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DETERMINING CRACK LENGTH AND CRITICAL LOAD USING VICKERS INTERSURFACE INDENTATION METHOD ON THE INTERFACE OF THE SUBSTRATE / COATING

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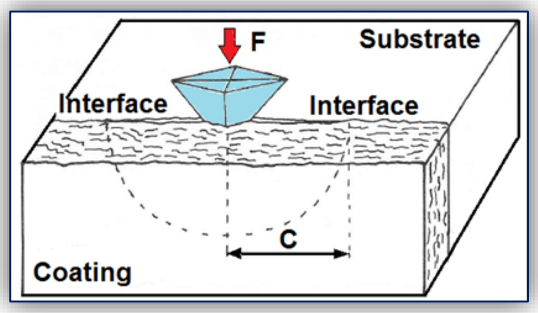
Abstract: The properties of plasma spray coatings are directly related to the type and characteristics of the powder, deposition parameters, type of material and the morphology of the substrate. Key properties of the coating are toughness, strength, shear, hardness, inter surface toughness and adhesion of the coating. Adhesive strength determines the quality of the coating, while the cohesive strength of layers determines the lifetime of the coating in service. These properties are very important for biomedical inert and reactive ceramics which are deposited on implants. Plasma spray coatings show inter surface complexity with the substrate due to the influence of residual stress, porosity and micro cracks at the inter surface. Therefore, for each coating, adhesion strength must be examined. There are several methods of measuring joint adhesion strength of thermal spray coatings of which most commonly used is the method of tensile testing. Presented in this paper is the Vickers method of inter surface diamond pyramid indentation testing at the interface substrate/coating, which is used to determine coating adhesion strength.

Keywords: plasma spray coatings, indentation test, adhesion, cohesion

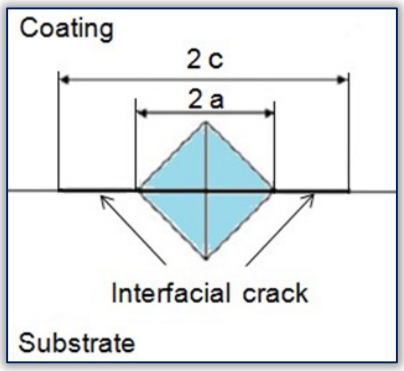
1. INTRODUCTION

For evaluation of adhesion strength of plasma spray coatings or surface/coating bond strength, many methods have been developed, of which the most commonly used are the tensile test method specified by standard (ASTMC633) and the scratch test according to standard (ISO/WD27307), [1]. The Standard that refers to the tensile test cannot be applied in the evaluation of the coating with adhesive strength greater than the strength of the glue which is used to prepare samples for the tensile test, [2]. A universal method for measuring the adhesion of coatings does not exist due to various combinations of substrate/coating systems. The best method of determining adhesion strength is the one that simulates the practical state of stress in the coating layers, [3]. The residual stresses in the coating layers, a result of mismatched thermal and mechanical properties of the substrate and the coating, are of great importance for adhesion strength. Adhesion strength is not a constant in real circumstances, but a complicated feature that depends on the load conditions applied to the designed coating thickness, [4]. Therefore, it is important to determine the adhesion strength because, based on the test values, the quality of the coating is assessed, understanding the degradation of the substrate/coating bond at the interface in operating conditions is determined as well as the evaluation of life span of the coatings on the coated working equipment. The obtained estimate results also allow, through testing of a batch of samples, to homologate optimal powder deposition parameters which will enable coating reproducibility of high and uniform quality. In addition to standard procedures of tensile testing of adhesion strength, among the most common methods applied is the indentation test, which is schematically shown in Fig.1. [5]. This method can easily evaluate the adhesion strength of the coating by a plurality of diamond pyramid indentations on the substrate/coating interface using the Vickers method, which has long been accepted and is commonly used in industry. When using thicker coatings, the fracture toughness (K_{ca}) at the boundary surface of the substrate/coating is determined using the Vickers method of measuring the total lengths of cracks (*Cc*) caused by ploughing with a diamond pyramid along the interface. Several measurements are conducted for the testing. Shorter cracks along the interface indicate that the coatings have higher adhesion strength and interface toughness. Also, a higher critical indentation force (*Fc*) which causes cracking at the intersurface confirms higher adhesion strength of coatings.

This article describes the Vickers method which is applied for testing adhesive strength of coatings. The paper shows measurement values of critical loads (*Fc*) and total lengths of cracks (*Cc*) at the interface of the substrate and the NiCrAlY coating, and their values and causes of fracture are discussed.



a)



b)

Figure 1. a) A schematic view of an indentation test; b) the geometry of the indentation

2. MATERIALS AND EXPERIMENTAL DETAILS

For the production of the coating the powder marked AMDRY 9624 (Ni22Cr10Al1Y) was used with a range of powder particles granulation of 11 to 37 μm, [6]. Before the process of depositing the substrate surface is roughened by Al_2O_3 corundum particles with a size of 0.7 to 1.5 mm. The coating was deposited with a thickness of 0.4 mm on a sample of stainless steel X15Cr13 (EN 1.4024). The material was used in soft annealed condition, 40x10x5 mm in size. The sample for the adhesion testing was prepared in the same way as for the hardness test, and the measuring was conducted on the cross section of the sample which prior to measuring was polished to a mirror shine. Measuring points were in the edge zone (A and C) with the highest content of residual stretching stresses and in the middle of the sample, zone (B) with the smallest proportion of the residual compression stresses. Indentation testing is based on the direct measuring of the length of radial cracks initiated by the Vickers indenter at the interface substrate/coating, as shown in Figure 2. The geometry of the imprint was of irregular shape with a crack initiated by the critical load at the substrate/coating boundary surface. The irregular shape of the imprint is due to the soft annealed condition of the substrate, which had hardness lower than the NiCrAlY coating.

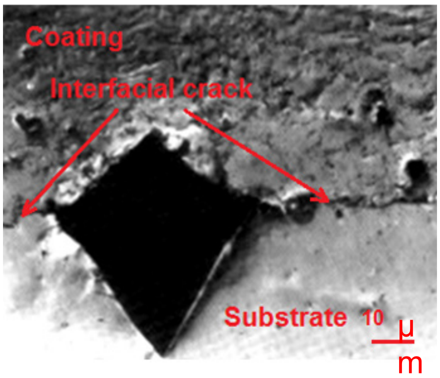


Figure 2. (BM) the geometry of the indentation with the crack at the interface initiated by the critical load

For obtaining reliable values six indentations in each zone of measurement were made, in order to reliably determine the mean length of a crack (C_C) and the mean value of the critical force (*Fc*). With the increase of the applied load (*F*) the depth of indentation and the length of the indentation half diagonal (a) increases. By gradually increasing the load, the diagonal of the indentation (a) at one point reaches a critical value initiating an intersurface crack. The crack is initiated from the corner of the indentation and extends along the interface, as shown in Fig.2. The ratio between the crack length (*c*) and the applied load (*F*) is a straight line which represents the crack curve for a particular substrate and coating thickness, [7]. The measured crack length at the interface is used to calculate adhesion and interface toughness. The critical load of indentation (F_c) and the corresponding crack length (C_c) define adhesion and interface fracture toughness of the interface (K_{ca}) which is expressed by Equation 1, [7], [8].

$$K_{ca} = 0.015\left(\frac{E}{H}\right)_I^{1/2} \frac{F_c}{C_c^{3/2}} \tag{1}$$

where $(E/H)_I^{1/2}$ is the square root of the ratio of the modulus of elasticity and hardness at the interface. This ratio is expressed by Equation 2, [7].

$$\left(E/H\right)_I^{1/2} = \frac{\left(E_S/H_S\right)^{1/2}}{1+\left(H_S/H_C\right)^{1/2}} + \frac{\left(E_C/H_C\right)^{1/2}}{1+\left(H_C/H_S\right)^{1/2}} \qquad (2)$$

In the equation (E_i) is the Young's modulus, (H_i) is hardness and the indices (*s*) and (*c*) represent the substrate and the coating. The method of interfacial indentation of the diamond pyramid is reliable for assessing the adhesion strength of the coatings as it is independent of the thickness of the coating.

3. RESULTS AND DISCUSSION

In Table 1 shown are crack lengths (C_c) in the measuring zones, which were made at the interface by indenting with the diamond pyramid using different critical loads (*Fc*) with respect to the measuring location. Fracture cracks of a certain length that separate the coating from the substrate, extend horizontally along the interface. Figure 2 shows an example of a crack at the substrate/NiCrAlY coating interface in zone (B). The measured crack lengths were directly related to the stress state of the deposited layers in the measuring zones. The largest lengths of the cracks were measured in the edge zones (A) and (C) caused by the smallest load (*Fc*) as expected, since the edge zones are the sites with the highest content of residual stresses at the interface.

Table 1. The measured crack lengths (Cc) and the mean value of the critical load (Fc) and (Cc)

Measuring zone	The length of the crack Cc [μm]						Fc [N]	Cc [μm]
A	1460	1550	1390	1410	1290	1370	21	1411
B	1350	1410	1200	1280	1220	1310	49	1295
C	1530	1480	1520	1380	1510	1310	19	1455

According to the results shown in Table 1 we can see that the length of the crack (the binding of coating to substrate) varies significantly with the location of indentation along the cross-section of the sample. This variation of crack lengths is explained by the fact that the droplets of melted powder progressively and unevenly transfer heat to the substrate during plasma spray deposition; due to axial movement of the plasma gun from zone (A) to zone (C). Because of the edge effect in the process of deposition the molten particles input more heat into the edge zone of the coating. As a result of mismatch of thermal and mechanical properties of the substrate and the coating, in the buffer zone of the substrate/NiCrAlY coating there are residual stresses unevenly distributed along the interface. Shorter crack lengths formed with indenting with a higher indentation force (*Fc*) indicate that the coating in the center of the sample (zone B) has greater adhesive strength and interface toughness compared to the edge zones (A and C).

4. CONCLUSION

In this paper conducted was measuring of crack lengths and the critical load on the substrate/NiCrAlY coating interface using the Vickers method in order to evaluate adhesion and fracture toughness of the interface. On the basis of the measurements it can be determined that the Vickers method is very simple and reliable for assessing the quality of the bond between the substrate and the plasma spray coating. Due to an uneven distribution of stress caused by the different amounts of heat input during the deposition of powder and the difference in the coefficient of elasticity of the substrate and the coating, the highest strain and the lowest fracture toughness with the longest cracks has the interface in the edge zones of the sample. The obtained values of the study show good adhesion of the coating because at the interface substrate/coating in deposited state there are no observed defects, such as discontinuity of the deposited layers, voids, micro pores and contamination from the corundum.

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