts, which are more complex and resource-consuming in manufacturing than FGM of the first group [4]. A direct energy deposition (DED) technology [5–7] is convenient, processable and efficacious way to produce bulk FGM with required gradient of physical and chemical characteristics [8–11]. One of the first mentions of usage of DED for FGM synthesis is 23 years old [12]. The common techniques of FGM synthesis are a straight joining, a method of a gradient transition and a method of an intermediate segment [8]. A straight joining is a simplest conjunction method with a sharp transition between two pure initial metals. A method of a gradient transition supposes producing of interjacent layers with incrementally variable concentration of original materials between a pure metal A and a pure metal B. A method of an intermediate section is similar to the previous one, but is notable for using of a segment of the third pure material deposited amongst first and second ones. Advantage of this technique is exception of forming of unnecessary phases like intermetallics, which can easily appear in the method of a gradient transition [8].

There are several important groups of conjoining metallic materials in FG structures, which have the highest significance in mechanical engineering (including aerospace, milling and nuclear industries), medicine, food industry and so on.
The first group is Ni-based FG superalloys (such as Diamalloy 1005 [13]) which are combinations of Ni, Cr and Al. These superalloys are commonly used for tools working in elevated temperatures like airfoils of turbines [14]. Such intermetallic phases as NiAl and Ni3Al, which have good oxidation resistance in intense heat conditions, even under influence of hot flows of various gases, find their application as protective coverings in aerospace and power mechanical engineering, including components of gas turbine units and engines for missiles of diverse purposes. The advantages of these materials are their moderate density, unique mechanical characteristics under high temperatures, chemical and erosion resistance within a wide temperature range [13].

The second group is Ti-based FG superalloys (Co-V-Ta-Ti, Co-Ni-Al-Ti, Nb-Ti, Ti-B-Cu, Ti-Al-V-C, Ti-W-C and so on), Ti & TiO2 [15], and aluminides of titanium. They are also widely used in aerospace and power industries, above all in high temperature conditions [10, 16]. The area of application of these FGM is larger in comparison with pure Ti alloys. The advantages of Ti-based superalloys are higher rigidity, hardness, thermal stability, and heat resistance [17, 18]. The results of investigation tests performed with TiB2-Cu FGM in conditions similar to missile operation with thermal shock influence demonstrated absence of brittle failure or cracking [19]. Ti alloys-based FG structures, as well as technically pure titanium, due to their biocompatibility also find their application in endoprosthetics [20].

The third group is aluminium-based FGM such as AlSi40 and Al [21] where particles of Si serve as hard reinforcements of an inhomogeneous Al-based structure. The resulted material has enhanced mechanical and chemical resistance properties in comparison with initial components [21].

The fourth important group is Fe-Al system (such as Fe3Al and SS 316L) FG alloys. These compounds have many special characteristics [22]: corrosion, creep and heat resistance, significant mechanical strength in high temperature conditions [23–26]. Manufacturing of these FGM is not so expensive partly owing to reasonable cost of substrates [27–29].

The fifth group is CoCrMo multimaterial alloys, which play an important role in biomedicine. They are used, in particular, in producing of implants for TKR and THR (total knee and total hip replacement) [30].

The sixth group is FG system of Ni-Cr-B-Si and steel such as SS 316L [31]. Ni-Cr-B-Si alloys are widely used in corrosive and high temperature conditions. These FGM have many different solid inclusions and are applicable for hardfacing [32].

The seventh group is Ti-Fe system FGM [33, 34]. They combine low density, high mechanical strength and heat resistance of Ti with specific properties of steel (such as processability and lower price) [33].

The eighth group we should mention is Ni-Cu (including Inconel-Cu) and Fe-Cu (including steel-bronze) FG systems [35, 36, 4]. Combining of two aerospace alloys such as, for example, GRCop-84 and Inconel 718 allow to enhance thermophysical properties of a resulted bimetallic structure [35]. Copper allow combining good oxidation resistance in aggressive atmospheres (such as alkaline and salt) with high values of heat and temperature conductivity coefficients [36]. Nickel is a common component of high temperature alloys. Perfect notch toughness, mechanical strength, and corrosion resistance in elevated temperature conditions characterize them. High temperature strength of Ni and heat conductivity of Cu provide usage of these FGM in extreme temperature conditions [36]. FGM sintered using tool steel H13 and copper can be used for purposes of casting industry as materials for injection moulding tools due to high mechanical strength, wear resistance and thermal conductivity [4]. The stainless steel and Cu combination find its application for producing of food processing, steam turbine and power nuclear plants, electronic components and so on because of mutually supportive characteristics, such as electrical and heat conductivity of copper along with good corrosion resistance and manufacturability of stainless steel [37].
FGM of the eighth group are widely used in manufacturing of space industry parts due to the mentioned physical and exploitation properties of copper and advantages of nickel alloys and stainless steel. The first steps in research of FGM usage for space industry were performed 18 years ago [19]. There is a description of FGM application for producing of thermal barrier materials for space shuttles, creating of air and gas vanes, thrust chambers, piston tops, nosetips, and so on. A radial laser deposition additive technology [38] allows producing axially symmetric gradients, such as metallic parts for spaceship elements created of carbonaceous filaments. Such gradient parts can be fabricated radially from the center of a sample to the outside.

Our research is devoted to synthesis of FG structures associated with the eighth group. The key problem is analysis of the reasons of cracking occurring in FGM created from aluminium bronze and stainless steel via DED, investigation of microstructure, microhardness and elasticity of the specimens and performing of numerical simulation of the stress-strain state forming in nonequilibrium conditions.

2. Materials and equipment
Deposition process was performed with stainless steel AISI 316L and aluminium bronze with 10% content of aluminium via the InnStek MX-1000 machine in the DMT (direct metal tooling) mode. Laser source was an IPG Photonics 1 kW ytterbium doped fiber laser (1064 nm). Focus of a beam was on 1 mm level under the surface of the specimens. Average laser radiation power was equal to 308 W (450 W maximal) for stainless steel and to 500 W (750 W maximal) for aluminium bronze. Power of laser radiation was chosen in accordance with the bronze infrared radiation absorption coefficient which was lower than the same one of stainless steel. Velocity of laser source was equal to 0.85 m/min, powder rate – 3.5 g/min, cooling time between layers – 5 s. We used Ar as a shielding and feeding gas. There was no surface preheat in the experiments. Configuration of single layer tracks formation is shown in the figure 1. Width of a single track was equal to 800 μm. Covering of the tracks was equal to 300 μm.

![Figure 1. Configuration of the tracks of a single layer (left picture – odd layers, right picture – even).](image)

Microstructure research was performed via an optical microscope Carl Zeiss and a scanning electronic microscope Quattro SEM. All samples were previously treated with the etch (the etchant - H₂SO₄ with etanol and CuCl₂, etch time – 5…7 s). Microhardness mechanical tests and stiffness measurements were performed in automatic mode via Nanovea mechanical hardness tester PB1000 with a triangular Berkovich diamond pyramid as an indentor. Number of test points was equal to 9 in each experiment, indentation force was equal to 3 N, load-relaxation cycle time was approximately 200 s per each measurement. Resulted values of microhardness were recalculated in a Vickers’s scale automatically by means of Nanovea equipment and software.

3. Results and discussion
Two different series of experiments were performed in our research.

3.1. Experiment №1.
The FG experimental specimen type №1 was created of 25 layers of steel, 1 layer of bronze and 25 layers of steel again (figure 2). The results of the experiment demonstrated cracking in the interface between steel and bronze. It was proved via optical microscopy (figure 2).

The results of microhardness and stiffness measurements (figure 3, table 1) of the specimen type №1 demonstrated local increase of microhardness on a border between steel and bronze. Supposedly the reason of this embrittlement was intermetallic growth (presence of intermetallics was proved by X-ray
diffraction analysis). Internal stresses and deformations in the transitional zone caused by difference
between temperature expansion coefficients of materials (up to $19.6 \cdot 10^{-6} \, ^\circ\text{C}^{-1}$ for AISI 316L and at the
most $17.5 \, ^\circ\text{C}^{-1}$ for aluminium bronze) were the reason of crack nucleus occurring on the grains of
intermetallics.

Figure 2. The experimental specimen type №1. In the right picture: dark area is bronze, bottom part
of the specimen is leftward.

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Figure 3. The microhardness measurement pattern of the experimental specimen type №1.

Table 1. The results of microhardness and stiffness measurements of the experimental specimen №1.

|       | 1  | 2  | 3  | 4  | 5  | 6  | 7  | 8  | 9  |
|-------|----|----|----|----|----|----|----|----|----|
| Microhardness, HV | 133 | 171 | 143 | 179 | 196 | 195 | 223 | 177 | 186 |
| Young’s modulus of elasticity, GPa | 32.6 | 34.9 | 29.8 | 34.2 | 33.8 | 33.7 | 36.7 | 33.5 | 34.4 |
3.2. **Experiment №2.**

The specific multilayer transition technique (figure 4) was used to cease cracking on the same treatment modes as in the experiment №1. Optical microscopy analysis proved absence of cracks (figure 4).

![Figure 4](image_url)

**Figure 4.** Experimental specimen type №2: multilayer bronze (dark layers) - steel (light layers) structure. Bottom part of the specimen is leftward in the right picture.

The microhardness research demonstrated even higher values of microhardness and lower elasticity (figure 5, table 2) in comparison with experiment №1. However, these phenomena didn’t cause cracking.

![Figure 5](image_url)

**Figure 5.** A microhardness measurement pattern of the experimental specimen type №2.

| Microhardness, HV | 170 | 266 | 257 | 242 | 191 | 198 | 203 | 188 | 238 |
|-------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Young’s modulus of elasticity, GPa | 37.8 | 43.2 | 42.1 | 40.3 | 35.6 | 36.2 | 37.1 | 36.6 | 40.6 |

**Table 2.** The results of microhardness and stiffness measurements of the experimental specimen №2.

Scanning electronic microscopy of the specimen type №2 allowed to observe dendritic structure (figure 6, left picture - dendritic crystals appear as herringbone dark patterns, two of which are marked with orange ellipses) and indicated nanometer porosity (figure 6, right picture - porosity is seen as a small black disks, some of which are marked with the red circles) which had no influence on the strength properties.
3.3. Numerical simulation
A numerical simulation in our research was performed via Dassault Systèmes Abaqus software. A purpose of analysis was confirmation that stress concentrators (which were the factor of cracking) were located in the interface area between steel and bronze in the specimen type №1. We researched a stress-strain state of the 1 mm diameter elementary analytical cylinder (imaginatively cut from the central area of the specimen to exclude edge effects from consideration). Influence of the temperature gradient was substituted by equivalent mechanical stresses. One tail of the cylinder was encastred (boundary conditions in finite elements analysis were: \( F_{x0} = F_{y0} = F_{z0} = M_{x0} = M_{y0} = M_{z0} = 0 \)); plane distributed tensile stresses, caused by inequal heating, acted at another tail; compressive stresses from metal around the cylinder acted at its envelope (figure 7). The value of all stresses (tensile and compressive) for a first approximation was set equal to 500 MPa [39, 40]. Exact value of resultant stresses did not play a significant role in the simulation results. Mechanical characteristics of materials were: of steel - mass density 7850 kg/m\(^3\), Young’s modulus 2.1·10\(^5\) MPa, Poissons ratio 0.3; of bronze - mass density 8960 kg/m\(^3\), Young’s modulus 1.1·10\(^5\) MPa, Poissons ratio 0.35. Parameters of mesh: 9·10\(^{-5}\) m approximate global size of seeds, 0.1 maximum deviation factor, linear geometric order C3D8R element type (an 8-node linear brick). The results of numerical simulation (figure 8) proved that mechanical stresses in the specimen type №1 had their ultimate values in the interface between steel and bronze (the red circles in the figure 8). The model in figure 8 is demonstrated with local angular cross section which shows distribution of stresses inside the cylinder. Blue area in the base of cylinder is a zone with the lowest value of mechanical stresses (what was certainly predictable because of boundary conditions). A light-green area in the figure 8 demonstrates a low-stressed zone.

Figure 6. Dendrites (left picture) and nanometer porosity (right picture) of the specimen type №2.

Figure 7. The loading pattern of the elementary cylinder (not to scale).
4. Conclusion. Key results and practical significance

A difference between thermal expansion coefficients of the materials along with rapid intermetallic growth, increase of microhardness (up to 223 HV), increase of elasticity modulus (up to 36.7 GPa), stress concentration and local embrittlement of the FG laser deposited specimens with two sharp transitions created of stainless steel AISI 316L and aluminium bronze in the DMT mode via the straight joining scheme lead to cracking on a border between stainless steel and aluminium bronze. These effects can be suppressed by producing (on the same regimes) a multilayer structure with alternating of steel and bronze layers, which will get comparatively more favourable thermal history during the process of laser treatment. Such multilayer structures can have even a higher level of microhardness (up to 266 HV) and Young’s modulus of elasticity (up to 43.2 GPa), but nevertheless, cracking of them can be entirely excluded because of reassignment of internal stresses and deformations in a volume of a detail. Porosity of such multilayer structures has nanometer level size and doesn’t effect on their mechanical strength properties. These phenomena and manufacturing method have a possibility to be used for purposes of space industry FG parts manufacturing of stainless steel and aluminium bronze via DED.

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