СТРУЧНИ ЧЛАНЦИ

ПРОФЕССИОНАЛЬНЫЕ СТАТЬИ

PROFESSIONAL PAPERS

# CHARACTERISTICS OF PLASMA SPRAY COATINGS

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*Summary:*

*The characteristics of plasma spray coatings are directly related to the type of process applied (APS - atmospheric plasma spray, VPS - vacuum plasma spray, and SPS - suspension plasma spray), the characteristics of the powder and the powder deposition parameters. The properties of plasma spray coatings can be changed and adjusted depending on the operating conditions, i.e. the purpose of coatings. Applying the same powder in the deposition process can lead to coatings of different properties, depending on their purpose. If the deposited coating is intended for corrosion protection and biomedical application, it is necessary that the coating is compact. When the coating is deposited for the purpose of thermal insulation, it should be porous. Powder particles in the plasma are spherical liquid drops which, in collision with the substrate, form a lamellar structure of the coating. A higher degree of powder melting and a higher velocity of molten droplets lead to a denser structure of deposited layers. When colliding with the substrate, deposited particles transfer heat to a working part until cooling to ambient temperature. If the working part is cooled by compressed air, molten droplets solidify extremely quickly with columnar crystals in lamellae. The aim of this paper is to describe the influence of temperature and velocity of powder particles on the deformation and hardening of particles defined by the D/d ratio, chemical changes in molten particles as well as porosity, crystal changes, and internal stresses in coatings.*

*Keywords: APS, VPS and SPS process, adhesion/cohesion, structure, porosity, internal stresses.*

## Introduction

The properties of plasma spray coatings depend on: the degree of melting of powder particles, the degree of deformation of individual particles in collision with the substrate, their interaction upon impact on the substrate surface and the interactions of lamellae at the contact during deposition and layer solidification. In the process of deposition, higher temperature and velocity of molten droplets increase coating adhesion, cohesive strength, tensile strength and resistance to friction fatigue, while reducing coating brittleness, which all affects the length of coating service life (Li et al, 2006, pp.1166-1172), (Sobieszczyk, 2010, pp.30-42). The characteristics of deformation and flattening of molten droplets of powder particles during deposition are of utmost importance, defined by the $D/d$ ($D$ - diameter of a deformed particle, and $d$ - diameter of an injected particle) ratio. One of important parameters which affect the coating characteristics is contact temperature ($Tc$) at the interface between the substrate and the deposited layer. An increase in contact temperature ($Tc$) increases the coating – substrate adhesion strength and the density of layers in the coating. Adhesive strength determines the coating quality, while the cohesive strength of layers determines the coating service life (Bull, Rickerby, 2001, pp.181-228). The melting point of powder particles is often higher than the melting temperature of the substrate on which the coating is deposited, so the contact temperature can increase the temperature of the substrate. The increase in the substrate temperature significantly affects the stress state of the substrate / coating interface, which directly reflects on the interface fracture toughness. In some cases, it can cause fatigue in deposited layers, which causes micro and macro cracks in coating layers followed by peeling or separation of coating layers from the substrate surface through coating layers. Also, due to large temperature differences, at some places on the coating surface, segmental micro cracks can occur due to large difference in tensile stress caused by the substrate expansion and shrinkage stress during the solidification of deposited particles (Mrdak, 2016, pp.411-430). In extreme cases, the substrate may be deformed. This can happen during the process of melting and depositing powder particles of refractory metals such as W, Nb, Ta, Mo, Ti, etc. The same phenomenon can occur in the deposition of inorganic bioinert ceramics: $TiO_2$, $Al_2O_3$, $ZrO_2$, $ZrO_2Y_2O_3$, and $Cr_2O_3$, as well as carbides WC, TC, BC, CrC, etc.

This paper describes the *D/d* ratio of the particles of $Al_2O_3$ inorganic bioinert ceramics depending on the temperature and velocity of powder particles of different granulation. The aim of this study was to describe the chemical changes in molten particles, the crystal changes in deposited particles, porosity in coatings and its effect on the mechanical properties of the deposited coatings, and stresses in coatings.

## Effect of temperature and velocity of powder particles on the D/d ratio

During powder deposition, it is necessary to set parameters so that particles have optimal temperature and velocity (kinetic energy) for a certain type of powder of specific physical characteristics and granulation. The substrate temperature also affects the quality of the coating and during deposition it is necessary to maintain the substrate temperature within certain limits without causing side effects. The contact temperature (*Tc*), which directly influences the *D/d* ratio, depends on the substrate preheating temperature and on the powder melting point, as well as on powder thermal conductivity and cumulativity. The parameters are always set to achieve the highest *D/d* ratio. Ignoring the effect of surface stresses and the assumption that molten particles flatten before solidification has led to the execution of the terms of the *D/d* ratio defined by equation 1 (Sivakumar, Nishiyama, 2004, pp.485-489),

$$D/d = 1{,}29 \left( \frac{\rho V d}{\mu} \right)^{0.2} \tag{1}$$

where: is $\rho$ - density of particles $\mu$ - viscosity of particles, *Vd* – velocity of particle impact on the substrate. For $Al_2O_3$ ceramics used in the process of biomedical coatings, temperature and velocity of powder particles are very important. The $Al_2O_3$ powder particle velocity of 100 m/s - 400 m/s is obtained by the ratio of *D/d* = 3 - 6 (Mishin et al, 1987, p.620). Figure 1 shows the *D/d* ratio for $Al_2O_3$ powder with a particle surface temperature of 2300 °C depending on particle velocity for the following granulation values: 5, 10, 20, 30 and 35 µm (Vardelle et al, 1993, pp.79-91). For a powder surface temperature of 2300 °C, the *D/d* ratio increases with an increase in particle velocity regardless of the granulation size.
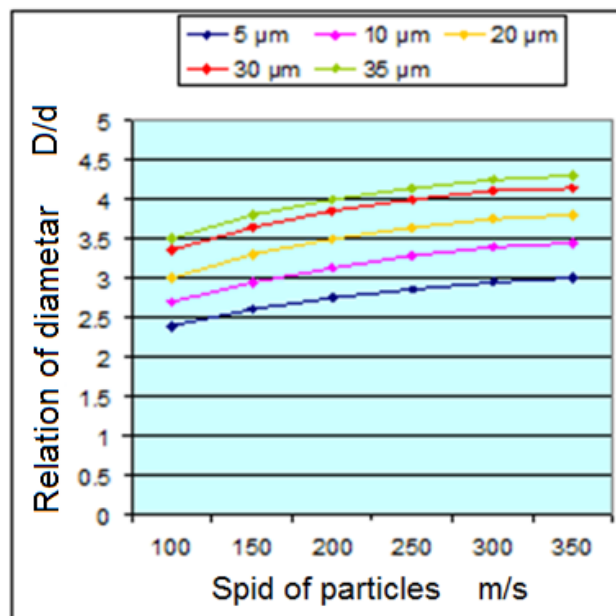
*Figure 1 – The D/d ratio depending on the velocity of Al₂O₃ particles*
*Рис. 1 – Соотношение диаметра D/d в зависимости от скорости частиц Al₂O₃*
*Слика 1 – Однос пречника D/d у зависности од брзине честица Al₂O₃*

With an increase in velocity, the particle kinetic energy increases, which causes a higher degree of flattening of molten particles on the substrate surface. The lowest value of the *D/d* ratio, i.e. 2.4, is found in particles of 5 μm deposited with a velocity of 100 m/s. For particles of 35 μm and a velocity of 100 m/s, this ratio is much higher and amounts to *D/d* = 3.5. For the finest particles with a maximum speed of 350 mm/s, the *D/d* ratio is 3.0 and does not reach the levels of the largest particles with a minimum velocity. The highest *D/d* ratio of 4.3 is achieved with particles of the grain size of 35 μm. Figure 2 gives the *D/d* ratio for Al₂O₃ powder with a particle velocity of 300 m/s depending on the surface temperature of molten particles for the granulation values of 5, 10, 20, 30 and 35 μm. The surface temperature of powder particles has a similar effect on the D/d ratio as the velocity of particles. With an increase in particle surface temperature, particle plasticity increases causing more pronounced flattening of molten droplets on the substrate surface. For the lowest particle surface temperature of 2000 °C, the lowest *D/d* ratio of 2.4 is found in the particles with the smallest size of 5 μm, and the highest *D/d* ratio of 3.5 is found in the biggest particles of 35 μm.
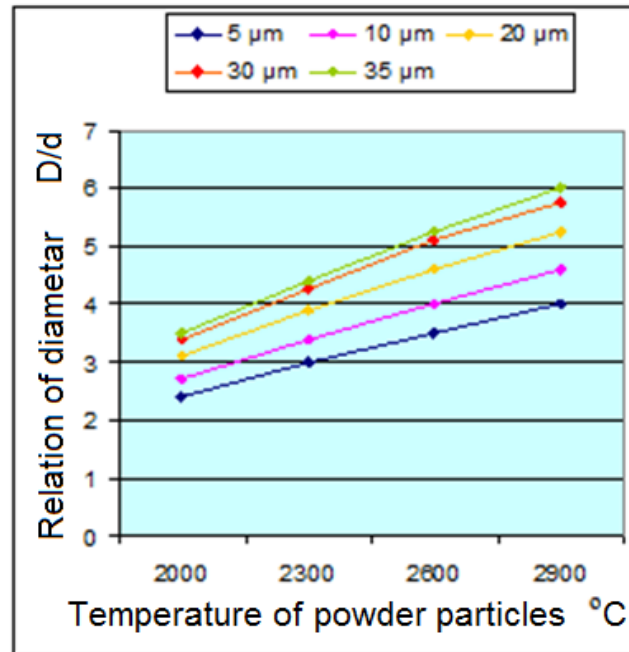
*Figure 2 – The D/d ratio depending on the temperature in $Al_2O_3$ particles*
*Рис. 2 – Соотношение D/d в зависимости от температуры частиц $Al_2O_3$*
*Слика 2 – Однос D/d у зависности од температуре честица $Al_2O_3$*

When temperature increases to 2900 °C, the *D/d* ratio increases to a maximum value. For the 5 μm granulation, the *D/d* ratio is 4.0, and for the granulation of 35 μm, the *D/d* ratio is 6.0. If we compare the effects of particle velocities and the surface temperatures of particles of different sizes on the flattening degree, it is clear that the *D/d* ratio is influenced more by the surface temperature of powder particles (Vardelle et al, 1993, pp.79-91).

## Chemical changes in molten particles and crystal changes in coatings

In molten powder particles, chemical changes can occur as a result of the reaction of liquid droplets with the plasma jet. High plasma temperature is suitable for the reactions such as oxidation, reduction and thermal decomposition of the initial powder phases. Many metals absorb a significant amount of oxygen during deposition, oxides lose oxygen and

nitrides lose nitrogen. The chemical reaction is determined by the plasma gas diffusion into the liquid phase of molten particles. When $H_2/Ar$ or $He/Ar$ are used as plasma gases, diffusion is carried out with a fairly low speed of $10^{-3}$ to $10^{-1}$sec in comparison to the particle melting time of $10^{-4}$sec. During the deposition of $Al_2O_3$ powder with a mixture of $N_2/Ar$ as plasma gas, aluminum from the oxide reacts with nitrogen. The result of this reaction is the formation of the AlN phase. Oxidation is a major problem, especially in metals such as tungsten, molybdenum and titanium. Some oxidized particles can be seen in coatings. The oxidation of powder particles is very important and affects porosity as well as cohesion and adhesion strength. The problem is greater in the oxidation of carbides. Oxygen binds carbon from the carbide so that the carbide decomposes. The carbide decomposition is favored by high temperature of plasma. Thermal decomposition of carbides is incomplete due to short time powder particles spend in the plasma jet (Qiao et al, 2003, pp.24-41).

Extremely rapid cooling of the liquid phase of the material deposited may affect the suppression of the crystallization of particular phases. A typical effect was tested with $Al_2O_3$ particles. The coating is predominantly composed of one or more metastable phases, with the mostly dominant $\alpha$-$Al_2O_3$ phase and a smaller share of the $\gamma$-$Al_2O_3$ phase. The formation of metastable phases can be explained by lower free energy for the creation of spinel cells from the liquid phase when the subcooling is below the equilibrium melting temperature due to rapid cooling. The phases are separated by transformation from one to another metastable phase. Different metastable phases are separated depending on the deposition parameters, the size and shape of particles and the substrate temperature. The ratio between substrate and coating cooling during powder deposition is very important. When the substrate is cooled during deposition, the cooling rate of particles is between $10^4$ and $10^6$ K/s. In order to obtain coatings which contain only the $\alpha$-$Al_2O_3$ phase, it is necessary to heat the substrate to a temperature of 1100 °C (Yang et al, 2006, pp.1649-1653). The $\alpha$-$Al_2O_3$ phase is always present in the coating with a share of 10% to 25%, because of the incomplete melting of powder particles which serve as centers of crystallization (Friis et al, 2001, p.115).

During deposition, the starting $\alpha$-$Al_2O_3$ phase with a density of 3.98 to 4.0 g/cm$^3$ is transformed into the $\gamma$-phase with a density of 3.3 to 3.4 g/cm$^3$, which is stable up to a temperature of 1050°C (Yang et al, 2006, pp.1649-1653). Due to its lower density, the separated $\gamma$-phase increases

porosity and decreases the mechanical properties of the coating. For metals and alloys, the cooling rate of molten particles is higher and ranges from $10^6$ to $10^8$ K/s (Johnston, 2009, pp.1004-1013). For superalloys with this cooling rate ratio, it is possible to obtain very fine grain sizes from 0.25 to 0.5 $\mu$m, which gives very good mechanical properties of the coating. If coatings are overheated at temperatures from 100 to 1200° C, higher density and better mechanical properties are obtained (Mrdak, 2015, pp.337-343). In some coatings, during the deposition of molten droplets, crystals become conveniently directed relative to the substrate surface. This refers primarily to $Cr_2O_3$ oxide, where the c - axis and the basal planes are conveniently directed relative to the surface, thus giving it high resistance to friction. This also applies to the hexagonal structures of the coatings of $W_2C$, CoW and CoMo which have a low friction coefficient. These coatings, therefore, have a friction coefficient lower than 0.15 (Hakan et al, 2008, pp.259-265).

## Porosity of coatings

Depending on coating purposes, the share and size of pores, together with their distribution through layers, are of key importance for the mechanical properties of the coating (Mrdak et al, 2015, pp.337-343). High porosity in layers reduces the coating strength: it increases the coating brittleness, reduces the brittleness of the interface fracture and friction resistance, as well as resistance to oxidation and corrosion. The cause of pore formation in the coating is the uneven granulation of powder particles injected into the plasma jet which move along different paths through the plasma jet. Porosity is present in the coatings which consist of tough and fully melted powder particles. Substrate surfaces and molten particles may contain air or gas which also causes the formation of pores at the interface, reducing adhesion. If particles have high temperature and high velocity when impinging the substrate, liquid drops of molten particles are sprayed causing high porosity in the coating layers. In the coatings deposited at atmospheric pressure, porosity is in the range from 5 to 20% (Mrdak et al, 2015, pp.337-343).

Materials such as $TiO_2$, $Al_2O_3TiO_2$, $Cr_2O_3$, and NiO form coatings with an average porosity below 4%. In $Al_2O_3$ and $ZrO_2$ coatings, porosity ranges from 4-8%, while porosity in thermal barriers ($ZrO_2Al_2O_3$, $ZrO_2Y_2O_3$, $ZrO_2CaO$, $ZrO_2MgO$, $ZrO_2Y_2O_3CeO_2$, etc.) ranges from 8 to 15 % (Mrdak et al, 2015, pp.337-343), (Mrdak, 2017, pp.30-44). Pore share and size in the coating are directly related to the angle at which the molten particles are deposited and to the tension of deposited particles.

TiO$_2$ powder particles have half the strength of the Al$_2$O$_3$ surface; therefore, TiO$_2$ coatings have for 2.5% lower porosity compared to Al$_2$O$_3$ coatings. Generally, the coating porosity is linked to the size of powder particles, their velocities and temperatures in the moment of impact with the substrate. It is clear that the lowest porosity is obtained with particles that are well melted and have high velocity. Coating layers have a porosity of 23% with Al$_2$O$_3$ particles of a granulation of 18 $\mu$m deposited on the substrate from a distance of 75 mm. When the distance to the substrate is reduced to 50 mm, powder particles gain maximum velocity and reduce the proportion of pores to 11% (Vardelle et al, 2001, pp.267-284). High-speed plasma jets are suitable for the preparation of coatings with low porosity. High power supply allows for a faster transfer of heat and velocity of plasma particles onto powder particles. The application of a high-power plasma gun is limited; the gun is not used for the deposition of powder with a small coefficient of thermal conductivity because the particle surface vaporizes while the particle core remains unmolten.

## Internal stresses in coatings and on the interface

Generally, it can be said that powders of tough materials give tough coatings with low percentage of residual stresses, while ceramic powders produce brittle coatings with a considerable share of residual stresses. The internal stresses in coatings are the result of different temperatures and coefficients of thermal expansion of substrates and coatings. The substrate bond with the ceramic coating is non-metallic; therefore, the bond strength values are lower than the values achieved with metal coatings. The increase in coating thickness leads to the reduction of bond strength. For some ceramic coatings the thickness of which is up to 0.1 mm, the bond strength values range from 30 to 40 MPa. For the thickness values up to 0.3 mm, these values are smaller and range from 5 to 10 Mpa. If the thickness values are over 0.5 mm, strength values reach only 4 MPa (Moridi et al, 2014, pp.449-459). Since coatings consist of several layers, stresses also increase with the coating thickness. In order to reduce stresses caused by the temperature difference and the coefficients of thermal expansion, it is necessary to cool down the substrate and the deposited layers during deposition. Also, if there is a low temperature difference between the substrate and the deposit, this may cause cracking and spalling of the deposit from the substrate. Within individual sections in the coating, micro-cracks can occur, as well as within the entire coating. Micro-cracks in the coating occur as a result of the influence of the substrate on the shrinkage of molten particles during

solidification. The shrinkage of molten powder particles is always limited because of the influence of the substrate. The values of residual stresses mostly depend on the ratio of thermal expansion coefficients and on the elasticity coefficient between the substrate and the coating (Mrdak et al, 2015, pp.337-343). Also, there is the influence of yield and plastic deformation of the coating and the substrate on the value of residual stresses at the interface. Macro cracks may occur In the coating after cooling the coating structure to ambient temperature due to a large temperature gradient between the substrate and the coating layer during deposition, as well as due to differences in the coefficients of thermal expansion (Mrdak, 2017, pp.30-44). High levels of stress at the interface could cause peeling of the coating, while high levels of tensile stresses could cause internal cracks in the deposit. Residual compressive stresses in ceramic coatings could even be beneficial for the increase of fracture strength. In order to reduce stresses in coatings, it is necessary to reduce the temperature gradient between the substrate and the coating. This allows the shrinkage of the coating at lower temperatures during solidification. Therefore, during the deposition of powder particles, coating primary and secondary cooling (after powder deposition) is applied. However, despite cooling both the coating and the substrate, it is extremely difficult to reduce stresses in the coatings with low coefficients of thermal conductivity, especially in the case of thicker coatings. Internal cracks in the deposited layers are formed as a result of lower resistance of layers to tension and compression. In ceramic materials, compressive strength is higher than tensile strength. Residual stresses are usually the result of different coefficients of expansion of the layers $\alpha 1$ and the substrate $\alpha 2$. If $\alpha 1 > \alpha 2$, the coating will be bent and deformed as shown in Figure 3 or it will be exposed to tensile stresses if the coating is deposited on thicker substrates. When ceramic powders are deposited on metal substrates, the coating will be distorted or subjected to compressive stress, as shown in Figure 4.
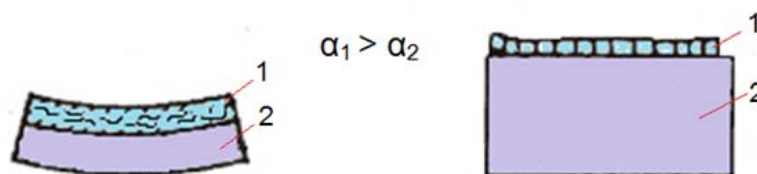


*Figure 3 – Stresses in the coating for $\alpha_1 > \alpha_2$*
*Рис. 3 – Напряжения в покрытии для $\alpha_1 > \alpha_2$*
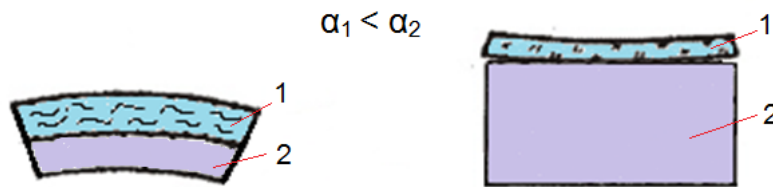*Слика 3 – Напони у превлаци за $\alpha_1 > \alpha_2$*

*Figure 4 – Stresses in the coating for α₁ < α₂*
*Рис. 4 – Напряжения в покрытии для α₁ < α₂*
*Слика 4 – Напони у превлаци за α₁ < α₂*

High stresses in layers will be reduced through cracks or through separation of the coating from the substrate. During deposition, controlling layer temperature is of primary importance, especially for materials with a low coefficient of thermal conductivity. If temperature during the deposition process is not under control, a large temperature gradient may appear between the upper and lower surface layers, which is undesirable. In the coatings with changes in volume due to phase transformations, residual stresses are increased. These stresses are also increased during exploitation as a result of cyclic phase transformations. For example, heating the metastable $\gamma$ - $Al_2O_3$ phase at temperatures above 1050 °C will transform it into the metastable $\alpha$ - $Al_2O_3$ phase followed by an increase in volume, which adds to residual stresses (Yang et al, 2006, pp.1649-1653). The same is the case with the metastable tetragonal t - $ZrO_2$ phase which, at temperatures above 1100 °C under the influence of stress, transforms into the monoclinic phase of m - $ZrO_2$ accompanied by an increase in volume. Phase transformations occurred in service will further increase stresses in coatings and reduce coating resource. Due to the above, porosity in coatings is preferred in e.g. filters and thermal barriers. Desired porosity levels can be easily obtained in coatings as well as controlled through powder granulation and the location of powder injection into the plasma jet.

## Conclusion

This article describes the impact of temperature and velocity of powder particles on the *D/d* ratio, chemical changes in molten particles and crystal changes in coatings, porosity in coatings and internal stresses in coatings and at the interface. Based on the above, the following conclusions can be drawn.

The powder deposition parameters are always adjusted so that the highest *D/d* ratio is obtained for injected particles. The *D/d* ratio increases with increasing particle velocity and temperature regardless of the size of granulation. The comparison of the effect of particle velocity and the effect of the temperature values of particle surfaces of different granulations on the flattening degree has shown that the surface temperature of powder particles has a greater impact on the *D/d* ratio.

In molten powder particles, chemical changes (oxidation, reduction and thermal decomposition of the powder initial starting phase) occur as a result of the reaction of liquid drops with the plasma yet. Extremely rapid cooling of the liquid phase of the depositing material may affect the suppression of crystallization of individual phases. Different metastable phases are separated depending on the deposition parameters, the size and shape of the particles and the substrate temperature. Chemical and crystal changes in the deposited particles significantly affect porosity, cohesion and adhesion strength, fatigue and wear, which is why it is necessary to apply strict control of the coating production process.

Porosity in layers reduceses coating strength, increases coating brittleness, reduces interface fracture brittleness and abrasion resistance as well as resistance to oxidation and corrosion. Pores in coatings are formed due to uneven granulation of powder particles that are injected into the plasma jet and move along different paths through the plasma jet.

The internal stresses in coatings are the result of different temperatures and coefficients of thermal expansion of substrates and coatings. If $\alpha_1 > \alpha_2$, coatings are to be bent and deformed on thicker substrates. If $\alpha_1 < \alpha_2$ when depositing ceramic powders on metal substrates, the coating will be deformed or subjected to compressive stress. High stresses in layers will be reduced through cracks or through separation of the coating from the substrate. In order to reduce stresses caused by the temperature difference between the coefficients of thermal expansion, it is necessary to cool down the substrate and deposited layers during deposition.

The article displayed some of the important parameters affecting the characteristics of plasma spray coatings, which must be taken into consideration when creating a coating for a given purpose.

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## ХАРАКТЕРИСТИКИ ПОКРЫТИЯ, НАНЕСЕННОГО ВОЗДУШНО-ПЛАЗМЕННЫМ НАПЫЛЕНИЕМ

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ЯЗЫК СТАТЬИ: английский

*Резюме:*

*Характеристики покрытий, нанесенных плазменным напылением напрямую взаимосвязаны с видом применяемого процесса (APS - atmospheric plasma spray, VPS - vacuum plasma spray и SPS - suspension plasma spray), свойствами порошка и параметрами осаждения порошка. Свойства покрытий, нанесенных плазменным напылением, могут быть изменены и приспособлены к условиям эксплуатации и их назначению. При использовании одного и того же порошка в процессе осаждения могут быть получены различные свойства покрытий в зависимости от их назначения. Так, например, если покрытие предназначено для антикоррозионной защиты или биомедицинских нужд, то необходимо чтобы оно было как можно более компактным. В то время как при нанесении покрытия для нужд термоизоляции благоприятным свойством будет считаться пористость. Частицы порошка в плазменной струе представляют собой капли жидкости, имеющие сферическую форму, которые при сцеплении с подложкой образуют ламинарную структуру покрытия. Чем выше температура плавления порошка и чем больше скорость капель расплава, тем плотнее будет структура нанесенных слоев. При столкновении с подложкой осажденные частицы передают тепло рабочей поверхности до момента их охлаждения до температуры окружающей среды. В случаях, когда рабочая поверхность охлаждается сжатым воздухом, отверждение капель расплава ускоряется максимально со столбчатыми*

кристаллами в ламелях. Целью данной работы являлось описание того, каким образом температура и скорость частиц порошка влияют на образование деформаций и затвердения частиц, которые определяют соотношение D/d, пористость в слоях покрытия, химические реакции в частицах расплава и на кристаллические изменения в слоях покрытия, пористость и внутреннее напряжение покрытия.

Ключевые слова: APS, VPS и SPS процесс, адгезия/когезия, структура, пористость, внутренние напряжения.

## КАРАКТЕРИСТИКЕ ПЛАЗМА СПРЕЈ ПРЕВЛАКА

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*Сажетак:*

*Карактеристике плазма спреј превлака су у директној вези са врстом примењеног процеса (APS – atmospheric plasma spray, VPS – vacuum plasma spray и SPS – suspension plasma spray), карактеристикама праха и параметрима депозиције праха. Својства плазма спреј превлака могу се мењати и подешавати у зависности од радних услова, односно намене превлака. При употреби истог праха у поступку депозиције могу се постићи различита својства превлаке, зависно од намене. Ако се превлака депонује за корозиону заштиту и биомедицинску примену, неопоходно је да буде компактна, а када се депонује ради топлотне изолације пожељно је да буде порозна. Честице праха у плазми су течне капи сферног облика које у судару са подлогом формирају ламеларну структуру превлаке. Већи степен топљења праха и већа брзина истопљених капи производи гушће структуре депонованих слојева. На контакту са подлогом депоноване честице преносе топлоту радном делу до хлађења на температуру околине. Ако се радни део хлади компримованим ваздухом, очврћавање истопљених капи одвија се екстремно брзо са стубастим кристалима у ламелама. Циљ рада јесте да се опишу: утицај температуре и брзине честица праха на деформацију и очврћавање честица које дефинише однос D/d, порозност у превлакама, хемијске промене у истопљеним честицама и кристалне промене у превлакама, порозност у превлакама и унутрашњи напони превлака.*

*Кључне речи: APS, VPS и SPS процес, адхезија/кохезија, структура, порозност, унутрашњи напони.*

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