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D. JANKURA*, D. DRAGANOVSKÁ*

ADHESION PROPERTIES OF SYSTEMS ON A COMPOSITE COATINGS BASIS – THE STEEL SUBSTRATE

ADHEZJA POWŁOK KOMPOZYTOWYCH NA PODŁOŻU STALOWYM

The paper deals with the assessment of systems properties on the heating coat basis – the steel substrate in terms of the adhesion and coatings structure. On S235JRG1 steel substrate the ceramic and composite coatings on the Al_2O_3 basis were made for research purpose by the technology of plasma heating spraying with a water stabilized arc. The content effect of Ni metallic component to the base Al_2O_3 powder was examined on the adhesion and composite coatings structure. Adhesion tests were examined in terms of STN EN 582 (Thermal spraying. Determination of tensile adhesive strength). Coatings were further evaluated by a hardness measurement, chemical and microscopic analysis. Adhesion tests proved the Ni metallic powder addition markedly increases the adhesion of composite heating sprayed coatings and allows their creation without an interlayer utilization. The composite coating contains fewer defects in term of pores and cavities occurrence and rifts in the first place which are caused by dilatation strains during a setting process.
Keywords: plasma spraying, coating, ceramics, composite coating, adhesive properties

Praca dotyczy oceny właściwości płaszcza grzewczego po względem przyczepności powłok do stalowego podłoża i ich struktury. Do celów badawczych na stalowym podłożu S235JRG1 metodą natryskiwania plazmowego wykonane zostały ceramiczne i kompozytowe powłoki na bazie Al_2O_3. Badano wpływ zawartości metalicznego niklu do bazowego proszku Al_2O_3 na przyczepność powłoki kompozytowej i jej strukturę. Badania adhezji wykonano zgodnie z normą EN 582 (Natryskiwanie cieplne – określanie przyczepności metodą odrywania). Powłoki były następnie poddane pomiarom twardości, analizie chemicznej i mikroskopowej. Badania adhezji dowiodły, że dodanie metalicznego proszku Ni znacznie zwiększa adhezję kompozytowych powłok i umożliwia ich tworzenie bez warstwy pośredniej. Powłoka kompozytowa ma mniej wad: porów, ubytków i szczelin, które powodowane są przez naprężenia objętościowe w trakcie procesu osadzania.

1. Introduction

Nowadays, thermal spraying technologies bring new possibilities into the production and renovation technology. Those technologies find a wide range of application in many of top industries such as in the area of machine industry, power industry, aircraft and automobile industry, chemistry and electrical technology. The aim is to increase the surface properties quality of individual components.

The plasma technology spraying comes into the front out of various ways of thermal spraying coatings. Plasma spraying technology practically allows lying on all technologically utilizable materials with suitable properties on both metallic and non-metallic substrates. Metallic, metallic-ceramic and ceramic coatings are applied on. Ceramic materials form the predominant part of additional coating materials on a ceramic basis of which Al_2O_3 is the dominant part. Other oxides (Cr_2O_3, $ZrSiO_4$, ZrO_2, MgO, CaO, HfO_3, and MoO2) are on the other positions and are applied either in a pure state or with various dopants and other chemical compounds (nitrides, borides, etc.) [1,2].

Ceramic coatings are characterized by specific properties such as high hardness, wear resistance, resistance against corrosion, temperature, heat etc. They allow optimally adjust components surface properties to operational conditions. It leads to the operational life extension and components reliability increase. Basic regularities of the plasma sprayed coatings production are stated in the thesis [3,4,5].

One of the most important factors which decide the coatings properties and operational life is their adhesion – an adhesive capacity to the basic substrate. By various means it is possible to achieve a high adhesion assurance. Besides the type of a coating application technology, substrate surface properties it is also the coating type and composition [6,7].

The contribution is focused on the possibilities of composite coatings production on the basis (Al_2O_3) with the metallic nickel component addition. The impact of metallic component content on coatings adhesion properties was determined. The coating production mechanism at coatings metallographic study was examined from the production aspects of the own layer, its bond to the basic substrate and the bond between the individual coating elements.

* DEPARTMENT OF TECHNOLOGY AND MATERIALS, FACULTY OF MECHANICAL ENGINEERING, TECHNICAL UNIVERSITY OF KOŠICE, MÄSIARSKA 74, 040 01 KOŠICE, SLOVAKIA

2. Materials and experimental methods

The examined coatings were applied on the front surfaces of the steel rollers with 25 mm diameter from S235JRG1 material. The powders were applied on steel samples with size 100×50×5 mm for the coatings structure study. Coatings' spraying was realized on a plasma device with water arc stabilization and the device producer is ÚFP Praha. Plazmathron stabilized by water has higher intensity of spraying (in $kg \cdot kW^{-1} \cdot h^{-1}$) compared to the basic one, devices stabilized by gas. It also has markedly (approximately two times) bigger plasma temperature (30 000 degrees of Celsius). Therefore the device is suitable for highly-meltable ceramic materials application and coatings production on larger surfaces, when producing the coatings with a bigger thickness or creation of self-supporting frameworks. For the plasma spraying with water plasma stabilization a plasma gas is generated from the liquid, i.e. water, but also from ethyl or methyl alcohol [10].

Realized experimental works were focused on the adhesion properties research, composite coatings composition and structure on the Al_2O_3 basis with metallic component addition with K 30 industry marking of which structure is stated in Table 1. Its fusion point is 1050°C and is used for technological process of various components hard surfacing from the steel and cast iron. Powder granularity given by the producer and confirmed by a sieve analysis was 40-90 μm.

TABLE 1

Chemical metallic powder composition – K 30 [%]

C	Si	B	Fe	Cr	Ni
max 0.1	max 3.5	max 2.5	max 0.5	max 2.5	residue

For the research the composite powders with the following volumetric proportion of components were prepared: Al_2O_3+ 5% K 30, Al_2O_3+ 12% K30 and Al_2O_3+ 30% K30. The pure ceramic coating Al_2O_3 was applied on the substrate without and with the BP NiCr interlayer for the properties comparison of the composite coatings. Its composition is stated in Table 2. The granularity listed by the producer is 45-90 μm.

TABLE 2

Chemical metallic powder composition – BP NiCr [%]

C	Si	Mn	Fe	Cr	Ni
max 0.25	max 1.5	max 2.5	max 1	18-22	residue

For the research the ceramic and composite coatings with the following marking were used – they are stated in Table 3.

Basic substrate surface was pre-modified by mechanical blasting with the help of a projectile wheel before the single spray application. On the basis of the initial knowledge a sharp-edged instrument – a corundum crushed material with a grain size d_z = 1 – 1.2 mm was chosen. The grain fly speed in the blasting process v = 80 $m \cdot s^{-1}$. Blasting was made on laboratorial blasting device of Di-2 type [8,9].

TABLE 3

Identification of the investigated coatings

Signature	Coating type
A0	Ceramic coating Al_2O_3 without interlayer
AM	Interlayer BP NiCr + ceramic coating Al_2O_3
A5K	Composite coating on the basis Al_2O_3+ 5% K30 (volumetric %)
A12K	Composite coating on the basis Al_2O_3+ 12% K30 (volumetric %)
A20K	Composite coating on the basis Al_2O_3+ 12% K30 (volumetric %)

Adhesion tests were realized on tensile test machine ZD 10 in compliance with the STN EN 582 Standart - Thermal spraying. Determination of tensile adhesive strength. The sample with the applied coating is glued together with a companion part (Fig. 1) and with the help of a fixative device clamps into bites of test device and is ballasted by the pull with a constantly increasing power till a joint interruption. The adhesion is determined as a stress perpendicular to the surface of basic material, needed to tear the coating from the substrate.

The measurement of the coatings micro-hardness was evaluated in terms of norm STN ISO 4516 by the help of digital micro-hardness tester LECO LM 700 AT.

The structure and chemical composition of the examined coatings was investigated using a light ray of microscope Olympus BXFM and an electron microscope JEOL JSM – 7000 F with microanalyser INCA.

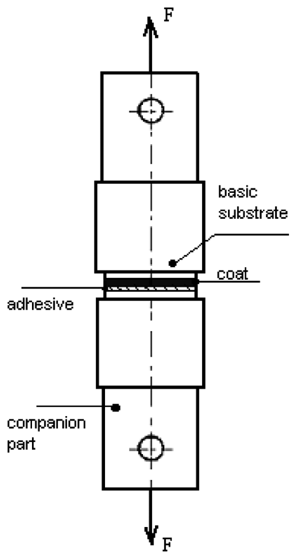

Fig. 1. The principle of a coatings adhesion test

3. Results and discussion

The adhesion of ceramic coatings was measured on 20 samples of which 4 samples from the same coating type were sprayed always in the one cycle. The same test conditions were maintained during individual tests series.

Calculated average values of the individual test samples adhesion are graphically worked on Fig. 2. From the picture it results that the lowest adhesion value was reached for the

Al_2O_3 coating without the inter layer. The interlayer increased the coating adhesion value approximately two times.

The addition of the Ni-based metal component (K 30) to basic Al_2O_3 powder has led to a substantial increase of the composite coatings adhesiveness. The coatings with 5% addition of K 30, formed without the interlayer, shows similar adhesiveness as the ceramic coatings with the interlayer (AM). The coating with 12% K 30 addition achieved the adhesiveness approximately 16 MPa what is almost a triple of Al_2O_3 coating (AM). The highest adhesiveness was recorded for a composite coating with 12% K30 whereby an average measured value achieved almost 25 MPa what is a distinct increase compared to a ceramic coating Al_2O_3 without the interlayer (to quintuple).

For more exact determination of the Ni addition content at which maximum adhesion is achieved, it will be necessary to extend the experimental program.

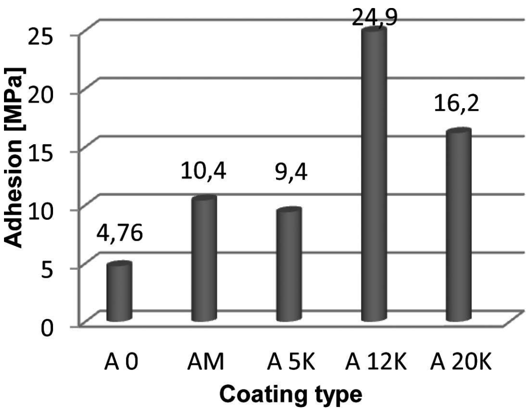

Fig. 2. Average adhesiveness values of coatings

The high-grade tracing of pre-modified surface by the ceramic and composite coating blasting is visible on the Fig. 3 and Fig. 4. A mechanical wedge increases the coatings adhesion.

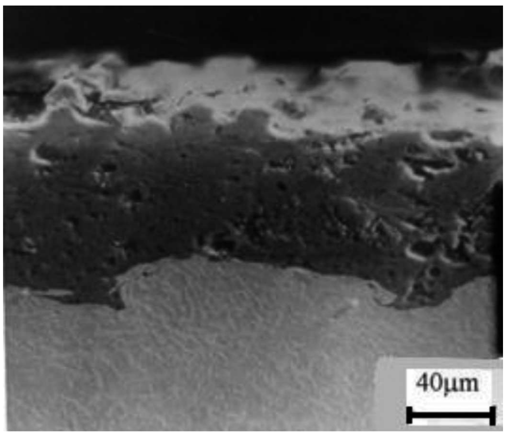

Fig. 3. Al_2O_3 coating anchoring to the steel substrate blasting surface

For the purpose of the nickel component presence verification in a sprayed coating, the evaluation of ceramic and composite coatings micro-hardness was made on metallographic samples. The composite coatings structure, Fig. 4 consists of two phases; hence the measurement was focused on the hardness values acquirement of light and dark phases. The results of the realized measurements are stated in Fig. 5. The average coating micro-hardness A0 achieved value 1419 HV0,05, powder K 30 on the nickel basis 420 HV0,05. By the comparing of the values with individual phases micro-hardness values in composite coatings we can assume that the dark phase responds to Al_2O_3 and the light phase responds to nickel component.

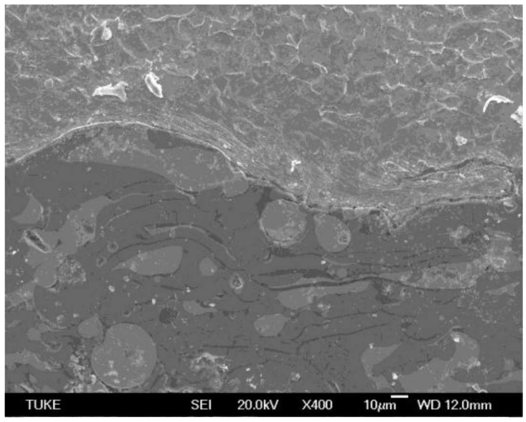

Fig. 4. Composite coating anchoring

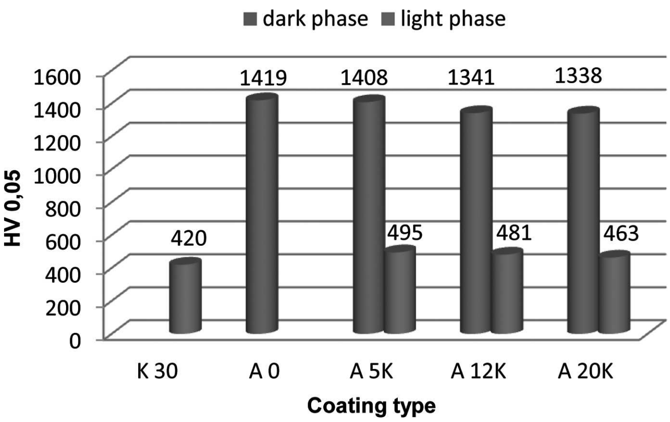

Fig. 5. Microhardness of of phases of individual coatings

The actual structure of the Al_2O_3 coating is documented on Fig. 6. The surface is characterized by a really heterogeneous structure. Individual particles are separated from each other by wide-angled boundaries. We can divide non-integrities in the layer structure to voids, pores, map disruption and massive insufficiently melted particles.

Fig. 7 shows the lamellar arrangement of particles on the fracture surface of the ceramic coating. The coating inner structure is created by the components interleaving at each other. It deals with a characteristic structure of the sandwich type with relatively small amount of mistakes. The bond between individual layers (splats in the disc shape) will be the better the fewer mistakes will contain such as pores, voids, and cold joints and the more components will be in the layer, wedged.

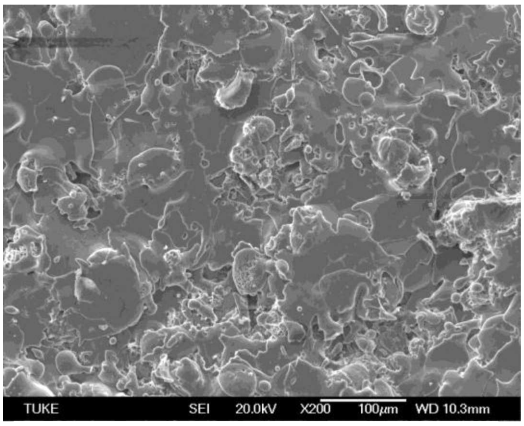

Fig. 6. Al_2O_3 coating surface

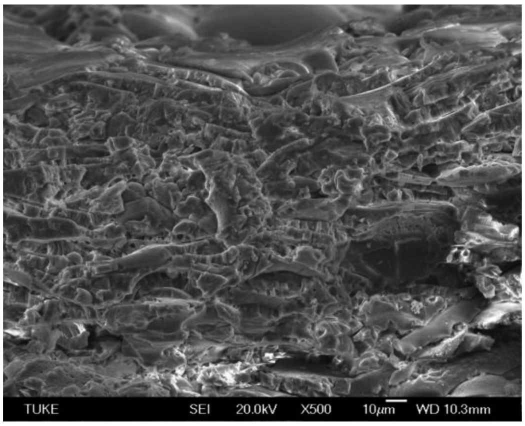

Fig. 7. Al_2O_3 coating sandwich structure on the fracture area

The overall view on the composite coating surface structure Al_2O_3 + 12% K30 is documented on Fig. 8. The snapshot presents globally more compact character of a composite coating surface with the lower occurrence of components cracks and disruption and the particles ability to replicate properly past the surface and to copy the previous components.

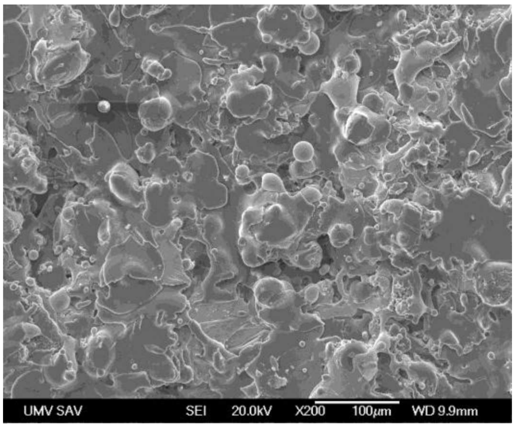

Fig. 8. The surface of composite coating

Fig. 9 presents typical composite coating lamellar structure which is similar to the ceramic coating created by the oxide alumina (Al_2O_3). The snapshot was made in the COMPO regime (the signal of backward electrons which is dependent on the Z element atomic number). Splats on the nickel basis (white areas) are markedly thinner compared to Al_2O_3 and as the nickel is plastic they copy them totally. Those nickel splats create inter-layer between the Al_2O_3 layers and thereby increase their cohesion. The composite coating contains fewer defects from the pores and voids occurrence point of view but in particular fractures caused by dilatation strains during a solidification process. Dilatation strains are caused by the different coefficient of the carbon steel thermal expansion (11.1 – 11.7×10^{-6} K^{-1}) and alumina (7 – 8×10^{-6} K^{-1}) [11, 12] as well as low ceramic deformation capability. The nickel thermal expansion coefficient is 12 – $13{,}5.10^{-6}$ K^{-1} [13]. In composite coatings that value and nickel plastic properties can eliminate the mentioned dilatation strain in coatings.

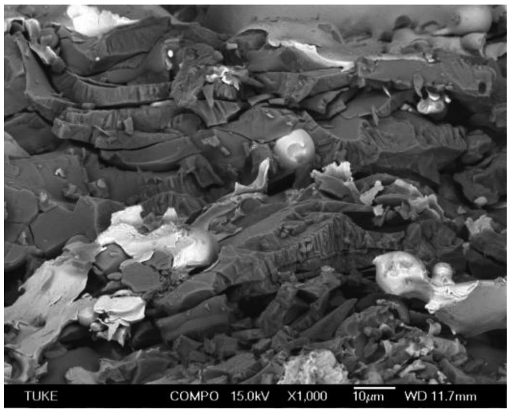

Fig. 9. Al_2O_3 coating sandwich structure on the fracture area

Stated facts probably increase the composite coating cohesion consistency as well as the adhesion to the steel substrate where what is more the diffusion character bond strengths can be applied.

For the complexity of microscopic examination the chemical analysis was made on the composite coating surface for the verification of metallic component K30 occurrence on the nickel basis in the composite coatings. Fig. 10 and 11 present the chemical spectral analysis of Al_2O_3+ 12% K30 composite coating surface. The chemical spectral analysis, made with the help of INCA micro-analyzer, confirmed the K30 metallic component occurrence on the nickel basis in the composite coating. Results of the chemical spectral analysis (Fig. 11) of a light area in the composite coating fracture confirm the assumption the light particles (spectrum 17) are on the basis of K30 metallic powder or nickel respectively.

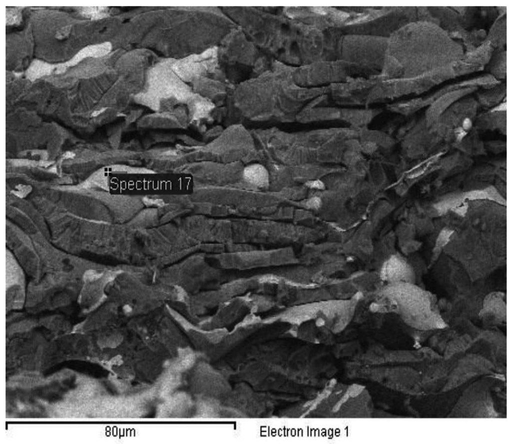

Fig. 10. The light area spectral analysis in the A12K coating fracture

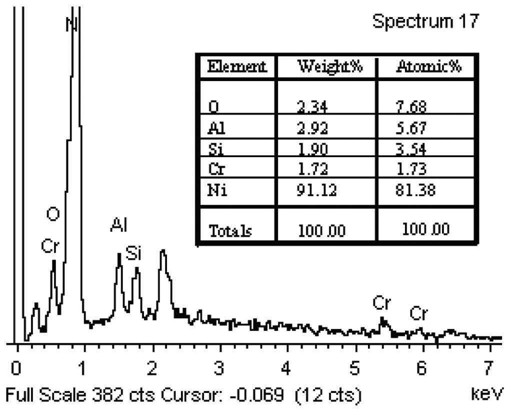

Fig. 11. Result microanalysis of light particles

4. Conclusion

The contribution was focused on the adhesion research and structures of the ceramic (Al_2O_3) and composite coatings on the Al_2O_3 basis with the K30 metallic component addition on the nickel basis with the components of various volumetric share. The coatings were created by the plasma spraying technology with water stabilization of the arc.

The results confirmed the nickel addition in Al_2O_3 coatings act very positively in terms of adhesion and cohesion properties of composite coating sprayed without the inter-layer use. The lowest adhesion was found out at the coating which was created from the pure Al_2O_3 without the inter-layer (4.8 MPa). By the nickel addition the adhesion is increased and the highest value was achieved for coating Al_2O_3 + 12% K30 (24,9 MPa).

The composite coatings application without inter-layers use increase economical effectiveness of composite coating application compared to the ceramic ones. The absence of the inter-layer brings time saving and the decrease of additional powder materials consumption.

The results of Al_2O_3+ K30 composite coatings structure showed the metallic component addition on the nickel basis into the ceramic matrix improve the coating structure in terms of the defectiveness what is expressed mainly in terms of the achieved adhesion.

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