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## High nitrogen steel powder formation for additive technologies

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# High nitrogen steel powder formation for additive technologies

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**Abstract.** Today, when manufacturing and restoring bulk products of almost any shape and complexity degree, additive technologies are in demand, which use metal-powder compositions of various alloys as a building material. In the present work, the possibility of obtaining powder by gas atomization from batch products of high-nitrogen 04Cr20Ni6Mn11Mo2NVNb steel used for selective laser melting is shown, its structure and properties are evaluated.

## 1. Introduction

The production technology of high nitrogen steels has been developed most intensively over the last 20 years [1-3]. Nitrogen is an austenite stabilizing element. Doping with nitrogen helps to increase static and cyclic strength, hardness, wear resistance, hardening of steel during plastic deformation. The main area of application for nitrogen steels are: the chemical and oil and gas industries, applications where alloys with high resistance to corrosion cracking in chlorides, halides, pitting corrosion, and marine corrosion (heat exchangers, absorbers, condensers, stop valves) are required. Steel with a high nitrogen content can be obtained, including powder metallurgy methods [4]. To do this, different schemes for obtaining powder are used. For example, the methods of plasma rotational spraying, gas atomization are applied. These and other production schemes have proven themselves in powder metallurgy, which makes them [4, 5] the most promising methods for obtaining materials with a high nitrogen content.

The use of high nitrogen powders in additive technologies by selective laser melting (SLM) can provide a high (possibly even non-equilibrium) nitrogen content in the 3D-modeling and at the same time can ensure uniform nitrogen distribution throughout the product section, but at the moment there are no powdered nitrogen steels used in the SLM technology [6]. The aim of this work is to study the possibility of obtaining powders with a high nitrogen content for additive technologies by melt spraying using melting ingots of high-nitrogen 04Cr20Ni6Mn11Mo2NVNb steel as an example.

## 2. Materials and experimental methods

All experiments to obtain powders of nitrogen steel were carried out using HERMIGA 75/3VI gas atomizer with induction heating of a 3 kg volume crucible. This type of atomizer has wide potential for the production of spherical metal powders, which makes it the most flexible tool for conducting highly efficient research activities and the production of small batches of special purpose metal



powders. HERMIGA 75/3VI is a high-performance closed-type monoblock gas sprayer that meets modern requirements:

- a) To provide greater purity and smaller particle size than the existing systems;
- b) Cooling rate with enhanced fast crystallization (RSP).

Powder of high-nitrogen steel 04Cr20Ni6Mn11Mo2NVNb with the following melt spraying parameters:

- Melt temperature – 1500 °C;
- Excess pressure in the melting chamber – 0.25 bar;
- Exposure time of the molten metal to spray – 40 minutes.

Melting was carried out by induction heating in a protective gas. Both nitrogen (pressure at the nozzle 50 bar) and argon (pressure at the nozzle 30 bar) were used as a spray and at the same time as a protective gas.

Table 1 presents the chemical composition of the powders and the initial charge, determined by X-ray fluorescence analysis on a Niton XL3t unit. Table 1 shows that the chemical composition of the obtained powders under various technological conditions corresponds to the chemical composition of the ingot.

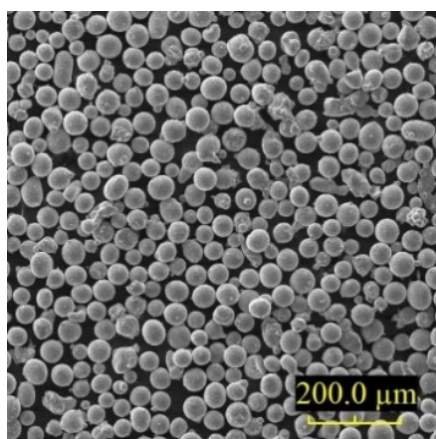
**Table 1.** Chemical composition of powders.

Material	Basic alloying elements, wt. %					
	Fe	Cr	Mn	Ni	Mo	others
Ingot	60.5	20.8	10.5	5.9	1.7	0.6
Powder (30 bar argon)	61.1	19.3	10.1	7.3	1.4	0.8
Powder (50 bar nitrogen)	59.7	20.6	10.0	6.7	2.0	1.0

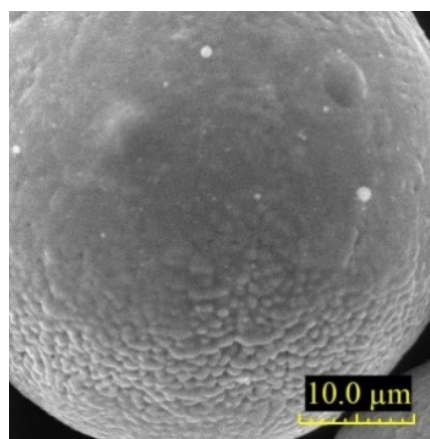
The granulometric composition of the powders was measured by laser diffraction using the Fraunhofer approximation by Malvern Mastersizer 2000. According to the results of particle size analysis, powder fractions of more than 80 µm were excluded, which are not suitable for using in SLM technology. The share of raw materials suitable for fusion was 70%, in which the 0-20 µm fraction was 1%, the 20-40 µm fraction was 40%, the 40-63 µm fraction was 10%, and the 63-80 µm fraction was 5%. Powder sieving by fractions used a sieve system on a shaker table. To determine the content of nitrogen and oxygen in the powder, the complex of gas analyzers LECO CS-230, TC-400 was used. According to the results of the nitrogen content measure before spraying, the value was 0.39%. Structural studies were performed by a TESCAN Vega 3 scanning electron microscope (SEM). The non-destructive testing of the elemental composition was performed by an Inka X-ray fluorescence attachment on a TESCAN Vega 5136-LM scanning electron microscope.

### 3. Experimental results and discussion

The particles morphology of the obtained high-nitrogen steel powder of 04Cr20Ni6Mn11Mo2NVNb is demonstrated in Figure 1. It can be concluded that the particles have a shape close to spherical, with no particles of irregular shape, which makes the powder suitable for using in the SLM technology. The selected pressure values at the nozzle provide particles of the specified form with a maximum proportion of available powder. In some powders production methods [7], on the surface of the powder particles, one may see foreign inclusions that the quality characteristics of the final product worse. Figure 2 shows the characteristic appearance of particles after gas atomization: it is clear that the surface of the powder particles does not contain satellites.



**Figure 1.** The morphology of the particles of the obtained high-nitrogen steel powder of 04Cr20Ni6Mn11Mo2NVNb



**Figure 2.** The characteristic appearance of high-nitrogen steel particles after gas atomization

The measurement results made by gas analyzers of nitrogen and oxygen in the powders obtained in different technological conditions are presented in table 2.

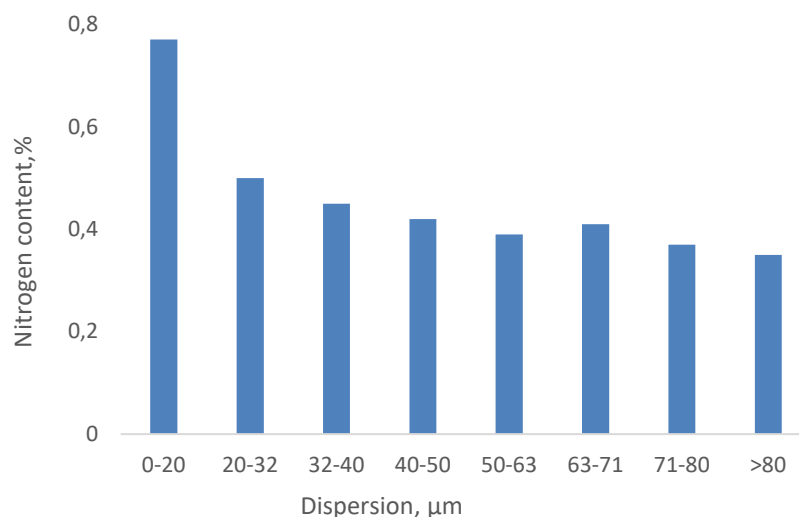
**Table 2.** The content of nitrogen and oxygen in the obtained powders of steel 04Cr20Ni6Mn11Mo2NVNb.

Pressure, Bar	Melt gas	Tmelt., ° C	Dispersity, μm	Oxygen,%	Nitrogen,%
30	Ar	1600	50-100	0.020	0.250
30	Ar	1600	50-100	0.020	0.270
			0-20	0.064	0.771
			20-32	0.061	0.500
			32-40	0.060	0.450
			40-50	0.062	0.420
50	N	1500	50-63	0.050	0.390
			63-71	0.050	0.410
			71-80	0.060	0.370
			>80	0.040	0.350

It is known [8] that at low pressure above the melt nitrogen is removed from steel. Therefore, an optimal pressure was chosen that impedes this process. Argon was used to study diffusion processes taking place with the melt in the crucible before spraying (Table 2 shows that the use of argon leads to the nitrogen content decrease in the melt and in the powder). The argon pressure above the melt was 0.25 bar, at the nozzle at a spray of 30 bar, with the nitrogen content decrease in the powder compared to the initial ingot (~ 0.26% versus 0.39%, respectively). It enables to suggest that the process of nitrogen diffusion from the melt proceeds. When nitrogen is used (at excessive pressure above the melt of 0.25 bar, as in case of using argon), the melt is saturated with nitrogen, and during the subsequent spraying (50 bar at the nozzle), the melt drops are further saturated.

The nitrogen content in the fractions of the nitrogen sprayed powder obtained by sieving through a sieve system, is graphically represented (Figure 3). A regular occurrence has been revealed: in particles of low dispersion, the nitrogen content exceeds that value even in the initial ingot. The decrease in nitrogen content depending on the particle diameter (fractional composition of the powder mixture) can be explained by diffusion processes, namely, the different depth of nitrogen diffusion into the powder particle from its surface at the moment of spraying, when the powder looks like metal

droplets (in the liquid phase). Accordingly, in a particle with a smaller diameter, the volume fraction of nitrogen is higher than in particles with a larger diameter. In this case, the time interval of the diffusion process is insignificant.



**Figure 3.** Nitrogen content in various powder fractions

The issue of high-nitrogen steels is to maintain a non-equilibrium nitrogen content (heat treatment modes, melt cooling rates). Therefore, obtaining the final part from high-nitrogen steels is associated with a number of challenges. Additive technologies provide prerequisites for solving this problem: at each stage, when the alloy undergoes a transition to a liquid state (sputtering in the atomizer and subsequent selective laser melting), it is possible to control the partial pressure above the melt, the cooling rate of the melt.

A further research continues with a more detailed study of the structure and properties of the additive samples (according to the SLM technology) from obtained powder materials. The nitrogen content and the phase composition of the melted sample are of importance. Samples are supposed to be grown in nitrogen to maintain (or even increase) their nitrogen content. The phase composition is determined by the ratio of alloying elements; therefore with nitrogen (nickel equivalent) and chromium contents one can obtain a fully austenite or an austenite-ferrite structure.

#### 4. Conclusions

Powder of high-nitrogen steel 04Cr20Ni6Mn11Mo2NVNb of necessary dispersion (less than 80 microns) and morphology (sphericity) that meets the requirements of equipment used for selective laser melting of metallic powder materials was obtained by melt spraying in a nitrogen atmosphere by HERMIGA 75/3VI atomizer.

It was determined that when argon is used during atomization of the high-nitrogen steel ingot 04Cr20Ni6Mn11Mo2NVNb, the nitrogen content in the final product (powder) decreases in comparison with the initial ingot.

The nonequilibrium content of nitrogen in a metallic powder material with dispersity of 0–20 μm with about 0.7% of nitrogen (in the initial ingot – 0.39% of nitrogen) is obtained. The increasing of nitrogen content in steel in powders production for additive technologies by HERMIGA 75/3VI gas atomizer is investigated. Thus, a number of technological challenges to obtain high nitrogen samples are addressed.

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