



Interfacial indentation and shear tests to determine the adhesion of thermal spray coatings

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Abstract

Adhesion is one of the most important parameters which influences the development of thermal spray coatings. Therefore, the level of adhesion should be known for a given application. Apart from the standardized Tensile Adhesive Test (TAT), more than 80 methods are reported to measure the coating adhesion. Most of them are energy consuming in terms of time, cost and equipment. Moreover, they do not fulfil the necessary requirements of accuracy, confidence and representation of the real delamination process observed in service. To address this problem, the interfacial indentation test is used here to initiate and propagate a crack at the interface between the substrate and the coating. Studying the extension of the crack, an interfacial toughness is defined and deduced analytically from the experimental results. The new shear test, developed in the frame of the EU-CRAFT-project "Shear Test for Thermally Sprayed Coatings", is also employed to assess the coating adhesion. Both tests are compared to the standardized TAT for various spraying systems, materials, substrate roughness and coating thickness. Advantages and disadvantages of the three tests are discussed. Correlations between the tests results obtained for different coating—substrate combinations are presented and general trends are described.

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1. Introduction

Adhesion of a coating on its substrate is an important parameter to be considered for the use of the thermal spray coatings during thermo-mechanical loading conditions. Industries still need a quantitative adhesion test as simple, reliable and representative as possible. In spite of the fact that the standard tensile adhesive test is widely used, still it does not fulfil all these requirements [1,2]. Furthermore, not only it necessitates the use of a bonding agent which has a limited strength resistance, but also does not represent the stress conditions met in service, which are more characteristic to shear rather than tension. These are the reasons why many other tests, more than eighty nowadays, were developed to quantify the adhesion that would be independent of these limitations. However, this

objective is still far from being reached. The test where the interface toughness, which characterises the resistance to crack propagation at the interface (i.e. delamination of the coating), is determined offers at least a mean to compare the results of some mechanical tests that are able to allow its calculation. Within the present work, we examine the results obtained by three test methods: the interfacial indentation test associated to an analytical model for the calculation of the interface toughness [3–5], the shear test of more recent development [6–8] and the tensile adhesive test, performed on different coating–substrate combinations. The results were discussed accordingly and some correlation between them has been found.

2. Tensile adhesive test

The tensile adhesive test (TAT) is a widely used standard method, which follows both the European EN 582 [1] (equivalent to international standard ISO 14916) and American ASTM C 633 [2] standards.

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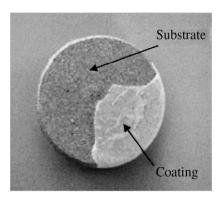


Fig. 1. Macrograph of a specimen coated with plasma sprayed Al_2O_3 after the tensile adhesive test [11].

2.1. Testing and interpretation

A cylinder of normalized geometry is coated on one of its flat face and, subsequently, glued with an epoxy resin to an identical cylinder, the later playing the role of the counterpart. After curing, the assembly is submitted to a progressive tensile loading until fracture occurs. Adhesion is characterised by the bonding strength calculated by dividing the maximum normal force necessary to separate the two cylinders by the cross section area of the cylinder.

2.2. Critical analysis

Although this test has been normalized for a long time and was recently improved [9], its interpretation is difficult and some problems remain still unsolved. Actually, the same bonding strength may represent different fracture processes since the fracture can occur: i) within the coating by cohesive failure, ii) within the glue if the adhesion strength and cohesion of the coating are comparable or higher than the adhesive one, iii) at the interface in ideal conditions or even, iv) in a combination of two or three of them, as it is often observed [10–12].

As an example, the surface of an alumina coated sample after testing is shown in Fig. 1. Some parts of the coating remain adhered to the surface, whereas the major part of it is kept glued to the counterpart. In this situation, it is not clear how to calculate the real adhesive strength, since there are at least two distinct contributions of the observed bonding strength such as the adhesion between coating and substrate and the adhesion between coating and the epoxy resin.

The main limitations of this test method are i) cost of the sample (machining, drilling, tapping) and epoxy resin, ii) preparation time (gluing, curing), iii) strength of the epoxy which should be higher than the adhesion strength of the coating to the substrate [13,14] and iv) porosity of the coating, which may invalidate the results due to penetration of the bonding agent into the coating towards the interface, modifying its physical properties [14–17]. In such a situation, the EN 582 standard [1] recommends the use of the bending test, but this test has also its own limitations and it is very sensitive to the samples preparation conditions.

3. Interfacial indentation test

The interfacial indentation test was used for the first time at the end of the 80's [18,19] and, since then, has received further development in particular for its interpretation in terms of the apparent interface toughness [3–5]. This test is now often used as an alternative to other tests [17,20,21]. The principle of the test is to perform a set of conventional Vickers indentations on the cross section of the sample in order to initiate and propagate a crack at the existing interface between the coating and substrate.

3.1. Testing and analysis

After careful preparation of the sample cross section, consisting in grinding with SiC paper from grade 80 to 1200 and, subsequently, followed by final diamond polishing until 3 or 1 μ m, different Vickers indentations are performed for 2 min at the interface using a discrete increase of the indentation load [3,22]. One diagonal of the Vickers indenter has to be in the alignment of the interface. The principle of the test is shown in Fig. 2. The crack generated by the penetration of the indenter is localized along the interface plane and have a semi-circular shape (half penny shape), as shown previously by Ostojic [18] and Choulier [19].

Démarécaux, Chicot and Lesage [3–5], using the $(P_{\rm c}, a_{\rm c})$ couple associated to the interfacial crack initiation, where $P_{\rm c}$ is the applied critical load, and $a_{\rm c}$ is the interfacial crack length, proposed the relationship shown below to calculate an "apparent interfacial toughness" $K_{\rm Ca}$, in analogy to the relationship put forward by Anstis, but taking into account the substrate and the coating mechanical properties:

$$K_{\text{Ca}} = 0.015 \frac{P_{\text{c}}}{a_{\text{c}}^{3/2}} \times \left(\frac{E}{H}\right)_{\text{I}}^{1/2},$$
 (1)

where the $(E/H)_I$ ratio, defined in Eq. (2), characterises the global behaviour of the coating/substrate system.

$$\left(\frac{E}{H}\right)_{I}^{1/2} = \frac{\left(\frac{E}{H}\right)_{S}^{1/2}}{1 + \left(\frac{H_{S}}{H_{C}}\right)} + \frac{\left(\frac{E}{H}\right)_{C}^{1/2}}{1 + \left(\frac{H_{C}}{H_{S}}\right)}, \tag{2}$$

where E is the Young modulus, H is the hardness and I, S, and C subscripts stand for interface, substrate and coating, respectively.

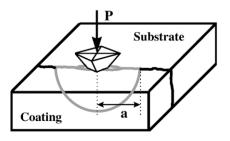


Fig. 2. Interfacial indentation test [4].

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