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Материалы и покрытия, получаемые методами аддитивных технологий

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Features of obtaining TiNi alloy samples from commercial powders with high oxygen content using the SLM technique

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Abstract. Additive technologies, in particular selective laser melting (SLM), enable to manufacture the products with complex geometries. The SLM technique can help to effectively expand the titanium nickelide scope of application. However, SLM is a complex process – numerous factors significantly affect the characteristics of the resulting alloy. When the SLM technique is used, as the material is subject to laser processing, the content of nickel in the alloy drops due to evaporation, which can lead to changes in the temperatures of martensitic transformations. This impact on the resulting alloy characteristics can be regulated by changing the parameters of the SLM process. The objective of our research was to develop the processing methods for manufacturing samples from two commercial TiNi alloy powders using the SLM technique and to analyze the factors causing defects in the obtained samples. At the same time, processing methods with low values of volumetric energy density were used to reduce possible evaporation of nickel during printing. The initial powders were examined for the presence of impurities or other factors affecting the quality of the manufactured samples. The processing method A4 that we have developed for powder *1* enables to obtain a defect-free sample with the density of 6.45 g/cm^3. It was found that none of the processing methods used enabled to obtain a defect-free sample from powder *2* due to presence of a large amount of oxygen impurities, including in particular $Ti_4Ni_2O_x$ secondary phase, which leads to embrittlement and destruction of the samples. Therefore, high content of oxygen in the initial powders has a negative impact on the quality of the samples manufactured using the SLM technique.

Keywords: selective laser melting, TiNi alloy, titanium nickelide, impurities, defects, defect-free samples

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Особенности получения образцов сплава TiNi методом СЛС из коммерческих порошков с повышенным содержанием кислорода

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Аннотация. Аддитивные технологии, в частности метод селективного лазерного плавления (СЛС, или SLM), позволяют изготавливать изделия со сложной геометрией. С помощью СЛС можно эффективно расширить области применения никелида титана. Однако процесс СЛС является комплексным – множество факторов оказывают серьезное влияние на характеристики получаемого сплава. В процессе лазерной обработки материала в технологии СЛС происходит снижение содержания никеля в составе сплава за счет испарения, что может приводить к изменению температур мартенситных превращений.

Регулирование данного влияния на результирующие характеристики сплава возможно за счет изменения параметров процесса СЛС. Цель работы состояла в разработке технологических режимов изготовления образцов из двух коммерческих порошков сплава TiNi методом СЛС и анализе факторов, влияющих на наличие дефектов в полученных образцах. При этом для снижения возможного испарения никеля в процессе печати применялись технологические режимы с невысокими значениями объемной плотности энергии. Исходные порошки исследованы на наличие примесей или иных факторов, влияющих на качество изготавливаемых образцов. В результате проведенного исследования для используемого порошка *1* разработан технологический режим А4, с помощью которого изготовлен бездефектный образец, плотность которого составила 6,45 г/см^3. Установлено, что ни один из применяемых режимов не позволил получить бездефектный образец из порошка *2* ввиду наличия в нем большого количества примесей кислорода, в частности вторичной фазы $Ti_4Ni_2O_x$, приводящей к охрупчиванию и разрушению образцов. Следовательно, высокое содержание кислорода в исходных порошках отрицательно влияет на результаты изготовления образцов методом СЛС.

Ключевые слова: селективное лазерное сплавление, сплав TiNi, никелид титана, примеси, дефекты, бездефектные образцы

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Introduction

Titanium nickelide (TiNi) is one of the most well-known smart materials famous for superelasticity and its ability to exhibit shape memory effect. It is widely used in the aerospace and automotive industries, medicine, microelectronics and other fields of science and technology [1–5]. Additive technologies, in particular the selective laser melting (SLM) method, enable to create highly complex geometries [6] and can dramatically increase applications of titanium nickelide. However, SLM is a complex process – numerous factors significantly affect the characteristics of the resulting alloy.

It is known that when the SLM technique is used for laser processing of the material, the nickel content in the alloy drops due to evaporation as the boiling points of nickel and titanium differ: nickel boils at 2913 °C, while titanium has a boiling point of 3287 °C [7–12]. Additionally, nickel has a higher partial pressure than titanium, and therefore nickel is more volatile at elevated temperatures [7]. It has been established that changes in the nickel content in the alloy can result in changing temperatures of martensitic transformations – the main parameters indicating that the functional properties of the alloy can manifest themselves at certain temperatures [13–15].

This impact on the resulting alloy characteristics can be regulated by changing the parameters of the SLM process – volumetric energy density (E) and its determining factors – scanning speed, distance between laser passes, laser power and layer thickness. It was found that as the E value increases, so does nickel evaporation during the SLM process [16–18]. Some researchers point out that an average volumetric energy density of more than 100 J/mm^3 is required to obtain dense products from nitinol using the SLM technique [3; 18–20]. At the same time, in some studies, defect-free samples were obtained with lower E values [11; 21; 22]. Such a spread in the values of volumetric energy density may indicate that the quality of the initial powder (presence of impurities or secondary phases in it) affects the characteristics of the resulting products.

Based on the above, the objective of our research was to develop the processing methods for manufacturing samples from two commercial TiNi alloy powders with enhanced oxygen content using the SLM technique and to analyze the factors causing defects in the obtained samples. At the same time, processing methods with low values of volumetric energy density were used to reduce the possible evaporation of nickel during printing [17]. The initial powders were examined for the presence of impurities or other factors affecting the quality of the manufactured samples. The results obtained will, in the future, enable to improve the quality of the resulting products and to simplify the selection of TiNi alloy powders, as well as the development of processing methods for manufacturing products from these powders.

Materials and methods

In this study, we used two commercially produced spherical powders (*1* and *2*, respectively) of TiNi alloy with similar chemical composition $Ti_{49}Ni_{51}$ (at. %). The chemical composition of these powders is presented in Table 1.

The cylindrical samples with a diameter of 10 mm and a height of 60 mm were fabricated for the study. Table 2 shows 4 methods of their production using the SLM technique. The processing methods with low values of volumetric energy density – $E < 100$ J/mm^3 – were selected to minimize nickel evaporation during the SLM process. The E values are changed by gradually reducing the scanning speed in increments

***Table 1.* Chemical composition (at. %) of the powders under study**

***Таблица 1.* Химический состав (ат. %) исследуемых порошков**

Powder	Ti	Ni	O	N	C
1	48.63	51.03	0.14	0.01	0.19
2	48.56	50.96	0.29	0.01	0.18

of 175 mm/s. The laser power, distance between passes, and layer thickness remained the same for all processing methods. For convenience, the samples manufactured using a certain processing method from powder *1* will hereinafter be marked as A1/1, A2/1, A3/1 and A4/1, and those from powder *2* – A1/2, A2/2, A3/2 and A4/2.

The SLM process was conducted on the SLM280HL printer (SLM Solutions GmbH, Germany), which uses an ytterbium fiber laser with a maximum power of 400 W, has a wavelength of 1070 nm, the minimum laser beam diameter of 80 μm and the maximum scanning speed of 15 m/s. The process was implemented in an inert gas atmosphere (argon). The chemical composition of the initial powders and the resulting samples was visually analyzed and determined using Tescan Mira 3 LMU scanning electron microscope (SEM) (Tescan, Brno, Czech Republic) with an energy-dispersive *X*-ray spectroscopy module "EDX X-max 80" (Oxford Instruments, Abingdon, United Kingdom). The microstructure of the obtained samples was evaluated on a Leica DMI 5000 light optical microscope (Leica Microsystems, Germany). The granulometric composition of the powders was assessed using an Analysette 22 NanoTec particle size analyzer (Fritsch, Germany). The phase composition of the initial powder and the resulting samples was determined on a Bruker D8 Advance *X*-ray diffractometer (Bruker, Bremen, Germany).

Results and discussion

Fig. 1 shows the granulometric composition of the powders under study. Volume distribution fractions for powder *1* – d_{10} = 28.7 μm, d_{50} = 48.1 μm, d_{90} = 76.5 μm, for powder *2* – d_{10} = 15.1 μm, d_{50} = 29 μm, and d_{90} = 52.6 μm.

Fig. 2 demonstrates SEM images of the powders under study.

Fig. 3 shows the samples made from powder *1* using various processing methods; Fig. 4 demonstrates the samples from powder *2*. In Fig. 3, we see that the samples manufactured at a lower volumetric energy density (A1/1, A2/1) have multiple defects in the form of cracks. As *E* was increased by reducing the scanning speed, first, the number of visually detectable cracks (sample A3/1) decreased, and as *E* reached 90 J/mm^3 (sample A4/1), no cracks were detected by visual inspection. The density of the resulting sample A4/1 was 6.45 g/cm^3. The nickel content in the defect-free sample A4/1 (50.85 at. %) dropped by 0.18 at. % against the original powder *1*. The results of our study are generally consistent with those previously obtained in [17].

The samples from powder *2*, which were manufactured using processing methods with low volumetric energy density, are severely deformed and partially destroyed (see Fig. 4). The increase in *E* resulted

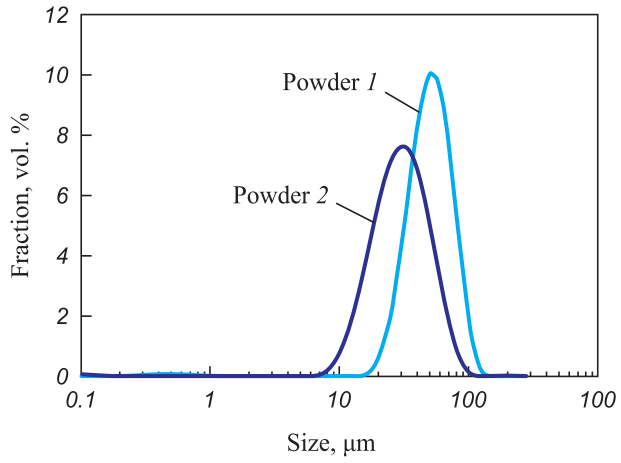

Рис. 1. Гранулометрический состав исследуемых порошков

Fig. 1. Granulometric composition of the powders under study

***Table 2.* Processing methods for manufacturing samples**

***Таблица 2.* Технологические режимы изготовления образцов**

Method	Power, W	Scanning rate, mm/s	Distance between laser passes, mm	Layer thickness, mm	Volumetric energy density, J/mm^3
A1	200	1450	0.08	0.03	57
A2	200	1275	0.08	0.03	65
A3	200	1100	0.08	0.03	75
A4	200	925	0.08	0.03	90

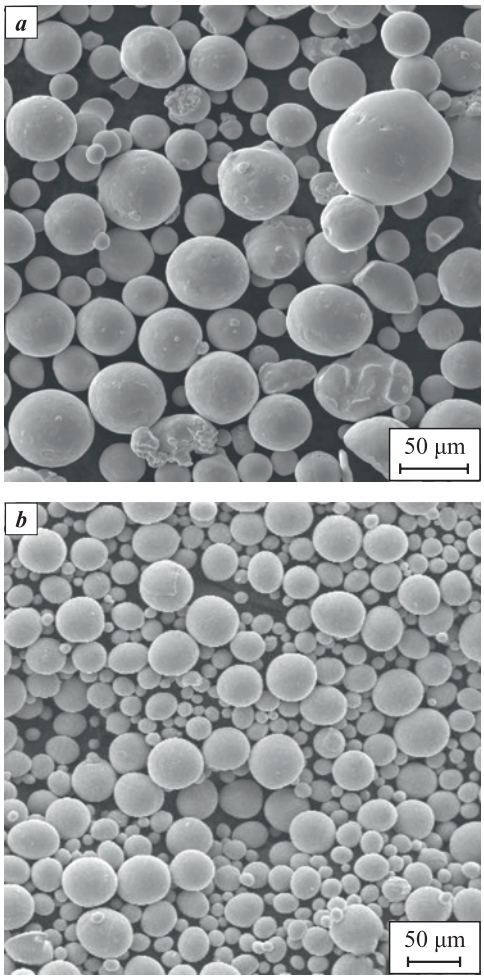

Fig. 2. SEM images of the powders used
a – powder *1*, ***b*** – powder *2*

Рис. 2. СЭМ-изображения используемых порошков
a – порошок *1*, ***b*** – порошок *2*

Fig. 3. Samples made from powder *1*
a – A1/1, ***b*** – A2/1, ***c*** – A3/1, ***d*** – A4/1

Рис. 3. Образцы, изготовленные из порошка *1*
a – A1/1, ***b*** – A2/1, ***c*** – A3/1, ***d*** – A4/1

in a decreased level of deformation; however, even at $E = 90$ J/mm^3 (sample A4/2), multiple cracks were observed along the entire length of the sample.

The large number of cracks in the samples is associated primarily with the features of the SLM process, namely with residual stresses caused by a high temperature gradient during the samples manufacture due to rapid heating, melting and, subsequently, rapid cooling and solidification of the material, as well as possible presence of defects in the form of micropores [23–25]. An increase in the scanning speed during the samples

Fig. 4. Samples made from powder *2*
a – A1/2, ***b*** – A2/2, ***c*** – A3/2, ***d*** – A4/2

Рис. 4. Образцы, изготовленные из порошка *2*
a – A1/2, ***b*** – A2/2, ***c*** – A3/2, ***d*** – A4/2

manufacture causes an expected drop in volumetric energy density. When the energy density is insufficient, micropores and discontinuities emerge in the sample structure due to the small size of the molten bath and presence of unmelted powder particles [3]. These defects are stress concentrators that contribute to active propagation of cracks in the samples. This phenomenon was observed in the samples made from powder *1*. When the processing method A1 was used with a scanning speed of 1450 mm/s, a large number of cracks was registered over the entire area of the sample; and when the scanning speed was reduced to 925 mm/s, cracks were not visually detected.

Fig. 5 shows the microstructure of samples from powder *2* with various defects. We can see multiple cracks at the edges of the sample (Fig. 5, *a* and *b*) and also the ones running through the entire sample (Fig. 5, *c*), up to 200 μm thick. In addition, spherical and non-spherical pores of various sizes (some larger than 100 μm) are visible. Some cracks penetrate through pores and propagate in them (Fig. 5, *a* and *b*).

The samples made from powder *2* underwent rather severe deformations, which is indicative of additional factors contributing to crack formation, along with the scanning speed. One of the hypothetic causes may be the presence of a large amount of oxygen in the initial powder *2* (more than in powder *1*), which may also be present in the form of $Ti_4Ni_2O_x$ secondary phase. In powder *2* under study, the oxygen content was initially 2 times higher than in powder *1*. The $Ti_4Ni_2O_x$ secondary phase is a Ti_2Ni phase with oxygen in the solid solution. The presence of this phase has a negative impact on the alloy and can lead to embrittlement and destruction of samples [26; 27]. It was noted [28] that initially cracks emerge in this very $Ti_4Ni_2O_x$ secondary phase. Therefore, a large amount of this phase in the composition of the initial powder can lead to increased cracking when the samples are manufactured. Another assumption is that a large amount of oxygen contributes to the formation of these secondary phases during the sample preparation process.

To determine the reasons more clearly, a cross-section of powder *2* was prepared. Fig. 6 shows SEM images of particles of powder *2* at high magnification. It is clearly visible that the particles contain dark inclusions, which may be $Ti_4Ni_2O_x$ secondary phases.

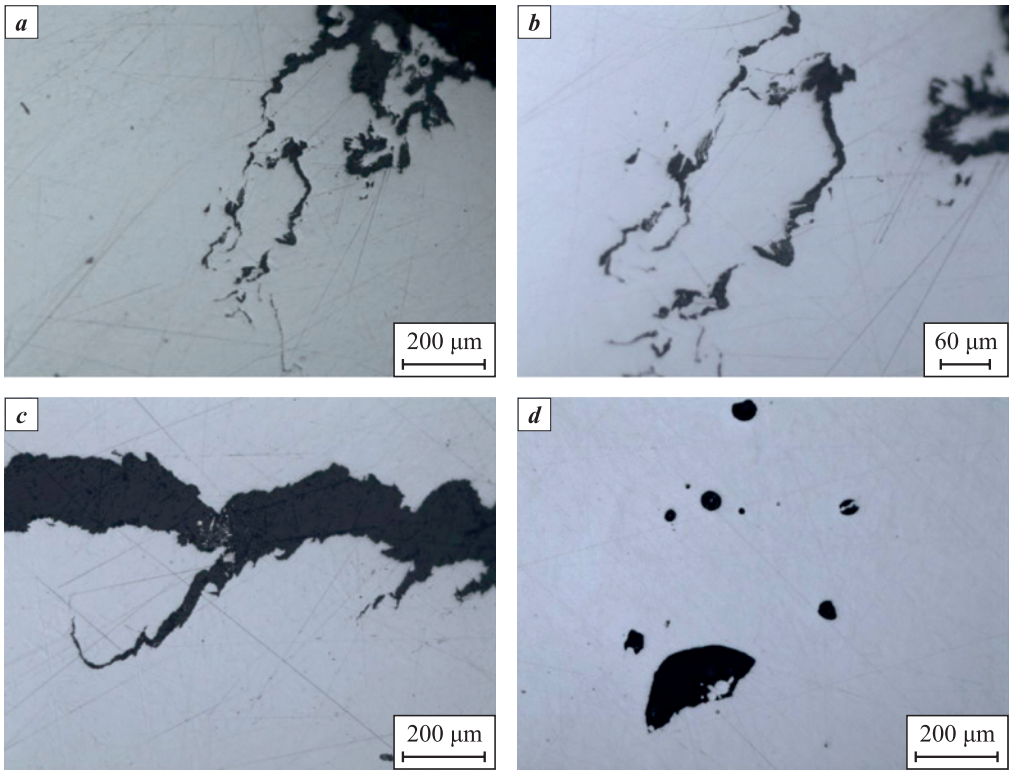

Fig. 5. Defects in the samples made from powder *2* using the SLM technique
a – cracks and pores at the edge of the sample; ***b*** – cracks penetrating through pores;
c – crack running through the entire thickness of the sample, ***d*** – spherical pores

Рис. 5. Дефекты в образцах, изготовленных из порошка *2* методом СЛС
a – трещины и поры с краю образца; ***b*** – трещины, проходящие через поры;
c – трещина, пересекающая всю толщу образца, ***d*** – сферические поры

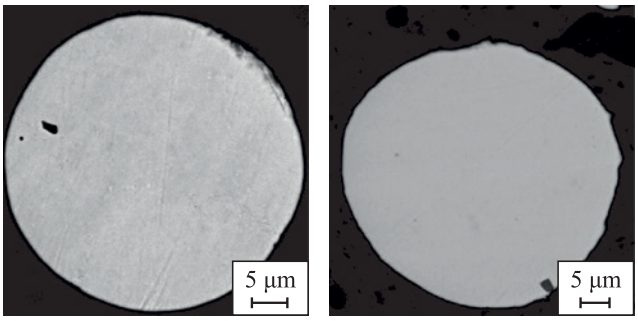

Fig. 6. SEM image at high magnification of powder particles *2* in the BSE mode with inclusions – secondary phases

Рис. 6. СЭМ-изображения с большим увеличением в режиме BSE частиц порошка *2* с наличием включений – вторичных фаз

Fig. 7 shows the results of *X*-ray phase analysis of powder *2* and a sample made from it.

The *X*-ray diffraction pattern of the sample (Fig. 7, *b*) shows that the lines of the B2 phase have widened

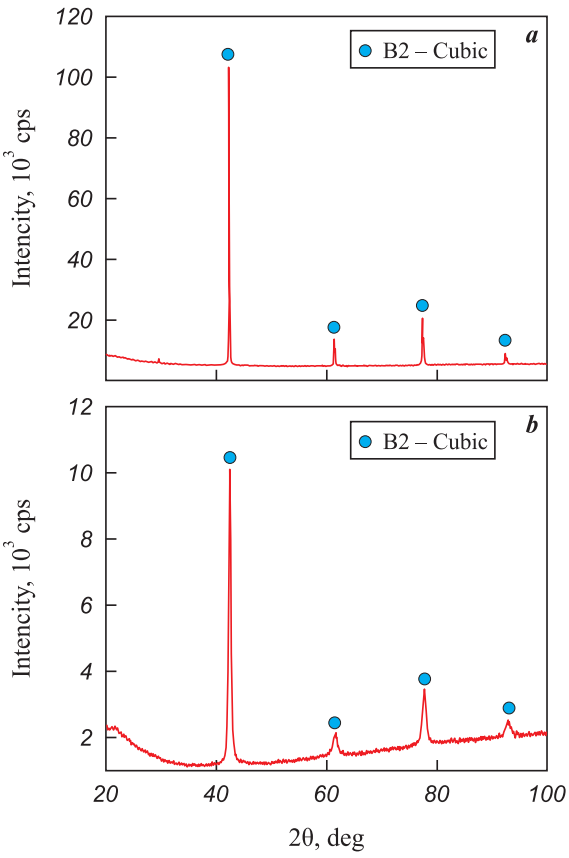

Fig. 7. *X*-ray diffraction patterns
a – powder *2*
b – sample made from powder *2* using the SLM technique

Рис. 7. Рентгеновские дифрактограммы
a – порошок *2*
b – образец, изготовленный из порошка *2* методом СЛС

Table 3. **Oxygen content in the samples**

Таблица 3. **Содержание кислорода в образцах**

Sample	Oxygen content, at. %
A4/1	0.26
A4/2	0.40

severalfold compared to the original powder. This widening is attributed to increased dislocation density and micro stresses. No secondary phases were detected in the composition of either the powder or the sample. This may suggest an extremely low content of the indicated $Ti_4Ni_2O_x$ phases in the composition of both powder *2* and the samples made from it – below the detection limit of the research method used. Moreover, as the lines of the B2 phase have widened, the coordinates of all the sought $Ti_4Ni_2O_x$ lines (39.0, 41.4 and 45.2) lie at the (110) B2 peak base. It can be assumed that the sample contains the indicated $Ti_4Ni_2O_x$ secondary phase, but its detection is hampered.

In general, based on the results obtained, we can confidently state that the increased oxygen content in the initial powder negatively affects the quality of the resulting samples, especially when they are manufactured with low values of volumetric energy density. To confirm this conclusion, we investigated the chemical composition of samples A4/1 and A4/2 for oxygen content. The results are presented in Table 3. It was found that the oxygen content in sample A4/2 reached 0.4 at. %, which is 0.14 at. % more than in sample A4/1. The oxygen level in the samples is higher than in the powder due to the capture of oxygen during the samples manufacture using the SLM technique. It can be noted that as the same processing method A4 was used, the oxygen content being high both in the original powder and in the manufactured sample, the sample from powder *1* had no defects, while that from powder *2* had numerous cracks. It confirms the assumption that oxygen content affects formation of defects in the samples manufactured using the SLM technique.

Conclusions

1. Having tested the processing methods, we came to conclusion that the defect-free sample from powder *1* can be obtained using the processing method A4. The density of the resulting sample A4/1 was 6.45 g/cm^3. Defect-free samples from powder *1* cannot be obtained using the processing methods with lower volumetric energy density.

2. We failed to obtain a defect-free sample from powder *2* using any of the processing methods. Probably

methods with higher values of volumetric energy density are required to obtain defect-free samples from this powder.

3. The presence of a large amount of oxygen impurities in powder *2* is one of the factors that hampered us to obtain defect-free samples when using the indicated processing methods. This is attributed to $Ti_4Ni_2O_x$ secondary phase present in the powder composition, which leads to embrittlement and destruction of the samples. That is, the increased oxygen content in the initial powders negatively affects the quality of samples manufactured using the SLM technique.

4. The study of the microstructure of samples obtained from powder 2 revealed the presence of spherical and non-spherical pores and cracks of various sizes. It was found that cracks propagate directly through the detected pores.

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