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TESTING ADHESIVE BOND STRENGTH AND FRACTURE MECHANISMS OF THICKER AND POROUS PLASMA SPRAY COATINGS

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Abstract: For the functionality of metal products protected with plasma spray coatings critical is the adhesive strength of the bond of the coatings with the substrate surfaces. For the working life of the coatings the cohesive strength is also very important, which is directly related to the strength of the inter-lamellar connections, share of pores, presence of cracks and residual stresses in the deposited layers of coating and at the interface with the substrate. To test the adhesion / cohesion strength the most frequently used were the standardized test methods, ASTM C633 and Pratt & Whitney (PN 582005). Access to control of deposited plasma spray coatings allows the development, mastering and application of new quality materials, powders at the micro and nano level for more effective performance of metal products in service. For this paper, selected for testing were coating systems NiCrAlCoY₂O₃/ZrO₂CeO₂Y₂O₃ deposited by plasma spray process at atmospheric pressure (APS). Tests were performed according to standard Pratt & Whitney (PN 582005) and shown were assessments of adhesive bond strength and fracture mechanisms of thicker and porous plasma spray coatings. **Keywords**: adhesion, cohesive strength, plasma spray coatings, APS

INTRODUCTION

Adhesive bond strength is the basic characteristic of thermal spray coatings. It is very important for porous ceramic coatings such as organic bio-reactive ceramic hydroxyapatite $Ca_{10}(PO_4)_6(OH_2)$ and inorganic bio-inert ceramics ZrO₂, ZrO₂Y₂O₃, Al₂O₃, TiO₂ and Cr₂O₃ which have a wide range of applications. The method for testing coating bond strength is a method of tensile tests [1,2]. The control procedure for adhesive / cohesive strength of the plasma spray coatings is required for the functional part to be installed and thus approved for use in operation. According to the Pratt & Whitney standard (PN 582005) samples for testing adhesive bond strength are made of AMS materials depending on the type of substrate on which the coatings are deposited. For substrates based on Fe, Ni and Co alloys, samples are made from AMS steel 5504 (0.15%C, 13.0%Cr)X15Cr13(EN1.4024) [1]. Based on the standard for testing adhesive / cohesive strength of the coatings, the thickness of the coatings to be tested is directly related to the proportion of micro pores in the coatings. The thickness of the tested coatings is increased with the proportion of micro pores as to prevent the penetration of the adhesive through the layers of the coating to the substrate. For coatings with a proportion of pores below 2% a coating thickness of 250 µm is recommended. For coatings with a high proportion of pores thickness should be minimum 380 µm or to have a maximum thickness for the designed coating on the functional part [1,2]. It should be noted that the adhesion strength is not a constant in real conditions, it is a complex trait that depends on: roughness of the substrate surface, coating deposition parameters,





residual stresses due to mismatches of thermal and mechanical properties between the coatings and the substrates and the load conditions on the thickness of the coating. Testing adhesion bond strength is done on the hydraulic tensile testing machine at room temperature at low and constant tension speed. Low tension speed and gradual loading is used because of the stress state in the coating layers and at the substrate / coating interface. Tensile load is normal to the surface of the substrate / coating interface. During testing the load is increased from zero to the load level causing fracture. Depending on the quality of the coating (coating surface roughness, proportion and size of pores in the coating, stress state of coating layers) and the quality of the bond of the coating with the substrate surface, fracture of the specimen can occur in several unwanted places as shown in Figure 1 [3]. The coating bond strength σ = F/A (MPa) is calculated as the maximum load (F_{max}) on the sample divided by the area (A) of the fracture of the coating. The fracture surface area of the samples (A) is calculated as follows A = 3.14 x R²/4, where R is the measured fracture diameter [1,2].



Figure 1. Undesirable possible fracture locations: a) through the bond coating, b) at the bond coating / ceramic coating interface c) through the ceramic coating and d) at the ceramic coating / glue interface [3].

This paper analyzes the adhesive / cohesive bond strengths, and fracture mechanisms of the NiCrAlCoY₂O₃/ZrO₂CeO₂Y₂O₃ plasma spray coating system using tensile testing according to most commonly applied standard test methods, Pratt & Whitney (PN 582005) and (ASTM C 633), for testing adhesion / cohesion bond strength of thermal spray coatings. The examination tests belong to the group of mechanical methods in which the adhesion is determined by using a force on the coating / surface system [4-6]. Coating materials used for the deposition are two different plasma spray coatings. Adhesive bond strengths were measured in the bond coating / ceramic coating system, whose values and fracture mechanisms were discussed.

MATERIALS AND EXPERIMENTAL DETAILS

In the experiment two spray powders were used, Sulzer Metco 461 (NiCr/Al/Co/Y₂O₃) and Metco 205NS (ZrO₂CeO₂Y₂O₃) [7,8]. The NiCrAlCoY₂O₃ bond layer was deposited with a plasma gun speed of 250 mm/s and the ZrO₂CeO₂Y₂O₃ ceramic coating layers at plasma gun speeds of 250 mm/s, 350 mm/s and 500 mm/s on cold and preheated substrates with temperature of 160-180°C. The specimen for tensile testing consists of two paired samples, \emptyset 25x50mm in size, on one of the samples a plasma spray coating was deposited and on the other adhesive was applied, Figure 2. Before pairing and gluing of the samples

a control and quality check of the deposited coatings on the substrates was performed, because for testing, the coatings in deposited state should not peel off, separate at the edges of samples or have cracks. In order to increase adhesion surface, the paired samples were roughened using the same parameters used for roughening the



samples on which the coatings were deposited. Samples with the deposited coatings are not roughened. For gluing of the sample an adhesive (glue) was made based on resin and additives. Components of the adhesive were: resin LY 556, herter NT 972 and additive Al₂O₃ TiO₂. Resin LY 556 was mixed with component NT 972 in a weight ratio of 100:27 and then the additive Al₂O₃ TiO₂ was added in weight percent of 20% of the total weight of the resin. The prepared adhesive was applied in a thin layer on the roughened samples. Fixation and gluing was done in a tool, made according to the Pratt & Whitney standard, of aluminum alloy so that five sets of specimens were glued in it at once [1,9]. After alignment and clamping of samples, the tool was put in a chamber furnace heated to a temperature of 80°C. During gluing the tool with the samples was held at an angle of 30 degrees in the furnace. Parameters for gluing of samples were: gluing at 80°C for 1 hour, gluing at 140°C for a period of 2 hours and 20 minutes, and cooling of the tool with glued specimens in the furnace to room temperature. Before the tensile testing the excess adhesive was carefully removed from each set of specimen joints. For tensile testing of





specimens adapters were used designed so that during the tests they ensure elimination of shear forces. Figure 2 shows the scheme of the adhesive tensile test [1,9].

Testing of specimens was done at room temperature with tension speed of 1 mm/min. Five specimens were used for testing each time. The paper presents the mean values of adhesive bond strength.

RESULTS AND DISCUSSION

Table 1 shows the rate of deposition of coating layers, the mean values (of coating thickness, porosity in the ceramic coatings and adhesive bond strength) and fracture mechanisms. In all test samples the crack fracture extended horizontally along the substrate / bond coating interface. There was no fracture through the bond layers at the bond coatings / ceramic coatings interface and through the layers of ceramic coating in any of the samples. The fracture mechanism for all deposited coating systems was adhesive at the substrate / bond coating interface. Figure 2 shows a representative example of a broken specimen. For all deposited coating systems high levels of adhesive bond strength were measured. The measured values were directly related to the stress states of the deposited layers and the proportions of porosity in the oxide lamellae. The stress states and porosity of the coating layers were controlled by the substrate temperatures and deposition rates of plasma spray layers.

Table 1. Adhesive bond strength, and fracture mechanisms of plasma spray NiCrAlCoY₂O₃ / ZrO₂CeO₂Y₂O₃ coatings

Plasma gun speed (mm/s)	Metal substrate temperature (°C)	Coating thickness (mm)	Ceramic coatings porosity (%)	Adhesive bond strength (MPa)	Fracture mechanism
250	21-23	0.557	17.89	40.9	Adhesive fracture at the substrate / bond coating interface
	160-180	0.543	15.11	47.5	Adhesive fracture at the substrate / bond coating interface
350	21-23	0.552	16.34	46.2	Adhesive fracture at the substrate / bond coating interface
	160-180	0.535	13.45	53.1	Adhesive fracture at the substrate / bond coating interface
500	21-23	0.567	15.9	48.7	Adhesive fracture at the substrate / bond coating interface
	160-180	0.549	12.63	54.3	Adhesive fracture at the substrate / bond coating interface

The highest values of adhesive bond strength have the coatings deposited with a plasma gun speed of 500 mm/s on preheated substrates, which enabled the deposition of layers with the lowest stress condition and proportion of micro pores.

Higher plasma gun speed deposits thinner layers of coatings which introduce less heat and reduce the temperature differences in depth of the deposited coating and the substrate, and thus input less residual stress into the coating and at the interface with the substrate [3,10].

CONCLUSIONS

Based on the obtained values of adhesive bond strength and fracture

mechanisms of the NiCrAlCoY₂O₃/ZrO₂CeO₂Y₂O₃ plasma spray coatings system the following conclusions may be drawn:

- Ξ Measured adhesive bond strengths between the coatings and the substrate surfaces, which are crucial to the functionality of the coated working parts, showed that the deposited coatings system meets all the criteria set in the standard Pratt & Whitney (PN 582005).
- Ξ Small amounts of heat input and uniform heat distribution in depth of the coatings in the ceramic coatings and on substrates surfaces influenced the formation of a smaller proportion of residual stresses and their more uniform distribution, as confirmed by the high values of adhesion strength.
- Ξ Examinations of the coatings systems have shown that the cohesive strength of all the deposited layers is good and that it is greater than the adhesive bond strength.



Figure 2. Fracture mechanism at the substrate / bond coating interface



Impeccably obtained test values of adhesive / cohesive bond strength of the NiCrAlCoY₂O₃/ $ZrO_2CeO_2Y_2O_3$ coatings ensures their durability and justifies their use on functional working parts in exploitation.

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Note

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