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Synthesis of a three-component aluminum-based alloy by selective laser melting

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ABSTRACT

Introduction. The technology of selective laser melting is one of the key technologies in *Industry 4.0*, which allows manufacturing products of any complex geometric shape, reducing significantly the amount of material used, reducing the lead time and obtaining a new alloy from elementary powders in the melting process. To understand the process of alloy formation under laser exposure, it is necessary to know the initial data of powders, which significantly affect the quality of the products obtained. **The purpose of this study** is to determine the requirements for the structural-phase state, elemental composition of aluminum, silicon and magnesium powders and further preparation of *Al-Si-Mg* (Al — 91 wt.%, Si — 8 wt.%, Mg — 1 wt.%) powder mixture for laser synthesis. The initial powders of aluminum *PA-4* (*GOST 6058-73*), silicon (*GOST 2169-69*) and magnesium *MPF-4* (*GOST 6001-79*) and powder composition *Al-Si-Mg* are studied using X-ray diffraction and X-ray phase analysis. The shape and sizes of particles are determined by the studies of raster electronic images. By the method of selective laser melting, samples are obtained from a powder composition under constant and pulsed laser exposure. The composition is prepared by mixing powders in a globe mill. **Results and discussion.** It is shown that the initial powders of aluminum, silicon and magnesium are single-phase. Particles with a size of 20–64 μm, recommended for selective laser melting, are used to obtain a powder composition. By mixing the powders for one hour, spherical particles are obtained, which is preferable for laser melting. The results of grinding the samples after laser melting showed that the samples obtained under constant laser exposure at the following mode parameters: P = 80 W, V = 300 mm/s, s = = 90 μm, h = 25 μm have the greatest mechanical strength. **Conclusions.** The described study shows the possibility of synthesizing products from a powder composition of aluminum, silicon and magnesium by selective laser melting.

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Introduction

The technology of selective laser melting is one of the key technologies in *Industry 4.0*, which allows manufacturing products of any complex geometric shape, reducing significantly the amount of material used, reducing the lead time and obtaining a new alloy from elementary powders in the melting process.

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Therefore, these technologies change the framework for the automobile, aerospace and mechanical engineering industries [1]. In spite of the intense introduction of additive technologies (*AT*) and industrial investments into it [2] there are difficulties that prevent the enormous potential of these methods from realizing. Many alloys, which possess perfect mechanical properties when being treated according to traditional production techniques, are prone to significant cracking when hardening during the laser melting [3]. The large gap in the scientific knowledge concerning the microstructures is also an obstacle limiting the use of *SLM* methods. The gap occurs due to the complex nonequilibrium processes associated with laser melting [4].

Currently, only a small number of aluminum alloys is used as basic materials for additive manufacturing [5], [6]. The most widely used ones are the hardening alloy *AlSiMg* [7, 8] and the eutectic alloy *AlSi12*. The mechanical properties of *AT* components produced from these two alloys compare well with those of cast or die-cast samples but it is definitely far below the properties of the deformed items produced from high-strength aluminum alloys, such as *Al7075* (*Zn* – 5.5 wt. %, *Mg* – 2.5 wt. %, *Cu* – 1.5 wt. %), which yield strength exceeds 500 MPa and its plasticity is 3–9 % [9]. Unfortunately, *SLM* of the last-named alloy is difficult due to its low weldability as well as due to its high reflectivity and low viscosity (an inherent disadvantage of most conventional aluminum alloys). To be more precise, thermal compression of the item when treated leads to its cracking [10]. Besides, the evaporation of the low-melting alloying elements, such as *Zn*, in the process of laser melting is crucial for the formation of strengthening phases, so it also results in mechanical properties deterioration.

Literature analysis shows that the composition of the alloy plays an important role in determining the final microstructure and the mechanical properties of the *SLM*-produced composites [11, 12]. *Al-Si-Mg* (*Al* – 91 wt. %, *Si* – 8 wt. %, *Mg* – 1 wt. %) alloy is close to the eutectic composition. It has perfect castability due to little change in volume when solidifying, which makes it suitable for manufacturing thin complex shape casts and a promising material for *SLM*-produced items with improved mechanical properties [13]. An increase in mechanical properties occurs due to an increase in the solids solubility and a decrease in the grain size of *Al-Si-Mg* alloys due to high rates of the powder material melting, cooling and solidification during the *SLM* process. Currently, globular powders produced from *Al-Si-Mg* alloy are used in *SLM* units [14, 15]. In the given paper the method of layer-by-layer laser synthesis will be applied to solve a fundamental problem – the possibility of synthesizing items and alloy of *Al-Si-Mg* system from the powder composition of aluminum, silicon and magnesium having principal difference in melting temperatures, density, thermal conductivity, etc.

Two methods are usually used to prepare mixtures of powdered metals: direct blending and mechanical alloying with a globe mill. Mechanical alloying is a nonequilibrium solid-state treatment method which can be used for preparing a powder composition at room temperature. The repeated deformation and destruction taking place under high-energy ball-milling lead to the change of morphology, size and microstructure of metal powders [16]. As the globe mill introduces a lot of energy into the powder mixture (in comparison to direct mixing), it can significantly influence the properties of the composite material after the laser treatment [17], that is why research is required to determine the powder morphology, the size and the size-wise distribution characteristics of particles in the initial powder.

The aim of the given study is to determine the requirements for the structural-phase state and element composition of aluminum, silicon and magnesium powders; further preparation of the mixture of *Al-Si-Mg* (*Al* – 91 wt. %, *Si* – 8 wt. %, *Mg* – 1 wt. %) powder composition to be used for the laser synthesis. Based on the purpose of the study, the following tasks were formulated: to study the initial powders and the powder composition by the methods of X-ray diffraction and X-ray phase analysis, scanning electron microscopy; prepare a powder composition for the *SLM* process and conduct an experiment of laser synthesis of the powder composition to determine the possibility of the process.

Study methods

The powders of aluminum, silicon, and magnesium were used as initial materials for creating the powder composition. Aluminum powder *PA-4* (produced according to *GOST 6058–73*), silicon powder

(*GOST 2169-69*) and magnesium powder *MPF-4 I (GOST 6001-79I)*. The powders differ in fusion temperatures, density, heat conductivity and other characteristics. The fusion temperature of aluminum is 660 °C, those of silicon and magnesium are 1,414 °C and 650 °C respectively. The density of aluminum is 2.7 g/cm^3, the density of silicon is 2.35 g/cm^3, the density of magnesium is 1.74 g/cm^3. At the same time the heat capacity of aluminum, silicon and magnesium does not differ much. The molecular heat capacity of aluminum is 24.35 J/(K·mol), those of silicon and magnesium are 20.16 J/(K·mol) and 24.9 J/(K·mol) respectively. The heat conductivity of aluminum under the room temperature is 237 W/(m·K), the heat conductivity of silicon and magnesium under the room temperature is 149 W/(m·K) and 156 W/(m·K) respectively.

The surface morphology of the particles was studied with the scanning electron microscope *LEO EVO* in Research Equipment Sharing Center «*Hanotekh*» ISPMS SB RAS under the following conditions: accelerating voltage – 20 kV, beam current – 1–2 nA, focal distance 8.5–10 mm, magnification – 100–2,000. The elemental composition of the surface of the sample was analyzed using the X-ray microanalyzer for dispersion analysis Oxford Instruments *INCA350*.

The x-ray diffraction studies were conducted on X-ray diffractometer *DRON-7* (Burevestnik, Russia) in CoK_α-rays (λ = 0.1789 nm). The voltage supplied to the roentgen tube was 35 kV, current – 22 mA. The imaging was conducted in the *Bragg-Brentano* geometry (*2theta-theta*) at the 2θ angle range of 10–165° with the sampling interval of 0.05° and rotation of the sample. Exposure time for each point was 5 s. To complete the X-ray diffraction studies, due to the vertical fixation of the sample on the diffractometer goniometer, the powders were glued with transparent zapon enamel into the viniplast forms.

The grain-size composition was established with the help of the screen test. The screen test ensures a simple approach to obtaining powder size-wise distribution by sieving the powder through the sieves (No. 0100, No. 0080, No. 0064, No. 0040, No. 0020) put on one another in the order of mesh size decreasing subjected to mechanical vibration during 60 min. Each screen holds the particles which are impassable for the next screen and this way each screen generates the size range of particles. The screen test of aluminum powder showed that the proportion of particles smaller than 20 μm is 6.5 wt. %, the proportion of particles smaller than 40 μm is 20 wt. %, 27 wt. % of particles are smaller than 64 μm in size, 17 wt. % of particles are smaller than 80 μm in size, 11 wt. % of particles are smaller than 100 μm in size, and particles over 100 μm make 25 wt. % of the powder. In the magnesium powder 3.6 % of particles are smaller than 20 μm in size, 15 % of particles are smaller than 40 μm in size, 27 % of particles are smaller than 64 μm in size, 26 % of particles are smaller than 80 μm in size, 25 % of particles are smaller than 100 μm in size. The powder loss when screening did not exceed 4%. The silicon powder was not sieved due to its poor flowability. To obtain the powder composition, particle size range of 20–64 μm was chosen as recommended for the selective laser melting. Smaller alloying elements ensure higher density of the powder layer, although the difference in the sizes of particles may result in undesired segregation. Three elementary powders were combined by weight *Al* – 91 wt. %, *Si* – 8 wt. %, *Mg* – 1 wt. % and then mixed with a globe mill during one hour in protective argon atmosphere. Balls of structural steel *ShKh15* with a diameter of 5, 7 and 8 mm served as grinding medium. A globe mill is an economically viable and widely used method of mechanical alloying of a powder composition.

The samples were grown at the selective laser melting unit *VARISKAF-100MVS* developed and made in Yurga Institute of Technology (branch) of Tomsk Polytechnic University [18, 19]. The conditions of the searching experiments were as follows: the constant laser power P = 80 W and the pulsed laser power P = 100 W, modulation m = 5,000 Hz, under the invariable mode parameters: the sampling interval S = 90 μm, the layer thickness t = 25 μm, and the varied laser beam scanning speed V = 100, 200, 300, 400 mm/s. The samples with a side of 10 cm were built on the aluminum base plate in a chamber preliminarily heated to 200 °C and filled with argon after preliminary degassing.

The metallographic samples were prepared by standard abrasive machining and diamond paste polishing to obtain the polished cross-section.

Results and discussion

In the recent years many studies have been concentrating on the optimization of the modes of selective laser melting of powder metallic alloys mainly made up of spherical particles. The problem of *SLM* alloy formation from metallic powdered materials made up of non-spherical particles is understudied. The properties of powders change both at the stage of preparation of the powder composition and during the *SLM* process under the influence of the environment, mechanical and thermal influences. All this affect the quality of the produced items. To determine the optimal *SLM* modes we need to know the particle distribution in terms of its size and chemical composition of the surface. Pollution of the powders is the major problem in *SLM*, especially when treating high-reactive materials, such as magnesium, titanium and aluminum alloys. Long-term exposure of the powders to the natural air leads to its oxidation and, as a result, to non-stable *SLM* process. Oxide layers prevent wetting and cause porosity.

Consequently, to understand the process of alloy formation in *SLM* we need to know the initial characteristics of the powders as it has a significant effect upon the quality of produced items. The important characteristics of the powder material to be used in *SLM* are morphology, grain sizing, surface chemistry, bulk density, rheology and thermal properties which are known to influence the material behavior when the material is exposed to the laser action [20]. Scanning electron microscopy, X-ray and computer tomography are used to study the form and the surface morphology of the powder particles.

In Figure 1 we provide scanning electron microscopy images (*SEM*) of the surfaces of aluminum, silicon and magnesium powders obtained when imaging the sample.

The aluminum powder is formed by conglomerates of irregular particles 1–20 μm in size and larger particles 30–140 μm in size (Fig. 1, *a*, *b*).

Single-phased magnesium powder is a mixture of particles with “scaly” structure, its size varies within the range of 30–400 μm (Fig. 1, *c*, *d*). Its particles have irregular form with rough texture of the surface which leads to reduced flowability. The element composition of the powder corresponds to magnesium with oxygen content less than 2 wt. %.

Single-phased magnesium powder consists of conglomerates 0.5–45 μm in size (Fig. 1, *e*, *f*). The proportion of large conglomerates in the powder does not exceed 15 vol. %. The powder also contains small amounts of aluminum, titanium, calcium and oxygen (not more than 4 %).

Figures 2–4 present X-ray diffraction patterns with the completed phase identification of magnesium, aluminum and silicon samples respectively. The phase compositions correspond to the single phases of *Mg*, *Al*, *Si*.

To form the *Al*-*Si*-*Mg* powder composition, the powders were subjected to mechanical mixing by placing the initial powders in a globe mill at the ratio *Al* – 91 wt. %, *Si* – 8 wt. %, *Mg* – 1 wt. % and activation in the protective argon atmosphere during one and two hours. Balls 5, 7 and 8 cm in diameter made of structural steel ShKh15 served as the grinding bodies at a mass ratio “powder-balls” of 1:10. The acceleration of the grinding bodies in the process of mechanical alloying was 40g.

The scanning electron images of the mixture of aluminum, magnesium and silicon powders obtained as a result of imaging the samples after one hour of mechanical activation are shown in Figure 5. The powder is presented by spherical and ellipsoidal aluminum particles which sizes vary within the range of 1–40 μm (Figure 5, *a*–*d*). There are also conglomerates of spherical particles 30 to 50 μm in size. The silicon particles in the powder mixture are presented by irregular agglomerates which sizes vary from 3 to 40 μm. The particles of magnesium powder are distributed throughout the powder (Fig. 6, *a*). A large amount of deformed powder particles is observed after one hour of mechanical activation of the powders (Fig. 5, *a*, *b*).

After two hours of mechanical activation the powder is also presented by spherical and ellipsoidal (elongated) aluminum particles which sizes vary within the range of 1–50 μm (Fig. 5 *c*, d, Fig. 6, *b*). There are conglomerates of spherical particles 30 to 50 μm in size. Silicon particles in the powder mixture are also presented by irregular agglomerates which sizes vary from 3 to 70 μm (Fig. 6, *b*). According to the mapping element analysis the magnesium particles are distributed throughout the powder and are also presented in the form of large (up to 70 μm) conglomerates (Fig. 6, *a*). After two hours of treatment the powder was

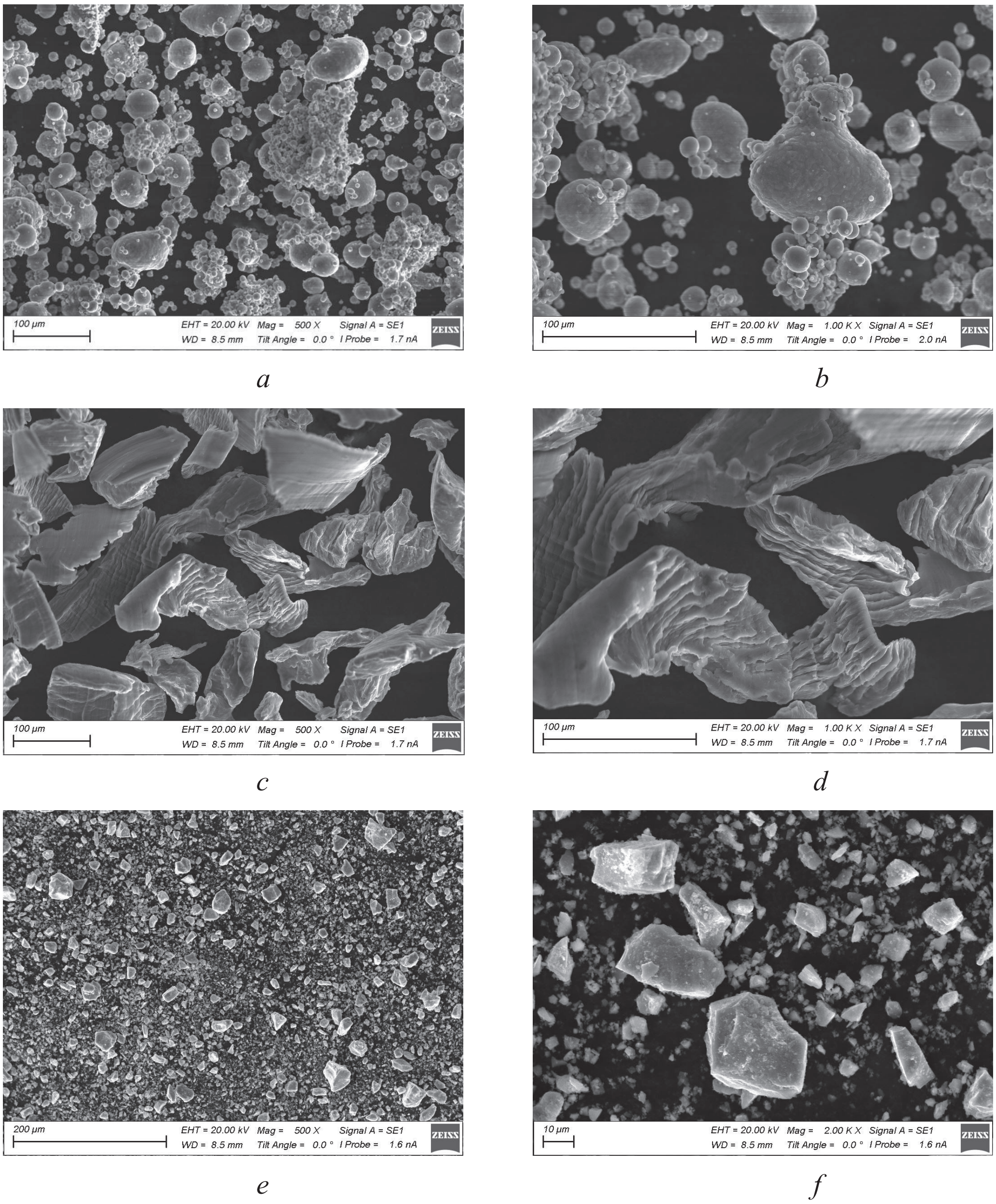


Fig. 1. SEM microphotograph of aluminum (*a*, *b*), magnesium (*c*, *d*) and silicon (*e*, *f*) powders

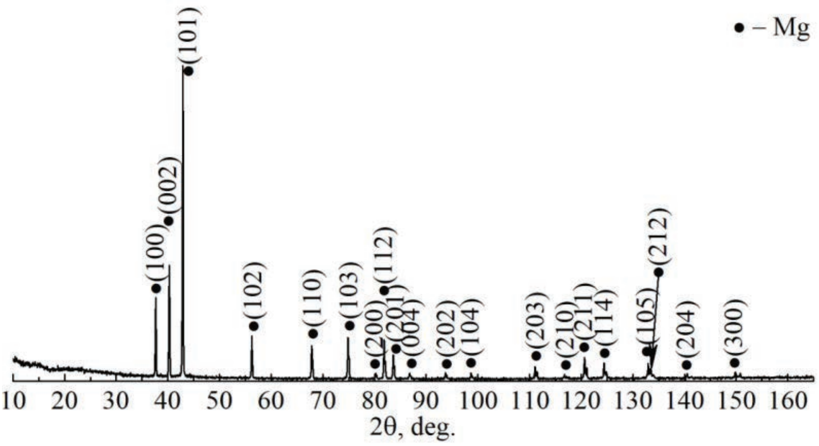


Fig. 2. X-ray diffraction pattern of a magnesium powder sample with phase identification performed

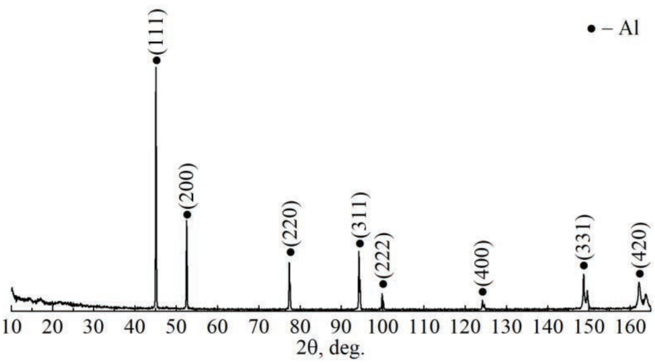


Fig. 3. X-ray diffraction pattern of an aluminum powder sample with phase identification performed

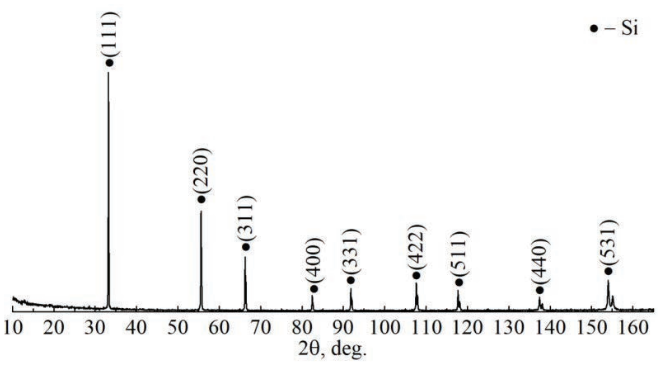


Fig. 4. X-ray diffraction pattern of a silicon powder sample with phase identification

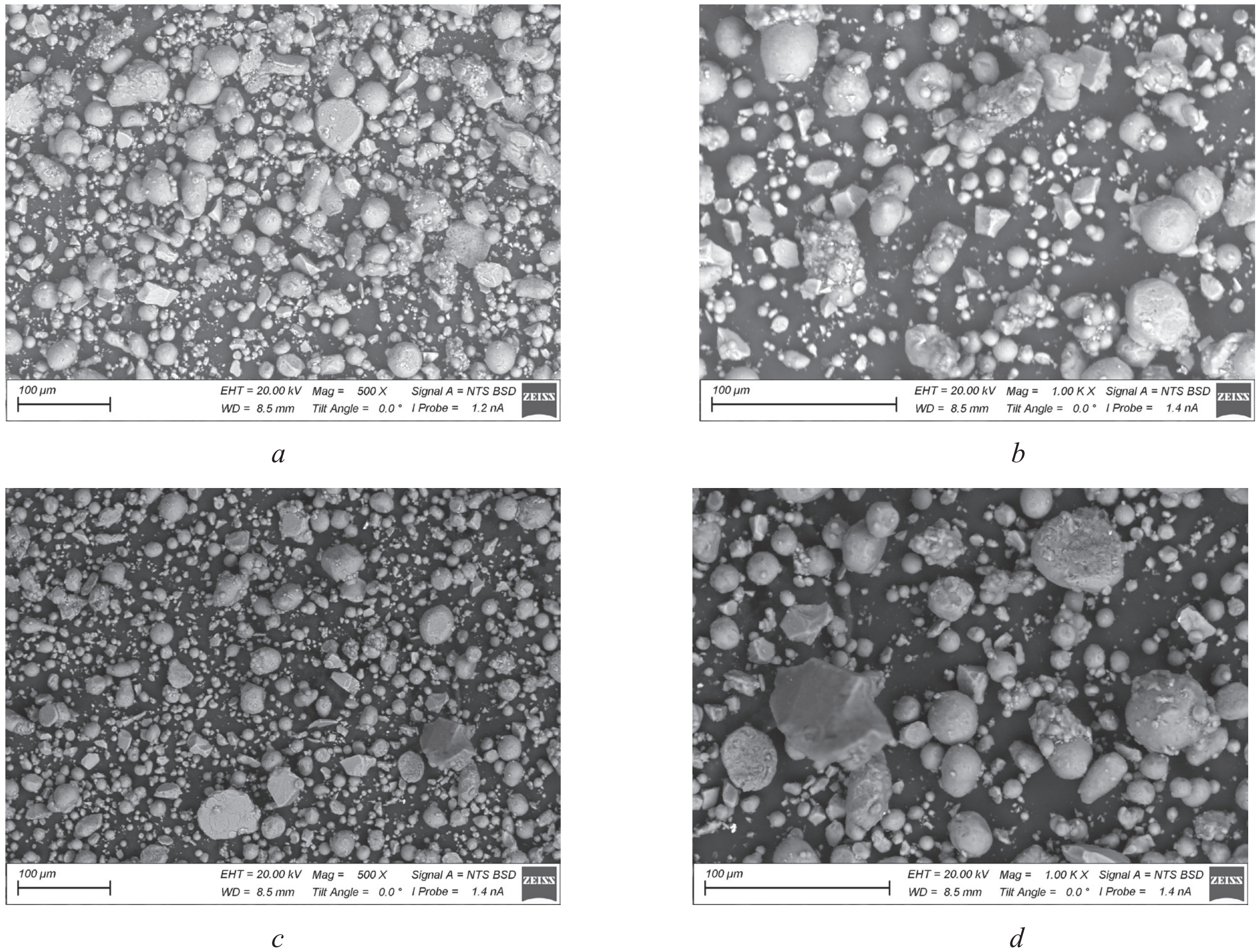


Fig. 5. SEM microphotograph of a mixture of aluminum, magnesium and silicon powders after 1 hour of mechanical activation (*a*, *b*) and after 2 hours of mechanical activation (*c*, *d*)

more oxygenated, so it was decided to reduce the activation time for further studies and complete the experiment in laser melting for the powder composition after one hour of mechanical treatment.

Thus, as a result of the experiment in formation of the powder composition *Al* – 91 wt. %, *Si* – 8 wt. %, *Mg* – 1 wt. % in the globe mill, globular powder was produced for further formation of an alloy in the process of selective laser melting. It is globular powder that is considered to be the qualified material for the *SLM* process as this form of particles allows increasing both the density of powder application and the rheological characteristics.

In Figure 7 we provide the X-ray diffraction pattern with the identification of the phases of the powder sample obtained by mixing for 1 hour. The phase composition is presented by two phases: *Al* and *Si*. The

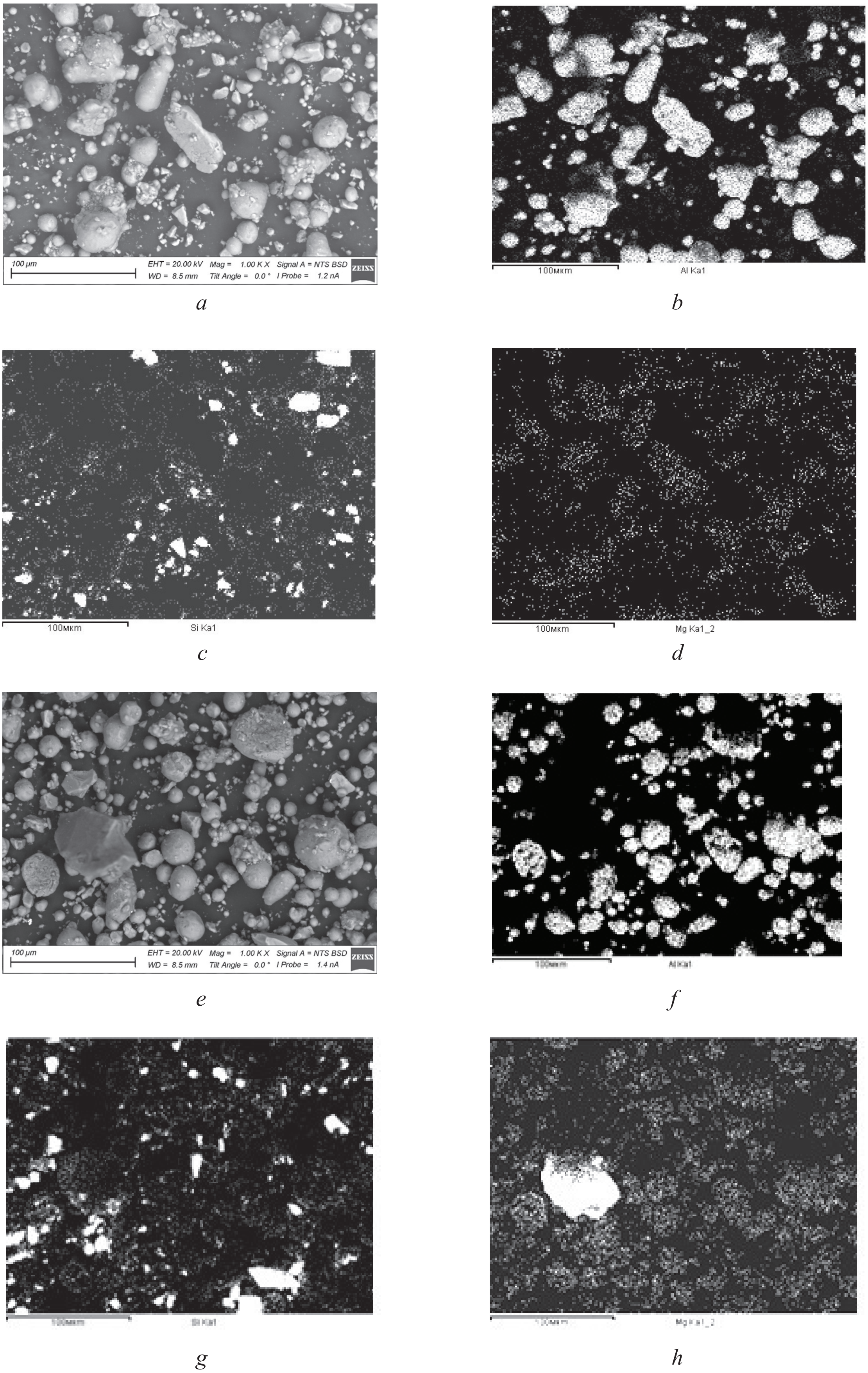


Fig. 6. SEM microphotograph and element distribution maps of a mixture of aluminum, magnesium and silicon powders after 1 hour of mechanical activation (*a*–*d*) and after 2 hours of mechanical activation (*e*–*h*)

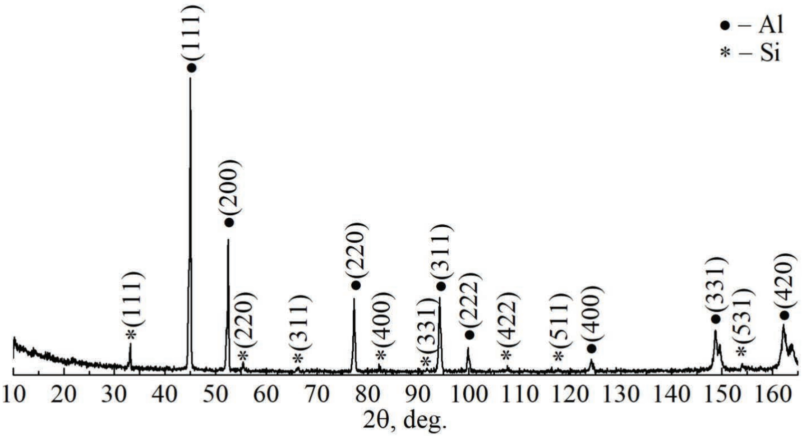
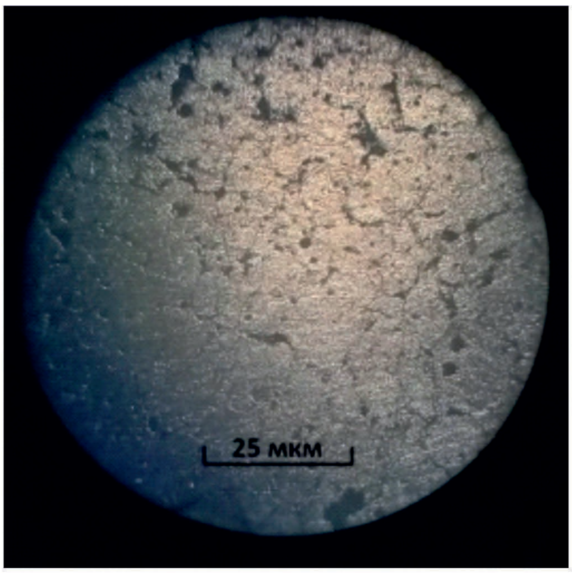


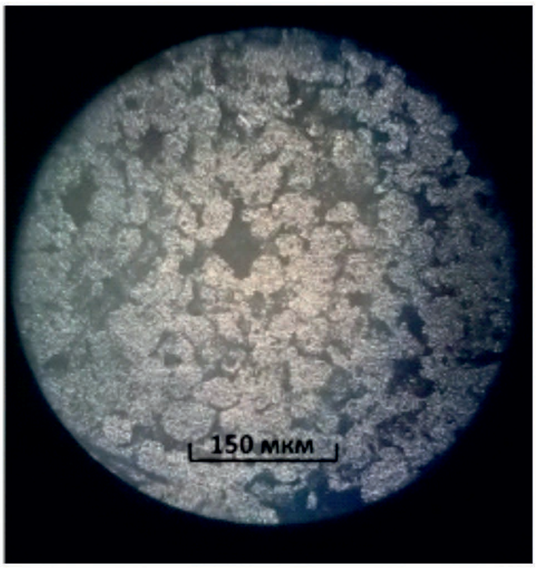
Fig. 7. X-ray diffraction pattern with the phase identification of the *Al-Si-Mg* powder composition obtained by mixing for 1 hour

volume fraction of aluminum is 90 %; that of silicon is 10%. The magnesium phase is not observed due to its small volume fraction in the studied powders.

The samples were grown at the selective laser melting unit *VARISKAF-100MVS*. The mechanical grinding of the samples showed that the sample produced under the following conditions: constant laser power of 80 W, the laser beam scanning speed of 300 mm/s, the sampling interval of 90 µm, the layer thickness of 25 µm – had the highest strength. The samples produced under the constant power, with other unchanged mode parameters, showed better strength (Fig. 8, a) than the samples produced under the pulse mode (Fig. 8, *b*). It broke off when being grinded; the microstructure analysis showed a large amount of non-melted powder. That is why it was decided to conduct the experiments only after the constant laser power.

Alloying the powders in the laboratory will significantly increase the range of materials that can be applied in *SLM*. It especially facilitates developing the composition of new alloys which are able to use the unique hardening method determined by the melting process and increase the productivity. However, to





a *b*

Fig. 8. Structure of samples obtained by the *SLM* method from powder after 1 hour of mechanical activation, mode parameters:

V = 300 mm/s, s = 90 µm, h = 25 µm: *a* – constant power P = 80 W; *b* – pulse mode P = 100 W, m = 5,000 Hz produce parts with uniform structure and properties it is extremely important to prepare the material with the controlled composition which is a difficult task when different-type elementary powders are mixed.

In the this paper the method of producing the powder composition by mechanical mixing was estimated to reduce the liability of powder elements to segregation. In the process of mixing finer powder is mixed with coarser one.

The process of mechanical mixing suggests a new interesting approach to producing more homogeneous initial powder material which can potentially simplify studying the alloy construction for *SLM* use and developing new alloy compositions with unique structure. To produce parts with uniform structure and properties it is extremely important to prepare the material with the controlled composition. The structure of *SLM* produced alloys differs from that of traditionally cast ones in terms of grain size, growth and phase formation morphology due to rapid heating and cooling cycles.

Extreme cooling speeds usually result in the formation of finer grains (≪1 μm) in comparison to the normal methods of hardening. The grain sizes in the *SLM* produced items can be controlled by changing the conditions of the melting process: higher scanning rate and lower resulting energy density allow obtaining finer microstructures. Increase of the scanning speed ensures larger amount of cooling in the molten pool which leads to higher hardening rate and slowing down of grain growth.

The results of the study show that the produced powders of magnesium, silicon and aluminum are single-phased. Presence of impurity phases is possible, its proportion does not exceed 5 vol. %.

The mechanical alloying of the powder composition (*Al* – 91 wt. %, *Si* – 8 wt. %, *Mg* – 1 wt. %) was conducted with the globe mill during one hour in the protective atmosphere of argon. The powder was made up of aluminum, silicon and magnesium with the particle size of 20–64 μm. Spherical particles were formed in the powder composition in the process of mixing.

In the recent years *SLM* production of the parts was thoroughly studied. The studies concerned the changes in the conditions of the process: laser power, the rate of laser scanning, the beam diameter, the sampling interval. The current research was carried out with the application of the process parameters approaching such optimal melting conditions of preliminary alloyed *Al-Si-Mg* with the grain-size composition of particles of 20–64 μm.

The results of the searching experiments show that the sample made at a speed of 300 mm/s showed the highest strength. The described study shows the possibility of synthesizing products from the powder composition of aluminum, silicon and magnesium by selective laser melting, but to produce samples with improved mechanical properties additional searching experiments are required with varying speed, diameter of the laser beam, changing the scanning strategy.

Conclusion

The initial powders of aluminum *PA-4* (*GOST 6058–73*), silicon (*GOST 2169–69*) and magnesium *MPF-4* (*GOST 60001–79*) were studied by the methods of X-ray diffraction and X-ray phase analysis which showed that the powders had single-phase structure. The study of *SEM* images showed that the aluminum powder consisted of conglomerates of irregular shape particles 1–20 μm in size, and larger particles 30–140 μm in size. The single-phased magnesium powder was a mixture of scaly particles, which size varied within 30–400 μm. The element composition of the powder corresponded to magnesium in the presence of oxygen no more than 2 wt. %. The single-phased silicon powder consisted of conglomerates 0.5–45 μm in size. The proportion of large conglomerates did not exceed 15 vol. %. Besides, it included small amounts of aluminum, titanium, calcium and oxygen (not more than 4%). The powder composition (*Al* – 91 wt. %, *Si* – 8 wt. %, *Mg* – 1 wt. %), which was used for producing the samples, was prepared by mixing the powders in the globe mill during one and two hours. The *SEM* images of the mixture of aluminum, magnesium and silicon powders after one hour of mechanical activation showed that the powder is presented by spherical and ellipsoidal aluminum particles which sizes vary within the range of 1–40 μm. There are also conglomerates of spherical particles 30 to 50 μm in size. The silicon particles in the powder mixture are presented by irregular agglomerates which sizes vary from 3 to 40 μm. The particles of magnesium powder are distributed throughout the powder. *SLM* method was used to produce samples from the powder composition under both constant and pulsed modes. Under the constant mode the parameters were as follows: P = 80 W, V = 100 mm/s, V = 200 mm/s, V = 300 mm/s, V = 400 mm/s, s = 90 µm, h = = 25 µm. The parameters of the pulsed mode were as follows: P = 100 W, m = 5,000 Hz, V = 100 mm/s, V = = 200 mm/s, V = 300 mm/s, V = 400 mm/s, s = 90 µm, h = 25 µm. The results of the searching experiments show that the sample produced under the constant power and V = 300mm/s has the highest strength and does not break off when being grinded.

The described study shows the possibility of synthesizing products from the powder composition of aluminum, silicon and magnesium by selective laser melting, but to produce samples with improved mechanical properties additional searching experiments are required with varying speed, diameter of the laser beam, changing the scanning strategy.

This paper presents the technology of mechanical mixing of powders as a methodology of producing homogenous raw material for *SLM*. Research on mixing elemental powders is of growing interest in the additive technology community to produce new materials. The promising new aluminum alloy (*Al* – 91 wt. %, *Si* – 8 wt. %, *Mg* – 1 wt. %) is developed for selective laser melting. The material allows forming highly dispersed structure with low porosity.

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Conflicts of Interest

The authors declare no conflict of interest.