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INFLUENCE OF LONG-TERM SOAKING ON THE STRUCTURE AND PROPERTIES OF IN625 ALLOY SAMPLES MADE BY SELECTIVE LASER MELTING

N.O. Lysenko, O.O. Pedash, V.V. Klochykhin, P.O. Kasai

JSC "MOTOR SICH", 69068, Zaporizhzhya, Ukraine

ABSTRACT

The paper presents the results of studying the influence of long-term soaking on the structure and properties of samples made by selective laser melting of Inconel 625 alloy powders, produced by the method of off-center plasma spraying of a rapidly rotating rod billet (PREP-process). Investigations of chemical composition, macro- and microstructure, mechanical and heat-resistant properties of the samples plotted in *xy* and *z* direction was performed. Samples after building and hot isostatic pressing with subsequent heat treatment by different variants were to be examined. Investigation results showed that performance of the operation of hot isostatic pressing of the samples with subsequent standard heat treatment promotes practically complete elimination of microporosity in inner volumes of the metal and obtaining a more uniform solid solution. It was established that long-term soaking at the temperature of 900 °C, facilitates lowering of ductile and heat-resistant properties of Inconel 625 alloy due to lamellar precipitates of δ-phase. Performed study was the basis for recommending the recovery heat treatment mode. In addition, it is shown that long-term soaking at the temperatures of 700 and 980 °C does not lead to precipitation of lamellar δ-phase.

KEY WORDS: heat-resistant alloy; additive technologies; selective laser melting; hot isostatic pressing; heat treatment

INTRODUCTION

Development of additive technologies, in particular those of selective laser melting (SLM), allows manufacturing metal products of a complex geometrical shape by 3D compacting directly from a computer model by layer-by-layer deposition of metal powder and its further meting. Additive technology is the general name of a group of technologies, which envisage manufacturing products by digital models by layer-by-layer addition of material [1–4]. The interest to additive technologies and direct "printing" or growing of metal parts, as an alternative to traditional technologies, arose first of all in aviation, space industry, medicine and power engineering. Here, economic activity was the main driving factor. This is particularly true for single complex products, moulds with intricate cooling channels, where manufacturing by the traditional methods is considerably more expensive, than using additive technologies [1]. In addition to lower cost of manufacturing unique products, additive technologies allow an essential reduction of the time, consumed in manufacturing the finished product. There is no need to make sophisticated fixtures. Proceeding from that, the time from the moment of creating the model to obtaining the finished part is reduced from several weeks to several days.

In this work, the influence of long-term soaking at different temperatures on the structure and properties of Inconel alloy (IN625) was studied on samples made by 3D printing (selective laser melting).

MATERIALS AND METHODS OF INVESTIGATION

Used as the object of study were samples from Inconel 625 alloy for testing mechanical properties and microstructure, produced by selective laser melting in 3D unit of EOS GmbH Company (Germany), with working chamber dimensions ($x \times y \times z$) of 400×400×400 mm, fitted with ytterbium laser of 1000 W power.

The powder was made by the method of off-center spraying of rapidly rotating billet (PREP-process). The fractions of granules used for making the samples were equal to 20–50 μm.

Laser sintering of powders is a multiple repetitive process which includes several stages: deposition of a powder layer and its leveling with a roller; laser processing (scanning) of the powder layer with full penetration of the powder mixture according to the geometry of the initial 3D model; cleaning of the produced layer; moving the platform with the part downwards by one layer thickness; repetition of the entire process, i.e. deposition of the next powder layer, laser scanning, etc. Processing is performed in the chamber with inert gas purging and is controlled by the computer to obtain the specified 3D geometry of the part.

The made samples were both cylindrical of 14 mm diameter, and of rectangular cross-section with

Table 1. Modes of heat-treatment of 3D samples after HIP

Variant	Homogenizing I		Aging		Homogenizing II	
	T, °C	τ, h	*T*, °C	τ, h	*T*, °C	τ, h
1	1190	1	–	–	–	–
2	Same	Same	900	16	–	–
3	–»–	–»–	Same	700	–	–
4	–»–	–»–	–»–	1000	–	–
5	–»–	–»–	–»–	2100	–	–
6	–»–	–»–	–»–	Same	1000	1
7а	–»–	–»–	–»–	1000	1180	Same
7б	–»–	–»–	–»–	2100	Same	–»–
8	–»–	–»–	700	700	–	–
9	–»–	–»–	980	1400	–	–

16×6 mm dimensions. Sample length was 65 mm. Sample building on 3D printer was performed in *xy*, as well as in *z* direction. To eliminate internal porosity and improve the sample density, the process of selective laser melting was followed by hot isostatic pressing (HIP) in hot isostatic press QIH 09×1.5-2070-1400 MURC (QUINTUS Company, Sweden). HIP was conducted in the following mode: heating up to 1160±10 °C temperature, soaking time of 3 h, 160 MPa working gas pressure in the high-pressure vessel. Sample cooling was performed with application of the function of high-rate uniform cooling.

After HIP samples were heat-treated in vacuum furnace IPSEN T²T in the dynamic vacuum environment, cooling was conducted in the flow of inert gas (argon).

Modes of sample heat treatment after gas stating are given in Table 1.

After treatment by different variants the billets were machined to ensure the dimensions, envisaged by the technical documentation for making samples for mechanical and high-temperature strength testing.

The alloy chemical composition was determined by the methods of spectral and chemical analysis. Mechanical properties (σ_t, $\sigma_{0.2}$, δ, ψ) of the samples were tested in tensile testing machine ZDMY 30 for correspondence to the requirements of AMS 7000 standard.

Impact toughness (*KCU*) was determined on impact samples, tested in pendulum testing machine Instron SI-1M.

Hardness was determined by Brinell method in LECO AMH-43 instrument.

The time to high-temperature destruction (τ_d) (characteristic which is determined at long-term strength testing) of the samples was found in Instron M3 unit at the temperature of 700 °C and constant loading of 333 MPa. At long-term strength testing the samples were taken to destruction.

Fractographic study of powder morphology, as well as fractures of rupture and impact samples after testing for mechanical properties was conducted in binocular microscope STEMI 2000-C and electron scanning microscope JEOL JSM 6360LA.

Microstructural studies were performed on unetched and etched microsections using Axio Observer. Dlm microscope.

ANALYSIS AND DISCUSSION OF THE OBTAINED RESULTS

Microstructural examination revealed that the powder used to obtain samples by 3D laser printing (SLP), is characterized by a practically complete absence of satellite granules, and good sphericity with a small number of irregularly shaped particles (Figure 1).

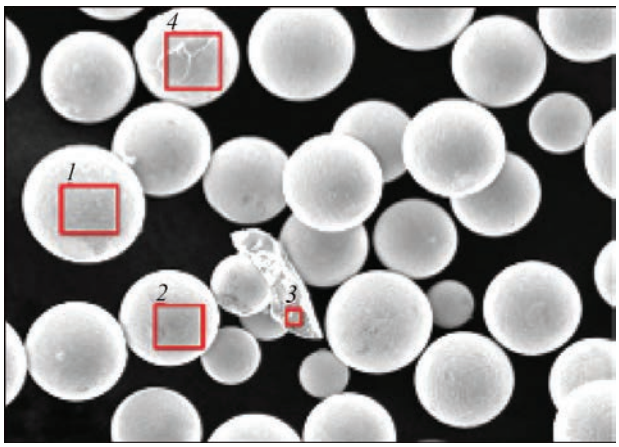

Figure 1. Morphology of IN625 alloy powders

Chemical composition of the made samples is satisfactory and meets the requirements of AMS 7000 for the studied alloy (Table 2).

IN625 alloy is a typical example of alloys with a low hardening (or non-hardenable alloys). The alloy is mainly used at up to 1000 °C temperatures, where oxidation resistance is the most important requirement [5]. More over, at temperatures above 550 °C, this alloy can undergo essential changes of the microstructure and mechanical properties [6].

It is found that mechanical properties of the samples grown in *xy* and *z* directions (variants 1, 2), meet

Table 2. Chemical composition of Inconel 625 samples, wt.%

Alloy grade	Element content,%						
	C	Cr	Al	Ti	Nb	Mo	Fe
IN625	0.03	21.20	0.20	0.20	3.88	8.55	3.03
AMS 7000 norms	≤ 0.10	20.0–23.0	≤0.4	≤0.4	3.15–4.15	8.0–10.0	≤5.00
Note. The balance is Ni.							

Table 3. Mechanical properties of IN625 alloy at T = 20 °C

Heat-treatment variant	Direction of sample building	σ_t, MPa	$\sigma_{0.2}$, MPa	δ, %	ψ, %	*KCU*, J/cm^2	*HB*
1	*xy*	882	438.5	56.4	57.7	281.3	201
	z	869	433.5	57.0	59.0	308.7	207
2	*xy*	881	448.0	48.4	53.9	281.3	Same
	z	866	433.0	52.0	53.7	308.7	–»–
AMS 7000 norms		≥827	≥344.7	≥30.0	–	–	–

the requirements of AMS 7000 for IN625 alloy. Values of relative elongation (δ) are approximately 2 times higher than the requirements of AMS 7000 (Table 3).

Long-term strength (T_{test} = 700 °C; σ = 333 MPa) of the samples after heat treatment by the 1st and 2nd variant (see Table 1) is at approximately the same level (Table 4); here the time to fracture (τ_{fr}) is equal to 437.5–655.4 h; in keeping with AMS 7000 τ_{fr} is not regulated.

It should be noted that performance of aging at the temperature of 900±10 °C for 16 h (variant 2) did not lead to any noticeable increase of the mechanical or high-temperature properties.

Metallographic examination showed that the sample microstructure in the condition after building (before performance of HIP and heat treatment operations of both vertical (*z*), and horizontal (*xy*) ones) is γ-solid solution of Ni–Cr–Mo–Nb–Fe with the presence of carbides and carbonitrides, and it corresponds to non-heat-treated state of IN625 alloy, manufactured by the method of 3D printing (Figure 2, *a*), A structural inhomogeneity is observed in the initial samples, which is due to formation of zones of layer-by-layer fusion, as well as thin dendrites elongated in the direction of sample growing, which formed due to high rates of heating and cooling, which are in place during the processes of melting and solidification in a short time.

Table 4. High-temperature strength properties of IN625 alloy

Heat-treatment variant	Direction of sample building	Long-term strength		
		T_{test}, °C	σ, MPa	Time-to-fracture (τ_{fr}), h
1	*xy*	700	333	446^{15}
	z	Same	Same	627^{20}
2	*xy*	–»–	–»–	517^{50}
	z	–»–	–»–	590^{30}

During high-temperature heating at HIP (1160±10 °C) and heat-treatment (1190±10 °C), a more uniform structure forms due to solid solution homogenizing as a result of equalizing of the chemi-

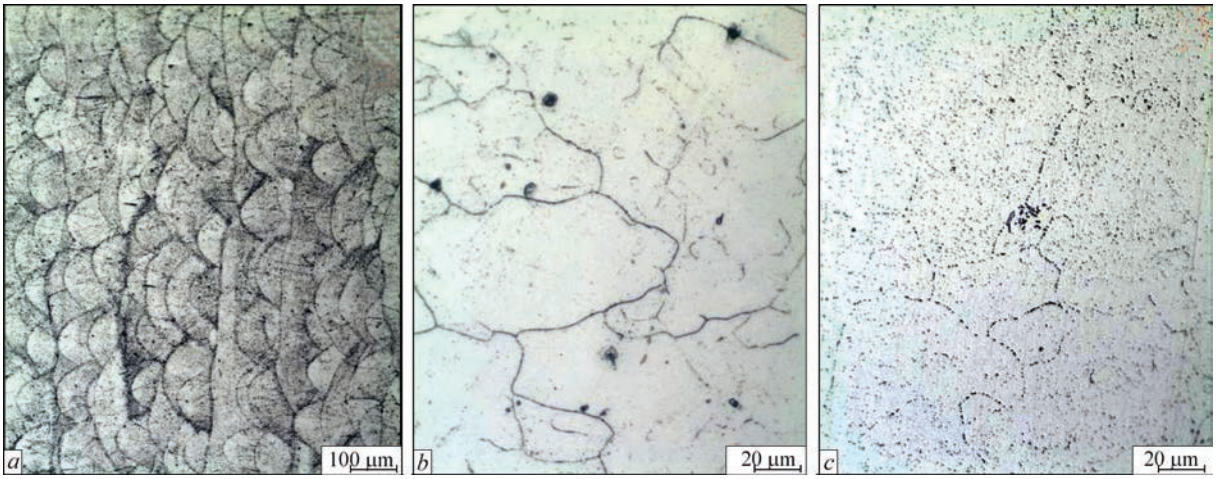

Figure 2. Microstructure of 3D samples from IN625 alloy: *a* — initial condition, ×100; *b* — variant 1, ×500; *c* — variant 2, ×500 (see Table 1)

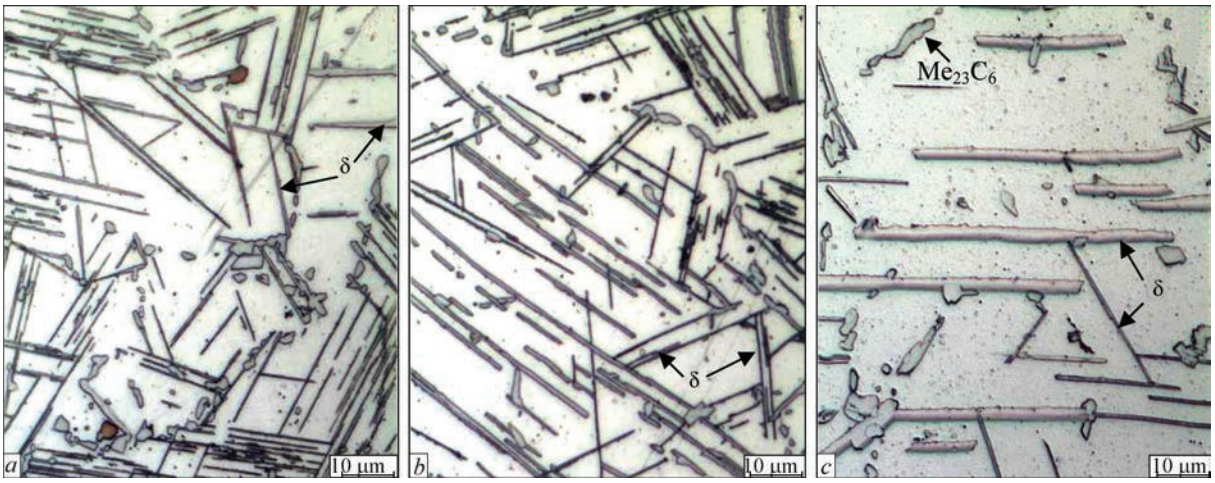

Figure 3. Microstructure of 3D samples of IN625 alloy after heat-treatment, ×1000: *a* — variant 3; *b* — variant 4; *c* — variant 5 (see Table 1)

cal composition between layer-by-layer fusion zones (Figure 2, *b*).

At heat-treatment by variant 2 (HIP + (homogenizing + aging at 900 °C, 16 h)) precipitation of hardening intermetallic γ″ (Ni_3Nb)-phase with BCC structure and slight coagulation of carbides of $M_{23}C_6$ type (Figure 2, *c*) are observed (Figure 2, *c*).

Lamellar precipitates of δ-phase, as well as structures, characteristic for overheated state of IN625 alloy in the form of partial melting along the grain boundaries, were not revealed in the initial samples heat-treated by the 1st and 2nd variants.

Performance of HIP operation by the above-given mode promotes practically complete "healing" of pores and microdiscontinuities, concentrated in the inner volumes of metal. Here, globular and (or) thin film oxides were found in the "healing" zones. Dimensions of oxide inclusions in the studied sample material were, mainly, equal to 7 μm (isolated, rarely found ones were up to ~ 20 μm). Carbides and carbonitrides precipitate in the form of discrete globular particles of approximately 5 μm size.

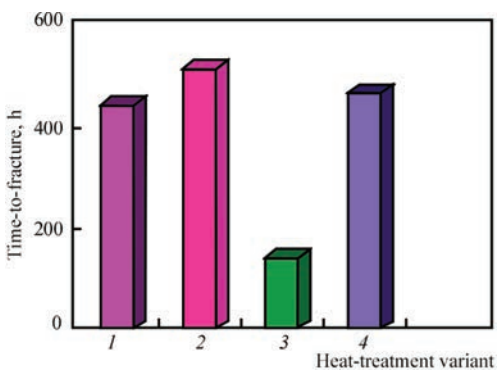

Figure 4. Long-term strength of 3D samples of IN625 alloy, depending on heat-treatment variant: *1* — variant 1; *2* — variant 2; *3* — variant 4; *4* — variant 7a (see Table 1)

Conducting long-term soaking at the temperature of 900 °C (variants 3, 4, 5) promoted precipitation of a considerable amount of lamellar particles of δ-phase from γ-matrix (Figure 3). With longer soaking time the number and dimensions of δ-phase plates increased: at soaking for 700 h (variant 3) the plate width reached approximately 0.85 μm (Figure 3, *a*); at soaking for 1000 h (variant 4) plates of the width of approximately up to 1.5 μm (Figure 3, *b*) were found; at soaking for 2100 h (variant 5) the maximum width of the plates is equal to approximately 3.75 μm (Figure 3, *c*).

It should be also noted that the material of the samples processed by variants 3–5, demonstrates coarsening of the structure as a result of growth and coagulation of carbides of $Cr_{23}C_6$ type, particularly, on grain boundaries. Precipitation of particles of hardening intermetallic γ-phase is negligible.

Testing samples treated by the 4th variant showed that long-term soaking (1000 h) at the temperature of 900 °C leads to lowering of the ductile characteristics (δ, ψ — 1.5 times, *KCU* — ~6 times) at a certain increase of strength (σ_t, $\sigma_{0.2}$) and hardness (*HB*) of the material, compared with the values, obtained after heat-treatment by variants 1 and 2 (Table 5).

Long-term strength of samples heat-treated by variant 4 (with soaking for 1000 h at aging at 900 °C), decreased more than 3 times (Figure 4).

In order to increase the ductility and high-temperature strength of IN625 alloy, and eliminate unfavourable phases (lamellar and large near-boundary carbides) formed during long-term soaking at 900 °C temperature, recovery heat treatment operations were performed at temperatures of 1000 °C (variant 6) and 1180 °C (variants 7a, 7b) with soaking for 1 h.

Fragmentation and thinning of δ-phase plates, as well as partial dissolution of δ-phase and $Cr_{23}C_6$ car-

Table 5. Mechanical properties of IN625 alloy at $T = 20$ °C

Heat-treatment variant	Direction of sample building	σ_t, MPa	$\sigma_{0.2}$, MPa	δ, %	ψ, %	*KCU*, J/cm^2	*HB*
1	*xy*	882.0	438.5	56.4	57.7	281.3	201
4	–»–	912.0	467.0	38.0	33.4	49.0	241
7a	–»–	885.0	456.7	60.0	64.2	262.2	207
AMS 7000 norms		≥827.0	≥344.7	≥30.0	–	–	–

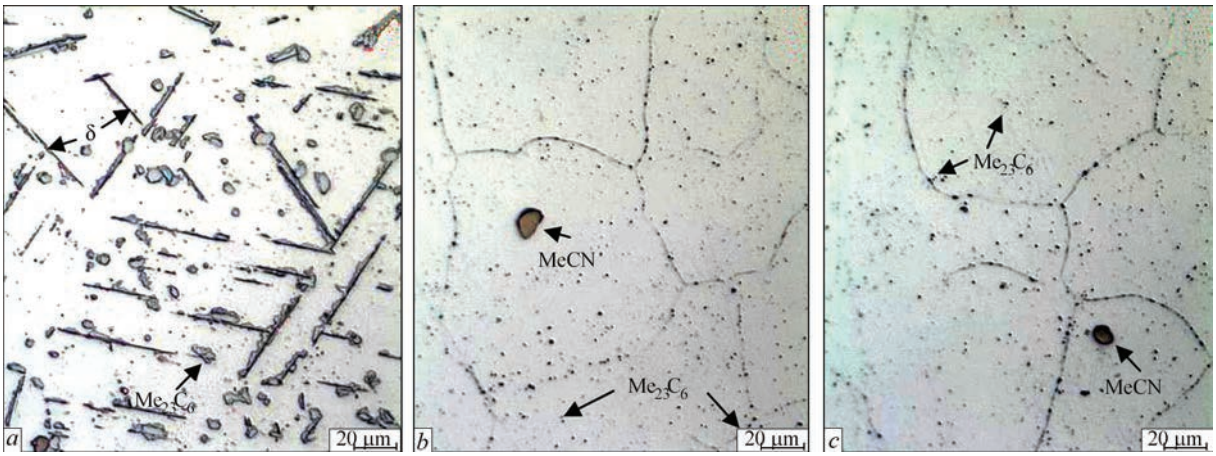

Figure 5. Microstructure of 3D samples of IN625 alloy after recovery heat-treatment, ×1000: *a* — variant 6 (variant 4 + heating at 1000 °C (1 h)); *b* — variant 7a (variant 4 + heating at 1180 °C (1 h)); *c* — variant 7b (variant 5 + heating at 1180 °C (1 h))

bides in γ-solid solution are observed in the microstructure of IN625 alloy samples, heat treated by variant 6 at 1000 °C temperature (1 h) (Figure 5).

Heat-treatment of samples by variants 7a, 7b at 1180 °C temperature (1 h) (after long-term soaking for 1000 h (variant 4) and 2100 h (variant 5)) promotes complete dissolution of lamellar δ-phase and $Cr_{23}C_6$ type carbides with further precipitation of these carbides in the form of fine discrete particles (Figure 5). The microstructure is Ni–Cr–Mo–Nb–Fe γ-solid solution, containing carbides, carbonitirides and a small quantity of intermetallic γ″-phase; it cor-

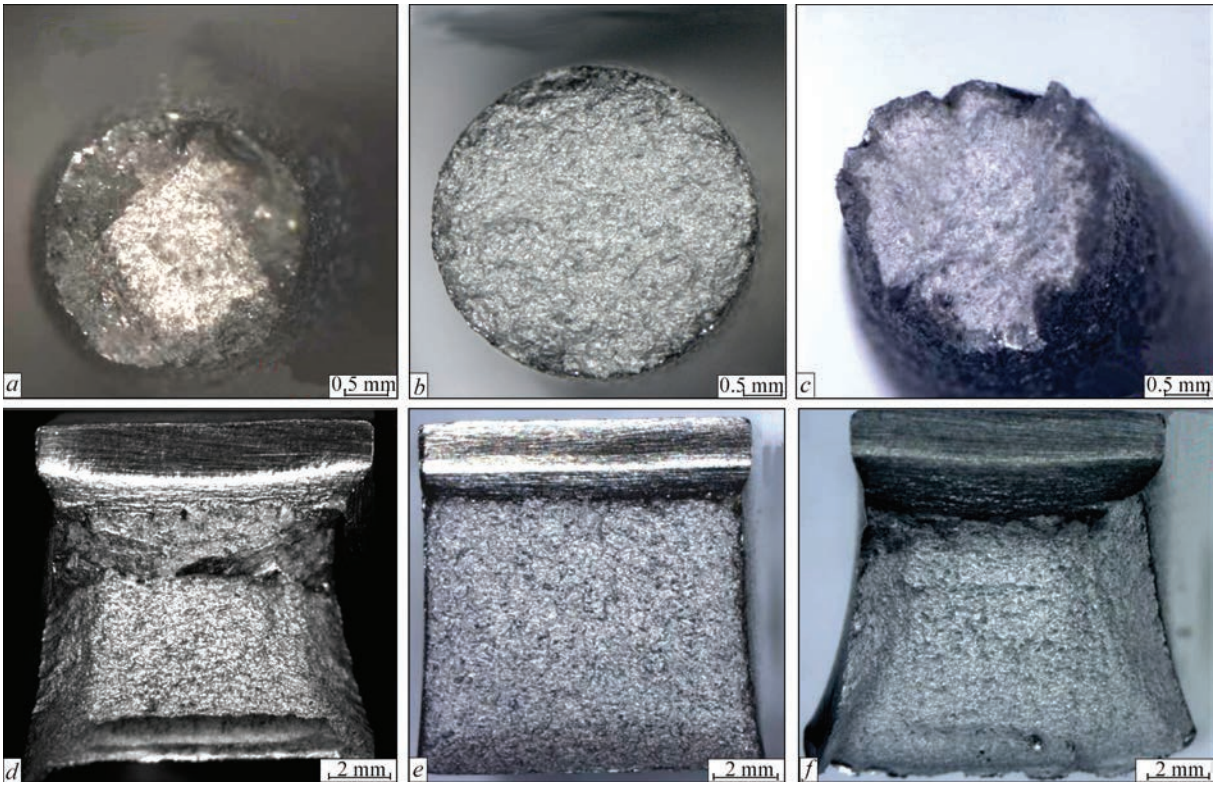

Figure 6. Fractographic structure of fractures of rupture (*a, b, c* — ×12.5) and impact (*d, e, f* — ×6.5) 3D samples of IN625 alloy: *a, d* — variant 1; *b, e* — variant 4; *c, f* — variant 7a

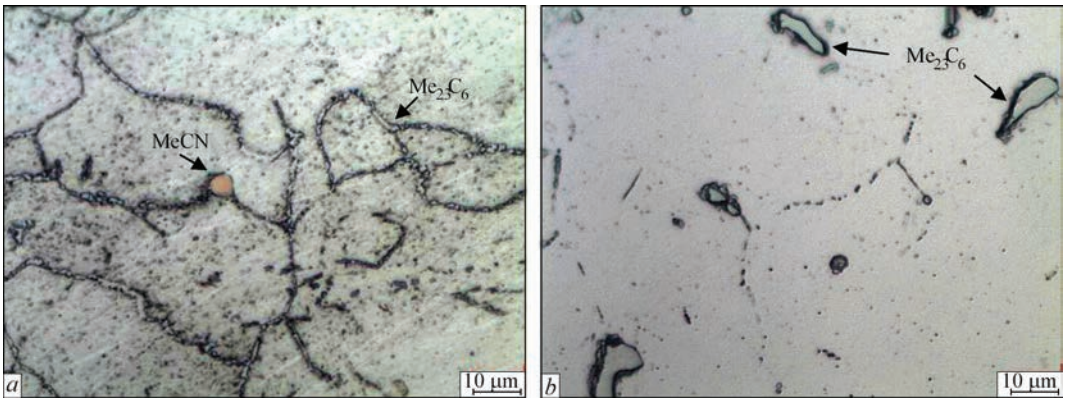

Figure 7. Microstructure of 3D samples of IN625 alloy after heat-treatment, ×1000: *a* — variant 8; *b* — variant 9 (see Table 1)

responds to homogenized state of IN625 alloy, and is similar to variant 1.

Figure 6 shows the structure of fractures of IN625 alloy samples (3D printing), heat-treated by variants 1, 4 and 7a, after tensile and impact toughness testing at room temperature. The cup-shaped structure of the fractures and formation of a "neck" in the destruction zone on rupture test samples and presence of beveling on impact test samples of variants 1 and 7a are indicative of a rather high ductility of the alloy (Figure 6, *a*, *c*, *d*, *f*). Practical absence of the "neck" in the destruction zone on rupture samples or beveling on impact samples treated by the 4th variant, is characteristic for material of a lower ductility (Figure 6, *b*, *e*).

As a result of the performed study, it was determined that heat treatment at 1180 °C temperature for 2 h (variants 7a and 7b) promotes recovery of mechanical and high-temperature strength properties that is due to homogenizing of IN625 alloy, at which dissolution of lamellar δ-phase and $Cr_{23}C_6$ type carbides in γ-solid solution is observed, with further precipitation of $Cr_{23}C_6$ type carbides and a small quantity of intermetallic γ″-phase in the form of fine discrete particles, strengthening the matrix.

In addition, the microstructural state of IN625 alloy samples produced by selective laser melting (3D printing) was studied after heat-treatment by variants 8 and 9 with long-term soaking at temperatures of 700 °C (700 h) and 980 °C (1400 h), respectively.

Metallographic examination of samples at up to ×1000 magnification showed that long-term soaking at temperatures of 700 °C (700 h, variant 8) and 980 °C (1400 h, variant 9) does not lead to formation of lamellar δ-phase. Here, it was noted that at treatment by 8th variant (700 °C, 700 h) coarsening of the structure takes place, mainly, due to growing and coagulation of $Cr_{23}C_6$ type carbides which precipitate predominantly on grain boundaries (Figure 7, *a*).

In samples treated by the 9th variant (980 °C, 1400 h) the grain boundaries are thin, and a considerable quantity of $Cr_{23}C_6$ type carbides are present in the form of coagulated particles of approximately 7–15 μm size, uniformly distributed in the metal volume (Figure 7, *b*).

CONCLUSIONS

1. Chemical composition of samples, made from IN625 alloy by 3D printing in EOS M400 unit, corresponds to AMS 7000 requirements for IN625 alloy.

2. It is found that long-term soaking at 900 °C temperature for 700 h (variant 3), 1000 h (variant 4) and 2100 h (variant 5) leads to lowering of ductility and high-temperature strength of IN625 alloy as a result of precipitation of a considerable number of lamellar particles of δ-phase from γ-matrix, as well as coarsening of the structure due to growth and coagulation of $Cr_{23}C_6$ type carbides, particularly on grain boundaries. Increase of soaking time promotes increase of the number and dimensions of δ-phase plates, as well as coarsening of the grain boundaries.

3. Heat treatment of samples with lower properties, presence of a large number of δ-phase plates and coarsening of the grain boundaries (variants 4, 5) at 1180 °C temperature for 1 h (variants 7a and 7b) promotes recovery of mechanical and high-temperature properties that is due to homogenizing of IN625 alloy, at which dissolution of lamellar δ-phase and $Cr_{23}C_6$ type carbides in γ-solid solution are observed with further precipitation of $Cr_{23}C_6$ type carbides and a small quantity of intermetallic γ″-phase in the form of fine discrete particles which strengthen the matrix.

4, Long-term soaking at temperatures of 700 °C (700 h, variant 8) and 980 °C (1400 h, variant 9) does not lead to lamellar phase formation.

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ORCID

N.O. Lysenko: orcid.org/0000-0003-2319-1260,
O.O. Pedash: orcid.org/0000-0003-1231-9951,
V.V. Klochykhin:orcid.org/0000-0002-0754-5543,
P.O. Kasai: orcid.org/0000-0003-2181-8381

CONFLICT OF INTEREST

The Authors declare no conflict of interest

CORRESPONDING AUTHOR

N.O. Lysenko
JSC “MOTOR SICH”, 69068, Zaporizhzhya, Ukraine. E-mail: tb.ugmet@motorsich.com

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