

Structure and mechanical properties of parts obtained by selective laser melting of metal powder based on intermetallic compounds Ni_3Al

V G Smelov^{1,2}, A V Sotov^{1,2}, A V Agapovichev¹, E A Nosova¹

¹ Samara National Research University, 34, Moskovskoye Shosse, Samara, 443086, Russia

² Mechanical Engineering Research Institute of the Russian Academy of Sciences, 4, M. Kharitonyevskiy Lane, Moscow, 101990, Russia

E-mail: SotovAnton@yandex.ru

Abstract. The structure and mechanical properties of samples are obtained from metal powder based on intermetallic compound by selective laser melting. The chemical analysis of the raw material and static tensile test of specimens were made. Change in the samples' structure and mechanical properties after homogenization during four and twenty-four hours were investigated. A small-sized combustion chamber of a gas turbine engine was performed by the selective laser melting method. The print combustion chamber was subjected to the gas-dynamic test in a certain temperature and time range.

1. Introduction

At present the high strength steels, titanium alloys and nickel-based alloys are the main materials used for the manufacture of gas turbine engines (GTE). Other materials are used to a much lesser extent. Among the perspective materials intermetallic alloys should be noted, including those based on nickel and aluminium, the use of which will allow one to solve a number of problems in the aircraft industry, rocket and other high-tech fields of technology [1].

In the number of nickel and aluminium compounds, intermetallic compounds Ni_3Al and NiAl are of the greatest interest, used as heat-resistant materials and materials having high refractoriness and heat-resistance along with relatively small density. Yield stress of intermetallic Ni_3Al increases with heating up to 700°C. This temperature dependence of the strength properties of the intermetallic compound is associated with a particular mechanism of dislocation motion as described in research [2, 3].

One of the factors breaking the nickel-aluminium compounds is the limited number of possible production methods used for the parts' manufacture from these materials, which greatly affects the cost of production. Massive blanks may be prepared by conventional methods of powder metallurgy, plasma spraying with or without the use of hot isostatic pressing. Arc melting and casting, followed by the high-temperature extrusion or cold rolling, are also used for large-sized products alternating with the recrystallization annealing. All other traditional methods of casting with subsequent deformation operations are recognized as unsatisfactory, since they result in a brittle material [4].

One of the ways of reducing the production cost is the use of new fuel-efficient, environmentally



friendly and resource-saving technologies, such as selective laser melting (SLM) since SLM technology allows one to produce parts of complex shape in a short time almost without the use of tooling. These factors make it possible to drastically reduce the production lead time [5-10].

The SLM technology uses a high power laser to melt a thin layer of powder to produce machine' parts based on the prepared three-dimensional model [11-16]. The SLM method is a promising method for manufacturing the parts from the materials, which processing methods are traditionally complex, expensive and sometime technically impossible.

Nowadays a question about the use of high-speed (600 ... 800 km / h) unmanned aerial vehicles arises. Such speeds can be achieved using the small-sized GTE [17]. Manufacturing the small-sized GTE is limited by large periods of creation, technological preparation of production, manufacture and finishing machining of highly loaded parts (for example, a combustion chamber (CC), a turbine, a compressor). In this paper, a way to reduce mentioned stages by applying additive technologies is proposed.

2. Materials and methods

The initial material used in the research was metallic powder of the Ni_3Al compound, rectangle samples and a combustion chamber, obtained by the SLM method.

Samples and the combustion chamber was produced on a unit SLM 280HL in the inert gas (nitrogen) environment. This equipment has a chamber for creation with size 280x280x350 mm and a ytterbium fiber laser with 400 watts of 70 micron spot.

Interval and scanning rate power values of the powder surface layer by laser radiation were determined based on a series of preliminary experiments. The amount of transmitted energy was evaluated using the linear energy density equal to radiation power, to the relative scanning speed. Twelve different linear energy density values were obtained in the range of 0.23 to 0.65 J/mm based on the certain process conditions of alloying.

The several specimens were made from the powder intermetallic alloy using the fusion at various modes to investigate the influence of the main alloying process parameters (laser power and scanning speed) on the structure and physical and mechanical properties of the alloy.

The study of mechanical properties of the rectangle samples was performed on a floor system for fatigue testing INSTRON model 8802. Tension tests were carried out according to ASTM E8/E8M-13: "Standard Test Methods for Tension Testing of Metallic Materials".

Samples X-ray analysis was provided and the cross-sectional optical microstructure was studied to explain the effect the fusing and annealing modes on the mechanical properties. To study the microstructure of the samples, optical microscope Metam LP-31, as well as scanning electron microscope Tescan Vega, was used.

The chemical composition of the sample was studied using the energy-top box of scanning electron microscope (SEM) Tascan Vega.

The fused objects' homogenization at a temperature of 1150°C during four and twenty-four hours was made to uniform the chemical composition of the alloy and dispersed phases.

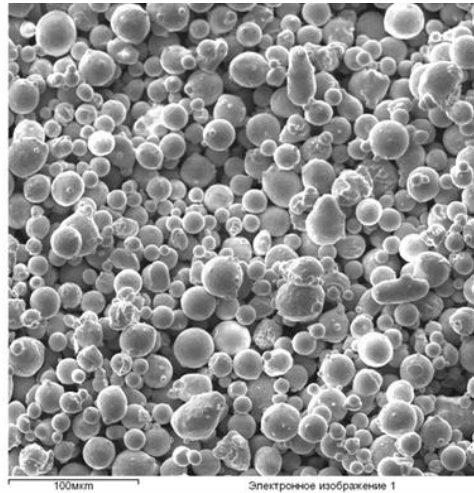
The gas-dynamic bench tests of the combustion chamber were made applying the heated air in the range of 573K to 1173K. Also, transient tests were conducted with the burner combustion in the range of excess air ratio changes from 3.25 to 1.86.

3. Results

The results of elemental analysis of the metal powder composition, based on the Ni_3Al intermetallic compound obtained by the electron microprobe analysis, are presented in Table 1. Figure 1 shows a morphology of powder, which was obtained by SEM.

Table 1. Chemical composition of used metal powder.

	Mass fraction, %										
	Ni	Al	Cr	Mo	W	Ti	Y	Hf	O	Si	C
Used powder	75.7	9.21	5.47	3.65	2.84	1.59	1.55	-	-	-	undefined
VKNA - 1V ^[18]	Base (77.28)	8.83	5.58	3.5	2.82	1.54	-	0.45	0.008	0.015	0.03

**Figure 1.** Ni₃Al powder surface morphology, x 400.

In Figure 1, particles are commonly spherical with a presence of defective grains. The powder's typical defects can be identified as satellites and an "amorphous" layer inherent in powders prepared by gas atomization [18].

Charts of tensile strength of the melted samples depending on LED are presented in Figure 2. In Figure 2, the maximum tensile strength takes place at LED interval 0.31 – 0.33 J/mm. When LED increases and goes down, the tensile strength drops, but at values of LED 0.45 J/mm, tensile strength rises slightly. The best plastic properties of samples are achieved in LED values interval 0.31-0.39 J/mm. Data of mechanical properties of the samples after heat treatment are presented in Table 2.

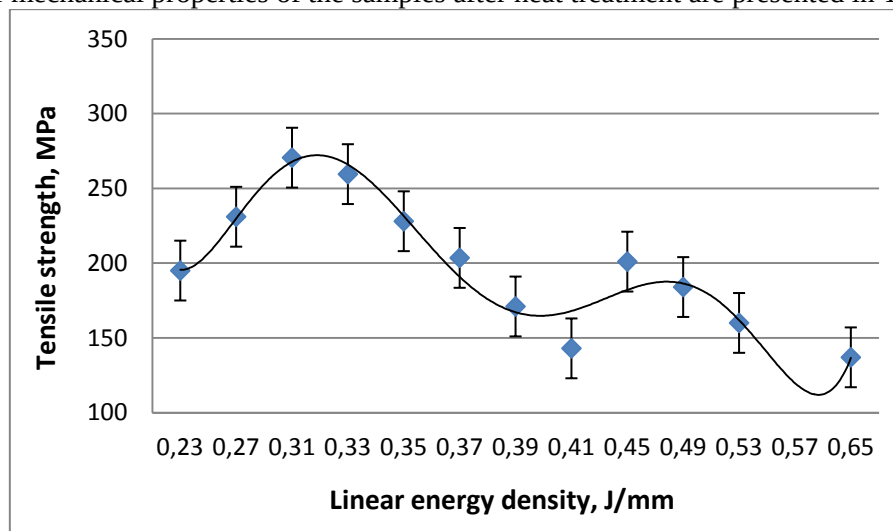
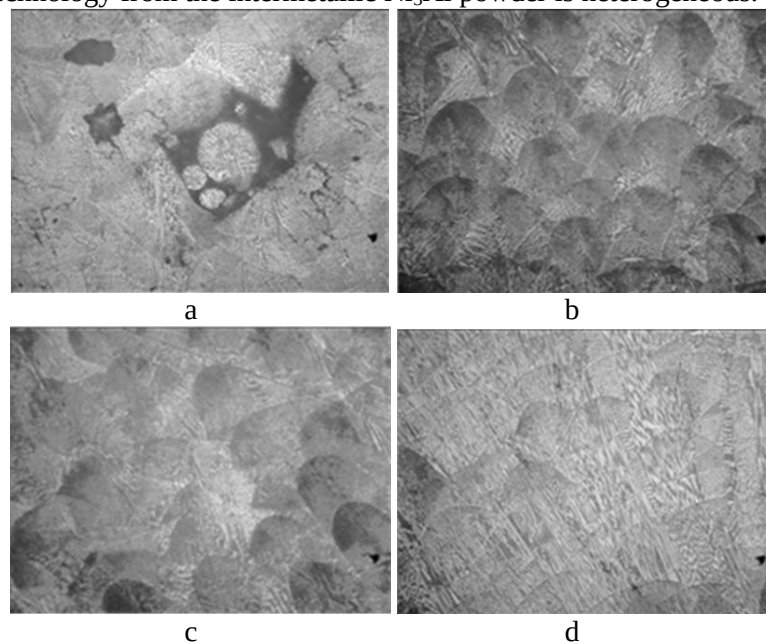
**Figure 2.** Dependence of tensile strength of intermetallic SLM samples on LED.

Table 2. Mechanical properties of samples after homogenization annealing.

State	Certificate data	Initial state				Homogenization during 4 hours at 1150°C				Homogenization during 24 hours at 1150 C
LED, η J/mm	-	0.31	0.37	0.41	0.49	0.31	0.37	0.40	0.40	0.30
Tensile strength, σ_b MPa	635 ^[18]	272	204	170	138	202	143	160	113	149
Elongation, δ %	13 ^[18]	1.2	1.3	1.2	1.1	1.1	0.6	0.5	0.4	0.5

In Table 2, the highest values of tensile strength ($\sigma_b = 272$ MPa) were obtained for samples, melted at the LED of 0.31 J/mm. Melting of samples at higher values of the LED leads to lower values of tensile strength (138...204 MPa). However, under these conditions, the equal data of elongation of 1,1...1,3% were kept. In samples, melted at LED B of 0.31 J/mm and annealed at 1150°C during 4 hours, a reduction of tensile strength by approximately 25% can be seen (from 272 to 202 MPa). This samples' elongation after annealing remained at values 1,1%. Annealed samples melted at other modes have the decreased tensile strength and elongation. The longer annealing during 24 hours leads to the further decreasing of the tensile strength down to 149 MPa and elongation - down to 0,5%, which corresponds to a drop in mechanical properties by approximately 60...65%.

Figure 3 shows the microstructure of samples melted by different modes. The structure of materials obtained by SLM technology from the intermetallic Ni₃Al powder is heterogeneous.

**Figure 3.** Microstructure of intermetallic Ni₃Al alloy obtained by different values of LED: a – 0.31 J/mm; b – 0.37 J/mm; c – 0.41 J/mm; d – 0.49 J/mm.

Some samples have pore defects. Figure 3a shows emptiness, incomplete fusion and cracks, obtained after melting at LED of 0.31 J/mm when the best combination of strength and elongation were obtained. Commonly, these defects are placed between particles. Also, there are a number of powder particles which retained their spherical shape. In Figure 3b, the sample after melting at 0.37 J/mm has less pores, cracks and incomplete fusion. Figure 3c shows a structure after melting at 0.41 J/mm, there are also no pores, cracks and incomplete fusion. After melting at maximum applied

LED of 0.49 J/mm (Figure 3d), emptiness and cracks were not observed. Particles changed their shape. The layer structure was found. The samples' microstructure after homogenization during 4 hours is presented in Figure 4; after homogenization during 24 hours – in Figure 5.

In Figure 4, there are cracks in the microstructure after homogenization during 4 hours. Their number and longitude are brightly seen after melting at LED 0.37 J/mm (Figure 4b). After melting at the LED 0.31 J/mm, longitude and a number of cracks are less (Figure 4a). Melting at LED of 0.41 and 0.49 J/mm leads to the minimum number and longitude of the cracks (Figures 4c and 4d).

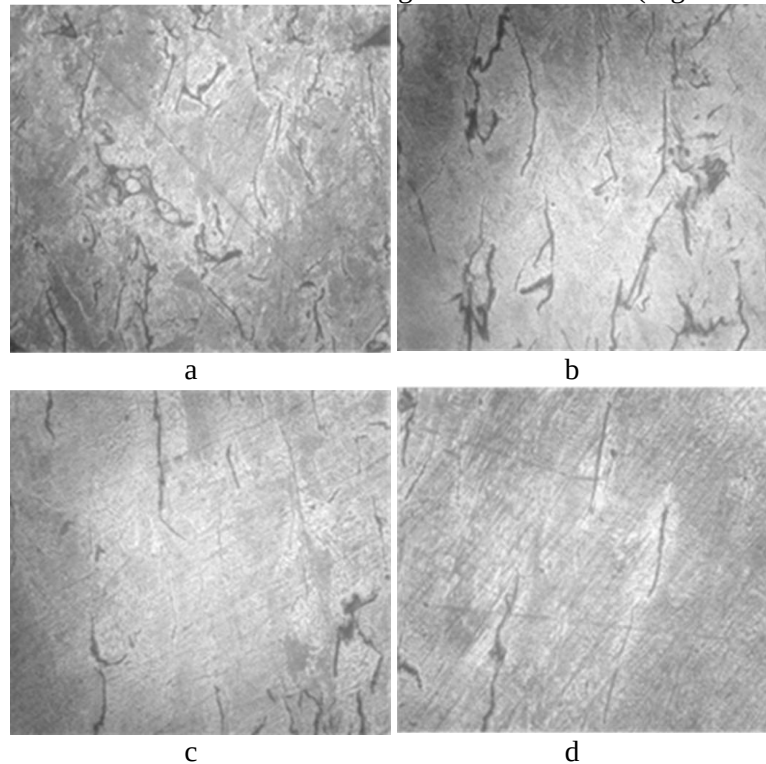


Figure 4. Microstructure of intermetallic Ni_3Al alloy after homogenization at 1150°C during 4 hours melted at LED: a – 0.31 J/mm; b – 0.37 J/mm; c – 0.41 J/mm; d – 0.49 J/mm.

In Figure 4a, incomplete melted particles are visible, which is evident because of the lack of energy for joining particles. Moreover, long high temperature of annealing at 1150°C during 4 hours did not lead to pores' disappearing.

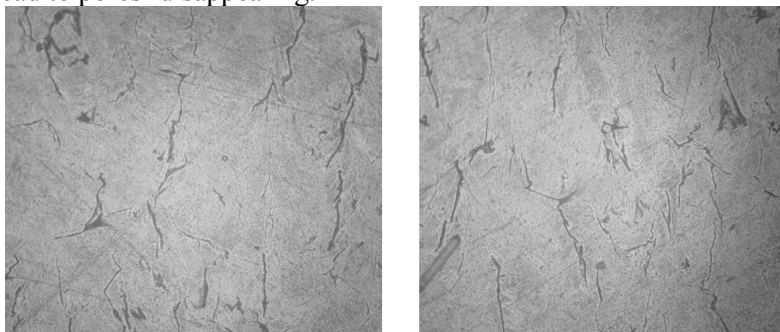


Figure 5. Microstructure of intermetallic Ni_3Al alloy obtained at LED 0.31 J/mm after homogenization at 1150°C during 24 hours.

Annealing of melted samples during 24 hours at 1150°C leads to the reducing of cracks' size. Besides, there is no certain morphology of particles and layers after annealing in the melted material,

which is visible in Figure 3.

X-ray phase analysis (Figure 6) of samples after fusing has shown that the surface layer consists of compound Ni_3Al . Other compounds have not been found.

After fusing and homogenization, X-ray pictures show peaks of Ni_3Al_2 in addition to peaks of Ni_3Al .

Inter plate spaces' change, calculated according to movement of interference lines of angles, is presented in Table 3.

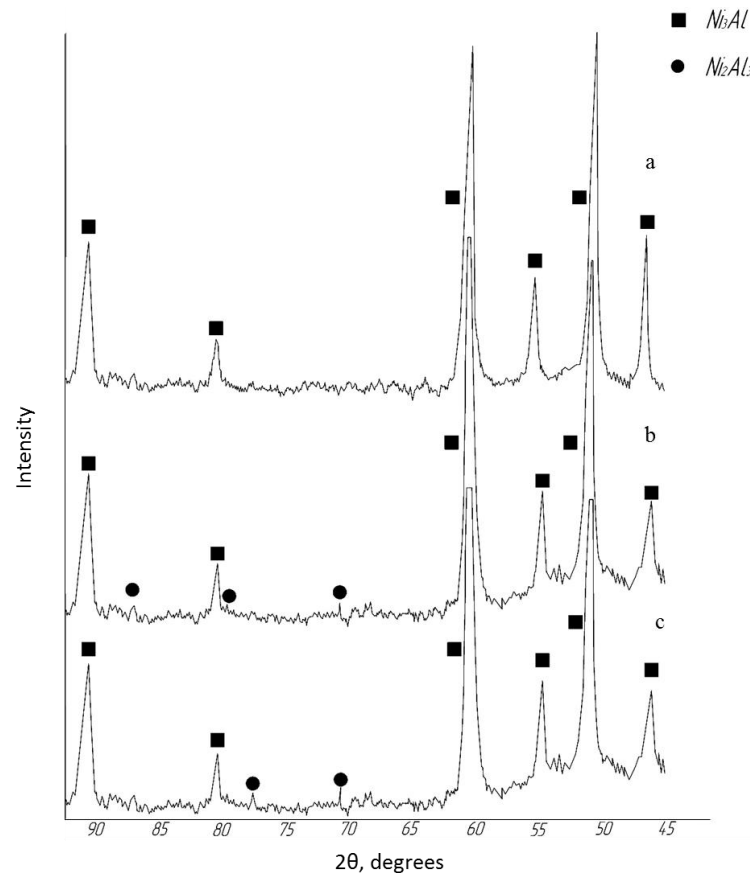


Figure 6. Diffractogram of fused and annealed samples: a - after fusing at linear energy density 0.31 J/mm; b – after fusing and annealing at 1150°C during 4 hours, c – after fusing and annealing at 1150°C during 24 hours.

Optimal values of the scanning speed of power and the powder' layer surface were obtained by the laser ray for producing the combustion chamber burner of the small-sized turbine engine from the intermetallic Ni_3Al powder by the SLM method based on the mentioned investigations. The obtained burner is represented in Figure 7. The common time of producing was 13.5 hours. The entering surface of the burner has small sized channels for air flow, where the substrate material cannot be used because of complicated removing from the inner cavity. Therefore, the burner was divided into three parts during fusing, which were joined by laser impulse welding technology.

Table 3. Inter plate spaces values for peaks of Ni_3Al and Ni_3Al_2 phases after homogenization

State	2θ, degrees	d/n	Δd, %
After fusing	51.5	2.060098	-
	60.3	1.781926	-
	90.3	1.26242	-

	97.6	1.189503	-
	51.6	2.056379	-0.18056
Homogenization 4	60	1.79	0.453107
hours	90.5	1.260234	-0.17317
	97.7	1.188596	-0.0763
	51.7	2.052674	-0.36104
Homogenization 24	60.5	1.77659	-0.29808
hours	90.4	1.261326	-0.08685
	97.8	1.18769	-0.15252

In Table 3, the inter plate spaces changing is negative, which is evidence of reducing inter plate spaces after homogenization.



Figure 7. Combustion chamber of small-sized GTE.

Gas-dynamic bench tests of the printed CC were made. The obtained sample was subjected to tests in the determined temperature and time intervals. The burner process and the extracted CC are presented in Figure 8.

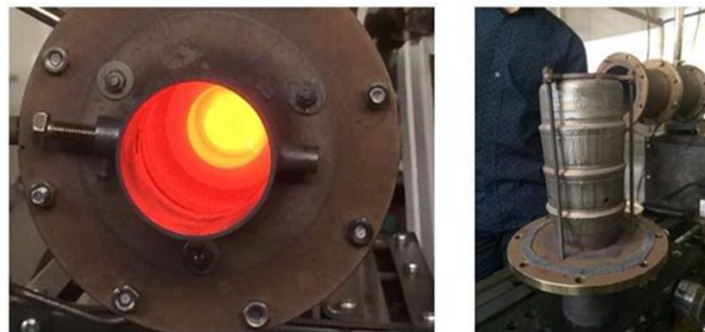


Figure 8. Bench tests of CC of short-time service engine.

4. Discussion

Cracks, appearing after melting and further annealing of the composite alloy, can be connected with residual stress, which may be a consequence of two factors: phase volume fraction changing and difference between linear coefficients of thermal expansion. If the Ni_3Al phase appears, volume fraction changing calculation will require following. The density of Ni_3Al = 7.35 g/cm³; the density of Ni_3Al_2 = 6.42 g/cm³. When the particle mass is the same, the volume of the Ni_2Al_3 particles will be by 12.6% greater than that for Ni_3Al . Since X-ray analysis has shown the presence of the Ni_3Al phase after homogenization, only a part of the Ni_3Al particle has transformed into Ni_3Al_2 . Table 4 presents the results of reaction products mass for transformation of Ni_3Al into Ni_2Al_3 .

Table 4. Calculation of reaction products for transformation of Ni₃Al into Ni₂Al for particles having the grading about 30...40 microns.

Reaction	2 Ni ₃ Al	→	Ni ₂ Al ₃	+	3Ni
Mass, g	1.04*10 ⁻⁸ 2.46*10 ⁻⁸		0.59*10 ⁻⁸ 1.39*10 ⁻⁸		0.45*10 ⁻⁸ 1.07*10 ⁻⁸
Molar mass, g/mol	230		203		58.7

Geometrical calculation shows if the shape of Ni₃Al particles having average 30...40 microns is spherical; their mass is nearly (1.04...2.46)*10⁻⁷ g. When 10% of this value (Table 4) will transform into the mix of Ni₂Al₃ and pure nickel on the surface of the Ni₃Al particle, the particle' diameter will increase to 0.06...0.010%. In this condition, the pure nickel thickness on the particle surface will be 0.07...1.5 microns. This changing of particle volumes can result in compressive residual stress on the border between particles, which can be seen from data in Table 3. Since ductility of initial intermetallic particles is low (see elongation values of samples in Table 2), changing of specific volume can lead to crack appearance, which can be observed in Figures 4 and 5. It must be noted that the plastic deformation mechanism of the ductile materials (such as austenitic steels or low alloyed aluminium alloys), thin films cannot be realized via dislocation sliding for the reason of the lack of space for dislocation moving. Therefore, elongation values of thin films may be lower than those for thick layers [19, 20, 21].

An annealing time increase is expected to result in the obtained intermetallic Ni₂Al₃ volume fraction growth and a further increase of compressive residual stress. From geometrics, 20 mass % of Ni₃Al transforming into Ni₃Al₂ can result in the particle having an initial size of 30...40 microns, which may grow up to 30.03...40.05 μm. In this case, the layer of nickel increases to 1.4...2.0 microns, which may lead to the reducing of length and width of cracks due to possibility of plastic strain of the nickel layer.

5. Conclusion

Samples obtained by SLM at LED values of 0.31 J/mm have the high level of tensile strength and ductility before other conditions of melting, but lower than those that have been written in the certificate of the producer, made for castings obtained from the chosen grade of powder.

Homogenization at temperature 1150°C during 4 and 24 hours leads to the decreasing of samples' tensile strength and elongation, which is connected to cracks, appeared in the microstructure.

X-ray analysis has shown that intermetallic Ni₃Al₂ was found after homogenization, but initial intermetallic Ni₃Al was kept. Presence of phase transformation leads to compressive residual stress. Increasing the annealing time for homogenization allows to the nickel layer grows up between particles, which in turn effects on longitude and width of cracks decreasing in samples' microstructure.

Increasing the homogenization annealing' temperature and/or the time can lead to cracks disappearance. For the confirmation of the supposed further research of main melting and heat treatment, technological modes influencing samples' mechanical properties are required, including at high temperatures in the interval of CC service conditions.

It was found that the metallic powder based on intermetallic Ni₃Al of the researched composition may be used for producing CC of small sized GTE by the SLM technology realized with unit SLM 280HL.

The bench test of small sized GTE, CC obtained by SLM were proved when feeding the air flow heated in the temperature range of 573...1173 K. The short time of the test of burning in CC of the excess air ratio ranges from 3.25 to 1.86 during 6 minutes.

6. Acknowledgments

This work was supported by the Russian Science Foundation grant (project no.15-19-00284). Standard samples for mechanical testing were manufactured on the equipment of the center of CAM common

use technology (RFMEFI59314X0003). Mechanical tests of the samples were carried out in the ONIL-1 laboratory of Samara University.

References

- [1] Eliseev Y S, Bojcov A G, Krymov V V and Khvorostukhin L A 2003 *Technology aircraft gas turbine engine production* (Moscow: Mashinostroenie)
- [2] Kolachyov B A 2005 *Metallurgy and heat treatment of non-ferrous metals and alloys* (Moscow: MISiS)
- [3] Youjun Y, Jiansong Z, Jianmin C, Huidi Z, Chun G, Lingqian W and Lianbin Y 2012 Preparation, microstructure and tribological behavior of laser cladding NiAl intermetallic compound coatings *Wear* **274-275** 298-305
- [4] Sims C T, Stoloff N S and Hagel W C 1987 *Superalloys II: High-Temperature Materials for Aerospace and Industrial Power* (New York: Wiley-Interscience)
- [5] Thijs L, Verhaeghe F, Craeghs T, Humbeeck J and Kruth J P 2010 A study of the microstructural evolution during selective laser melting of Ti-6Al-4V *Acta Materialia* **58** 3003-3012
- [6] Yadroitsev I, Smurov I. 2010 Selective laser melting technology: From the single laser melted track stability to 3D parts of complex shape *Physics Procedia* **5(2)** 551-560
- [7] Kruth J P, Froyen L, Van Vaerenbergh J, Mercelis P, Rombouts M and Lauwers B 2004 Selective laser melting of iron-based powder *Journal of Materials Processing Technology* **149(1-3)** 616-622
- [8] Agapovichev A V, Knyazeva A G and Smelov V G 2017 Numerical research of influence of laser radiation parameters on the formation of intermetallic phases from metal powders in selective laser melting technology *AIP Conference Proceedings* **1893(1)**
- [9] Vdovin R A, Smelov V G 2017 Design and optimization of the micro-engine turbine rotor manufacturing using the rapid prototyping technology *IOP Conference Series: Materials Science and Engineering* **177(1)**
- [10] Alexeev V P, Balyakin A V and Khaimovich A I 2017 Influence of the direction of selective laser sintering on machinability of parts from 316L steel *IOP Conference Series: Materials Science and Engineering* **177(1)**
- [11] Dholakia K, Cizmar T 2011 Shaping the future of manipulation *Nature Photonics* **5(6)** 335-342
- [12] Kruth J P, Froyen L, Vaerenbergh J, Mercelis P, Rombouts M and Lauwers B 2004 Selective laser melting of iron-based powder *Journal of Materials Processing Technology* **149** 616-622
- [13] Gu D D, Meiners W, Wissenbach K and Poprawe R 2012 Laser additive manufacturing of metallic components: Materials, processes and mechanisms *International Materials Reviews* **57(3)** 133-164
- [14] Smelov V G, Sotov A V, Agapovichev A V and Tomilina T M 2016 Selective Laser Melting of Metal Powder of Steel 316L *IOP Conference Series: Materials Science and Engineering* **142(1)**
- [15] Smelov V G, Sotov A V and Agapovichev A V 2016 Research on the possibility of restoring blades while repairing gas turbine engines parts by selective laser melting *IOP Conference Series: Materials Science and Engineering* **140(1)**
- [16] Sufiyarov V S, Popovich A A, Borisov E V and Polozov I A 2015 Selective laser melting of heat-resistant nickel alloy *Non-ferrous metals* **1(38)** 79-84
- [17] Schneider C, Rose M G, Staudacher S, Heisler A, Lippl F and Raab I 2012 On the assessment of turbine efficiency from experimental data for low pressure turbines at low reynolds numbers *ASME Turbo Expo 2012: Turbine Technical Conference and Exposition (Electronic Materials vol 8)* pp 1487-1496
- [18] Bazyleva O A, Bondarenko Y A, Morozova G I and Timofeeva O B 2014 Structure, chemical composition, and phase composition of intermetallic alloy VKNA-1V after high-temperature heat treatment and process heating *Metal Science and Heat Treatment* **56(5-6)** 229-234
- [19] Valiev R Z 2004 Nanostructuring of metals by severe plastic deformation for advanced

- properties *Nature Materials* **3(8)** 511-516
- [20] Valiev R Z, Korznikov A V and Mulyukov R R 1993 Structure and properties of ultrafine-grained materials produced by severe plastic deformation *Materials Science and Engineering A* **168(2, 31)** 141-48
- [21] Sun C Q 2007 Size dependence of nanostructures: Impact of bond order deficiency *Progress in Solid State Chemistry* **35(1)** 1-159