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Structure and properties of samples made from XH50BMTJuB-VI (EP648-VI) alloy produced by using selective laser melting process

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Abstract. *The paper presented examines the composition, the structure and properties of samples made from the XH50VMTJuB-VI (hereinafter EP648-VI) alloy obtained by using the selective laser melting process (the SLM-process) in relation to manufacturing parts for aviation purposes. The authors carried out a comparative study of the samples' structure and properties upon conducting such operations as depositing in two directions (horizontal and vertical), separate heat treatment and after hot isostatic pressing (HIP) followed by a standard heat treatment process applied for deformable semi-finished products made from the XH50VMTJuB-VI (EP648-VI) alloy. The authors inform that the manufacturing of samples by means of the SLM-process involved powders obtained through the technology of the vacuum-induction spraying of the molten metal jet with an inert gas (argon). The paper has established that samples obtained by using the HIP process with the application of the heat treatment (a vacuum high-temperature homogenization followed by a long-term aging) demonstrated the best set of mechanical properties, since the implemented complex process ensured the "healing" of pores and discontinuities in the structure, and strengthening by means of the intermetallic γ'-phase, while separations of the excess, needle-shaped α-Cr phase are fine and evenly distributed in the material structure. The authors noted that mechanical properties of samples under analysis were generally in compliance with the requirements set forth in the regulatory documentation for deformable semi-finished products made from the XH50VMTJuB-VI (EP648-VI) alloy, while underlining the increase in the level of impact strength of samples that underwent the HIP process, and the long-term strength of samples manufactured in the vertical direction compared to other options studied. Following the results of the analysis, the authors established that the SLM-process made it possible to manufacture products whose level of mechanical properties was close to the level of the deformable material, and even exceeded it in some cases.*

Keywords: *additive processes, selective laser melting, hot isostatic pressing, vacuum heat treatment, a heat-resistant nickel alloy, the γ'-phase, the α-Cr phase.*

Introduction

Additive processes are increasingly applied in the production chain of manufacturing aerospace parts, in the sphere of producing both airframes [1]–[4] and individual components of aircraft gas turbine engines [5]–[7] due to a number of advantages, primarily the reduction in time and the production labor intensity.

The selective laser melting (SLM) process is one of processes to become widely used in industries above. A number of research works [8]–[12] on testing the manufacturing of parts for aviation purposes shows that the SLM process makes it possible to manufacture parts with a given level of mechanical characteristics and a satisfactory surface condition.

Note that the introduction of additive processes in relation to aviation materials is limited because of the need to conduct comprehensive research works at all stages of manufacturing aircraft parts. The manufacturing of components of the hot section of gas turbine engines foresees the use of the XH50VMTJuB-VI (EP648-VI) heat-resistant nickel-chromium alloy, one of the most popular materials. The alloy is traditionally used for manufacturing components of combustion chambers and nonrotational parts of hot sections of aircraft engines, as the alloy ensures a high level of ductility and resistance to oxidation at high temperatures. The optimal content of Ti, Al and Nb ensure a good weldability of the alloy, a critical feature when applying the SLM process.

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At the same time, the experience of using the alloy above in traditional applications has shown that a high content of Cr in the alloy composition (32–35%), makes the alloy prone to the formation of excess phases (usually of needle-shaped or plate-like morphology) during a prolonged temperature exposure, with the phases contributing to a decrease in ductility, which can ultimately lead to the premature destruction of hot section components of the gas turbine engine. A further improvement of the alloy characteristics is, therefore, associated with ensuring the specified morphology and regulating the degree of dispersion of structural components of the alloy. When parts are manufactured using additive processes, the microvolumes of material (powder particles with a fractionation of 10–60 microns as a rule) are heated, melted and cooled at high speeds (10^3–10^5 °C/sec.), thus ensuring a uniform distribution of fine structural components in the volume of the end product [13]–[16].

In this case, the paper is aimed at illustrating the testing of the manufacturing of samples made from the XH50VMTJuB-VI (EP648-VI) alloy [in the aviation industry] using the SLM process, and the subsequent assessing of the microstructure and mechanical properties to determine the possibility of using the SLM process in manufacturing parts for aviation purposes.

Experimental Section

Research subjects are samples made from the XH50VMTJuB-VI (EP648-VI) alloy produced by means of the SLM process using the EOS M400 installation [working chamber dimensions (X/Y/Z) 400×400×400 mm] equipped with the ytterbium laser of 1000 watts, with the research aimed at testing mechanical properties and microstructure of samples. The fraction of granules of the alloy used for producing samples amounted to 20–53 microns and was obtained using the process of the vacuum gas-ejection spray of smelt with an inert gas (argon). Sample blanks were made both cylindrical Ø 12 mm and square with side 12 mm. The sample length was 60 mm. Samples were deposited with a 3D printer both in the XY (horizontal) the Z (vertical) directions.

The SLM process was followed by the hot isostatic pressing (HIP) using the QIH 09×1.5-2070-1400MURC hot isostatic press under the specific regime [heating at 1180±10 °C, with the subsequent 3-hour exposure at the working gas (argon) pressure of 160 MPa in a high-pressure vessel; the cooling process: a high-speed uniform cooling] to eliminate internal porosity and increase the density of samples

The samples, having undergone the HIP process, were subjected to thermal processing in the IPSEN T^2T vacuum furnace under the regime as follows: heating at 1140±10 °C with a subsequent 1-hour aging in a dynamic vacuum; the cooling process: in an inert gas (argon); aging at 900±10 °C; soaking of 16 hours in the dynamic vacuum followed by cooling in the argon stream (the cooling speed is identical to the cooling in the air).

Some samples did not undergo the HIP process for the purpose of comparative studies, yet these samples were subjected to the heat treatment immediately after the SLM process. Samples were also studied in the condition immediately after their deposition.

Workpieces, having undergone the processing under options above, were subjected to machining to ensure the size foreseen by the technical documentation for producing samples for mechanical tests.

The alloy chemical composition was determined by methods of spectral and chemical tests.

Mechanical properties (σ_V, σ_{02}, δ) of samples were tested using the ZDMY 30 tensile machine to ensure the compliance with the requirements set forth in the regulatory documentation for the EP648-VI deformable material.

The KCU shock viscosity was determined on shock samples tested using the Instron SI-1M pendulum machine.

The HB ($d_{OPP.}$) hardness of blanks was determined using the Leco AMH-43 solidomer.

Time to high-temperature destruction (the indicator determined during long-term strength tests) τ_σ^T was determined to ensure the compliance with the requirements set forth in the technical documentation for the deformable rod material using the Instron M3 installation at a temperature of 800 °C and the constantly applied load of 176 MPa (18 kgf/mm^2). When samples were undergoing long-term strength tests, they were brought to destruction.

The fractographic study of fractures of shock samples (upon undergoing shock viscosity testing) was conducted under the STEMI 2000-C binocular microscope.

The microstructure study was conducted on unsuitable and pickled micro-grinders under the “Axio Observer. DLM” microscope and using the raster electron microscopy method under the “Jeol JSM 6360LA” scanning electronic microscope.

Research Results: Analysis & Discussion

Study results established that the chemical composition of manufactured samples was in compliance with the requirements set forth in the technical documentation for deformable rod materials made from the XH50VMTJuB-VI (EP648-VI) alloy, and was approximately at the same level for all options studied in terms of the main alloying elements (Table 1).

When studying the structure of fractures of the original (immediately after deposition) shock samples produced in a horizontal direction by using the SLM process, the structural banded orientation with rough relief was registered, with the relief formed due to either the presence of areas of spherical structure and pores formed at the site or the detachment of granules (Fig. 1, *a*). The thermal processing made it possible to reduce the share of the banded orientation above (Fig. 1, *b*), while the hot isostatic pressing

Table 1. Chemical composition of samples from the EP648-VI alloy of the studied options

Direction of Construction	Content of Elements, % (wt.)							
	C	Cr	Al	Ti	Mo	Nb	W	Fe
XY	0.07	32.6	0.95	0.89	2.95	0.98	4.82	< 0.5
Z	0.07	32.5	0.96	0.88	2.98	0.99	4.85	< 0.4
Norms set forth in regulatory documentation regarding deformable materials	≤ 0.10	32.0–35.0	0.50–1.10	0.50–1.10	2.3–3.3	0.50–1.20	4.3–5.3	≤ 4,0

Note: Ni is base material; Content of Mn < 0,01; Content of Si < 0,4; Content of S, P < 0,005.

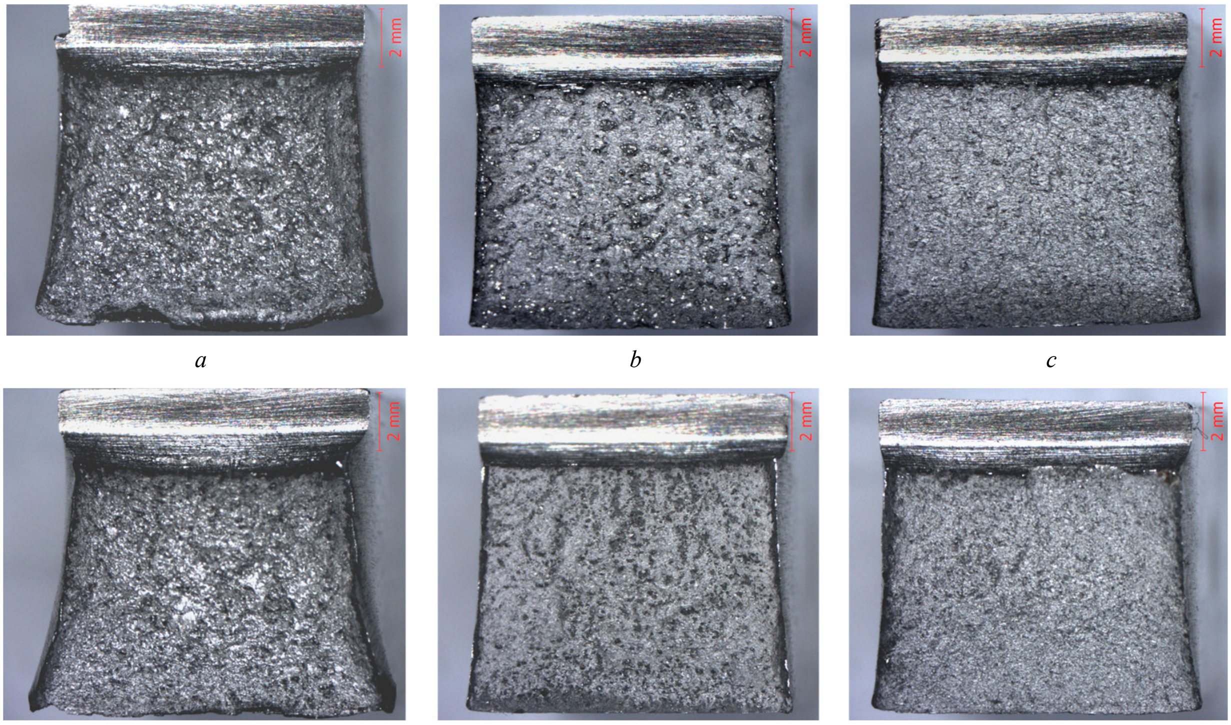
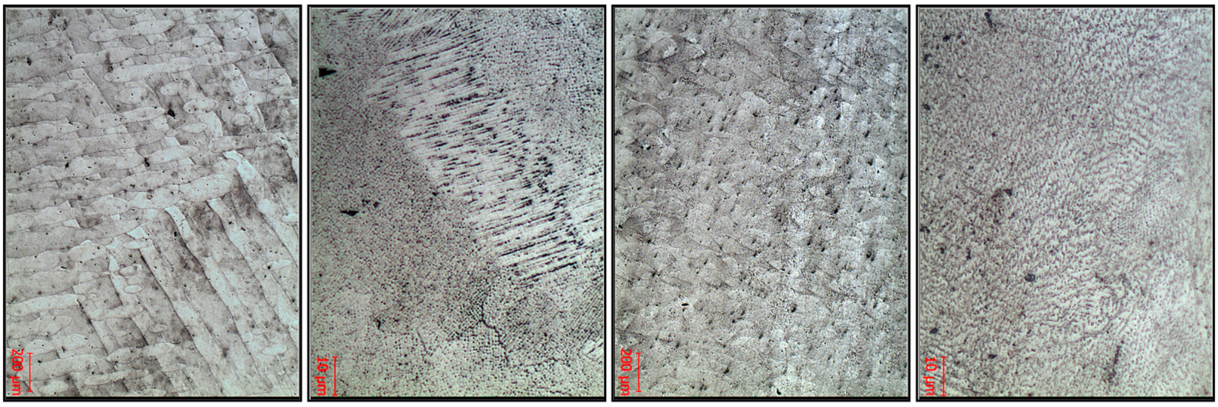


Fig. 1. Structure of fractures of horizontal (*a*, *b*, *c*) and vertical (*d*, *e*, *f*) samples made from the EP648-VI alloy of the studied options upon undergoing impact tests, ×6.5: *a*, *d* – state of construction; *b*, *d* – heat treatment; *c*, *e* – HIP + heat treatment

conducted prior to the standard thermal processing contributed to nearly the complete elimination of the banded orientation (Fig. 1, *c*) with the formation of a more homogeneous fracture structure. No banded orientation was observed on transverse fractures of samples deposited in the vertical direction due to a change in the sample deposition direction (Fig. 1, *d*–*f*). Significant chamfers along contours of samples in the state of their deposition (Fig. 1, *a*, *d*) indicate a sufficiently high plasticity in comparison with other options studied. Note that fractures of all options under study were generally characterized with fine granulation.

The metallographic study established that the microstructure of the material of samples (both vertical and horizontal ones) made from the EP648-VI alloy by using the SLM process was a γ-solid solution with the presence of a small amount of carbides, carbonitrides, as well as intermetallic phases and was characteristic of the non-heat-treated state of the EP648-VI alloy (Fig. 2, *a*, *c*) in the state of deposition (prior to the HIP and heat treatment). The microstructure of samples under study is characterized with the structural heterogeneity due to the formation of layer-by-layer (build-up) fusion zones that are grains and subgrains oriented by the competitive movement of crystallization fronts, and thin dendrites elongated in the direction of the sample deposition, which are formed due to high rates of heating and cooling during melting and solidification processes in a short time (Fig. 2, *b*, *d*). No cracks were registered in the volume of the forming fusion tracks.

The thermal treatment of the studied SLM samples led to the homogenization of the solid solution and the formation of a generally more homogeneous recrystallized structure due to the equalization of the chemical composition between layer-by-layer (build-up) fusion zones (Fig. 3). During the long-term aging (~ 16 hours), the research team



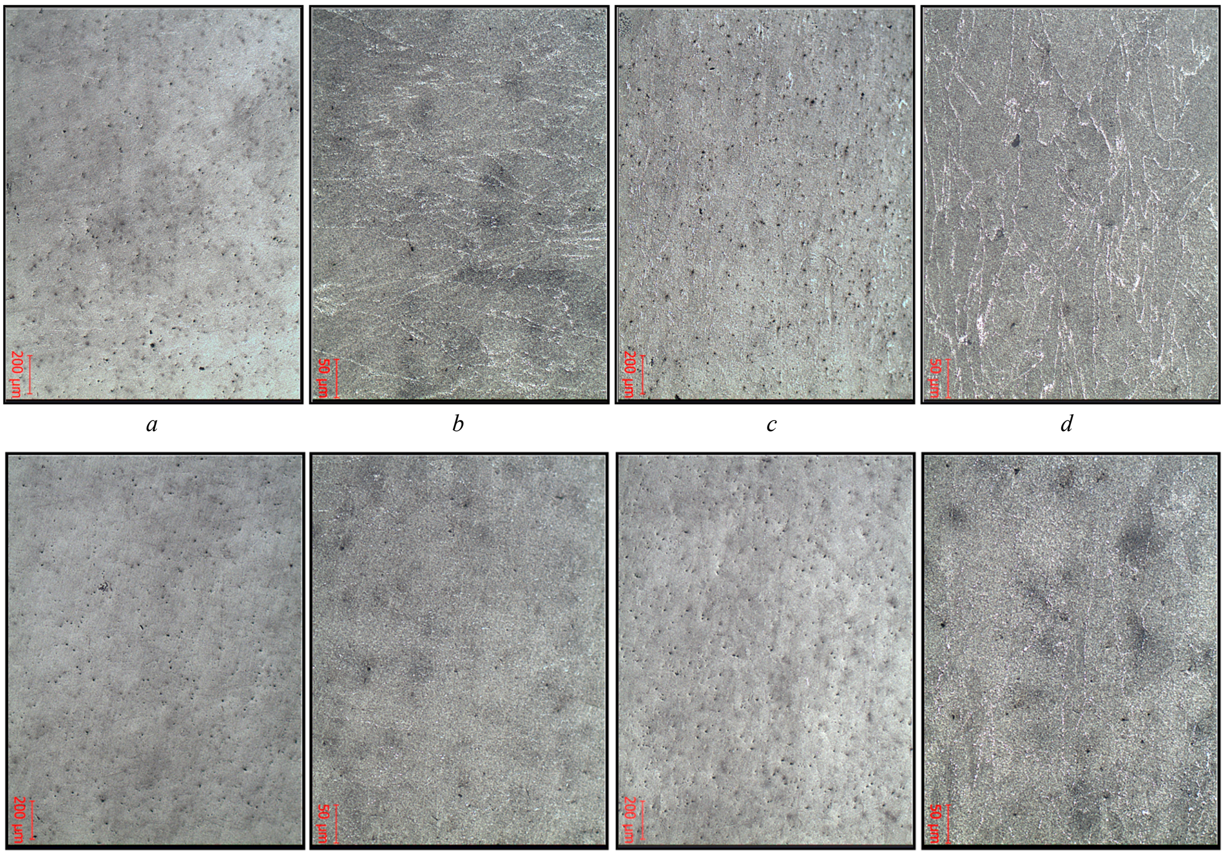
a *b* *c* *d*

Fig. 2. Microstructure of samples made from EP648-VI alloy (SLM) in state of deposition: *a*, *b* – horizontal direction; *c*, *d* – vertical direction

witnessed the separation of the following components from the supersaturated solid solution: the intermetallic γ′-phase of the stoichiometric composition $(Ni, Cr)_3(Al, Ti, Nb)$, carbides of the MC and $Me_{23}C_6$ types, and particles of needle-shaped morphology representing an excess phase based on Cr (α-Cr) – Ni(Cr, Mo, W) (Fig. 4).

The microstructural study using the method of scanning electron microscopy at magnifications up to ×10000 established that the amount of strengthening intermetallic phase in the alloy under analysis did not exceed 10% (Fig. 4). Note that the intermetallic γ′ phase and the α-Cr phase are released in a finely dispersed state with sizes of ~0.2 µm and ~ 2–3 µm respectively during the aging process.

Thu study did not register structures characteristic of the overheated state of the EP648-VI alloy in the form of meltings along grain boundaries.



a *b* *c* *d*

e *f* *g* *h*

Fig. 3. Microstructure of samples made from EP648-VI alloy (SLM) upon undergoing heat treatment (*a*–*d*) and upon undergoing HIP and heat treatment (*e*–*h*): *a*, *b*, *e*, *f* – horizontal direction of construction; *c*, *d*, *g*, *h* – vertical direction of deposition

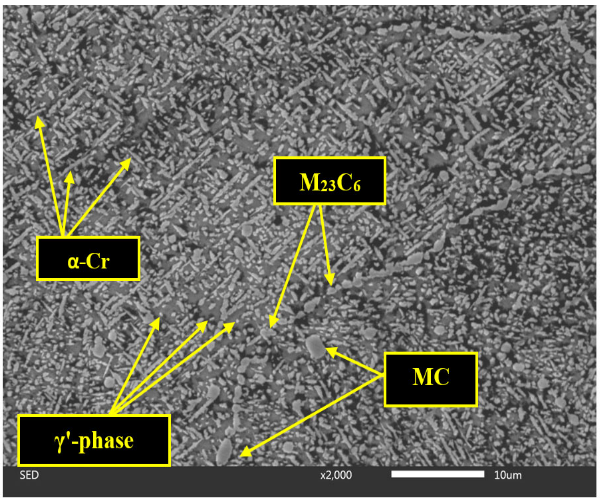


Fig. 4. Phase composition of samples made from EP648-VI alloy (SLM) obtained using SLM-process upon undergoing HIP and heat treatment

The metallographic study established that the material of the samples, upon both deposition and the heat treatment, was characterized by microporosity with a pore size of up to ~ 15 µm (Fig. 5 *a*), with single local discontinuities also registered at the interface with spherical powder particles (Fig. 5 *b*). The microporosity registered in samples produced by using the SLM-process is apparently associated with the burn-out of small particles of satellite granules (<10 µm in size, usually present in powders produced using gas jet spraying process) when a high level of energy is supplied to the fusion zone, and the heredity due to the presence of internal porosity in granules, which is formed when granules are sprayed with an inert gas under the influence of surface tension forces.

The microstructure of samples of all options studied is characteristic of the EP648-VI alloy in a normally heat-treated state. Carbides and carbonitrides in heat-treated samples are released mainly in the form of small discrete globular particles (Fig. 3).

Moreover, samples that failed to pass the HIP process contain oxide inclusions in the form of globular and elongated (Fig. 5 *c*) particles up to ~ 8 µm in size and particles with sizes up to ~ 26 µm (rarely found).

The HIP process contributes to almost the complete "healing" of pores and micro-discontinuities concentrated in internal volumes of the metal. At the same time, "healing" zones (Fig. 5, *d*) had globular and/or film oxides registered in them.

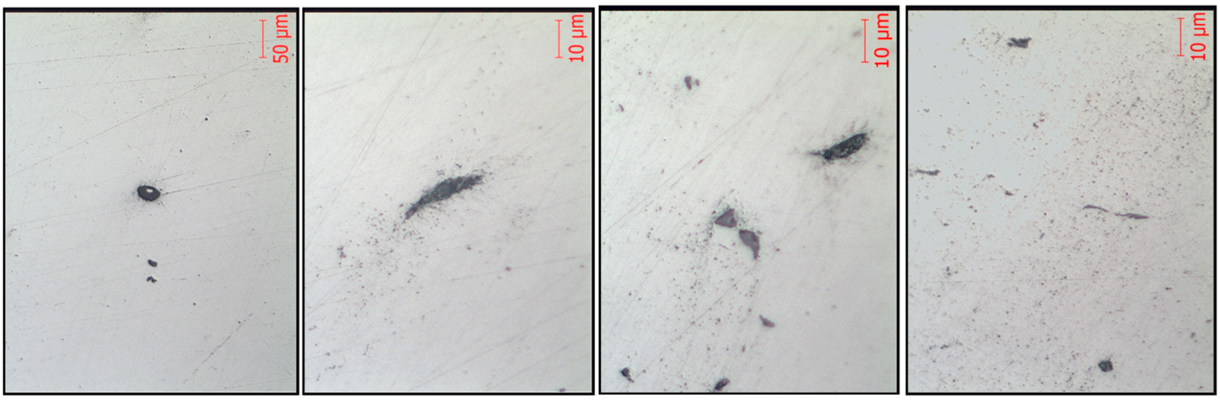
The microstructure of the material of samples, upon undergoing the HIP process and following the subsequent standard heat treatment, is characterized by a greater homogeneity due to samples' undergoing high-temperature homogenizing heatings, which ultimately led to the precipitation of noticeably smaller carbides along grain boundaries (Fig. 3, *f*, *h*) compared to samples that underwent only the heat treatment (Fig. 3, *b*, *d*).

Mechanical properties of samples (both vertical and horizontal) after being subjected to technological transitions under study are in compliance with the requirements set forth in the regulatory documentation for deformable materials (Table 2). Samples tested in the state of deposition (without the heat treatment) showed significantly higher (2–3 times) indicators of the impact strength of the material under study, while a decrease in strength indicators was registered in samples of both directions of deposition under study.

The heat treatment made it possible to increase both the strength and hardness of the material while reducing its impact strength and ductility. Note that horizontal samples have a higher level of impact strength, especially upon undergoing the HIP and the heat treatment processes.

Long-term strength indicators were studied in the heat-treated state to determine the compliance with the state of the material, which is foreseen by the regulatory documentation for deformable materials.

The research carried out established that the indicators above exceeded the requirements set forth in the regulatory documentation by more than 2–3 times, depending



a *b* *c* *d*

Fig. 5. Micropores (*a*, ×200), microdiscontinuities (*b*, ×1000), oxide inclusions (*c*, ×1000) and "healing" zones (*d*, ×1000) in samples made from EP648-VI alloy (SLM): *a*, *b*, *c* – state of deposition, *d* – HIP+ heat treatment

Table 2. Mechanical properties (average) of SLM samples made from EP648-VI alloy of studied options

Option	Deposition Direction	Mechanical Propertie at t = 20°C				
		σ_v, MPa	σ_{02}, MPa	δ, %	KCU, J/cm^2	HB, $d_{opp.}$, mm
In State of Deposition	XY	975.0	732.0	28.0	136.0	3.70
	Z	888.0	658.0	36.0	144.1	3.70
Heat Treatment	XY	1177.0	824.0	18.0	53.4	3.30
	Z	1139.0	810.5	22.0	41.0	3.25
HIP + Heat Treatment	XY	1197.0	755.0	26.0	70.5	3.50
	Z	1124.0	679.0	22.0	50.2	3.50
Norms set forth in regulatory documentation regarding deformable materials		≥ 780.0	≥ 345.0	≥ 25.0	≥ 34.0	–

Table 3. Indicators of long-term strength (average) of SLM samples made from the EP648-VI alloy of studied options

Option	Deposition Direction	Long-Term Strength		
		$T_{testing.}$, °C	σ, MPa	Time to fracture, τ, h
Heat Treatment	XY	800	176	69.5
	Z			104.5
Norms set forth in regulatory documentation regarding deformable materials	–	800	176	≥ 30

on the direction of the sample deposition under study (see Table 3). At the same time, samples deposited in the vertical direction demonstrated higher time-to-fracture values.

In case with the production of the EP648-VI alloy as per the traditional process, an increase in heat-resistant and strength characteristics is achieved due to the separation of finely dispersed carbides along grain boundaries and the intermetallic γ′-phase coherently associated with the alloy matrix. Note that, since the γ′-phase volumetric content in the alloy is small (6–8%), the dispersed α-Cr phase, whose finely needle-shaped precipitations are uniformly distributed throughout the volume, will certainly contribute to the overall strengthening of the alloy. Note that the formation of large and coarse deposits can lead to a decrease in mechanical properties of the material, primarily the ductility and the heat resistance. Therefore, regulating the degree of dispersion of this phase in the alloy structure requires carefully controlling the introduction of alloying elements into the melt (especially Cr), and complying with temperature-time regimes of the HIP process and the heat treatment.

Conclusion

The paper analyzed results of studying the microstructure and mechanical properties of samples obtained in the horizontal and vertical directions after various technological transitions by means of using the selective laser melting process. The authors studied mechanical characteristics of the SLM samples and compared them with the requirements for deformable semi-finished products made from the XH50VMTJuB-VI (EP648-VI) alloy.

The study showed that the SLM process followed by the hot isostatic pressing and the heat treatment ensures the following processes in the samples' structure: the formation of the structure of a fine-grained homogeneous γ-solid solution; the distribution of fine carbides along grain boundaries; and the distribution of dispersed precipitates of the intermetallic γ′-phase and the α-Cr phase.

Moreover, the use of hot isostatic pressing made it possible to significantly reduce the number of internal defects (pores, looseness, discontinuities), which, combined with the subsequent heat treatment, enabled the obtaining of the level of mechanical properties of the in the SLM material, which are posed to deformable semi-finished products made from the EP648-VI alloy in accordance with traditional manufacturing processes. The study registered an increase in the level of impact strength in samples that underwent the HIP, and a long-term strength in samples manufactured in the vertical direction compared to other studied options.

Despite a plethora of research work on the influence of additive processes on the structure and properties of traditional aviation materials, the introduction of additive processes and their application in the technological chain of manufacturing aircraft parts require a large-scale R&D and testing campaign to confirm the stability of characteristics for the subsequent procedure of the mandatory certification of manufacturing processes as per the requirements set forth in aviation regulations.

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Структура та властивості зразків зі сплаву ХН50ВМТЮБ-ВІ (ЕП648-ВІ) виготовлених технологією селективного лазерного плавлення

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Анотація. *У статті досліджено склад, структуру та властивості зразків зі сплаву ХН50ВМТЮБ-ВІ (далі ЕП648-ВІ), отриманих методом селективного лазерного плавлення (СЛП-процес), при виготовленні деталей авіаційного призначення. Авторами проведено порівняльне дослідження структури та властивостей зразків після проведення таких операцій, як осадження в двох напрямках (горизонтальному та вертикальному), роздільна термічна обробка та після гарячого ізостатичного пресування (ГІП) з подальшою стандартною термічною обробкою, що застосовується для деформівних напівфабрикатів зі сплаву ХН50ВМТЮБ-ВІ (ЕП648-ВІ). Автори повідомляють, що для виготовлення зразків за допомогою СЛП-процесу використовували порошки, отримані за технологією вакуумно-індукційного розпилення струменя розплавленого металу інертним газом (аргоном). Встановлено, що найкращий комплекс механічних властивостей продемонстрували зразки, отримані за допомогою ГІП-процесу із застосуванням термічної обробки (вакуумна високотемпературна гомогенізація з подальшим тривалим старінням), оскільки реалізований комплексний процес забезпечив "заліковування" пор і розривів у структурі, зміцнення за рахунок інтерметалідної γ'-фази, а виділення надлишкової голкоподібної α-Cr-фази є тонкими і рівномірно розподіленими в структурі матеріалу. Автори відзначили, що механічні властивості досліджуваних зразків в цілому відповідають вимогам нормативної документації до деформівних напівфабрикатів зі сплаву ХН50ВМТЮБ-ВІ (ЕП648-ВІ), підкресливши при цьому підвищення рівня ударної в'язкості зразків, які пройшли процес ГІП, і тривалої міцності зразків, виготовлених у вертикальному напрямку, в порівнянні з іншими досліджуваними варіантами. За результатами аналізу автори встановили, що СЛП-процес дозволив виготовити вироби, рівень механічних властивостей яких був близький до рівня деформівного матеріалу, а в деяких випадках навіть перевищував його.*

Ключові слова: *адитивні процеси, селективна лазерна плавка, гаряче ізостатичне пресування, вакуумна термообробка, жароміцний нікелевий сплав, γ'-фаза, α-Cr фаза.*