СТРУЧНИ ЧЛАНЦИ

ПРОФЕССИОНАЛЬНЫЕ СТАТЬИ

PROFESSIONAL PAPERS

ANALYSIS OF RESIDUAL STRESSES IN BIOINERT INORGANIC PLASMA SPRAYED CERAMIC COATINGS

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Summary:

One of the factors for a successful application of ceramic coatings on biomedical implants is the compatibility of the physical and mechanical properties of coatings with the metal substrates of implants. Temperature and temperature gradient in the coating during powder deposition play an important role in the final quality of the coating. The coefficients of thermal expansion and thermal conductivity of the coating and the substrate are different, which affects the growth of residual stresses in coatings. To reduce the difference between the physical characteristics of the coating and the substrate to a minimum, the coating surface temperature and the substrate surface temperature must be kept under control during the deposition of powder. It is therefore of particular importance to control residual stresses in ceramic coatings in order to secure service life of coatings and implants. The paper describes a model of plasma heat transfer and predicts the distribution of residual stresses in the deposited coatings; it also describes the radiography techniques for measuring residual stresses in ceramic coatings. The aim of this paper is to describe the effect of powder deposition rates as well as the effect of the changes in the thickness and the thermal conductivity of the ZrO_2CaO coating on the level and the sign of residual stresses.

The paper also presents the influence of the bonding coating, and the changes in the thickness of bonding and the ceramic ZrO$_2$MgO coating as well as the heat treatment on the level and the sign of residual stresses. It was found that the increase of the total thickness of the coating increases the proportion of residual stresses on the surface and the edges of the coating.

Keywords: residual stresses, positive tensile stresses, negative compression stresses, ZrO$_2$CaO, ZrO$_2$MgO, heat treatment.

Introduction

Distribution of residual stresses in biomedical ceramic coatings based on hydroxyapatite (HA), TiO$_2$, Al$_2$O$_3$, ZrO$_2$Y$_2$O$_3$, CaO and MgO is of decisive influence on coating adhesion, cohesive strength and toughness which define coating quality and service life. The share and the distribution of residual stresses in coatings is directly related to the ratio of coefficients of thermal expansion of substrates and coatings. To minimize the influence of thermal expansion coefficients of the residual stress in the coating layer, it is necessary to reduce the temperature difference ΔT between the substrate and the layers during powder deposition. During the plasma spray process, there is a large temperature gradient between molten powder and base metal substrates. Heat shrinkage during coating curing is hindered by the base metal, so that residual stresses are formed inside the coating. Because of the low thermal conductivity of ceramics, powder particles which melt in the plasma before they hit the substrate surface cool rapidly from 10^5°C/s do 10^6 °C/s. During the deposition, primary cooling of layers and secondary cooling of the coating are performed to the substrate temperature to minimize the influence of thermal expansion of the coating substrate. Residual stresses in coatings can be kept under control by constant cooling of the substrate from the back side and by an additional air-cooled front on which coating layers are deposited (Limarga et al, 2011). For the substrates of implants based on Ti6Al4V superalloy, the coefficient of thermal expansion is α = 8.7-9.1×10^{-6}/°C. For coatings used in biomedicine, the coefficients of thermal expansion are approximate values, and their values are: hydroxyapatite (HA) (α = 10.6×10^{-6}/°C), TiO$_2$ (α = 8.5×10^{-6}/°C), Al$_2$O$_3$ (α = 8.8×10^{-6}/°C), ZrO$_2$ (α = 9,0×10^{-6}/°C), ZrO$_2$Y$_2$O$_3$ (α = 10.0×10^{-6}/°C), ZrO$_2$MgO (α = 11.0×10^{-6}/°C), ZrO$_2$CaO (α =1 0.5 ×10^{-6}/°C) (Miyazaki et al, 2008, pp.1463-1466), (Mrdak, 2017, pp.924-936). Besides the physical properties of the substrate material and the powder to be deposited, powder deposition parameters significantly influence the stress states in coatings. The study

of stresses in layers by nondestructive testing and the examination of the coating morphology using tensile testing can show the optimum parameters of powder deposition. The residual stresses in coatings are distributed in the middle of the sample (negative compression stresses), and on the edges of the sample (positive tensile stresses). The examination of residual stresses includes their distribution and their values, s well as the way how their control can affect the life of coatings (Hobbs & Reiter, 1988, pp.33-42). Residual stresses in plasma sprayed coatings are commonly measures by the X-ray diffraction method and by the tensile testing method. High resolution of X-rays through intense long wavelengths offers a significant potential for the measurement of residual stresses in coatings. Other methods used are neutron diffraction (Kesler et al, 1998, pp.215-224), (Matejicek et al, 1999, pp.607-617), the method of measuring the curvature, the method of removing material layer by layer and by tensile testing (Teixeira et al, 1999, pp.209–216), (Greving et al, 1994, pp.379-388), (Clyne & Gill, 1996, pp.401-418), (Zhu et al, 2014, pp.127-136). These methods make it possible to establish the average stresses in coatings. However, the distribution of residual stresses through the coating thickness is rarely published (Otsubo et al, 2005, pp. 2473-2477).

The aim is to explain the residual stresses in the ceramic coatings on the basis of ZrO_2CaO and ZrO_2MgO used in biomedical applications to improve the mechanical properties of hydroxyapatite (HA). This paper presents a model of heat transfer from plasma to the coating, powder deposition rate impact, changes in the thickness and thermal conductivity of ZrO_2CaO coatings on the level and sign of residual stresses. It also shows the effect of bond coating, changes in the thickness of the bond and the ceramic ZrO_2MgO coating and heat treatment on the level and sign of residual stresses. Generally, with increasing the total thickness of the coating, the proportion of residual stresses on the coating surface and its edges also increases.

Model of plasma heat transfer and the prediction of the distribution of residual stresses in ceramic coatings

To connect residual stresses in coatings with the deposition parameters, a model of heat transfer, shown in Figure 1, was developed. Research conducted for ZrO_25CaO ceramics may be applied to another type of coating (Rickerby et al, 1988, pp.267-276). As mentioned before, the powder deposition process by plasma consists of the injection of powder particles into high-temperature plasma made of inert gases

(Ar/He) with an electric arc in the nozzle cooled with water. Powder particles injected into the plasma jet melt and then hit the surface of the substrate where they are cooled at a speed of $10^{5}°C/s$ to $10^{6}°C/s$. Molten powder particles arrive onto a stable surface where they collide, deposit and bind to the surface to be cooled. These sites are graded and increased while powder particles deposit on the surface. The thickness of the coating increases with speed (η). During the deposition, there was a simulation of a model of transferring heat from the coating to the substrate surface and the heat transfer by radiation from plasma to the deposition surface and by radiation of the deposition surface into the environment. When defining the heat transfer model, the basic assumption was that the plasma spray process is continuous, the thickness of the substrate is greater than the thickness of the coating, the heat loss is presented with a standard convection and radiation, and thermal and electrical properties are temperature-invariant.
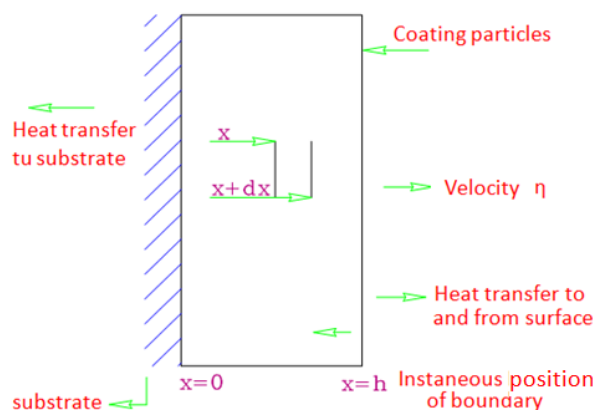

Figure 1 – The basic elements of the model of heat transfer in the process of deposition
Рис. 1 – Основные элементы теплообмена в процессе нанесения покрытия
Слика 1 – Основни елементи модела преноса топлоте у процесу депозиције

In order to achieve the above mentioned cooling rate ($10^{5}°C/s$ - $10^{6}°C/s$), methods of rapid heat removal were used together with a controlled stream of dry air (primary cooling). The main parameters used in the modeling of residual stresses in the plasma deposited coatings are shown in Table 1.

Figure 2 shows the diagram providing the variants of distribution of residual stresses in the plasma deposited $ZrO_2 5CaO$ coatings as a function of the powder deposition rate (Rickerby et al, 1988, pp.267-276).

Table 1- Parameters of the modeling of residual stresses in plasma coatings
Таблица 1 – Параметры моделирования остаточных напряжений в покрытиях плазменного напыления
Табела 1 – Параметри моделирања заосталих напона у плазма превлакама

| Process parameters	Parameters of the coatings / surfaces
Deposition speed	Thermal conductivity of the coatings, specific heat and latent heat
Flame temperature	
Surface temperature	Coating density and Poisson number
Final coating thickness	Capacitance and the coefficient of the thermal expansion of the substrate
Ratio of the coating surface and the substrate surface	
Type and morphology of the substrate	
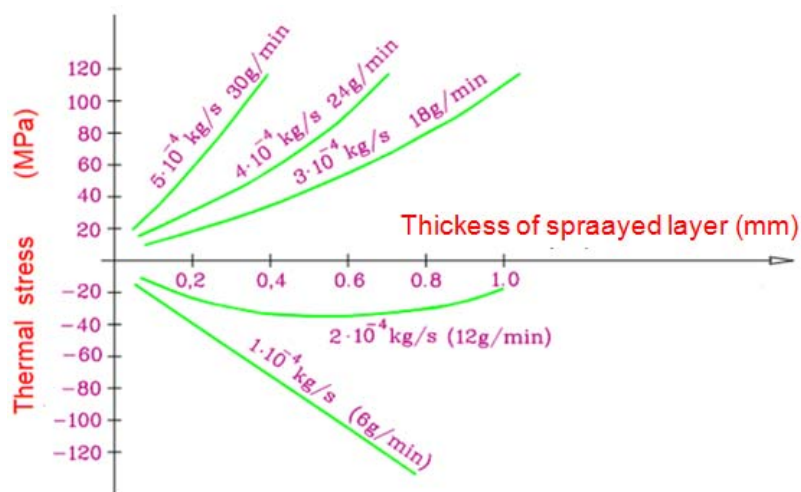
Figure 2 – Residual stresses in the ZrO$_2$5CaO coating as a function of deposition speed
Рис. 2 – Остаточные напряжения в покрытии ZrO25CaO в зависимости от скорости осаждения
Слика 2 – Заостали напони у превлаци ZrO$_2$5CaO у функцији брзине депозиције

The diagram shows that the increase of the deposition speed of powder in ZrO$_2$5CaO coating layers produce a change of state of calculated stresses. For low powder deposition speed of 6 g/min and 12 g/min, negative compression stresses are present in the layers. With the increase of the powder deposition speed to 18 g/min, stresses change

the sign from negative to positive tensile stresses (Rickerby et al, 1988, pp.267-276). Also of great importance is the effect of coating thickness on residual stresses. It is noted that a coating does not meet the requirements at a critical thickness which can be defined as the moment when the coating itself begins to separate from the substrate.

Figure 3 shows the change in the sign of residual internal stresses with the change of the total thickness of the ceramic $ZrO_2 5CaO$ coating. For coating thicknesses from 0.5 mm to 1.0 mm, residual internal stresses in the layers are negative compression stresses. By increasing the coating thickness, it is possible to create different zones of internal tensions, so that negative compression stresses remain in a part of the deposited layer on the boundary with the substrate, while in the zone close to the coating surface, positive tensile stresses can occur (Rickerby et al, 1988, pp.267-276).
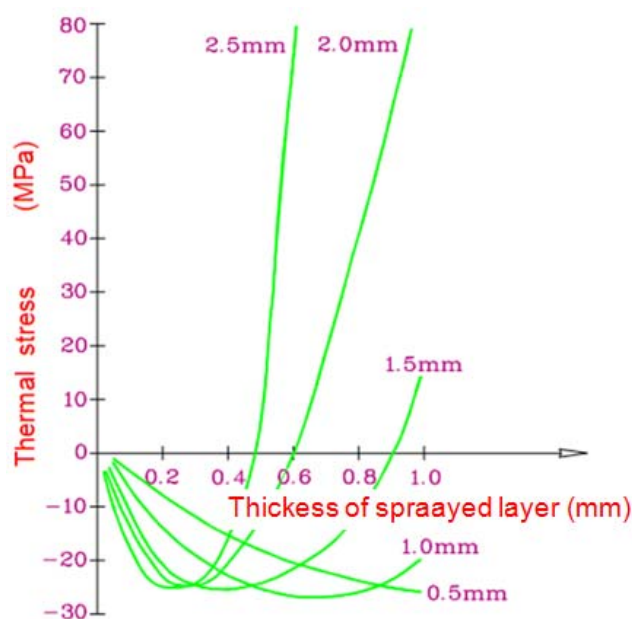

Figure 3 – The change of stresses with changing the thickness of $ZrO_2 5CaO$
Рис. 3 – Изменение напряжения при изменении толщины слоя покрытия $ZrO_2 5CaO$
Слика 3 – Промена напона са променом дебљине слоја $ZrO_2 5CaO$

One of the important factors that influence the level of residual stresses is the thermal conductivity of the coating. Figure 4 shows how small changes in the thermal conductivity of $ZrO_2 5CaO$ coatings can

have a significant effect on the level of residual stresses. Deviation values of thermal conductivity from 1 W/mK to 0.75 W/mK can be achieved by changing the degree of porosity in the ceramic coating which affects the value and sign of residual stresses. The increase of the thermal conductivity of the coatings from 1 W/mK to 2 W/mK leads to an increase in the value of negative compression stresses. Such increase can be beneficial to the life of the coating as a counter-balance to positive tensile stresses occurring in exploitation. However, a large increase in negative compression stresses can cause cracks in the coating during the coating deposition phase. Bearing in mind different conditions regarding the temperature of the outer surface and the ceramic coating heating rate, deposition conditions must be carefully chosen since they affect internal residual stress in order to obtain an optimal state of stress in the ceramic layer upon which the coating service life depends. Therefore, recommendations for thermal conductivity as low as only 0.5 W/mK can be found in the literature (Rickerby et al, 1988, pp.267-276).
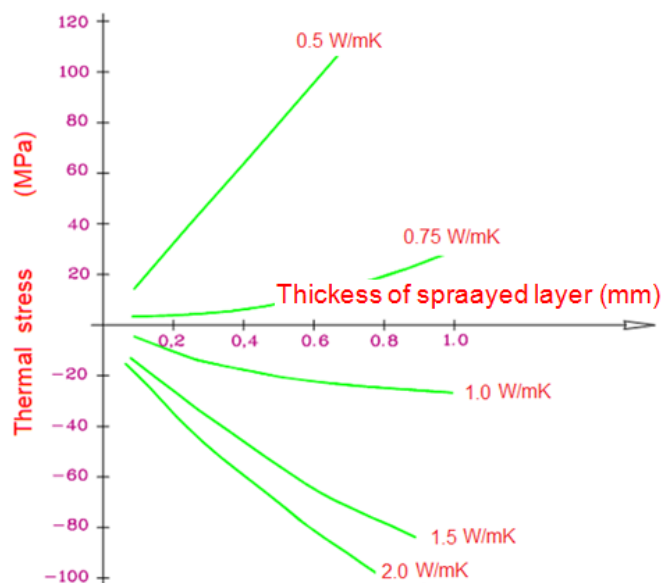

Figure 4 – Influence of thermal conductivity of residual stresses in the $ZrO_2 5CaO$ coating
Рис. 4 – Влияние теплопроводности на остаточные напряжения в покрытии $ZrO_2 5CaO$
Слика 4 – Утицај топлотне проводљивости на заостале напоне у превлаци $ZrO_2 5CaO$

The effect of bond coating and changes in coating thicknesses on the level of residual stresses

The level of residual stresses in deposited coatings is significantly influenced by bond coatings, changes in the thickness of bond coatings with respect to the outer ceramic coating, as well as changes in the thickness of ceramic coatings. Overall, the increase in the total coating thickness increases the proportion of residual stresses on coating surfaces and coating edges. Residual stresses in coatings can be significantly reduced by heat treatment (Zhuang & Gu, 1988, pp.277-284). D-9C is one of the instruments with which residual stresses are successfully measured with a change angle of 0.01° in coatings by the X-ray diffraction method. The theory of measuring residual stress in the coating is such that, when passing through the coating, the X-ray is reflected from a particular crystallographic plane and reduces its intensity. The measuring parameters are: operating voltage KV; operating current; CuK$_\alpha$ radiation and filter material (Zhuang & Gu, 1988, pp.277-284). During measurements, the angle (Ψ) between the X-ray and a vertical line on the sample surface is changed. Stress measurements are carried out in two directions $\Psi = 0°$ and $\Psi = 45°$, so that a corresponding dual angle of ray reflection of 2θ can be obtained. Using the theory of elasticity and the Bragg formula, the values of the residual stress along the (Ψ) direction can be calculated using the formula (1):

$$\sigma_\psi = -\frac{E}{2(1+\nu)} \cdot \frac{\pi}{180} \cdot ctg\,\theta_0 \frac{\partial(2\theta)}{\partial(\sin^2 \psi)} \tag{1}$$

where is: E - modulus of the coating elasticity, ν - Poisson share for the coating, 2θ - standard Bragg angle without stresses, 2θ - double reflected angle and Ψ - X-ray angle direction.

The effect of the bonding Ni20Cr coating of a thickness of 0.2 mm with a change in the thickness of the ceramic ZrO$_2$24MgO coating of: 0.1; 0.2; 0: 3; 0.4 and 0.5 mm on the level of residual stresses is shown in Figure 5. The residual stresses were measured in the middle of the samples by the X-ray diffraction method. Increasing the thickness of the ceramic coating increases the value of positive tensile stresses. Residual stresses are partially absorbed on the samples with the bond coating, which is a reason why two-layer coating systems have a much lower

share of residual tensile stresses compared to deposited coatings of pure $ZrO_2 24MgO$ ceramics (Zhuang & Gu, 1988, pp.277-284).

Figure 6 shows the influence of the thickness of the ceramic $ZrO_2 24MgO$ coating of 0.3; 0.4; 0.5; and 0.6 mm without the bond coating on the level of residual stresses in the middle and on the edge of the samples Ø55x5 mm. An increase in the thickness of ceramic coatings increases the values of negative compressive stresses in the middle and on the edge of the samples, which reach a maximum value for the thickest coating of 0.6 mm (Zhuang & Gu, 1988, pp.277-284). Figure 7 shows the influence of the change in the thickness of the bond layer of 0.1; 0.2; 0.3; 0.4 and 0.5 mm on the residual stresses in the two-layer system with a ceramic $ZrO_2 24MgO$ coating with a thickness of 0.2 mm.
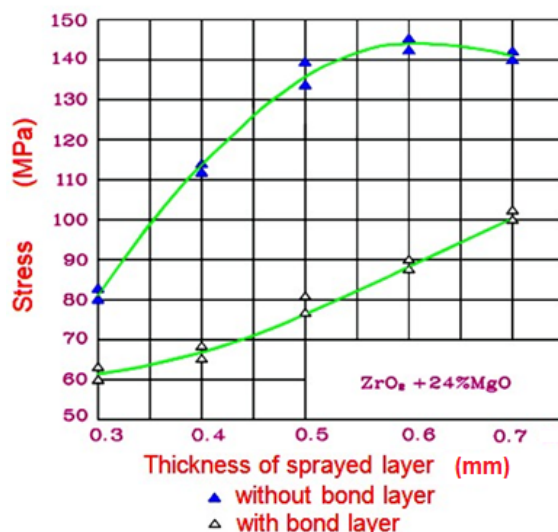

Figure 5 – Effect of the bond coating with a change in the thickness of the ceramic coating on residual stresses
Рис. 5 – Влияние связующего покрытия с измененной толщиной керамического покрытия на остаточные напряжения
Слика 5 – Утицај везне превлаке са променом дебљине керамичке превлаке на заостале напоне
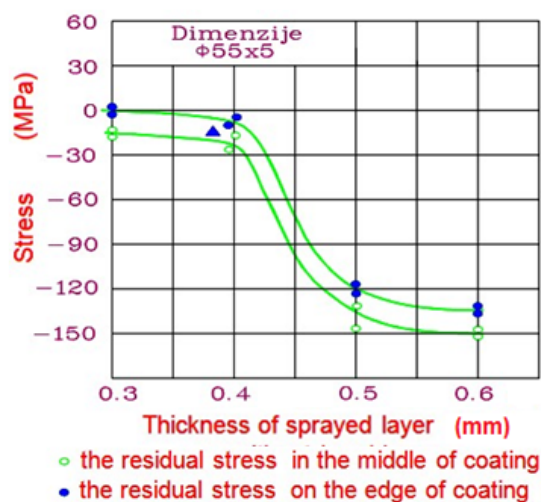
Figure 6 – Effect of the change in the thickness of the ceramic coating without the bond coating on residual stresses
Рис. 6 – Влияние изменения толщины керамического покрытия без связующего покрытия на остаточные напряжения
Слика 6 – Утицај промене дебљине керамичке превлаке без везне превлаке на заостале напоне
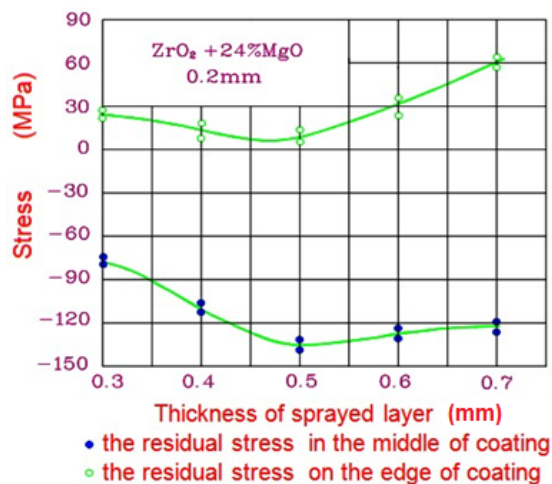

Figure 7 – The influence of the change in the bond layer thickness in the system with a ceramic coating on residual stresses
Рис. 7 – Влияние изменений толщины связующего покрытия в системе с керамическим покрытием на остаточные напряжения
Слика 7 – Утицај промене дебљине везног слоја у систему са керамичком превлаком на заостале напоне
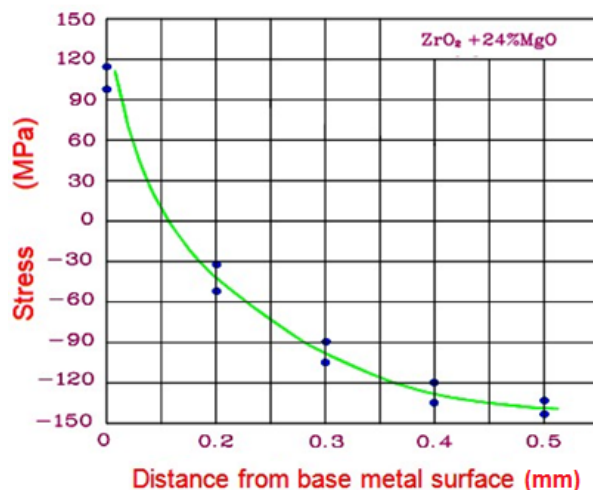
Figure – 8   Distribution of residual stresses along the depth of the ceramic coating without a bond layer
Рис. 8 – Распределение остаточных напряжений по глубине керамических покрытий без связующего слоя
Слика 8 – Расподела заосталих напона по дубини керамичке превлаке без везног слоја

Residual stresses on the edge of the samples were positive tensile stresses, while negative compression stresses were present in the middle of the samples. The distribution of residual stresses in the direction of the growth of coating thickness or coating depth is of great importance (Rickerby et al, 1988, pp.267-276). To determine the distribution and the sign of residual stresses in the coating depth, a layer after a layer was removed by 0.1mm grinding after each measurement. Figure 8 shows the stress distribution in the depth of the ceramic $ZrO_2 24MgO$ coating of a thickness of 0.5 mm and without the bond layer, deposited on the 5 mm thick substrate. The highest values of residual stresses are found on the coating surface. Stresses on the surface and on the coating boundaries are negative compression stresses whose values decrease with the decrease of the coating thickness. When the coating thickness is reduced to 0.1 mm, the stress sign is changed, i.e. negative compression stresses change into positive tensile stresses. The highest value of positive tensile stresses is found in the coating on the interface with the substrate (Zhuang & Gu, 1988, pp.277-284). Significant reduction of residual stresses in coatings can be achieved by heat treatment. Figure 9 shows the effect of the heat treatment of the two-

layered Ni20Cr coating of a thickness of 0.2 mm and the ceramic $ZrO_2$24MgO coating of a thickness of 0.4 mm on residual stresses.
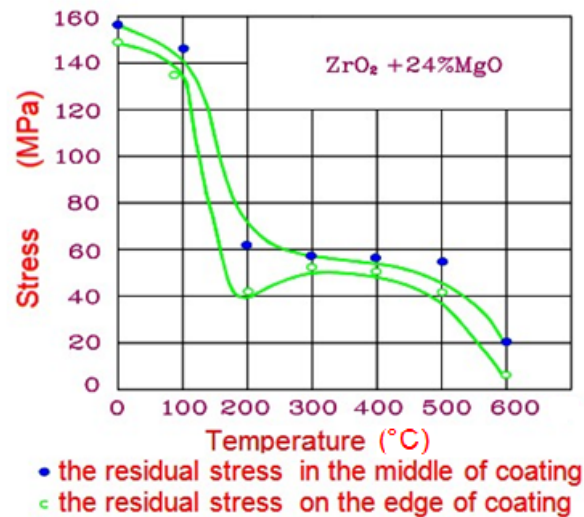
Figure 9 – Effect of coating heat treatment on residual stresses
Рис. 9 – Влияние термической обработки покрытия на остаточные напряжения
Слика 9 – Утицај термичке обраде превлака на заостале напоне

The thermal treatment of coatings at 100°C; 200°C; 300°C; 400°C; 500°C and 600°C for a period of 2h, followed by coating cooling in the furnace to room temperature, brought about a significant reduction in residual stresses. Residual stresses in the coating decrease with the increase in the heat treatment temperature. The curves of residual stresses in the middle and at the edge of the sample are identical, indicating a more uniform stress distribution in the middle and at the edge of the coating after heat treatment.

Conclusion

The paper presents the modeling and prediction of residual stresses in the plasma sprayed ceramic ZrO_2CaO coating as well as the analysis of the stress state in the ZrO_2MgO coating. The analysis found out that the residual stresses in the ZrO_2CaO coating are significantly affected by the deposition speed. For low powder deposition speed values, negative compression stresses are present in coating layers and they change the sign into positive tensile stresses with the increase of the deposition speed. The increase of the coating thickness over 1mm results in

residual stresses changing values and the sign from compression to tensile. The increase in the coating thermal conductivity leads to an increase in the value of negative compressive stresses, which can cause cracks in the coating during coating deposition. The bond coating in a combination with the ceramic ZrO_2MgO coating partially reduces residual stresses in a two-layered coating system, which is why the coating has a lower share of residual tensile stresses compared to deposited coatings from pure ceramics. The highest stresses are on the surface of the coating. For up to 5mm-thick substrates, stresses on the surface and the edge of the coating are negative compression stresses whose value decreases with the decrease of the coating thickness. With the reduction of the coating thickness to 0.1 mm, stress changes the sign and negative compression stresses change into positive tesile stresses. The highest value of positive tensile stresses is found in the coating on the interface with the substrate. Significant reduction of residual stresses in coatings is achieved by heat treatment. The increase in the heat treatment temperature leads to the reduction of the residual stresses in the coating and their distribution is more uniform in the middle and at the edge of the sample.

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АНАЛИЗ ОСТАТОЧНЫХ НАПРЯЖЕНИЙ В БИОИНЕРТНЫХ НЕОРГАНИЧЕСКИХ КЕРАМИЧЕСКИХ ПОКРЫТИЯХ, НАНЕСЕННЫХ ПЛАЗМЕННЫМ НАПЫЛЕНИЕМ

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Резюме:

Один из факторов успешного применения биомедицинской керамики в области покрытия имплантов заключается в соответствии физических и механических характеристик покрытия с металлической основой импланта. Весьма важную роль при нанесении порошка играют температура и

температурный градиент покрытий, так как от них в большей мере зависит конечное качество покрытия. Коэффициенты термического расширения и теплопроводности покрытия и основания различаются, а это влияет на увеличение остаточных напряжений в покрытиях. Для минимизирования разницы физических и механических характеристик покрытия и основания, необходимо вести постоянный контроль за температурами покрытия и основания в течение всего процесса нанесения порошка и его осаждения. В целях продления срока службы как покрытия, так и самого импланта необходимо обеспечить соответствующий контроль за остаточными напряжениями в керамических покрытиях. В данной статье представлена модель теплообмена плазмы, с предусмотренным распределением остаточных напряжений в нанесенных покрытиях, а также техника рентгенографии для измерения остаточных напряжений в керамических покрытиях. Целью данной статьи было описание эффекта скорости осаждения порошка, изменения толщины и теплопроводности ZrO$_2$CaO покрытий к уровню и значениям остаточных напряжений. В статье также представлено каким образом связующие покрытия, изменения толщины связующих и керамических покрытий ZrO$_2$MgO и термическая обработка влияют на уровень и значения остаточных напряжений. Выявлено, что при увеличении общей толщины покрытия пропорционально увеличивается доля остаточного напряжения на поверхности и краях покрытия.

Ключевые слова: основания, напряжения, покрытия, керамика.

 АНАЛИЗА ЗАОСТАЛИХ НАПОНА У БИОИНЕРТНИМ НЕОРГАНСКИМ КЕРАМИЧКИМ ПЛАЗМА СПРЕЈ ПРЕВЛАКАМА

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Сажетак:

Један од фактора за успешну примену биомедицинских керамичких превлака на имплантима јесте усклађеност физичких и механичких карактеристика превлака са металним подлогама импланата. Температуре и температурни градијент у превлакама током депозиције праха имају важну улогу на коначни квалитет превлака. Коефицијенти топлотног ширења и топлотне проводљивости превлаке и подлоге се разликују, што утиче на повећање заосталих напона у превлакама. Да би се разлика физичких карактеристика превлаке и подлоге свела на минимум,

температуре површине превлаке и подлоге морају се држати под контролом током депозиције праха. Због тога је од посебног значаја контрола заосталих напона у керамичким превлакама уколико се жели постићи корисни век превлаке и импланта. У раду је описан модел преноса топлоте плазме са предвиђањем расподеле заосталих напона у депонованим превлакама и техника рендгенографије за мерење заосталих напона у керамичким превлакама. Циљ рада јесте да се опише ефекат брзине депозиције праха, промене дебљине и топлотне проводљивости ZrO_2CaO превлаке на ниво и предзнак заосталих напона. Приказан је и утицај везне превлаке, промена дебљине везне и керамичке превлаке ZrO_2MgO и термичке обраде на ниво и предзнак заосталих напона. Установљено је да се са повећањем укупне дебљине превлака повећава удео заосталих напона на површини и ивицама превлака.

Кључне речи: подлоге, напони, превлаке, керамике.