Analysis and development of methods for obtaining metallic powders for selective laser melting

R N Kashapov\textsuperscript{1,2}, L N Kashapov\textsuperscript{1} and N F Kashapov\textsuperscript{1}
\textsuperscript{1} Engineering Institute, Kazan Federal University, Kazan, 420008, Russia
\textsuperscript{2} Laboratory of Radiation Physics, Kazan Physical-Technical Institute of the Kazan Scientific Center of the Russian Academy of Sciences, Kazan, 420029, Russia

E-mail: kashramiln@gmail.com

Annotation

The paper compares the existing methods for obtaining metal powder compositions and reveals their shortcomings. A technique for evaluating the properties of powders for suitability for use in selective laser melting (SLM) processes is proposed. Analysis of methods for obtaining powders has shown the need to investigate the possibility of using a plasma-electrolyte process in the production of consumables for SLM technology.

Introduction

Additive technologies, in particular the method of selective laser melting (SLM), are increasingly used in industry [1]. This is primarily due to the appearance of new materials for 3D printing of metal and ceramic products. [2]. In installations of selective laser melting, powder materials are used which must satisfy the following requirements: have a certain dispersion composition, the particles must be spherical. At the moment, several technologies are known for the production of metal powders for SLM: gas, water, plasma with a rotating electrode, plasma in crossed flows, and high-frequency discharge of atomization. In the first case, the molten metal is sprayed in a supersonic jet of an inert gas. In the second case, the spray is produced in a liquid stream. In the third one, an arc discharge is ignited between the electrodes, rotation of the melted electrode around its axis takes place, which leads to spattering of the metal. In the fourth case, the metal rod is sprayed in crossed plasma streams. In the fifth case, the pre-ground metal particles are passed through the installation with a high-frequency induction or capacitive discharge, and as a result, they take the form of a sphere. Disadvantages of these technological productions: a high dispersion of particle sizes from 1 μm to 200 μm, which requires additional cleaning and separation, the difficulty of transition of the plant to the production of another type of material, high energy consumption of the process, the difficulty of controlling and extracting particles of the nanometer range, the use of expensive equipment. The concept of additive manufacturing involves individual product geometry, as well as the ability to use a wide range of materials [3]. Now the main limitations are the shortcomings in the production of powders and the difficulty of a rapid transition from one type of metal to another. Thus, the next stage in the development of the SLM process is the creation of technologies for the production of micro and nanopowders, which are devoid of the above disadvantages. One solution is to use a gas discharge with liquid electrodes [4,5,6], this method is simple and does not require expensive equipment. Therefore, the aim of the work was to determine the possibility of its applicability by comparing the plasma-electrolyte method with the existing atomization technologies for powders.
Main part

Due to the fact that powders used in additive technologies are used in conditions other than standard technologies (powder metallurgy, powder coating) it is necessary to determine which of their properties are the most important and to choose the methods for measuring these parameters. Methods for determining the properties of powders used in standard technologies are known: determination of bulk density, powder flowability according to ASTM B213, measurement of the natural bevel angle. Of interest is the paper [7], which describes the device FT4 Powder Rheometer® (Freeman Technology) allowing to determine a wider class of dynamic and volumetric characteristics of the powder. The authors of the paper showed the effectiveness of studying the powder properties by the "shear cell" methods, measuring the possibility of passing a powder rotating through the flow, measuring the fluidity of the powder through a screw feeder, aerating to create a uniform powder distribution, determining the cohesion of powder particles, measuring the effect of air flow velocity on powder motion, determination of density, compressibility, permeability and air release. Also, an important conclusion is made in the paper about the deterioration of the dynamic properties of the powder after use in additive production machines. Thus, monitoring the properties of the powder is a factor in obtaining the product without internal defects.

Attention should be drawn to the paper [8], in which a comparative analysis of powders 17-4PH obtained by gas and water atomization is carried out. The morphology of particles about 3 μm in size is similar and has a spherical shape. However, for the powder obtained by the water atomization method, with an increase in the particle size to about 20 μm, the particle sphericity is disturbed. This, in turn, does not allow using it in laser melting technologies. Therefore, the main methods of powder production for SLM and LMD are methods of gas atomization, plasma and electric arc with a rotating electrode. These methods make it possible to control the size of the powder obtained while maintaining its sphericity.

It is of interest to obtain a powder in a gas discharge with liquid electrodes. Gas discharges with liquid electrodes have been studied quite well since the middle of the 19th century, and have found wide application in practice: the formation of surface microrelief, heat treatment of products, the formation of functional coatings, the cleaning of metal surfaces, water sterilization, nanoparticle production etc. Burning discharge can occur both on the metal anode and cathode, while the electrode can be immersed in the liquid, and may be above it. The combination of all these conditions with the variation in the shape of the current and the magnitude of the applied voltage provides a variety of options for searching for micron-size powder production regimes. Our starting point for the studies was a survey paper [9], which describes the main methods for obtaining metal nanoparticles and their oxides. Proceeding from this, the set of possible variants of studies was greatly reduced, and the experimental setup was optimized.

Figure 1 shows the functional scheme of the experimental setup, which consists of an electric power supply system 1, an electrolytic bath 2, an electrode system 3, an oscilloscope 4, an additional resistance 5, a voltmeter 6, an ammeter 7 and a thermocouple 8. With the help of the electrode system, the distance between the anode and the electrolyte solution was monitored. With the help of an oscilloscope 4, the shape of the applied voltage and current was monitored, the voltmeter and ammeter were used to measure the voltage and discharge current. The electric power supply system represents a high-voltage DC power source for creating and maintaining electric discharge combustion with smooth regulation of the output voltage in the range from 0 - 3 kV and current 0-10A. Voltage and discharge current were measured by means of two digital universal measuring devices MMH-930 and APPA 109N, the relative error of measurement is 0.8%.
Burning gas discharge occurs between the metal anode made of steel grade S17400 / 630 and an electrolytic cathode. The anode is a metal cylinder with a diameter of 5 mm, located above the surface of the electrolyte at a height of 1 to 5 mm. As the electrolytic cathode, aqueous solutions of NaCl and Na₂CO₃ with a concentration of 0.1-1% by weight were used.

When certain current and voltage values are reached, a process of spraying the metal anode is observed, most of the powder enters the electrolytic cathode and crystallizes. In parallel, there is a process of evaporation of the liquid electrode. The resulting powder was washed with deionized water and dried in a drying oven. The morphology of the powder was studied using a scanning electron microscope, Carl Zeiss EVO 50. The dispersion composition was determined by sieving with a sieve set of sieves of from 10 to 300 microns. The average sieving time was 30 minutes. The microhardness was measured with a PMT-3M device, using the Vickers method. The powder was mixed with cyanoacrylate and pressed into tablets, which were then ground and polished. The resulting microsections were examined at a load of 50 gauss, a holding time of 25 s and 5 prints. Analysis of the chemical composition was determined with the aid of a scanning electron microscope attachment of an energy-dispersive electron-probe spectrometer Oxford Instruments Inca X-act.

The main parameters of plasma-electrolyte production of steel powder are voltage, current, discharge power, used for heat generation, physical and chemical properties of the metal anode. The power put into the discharge is determined by the current-voltage characteristic, on the basis of which it is possible to estimate the energy contribution. Figure 2 shows the current-voltage characteristic of the plasma-electrolyte process for three interelectrode distances.

Fig. 1. The scheme of the experimental setup

Fig. 2. Volt-ampere characteristics of the plasma-electrolyte process

Fig. 3. Dependence of the anode temperature on the power
The significant effect on the discharge combustion process is exerted by the electrolyte concentration, it is established that the electric field strength decreases with increasing concentration. Therefore, for stability of the results, solutions with a concentration of less than 1% were used.

Figure 3 shows the linear dependence of the change in anode temperature with increasing discharge power.

The formation of the powder occurs when the surface temperature of the anode reaches the appropriate solidus temperature. When the liquidus temperature is reached, the electrode is melted and large drops of metal are formed. Proceeding from this, the powder can be produced in the temperature range of the liquidus and solidus anode. An increase in temperature will lead to an increase in the productivity of the powder, but with an increase in the particle size of the powder. Figure 4 shows the dependence of the powder performance on the anode temperature. It has a nonlinear dependence and increases with increasing temperature. A linear increase in the average particle size with increasing anode temperature is established. Figure 5 shows the histogram of the granulometric composition of the obtained powder. The largest amount of powder is obtained by a size smaller than 40 μm. Figure 6 shows SEM images of the resulting powders. The powder has a spherical shape, the smallest particle size is 0.5 μm. The resulting powder is suitable for use in selective laser alloying plants. However, the need for further research related to the increase of the process productivity by increasing the anode area definition and dynamic characteristics of the powder.
Fig. 6. SEM images of steel powder obtained in the plasma-electrolyte process

**Conclusion**

Studies of the combustion of a gas discharge between a metal electrode S17400/630 and liquid electrodes have shown the possibility of obtaining a spherical powder with a particle dispersion from 0.5 to 40 μm. Analysis of the obtained SEM photographs shows similarity with the powder obtained by the gas atomization method. The influence of the gas discharge parameters on the process productivity and the size of the particles obtained is determined. In the voltage range from 500 to 800 V, particles smaller than 40 μm in size with a productivity of 1 • 10⁻² g / s occur. Above this range, the electrode is melted and the process for obtaining the powder ceases. The location of the metal electrode affects the physicochemical processes occurring in the discharge and the mechanism of particle formation.

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**References**

1. Journal of Alloys and Compounds Volume 691, 15 January 2017, Pages 316-322
2. International Journal of Heat and Mass Transfer Volume 104, 1 January 2017, Pages 665-674.
3. Scripta Materialia Volume 126, 1 January 2017, Pages 41-44.
4. L. Kashapov, N. Kashapov and R. Kashapov, Journal of Physics: Conference Series. Volume 479, Issue 1, 2013, Article number 012011
5. D. Denisov, N. Kashapov and R Kashapov, IOP Conference Series: Materials Science and Engineering, Volume 86, Issue 1, 26 June 2015, Article number 012005.
6. L. Kashapov, N. Kashapov, R Kashapov and D. Denisov, Journal of Physics: Conference Series. Volume 669, Issue 1, 14 January 2016, Article number 012029
7. J. Clayton, Metal Powder Report. Volume 69, Issue 5, September–October 2014, Pages 14–17
8. B. Hausnerova, B. Mukund, D. Sanetrnik, Powder Technology, 312 (2017), p. 152 – 158.
9. T. Abdul Kareem, A.Anu Kaliami, Ionics (2012) 18: 315 - 327.