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SYNTHESIS AND SPHEROIDIZATION OF DISPERSE HIGH-MELTING (REFRACTORY) POWDERS IN PLASMA DISCHARGE

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ABSTRACT

One of the areas of using arc and radio-frequency induction (RFI) plasma in metallurgy is to obtain refractory metals and materials with spherical shape of particles. The main advantages of using spherically shaped particles are high purity of particle surface, high bulk density (minimum surface/volume ratio) and ability to gain control on porous article properties and to separate the particles in fractions. Spherical particles are needed in the formation of powder-metallurgy elements with desired and uniform porosity, which are operated at high temperature, in highly aggressive media and at high velocity fluid flows.

The present work considers the possibilities of using arc and RFI-plasma in metallurgy to obtain high-melting point metals and materials with spherical shape of particles.

KEYWORDS: Arc plasma, RFI plasma, refractory materials, spherically shaped particles

1. Using powder-like materials with spherical particle shape in powder metallurgy and surface welding

One of the areas of using arc and radio-frequency induction (RFI) plasma in metallurgy is to obtain spherical and refractory metals and materials, [1, 6, 9, 12÷17].

It is known that plasma treatment of the initial materials aimed at producing disperse powders with particles of spherical-shape still finds broader application and has become a very perspective method, allowing to produce powders from W, Mo, Cr, Ta, B, Al, oxides, carbides, silicates, etc.

The main advantages of using spherically shaped particles are high purity of particle surface, high bulk density (minimum surface/volume ratio) and ability to gain control on porous article properties and to separate the particles in fractions.

Spherical particles are needed in the formation of powder-metallurgy elements with desired and uniform porosity, which are operated at high temperature, in highly aggressive media and at high velocity fluid flows.

Powders with spherically shaped particles of high-melting materials make it possible to substantially enhance the performance parameters of the produced articles. For example, porous filters of high-melting-point materials intended for purification of fuels, oils, aggressive liquids and gases contribute to attain higher effectiveness of the processes in chemical, metallurgical, food, pharmaceutical and a number of other industrial areas.

Another branch of high-temperature technique, where powders with spherical particle form still find wider application, is surface welding of complexly shaped elements intended for heavy operation conditions.

The quality of the surface-welded layer depends on the regular feeding of the welded material in the gas-flame or plasma devices, [5, 8].

Only powders with particles of spheroid shape and fixed within a narrow range of granulometric distribution ensure high operation properties of the surface-welded layer, [12, 15], (*Fig. 1*).

The standard fluidity of the material makes it possible to use dosing equipment with simplified construction and high reliability in the process of operation if steady flow of the spheroidized material is achieved, and if constant temperature of the particle is ensured in the moment of its contact with the welded surface.

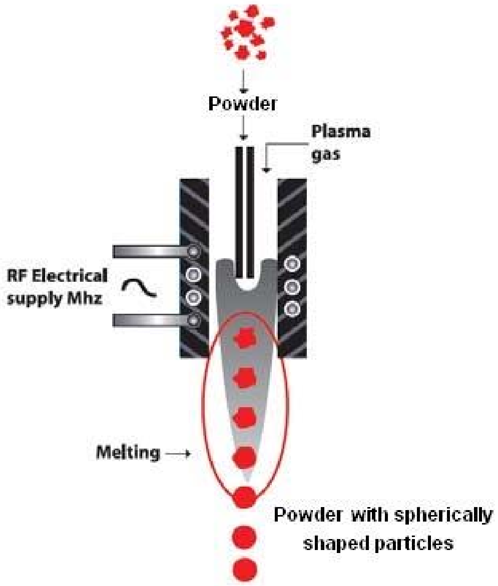


Fig.1. *Principle of surface-welding with RFI-plasmotron*

The process of plasma spheroidization is based on the intensive heating of the treated material and rounding of the liquid particles under the effect of surface tension forces, (*Fig. 2*).

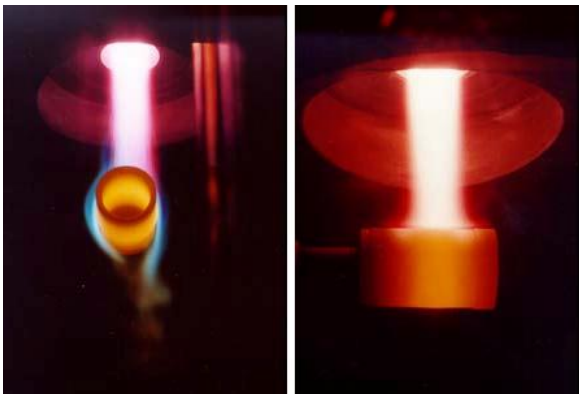


Fig.2. *Plasma surface welding of elements with refractory materials*

The single stages of the process are:

- Melting and pulverization of the processed material;
- Spheroidization of the molten particles in a hot gas flux;
- Solidification of the particles;
- Cooling and collecting the spheroidized material.

Arc or high-frequency (RF) plasmotron is used for realizing the process.

2. Treatment of powders in an arc plasmotron

The treatment of powders is carried out in ordinary or multi-chamber plasma generators (plasmotrons). This provides the possibility of producing spherical particles with sizes from 1 to 200μm.

The use of protective medium ensures the possibility preserve the chemical composition of the processed powder-like material.

When spheroidizing preliminarily prepared granules, the particles acquire not only a spherical shape, but are also formed with sufficiently uniform grain-size distribution, (*Fig.3*). At low velocities of the transporting gas, particle kinetic energy is insufficient for their deep penetration in the plasma and the main part of the material passes through the peripheral section of the stream and along the walls of the plasma reactor.

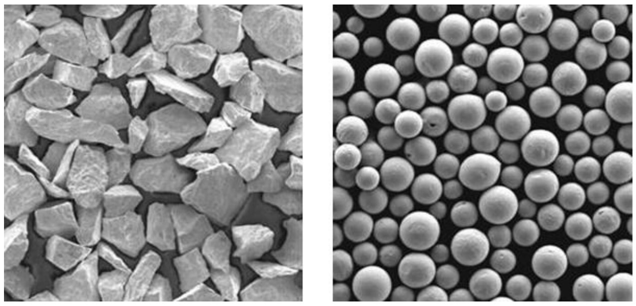


Fig.3. *Powdery corundum (Al_2O_3) prior to and after spheroidization, x120*

At very high velocities of the transporting gas the particles "break through" the jet, [3, 8, 10].

It has been established that when processing TiC the relative amount of spherical particles increases with increasing the electric current intensity (as a result of particle intensive heating and increasing jet geometric dimensions).

The processing of initial powder with grain sizes of 30μm yields spheroidization of 40% of the particles, the average dimension being reduced to 25μm. It is observed that the amount of the spheroidized particles (C, %) depends on the initial sizes of the powder (in the case of TiC pulverization), (*Table 1*), [1÷3, 14].

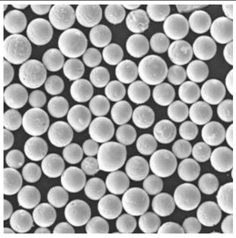
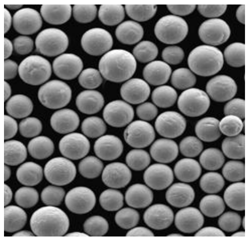
Physical properties		
Chemical formula Molecular Weight Melting point Boiling point Density Heat Capacity Thermal conductivity	Ti 47.867 g/mol 1668^{o}C 3287^{o}C 4.506 g/cm^3 523 J/(кgК) 22 W/(mK)	

Table 1. *Quantity of particles on the initial sizes of the powder*

Particle size, µm	2	2-5	5-10	10-15	15
C, %	90	90	80	50	30

When processing WC powder with an initial particle size of 5µm, the concentration of spherically shaped particles is 95%, and the average size is 3÷5µm. The studies on TiC and WC behavior during plasma spheroidization in argon show that the impact of high temperature leads to reducing the carbon content in carbides [14, 15]. The spheroidization is carried out in hermetically sealed columns, where the processing zone is isolated from atmospheric oxygen and nitrogen.

The greatest effect is achieved during treatment of carbide powder with 5% of carbon black in a plasma stream. The spherical particles at the outlet amount to 60%.

Physical properties		
Chemical formula Molecular Weight Melting point Boiling point Density	WC 195,9 g/mol ~ 2800^{o}C ~ 6000^{o}C 15.7 g/cm^3	

The conclusion is made that the lower the particle heat conductivity and the larger the particle size, the more is the spheroidization process inefficient.

By selecting the appropriate regime of powder pulverization and transportation it is possible to find conditions, under which the processed material passes through the central parts of the jet.

When high-melting-point materials are pulverized and spheroidized in plasma stream, the material often adheres to the internal walls of the die channel and coating is formed [14, 17].

In an arc plasmotron of ordinary construction the powder is introduced in the transporting gas under an angle of 90° with respect to the plasma stream direction.

At low values of the kinetic energy the powder particles move under the impact of the plasma stream along a trajectory, which prevents them from sticking to the opposite channel wall. If the particles possess sufficient kinetic energy to break through the plasma jet, they move along a trajectory that leads to the formation of incrustations on the opposite die wall.

Depending on the growth of the external layers of this deposition, it starts melting and is pulverized by the plasma stream in large drops, i.e. the process of powder pulverization is replaced by pulverization of a material in the liquid state. This phenomenon, which is, for example, undesirable when placing coatings (as the evaporation of larger particles exerts adverse impact on surface quality and other characteristics of the coating), could be used for producing coarser powder than the fine-grained one [1÷5, 7, 8, 12, 13].

The experimental results prove that granules with lower density should be applied in the processing in order to ensure conditions for their transportation in the plasma stream without noticeable evaporation. The size of the initial granules should be with 40-50% larger than that of the obtained spherical particles.

Disturbance in chemical composition is often observed during spheroidization of oxide powders in neutral (argon) plasma, and hence partial recovery is needed. In cases when such disproportioning is undesirable, the spheroidization is carried out in oxygen plasma. With this approach it is natural that preference is given to RFI-plasma heating of oxygen or oxygen-containing gas [9].

3. Powder processing in RFI-plasmotrons

The low velocity of the plasma stream and the large volume of plasma in both its transverse and longitudinal cross section allow for very effective spheroidization of the particles of different refractory materials. The specificity of Radio-Frequency-Induction (RFI) plasma discharge provides the opportunity of introducing the treated powder immediately along the plasma volume axis and using most effectively the energy of the RFI-discharge.

The current density along the RFI-discharge axis is equal to zero and therefore powder introduction in the central zone does not disturb the stability of discharge burning [4].

Temperature distribution in the plasma discharge volume is not uniform. Its maximum value reaches ≈ 9÷10000 K. There are places along the discharge axis, where temperature is reduced to 500÷1000 K. When the transporting gas is fed, the gas temperature along the RFI-discharge axis is also significantly reduced.

The productivity of the RF-plasma equipment is sufficiently high: when the power of the RFI-discharge is 6.5 kW, about 1÷2 kg/h Al_2O_3 powders with sizes of 63÷100μm are spheroidized, while the analogous productivity in the arc plasmotron is achieved by power of 100 kW.

The efficiency of spheroidization of plasma heated powder is determined by the duration of particle sojourn in the high-temperature (HT) area of the stream. In the case of RFI-plasma, characterized by low gas velocity, the treated particles move slowly with 2÷5 m/s.

The length of the plasma torch of the RFI-discharge is considerably larger than the length of the arc plasma stream of the same power.

Furthermore, plasma dynamics in RFI-discharge intensifies the heat exchange of the processed powders.

4. Conclusion

All mentioned factors indicate that the duration of particle sojourn in the plasma RFI-discharge is significantly increased, (*Table 2*). In its turn, this is the basis for the high efficiency of the RF-plasma processes for powder spheroidization, (*Table 3*).

Table 2. *Technological characteristics*

PLASMOTRON	**Stream length,** mm	**Particle velocity,** m/s	**Duration of particle sojourn in the HT-zone**, m/s
One-chamber	50	100	0.5
Multi-chamber	100	25	4
RF-induction	300	3	100

Table 3. *Thermal efficiency of heated powders*

Spheroidization method	**Thermal efficiency of heated powders**, %
Arc plasma, neutral wire	1-1.5
Arc plasma, granulated powder	1-5
Arc plasma, conductive wire	8-10
RF-plasma	25-30

RFI-plasma not only enhances the efficiency of the spheroidization process, but also provides the possibility of rounding the larger particles. For example, in arc plasma Al_2O_3 powders with spherical particle shape and sizes of up to 60÷70μm are obtained, while in RFI-plasma the size of the treated particles may be increased to 600÷800 μm. If coarser Al_2O_3 fractions or powders with the same sizes but of higher-melting-point materials are processed, it will be necessary to increase the plasma heat flux towards the particles.

In oxygen plasma it is possible to spheroidize with RFI-discharge MgO and ZrO_2 powders, which cannot be processed in argon plasma and RFI-discharge of the same power.

The analysis of powder spheroidization processes of high-melting-point materials in arc and RFI-plasma proves the undisputable advantage of the latter compared to the shortcomings of the process, realized in arc plasma, namely:

- non-uniform heating of the particles;
- powder feeding devices with complex construction;
- impossibility of ensuring flawless operation of the plasmatron;
- restrictions concerning the type of plasma forming gas;
- possibility for contamination of the processed material with elements from the arc plasmatron electrodes.

The advantages of the RFI-plasma are:

- uniform heating of the particles due to their facilitated introduction in the desired point of the plasma volume;
- possibility of heating particles with relatively large sizes due to the low velocity of the plasma-forming gas and long time of sojourn of the particles in the plasma volume;
- possibility for operation with different technological and plasma forming gases (from neutral to highly aggressive);
- possibility for high purity of the obtained product due to the non-electronic nature of generation of RFI-plasma.

The enumerated facts lead to the still broader application mainly of RFI-plasma for treatment of powders from high-melting point materials, aimed at spheroidization of their ingredient particles. The calculations and experimental results concerning RFI-plasma generation and the possibilities for its management will be the subject of another publication.

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