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## Features of the impact of hot isostatic pressing and heat treatment on the structure and properties of maraging steel obtained by selective laser melting method

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**Abstract:** Using the SLM method in a nitrogen blanket with heating to a temperature of 200 °C, samples were obtained at a position of 0° against the build plate. The effect of the hot isostatic pressing (HIP) and heat treatment (HT: hardening + aging) on the structure and mechanical properties of maraging steel CL50 WS was studied (the Russian analogue is ChS4 grade). To analyze the effect of post-processing on the strength characteristics ( $\sigma_b$ ,  $\sigma_{0.2}$ ,  $\delta$ ,  $\psi$ ), tensile tests were conducted. Their results indicated high values of strength and ductility. It has been established that as a result of HT in the steel structure, in addition to  $\alpha$ -Fe,  $\gamma$ -Fe, dispersed precipitates of the  $\text{NiTi}_3$  strengthening phase are formed, the identification of which was carried out by high-resolution transmission electron microscopy. Through the  $\text{NiTi}_3$  intermetallic phase, the steel has acquired increased tensile strength and yield strength required for the production of critical components and parts for highly loaded turbomachine disks. The change in the porosity of the samples before and after the HIP was analyzed. The microstructure of the samples and the changes that occur under the influence of various post-processing options are studied. The fine-grained homogeneous structure obtained by combining the SLM, HIP and HT provided optimal strength and ductility. Analysis of fractures after mechanical testing showed that the samples after post-processing are destroyed according to the viscous-pitting mechanism with the formation of a neck.

**Keywords:** selective laser melting, maraging steels, HIP, heat treatment, microstructure, phase composition, mechanical properties.

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## Особенности влияния горячего изостатического прессования и термообработки на структуру и свойства мартенситно-старееющей стали, полученной методом селективного лазерного сплавления

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**Аннотация:** Методом селективного лазерного сплавления (СЛС) в среде азота при подогреве до температуры 200 °C получены образцы в положении 0° относительно плиты построения. Изучено влияние горячего изостатического прессования (ГИП) и термообработки (ТО: закалка + старение) на структуру и механические свойства мартенситно-старееющей стали CL50 WS (российский аналог — ЧС4). Для анализа влияния постобработки на прочностные характеристики ( $\sigma_b$ ,  $\sigma_{0.2}$ ,  $\delta$ ,  $\psi$ ) проведены испытания на разрыв. Их результаты показали высокие значения прочности и пластичности.

Установлено, что в результате ТО в структуре стали, помимо  $\alpha$ -Fe,  $\gamma$ -Fe, образуются дисперсные выделения упрочняющей фазы  $\text{NiTi}_3$ , идентификацию которой проводили методом просвечивающей электронной микроскопии высокого разрешения. Благодаря интерметаллидной фазе  $\text{NiTi}_3$ , сталь приобрела повышенные предел прочности и предел текучести, требуемые для производства ответственных узлов и деталей высоконагруженных дисков турбомашин. Проанализировано изменение пористости образцов до и после ГИП. Исследованы микроструктуры образцов и изменения, происходящие под влиянием различных вариантов постобработки. Мелкозернистая однородная структура, полученная при сочетании СЛС, ГИП и ТО, обеспечила оптимальные показатели прочности и пластичности. Анализ изломов после механических испытаний показал, что образцы после постобработки разрушаются по вязко-ямочному механизму с образованием шейки.

**Ключевые слова:** селективное лазерное сплавление, мартенситно-стареющие стали, горячее изостатическое прессование, термическая обработка, микроструктура, фазовый состав, механические свойства.

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## Introduction

High-strength maraging steel (MS) belongs to the group of high-alloy steel grades that are based on carbon-free martensite. Compared to classic carbon-free steels, maximum hardening is achieved by heat treatment (HT) in the aging mode due to the dispersion hardening of highly ductile martensite.

The worldwide experience in the application and operation of MS steels has proven that they provide a high degree of reliability, manufacturability and other advantages in comparison with carbon steels. The low hardness of low-carbon martensite contributes to good machinability and deformability in the initial and hardened states, while aging provides a high level of strength, ductility and permanent deformation. The hardening heat treatment of complex shaped thin-walled parts is accompanied by a small change in linear dimensions, i.e. no warpage is caused.

MS steels exhibit the feature of throughout hardenability at the austenitization temperature, i.e. martensitic structure formation is assured regardless of the cooling rate and cross-sectional dimension of a finished product or a part. Such steels are well deformed without heating during sheet stamping and rotary drawing and forging.

MS steel of ChS4 grade with strength  $\sigma_V = 1950 \div \pm 2150$  MPa has increased ductility, especially under conditions of local deformation, and, as a consequence, lower sensitivity to stress concentrators, which ensures

higher reliability during operation under extreme loads compared to high-strength carbon steels treated for the equivalent strength.

The structure of carbon-free martensite in all grades of MS steel, including ChS4, belongs to the type of massive martensite, which differs from carbon martensite in the absence of tetragonality in the  $\alpha$ -BCC lattice, high dislocation density, and the presence of a significant number of twins. This type of structure is not exposed to tempering and softening processes, but is only capable of intensive strengthening due to precipitation of dispersed phases.

In powder metallurgy, MS steel products are obtained from atomized alloy powders by thermoforming methods. This allows to reduce segregational heterogeneity as well as to ensure high strength properties. The parts produced by sintering exhibit high ductility, toughness and can be used under high-temperature contact conditions.

For the manufacture of geometrically-complex special-purpose products of MS steels, it is highly promising to use the technology of selective laser melting (SLM), which significantly reduces the production time and the material consumption. Interest in using MS steels in SLM technology is caused not only by a high complex of physical and mechanical properties, but also by almost complete absence of warping in the printing process due to the unique nature of the steel. The forma-

tion of the product during SLM is performed due to the successive melting and crystallization of layers of metal powder. Therefore, the formation of interfaces, which are a structural defect for this technology and may contain discontinuities, occurs.

The SLM process is characterized by such defects as an incomplete fusion of particles, a residual porosity, microcracks, high residual stresses, and a formation of supersaturated solid solutions. The presence of defects results in a decrease in the mechanical and operational properties of products. In this regard, the use of post-processing techniques, in particular hot isostatic pressing (HIP), is reasonable and economically feasible since it reduces residual porosity, heals structural defects, ensuring a fine-grained steel structure with the effect of dispersion hardening [1–8].

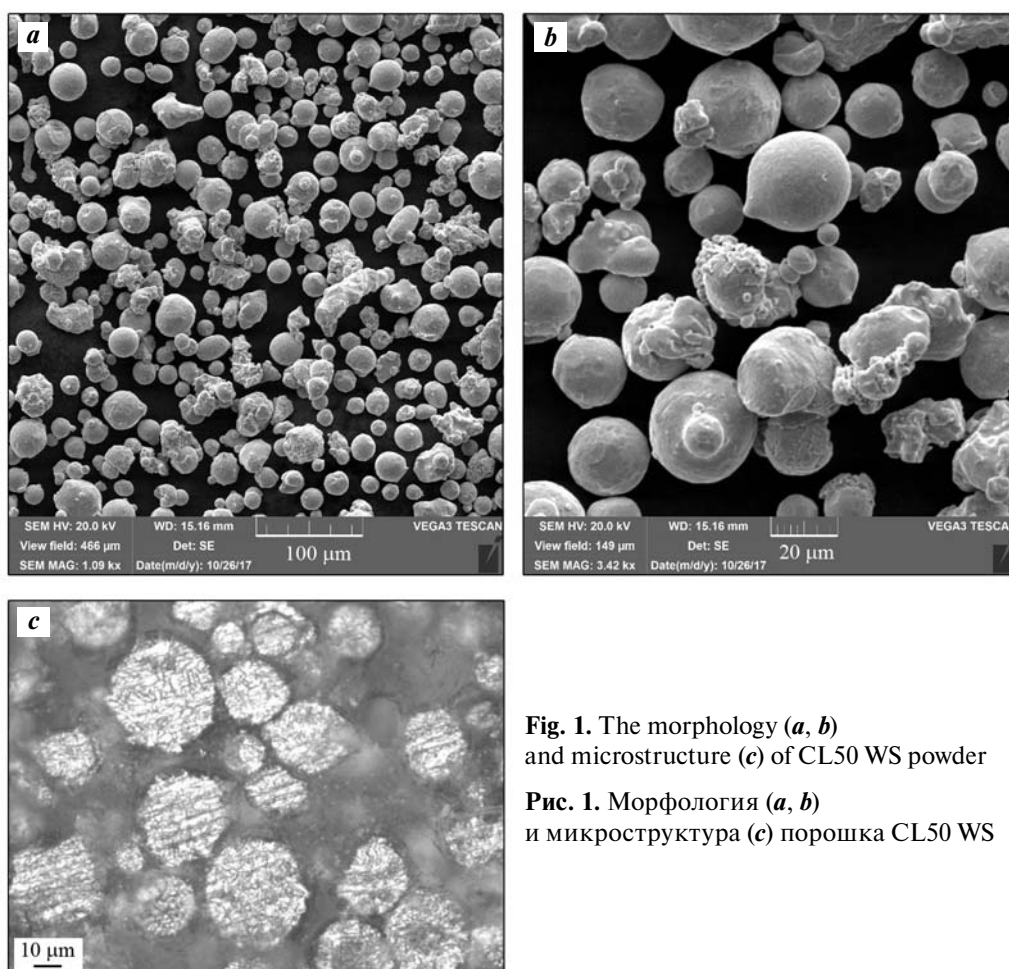
The purpose of this paper is to study the effect of HIP and heat treatment by aging on the structural phase transformations and properties of SLM samples made of maraging steel.

## Research methodology

The metal powder of maraging steel of CL50 WS grade (Germany) was used for the research. Its Russian analogue is steel of ChS 4 grade. The chemical composition of CL50 WS alloy is presented below, wt.%:

Fe .....	Base	Si .....	≤ 0.1
Mo .....	4.5–5.2	Mn .....	≤ 0.15
Ni .....	17.0–19.0	P .....	≤ 0.01
Ti .....	0.8–1.2	S .....	≤ 0.01
Co .....	8.5–10.0	Cr .....	≤ 0.25
C .....	≤ 0.03		

The concentrations of gas impurities in the powder for oxygen, nitrogen and hydrogen are 0.146, 0.021 and 0.0075 wt.%, respectively. The particle size of the powder is in the range of 5–45  $\mu\text{m}$ , while the distribution quantiles  $d_{10}$ ,  $d_{50}$  and  $d_{90}$  are 17.7, 29.4 and 48.0  $\mu\text{m}$ , re-



**Fig. 1.** The morphology (*a, b*) and microstructure (*c*) of CL50 WS powder

**Рис. 1.** Морфология (*a, b*) и микроструктура (*c*) порошка CL50 WS

spectively. The bulk density is  $4.544 \text{ g/cm}^3$ . The powder contains irregularly shaped particles up to  $50 \text{ }\mu\text{m}$  in size. There are satellites on the surface of individual particles (Fig. 1, *a, b*). The microstructure of the powder is represented by small dendritic crystals, no closed gas pores are detected (Fig. 1, *c*).

The samples were obtained on the «Concept Laser M2» machine (Germany) under a nitrogen blanket with the blanks being horizontally positioned against the build plate and subjected to the following process parameters: the thickness of the fused layer is  $30 \text{ }\mu\text{m}$ , the laser power is  $180 \text{ W}$ , the scanning speed is  $800 \text{ mm/s}$ , the temperature of the build plate during the printing process is  $200 \text{ }^\circ\text{C}$ . The control of blank samples was performed using XTH450 LC X-ray tomography system (Nikon Metrology, Japan) with a sensitivity of  $0.1 \text{ mm}$ .

The SLM samples were gasostated on ABRA HIRP 10/26-200-2000 unit (Sweden). The HIP mode consisted of heating to the hardening temperature and holding for 2 h at a constant pressure. Following the HIP, additional heat treatment was conducted in a chamber furnace with a blanketing atmosphere in two modes: HT1 — hardening followed by aging, HT2 — aging.

Porosity was determined by the layer-by-layer analysis in the cross section with the calculation of the average index for three layers with a step of  $3 \text{ mm}$ . Sections on samples for determining porosity and structural studies were made parallel (section YZ) and perpendicular (section XY) to the direction of synthesis.

To evaluate the mechanical properties of steel, cylindrical test-pieces were cut out of blanks for tensile tests (under GOST 1497-84, type IV, No. 8). The tests were conducted on «Shimadzu 100kN» unit (Japan), offset yield strength ( $\sigma_{0.2}$ ), tensile strength ( $\sigma_V$ ), percentage of elongation ( $\delta$ ) and percentage of reduction ( $\psi$ ) were determined.

The fractographic analysis of fractures was performed on «Vega 3» scanning electron microscope (Tescan, the Czech Republic), and the study of the microstructure was performed on «Hitachi S-3400N» scanning electron microscope (Japan). The fine structure was studied by transmission electron microscopy (TEM) method on JEM-2100 instrument (Jeol, Japan), including *in situ* at high resolution. Foils for TEM were prepared by ion etching in «PIPS II System» (Gatan, United States).

## Research results

The appearance of SLM blanks of MS steel is shown in Fig. 2.

The absence of discontinuities and cracks in the blanks was established using X-ray tomography (Fig. 3).

Hot isostatic treatment allowed to reduce the residual porosity from 0.43 to 0.20. The analysis of mechanical tests of samples in the state of SLM + HIP + HT1 demonstrated an increase in the yield strength by 58 % and tensile strength by 50 % relative to the state of SLM + HIP, and in the case of the state of SLM + HIP + HT2, the increase in these indicators was 58 % and 48 %, respectively. Thus, it has been established that heat treatment modes ensure optimal

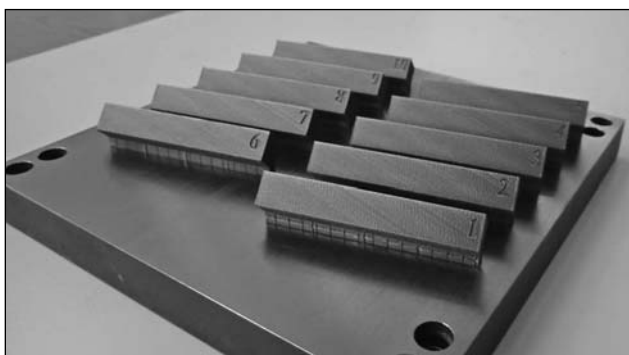


Fig. 2. The location of blanks on the build plate

Рис. 2. Расположение заготовок на плите построения

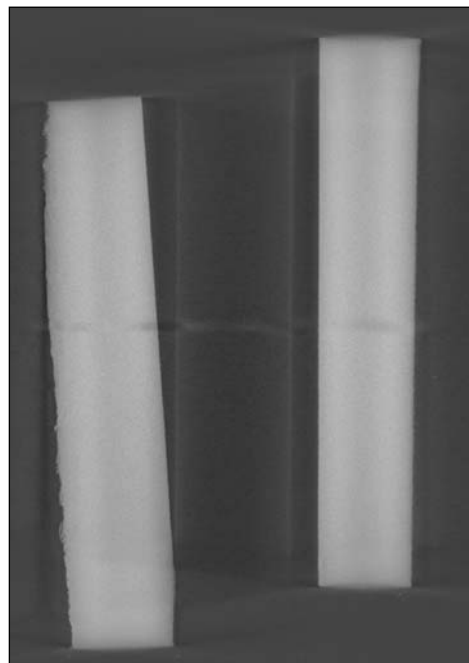


Fig. 3. The X-ray tomography of SLM samples

Рис. 3. Рентгеновская томография СЛС-образцов

indices of strength and ductility [9]. Fig. 4 shows the deformation curves for uniaxial tension of samples in the states of SLM + HIP + HT1, SLM + HIP + HT2, and SLM + HIP.

The samples subjected to post-processing under SLM + HIP + HT1 and SLM + HIP + HT2 modes are characterized by a uniform plastic deformation area and high strength and ductility.

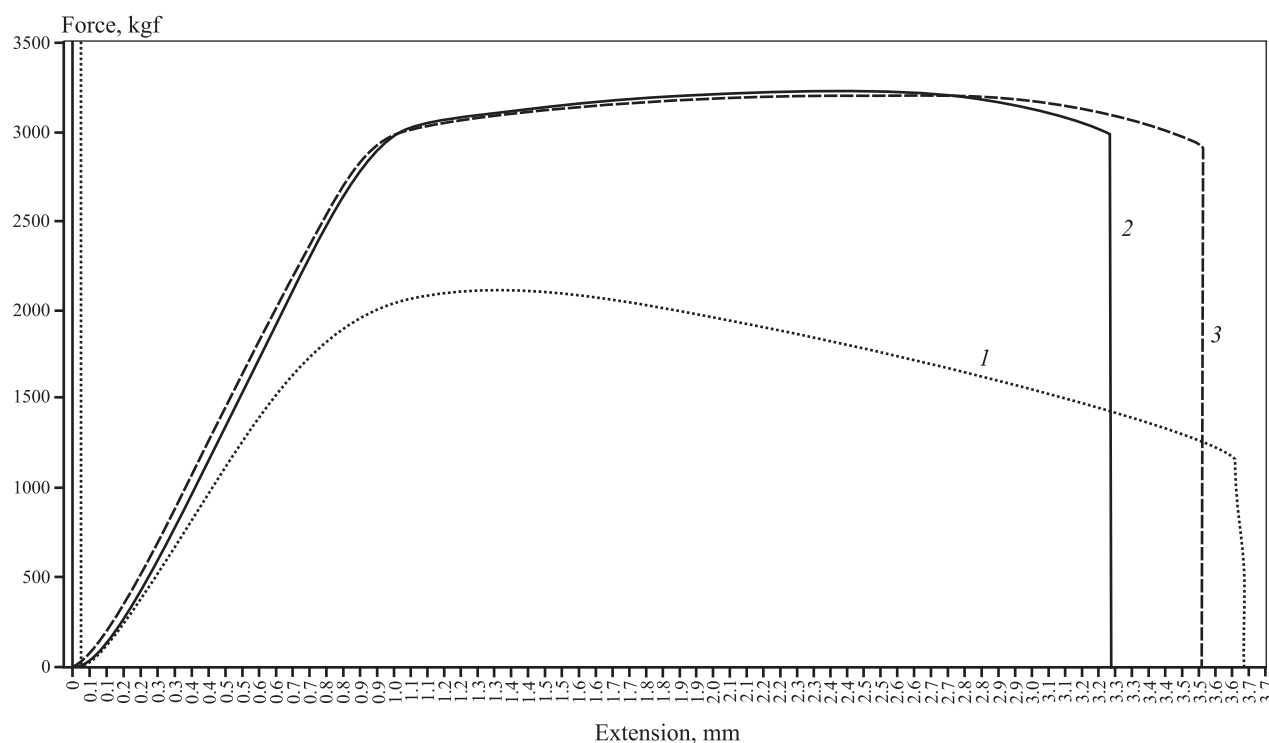
A noticeable increase in strength and ductility was also observed for the samples in SLM + HIP state ( $\psi = 58.9\%$ ). The obtained indices of percentage of reduction exceed the value for this steel in the state of hot-rolled and forged bar (TU 14-1-811-73,  $\psi = 40\%$ ).

In works [10–12], it is shown that the mechanical properties of MS steel of 18Ni300 grade (the analogue of steel of 01KhN18L9M5TYu grade) in SLM state are in the following intervals:  $\sigma_{0.2} = 500 \div 900$  MPa,  $\sigma_V = 800 \div 1100$  MPa,  $\delta = 10 \div 30\%$ ,  $\psi = 11 \div 25\%$ , in SLM + HT state ( $t = 425 \div 900$  °C) they are as follows:  $\sigma_{0.2} = 370 \div 1000$  MPa,  $\sigma_V = 700 \div 1200$  MPa,  $\delta = 15 \div 35\%$ ,  $\psi = 20 \div 50\%$ . Thus, the mechanical properties

obtained for MS steel of CL50 WS grade correspond to the international standard.

Fig. 5 shows the microstructure of SLM sample after HIP with high structural homogeneity. There is no sub-grain structure typical for SLM samples that indicates the completion of the grain recrystallization process during HIP.

Aging after gasostatic processing also results in the formation of a uniform martensitic structure, but with a smaller grain size (Fig. 6). As a result of «martensitic» aging, the alloying elements form a plastic matrix phase being a substituted martensite reinforced with uniformly distributed high-strength dispersed precipitates of the excessive  $\text{NiTi}_3$  intermetallic phase with an average crystallite size of  $10\ \mu\text{m}$ . The identification of this phase was confirmed by TEM method in an *in situ* study of structural transformations during heating of the lamella. The precipitation of  $\text{NiTi}_3$  phase from a supersaturated solid solution begins at a temperature of  $570\text{ °C}$ , which corresponds to the aging temperature (Fig. 7).

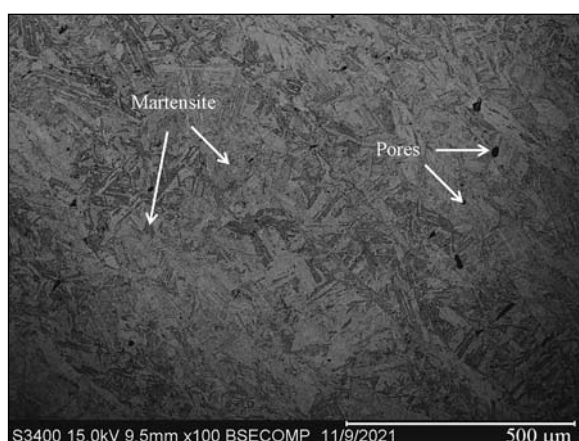


**Fig. 4.** The deformation curves in uniaxial tension of samples made of MS steel of CL50 WS grade depending on the type of post-processing

1 — SLM; 2 — SLM + HIP + HT1; 3 — SLM + HIP + HT2

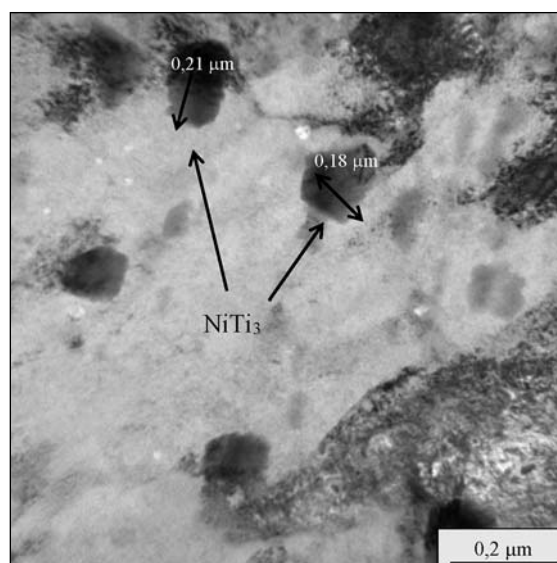
**Рис. 4.** Деформационные кривые при одноосном растяжении образцов из МС-стали CL50 WS в зависимости от вида постобработки

1 — СЛС; 2 — СЛС + ГИП + ТО1; 3 — СЛС + ГИП + ТО2



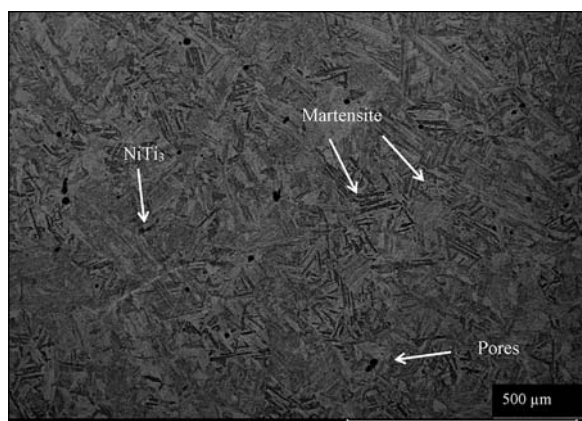
**Fig. 5.** The microstructure of MS steel in SLM + HIP state

**Рис. 5.** Микроструктура МС-стали в состоянии СЛС + ГИП



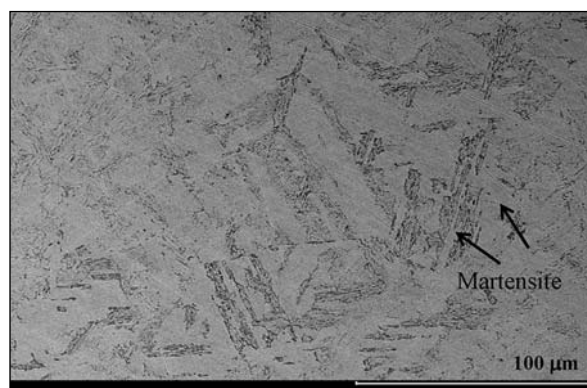
**Fig. 7.** TEM image of the precipitated phase

**Рис. 7.** ПЭМ-изображение выделившейся фазы



**Fig. 6.** The microstructure of MS steel in the state of SLM + HIP + HT2

**Рис. 6.** Микроструктура МС-стали в состоянии СЛС + ГИП + ТО2



**Fig. 8.** The microstructure of SLM sample after HIP + HT1

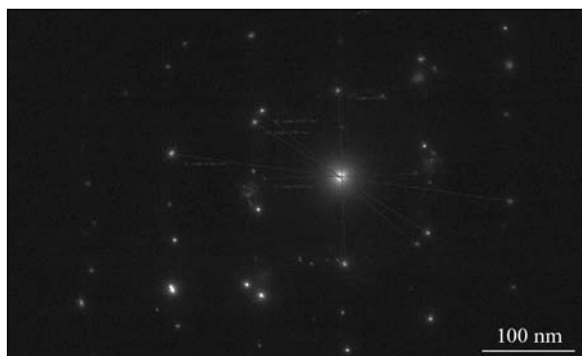
**Рис. 8.** Микроструктура СЛС-образца после обработки ГИП + ТО1

Post-processing under HIP + HT1 mode also resulted in the formation of a hardening phase, however, additional hardening after HIP contributed to the coarsening of the martensitic structure (Fig. 8).

The studies of the fine structure of SLM samples by TEM method revealed the grains of  $\text{NiTi}_3$  phase in the interdendritic space. As a result of calculating the parameters of crystal lattices of particles by Fourier transforms, the parameters of BCC-lattice  $a = 0.210 \pm 0.247$  Å were determined at tabular values  $a = 0.289 \pm 0.607$  Å. The formation of twins is specific for this type of steel (Fig. 9). A slight deviation of crystal lattice parameters of the identified phases from the tabular values is associated

with the dissolution of alloying and impurity elements in them.

The fractographic analysis revealed that the samples are destroyed with necking, i.e. the plastic component of deformation prevailed, and the destruction proceeded from the surface. Initially, a viscous shear occurred, resulting in separation. The macroplastic fracture was formed by the shear mechanism. The micromechanism of destruction is a viscous-pitting one. This type of failure is typical for maraging steels, regardless of the method of obtaining the material [13–22]. Fig. 10 exhibits the micromechanism (a) and the appearance of fractures (b–d).



**Fig. 9.** The Fourier transforms separated from the surface of phase

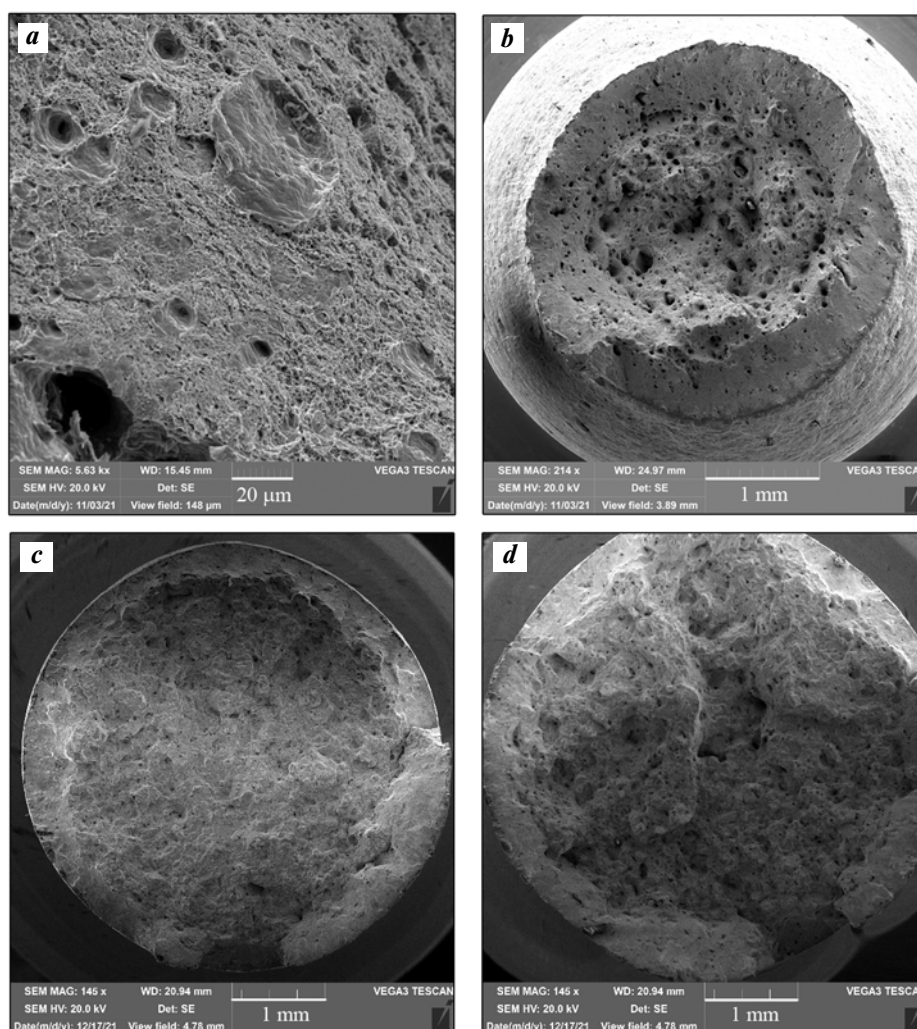
**Рис. 9.** Фурье-трансформации выделившейся с поверхности фазы

## Conclusions

1. In the process of hot isostatic pressing according to the selected modes, the subgrain structure of SLM samples of maraging steel is recrystallized with the formation of a homogeneous structure. As a result of aging, the alloying elements form a plastic matrix phase being a substituted martensite, dispersion-hardened by the precipitates of excessive phase  $\text{NiTi}_3$ .

2. HIP in combination with heat treatment (hardening + aging) provides a 2-fold reduction in residual porosity and a 1.5-fold increase in tensile strength and offset yield strength.

3. The SLM samples made of MS steel are destroyed



**Fig. 10.** The appearance of fractures

**a** — micromechanism; **b** — HIP; **c** — HIP, hardening and aging; **d** — HIP and aging

**Рис. 10.** Внешний вид изломов

**a** — микромеханизм; **b** — ГИП; **c** — ГИП, закалка и старение; **d** — ГИП и старение

with necking according to the viscous-pitting mechanism, which is typical for maraging steels, regardless of the method of their production.

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