

Plasma electrolyte produce 17-4PH powder for use in 3D MicroPrint technology

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Abstract. The work is devoted to the investigation of the processes of obtaining fine powders from steel 17-4PH, for the purpose of their further application in 3D MicroPrint technology. This technology is a type of selective high-resolution laser melting of $\sim 30\text{ }\mu\text{m}$. In 3D MicroPrint technology, a powder of less than 5 microns is used, which in turn necessitates the development of new cheap methods for obtaining spherical fine powders. The process of plasma-electrolyte powder production of 17-4PH steel was studied, the discharge burning conditions and conditions for the arrangement of the electrode system for obtaining powders with dimensions less than $5\text{ }\mu\text{m}$ were selected. The proposed method is simple and does not require expensive equipment.

Introduction

The technology of selective laser melting proved itself as capable of manufacturing complex metal products in one production cycle. It is layered formation that makes it possible to manufacture complex and hollow products that can't be done by traditional methods. Despite all the advantages, SLM technology has drawbacks due to high surface roughness, cracking, distortion of geometry and the inability to grow parts with a wall thickness of less than $140\text{ }\mu\text{m}$. At the moment, commercial machines have a high level, they have eliminated the shortcomings associated with the formation of cracks and distortion of geometry. All this allowed us to start creating factories of additive production. It is the ability to form complex geometric products that makes this technology economically viable and competitive by combining a large number of operations in a single step. The technology of laser melting isn't used only in the manufacture of prototypes, but serial products of small batches.

The need for engineering in the creation of micron products with a microscopic structure only increases. Therefore, part of the research in the field of additive technologies is aimed at creating new devices capable of producing micron-sized parts by selective laser fusion. There are works by the authors Chen J. [1], Exner H. [2], Kathuria Y.P. [3], in which laser fusion of various powders with pulsed and q-switched pulse regimes was investigated. And in the work of Regenfuss P. and the authors of [4,5,6] a new experimental setup was presented, which allows to create details with a resolution of $30\text{ }\mu\text{m}$ and a roughness R_a of $1.5\text{ }\mu\text{m}$. The installation consisted of a sintering



chamber from which a turbomolecular vacuum pump was pumped out, ScanLab scanning system of 25 * 25 mm in size, a Q-switched Nd: YAG laser ($\lambda = 1,064 \mu\text{m}$) with an output power of 0.1-1 W in TEM mode and 0.5 - 100 kHz pulsed. In the installation there are three pistons, one - serves as the platform on which the part is grown, and it gradually moves downward, the remaining two are necessary for feeding the powder to the construction zone. These pistons go up and squeeze out the powder, which is carried by a knife to the working platform. As a result of the research, miniature products were obtained, which were tested in practice.

At Formnext 2017 (Frankfurt / Main), 3D MicroPrint GmbH presented the first DMP63 commercial machine capable of producing products with the above listed properties. This unit is equipped with a fiber laser power of 50 W, which works in CW and pulsed mode. Scanning is performed using a high-speed galvanometer, the laser spot diameter is 30 μm , and the device has a built-in laser power measurement system. The size of the platform is 60 * 60 mm, the height of construction is 30 mm. The particle size of the powder used is $D_{90} \leq 5 \mu\text{m}$, the layer thickness of the applied powder is $\leq 5 \mu\text{m}$, the tolerance of the dimensional deviation is $\pm 5 \mu\text{m}$ by 10 mm. Selective laser melting takes place in an inert argon medium, the gas flow rate is 30 liters per hour, the concentration of oxygen and water vapor is less than 0.1 ppm. Comparison of the claimed characteristics of the obtained products with the capabilities of other machines shows the advantages in the ability to make thin-walled structures with a thickness of 32 microns, a roughness of $R_z 13 \mu\text{m}$, $R_a 2 \mu\text{m}$ and a bulk density of 99.9%. However, for the operation of this machine, strict requirements are imposed on the powder, namely its dimensions $D_{90} \leq 5 \mu\text{m}$ and the geometry - the spherical shape. The expected widespread use of such DMP63 in the production of precision medical products, aerospace, automation, electronics, energy and chemical industries requires us to develop new alternative methods for obtaining submicron powders. Existing methods of gas, plasma, water atomization make it possible to obtain powders in which the fraction of particles smaller than 5 μm is less than 1%. This shows the ineffectiveness of existing methods. A solution can be found in the joint use of plasma methods and fluid systems. The phenomenon of combustion of a gas discharge with liquid electrodes is known. The use of this phenomenon made it possible to obtain powders with a dispersion up to 100 μm . But it is also known that by changing the combustion parameters of a gas discharge, it is possible to control the size of the particles produced.

The aim of the work is to investigate the possibilities of plasma electrolyte systems for obtaining submicron powders suitable for use in 3D MicroPrint technology.

Main part

The phenomenon of obtaining micro-nanoparticles of metals and their oxides in a discharge with a liquid electrode has become known recently and is actively developing now [7]. However, nanoparticles obtained by this method are partially oxidized, agglomerated and have a wide spread in size. In [8, 9], a method of obtaining nickel oxide particles by Contact Glow Discharge Electrolysis (CGDE) was investigated when a metal electrode is located inside the electrolyte and the discharge burns in a vapor-air shell. In this case, the discharge combustion process takes place under the following conditions: $U \sim 30-42 \text{ V}$, $j \sim 0.1 \text{ A / mm}^2$. The nanoparticles obtained were 20 to 200 nm in size. As can be seen, there is a large enough spread. A method for obtaining micro- and nanoparticles using Glow Discharge Electrolysis (GDE) was also performed when the metal electrode is located above the surface of the electrolyte and they do not touch. In this way, Ag particles were obtained in [10, 11]. In work [12] it is assumed that it is possible to control the dimensions of nanoparticles by using a rotating electrode.

During the combustion of a discharge with liquid electrodes, various physical phenomena occur, including the formation of shock waves [13], while the electrolyte properties [14] can radically change the combustion process. Surface polishing [15] or the formation of a microrelief can occur. In [16, 17, 18], a positive effect was obtained for the production of powder microparticles with sizes up to 100 μm . All this suggests that a gas discharge can be used to produce particles smaller than 5 μm .

The experimental setup was used in the work, the functional scheme of which is shown in Figure 1.

The installation 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, 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 constant current source for creating and maintaining electric discharge burning with smooth regulation of the output voltage in the range from 0 - 3 kV and current 0-10A. Measurement of voltage and discharge current was carried out using two digital universal measuring devices MMH-930 and APPA 109N, the relative error of measurement is 0.8%.

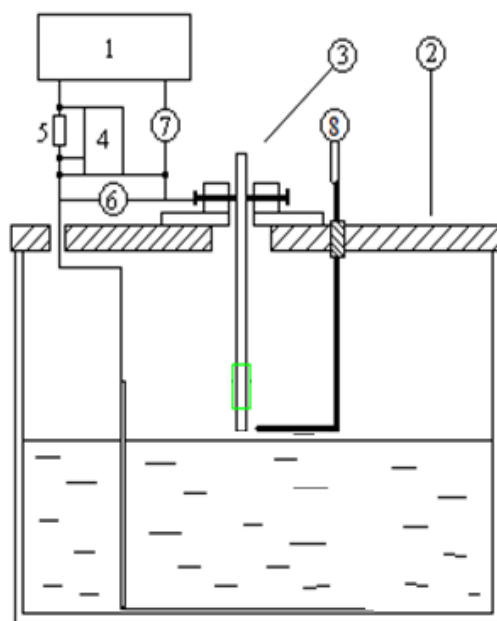


Figure 1 - Diagram of the experimental setup

The combustion of a gas discharge occurs between a metal anode made of 17-4PH steel 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 were used with a concentration of 0.1-1% by weight.

The main parameters of plasma-electrolyte steel powder production 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. All these factors affect the size of the particles obtained [16]. In our work, we added one more factor - the rotation of the electrode. This technique was previously used in [12] to obtain Pt and Au nanoparticles with dimensions of 10 -150 nm. Thus, by increasing the frequency of rotation of the electrode, we must come to reduce the size of the particles obtained.

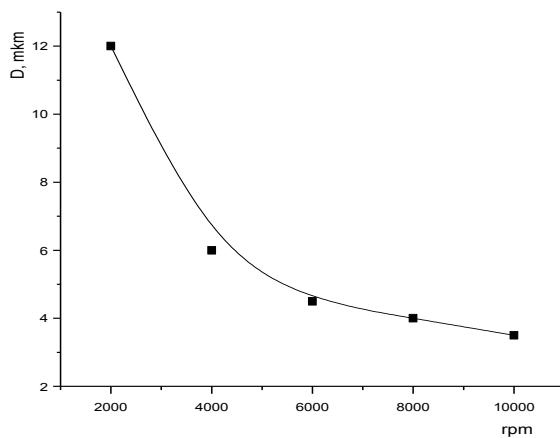


Figure 2 - Dependence of particle size on electrode rotation frequency

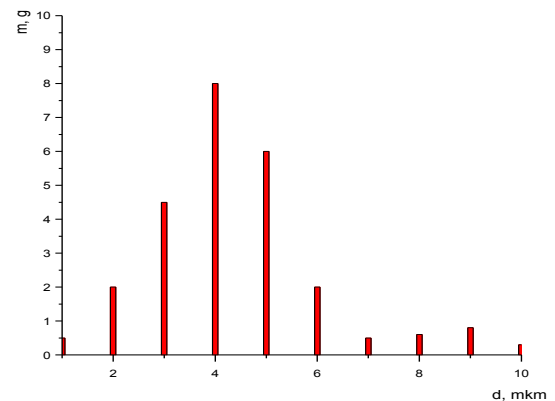


Figure 3- Powder distribution histogram

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 the stability of the results, solutions with a concentration of less than 1% were used.

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. A linear increase in the average particle size with increasing anode temperature is established. With an increase in the rotation frequency of the electrode, a nonlinear decrease in the dimensions of the produced particles is observed. Figure 2 shows the dependence of the particle size on the rotational speed. At a maximum speed of 10,000 rpm, the average particle size was 3.5 μm. Reducing the speed to 2000 rpm leads to a mean particle size of 12 microns. Figure 3 shows the histogram of the granulometric composition of the powder obtained. The largest amount of powder is obtained by a size of less than 4 μm. The powder has a spherical shape, the smallest particle size is 0.1 μm. The resulting powder should be suitable for use in selective laser melting plants using 3D MicroPrint technology. However, further research is needed to increase the productivity of the process by increasing the anode area and determining the powder dynamic characteristics.

Conclusion

Studies of the combustion of a gas discharge between a metal electrode of grade 17-4PH and liquid electrodes with a rotating electrode have shown the possibility of obtaining a spherical powder with a particle size of 3.5 μm in average. The influence of the gas discharge parameters on the process productivity and the size of obtained particles are determined. In the voltage range from 500 to 800 V, particles of less than 12 μm in size with a productivity of $1 \cdot 10^{-2}$ g/s occur. Above this range, the electrode melts and the process for obtaining the powder ceases. The electrode rotation can be considered as another effective tool for controlling the size of the particles produced.

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