

Influence of temperature and time factor on process of spraying of metallic powders in a plasma atomizer

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Abstract. The development of additive technologies is hampered by the almost complete absence of domestic systems for the production of metal powders. A prototype installation of a plasma atomizer was developed and manufactured, which makes it possible to obtain low-tonnage batches of metal powders of various chemical compositions. However, when the technology was transferred from the laboratory to production, a number of additional issues arose associated with a decrease in the quality of commercial products as the time of continuous spraying increases. It was found that the reason for this is the formation of powder deposits on the walls of the spray chamber and the increase in its temperature. Using the method of mathematical modelling, the maximum permissible temperature of the walls of the spraying chamber was determined, ways of stabilizing the quality of the powder during industrial production were shown.

1 Introduction

Modern mechanical engineering is rapidly changing, abandoning mass production of so-called universal devices with average technical characteristics, towards the creation of machines and mechanisms clearly oriented to a specific consumer with a predetermined set of useful properties. Such changes in the approach to production led to the need to change the ideology of production, abandoning multi-batch operations of subtractive technologies, for example, turning, milling, drilling, in favor of manufacturing parts using additive technologies. This is indicated by the intensive growth of additive manufacturing, whose annual world growth is tens of percent [1, 2] .

It should be emphasized that the rejection of traditional subtractive technologies, not only simplifies and dramatically reduces the production cycle and parts production, but also contributes to the improvement of the environmental situation by reducing the volume of production waste. So, if with the traditional production method the metal utilization rate rarely exceeds 30-45% - that is, more than a half of the produced metal is sent to waste and recycled, then in the case of additive technologies the metal utilization rate does not fall below 90-95% [3-7] .

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Intensively increasing use of machinery required to revise additive relation to the entire production cycle - from the preparation of starting powders to mechanical and thermal treatment obtained in 3D Printer, and final products.

One of the areas, hampering the development of additive technology, is an underdeveloped production base metal powders. The difficulties associated the weak production powder supply have led to the fact that in recent years there has been a gradual decrease in the growth rates of additive manufacturing, a gap arises between the volume of products produced by the world's leading centers of additive manufacturing and domestic enterprises.

Peter the Great St. Petersburg Polytechnic University adopted a policy of the most promising areas of engineering, including the creation of equipment for additive technologies - from powder production, to the creation of machines and products of production technologies on the 3D Printer, using different printing methods. In particular, research has been carried out and technology and equipment have been developed for the production of spherical metal powders by the method of plasma spraying. A schematic diagram of a plasma atomizer is described in [8-11]

At present, the technology for the production of powders is at the stage of transition from a laboratory facility to a industrial production. Such transition demanded partially revise the design of one of the basic units atomizer – column sputtering , as during long continuous (order of one work shift) process sputtering metal feedstock was observed a gradual decrease the quality of produced powders, increasing the average diameter of spray powder and reduce yield and commercial fractions to 40-140 microns. In order to clarify the reasons for this phenomenon, physical and mathematical experiments were carried out, which made it possible to stabilize the production process.

2 Materials and methods

As material for the experiments it was used a stainless wire hoist brand AISI 304/304L (08Cr18Ni10) diameter 2 and 4 mm. The wire was sputtered in a plasma atomizer developed and manufactured at Polytechnic university according to one and three plasma sputtering schemes [12].

The spraying was carried out in the following modes: the current supplied to the plasmatron was 300A, the voltage was 130V, the plasma-forming and shielding gas was high-purity argon 5.5. Wire feed speed 3 m / min, plasma gas feed speed 50 l / min for each plasma torch. Shielding gas flow rate from 10 to 20 l / min (or approximately 1-2 l / min per nozzle). The experiment lasts from 10 to 300 minutes.

To measure the temperature of the walls of the spray chamber, 11 thermocouples were installed on its surface - from the upper cover of the spray chamber (point 1) to the lower point of the powder collection system (point 11).

3 Results

Table 1 shows the results of measurements of the wall temperature of the spraying chamber during operation of the atomizer in one- and three-plasmatron schemes, and Table 2 shows the change in the proportion of the yield of commercial fractions during prolonged spraying. To obtain an objective picture, the sampling of the powder was carried out within 5 minutes before and after the control time, that is, when assessing the quality of the powder, for example, after 180 minutes of spraying, powders were taken, obtained in the period from 175 to 185 minutes of the process.

Table 1. Temperatures of the top cover and walls of the spraying column depending on the duration of the spraying process - numerator - when one plasmatron is operating, installed in the center of the top cover; the denominator - three plasmatrons.

Point No.	Temperature, °C, measured after spraying for						
	5 minutes	10 minutes	30 minutes	60 minutes	120 minutes	180 minutes	300 minutes
1	39/47	47/56	95/128	154/176	181/197	206/218	207/222
2	41/51	51/59	98/125	161/178	180/202	211/231	210/233
3	42/54	51/59	99/125	165/191	182/207	210/233	209/235
4	41/56	52/61	98/123	164/189	177/197	204/228	203/231
5	39/54	50/58	97/122	165/188	175/197	190/229	189/230
6	39/48	48/57	94/119	157/164	172/193	183/219	187/220
7	36/48	48/54	91/115	148/160	156/185	167/202	170/204
8	34/44	47/52	87/112	142/159	154/171	164/183	167/184
9	33/42	45/48	86/99	123/138	142/156	159/177	163/178
10	29/35	42/45	80/91	121/126	132/153	145/168	145/168
11	29/35	40/41	81/89	117/119	127/148	129/154	129/155

Table 2. Percentage of yield depending on the duration of the spraying process.

Number of plasmatrons	Percentage of usable yield after spraying within						
	5 minutes	10 minutes	30 minutes	60 minutes	120 minutes	180 minutes	300 minutes
1	31	30	27	25	24	23	23
3	29	29	26	23	22	20	19

The analysis of the results of monitoring the ratio of the chamber temperatures and the percentage of the yield of suitable powder allows us to assert that an increase in the temperature of the spray chamber wall leads to a decrease in the quality of the product. During the separation of the sprayed mass of the powder into fractions, it was noted that a decrease in the percentage of the yield of the suitable one with an increase in the spraying time is associated with a change in the particle size distribution of the powder. When conducting a sieve analysis of the sprayed mass, it was found that with an increase in the spraying time, the average particle size of the powder increases, and the process of intensive growth of particles of large-sized fractions proceeds most intensively in the time interval of 30-120 minutes. With a longer spraying time, there is a gradual stabilization in the yield of marketable fractions, although the total proportion of particles of coarse fractions is gradually (but with less intensity) still increasing. The results of studying the effect of the spraying time on the particle size distribution and the sprayed mass of the powder are shown in Table 3.

A review of the inner surface of the column showed that with an increase in the spraying time, deposits of powder masses accumulate on its surface , moreover, if after 10-60 minutes of spraying, these are powders of fractions less than 20 microns, then with an increase in the spraying time, particles of larger particles appear in the powder deposits - commercial fractions 40-140 microns - Fig . 1. Since the oxidation of powder particles is possible during the opening of the column and access to its cavity of atmospheric air, the deposits on the walls of the column cannot be used in the finished product. Thus, a decrease in the performance of the atomizer and an increase in d₅₀ powder product is associated with the appearance of deposits of the sprayed mass of powder on the walls of the chamber.

As the spraying process lengthens, the thickness of the loose deposits of the powder product increases, which leads to an increase in the temperature of the chamber wall. Attempts to determine the areas where sediments accumulate most intensively, empirically

did not lead to an unambiguous answer, therefore, it was decided to conduct an analysis using the method of mathematical modeling of the process. The formation of deposits is associated with the transfer of sprayed particles by gas streams inside the spraying chamber, therefore, using the method of mathematical modeling of the process in the software ANSIS 2019 R1, the movement of gas flows in the spraying chamber was simulated depending on the spraying time - that is, on the temperature of the chamber wall in [13-15]. The simulation results are shown in Fig. 2.

Table 3. Influence of the spraying time on the dispersion of the powder mass obtained by spraying stainless steel wire (as a percentage, rounded to the nearest whole).

Fraction, μm	Spraying time, min						
	5	10	30	60	120	180	300
≤20 *	3	4	2	<2	<1	<1	<1
20-40	5	4	3	<2	<1	<1	<1
40-60	7	6	5	3	2	2	1
60-80	8	7	6	4	3	2	3
80-100	8	8	7	8	9	9	8
100-140	8	9	9	10	10	10	11
Total fractions 40-140	31	30	27/26	25	24	23	23/19
140-200	14	15	18	19	16	16	15/17
≥200	1	1	1	1	1	1	rest

• Note - the accuracy of determining the volume of the fraction "less than 20 microns" is limited by the complexity of collecting powders of fine fractions and is not accurate enough

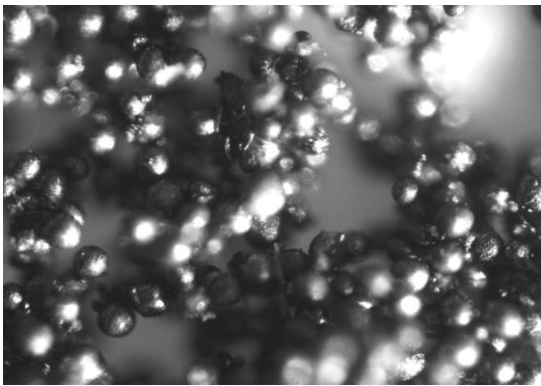


Fig . 1 . Deposits from oxidized powder particles on the side surface of the chamber after 300 min of the process.

As follows from the data in Fig. 2, with an increase in the temperature of the chamber wall, looped vortex gas flows appear inside it, leading to the displacement of fine powder fractions into the peripheral zone and the formation of deposits on the chamber walls. Moreover, the higher the wall temperature, the more intense the vortex flows, and, consequently, the greater part of the powder settles on the walls.

It was found that the process time is one of the most significant factors in the process of powder spraying in industrial conditions - with continuous operation of the atomizer for from one hour to one 8-10 hour shift - with an increase in the process time, the average particle size of the powder grows and the percentage of the yield of suitable - marketable fractions in the atomizer powder collection chamber decreases, the volume of deposits on the inner surface of the chamber walls increases.

The optimal parameters of the atomizer operation (in terms of the yield and particle size distribution of the powder), according to the results of physical and mathematical experiments, were obtained at an atomizer wall temperature not higher than 100-105°C. Maintaining this temperature level is possible only by using a forced cooling system for the walls of the spray chamber. Such cooling is achieved by using a forced water cooling system for the chamber walls and supplying a cold protective gas to its middle part. Taking into account the complexity of wall temperature control over the entire height (more than 3 m) in industrial conditions, mathematical modeling of thermal processes was carried out in order to determine the most heat-stressed zones of the spray chamber during its forced cooling. The simulation results for a single-plasma sputtering scheme are shown in Fig. 3. Temperature data are taken from the results of experiments - Table 1.

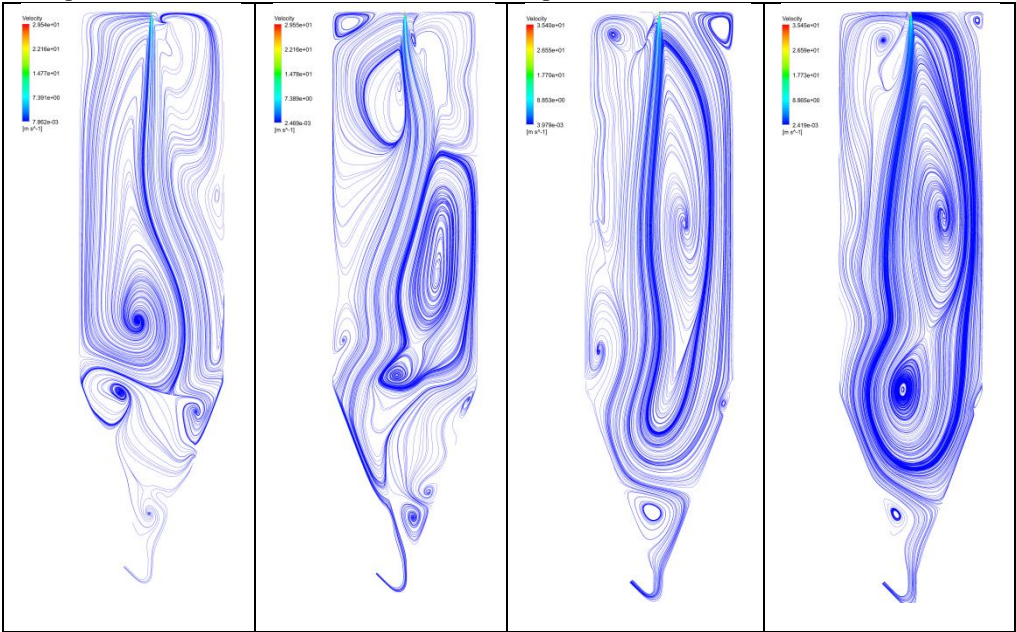


Fig. 2 . Influence of the chamber wall temperature on the directions and velocity of gas flows.

Calculations have shown that the maximum temperature of the chamber walls, subject to the condition that the temperature of the coolant at the outlet from the chamber does not exceed 55°C, is achieved in its middle part. To reduce the heat stress in this zone, additional cooling is required, created by the flow of cold shielding gas introduced into the column volume through the counter-flow system located in the middle of the column.

Simulation has shown and experimentally confirmed that to provide the necessary conditions for spraying, leading to a quality product, 10-12 l/min of protective gas is sufficient. If necessary, for example, in the event of a sharp increase in the outside air temperature, ensure high-quality operation of the atomizer by increasing the supply of shielding gas to 15-20 l/ min.

Another factor affecting the quality of the powder obtained is the temperature of the gas filling the column volume during spraying. This gas serves as the main source of heat transfer from plasma torches and molten metal particles to the walls of the sputtering chamber. The gas volume consists of two components - high-temperature - plasma-forming gas and low-temperature - protective gas supplied to the column through the counterflow system.

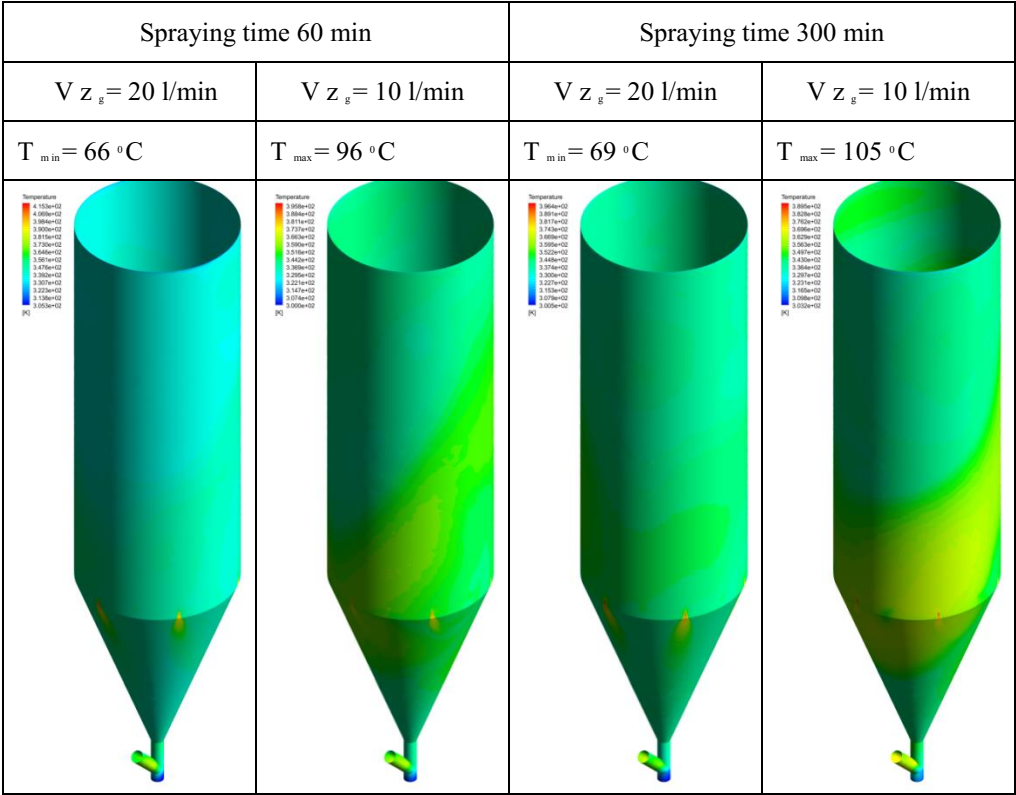


Fig. 3. Influence of the process time and volume of shielding gas supply on the formation of zones of local increase in the temperature of the walls of the spray chamber

The temperature of the gas inside the spraying column determines the rate of crystallization of powder particles, its mechanical strength, which makes it possible to maintain a spherical shape when the particles collide with the surface of the cone of the powder collection system. The lower the gas temperature, the more likely it is to expect spherical particles without internal pores and satellites. Calculations have shown that the gas temperature inside the spraying column increases with an increase in the plasma-forming gas feed rate, but the absolute values of temperatures are relatively low and outside the plasma torch are 330-380°C.

4 Conclusions

1. It has been shown that the automatic transfer of the results of laboratory developments to the industrial production of powders is impossible, since with an increase in the spraying time beyond 30 minutes, a gradual decrease in the yield of commercial fractions (40-140 μm) and an increase in the average particle size (d_{50}) in the commercial product. With an increase in the spraying time to 300 minutes, the yield decreases by 3-5%, and d_{50} increases by 20-40 microns.
2. The reason for the decrease in the output of commercial fractions is a sharp rise in the temperature of the chamber wall. It is found that when dispersed in an uncooled chamber in the range of 0-120 minutes, a sharp increase in the wall temperature - to temperatures over

200°C. Subsequently observed gradual e constant prices stabilization temperatures and after 300 minutes races dusting they reach 235°C.

3. The reason for the increase in the temperature of the chamber wall and a decrease in the yield of commercial fractions is the formation of deposits of sprayed particles on the walls and the lid of the chamber. Loose powder deposits create an additional thermal insulating effect and, in turn, further increase the temperature of the chamber wall. At wall temperatures up to 130-150 °C, deposits are mainly formed due to small (up to 20 microns) powder fractions, when exceeding 150 °C, particles of commercial fractions are also observed in the deposits. Removal of deposits is possible only after opening the chamber, while the particles that form the deposits are oxidized and cannot be used in a commercial product.

4. By mathematical modelling of gas flows in the spraying chamber, it was found that with an increase in the temperature of the chamber walls, gas vortices develop in it, which, with an increase in temperature, form closed loops stretched over the entire length of the chamber and passing along its walls. In this case, the gas streams capture a part of the sprayed material and throw it onto the chamber walls, forming deposits.

5. The conducted experiments and the results of calculations determined the maximum allowable temperature of the chamber wall, at which the stability of the yield of commercial fractions is ensured during a long spraying process. It is shown that these conditions are provided at a maximum temperature of the chamber wall of 105 °C. This led to the need to design a two-layer water - cooled chamber for an industrial installation.

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