Interaction of pulsed laser radiation with a powder complex based on the Al-Mg-C matrix

A. Voznesenskaya¹, K. Khorkov¹, D. Kochuev¹, A. Zhdanov¹, V. Morozov¹
¹Vladimir State University named after A. G. and N. G. Stoletovs, Vladimir, Russia

E-mail: 2obk@bk.ru

Abstract. Experimental work on laser melting of the Al powder composition has been carried out. The influence of the duration of the laser pulse on the result of processing the powder composition has been studied. In this work, the powder material was obtained by the joint mechanical activation of matrix material and filler particles in high-energy ball mills. The research work consisted of analyzing the starting material, the phase composition, the particle size distribution, and the morphology of the powder particles. The obtained samples also studied the phase composition, the presence of pores, cracks, the surface of the formed coating, the average height of the roller. The obtained samples were studied by X-ray diffractometry, Raman spectroscopy, and microsections of the structures obtained by optical microscopy. On the basis of the data obtained, conclusions were drawn about changes in the structural-phase composition, the nature of the distribution, the localization of alloying additives in the course of phase-to-phase transitions, and the change in the phase states of alloying additives.

1. Introduction
Currently, additive technologies are actively developing both abroad and in our country. The layered laser sintering of metal powder compositions is one of the directions. The popularity of the application of this method of synthesis of details is growing every day. This can be attributed to the high flexibility of production (from the time of creation of the 3D model to the time of its production, an insignificant time passes), the ability to modulate the properties obtained was made by changing the concentrations of various powders used. A small amount of production waste and minimal subsequent machining are also attractive aspects of layer-by-layer technology.

The essence of the method of selective laser sintering consists in the metered delivery of powder to the region of action of laser radiation. This is done using a special laser head. Further, it moves in space through a system of drives along a given path, then a part of the given geometry is formed, or a coating is applied from the fused powder particles. The accuracy of the manufacturing of parts depends on the thickness of the forming layer (usually it is from 50 to 100 μm). This is due to the size of the spheres in the powder used, the features of the focusing system. The most common is 20 to 60 microns. Modern trends in mechanical engineering require reducing the particle size of the powder to 10 microns, which will increase the accuracy of processing (synthesis of the part or coating), reduce the porosity of the resulting material.

In modern industry, more often than not metal alloys are used, but metal-ceramic compositions. For the production of which various materials (alloying additives) are used, they are introduced into the metal matrix as directly in the process of fusion (as a result of exposure to high temperatures they form
a connection with the base metal powder), and it is possible to introduce into the process of powder synthesis directly ceramic elements, carbides, nitrides, oxides. With this approach, the contribution of chemical reactions to the synthesis of the part does not take place, and the effect of laser radiation on such a powder results in the fusion of particles with a minimum transition of the material to the vapor-gas phase from the excess energy produced as a result of the different absorbing capacity of metals and introduced impurities.

The creation of hybrid materials is a new trend in additive engineering applications. The property of this material is laid in the course of its synthesis, i.e. the item is made of materials having different properties (coefficient of thermal expansion, hardness, plasticity, heat resistance). Aluminum matrices filled micro- and nanodispersed carbon components planned to be used as a working material. Carbon nanotubes, fullerenes, onions, nanodiamonds, graphene, and graphite are used as promising reinforcing nanosized filler. Composite materials based on an aluminum matrix filled with carbon "components" [1] are promising for high-tech industries. At present, a significant number of studies have been carried out aimed at obtaining carbon-based composites based on aluminum and its alloys [2-4]. Thus, the problem of the difference melting temperature is serious and requires special attention. In the framework of this paper, it is planned to establish the possibility of using some absorbing materials capable of reducing the necessary energy of laser radiation to form a homogeneous fused area of powder material. It is also planned to investigate the dependence of the influence of the morphology of the powder granules and energy spent on melting the area of the powder material. The application of this approach will allow to provide by reflow due to a change in the absorption coefficient of laser radiation for each material of the powder composition. This will allow to align the energy threshold leading to the reflow of these or other granules. This approach is also possible to use when using powder materials with a large dispersion of the components of the compositions.

Aluminum heat-resistant powders were used as the processed materials. The classic powder for surfacing has a highly spherical shape, a smooth surface of granules, a low dispersion in size. Carbon is planned to be used as an absorbing material. The carbon saturation of the surface of the powder materials was carried out by co-grinding in a planetary mill. As a result of this approach, the surface and shape of the granules has been greatly altered. To establish the phenomenon, which is the dominant role in the absorption process, aluminum was also processed in a ball mill with the addition of corundum. Thus, it was possible to obtain a powder-like material similar in morphology to the surface and form of granules, but without carbon content. In the literature, no data were found concerning the increase in the absorptive capacity of materials with the use of alumina powders for processing with laser radiation with a wavelength of 1.06 μm. In turn, carbon has good absorption in a wide range of wavelengths [5]. The well-developed morphology of the powder granules must absorb laser radiation to a much greater extent than a smooth surface.

When introducing various ceramic components into the composition, there are many difficulties associated with different melting points, phase transitions of the materials used [6].

2. Experimental work

Laser fusion was carried out using a millisecond laser system operating in a pulsed mode of radiation generation. The treatment was carried out in argon: a powder composition was applied to the surface of the metal substrate, the thickness of the powder layer was 150 μm. This thickness of the layer is due to the need to determine the condition for the maximum effective absorption invested in the melting of energy. The efficiency of reflow at the stage of selection of regimes was determined by visual control of the surface of the formed roller. The resulting surface was subjected to pulsed laser action, the radiation wavelength was 1060 nm, the spot size was 2 mm, the overlap when scanning 60%, pulse repetition frequency 25 Hz, the energy in the pulse varied from 1 to 30 joules in the pulse. The diameter of the laser beam on the treated surface was 1.8 mm. The treatment was carried out in 1 layer. The surface treatment of the powder layer was carried out without the use of additional heating. During the action of the laser pulse, a liquid phase was formed, followed by rapid cooling and solidification. The
nature of the treatment did not cause a significant heat load on the melted powder-substrate system. The scheme of the experimental setup is shown in Fig 1.

![Experimental setup](image)

**Fig.1.** Experimental setup: 1) powder, 2) Metal substrate, 3) The argon supply system, 4) Laser system, 5) Laser radiation

Optimum regimes for uniform reflow of powder materials by the criterion of the minimum sufficient power: 1 - (Al + Mg + 9C) - 4 J, 2 - (Al + Mg + 1C) - 5 J, 3 - (Al + 2Mg + 1ANT) - 5 J, 4 - (Al (heat-resistant)) - 9 J. During processing, materials 1-3 were melted at an energy almost 2 times lower than pure aluminum powder. It is worth noting a fairly narrow window at which a uniform roller is formed (+/- 0.2-0.4 J). When exposed at a lower energy, the powder is collected in droplets, the wettability of the components of the composition is poor; when the energy is exceeded, intense combustion of the material takes place with a characteristic dynamic gassing. In the treatment of pure aluminum, the stable formation of the roller occurs at 9 J, continues to about 12 J, further, cracks are formed, caused by a high temperature load. Probably, if the surface is heated up to temperatures of 250-350 degrees Celsius, according to [7], the formation of cracks can be avoided. At energies above 15 J, active gas formation and combustion of the powder material occur.

### 3. Research

The research work consisted of the analysis of the initial material, the particle size distribution and the morphology of the surface of the powder particles. The morphology of the surface and the granulometric composition of the powders after mechanochemical treatment in a ball mill were investigated by scanning electron microscopy (Fig. 2). The photographs of SEM show that the particle sizes of the powder differ significantly. The particle sizes of Al + Mg + 9C vary from 10 to 100 μm, Al + Mg + 1C is 40-150 μm, Al + 2Mg + 1ANT is 50-120 μm, Al (heat-resistant) is 10-120 μm. Particles of powders have a complex, ambiguous form.
Fig. 2. SEM images of powders: 1) Al + Mg + 9C, 2) Al + Mg + 1C, 3) Al + 2Mg + 1ANT, 4) Al (heat-resistant)

After the work on laser melting of the surface, a study was made of the surface of the Raman scattering (see Fig. 3) and optical microscopy (see Fig. 4), which was fused with spectroscopy. The surface structure, the presence of carbon, aluminum carbide, aluminum oxide were studied. The distribution of these substances on the formed surface was investigated. On the surface of sample 1 there were peaks of aluminum carbide, graphite grains were encountered, islands of fine-grained graphite were encountered on the surface of the sample (Al + Mg + 1C), alumina was recorded on the surface of the sample (Al + 2Mg + 1ANT) at the boundary of the reflow points. For a sample of pure aluminum, aluminum oxide was not recorded.

Fig. 3. Raman spectroscopy of the obtained samples: 1) Al + Mg + 9C, 2) Al + Mg + 1C, 3) Al + 2Mg + 1ANT, 4) Al (heat-resistant)
The next step was to study the formed roller in the cross section by creating microsections with subsequent etching of the surface. This study was aimed at studying the structure of the formed material in a cross section to detect pores, cracks, foreign inclusions by means of metallographic analysis. The results of the investigation of microsections are shown in Fig.4.

![Microsections:](image1)

![Morphology:](image2)

Fig.4. Metallographic examination of sintered aluminum powder by SLS-method: 1) Al+Mg+9C, 2) Al+Mg+1C, 3) Al+2Mg+1ANT, 4) Al (heat-resistant)

Information on the nature of the ongoing sintering processes can be obtained as a result of an analysis of the geometric characteristics of the laser exposure zones - the depth and width of the HAZ. The width and depth of the zone of thermal laser action in the general case depends both on the parameters of laser processing, and on the reflectivity, density, and thermophysical properties of the material being processed.

Fig. 4.1 shown that on the surface of melted zone contain free carbon and the inclusion of aluminum carbide which prevent the wetting between layers. On Fig. 4.2 the observed processes are similar to the first sample, but a smaller content of carbon not lead to formation a expressed carbon-containing layer, preventing to wetting by the melt, there is a possibility of applying several layers. On Fig. 4.3 present needle-like or columnar crystals growing in the direction of intensive cooling. There is a significant number of cracks and low adhesion resistance as the substrate and in the formation of new layers. For Fig. 4.1-4.3 is characterized by a much smaller thickness of the layer formed. This is probably due to the presence of an excessive amount of carbon and corundum that prevents the wetting of the components of the composition between themselves. Also, the developed surface of the powder materials used is a sufficiently gas-saturated medium, which is accompanied by a dynamic gas yield under the action of laser radiation, as a result of the removal of the material from the region of action, a small thickness of the formed layer (about 30% of the powder thickness). On Fig. 4.4 shown the formation of fine-grained structure, the porosity is not observed. However, achieving a stable melting takes 2 times more power than material processed in the case of Fig.1-3.

On the basis of the data obtained, conclusions were drawn about changes in the structural-phase composition, the nature of the distribution, the localization of alloying additives in the course of phase-to-phase transitions, and the change in the phase states of alloying additives. Data are obtained on the pronounced effect of the morphology of the powder granules and on the presence of absorbing materials on the surface of the granules on the absorption of laser radiation energy.

4. Conclusion
In the work done, the effect of surface structuring of powder microgranules was investigated to increase the absorption of laser radiation by laser fusion. Also, an absorbent coating based on finely divided
carbon was used. Thus, the use of absorbing materials (carbon) and the change in the morphology of the surface of the granules made it possible to reduce the energy of formation of a uniform roller from the powder by almost 50%, which is a very tangible result. The structure of the formed fusion layer is practically the same as the original material. When using corundum as an abrasive material to change the morphology of the surface of the powder granules, the structure of the formed weld layer has pronounced cracks. This is probably caused by poor wettability of corundum with aluminum. The result of this paper qualitatively shows the possibility of changing the absorption capacity of the granules of the powder composition by modifying the surface of the granules or using highly absorbing materials. Increasing the effectiveness of this approach is possible by improving the technology of changing the morphology of the surface by selecting the optimal phase and quantitative composition of the abrasive material, the material of the absorbing coating, and the use of other physical mechanisms of action.

This study was performed as a part of the state task VISU 1106/17 and grants of the RFBR number 16-48-330031, 17-20-03084 and 16-08-01226.

References

[1] Aborkin A.V. et al. 2016 J. Russian nanotechnologies. The effect of the mechanical activation regime on the morphology and phase composition of the nanostructured composite material Al-2Mg-nC № 5-6. P 30-36
[2] Bashkirov E.R., Kochuev D.A., et al. 2015 Journal of Magistracy 3D synthesis of composite materials and parts P 39
[3] Grigoryev S.N., Tarasova T.V. 2015 J. Metallurgy and heat treatment of metals. №10 P 5
[4] Yan S.J., Dai S.L., Zhang X.Y. et al. 2014 J. Materials Science & Engineering V. 612 P 440
[5] Santos-Beltran A., et al. 2004 J. Metastable and Nanocrystalline Materials Microstructural and mechanical characterization of aluminium-graphite composites V. 20–21 P 133–138
[6] Kuznetsov S.I., Nefedov S.A., et al. 2005. Izvestiya of the Samara Scientific Center of the Russian Academy of Sciences Influence of composition of ti-al powder composition on the processes of laser sintering and intermetalid synthesis V.7 № 1 P 35-42
[7] Shishkovsky I.V. 2016 Basics of additive high-resolution technologies (SPb: Pete.)
[8] Grigoriants A.G., Safonov A.N. 1987 Laser technology and technology: In 7 books. Book. 3. Methods of surface laser treatment Ed. A.G. Grigor’yants (Moskow: Higher School) pp 159
[9] Popov M., Medvedev V., et al. 2010 J. Applied Physics. Fulleride of aluminum nanoclusters V. 108. № 9 P 094317
[10] Delone N.B. 1989 Interaction of laser radiation with matter - Course Lectures (Moskow: Science) pp 280