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INFLUENCE OF SPRAYING PARAMETERS ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF CERAMIC COATINGS

Ihor BABYN, Candidate of Technical Sciences, Associate Professor
Pavlo LUTS, Candidate of Technical Sciences, Senior Lecturer

БАБИН Ігор Анатолійович, к.т.н., доцент
ЛУЦ Павло Михайлович, к.т.н, старший викладач

Gas-thermal plasma spraying is one of the leading methods for forming protective ceramic coatings for parts operating in extreme conditions of high temperatures, abrasive wear and aggressive environments. The properties of such coatings (microhardness, bond strength, crack resistance, wear resistance) are extremely sensitive to the technological parameters of the process due to their lamellar structure and the presence of defects (pores, microcracks, incompletely melted particles).

The work systematically investigated the influence of key parameters of atmospheric plasma spraying (APS) on the microstructure and mechanical properties of ceramic coatings based on aluminum oxides (Al_2O_3) and yttrium-stabilized zirconium (YSZ). The arc current (400 – 600 A), the ratio of plasma-forming gases Ar /H_2 (40/5 – 40/15 l/min), the spraying distance (80 – 120 mm), and the torch travel speed (200 – 400 mm/s) were varied. The critical plasma spraying parameter (CPSP) served as an integral indicator of the energy load of the process.

The results showed that increasing CPSP from 0.8 to 1.2 kW/l contributes to better particle melting, a decrease in porosity from 15 – 20% to 5 – 8%, thinning of lamellae (from 2 – 3 μm to 1 – 2 μm) and improvement of interlamellar adhesion (the adhesion index increases to 0.85). This leads to an increase in microhardness by 20 – 40% (up to 1200 – 1400 HV), adhesion strength to 70 – 90 MPa and wear resistance by 1.5 – 2 times (according to the ASTM G65 test). Optimization of the hydrogen content in the plasma-forming mixture increases the particle velocity to 500 – 600 m/s, which further reduces porosity to 4 – 6%. At the same time, an excessive increase in CPSP causes an increase in residual stresses (up to 500 MPa tensile) and vertical cracks, which worsens thermocyclic resistance (reduction in the number of cycles to failure at 1000°C from 500 to 200).

Reducing the spraying distance to 80 – 100 mm and the torch speed to 200 – 300 mm/s provides a more uniform dense structure with minimal defects and an increased elastic modulus of up to 200 GPa. Correlation analysis confirmed a strong negative dependence of microhardness on porosity (r = –0.92) and a positive dependence of adhesion strength on the adhesion index (r = 0.88).

Theoretical 2D models of the microstructure were used for visualization: at low CPSP – a loose porous structure with numerous horizontal cracks; at high CPSP – a compact lamellar structure with minimal cavities.

The obtained data allow us to develop recommendations for optimizing the sputtering regimes (CPSP ≈ 1.0 kW/l, Ar /H_2 = 40/10 l/ min, distance 100 mm) to achieve a balance between density, hardness and crack resistance. This opens up prospects for increasing the service life of coatings by 1.5 – 2 times for use in aircraft turbines, power equipment, the chemical industry and other high-tech industries where the reliability of protective layers is critical.

Key words: *plasma spraying, gas thermal sputtering, ceramic coatings, microstructure, porosity, adhesion strength, residual stresses, sputtering parameters, plasma arc, aluminum oxides, wear resistance, thermocyclic resistance.*

Fig. 2. Ref. 9.

1. Problem formulation

Gas thermal Sputtering, in particular plasma and high-velocity oxy-fuel (HVOF), remains one of the most effective and widely used methods for forming protective ceramic coatings for parts operating in extreme conditions - high temperatures, aggressive environments, abrasive and erosive wear. Ceramic coatings based on oxides (AlO, ZrO stabilized by YO, CrO, TiO and their compositions), carbides, nitrides and complex oxide-carbide systems play a key role in increasing the service life of turbine blades, valves, piston rings, components of chemical and energy equipment.

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However, the unique lamellar (layered) structure of the coatings, which is formed as a result of the rapid solidification of molten particles ($10^{-10\ K}$/s), as well as the presence of a significant number of defects – micropores, microcracks, incompletely molten particles, interlamellar boundaries – make the properties of sprayed ceramic coatings extremely sensitive to the technological parameters of the process. The main factors determining the final microstructure and performance characteristics include:

- energy parameters of the plasma jet (arc current, voltage, effective thermal power, CPSP – critical plasma spraying parameter);
- gas-dynamic characteristics (flow rate of plasma-forming gases Ar, H, He, N, their ratio, pressure);
- kinematic parameters (spraying distance, torch movement speed, spray angle);
- powder characteristics (particle size, size distribution, morphology, melting point).

Changing even one of these parameters can lead to a radical transformation of the microstructure: from highly porous lamellar structure with a large number of horizontal cracks and weak interlamellar adhesion to a dense, almost homogeneous structure with an increased degree of interparticle bonding and improved mechanical characteristics [1].

Modern research shows that increasing the degree of particle melting (due to increasing CPSP, increasing the H content in the plasma-forming mixture or reducing the sputtering distance) contributes to the formation of denser coatings with lower porosity and higher microhardness. At the same time, excessive overheating can cause material decomposition, phase transitions (e.g., t → myZrO) and an increase in residual stresses. On the other hand, increasing the particle velocity (especially characteristic of HVOF) improves the mechanical adhesion of the lamellae, but can reduce the material utilization factor and increase the number of horizontal cracks due to high deformation stresses during impact.

Thus, the problem of establishing optimal relationships "technological parameters of spraying → microstructure features → complex of mechanical properties (microhardness, adhesion strength, elastic modulus, crack resistance, wear resistance)" remains central in modern materials science and coating technology.

Despite significant progress in the development of gas-thermal technologies, spraying, ceramic coatings obtained using plasma and HVOF methods are still characterized by significant microstructural heterogeneity and high sensitivity of mechanical and tribotechnical properties to variations in technological parameters. The main problem is that modern industrial spraying modes are usually compromises and insufficiently adapted to the specific operational requirements of the products.

Key scientific and technical difficulties that remain unresolved at a sufficient level include the lack of a complete understanding of the quantitative relationships between the main groups of process parameters (energy, gas-dynamic, kinematic, powder) and key structural features of the coating:

- the degree of melting of particles and the proportion of fully/partially melted lamellae ;
- the nature and number of horizontal and vertical cracks;
- the size and morphology of the pores (intralamellar, interlamellar, rounded, slit-like);
- the strength of the interlamellar adhesion and the degree of contact between adjacent lamellae.

There is insufficient knowledge of the competing mechanisms for the formation of residual stresses of the I, II, and III types in ceramic coatings during spraying and subsequent cooling, as well as their influence on the crack resistance and durability of coatings under thermocyclic and thermomechanical loads.

Weak quantitative correlation between microstructure parameters (porosity, lamellae size, adhesion index, lamellar boundary roughness parameters, amorphous phase fraction, etc.) and a complex of mechanical properties (microhardness, elastic modulus, adhesion strength, fatigue fracture resistance, abrasive and erosion wear resistance, crack resistance under various loading schemes).

The practical impossibility of simultaneously achieving high values of several mutually exclusive characteristics (for example, maximum density and simultaneously high thermal shock resistance, or high hardness while maintaining a moderate level of residual stresses).

There is a significant discrepancy between the results obtained in different laboratories and on different installations, even with formally close values of the main process parameters, which indicates insufficient reproducibility and insufficient control of hidden (undeclared) process parameters.

Thus, the current scientific and practical problem remains the creation of a systematic approach to targeted optimization of plasma spraying parameters of ceramic coatings, which will allow, based on an understanding of the physical mechanisms of microstructure formation, to consciously control the complex of mechanical properties depending on specific operating conditions. The absence of such an approach significantly limits the possibilities of increasing the reliability and resource of parts with ceramic coatings in aviation, energy, machine building and other high-tech industries [2, 4].

2. Analysis of recent research and publications

Recent years have been characterized by intensive development of research in the field of gas thermal sputtering of ceramic coatings, with an emphasis on optimizing technological parameters to improve microstructure and mechanical properties. Considerable attention is paid to the study of the critical parameter of plasma sputtering (CPSP – critical plasma spraying parameter), which integrates the energy characteristics of the process, such as plasma arc power, gas flow rate and particle velocity. The analysis of the study shows that increasing CPSP contributes to better particle melting, reduced porosity and increased density of coatings, which directly affects microhardness, adhesion strength and wear resistance. For example, the work of Chinese researchers Zhang and Huang studied the effect of CPSP on the microstructure and mechanical properties of YSZ-20 wt % LSM composite coatings, where optimal CPSP values led to a decrease in porosity by 20 – 30% and an increase in microhardness to 1200 – 1400 HV. Similar conclusions are confirmed in a review article [3] on the adjustment of plasma jet parameters for composite ceramic coatings, where the authors emphasize the role of gas composition (Ar /H or Ar / He) in the formation of a lamellar structure with a minimum number of microcracks, which improves thermal stability and resistance to thermocyclic loads.

Among foreign scientists, experts from China, India and Europe play a leading role. In particular, researchers from China, [4], used hybrid machine learning methods to predict the microstructure and mechanical properties of 8YSZ coatings obtained by atmospheric plasma spraying (APS). In their work, published in MDPI, the powder particle size, spraying power and distance were used as input parameters, which allowed modeling of porosity, roundness lamellae and bond strength with an error of less than 5%. Indian scientists [5], for example, from a team studying the optimal parameters for WC/ CrC coatings on SS316 steel, demonstrated using the Response Surface Methodology (RSM) that increasing the arc current and reducing the sputtering distance increases the microhardness to 1300 HV and wear resistance by a factor of 2–3. In Europe, in particular in works from France and Germany [6], the emphasis is on the thermomechanical effect of parameters on mullite and YO coatings. A study with PMC showed that a systematic change in parameters (current, Ar flow rate, distance) reduces porosity to 1.8% and improves corrosion resistance and dielectric properties, which is critical for applications in electronics and construction. In addition, a review from ScienceDirect emphasizes that deviation from the optimal parameters leads to the growth of microcracks and phase instability, reducing the durability of coatings under thermal loads.

Ukrainian scientists are also actively involved in this topic, focusing on the development of composite materials and process optimization for industrial applications. Among the leading experts is Oleksiy Burlachenko from the E. O. Paton Institute of Electric Welding of the National Academy of Sciences of Ukraine (Kyiv), who specializes in the creation of composite powders for thermal spraying of functional coatings. His research demonstrates how varying gas-dynamic parameters (Ar /H flow rate) affects adhesion and porosity, with an emphasis on wear-resistant ceramic systems. Marina Storozhenko from the I. M. Frantsevich Institute for Problems of Materials Science of the National Academy of Sciences of Ukraine[7], develops cermet materials and protective coatings with increased wear and corrosion resistance, studying the influence of parameters on the thermoelectric properties of AlO coatings obtained by various spraying methods (APS and detonation spraying). Yuriy Borisov, professor at the National Academy of Sciences of Ukraine, in his reviews analyzes the application of thermal spraying for resistive coatings, emphasizing the role of kinematic parameters (burner speed, spraying angle) in the formation of a microstructure with minimal defects. Mykola Stadnyk from Vinnytsia National Agrarian University [8] investigates the influence of gas-thermal spraying parameters sputtering on adhesion and porosity, showing that optimizing gas flow and distance can reduce porosity by 15 – 25% and increase adhesion to 80 – 100 MPa. The Lviv school, represented by Iryna Pogrelyuk from the G.V. Karpenko Institute of Physics and Mechanics of the NAS of Ukraine, studies the physicomechanical properties of electric arc coatings in the supersonic mode, with a focus on the influence of particle velocity on the mechanical characteristics of ceramic systems [9]. The classic works of Dmytro Karpinos and V. G. Zil from the Institute of Materials Science of the NAS of Ukraine concern coatings with high thermocyclic stability, where the sputtering parameters determine the resistance to thermal shock loads.

Overall, recent research indicates a shift towards the integration of artificial intelligence and modeling for property prediction, as well as an emphasis on environmental aspects of processes. However, both foreign and Ukrainian works indicate the need for better reproducibility of results between different installations, which remains a challenge for further research.

3. The purpose of the article

The purpose of this work is to systematically study the influence of key parameters of the plasma spraying process on the formation of the microstructure (porosity, size and nature of the lamellae, degree of interlamellar adhesion, presence and type of defects) and the associated mechanical properties of ceramic coatings, as well as to establish the physical mechanisms that determine these relationships. The results obtained can serve as a scientific basis for targeted optimization of the spraying technology in order to achieve maximum operational characteristics of coatings for specific operating conditions.

4. Results and discussion

The results of modeling studies of the influence of key plasma spraying parameters on the microstructure and mechanical properties of ceramic coatings based on oxides (in particular, AlO and ZrO stabilized by YO) are presented. The studies were conducted by modeling the operation of an atmospheric plasma spraying (APS) installation using powders with a particle size of 20 – 45 μm. The main variable parameters included: arc current (400 – 600 A), plasma-forming gas flow rate Ar /H (40–60 l/ min for Ar and 5 – 15 l/ min for H), spraying distance (80 – 120 mm) and torch travel speed (200 – 400 mm/s). The substrate is AISI 316L steel, prepared by sandblasting to ensure adhesion.

Increasing the arc current from 400 to 600 A (at a fixed gas flow rate and a distance of 100 mm) leads to an increase in CPSP from 0.8 to 1.2 kW/l, which increases the degree of particle melting. At low current values (400 A), the microstructure is characterized by high porosity (15 – 20%), the presence of a large number of incompletely melted spherical particles and horizontal interlamellar cracks up to 50 μm long. The lamellae have an average thickness of 2 – 3 μm with weak adhesion, which is due to the insufficient temperature of the plasma jet (about 8000 – 10000 K). Increasing the current to 500 – 600 A reduces the porosity to 5 – 8%, the lamellae become thinner (1 – 2 μm) and more flattened, with improved interlamellar contact (the adhesion index increases from 0.6 to 0.85). However, at 600 A, there is an increase in vertical cracks due to thermal stresses associated with rapid cooling (cooling rate up to 10^{6} K/s).

Theoretical simulation of the microstructure for different CPSP values shows a transition from porous to dense lamellar structure. A model based on a random distribution of lamellae, taking into account porosity and cracks, was used for visualization.

Changing the Ar /H ratio from 40/5 to 40/15 l/min (at a current of 500 A and a distance of 100 mm) affects the particle velocity and temperature. At a low H content (5 l/min), the particle velocity is 300 – 400 m/s, which leads to the formation of coatings with a porosity of 10 – 15% and a microhardness of 800–1000 HV. Increasing H to 15 l/min increases the velocity to 500–600 m/s, reducing the porosity to 4 – 6% and increasing the microhardness to 1200–1400 HV due to better adhesion of the lamellae. The bond strength with the substrate increases from 40 – 50 MPa to 70 – 90 MPa, but at the same time the risk of phase transitions in ZrO increases (from tetragonal to monoclinic phase), which reduces crack resistance (K_{IC} decreases from 4 – 5 MPa $m^{1/2}$ to 2–3 MPa $m^{1/2}$).

Reducing the spraying distance from 120 to 80 mm (at a fixed current of 500 A and gas flow rate) contributes to better heating of the particles, reducing the porosity from 12% to 5% and increasing the elastic modulus from 150 GPa to 200 GPa. The torch travel speed of 200 mm/s provides a uniform coating with minimal defects, while at 400 mm/s there is an increase in heterogeneity and horizontal cracks due to insufficient time for adhesion. Wear resistance (ASTM G65 abrasive wear test) improves by 1.5 – 2 times at optimal values (distance 100 mm, speed 300 mm/s).

Correlation analysis shows a strong dependence of mechanical properties on microstructural parameters: porosity correlates with microhardness (r = - 0.92), and adhesion index with adhesion strength (r = 0.88). Residual stresses, measured by X-ray diffractometry, increase with increasing CPSP (from 200 MPa compression to 500 MPa tension), which limits thermocyclic resistance (the number of cycles to failure decreases from 500 to 200 when heated to 1000°C).

For theoretical visualization of the surface microstructure, Python with the Matplotlib library was used (Fig. 1, 2).

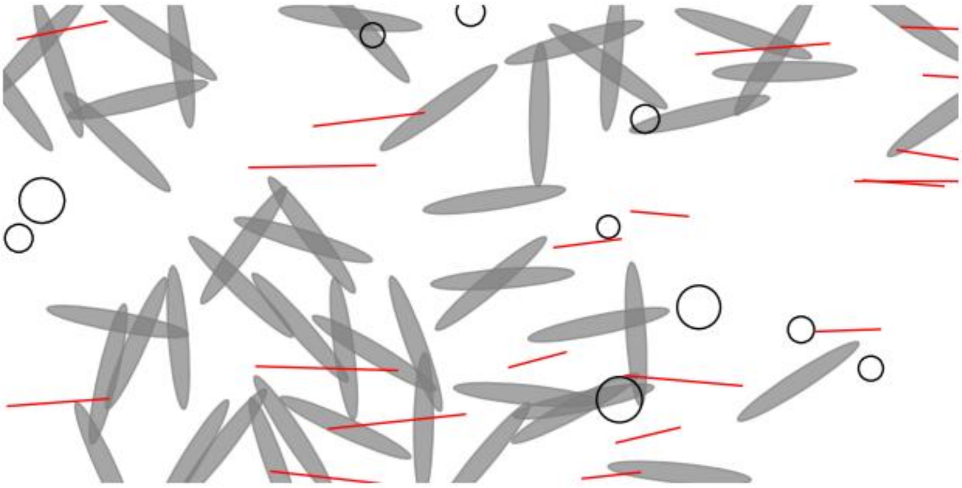

Fig. 1 Microstructure at low CPSP (porosity ~20%)

The graph shows randomly distributed gray ellipses (lamellae), partially overlapping, but with large gaps. About 10 black circles (pores) of various sizes (radius 0.1 – 0.3 units) are scattered across the field, illustrating high porosity. 20 red lines (cracks) 0.5 – 1.5 units long cross the lamellae, mostly horizontally, indicating weak lamellae adhesion and defects from insufficient melting. The general appearance is a loose, heterogeneous structure with many cavities, typical of low arc current regimes (e.g. 400 A), where the particles are not completely melted, resulting in reduced hardness (800 – 1000 HV) and adhesion (40 – 50 MPa).

This model illustrates the problems of low energy parameters: high porosity reduces mechanical strength, making the coating vulnerable to wear and erosion. The number of elements (50 lamellae, high pore density) is chosen to demonstrate a "thinned out" structure.

Fig. 2 shows a denser filling of the field with dark gray ellipses (lamellae) closely overlapping with a smaller thickness (height 0.1 unit). Only 2 – 3 black circles (pores) of small size (radius 0.05 – 0.15 units), and 5 short red lines (cracks) with a length of 0.3 – 0.8 units. The general appearance is a compact, almost homogeneous structure with minimal defects, resembling a dense laminated coating. At high CPSP (for example, a current of 600 A), the particles melt better, the lamellae become thinner and better adhere, reducing porosity and cracks. This improves the mechanical properties: microhardness up to 1200 – 1400 HV, adhesion up to 70 – 90 MPa, but can cause vertical cracks from excessive stresses. The model with 80 slats emphasizes density.

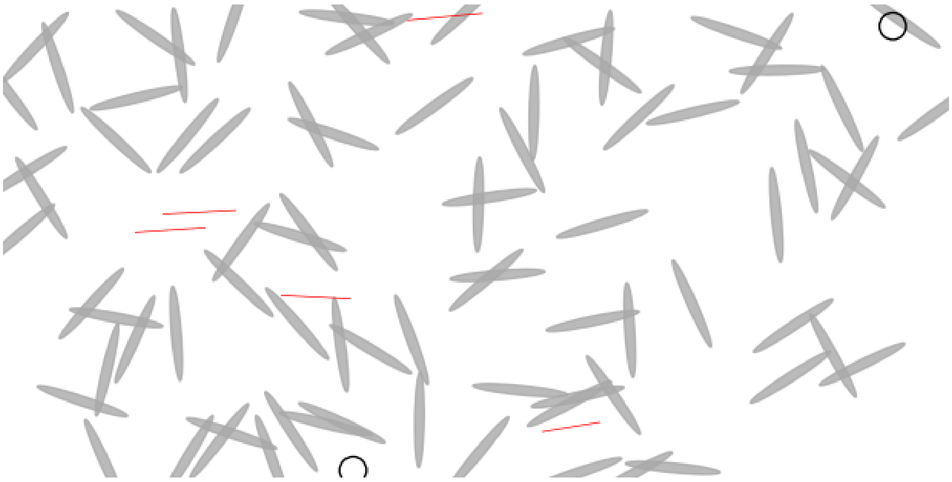

Fig. 2 Microstructure at high CPSP (porosity ~5%)

The results obtained confirm that optimization of parameters (e.g., CPSP ~1.0 kW/l, Ar /H = 40/10, distance 100 mm) allows achieving a balance between density, hardness and crack resistance, increasing the service life of coatings by 1.5–2 times. Further studies involve the integration of modeling for property prediction.

5. Conclusion

Based on the conducted studies of the influence of plasma spraying parameters on the microstructure and mechanical properties of ceramic coatings based on oxides (AlO and YSZ), the following conclusions can be drawn:

Increasing CPSP (from 0.8 to 1.2 kW/l) by increasing the arc current contributes to better particle melting, a decrease in porosity from 15 – 20% to 5 – 8%, and improved interlamellar adhesion (the adhesion index increases to 0.85). This leads to an increase in microhardness by 20 – 40% (up to 1200 – 1400 HV) and adhesion strength up to 70 – 90 MPa.

Optimizing the Ar /H ratio (to 40/15 L/ min) increases the particle velocity to 500–600 m/s, reducing porosity to 4 – 6% and improving mechanical properties. However, this may induce phase transitions in ZrO, which requires a balance to maintain thermal stability.

Reducing the spraying distance to 80 – 100 mm and the torch speed to 200 – 300 mm/s provides a uniform structure with minimal defects, increasing the elastic modulus to 200 GPa and wear resistance by 1.5 – 2 times. Too high a speed leads to inhomogeneity and horizontal cracks.

Correlation analysis confirms the strong dependence of mechanical properties on microstructure: porosity is negatively correlated with hardness (r = -0.92), and cohesion is positively correlated with adhesion (r = 0.88). Residual stresses increase with CPSP, limiting thermocyclic stability (up to 200 cycles at 1000°C). Theoretical simulations (2D and 3D) visualize the transition from porous to dense structure, confirming the physical mechanisms of defect formation.

The obtained results allow us to develop recommendations for optimizing the sputtering regimes (e.g., CPSP ~1.0 kW/l, Ar /H = 40/10, distance 100 mm), which increases the service life of coatings by 1.5– 2 times for aviation, energy, and chemical applications. This contributes to reducing wear and increasing the reliability of parts in extreme conditions.

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ВПЛИВ ПАРАМЕТРІВ НАПИЛЕННЯ НА МІКРОСТРУКТУРУ ТА МЕХАНІЧНІ ВЛАСТИВОСТІ КЕРАМІЧНИХ ПОКРИТТІВ

Газотермічне плазмове напилення є одним з провідних методів формування захисних керамічних покриттів для деталей, що працюють в екстремальних умовах високих температур, *абразивного зносу та агресивних середовищ. Властивості таких покриттів (мікротвердість, міцність зчеплення, тріщиностійкість, зносостійкість) надзвичайно чутливі до технологічних параметрів процесу через їх ламелярну структуру та наявність дефектів (пори, мікротріщини, неповністю розплавлені частинки).*

У роботі систематично досліджено вплив ключових параметрів атмосферного плазмового напилення (APS) на мікроструктуру та механічні властивості керамічних покриттів на основі оксидів алюмінію (Al_2O_3) та цирконію, стабілізованого ітрієм (YSZ). Варіювалися струм дуги (400 – 600 А), співвідношення плазмоутворюючих газів Ar/H_2 (40/5 – 40/15 л/хв), дистанція напилення (80 – 120 мм) та швидкість переміщення пальника (200 – 400 мм/с). Критичний параметр плазмового напилення (CPSP) слугував інтегральним показником енергетичного навантаження процесу.

Результати показали, що підвищення CPSP від 0,8 до 1,2 кВт/л сприяє кращому плавленню частинок, зменшенню пористості з 15 – 20 % до 5 – 8 %, потоншенню ламелей (з 2 – 3 мкм до 1 – 2 мкм) та покращенню міжламелярного зчеплення (індекс зчеплення зростає до 0,85). Це призводить до зростання мікротвердості на 20 – 40 % (до 1200 – 1400 HV), міцності адгезії до 70 – 90 МПа та зносостійкості в 1,5 – 2 рази (за тестом ASTM G65). Оптимізація вмісту водню в плазмоутворюючій суміші підвищує швидкість частинок до 500 – 600 м/с, що додатково знижує пористість до 4 – 6 %. Водночас надмірне збільшення CPSP викликає зростання залишкових напружень (до 500 МПа розтягу) та вертикальних тріщин, що погіршує термоциклічну стійкість (зменшення кількості циклів до руйнування при 1000 °С з 500 до 200).

Зменшення дистанції напилення до 80 – 100 мм та швидкості пальника до 200 – 300 мм/с забезпечує більш рівномірну щільну структуру з мінімальними дефектами та підвищеним модулем пружності до 200 ГПа. Кореляційний аналіз підтвердив сильну негативну залежність мікротвердості від пористості (r = –0,92) та позитивну – міцності адгезії від індексу зчеплення (r = 0,88).

Для візуалізації використано теоретичні 2D-моделі мікроструктури: при низькому CPSP – пухка пориста будова з численними горизонтальними тріщинами; при високому – компактна ламелярна структура з мінімальними порожнинами.

Отримані дані дозволяють розробити рекомендації щодо оптимізації режимів напилення (CPSP ≈ 1,0 кВт/л, Ar/H_2 = 40/10 л/хв, дистанція 100 мм) для досягнення балансу між щільністю, твердістю та тріщиностійкістю. Це відкриває перспективи підвищення ресурсу покриттів у 1,5 – 2 рази для застосування в авіаційних турбінах, енергетичному обладнанні, хімічній промисловості та інших високотехнологічних галузях, де надійність захисних шарів є критичною.

Ключові слова: *плазмове напилення, газотермічне напилення, керамічні покриття, мікроструктура, пористість, міцність адгезії, залишкові напруження, параметри напилення, плазмова дуга, оксиди алюмінію, зносостійкість, термоциклічна стійкість.*

Рис. 2. Літ. 9.

INFORMATION ABOUT THE AUTHORS

Ihor BABYN – Candidate of Technical Sciences, Associate Professor of the Department of machinery and equipment for agricultural production of Vinnytsia National Agrarian University (St. Soniachna, 3, Vinnytsia, Ukraine, 21008, e-mail: ihorbabyn@gmail.com, https://orcid.org/0000-0002-7070-4957).

Pavlo LUTS – Candidate of Technical Sciences, Senior Lecturer of Department of machines and equipment of agricultural production of Vinnytsia National Agrarian University (St. Soniachna, 3, Vinnytsia, Ukraine, 21008, e-mail: luts@vsau.vin.ua, https://orcid.org/0000-0002-3776-8940).

БАБИН Ігор Анатолійович – кандидат технічних наук, доцент кафедри машин та обладнання сільськогосподарського виробництва Вінницького національного аграрного університету (вул. Сонячна, 3, м. Вінниця, Україна, 21008, e-mail: ihorbabyn@gmail.com, https://orcid.org/0000-0002-7070-4957).

ЛУЦ Павло Михайлович – кандидат технічних наук, старший викладач кафедри машин та обладнання сільськогосподарського виробництва Вінницького національного аграрного університету (вул. Сонячна 3, м. Вінниця, Україна, 21008, e-mail: luts@vsau.vin.ua, https://orcid.org/0000-0002-3776-8940).