Research of the possibility of using an electrical discharge machining metal powder in selective laser melting

A A Golubeva, A V Sotov, A V Agapovichev, V G Smelov and V N Dmitriev

Samara University, 34, Moskovskoe sh., Samara, 443086, Russia

E-mail: agapovichev5@mail.ru

Abstract. In this paper the research of a Ni-20Cr-10Fe-3Ti (heat-resistant) alloy metal powder conducted for use in a selective laser melting technology. This metal powder is the slime after electric discharge machining. The technology of cleaning and melting the powder discussed in this article. As a control input of the powder, immediately before 3D printing, dimensional analysis, surface morphology and the internal structure of the powder particles after the treatment were examined using optical and electron microscopes. The powder granules are round, oval, of different diameters with non-metallic inclusions. The internal structure of the particles is solid with no apparent defects. The content of the required diameter of the total volume of test powder granules was 15%. X-ray fluorescence analysis of the powder materials carried out. The possibility of powder melting was investigated in the selective laser melting machine ‘SLM 280HL’. A selection of the melting modes based on the physical properties of the Ni-20Cr-10Fe-3Ti alloy, data obtained from similar studies and a mathematical model of the process. Conclusions on the further investigation of the possibility of using electric discharge machining slime were made.

1. Introduction

Modern industrial production has a tendency to search new materials and methods for their preparation. Currently, additive technologies introduced in all areas of production. Today, it is one of the most dynamically developing directions of “digital” manufacture. These technologies accelerate research and development, solve the problems of pre-production, and in some cases actively applied for the production of a functional product [1-6].

One of the directions of additive technology is the selective laser melting (SLM) of a metal powder composition. The development of this technology area has stimulated the development of technologies for producing metal powders. Today, the nomenclature of metal compositions has a wide range of materials based on Ni and Co (Co-Cr-MO, Inconel, Ni-Cr-Mo), on Fe (tool steel: 18%Ni300, H13; stainless steel: 316L), on Ti (Ti6-4, CpTigr1) and on Al (Al-10%Si-Mg, Al-12%Si).

Overall, the situation concerning the introduction of additive technologies in the Russian industry is extremely unfavorable. The aerospace production is in need of the introduction of this technology, because difficult-to-cut, refractory materials and alloys are used in the production of parts for rockets and aircraft engines.

The basic problem is production of powder materials for additive technologies [7]. Installations for the production of metal powders existing today in Russia are using different methods. The most widely used method is spraying a plasma jet of the rapidly rotating rod of the corresponding alloy in
an argon atmosphere. Such installations are expensive, and the possibility of the use of such powders in additive technology has not been studied.

In this paper, the treatment technology of electric discharge machining (EDM) of slime made of graphite, copper and mineral oils has been developed, the basic technological properties of the purified powder were studied and the use of EDM slime in SLM process has been investigated by the example of the Ni-20Cr-10Fe-3Ti alloy.

2. The methodology of the researches

The object of the research is the Ni-20Cr-10Fe-3Ti alloy in the form of EDM slime. EDM slime is a difficult-to-cut material [8-10]. This slime consists of micro powder, which consists of spherical particles with impurities of graphite, copper, and mineral oil. For cleaning the slime, the method for removing contaminants has been developed.

Dimensional analysis, the surface morphology and the internal structure of the powder particles after treatment have been investigated using electron and optical microscopes. These researches are necessary as the input of the powder should be controlled immediately before the SLM process.

For a detailed analysis of the chemical composition of the purified Ni-20Cr-10Fe-3Ti powder, spectral analysis using the optical emission spectrometer with inductively coupled plasma (PlasmaQuant PQ9000) conducted. This is a high-resolution spectrometer designed for high-precision multi-element analysis of liquid samples of various types with the minimum requirements for sample preparation.

The spectrometer has a range of 0.002 nm, which allows using the device for solving the most complex analytical problems. Unique optics and built-in database allow defining more than 75 elements. The possibility of melting the particles of the metal Ni-20Cr-10Fe-3Ti powder alloy was used on the basis of 3D machine SLM 280HL. The installation uses an infrared fiber laser with a wavelength of 1075 nm and a maximum power of 400 W, operated in a continuous mode. This machine has an open system that allows using its own production powders for the manufacture of parts.

3. Results and discussion

Since the additive manufacturing of metal powders sets sufficiently high quality requirements [1, 11], in particular, the requirements for morphology (spherical, the allowable number of defects), the grain size (the upper and lower limit of the particle size range is 0.03-0.05 mm), the requirements for process properties, the requirements for the chemical composition of the gas and impurities, the powder input control was conducted. In addition, the internal structure of the powder particles in the presence of pores investigated. Figure 1 shows an image of the Ni-20Cr-10Fe-3Ti alloy powder with pollution included therein.

![Figure 1. Slime of EDM.](image)

The preliminary analysis showed that the powder granules are round, oval and of different diameters with nonmetallic inclusions. The internal structure of particles is solid with no apparent defects. The content of the granules of the required diameter is 15% of the total amount of the investigated powder.
For obtaining a pure powder from slime, the clean technology of mineral oil, graphite and copper was developed and applied. The technology is based on the knowledge of chemical properties of the Ni-20Cr-10Fe-3Ti alloy metals and derivatives of EDM. Distilled water, baking soda, technical soap used as a chemical reagent. The centrifuge, the drying cabinet, the screen were used as equipment.

After cleaning the slime, the obtained powder materials sent to the X-ray fluorescence analysis (XRF). This method of analysis is one of the modern spectroscopic methods for studying the substance in order to obtain its elemental composition. It allows detecting various elements beginning from beryllium (Be) up to uranium (U). An XRF method is based on the collection and subsequent analysis of the spectrum upon irradiation of X-ray radiation of the material. The results of this study are shown in Figures 2 and Table 1 and 2. Figure 3 shows an image of the surface morphology of the particles of the Ni-20Cr-10Fe-3Ti alloy powder.

![Figure 2. Analysis of some quantity of the particles powder.](image_url)

| Element | ke, V | Mass, % | Counts | Error, % | Atom, % | K  |
|---------|-------|---------|--------|----------|---------|----|
| C K*   | 0.277 | 14.89   | 6311.02| 0.01     | 45.05   | 0.8603 |
| O K*   | 0.525 | 0.99    | 1103.47| 0.21     | 2.25    | 0.3273 |
| Al K*  | 1.486 | 0.64    | 937.85 | 0.42     | 0.86    | 0.2476 |
| Ti K*  | 4.508 | 5.01    | 3650.23| 0.11     | 3.80    | 0.5002 |
| Cr K*  | 5.411 | 19.35   | 11579.05| 0.04    | 13.52   | 0.6092 |
| Fe K*  | 6.398 | 7.03    | 5093.45| 0.08     | 7.00    | 0.7707 |
| Ni K*  | 7.471 | 38.29   | 13963.46| 0.03    | 23.70   | 1.0000 |
| Mo K*  | 2.293 | 10.07   | 3348.89| 0.13     | 3.81    | 1.0964 |
| Total  |       | 100.00  |        |          |         |     |

| Element | ke, V | Mass, % | Error, % | Atom, % | K  |
|---------|-------|---------|----------|---------|----|
| C K*   | 0.277 | 26.97   | 2.05     | 63.56   | 2.1505  |
| Al K*  | 1.486 | 1.09    | 0.91     | 1.14    | 0.1773  |
| Ti K*  | 4.508 | 3.18    | 0.47     | 1.88    | 1.7552  |
| Cr K*  | 5.411 | 15.81   | 0.62     | 8.61    | 9.0372  |
| Fe K*  | 6.398 | 7.03    | 0.70     | 3.56    | 4.0189  |
| Ni K*  | 7.471 | 41.22   | 1.08     | 19.87   | 22.3395 |
| Mo K*  | 2.293 | 4.69    | 1.27     | 1.38    | 1.8602  |
| Total  |       | 100.00  | 100      |         |     |
For a detailed chemical composition of the purified powder, a unique PlasmaQuant PQ9000 spectrometer used. The spectrograph deals with the solutions of any substance. To obtain the solution, the powder boiled in the hydrochloric acid solution (figure 4). The resulting solution filtered and loaded into the spectrograph. The chemical composition identified after analysis of the solution of the purified powder (table 3).

**Table 3.** The chemical composition of the purified powder.

| Chemical element | Mass, % |
|------------------|---------|
| Cr   | 15.82   |
| Ni   | 65.34   |
| Co   | 0.19    |
| Cu   | 0.14    |
| Zn   | 0.02    |
| Mn   | 0.01    |
| Fe   | 11.45   |
| Al   | 1.28    |
| Ti   | 1.47    |
| Mo   | 0.17    |

The precipitate (the powder remained after boiling in the hydrochloric acid) was dissolved in nitric acid. Then, the bulk of powder was analyzed with a spectrograph. The composition of the slime appeared to be similar to that of the bulk powder. The mass fraction of minor elements is not more than 0.01 %. Thus, a certain chemical composition of a purified powder should not prevent melting of the particles in the SLM machine.

After cleaning and researching of the powder, the melting process was conducted with the use of 3D machine SLM 280HL. The empirical search of the laser processing modes, which provide the necessary structural and phase composition, is extremely difficult and labor intensive. In fact, the
required quality layers formed in a very narrow range of laser modes. The selection of melting modes was carried out based on the physical properties of the Ni-20Cr-10Fe-3Ti alloy.

The results of melting were unsatisfactory. The synthesized material was sufficiently fragile, leading to detachment from the building platform (Figure 5). Selected laser modes were to ensure the melting of the powder particles, and this suggests an idea that the oxide film is present on the surface of the particles.

For the analysis of oxides, the additional X-ray analysis, which will identify the crystal structure of the powder inside and a classification of relations between the elements of the crystal lattice, is required. The results of X-ray diffraction are expected in the near future. The nitrogen as a protective gas could interfere with melting. Further melting will be carried out using argon as a protective gas.

![Figure 5. The melting results of the powder in the SLM machine.](image)

4. Conclusion

In this paper, the research on the possibility of using EDM slime in the selective laser melting technology showed in general a positive result. As a result of this work, we can conclude the following:

1. The treatment technology of EDM slime made of graphite, copper and mineral oils has been developed. Distilled water, baking soda, technical soap were used as chemical reagents. The centrifuge, the drying cabinet and the screen were used as equipment.

2. The surface morphology of powder investigated after cleaning EDM slime. The results of analysis showed that the granules of powder have a round shape with different diameters. The content of the required diameter granules (25 ... 35 microns) of the total powder volume was 15%. The mass fraction of minor elements is not more than 0.01 %.

3. After analyzing of the properties, the metal powder was melted in the SLM machine, where the synthesized material was sufficiently fragile. These data suggest the presence of the oxide film on the powder surface, preventing melting.

Acknowledgements

These studies were conducted on the basis of the equipment of CAM Technology Center (RFMEFI59314X0003). This work was supported by the Ministry of Education and Science of the Russian Federation in the framework of the implementation of the Program “Research and development on priority directions of the scientific-technological complex of Russia for 2014-2020”.

References

[1] Zlenko M A, Popovich A A and Mutylin I N 2013 *Publishing house of the Polytechnic University* (Saint Petersburg)
[2] Vdovin R A and Smelov V G 2014 *Int. J. of Eng. and Tech.* **6(5)** 2269-2275
[3] Smelov V G, Sotov A V and Agapovichev A V 2016 *Key Eng. Mat.* **684** 316-322
[4] Grjaznov M U, Shoshin S V and Chuvildeev V N 2012 *Bulletin of the Nizhny Novgorod University named after N. Lobachevsky* **5(1)** 45-50
[5] Smelov V G, Sotov A V, Agapovichev A V, Tomilina T M 2016 *IOP Conf. Series: Materials Science and Engineering* **142**
[6] Sufiianov V Sh, Popovich A A, Borisov E V and Polozov I A 2015 *Tsvetnye Metally* **1** 79-84
[7] Gu D D, Meiners W, Wissenbach K, Poprawe R 2012 *Mat., Proc. and Mech. Int. Mat. Rev.* **57(3)** 133-164
[8] Ivanova I I, Serebrovskiy V I, Kolmykov V I 2012 *Southwestern State University (Kursk)*
[9] Fominskiy L P, Levchuk M V, Tarabrina V P 2010 *Powder metallurgy* **4** 1-6
[10] Fominskiy L P 2016 *Electrophysical and electrochemical methods of processing* **8** 6-8
[11] Kruth J P, Froyen L, Van Vaerenbergh J, Mercelis P, Rombouts M and Lauwers B 2004 *J. of Mat. Proc. Techn.* **149** 616-622