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Estimation of Additive Manufactured Steel Hardness and Elastic Modulus by Instrumented Indentation Test

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Abstract

This work is devoted to mechanical properties estimation of steel samples produced by additive and classic technologies via instrumented indentation test. Features of 3D printing, physical and mechanical properties, methods and means of additive manufacturing products control are described. The experimental part presents a series of indentation tests to determine the parameters of elastic-plastic deformation: hardness and elastic modulus, which are compared to reference data from other studies. The difference in the properties of the material produced by classical technologies and synthesized by direct metal laser sintering (DMLS) from a powder of identical chemical composition is discussed. The elastic modulus varied less for DMLS samples (208–213 GPa) than for cast ones (203–234 GPa). Hardness of the additively manufactured 12Cr18Ni10Ti was also more consistent relative to the differently remelted samples.

Keywords: Additive manufacturing; Instrumented indentation; Mechanical properties; Hardness; Elastic modulus

1. INTRODUCTION

One of the goals of sustainable development approved in 2015 by the UN General Assembly was the creation of conditions for industrialization and innovation, as well as infrastructure development. One of the ways to achieve this goal is the fourth industrial revolution (Industry 4.0) through the introduction of software and computation into the industrial sector [1]. As noted in Ref. [2], the shift toward functional part production demands advanced non-destructive evaluation and mechanical property characterization integrated into the digital manufacturing workflow. And one of its most promising directions is additive manufacturing, where the product is made layer by layer. Interest in 3D printing increased when it became possible to produce parts with structural materials, and also due to the reduction in the cost of technology, i.e. wider availability for companies [3,4]. For example, the use of steels has significantly increased the performance properties of materials, which allowed the technology to move from a tool for prototyping to a full-fledged method for producing critical parts and assemblies of complex geometry [5,6]. Basically, quality improvement is ensured by the accumulation of a knowledge base and research on the materials that are used in the production process, as well as by conducting non-destructive testing of the products themselves [7,8]. The combination with information technology has significantly reduced the production cycle time, since most of its stages are in the digital environment – such as modeling in computer-aided systems that is the basis of the technology.

3D printing can be used in many areas of life: in medicine – as the production of individual artificial limbs and surgical implants of any geometry; in tech industries – as a way to improve the performance characteristics of complex units, critical parts and assemblies. For example, in the oil and gas industry, where one of the priority areas is the replacement of components of old equipment and reusable waste [9,10]. Most mining enterprises use aged equipment that needs to be maintained [11]. During the repair process, it is often the case when the necessary structural elements are no longer produced and replacement products must be ordered from third party companies.

To properly represent quality, it is necessary to analyze the properties that provide it. It is obvious that using the rapid prototyping approach significantly reduces the

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dispersion of the obtained results. It is difficult to reliably determine how much the characteristics of the finished product will differ from its model. The issue requires not only improving digital technologies, but also improving the quality control system and production technologies themselves, which allow operations to be performed with maximum precision. Determining whether quality meets the requirements is possible by measuring specific parameters of the finished product. Informative data, or data that reduces uncertainty, can be physical and mechanical properties that describe the object of study [12]. They are determined during inspection or testing. For the most part, these properties reflect future performance characteristics that are important to the consumer.

Non-destructive testing of products of additive manufacturing is a complex engineering task discussed in Ref. [13]. Classical approaches do not allow presenting such products as standard test units. Layer-by-layer manufacturing technologies provide heterogeneous material structure with certain manufacturing defects. A recent review [14] highlights that structural heterogeneity inherent to metals produced by additive manufacturing directly affects mechanical performance, reinforcing the need for reliable property assessment methods. The main question is the selection of means and methods of control, since the anisotropy of the obtained structure determines the uneven distribution of mechanical properties [13,15]. To find defects, a radiographic method (tomography) is often used, which is suitable for searching some inclusions and pores [16–18]. Thus, porosity is indirectly associated with the mechanical properties of the final product, since it reduces the resistance of the material to fatigue loads. Ultrasonic testing makes it possible to indirectly determine the elastic modulus of a material [17–19], but this method is complicated by the anisotropy of the material properties and the design features of products with complex geometry [20,21]. In particular, the results of ultrasonic testing are also affected by the attenuation of elastic waves in the material [22]. It is also relevant to use the acoustic emission method for local determination of the stress state area by analyzing the parameters of elastic waves [23,24]. However, strong acoustic attenuation in the material limits the application of these methods [25]. A possible solution to this problem may be the use of modern methods for processing acoustic testing data, which is already finding its application in the field of civil engineering [26].

In this work, the mechanical properties of steel samples obtained by additive technologies were studied using the instrumented indentation method. Properties provided by such hardness tests allow to determine the technical state of products in use, as well as serve in improving production technologies [27]. The study is carried out in order to determine the influence of various processes on the quality of the resulting product.

2. MATERIALS AND METHODS

2.1. Materials

A wide range of additive manufacturing technologies are currently available. The main differences are the layering method and the materials. There are also features of consolidation, i.e., the process of combining individual elements. In some cases, laser energy is used, for example in selective laser sintering (SLS) and selective laser melting (SLM). Polymer materials are characterized by the use of printers based on photopolymerization, where the hardening process occurs under the influence of radiation in the ultraviolet spectrum. However, each printing method is characterized by different properties for different input data: printing parameters, materials used and methods for its production [28]. All this can affect the quality and strength characteristics of the finished product.

This work examines products manufactured by direct metal laser sintering (DMLS) which is one of the most common methods for metals. In this method, fine powder with a particle diameter of $d = 20...40$ μm is deposited as a thin layer onto the substrate and leveled with a roller. The powder is consolidated under the influence of infrared laser beam in accordance with the current cross-section of a given model. The optical system of rotating mirrors determines the direction of radiation for achieving a certain trajectory. It is important that in DMLS there is no need to form supporting structures, since the material is evenly distributed over the surface of the substrate. The remaining powder is not removed and serves as a support for subsequent layers.

After exposure to energy emission, the material cools and shrinks. Together with the complex geometry of the part and residual stresses, this causes a local stress-strain state, which in turn causes the appearance of discontinuities [29]. If the printing powder has a large variation in dispersion, then when the particles melt, a local shortage of material may be observed [30]. This causes the appearance of a large number of pores and cavities in the volume of the resulting product. Porosity is a factor that reduces the resistance of a material to fatigue loads and, accordingly, worsens the mechanical properties [31,32]. Pores may also result from the entrapment of gas molecules or particles formed during the consolidation of the material by the laser, so the production chamber is maintained in an inert gas environment to counteract oxidation when exposed to high temperatures [33]. Porosity is also a factor for classical indentation test [34]. Crystallization stresses can cause cracks to appear in the volume or on the surface of products. However, this type of defect is caused by the material itself, and not by technology [35]. Alloys that can be easily melted do not tend to crack under the specified

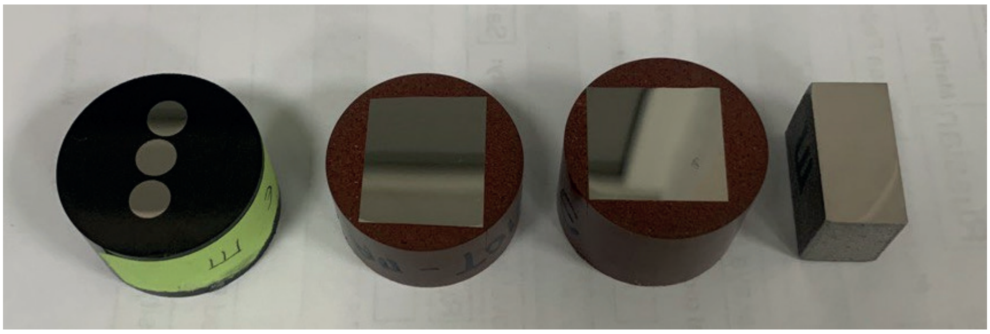


Fig. 1. Test samples.

printing parameters. The use of new materials and compositions requires greater attention in quality control, since cast alloys are more likely to crack. The use of materials with such a sequence of properties is an advantage when introducing them into technological systems that comply with operating conditions [36,37].

This work uses 12Cr18Ni10Ti (AISI 321 analog) powder and DMLS method for production of samples (Fig. 1). For proper comparison the same austenitic steel was obtained by rolling. The chemical composition corresponds to the 12Cr18Ni10Ti grade composition according to standard GOST 5632-72. The material refers to the class of cryogenic structural steel with corrosion-resistant, heat-resistant, heat-stable. The choice of material is determined by the existence of similar works on the study of its properties in additive manufacturing [38]. Properties of the material determine the thread of application: petrochemical industry, medicine (medical instruments), food and other industries with aggressive environments (alkaline, saline and some acidic solutions). Before powder atomization [39], the material was melted by three methods: vacuum-arc melting, electron beam melting, and electroslag melting.

The sample produced using additive technology contained three cylinders manufactured using DMLS. EOSINT M270 system was used for printing with the following synthesis parameters: laser wavelength $\lambda = 1063$ nm, laser power $P = 200$ W; particle size of spherical powder $d = 20...40$ μm.

Samples were put through three steps of preparation for instrumental indentation test: cutting, mold pressing, and sanding with polishing. These steps used low-speed tool action since high temperatures can affect material with structural changes. The samples were processed under the same treatment to avoid deviations due to different conditions.

The requirements for surface roughness for instrumented indentation are limited by its contribution to the uncertainty of the result for measuring the indentation depth (less than 5%) [40]. The h value should satisfy $Ra \leq 20h$, where Ra is the average roughness of a surface (the arithmetic mean deviation of the profile). The surface roughness was measured by Hommel Tester T1000 profilometer and plotting the surface profile. The arithmetic mean of the absolute values of profile deviations within the base length of 4 mm was no more than 0.02 μm (20 nm). The indentation depth during testing is subsequently selected based on this value.

2.2. Methods

The work uses the instrumented indentation method described by Oliver-Pharr [41]. This testing type is widely applicable for additive products [42–44]. The instrumented indentation method is based on the penetration of a solid diamond indenter into the surface of the object and the subsequent determination of the dependence of the load P on the indentation depth h. This experiment uses the Berkovich type indenter — a diamond pyramid with a 65.3° angle between the penetration axis and its edge. The load and depth are recorded continuously to build the P-h diagram. Based on the obtained from the diagrams data, hardness H and elastic modulus E are calculated. The mechanical module of the nanohardness tester "NanoScan 4D" (FSBI TISNCM, Troitsk, Russia) was used for testing [45].

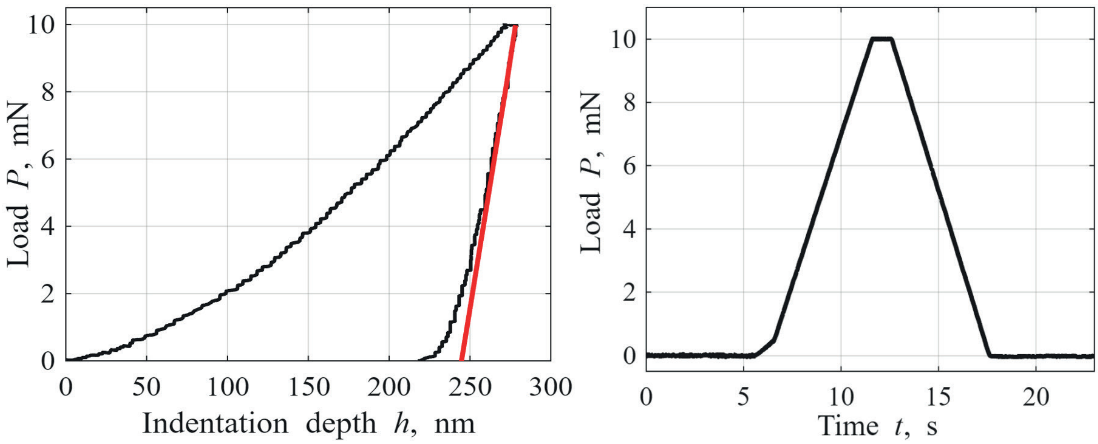
Hardness H_{IT} and elastic modulus E_{IT} values are calculated according to ISO 14577-1:2015 [40]. Hardness during indentation is determined by the ratio of the maximum applied force P_{max} to the contact surface area A_c:

$$H_{IT} = P_{max} / A_c. \quad (1)$$

The elastic properties are determined by Oliver-Pharr methodology [46]. Elastic modulus E_{IT} is determined by Eq. (2) through the reduced elastic modulus E_r (Eq. (3)) in the indentation area and the Poisson's ratios of the indenter and studied material. The contact stiffness function S depends on the incline angle of the tangent to the unloading curve at the point of maximum load as in Eq. (4).

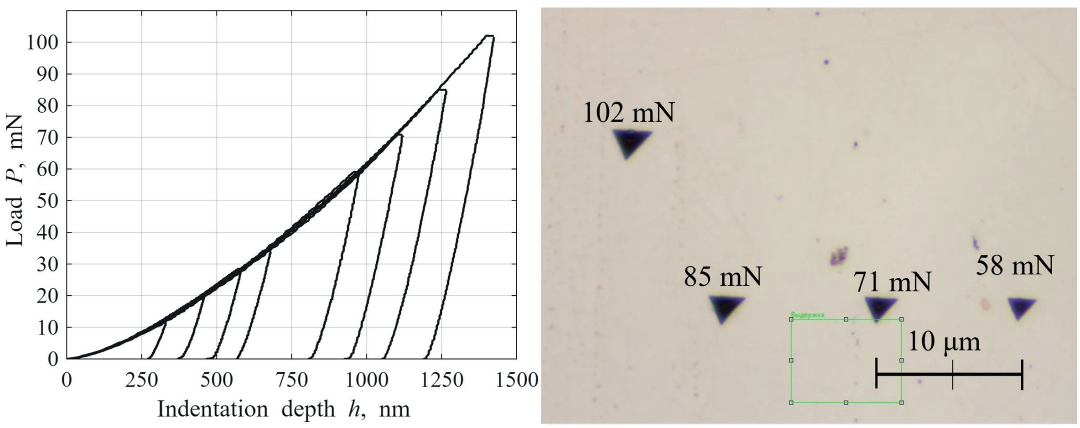
$$E_{IT} = \frac{1-\mu_s^2}{1/E_r - (1-\mu_s^2)/E_i}, \quad (2)$$

where μ_s is Poisson's ratio of the tested material (0.27 for steel alloy); μ_i is indenter Poisson's ratio (0.07 for diamond); E_i is indenter elastic modulus (1140 GPa = $1.14 \cdot 10^6$ N/mm^2).



(a) **(b)**

Fig. 2. $P(h)$ curve of one of the measurements (a) and trapezoidal load function (b).



(a) **(b)**

Fig. 3. Tests with different loads: (a) example of $P(h)$ diagram; (b) imprints for maximum loads.

$$E_r = \frac{1}{2}\pi^{1/2} S A_c^{1/2}, \tag{3}$$

$$S = \left(\frac{dP}{dh}\right)_{P=P_{max}}. \tag{4}$$

3. RESULTS AND DISCUSSION

An experimental study of the physical and mechanical properties was carried out by hardness testing using the instrument indentation method based on previously developed procedure [40]. Movement control system was used to measure depth and apply force to the indenter. Each measurement (imprint) was accompanied by automated construction of an indentation curve, an example of which is illustrated in Fig. 2a. The trapezoidal loading pattern (Fig. 2b) is applied for a specified period of time before a peak load, then holds that peak load constant for another specified time to reduce the effects of temperature drift and material creep, allowing the material to stabilize before unloading. The nonlinear section observed in Fig. 2b characterizes the time duration when a given load is applied to the indenter, but contact with the surface has not yet been made.

Ideally, the correlation between contact area A_c and contact depth h_c for a Berkovich indenter corresponds to a quadratic increasing function $A_c = 23.96h_c^2$. However, the indenter is rounded, therefore the specific area function of each indenter must be calibrated. Calibration of the system is carried out by forming a function of the shape of the indenter area and stiffness depending on the contact depth. It is conducted on a calibrated reference material with known assigned metrological characteristics: hardness 9.3 ± 0.3 GPa; elastic modulus 72.7 ± 0.4 GPa.

Preliminary tests (Fig. 3) with different loads of 0.45–106 mN and depths of 49…1278 nm showed no strong distortion of the indent shape and no plastic deformation of the material around, as when measuring microhardness of 12Cr18Ni10Ti in the study [47]. Heterogeneity of properties was observed when one sample was tested under different loads (loading and unloading curves at the same angle).

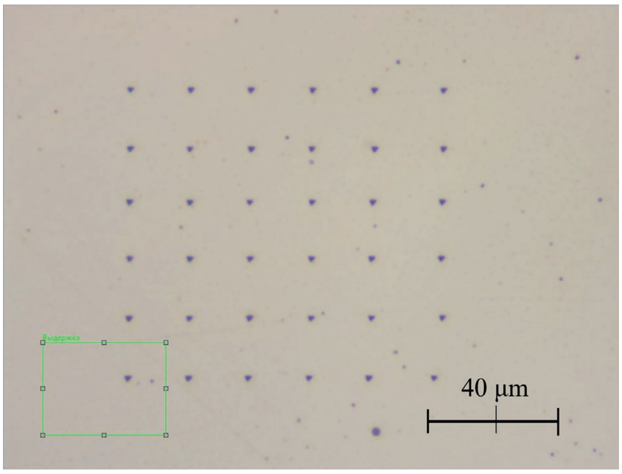


Fig. 4. Imprints on the surface of the DMLS sample.

The parameters and conditions of the main test series are set the same for all samples:

− load force P_{max} = 10 mN;
− load time 5000 ms;
− holding time 1000 ms;
− distance between points $dx = dy$ = 20 μm.

To reduce the effect of plastic deformation, it is recommended to choose the distance between testing points within more than three imprint width sizes. To distribute the field evenly and reliably determine the scale of the area (Fig. 4), the distance between measurements was chosen to be 20 μm. With this value and six measurements along each axis, a field of 100×100 μm is received.

All the parameters of hardness and elastic modulus for different technologies were determined from the data obtained from the diagrams using the standard methodology. The results are presented in Table 1 and in Fig. 5.

The average indentation depth in series with the same load on additively manufactured samples was 290 nm with a spread of 30 nm (10%). A deviation of the given load was observed: the minimum was 9.8 mN, the maximum was 9.99 mN (average 9.92 mN, dispersion less than 2%).

There is a potential for the hardness value to decrease on the additive produced samples. There is a difference in hardness between additive and conventional samples as in Ref. [48]. It is noticeable that samples produced using additive technology have more stable characteristics, regardless of the method for obtaining the starting material. For the DMLS sample of electron-beam and electroslag remelting, the hardness values coincided and were equal to 3.6 GPa, which was also comparable to the vacuum-arc remelting within the accuracy limits. Identical effect for the elastic modulus — the values ranged from 203 to 234 GPa for classical technologies, and 208–213 GPa for additive ones. A similar decrease was described in Ref. [49] and can be explained by the presence and influence of a large number of forced dislocations, which prevent enhancement in the yield strength and hardness. This issue can be possibly solved by heat treatment (aging) procedures to reduce the internal stresses that occur in the elongated grains of the additive structure, which will be investigated in further studies.

Experimental data show that some mechanical properties for additive technology can vary. This indicates that remelting during printing takes place and the resistance to deformation properties also change. However, the value of the elastic modulus remains close to the standard value for this steel (210 GPa). The hardness value for additive samples remains constant within the absolute accuracy error (±0.1 GPa), which indicates the reproducibility of the DMLS technology. With the further development of indentation methods, it is important to understand that current method showed a slight scatter, however, when switching to sample-free control methods, it is important to maintain the order of this scatter when using portable devices.

4. CONCLUSIONS AND FUTURE WORK

3D printing is actively becoming an applicable technology. Products obtained by the method of step-by-step synthesis are considered not only as an object of prototyping, but also as a full-fledged product with a specific set of properties. Typical problems of additive manufacturing are mostly related to the structure of the resulting products. The issue of quality control immediately comes up, since classical approaches are not always applicable due to anisotropy and other characteristics.

The described steps of the instrumental indentation were used for determining the mechanical properties of additively manufactured products. An experimental part was carried out in the form of testing the properties of elastic-plastic deformation in small load ranges. The instrumental indentation method made it possible to fully

Table 1. Test results (VA – vacuum-arc melting, E – electron beam melting, SH – electroslag melting).

Melting type	Cast sample		DMLS sample	
	H_{IT}, GPa	E_{IT}, GPa	H_{IT}, GPa	E_{IT}, GPa
VA	5.1 ± 0.1	234 ± 11	3.8 ± 0.1	210 ± 10
E	4.8 ± 0.1	203 ± 7	3.7 ± 0.1	208 ± 10
SH	3.5 ± 0.1	221 ± 10	3.7 ± 0.1	213 ± 12

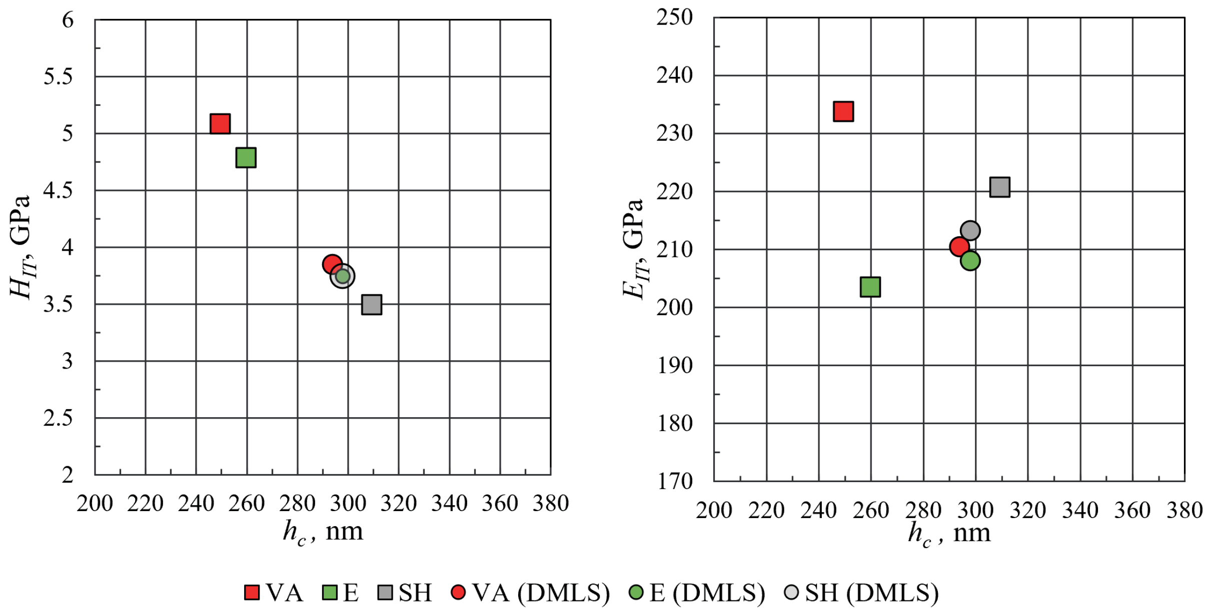


Fig. 5. Obtained values of hardness and elastic modulus.

determine such mechanical properties of DMLS samples material as hardness and elastic modulus. DMLS samples showed a smaller variation in elastic modulus (208–213 GPa) than casting ones (203–234 GPa). Hardness for additively manufactured 12Cr18Ni10Ti is more stable comparing to the samples which were remelted by different technologies.

Comparing the data with the source material concluded that the choice of steel production and melting technology for powder production has almost no affect the parameters of hardness and elastic modulus. DMLS at constant printing parameters ensures reproducible characteristics, in contrast to classical technologies, where a variation in hardness is observed. The obtained results can help to reduce measurement uncertainty while conducting studies using other physical testing methods, including non-destructive ones.

Future work will be devoted to characterize mechanical properties for different printing parameters within one material and porosity analysis. Another task is implementation of the dynamic instrumented indentation. There is a potential transition to sample-free testing of such products using Leeb-based sensors to measure physical and mechanical properties, but it is necessary to develop a proper method and algorithm for determining elastic properties.

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Определение твердости и модуля упругости стали аддитивного производства методом инструментального индентирования

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Аннотация. Данная работа посвящена оценке методом инструментального индентирования механических свойств стали образцов, произведенных аддитивными и классическими технологиями. Описаны особенности 3D-печати, физико-механические характеристики, методы и средства контроля изделий аддитивного производства. Экспериментальная часть представлена испытаниями по определению параметров упругопластической деформации: твердости и модуля упругости, которые затем сравниваются со справочными данными из других исследований. Обсуждается разница в свойствах материала, полученного классическими технологиями и синтезированного методом прямого лазерного спекания металла (DMLS) из порошка идентичного химического состава. Модуль упругости имеет меньший разброс для образцов, полученных методом DMLS (208–213 ГПа), чем для отлитых образцов (203–234 ГПа). Твердость аддитивно изготовленного сплава 12Х18Н10Т также более стабильна по сравнению с твердостью материалов образцов, подвергнутых различному виду переплавления.

Ключевые слова: аддитивные технологии; инструментальное индентирование; механические свойства материалов; твердость; модуль упругости