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# MORPHOLOGY OF POWDER PARTICLES PRODUCED BY SPRAY ATOMIZATION AND OTHER PROCESSES

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*Summary:*

*The physical and mechanical properties of coatings are heavily influenced by the technologies of powder production and by the parameters of the plasma spray process. One of the most important parameters that affects the physical and mechanical properties of coatings is the morphology of powder particles - homogeneity, granulation and granulation range, directly related to the technologies of powder production. This paper describes the technological processses of powder production most commonly used and shows the SEM micrographs of powder morphologies. Depending on the manufacturing process, powder particles have different characteristics regarding their shape, size, specific gravity, purity, etc. Since these characteristics have a significant impact on the quality and properties of deposited coatings, it is necessary to possess knowledge about the characteristics of powders in order to better control the behavior of particles in the plasma jet, in order to produce the expected characteristics of the coating.Powders have a variety of characteristics to be set for the operating parameters of the deposition in order to obtain the desired coating characteristics.*

Key words: *property, powders, particles, mechanical properties, coatings, characteristics.*

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# Introduction

The quality of deposited APS and VPS plasma spray coatings is influenced by several mutually dependable parameters, one of which is the powder quality, directly related to the powder production technology. Coatings of the optimal quality for respective purposes require the application of powders produced by certain technological processes. To ensure optimal properties of the coating, the powder with its chemical composition, morphology and distribution of the granulate particles must be adapted to the coating exploitation conditions. Powder manufacturers have different variations of manufacturing processes of powders in terms of morphology, density and chemical properties, which indicates that different types of powders are used for different exploitation conditions, as published in the works of authors (Vencl, et al., 2009, pp.398-405), (Vencl, et al., 2010, pp.591-604), (Vencl, et al., 2011, pp.1281-1288), (Mrdak, 2012, pp.182-201), (Mrdak, 2013b, pp.68-88), (Mrdak, 2014, pp.7-22), (Mrdak, 2013a, pp.7-25), (Mrdak, et al., 2013, pp.559-567). Powder production techniques greatly influence on: powder chemical composition and homogeneity, powder particle morphology, particle size and span granulation, porosity, specific density, cleanliness, etc. (Sampath, et al., 1996, pp.629-636). Powders of the same chemical composition but produced by different manufacturing processes have different powder morphologies and characteristics. Particles of a spherical shape have a higher flowability compared to powder particles of an irregular shape. Consequently, the resulting coating properties are different, even if the deposition conditions are constant. This is due to the differences in behavior during the injection of particles into the plasma jet and the flow of molten particles to the substrate where they are deposited. Irregularly shaped particles often cause blockage in the powder inlet pipe, which leads to a reduction in the deposition rate, overheating of the substrate and the coating separation from the substrate. The situation is similar in the case of the insertion of fine particles into the plasma jet. Accordingly, it is very important to possess knowledge about the powder characteristics in order to better control the behavior of particles in the plasma jet, in order to produce the expected coating characteristics. The particle morphology, the particle size and the range of granules are the key parameters that affect: degree of melting in the plasma, deposition effect, layer density, coating stress state, and adhesive and cohesive strength. Depending on the powder production technology, the morphology and density of powder particles can be quite different, so it is necessary for their application to adjust the essential deposition parameters to obtain the desired coating properties, as described in the works of authors (Vencl, et al., 2009, pp.398-405), (Vencl, et al., 2010, pp.591-604), (Vencl, et al., 2011, pp.1281-1288), (Mrdak, 2012, pp.182-201), (Mrdak, 2013b, pp.68-88), (Mrdak, 2014, pp.7-22), (Mrdak, 2013a, pp.7-25), (Mrdak, et al., 2013, pp.559-567). In most cases, one powder production technology can be used for a number of different materials. It is also important to point out that, in the powder production process, combinations of two or more techniques of powder manufacturing are used. This is particularly benefitial when developing multi-component powders the components of which have different physical properties. By applying different techniques of powder manufacturing, significantly greater homogeneity and density of particles are achieved with a minimum share of gases. It is particularly important to note that introducing more technological methods of powder making results in a more uniform distribution of granules and a constant powder surface / volume ratio, which has a positive effect on their uniform melting in plasma and on their deposition on the substrate.

The aim of this study was to describe the technological processes of powder manufacturing used for the deposition of coatings by atmospheric and vacuum plasma spray processes. The paper presents the SEM micrographs of powder morphologies influencing the powder deposition effect, coating density, stress state of the coating and adhesive/cohesive strength of the bond. The chosen powders are those commonly used for making coatings resistant to wear, abrasion, erosion, corrosion and fatigue at low and elevated temperatures.

## Powder production technologies and powder particle morphologies

Depending on powder manufacturing processes, powder particles have different characteristics relating to: microstructure, homogeneity, specific gravity, purity, morphology, particle size and particle size distribution. These characteristics have a significant impact on the quality and properties of deposited coatings in exploitation; therefore, it is necessary to possess knowledge of powder production technologies and the characteristics of produced powders in order to facilitate the control of the plasma spray parameters and the behavior of powder particles in the plasma, so as to produce high quality coatings. The most common and best-known powder production technologies are:

Agglomeration - spray drying: metals, carbides and ceramic powders;

Cladding of composite powders: metals, ceramics, metal powders carbide composite powders;

Melt atomization: metals and alloyed powders;

Electric arc melting: metal powders, ceramic powders;

Agglomeration and sintering: metal powders, ceramic powders and metal - carbide powders;

Plasma treatment (Plasma remelting) to achieve the spheroidization of metal and ceramic powders;

High Temperature Synthesis - SHS, which is obtained by the reaction of a mixture of elemental powders: powders of carbide, cermet powders, composite powders, etc.

## *Powder production by spray drying*

Spray drying is the technological process of agglomeration or consolidation of fine powder particles in the range of 1μm to 40μm. For agglomeration, ultra fine powder particles sized less than 1μm can also be used since they enable the production of powders of higher purity and density. Depending on the type of material and its purpose, there are various granulation powders for consolidation. By concentrating fine particles, spherical powder with an approximately constant surface area/volume ratio is produced. This procedure can also result in homogeneous single-component and multi-component powders. Fine powder particles mix with a liquid agent silicasol creating a suspension. In the process of plasma spray deposition of powders, silica sol evaporates and does not affect the properties of the deposited layers. Such suspension is sprayed with an appropriate nozzle under pressure into a chamber heated between 400 and 600 °C making dry powder particles which are agglomerated. The chamber temperature depends on the type and properties of the suspension. This process generally produces two fractions: coarse fraction collected at the chamber and the fine fraction collected in the cyclone. The fraction ratio is regulated by choosing the nozzle type and pressure in the process. The dried powder is then sintered in a gas furnace at a temperature of 1450 to 1600 °C , depending on he powder type, to enhance the density and provide uniform powder flowability in the deposition process. In this way, a range of powder systems of different quality and characteristics is produced, such as: WC/Co, WC/CoCr, WC/NiMoCr, Ni/SiC, $ZrO_2Y_2O_3$, $ZrO_2TiO_2Y_2O_3$, $ZrO_2CaOAl_2O_3$ and powders for special purposes. Fig. 1 shows the morphologies of the powder particles of $ZrO_220\%Y_2O_3$ and WC17%Co produced by spray drying. The particles of ceramic powders $ZrO_220\%Y_2O_3$ have a spherical morphology with a range of the granulation of 16μm to 90μm, and they are used to create the upper ceramic layer of thermo - barrier coatings $TBC_s$ (Mrdak, et al., 2013, pp.559-567). WC17%Co powder particles are more porous due to larger WC and Co particles which were used for the consolidation of spray drying. The powder is of a spherical shape with a range of grain size of 11μm to 53μm (Mrdak, 2013a, pp.7-25).

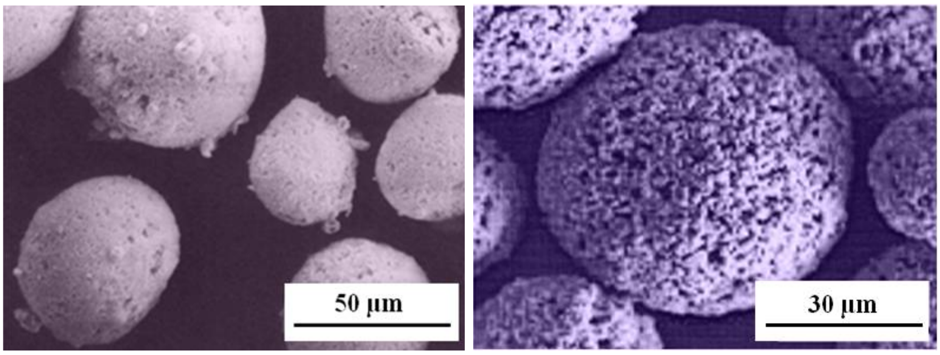


*Figure 1* – (SEM) micrography of the $ZrO_2 20\%Y_2O_3$ and WC17%Co powder particles
*Slika 1* – (SEM) mikrografija čestica praha $ZrO_2 20\%Y_2O_3$ i WC17%Co
*Рис. 1* – (SEM) микрография частиц порошка $ZrO_2 20\%Y_2O_3$ i WC17%Co

## Production of clad powders

Cladding is a technological process in which particles of a size of 40µm to 50µm are used as a powder core for cladding binary or multi-component composite materials. The powder particle cores can be produced by a process of spray drying or atomizing the liquid melt with an inert gas.The particle cores are coated with smaller particles of a size of 1 µm to 10 µm with the use of silica sol as a binder. Powders obtained by the cladding process prevent the segregation of individual components in deposited coatings, which can happen with a mechanical mixture of powders. Two major classes of powders produced by cladding are multicomponent composite metal powders of the self-adhesive Ni/Al, NiCr/Al and $NiCr/Al/Co/Y_2O_3$ types and soft sealant materials of the Ni/ graphite type. The goal of producing cladded Ni/Al, NiCr/Al and $NiCr/Al/Co/Y_2O_3$ composite powders is that the cladding enables the exothermic reaction between the components of the powder in the deposition process, to obtain a higher adhesive strength of the coating to the metal substrate (Mrdak, et al., 2013, pp.559-567), (Mrdak, 2013c, pp.7-22). The sealing material Ni/25%graphite is composed of a graphite corecladded with nickel. This prevents the combustion of graphite in plasma and enables a uniform and homogenous distribution of Ni and graphite in the coating (Mrdak, 2013b, pp.68-88). Fig. 2 shows the morphologies of the cross sections of Ni/25%graphite and Ni/20%Al powders. The cross section shows the graphite cores cladded with fine particles of nickel in the form of wrappers (Mrdak, 2013b, pp.68-88). The powder particles are of a granulate range of 30µm - 90µm and irregular in shape. Composite NI20%Al powder consists of Ni particle cores cladded with fine Al particles with a range of grain size of 53µm to 90µm. The powder particles are approximately spherical (Mrdak, 2013c, pp.7-22).

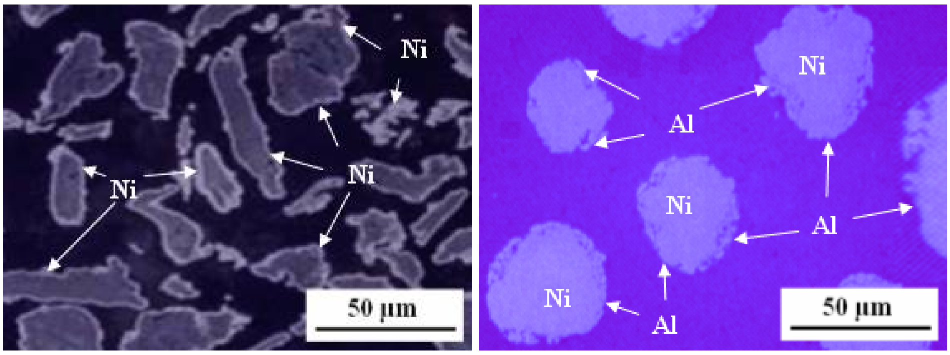


*Figure 2* – (SEM) micrography of the Ni/25% graphite and Ni/20%Al powder particles
*Slika 2* – (SEM) mikrografija čestica praha Ni/25% grafit i Ni/20%Al
*Рис.2* – (SEM) микрография частиц порошка Ni/25% графит и Ni/20%Al

## Preparation of powders by the atomization of a liquid melt

The dispersion of a liquid melt by an inert gas or atomization (Melt atomization) is a technological process mostly used for the production of powders of pure metals and their alloys such as: Al, Cu, Ni, CuAl,CuNi, CuNiIn, CuAlFe, FeCrNiC, FeCrNiMoSiC, CoCrNiWC, CoCrNiAlTaY, CoNiCrWC, CoMoCrSi, MeCrAlY, etc. The process consists of melting a metal or an alloy of a particular composition and the atomization of a liquid melt by the inert argon gas, where the products are powder particles of a spherical shape. The melting may be performed at atmospheric pressure or in vacuum. If the melting is performed in the presence of air, it is necessary to perform degassing of the liquid melt by a suitable inert gas prior to spraying. Powders made of elements sensitive to oxidation (such as MeCrAlY alloys containing Al and Y) are produced by melting in an induction furnace in vacuum, where the melt is degassed and sprayed by the inert argon gas of high purity. The produced powders are dense and of spherical morphology with insignificant amount of oxide impurities. Fig. 3 shows SEM micrographs of the morphology of the Ni22%Cr10%Al1%Y powder particles produced by the atomization of a liquid melt by an inert gas intended for deposition at atmospheric pressure and in vacuum (Mrdak, 2012, pp.182-201). The powder with a grain size range of 53μm to 106μm is used for the deposition of coatings at atmospheric pressure (Mrdak, 2012, pp.182-201).

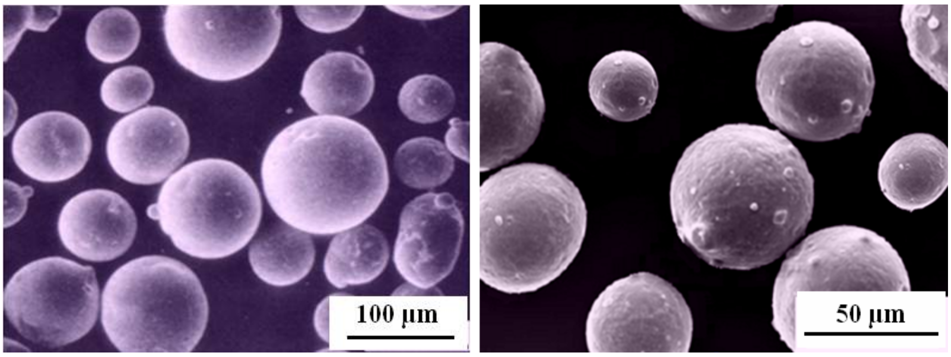


*Figure 3* – (SEM) micrography of the Ni22%Cr10%Al1%Y powder particles
*Slika 3* – (SEM) mikrografija čestica praha Ni22%Cr10%Al1%Y
*Рис. 3* – (SEM) микрография частиц порошка Ni22%Cr10%Al1%Y

The powder with a grain size range of 11μm to 37μm is used for the deposition of coatings in vacuum. The powder particles are spherical in shape and homogeneous.

## *Preparation of powders by electric arc melting*

Electric arc melting is a general method of producing powders, in which, after melting the powder components and their casting into blocks, the molten melt is rapidly cooled to room temperature. The cooled blocks are ground. After grinding, the powder is classified by a desired particle size distribution. The advantage of this method is pre-alloying and the natural homogenization of powder particles. The consequence of grinding is a typical irregular and angular shape of powder particles. This technological process is quite frequent in the production of ceramic powders: $Al_2O_3$, $TiO_2$, $ZrO_2$, $Al_2O_3TiO_2SiO_2$, $Al_2O_3TiO_2$, $Al_2O_3SiO_2$, $Cr_2O_3$, $Cr_2O_3TiO_2$, $Cr_2O_3SiO_2TiO_2$, $ZrO_2CeO_2Y_2O_3$, $ZrO_2Y_2O_3$, $ZrO_2MgO$, $CeO_2Y_2O_3$ and other ceramicsas well as in the production of metal and alloy powders. Fig. 4 shows the morphology of the $Al_2O_3$40%$TiO_2$ and $ZrO_2$24%MgO powder particles. Fast sub-cooling of the molten melt allows the formation of a suitable structure in polymorphous ceramic materials. In the structure of the $Al_2O_3$40%$TiO_2$ ceramic powder, the α $Al_2O_3$ hard phase is present in a greater proportion while there is an insignificant proportion of the γ - $Al_2O_3$ softer phase, which is very suitable for the production of coatings resistant to wear. The $Al_2O_3$40%$TiO_2$ powder is in a range of particle granulation of 15μm 45μm and it is used to protect surfaces from friction, abrasion and erosion of particles (Mrdak, 2014, pp.7-22).

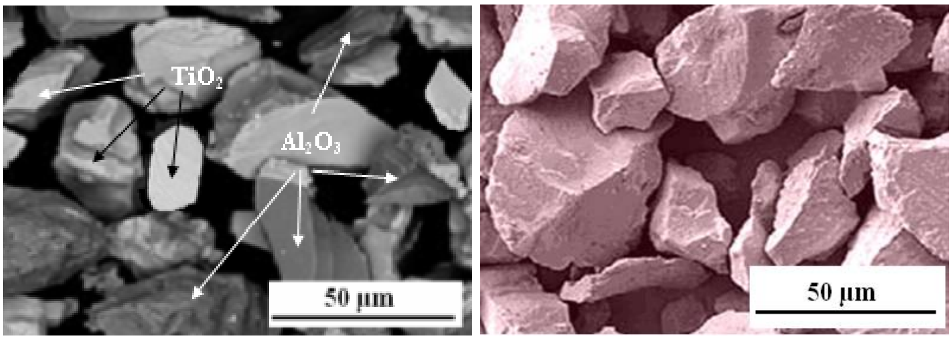


*Figure 4 – (*SEM) micrography of the $Al_2O_3$40%$TiO_2$ and $ZrO_2$ 24%MgO powder particles
*Slika 4 – (*SEM) mikrografija čestica praha $Al_2O_3$40%$TiO_2$ i $ZrO_2$ 24%MgO
*Рис. 4* – (SEM) микрография частиц порошка $Al_2O_3$40%$TiO_2$ i $ZrO_2$ 24%MgO

Quick subcooling of the $ZrO_2$24%MgO ceramic melt also provides a small fraction of the monoclinic phase (below 5%) which is not desirable in the deposited layers. The $ZrO_2$24%MgO powder has a range of the granulate of 10µm - 53µm and is applied for the preparation of the upper ceramic layer of thermal - barrier coatings (TBCs) (Kakaš, et al., 2005, pp.335-340).

## *Preparation of powder agglomeration - sintering*

Agglomeration - sintering is a technique in which agglomerated powder particles are condensed by a suitable heat treatment (gas or plasma) below the melting point of the components. This technology is used for the production of metal, metal - carbide and ceramic powders. Agglomeration and sintering do not produce completely alloyed materials, which is why this technique is used only for powders for the given purpose. Fig. 5 shows the morphologies of the agglomerated and sintered powder particles of $Al_2O_3$13%$TiO_2$ and Mo. The $Al_2O_3$13%$TiO_2$ powder particles consist of fine agglomerated and sintered particles of $Al_2O_3$ and $TiO_2$. Because of the fine granulation of the initial components of $Al_2O_3$ and $TiO_2$ powders, theagglomerated and sintered particles are quite dense, homogeneous and spherical.

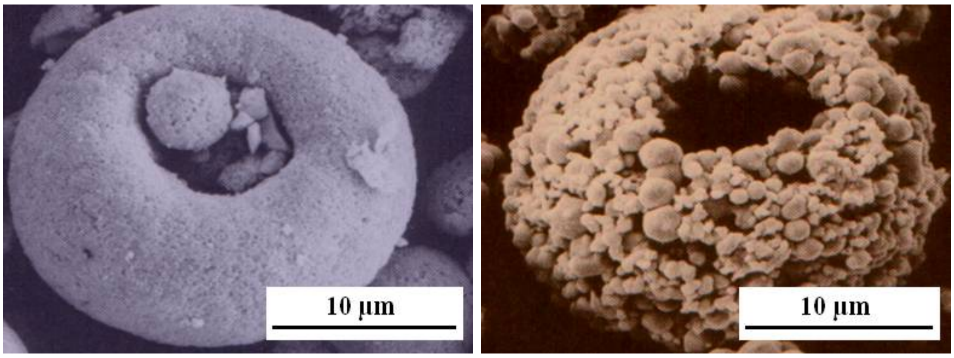


*Figure 5* – (SEM) micrography of the $Al_2O_3 13\%TiO_2$ and Mo powder particles
*Slika 5* – (SEM) mikrografija čestice praha $Al_2O_3 13\%TiO_2$ i Mo
*Рис. 5* – (SEM) микрография частиц порошка $Al_2O_3 13\%TiO_2$ i Mo

The powder is produced with the distribution of granules from 15µm to 45µm. The Mo agglomerated powder particle is more porous compared to the $Al_2O_3 13\%TiO_2$ powder particle because it consists of bigger particles of sintered and agglomerated Mo. The powder is produced with a granulation of the distribution of 16µm to 45 µm and is used for producing coatings with increased resistance to wear (Mrdak, et al., 2005, pp.235-239). The powder particles have quite a regular shape, taking into account that sintered components have quite different melting temperature and other physical properties.

## *Preparation of powders by the plasma treatment*

The process of plasma arc remelting has been developed as a new method to improve the existing quality of powders and to provide the best possible reproducibility of coatings with optimum properties. This primarily refers to the shape of particles, homogeneity, density, size, and the oxygen content as compared to the starting powder prior to the treatment with plasma. Remelting of plasma powders can result in powders with completely different specific properties. A constant powder flow is not the only parameter that affects the reproducibility of the coating quality. Since the melting process must be reproduced, it is also necessary to control the powder reactivity. All powder particles should have a constant ratio of surface/volume without gas content. The grain size uniformity can be achieved by careful screening and classification of powders by granulation. Most commercial powders are produced with different surface area/volume ratios, causing non-uniform melting of powder particles. This has led to the development of a new technique of powder production, which also applies plasma energy in the production. This technique can produce dense and spherical particles with a constant ratio of surface area/volume. Powders of different grain sizes can be easily sieved and classified in accordance with the desired granulation. This is not the case in conventional methods of powder production that produce powders with different morphologies and variable surface/volume ratios. The production of spherical particles by the powder remelting process in the plasma has great advantages. The spherical shape and a constant surface/volume ratio also provide a constant flow of powder in the plasma deposition process. The gas content in the powder particles is significantly reduced and in the deposition process there is no evaporation of particles as in the case of angular grains. The process of plasma arc remelting is carried out in a furnace with controlled temperature and atmosphere. The chamber must not contain oxygen. The most commonly used gas for protection is inert gas argon of high purity. The furnace is protected from noise with a material on the furnace outer part. A required quantity of powder to be treated is uniformly injected into the plasma jet of defined characteristics. Powder particles injected into the plasma zone are remelted and, at the exit of the jet, they are undercooled quickly in some of cryogenic liquids. The treated powder particles of different granulation pass through cyclones to be collected in the powder chamber. After the treatment, the powder is classified in accordance with grain sizes. If the desired effect has not been obtained during the initial treatment, ie. If all the particles are not spherical, the process is repeated. The starting materials are powders of different chemical compositions and morphologies of different particle types produced by different technological processes. Multi-component powders cannot be produced by other technological methods. In the process, a number of particles of different powders of chemically determined granulation are mixed and then micro pelleted into a new particle. Thus formed particles are used as a starting material for the plasma treatment and for the preparation of multicomponent particles with novel properties (Hansz and Tourenne, 1988, pp.35-40). Typical examples of multi-component powders are WC-TiC-NbC-TaC and TiC-TiN-$MoSi_2$-$Cr_3C_2$ powder. The produced powders are dense and homogeneous without a share of impurities and gases. Because of a high density and purity that can be achieved in powder particles, this procedure is widely used for thickening powder particles of metals susceptible to oxidation. Fig. 6.1 shows the morphology of a multicomponent sintered particle of WC-TiC-NbC-TaC before the plasma treatment and the morphology of multicomponent powder particles after the plasma treatment (Lugscheider, 1988, pp.23-48).

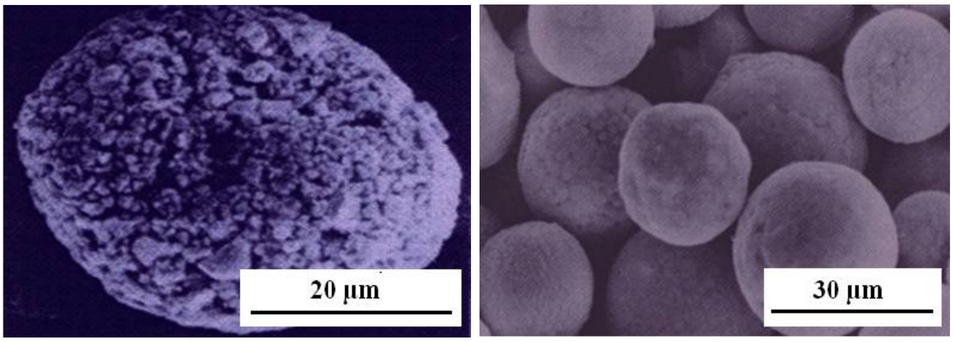


*Figure 6.1* – (SEM) morphology of the WC-TiC-NbC-TaC powder particles before and after the treatment with plasma
*Slika 6.1* – (SEM) morfologija čestica praha WC-TiC-NbC-TaC pre i posle tretmana sa plazmom
*Рис. 6.1* − (SEM) морфология частиц порошка WC-TiC-NbC-TaC до и после плазменной обработки

The process of powder plasma remelting is suitable for the production of powders of zirconium oxide stabilized with other types of oxides. High remelting temperature helps to improve prealloying zirconium oxide, which significantly improves the oxide stabilization. This technological process results in systems of ceramic powders such as $ZrO_2CeO_2Y_2O_3$, $ZrO_2Y_2O_3$, $ZrO_2MgO$, $CeO_2Y_2O_3$ and other ceramic powders. Fig. 6.2 shows the particle morphology of the $ZrO_2$25%$CeO_2$3%$Y_2O_3$ (Mrdak, et al., 2013, pp.559-567) and $ZrO_2$8%$Y_2O_3$ ceramic powders treated by plasma in order to spheroidize powder particles with a constant surface area/volume ratio. The spherical powder of $ZrO_2$25%$CeO_2$3%$Y_2O_3$ is fully prealloyed with particles ranging from 11 µm to 90 µm. The figure also shows the morphology of the powder particles of $ZrO_2$8%$Y_2O_3$ with a grain size of 15 µm to 45 µm. The powder particles have a regular spherical shape.

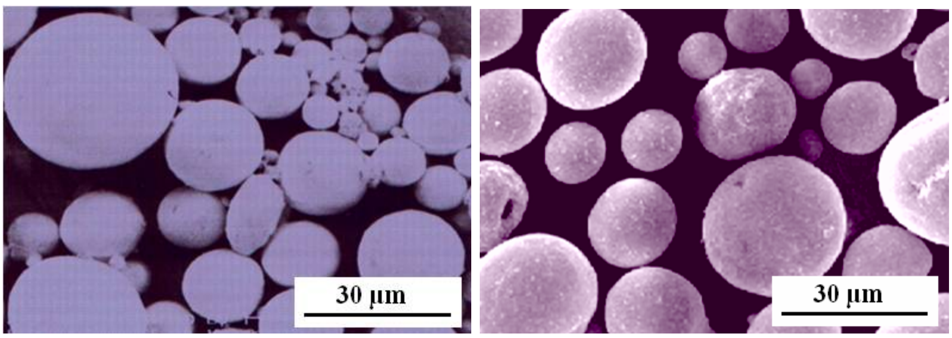


*Figure 6.2* – (SEM) morphology of the $ZrO_2$25%$CeO_2$3%$Y_2O_3$ and $ZrO_2$8%$Y_2O_3$ powder particles
*Slika 6.2* – (SEM) morfologija čestica praha $ZrO_2$25%$CeO_2$3%$Y_2O_3$ i $ZrO_2$8%$Y_2O_3$
*Рис. 6.2* − (SEM) морфология частиц порошка $ZrO_2$25%$CeO_2$3%$Y_2O_3$ и $ZrO_2$8%$Y_2O_3$

## Mechanical alloying

The technological process of making powders by mechanical alloying - MA aims at producing homogeneous powder particles. It produces very homogeneous coatings with a fine microstructure. Mechanical alloying (MA) is described as a high - energy grinding process in which powder particles are subjected to repeated cold-welding, breaking and re-welding. The transfer of mechanical energy to powder particles results in the introduction of stresses in powder particles through the creation of dislocations and other defects that act as fast diffusion paths. In addition, grains are filtered and grain size is processed, which decreases the diffusion distance. During grinding, there is a slight increase in powder temperature. All these effects lead to the alloying of the powder mixture components, during the grinding process (Suryanarayana, 2004, p.466). Since mechanical alloying can lead to the formation of intermetallic phases, which are often difficult to form even at high temperatures, plasma spray powders prepared by high energy grinding are a great alternative to the formation of deposits of this type of composite phases. The versatility of mechanical alloying enables the production of composite powders of hydroxyapatite - HA reinforced with zirconium oxide - $ZrO_2$ stabilized with yttrium oxide $Y_2O_3$, Ni/Al/Mo, Cu/Al, Cu/$Al_2O_3$, Tl/Al/$Si_3N_4$, etc. (Fukumoto and Okane, 1992 pp.595-600). In the case of systems with an explosive material such as aluminum powder with a very fine particle size of < 3 μm, short-term mechanical alloying reduces the reactivity of the powder due to the inclusion of hard phase particles ($Al_2O_3$ and / or SiC) within Al particles or fine $Al_2O_3$ and SiC particles sticking on the surface of aluminum (Bach, et al., 2000, pp.299-302). Fig. 7 shows a (SEM) morphology of the Ni/5.5%Al/5%Mo and Cu/10%Al powder particles produced by mechanical alloying. The Ni/5.5%Al/5%Mo powder particles have a spherical shape which allows an even flow of the powder in the plasma jet. The range of granulation of the powder particles is in the range of 45 μm to 90 μm.

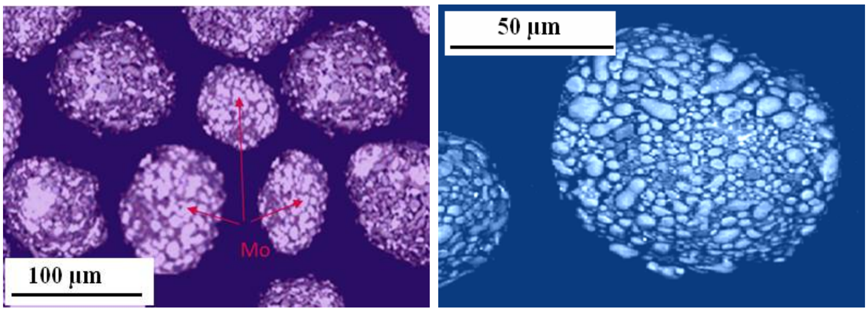


*Figure* 7 – (SEM) morphology of the Ni/5,5%Al/5%Mo and Cu/10%Al powder particles
*Slika 7* – (SEM) morfologija čestica praha Ni/5,5%Al/5%Mo i Cu/10%Al
*Рис. 7* − (SEM) морфология частиц порошка Ni/5,5%Al/5%Mo и Cu/10%Al

The powder particles of Cu/10%Al are also of a spherical shape in the granulate range from 45 μm to 106 μm.

### *Sintering, agglomeration by high-temperature synthesis*

For the production of carbide and cermet powders, different technological processes are used, eg: high-temperature synthesis - SHS, agglomeration and sintering, agglomeration and densification of powder by plasma and mechanical mixing of carbide and bonding alloys - MA. Fig. 8 shows the SEM - photomicrographs of the morphology of the TiC (Garrett, et al., 2012) and $TiB_2$ powder particles (Logan and Villalobos, 2006, pp.249-257) with the grain size of 1 µm to 5 µm, produced by the high temperature synthesis process - SHS. Carbide and cermet powders are often produced by the high temperature synthesis - (SHS), through the reaction of a mixture of elemental powders of Ti and C or B, which comprises a binder phase of Ni,Fe(Cr) and a hard cermet phase of TiC or $TiB_2$. This process uses an exothermic reaction of a certain reactive mixture of flammable Ti and C or Ti and B powders. Because of high temperatures that can be reached during the reaction (~1200 - 6000 ºC), impurities can evaporate and improve the bond strength between the hard carbide phase and the alloy used as a binder (Smith, et al., 1995, pp.1121-1126).

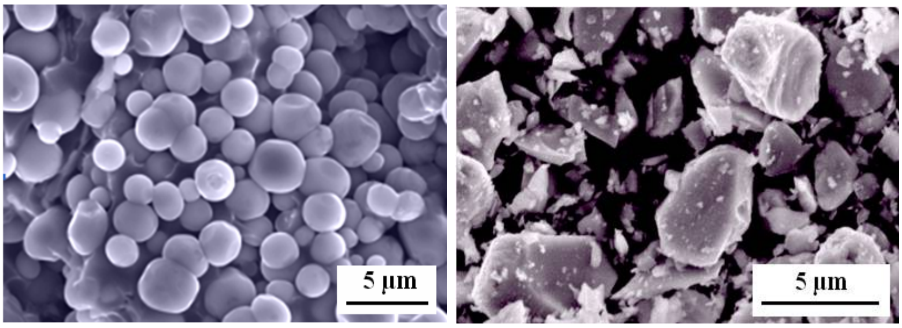


*Figure 8* – (SEM) morphology of the TiC and $TiB_2$ powder particles (Garrett, et al., 2012), (Logan and Villalobos, 2006, pp.249-257)
*Slika 8* – (SEM) morfologija čestica praha TiC i $TiB_2$ (Garrett, et al., 2012), (Logan and Villalobos, 2006, pp.249-257)
*Рис. 8* − (SEM) морфология частиц порошка TiC и $TiB_2$ (Garrett, et al., 2012), (Logan and Villalobos, 2006, pp.249-257)

This technological process allows the production of powders with a particle size of 1 µm to 5 µm in diameter and smaller than 100nm.

## Conclusion

This paper describes the technological procedures commonly used for the production of powders intended for thermal spray processes, as well as the morphologies of the powder particles tested by scanning electron microscopy - SEM.

The technological procedures commonly used for the production of powders are: agglomeration spray drying, powder cladding, spraying the liquid melt, electric arc melting, agglomeration and sintering, powder spheroidization by plasma remelting and high-temperature synthesis.

This paper has shown that various technological processes can produce powders with different morphologies of powder particles whose characteristics are directly related to the structure and physical - mechanical properties of deposited coatings.

Each technological procedure gives some specific characteristics to the produced powders, on the basis of which the powders are selected for protecting functional surfaces in the production of new machine parts, for repairing surfaces worn by mechanisms of wear, cavitation and corrosion at low and high temperatures as well as for special purposes.

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## ТЕХНОЛОГИЯ ИЗГОТОВЛЕНИЯ ПОРОШКОВЫХ ИЗДЕЛИЙ И МОРФОЛОГИЯ ЧАСТИЦ ПОРОШКА

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ОБЛАСТЬ: химические технологии
ВИД СТАТЬИ: обзорная статья
ЯЗЫК СТАТЬИ: английский

*Резюме:*

*На физические характеристики и механические свойства покрытий большое влияние оказывают, применяемая технология изготовления порошка и параметры, используемые в процессах плазменной обработки.*

*Ключевыми параметрами, влияющими на физические и механические свойства покрытий, являются такие производственно-технологические параметры порошковых изделий, как: морфология частиц порошка, однородность, грануляция порошка и диапазон грануляции, являющихся неотъемлимой частью технологического процесса при изготовлении порошковых изделий.*

*В данной статье освещены технологические процессы производства порошковых изделий, приведена морфология частиц порошка (ведущих произодителей порошковых изделий), изученная методом электронной сканирующей микроскопии – SEM.*

*Во многих публикациях объясняется корреляционная зависимость между морфологией частиц и технологическими процессами, применяемыми в производстве порошковых изделий, а также параметрами нанесения покрытия. Различные характеристики частиц, такие, как форма, величина, размер, густота, чистота и пр. непосредственно зависят от производственного процесса при изготовлении порошковых изделий.*

*Так как от перечисленных характеристик зависят качество и свойства нанесенного покрытия, для лучшего конроля частиц в плазменной струе и обеспечения желаемых результатов покрытия, необходимо владеть знаниями о характеристиках и свойствах порошка.*

*При нанесении покрытия необходимо учитывать, различия характеристик порошковых изделий.*

Ключевые слова: *технологии, порошки, морфология, микроструктура.*

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TEHNOLOGIJE IZRADE PRAHOVA I MORFOLOGIJE ČESTICA PRAHOVA

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OBLAST: hemijske tehnologije
VRSTA ČLANKA: pregledni članak
JEZIK ČLANKA: engleski

*Sažetak:*

*Fizičke osobine i mehanička svojstva prevlaka su pod velikim uticajem tehnologije izrade prahova i parametara plazma sprej procesa. Jedan od veoma bitnih parametara koji utiče na fizička i mehanička svojstva prevlaka su morfologije čestica praha, homogenost, granulacija praha i raspon granulacije, koji su u direktnoj vezi sa tehnologijama izrade prahova. U ovoj studiji opisani su tehnološki procesi proizvodnje prahova koji se najviše koriste i prikazane su morfologija čestica prahova renomi-*

*ranih proizvođača praha, koje su ispitane skening elektronskom mikroskopijom – SEM. Postoji mnogo studija koje su objavile korelaciju između morfologije i tehnologije izrade prahova sa parametrima depozicije prevlaka. U zavisnosti od procesa proizvodnje praha, čestice poseduju različite karakteristike koje se tiču oblika, veličine, specifične gustine, čistoće, itd. Pošto ove karakteristike imaju značajan uticaj na kvalitet i svojstva deponovanih prevlaka, potrebno je posedovati znanje o karakteristikama praha radi bolje kontrole ponašanja čestica u mlaz plazme, kako bi se proizvele prevlake očekivanih karakteristika. Prahovi imaju raznovrsne karakteristike za koje treba podesiti operativne parametre depozicije kako bi se dobile prevlake željenih osobina.*

## Uvod

*Kvaliteti deponovanih APS i VPS plazma sprej prevlaka su pod uticajem više međusobno uslovljenih parametara, a jedan od njih je kvalitet praha koji je u direktnoj vezi sa tehnologijom proizvodnje prahova. Da bi se obezbedila optimalna svojstava prevlake, prah sa svojim hemijskim sastavom, morfologijom i raspodelom granulata čestica mora biti prilagođen eksploatacionim uslovima prevlake. Od tehnike proizvodnje prahova umnogome zavisi: hemijski sastav i homogenost praha, morfologija čestica praha, veličina čestice i raspon granulacije, poroznost, specifična gustina, čistoća i dr. (Sampath, et al., 1996, pp.629-636). Za proizvedene prahove istog hemijskog sastava sa različitim procesima izrade dobijaju se različite morfologije i karakteristike prahova (Kubel, 2000, pp.12-32). Čestice sfernog oblika imaju bolju protočnost u odnosu na čestice praha nepravilnog oblika. Shodno tome, dobijena svojstva prevlaka se razlikuju, čak i ako su uslovi za depoziciju konstantni. To je zbog razlike u ponašanju tokom ubrizgavanja čestica u mlaz plazme i toka istopljenih čestica do podloge gde se deponuju. Čestice nepravilnog oblika često izazivaju blokadu u dovodnoj cevi za prah, što dovodi do smanjenja stope depozicije, pregrevanja substrata i odvajanja prevlake sa podloge. Slična je situacija i sa ubacivanjem finih čestica u mlaz plazme. Shodno tome, veoma je bitno posedovati znanje o karakteristikama praha radi bolje kontrole ponašanja čestica u mlaz plazme, kako bi se proizvele prevlake očekivanih karakteristika. Zavisno od tehnike izrade praha, morfologije i gustine čestica praha mogu biti sasvim različite, pa je za njihovu primenu potrebno podesiti vitalne parametre depozicije da bi se dobile prevlake željenih osobina, kako je opisano u radovima autora (Vencl, et al., 2009, pp.398-405), (Vencl, et al., 2010, pp.591-604), (Vencl, et al., 2011, pp.1281-1288), (Mrdak, 2012, pp.182-201), (Mrdak, 2013b, pp.68-88), (Mrdak, 2014, pp.7-22), (Mrdak, 2013a, pp.7-25), (Mrdak, et al., 2013, pp.559-567). Važno je naglasiti da se sa primenom više tehnoloških postupaka izrade prahova postiže ujednačenija raspodela granulata i konstantan odnos površina –zapremina čestica, što povoljno utiče na njihovo ravnomerno topljenje u plazmi i deponovanje na podlozi.*

*Cilj rada jeste da se opišu tehnološki postupci izrade prahova, koji se koriste za depoziciju prevlaka atmosferskim i vakuum plazma sprej procesima. U radu su prikazane SEM mikrofotografije morfologija prahova, od kojih zavisi efekat depozicije praha, gustina prevlake, naponsko stanje prevlake i adheziono-koheziona čvrstoća spoja. Odabrani su prahovi, koji se najčešće koriste za izradu prevlaka otpornih na habanje, abraziju, eroziju, koroziju i na zamor na niskim i povišenim temperaturama.*

## Tehnike proizvodnje prahova i morfologije čestica prahova

*U zavisnosti od procesa proizvodnje praha, čestice poseduju različite karakteristike koje se odnose na: mikrostrukturu, homogenost, specifičnu gustinu, čistoću, morfologiju, veličinu i raspodelu veličine čestica. Ove karakteristike imaju značajan uticaj na kvalitet i svojstva deponovanih prevlaka u eksploataciji, zbog čega je neophodno posedovati znanje o tehnologijama izrade prahova i karakteristikama proizvedenih prahova radi lakše kontrole plazma sprej parametara i ponašanja čestica praha u plazmi, kako bi se proizvele prevlake visokog kvaliteta. Najčešće i najpoznatije tehnologije proizvodnje prahova su:*

*– aglomeracija suvim raspršivanjem (Agglomeration – spray draying): metalni, karbidni i keramički prahovi;*

*– oblaganje prahova (Clad composite powders): metalni, keramički prahovi metalokarbidni, kompozitni prahovi;*

*– raspršivanje tečnog rastopa (Melt atomization): metalni i legirani prahovi;*

*– topljenje električnim lukom (Electric arc melting): metalni prahovi, keramički prahovi;*

*– aglomeracija i sinterovanje (Agglomerating and sintering): metalni prahovi, keramički prahovi i metalokarbidni prahovi;*

*– plazma tretman (Plasma fusion) radi sferoidizacije metalnih i keramičkih prahova;*

*– proces visokotemperaturne sinteze (High Temperature Synthesis – SHS) koja nastaje reakcijom smeše elementarnih prahova: karbidni prahovi, kermet prahovi, kompozitni prahovi i dr.*

## Izrada prahova suvim raspršivanjem

*Suvo raspršivanje (Spray drying) jeste tehnološki proces aglomeracije ili okrupnjavanja finih čestica praha u opsegu od 1μm do 40 μm. Za aglomeraciju se mogu koristi i ultrafine čestice praha, veličine manje od 1 μm, koje omogućavaju izradu prahova veće čistoće i gustine. Fine čestice praha vezuju se sa tečnim sredstvom silikasolom u jednu celinu, praveći pri tome suspenziju. Takva suspenzija raspršuje se sa odgovarajućom mlaznicom pod određenim pritiskom u komoru zagrejanu od 400 do 600°C, praveći suve čestice praha koje su okrupnjene – aglomerisane. Temperatura u komori zavisi od vrste i svojstava suspenzije. Ovaj proces proizvodi generalno dve frakcije: grubu frakciju skupljenu u komori i finu frakciju skupljenu u ciklonu. Odnos frakcija reguliše se izborom tipa mla-*

*znice i radnim pritiskom u procesu. Osušeni prah se zatim sinteruje u gasnoj peći na temperaturi od 1450 do 1600$^{o}$C u zavisnosti od vrste praha, kako bi se poboljšala gustina i obezbedila ujednačena protočnost praha u procesu depozicije. Na ovaj način proizvodi se čitav niz sistema prahova različitog kvaliteta i svojstava kao što su: WC/Co, WC/CoCr, WC/NiMoCr, Ni/SiC, $ZrO_2Y_2O_3$, $ZrO_2TiO_2Y_2O_3$, $ZrO_2CaOAl_2O_3$ i prahovi za specijalne namene. Proizvedeni prah je sfernog oblika.*

Izrada prahova oblaganjem

*Oblaganje prahova (Clad composits) jeste tehnološki proces u kojem se za oblaganje dvokomponentnih ili višekomponentnih kompozitnih materijala, kao jezgra praha, koriste čestice veličine od 40 µm do 50 µm. Jezgra čestica praha mogu se proizvesti postupkom suvog raspršivanja ili atomizacijom tečnog rastopa inertnim gasom. Jezgra čestica oblažu se manjim česticama veličine od 1 µm do 10 µm uz upotrebu silikasola kao vezivnog sredstva. Dve najvažnije klase prahova izrađene oblaganjem su višekomponentni samovezujući kompozitni metalni prahovi tipa Ni/Al, NiCr/Al i NiCr/Al/Co/$Y_2O_3$ i meki zaptivni materijal tipa Ni/grafit. Cilj izrade obloženih kompozitnih prahova Ni/Al, NiCr/Al i NiCr/Al/Co/$Y_2O_3$ jeste što oblaganje omogućuje egzotermne reakcije između komponenti praha u procesu deponovanja, kako bi se dobila što veća adheziona čvrstoća prevlake sa metalnom podlogom (Mrdak, et al., 2013, pp.559-567), (Mrdak, 2013c, pp.7-22). Zaptivni materijal Ni/25% grafit sastoji se od jezgra grafita obloženog niklom. Na taj način sprečava se sagorevanje grafita u plazmi i omogućuje ravnomerna i homogena raspodela Ni i grafita u prevlaci (Mrdak, 2013b, pp.68-88).*

Izrada prahova atomizacijom tečnog rastopa

*Raspršivanje tečnog rastopa inertnim gasom ili atomizacija (Melt atomization) tehnološki je proces koji se najviše koristi za proizvodnju prahova čistih metala i njihovih legura kao što su: Al, Cu, Ni, CuAl,CuNi, CuNiIn, CuAlFe, FeCrNiC, FeCrNiMoSiC, CoCrNiWC, CoCrNiAlTaY, CoNiCrWC, CoMoCrSi, MeCrAlY i dr. Proces se sastoji od topljenja metala ili legure određenog sastava i atomizacije tečnog rastopa inertnim gasom argonom, pri čemu se proizvode čestice praha sfernog oblika. Topljenje se može izvesti na atmosferskom pritisku ili u vakuumu. Ukoliko se topljenje izvodi uz prisustvo vazduha, obavezno se pre raspršivanja vrši degazacija tečnog rastopa pogodnim inertnim gasom. Prahovi izrađeni od osetljivih elemenata na oksidaciju, kao što su legure MeCrAlY koje sadrže Al i Y, proizvode se topljenjem u indukcionoj peći u vakuumu, u kojoj se tečni rastop degazira i raspršuje inertnim gasom argonom visoke čistoće. Proizvedeni prahovi su gusti i sferne morfologije sa neznatnim udelom oksidnih nečistoća.*

## Izrada prahova topljenjem električnim lukom

*Topljenje električnim lukom (Electric arc melting) opšta je metoda proizvodnje prahova, kod koje se, nakon topljenja komponenti praha i livenja u blokove, istopljeni rastop brzo hladi do sobne temperature. Ohlađeni blokovi se melju, nakon čega se vrši klasifikacija praha po željenoj raspodeli veličine čestica. Prednost ove metode je predlegiranost i prirodna homogenizacija čestica praha. Posledica mlevenja je tipičan nepravilan i uglast oblik čestica praha. Ovaj tehnološki postupak često je zastupljen u izradi kako keramičkih prahova:* $Al_2O_3$, $TiO_2$, $ZrO_2$, $Al_2O_3TiO_2SiO_2$, $Al_2O_3TiO_2$, $Al_2O_3SiO_2$, $Cr_2O_3$, $Cr_2O_3TiO_2$, $Cr_2O_3SiO_2TiO_2$, $ZrO_2CeO_2Y_2O_3$, $ZrO_2Y_2O_3$, $ZrO_2MgO$, $CeO_2Y_2O_3$ *i drugih keramika, tako i metalnih i legiranih prahova.*

## Izrada prahova aglomeracija – sinterovanje

*Aglomeracija – sinterovanje praha (Agglomerating – Sintering) jeste tehnika kojom se aglomerisane čestice praha zgušnjavaju pogodnim toplotnim tretmanom (gasom ili plazmom) ispod temperature topljenja komponenti. Ova tehnologija primenjuje se za proizvodnju metalnih, metalo-karbidnih i keramičkih prahova. Aglomeracijom i sinterovanjem ne proizvode se sasvim legirani materijali, zbog čega se ovom tehnikom proizvode samo prahovi za datu namenu. Čestice praha imaju dosta pravilan oblik, s obzirom na to da se sinteruju komponente koje imaju različite temperature topljenje i druga fizička svojstva.*

## Izrada prahova plazma tretmanom

*Postupak pretapanja praha mlazom plazme (Plasma fusion) razvio se kao novi postupak iz zahteva da se unaprede postojeći kvaliteti prahova i obezbedi što bolja reproduktivnost prevlaka sa optimalnim svojstvima. To se, pre svega, odnosi na oblik čestica, homogenost, gustinu, veličinu i sadržaj kiseonika u poređenju sa polaznim prahom pre tretmana sa plazmom. Ovom tehnikom proizvode se guste i sferne čestice sa konstantnim odnosom površina– zapremina. Prahovi sa različitom raspodelom veličine zrna mogu se lako prosejati i klasifikovati po željenoj granulaciji. To se ne odnosi na konvencionalne postupke izrade prahova koji proizvode prahove različitih morfologija i sa promenljivim odnosom površina –zapremina. Proizvodnja sfernih čestica praha postupkom pretapanja u plazmi ima velike prednosti. Sferičan oblik i konstantan odnos površina–zapremina omogućava, takođe, konstantan protok praha u procesu plazma depozicije. Sadržaj gasa u česticama praha znatno je smanjen i u procesu depozicije ne dolazi do isparavanja čestica kao što je slučaj za uglasta zrna. Proces pretapanja praha plazmom radi se u kabini sa kontrolisanom temperaturom i atmosferom. U mlaz plazme definisanih karakteristika ravnomerno se ubacuje određena količina praha koji se tretira. Ubrizgane čestice praha u zoni plazme pretapaju se i na izlasku iz mlaza brzo pothlađuju – gase u nekoj od kriogenih tečnosti. Tretirane čestice praha različitih granulacija prolaze kroz ciklone i* *skupljaju u komoru za prah. Nakon izvršenog tretmana prah se klasifikuje po granulacijama. Ukoliko prvim tretmanom nije postignut željeni efekat, tj. sve čestice nemaju pravilan sferičan oblok, proces se ponavlja.*

## Mehaničko legiranje

*Tehnološki postupak izrade prahova mehaničkim legiranjem (Mechanical alloying - MA) ima za cilj izradu homogenih čestica praha, koji proizvodi prevlake veoma homogene i fine mikrostrukture. Mehaničko legiranje (MA) opisano je kao visokoenergetski proces mlevenja u kojem su čestice praha podvrgnute višestrukom hladnom zavarivanju, lomljenju i ponovnom zavarivanju. Prenos mehaničke energije na čestice praha rezultira uvođenjem naprezanja u čestice praha kroz stvaranje dislokacija i drugih grešaka koje deluju kao brze difuzione staze. Pored toga, javlja se prečišćavanje čestica i prerada veličine zrna, a samim tim se smanjuje i razdaljina difuzije. Tokom mlevenja javlja se blagi porast temperature praha. Svi ovi efekti dovode do legiranja komponenti mešavine praha tokom procesa mlevenja (Suryanarayana, 2004, p.466). Pošto mehaničko legiranje može dovesti do stvaranja intermetalnih faza, koja se često teško formiraju čak i na visokim temperaturama, plazma sprej prahovi pripremljeni visokom energijom mlevenja odlična je alternativa za formiranje depozita ove vrste kompozitnih faza.*

## Sinterovanje, aglomeracija visokotemperaturnom sintezom

*Za proizvodnju karbidnih i kermet prahova koriste se različiti tehnološki procesi kao što su: visokotemperaturna sinteza (High Temperature Synthesis – SHS), aglomeracija i sinterovanje, aglomeracija i zgušnjavanje praha plazmom (Plasma fusion) i mehaničko mešanje karbida i vezne legure (Mechanical alloying – MA). Karbidni i kermet prahovi često se proizvode visoko- temperaturnom sintezom – High Temperature Synthesis (SHS) reakcijom smeše elementarnih prahova Ti i C ili B, koja sadrži vezivnu fazu Ni,Fe(Cr) i tvrdu kermet fazu TiC ili* $TiB_2$*. Ovaj proces koristi egzotermnu reakciju određene reaktivne smeše zapaljivih prahova Ti i C ili Ti i B. Zbog visokih temperatura koje se postižu tokom reakcije (~1200 – 6000ºC), nečistoće mogu da ispare i poboljšaju čvrstoću veze između tvrdih karbidnih faza i legure koja se koristi kao vezivno sredstvo (Smith, et al., 1995, pp.1121-1126). Ovaj tehnološki postupak omogućuje izradu prahova veličine čestica prečnika od 1 μm do 5μm i manjeg od 100nm.*

## Zaključak

*U radu su opisani tehnološki postupci koji se najčešće koriste za proizvodnju prahova namenjenih za termosprej procese, kao i morfologije čestica prahova koje su ispitane skening elektronskom mokroskopijom – SEM.*

*Tehnološki postupci koji se najčešće koriste za proizvodnju prahova su: aglomeracija suvim raspršivanjem, oblaganje prahova, raspršivanje tečnog rastopa, topljenje električnim lukom, aglomeracija i sinterovanje, sferoidizacija praha pretapanjem plazmom i proces visokotemperaturne sinteze.*

*Ovaj dokument je pokazao da se sa različitim tehnološkim postupcima mogu proizvesti prahovi sa različitim morfologijama čestica praha, čije su karakteristike u direktnoj vezi sa strukturom i fizičko-mehaničkim karakteristikama deponovanih prevlaka.*

*Svaki od tehnoloških postupaka daje proizvedenom prahu specifične karakteristike, na osnovu kojih se vrši odabir praha za zaštitu funkcionalnih površina u proizvodnji novih mašinskih delova i za reparaciju pohabanih površina mehanizmima habanja, kavitacije i korozije na niskim i povišenim temperaturama i za specijalne namene.*

Ključne reči: *tehnologije, prahovi, morfologija, mikrostruktura.*

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