CHARACTERIZATION OF THERMALLY SPRAYED COATINGS COHESION

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1. INTRODUCTION

The major limitation for the development of ceramic coatings and their full application on an industrial scale, as well as expanding their range of applications, lies in their poor fracture toughness. The coating is usually considered to be a homogeneous and monolithic deposit that is constrained and controlled by the engineering properties of the substrate, but in most cases, taking into account its real behaviour, it should be considered as planar oriented composite material.

Plasma sprayed coatings have been developed and optimized over the last decade. The engineering properties which have been taken advantage include corrosion protection, control of the wear rate and thermal barriers. The main result was an extended life and/or operating limits of the coated component.

Very often the coating is considered as an “add-on”, but the present technology of thermal spraying processes does not permit coatings to be used in application role where the failure leads to the immediate removal of the component from service. Therefore the coating should be considered as one part of the overall system and the current trend is to design the coating as an integral part of the component assembly rather than as an add-on to the substrate.

In effect most coatings are well known to be not very reliable. The low reliability means that the material properties of these coatings are highly variable, due to the lack of enough scientific knowledge of how their microstructure is formed during deposition and to the highly anisotropy of the resulting layered structure. Plasma sprayed coatings exhibit a lamellar morphology consisting of flattened “saucer-shaped” particles. The coatings are normally greater than 25 μm in thickness and can thus be described as bulk coatings. The minimum microstructural detail would be a single splat (lamella) which is about 5 μm in thickness and up to 80 μm in diameter.

The lamellar microstructures are complex and they are the result of the deposition and cooling of successive molten droplets where numerous parameters are involved such as temperature of the substrate, spraying atmosphere,
plasma gas properties (velocity, enthalpy), particle dimensions. The mechanical properties of the coatings depend on defects, such as porosity, cracks, lamellae size, etc. These aspects have a strong influence on the important properties of the deposit such as adhesion between coating and the substrate and cohesion inside the coating. The service failure mode can be described as interfacial, cohesive, or mixed interfacial-cohesive.

2. State of the Art

Many theories and mechanisms have been proposed, none however cover all situations and therefore the best test method is often that which simulates practical stress conditions. Laboratory tests should induce the observed service failures with a scale down criteria as concerns the working time, otherwise such tests will be of limited application to engineering design and quality control.

When critical thermomechanical loading conditions are exceeded, two different patterns of damage can occur:

a) if the adhesion strength of the coating to the base material is exceeded, the coating peels from the parent body;

b) if the internal coating strength is exceeded, separation occurs within the sprayed coating and the parent body is still covered with coating material in the damaged area.

Cohesive strength is mainly determined by residual stresses, friction and damping inside the compounds. In order to achieve high cohesion, compressive strains inside the coatings should be near the yield limit of compression and particle size should be low.

The yield limit of compression can be tailored by:

1. volumetrical stiffness of the substrate
   length*width*heigth*Young modulus
2. geometrical stiffness of the substrate
   radii or ring for instance
3. triaxial stress state.

The adhesion of thermally sprayed coatings is not only an interfacial problem of each lamella within a coating, but also involves the integrity of the interface between the substrate and coating, residual stresses, crack population and pore size and distribution.

Different tests methods have been proposed based on macroscopic or microscopic approaches. Two properties are generally distinguished: strength and toughness. Most of measurements are carried out with tensile adhesion tests, double cantilever beam tests and scratch tests. These methodologies must be take into account the difficulty involved with any mechanical property assessment of coating due to the need of attachment of a loading device to the coat-
ting without influencing the properties that are being measured. On the micro-
scoplc level the adhesion properties were investigated by shearing individual par-
ticles from the substrate surface and further theoretical work has treated adhe-
sion as a stochastic process that depends on the formation characteristics of the
first monolayer of material. Acoustic emission studies were also proposed in
order to assess the crack density function, a product of the number of cracks
and crack size.

These methods provide many different approaches to evaluate the adhesion or cohesion strength of coating systems. However each has disadvantages and none can elucidate the difference of in service performance. As an example, tensile adhesion test results could be influenced by epoxy penetration (epoxy is used to attach a coated specimen to a support fixture) and probe alignment, the double cantilever beam test requires elaborated and expensive test equipment and special specimen preparation, for the scratch test the theory developed for thin coatings may no longer be applied for bulk coating.

Indentation technique such as micro-Vickers hardness tests have been used to evaluate the fracture toughness of coatings and to demonstrate that the properties of coating are generally anisotropic. These technique, among others, may be used to gain a fundamental understanding of coating performance or quality control.

Other tests such as the ACT-JP test require a pre-blasted sprayed specimen setted to an angle $\theta$ with a jet stream of alumina particles of suitable weight used as the erosion material to evaluate the resulting weight loss and surface state, so giving qualitative information on internal strength of the coating. Wear tests — Ogoshi’s wear test, ring-on-disk wear and scratch wear — are often used to evaluate coating resistance to wear and to obtain indirect information on coating scaling off.

3. FRETTEING WEAR RIG

In order to evaluate the “in service” behaviour of the coating, fretting-cor-
rosion tests, a particular class of wear tests, represent an interesting mean of
investigation. A dedicated fretting wear rig, schematically shown in fig. 1, was
designed together with the Department of Metallurgy and Materials Science of
Nottingham University.

Tests at high pressure in superheated steam (400°C, 50 bar) and at high
temperature (up to 1300°C) in air or corrosive atmosphere (H$_2$S, H$_2$, NO, NO$_3$, SO$_2$,
H$_2$) can be carried out. During the test the applied value of normal load, fre-
quency and amplitude of oscillation can be recorded. After the test the failure
morphology can be examined by optical microscopy or SEM.

Preliminary tests were carried out in air at room temperature on piston rings coated with YSZ (yttria-partially stabilized zirconia) subjected to an initial
static load that approximately corresponds to the installed bending stress of the ring at nominal diameter and to a dynamic load with an oscillation frequency of 50 Hz. The progressive break-out of the coatings observed during the test in the previous cyclic conditions could be correlated with the coating fracture toughness and with coating porosity and microstructure.

A modified device of measurement has been developed in order to evaluate directly the variation of the friction coefficient during the test. The aim is to assess the progressive coating scaling off by monitoring the friction coefficient variation, in order to obtain information on coating cohesion properties. This can represent a more powerful information than weight loss and surface morphology after the test and can lead to a correlation with coating hardness, fracture toughness and spraying parameters, because local loads due to friction
are responsible of coating scaling off when coating cohesion strength is overcome. Tests are now under development.

A significant result of this kind of test lies in the possibility to reproduce the environmental conditions which characterize the operating environment, so that it must be considered as a complementary method respect to indentation and scratch tests, more focused on fracture toughness investigation of the as sprayed coatings.

Cohesion properties may be strongly influenced by the corrosion resistance of the coating, and the damages due to these phenomena (e.g. destabilization of YSZ due to reactions yttria-S, yttria-V) can be responsible for coating failure. The interaction between these different mechanisms needs to be considered in order to achieve a satisfactory coating design, both microstructural and mechanical. The design should be based on lifetime modelling because this is the critical information that should be forthcoming from any test method. Such design will increase understanding of thermal sprayed materials and coatings so their reliability and application will grow.

A further point to underline is that due to the highly variable nature of the material properties of coatings, statistical analysis of results (i.e. Weibull distribution) is necessary. This requires a suitable number of tests.

4. CONCLUSION

The adhesion strength and cohesion strength of thermal spray coatings can be influenced by many factors, such as spray variables, powder characteristics, substrate preparation, post treatments and service conditions. It is very important for materials designers to remember that these properties are strongly related to service conditions and therefore characterization tests which can reproduce real operating conditions are of primary importance. A fretting wear rig was designed and developed with the aim to reproduce the in-service conditions and to obtain information on the previous properties. Preliminary tests indicate a correlation between coating behaviour and micro-hardness measurements carried out in air at room temperature. Further developments have been implemented on the measurement device to investigate better the possible correlation by monitoring the friction coefficient variation during the test and realize tests at high temperature and high pressure.
REFERENCES