# Mechanical and Fracture Properties of Thermal Barrier Coatings Fabricated using Slurry Spray Technique

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**ABSTRACT.** Thermal barrier coatings were introduced to reduce operating temperatures and thermal stresses in structures and machine components across a wide range of industries and applications more than 40 years ago. Recently a new, relatively simple and low cost manufacturing method of thermal barrier coatings based upon the slurry spray technique has been developed with a focus on aerospace applications. The challenge in the development of this technique was to achieve the coating quality comparable to the existing manufacturing methods, which are often expensive and inapplicable to coat large or curved surfaces. This paper describes the developed technique and presents selected results of thermo-mechanical and fracture testing of the thermal barrier coatings including graded coatings fabricated using this new method.

## **INTRODUCTION**

Thermal Barrier Coatings (TBCs) represent a relatively thin layer of a material with high insulating properties, such as ceramics, that is bonded to a substrate, which is usually metal, to protect the metal load carrying structure during temperature excursions. The application of TBCs can significantly increase the operating temperatures up to 1400-1500°C, increase efficiency and improve the durability of the components. There are many applications, which have benefited from adopting TBCs. These include the aeronautical, aerospace, automotive and nuclear industries and heavy-duty utilities such as diesel trucks [1, 2, 3].

The development of TBCs has centred mostly on Partially Stabilised Zirconia (PSZ) due to its unique physico-mechanical properties and has been led by its use in aircraftengine combustion-path components [4]. The significant advance in the development of an effective protective coating was associated with the development of Functionally Graded (FG) TBCs. FG-TBCs are multiphase composite materials that are engineered to a have a spatial variation of material constituencies. Using FG-TBCs, as an alternative to joining directly together two dissimilar materials such as ceramics and metal, carries several advantages including: much lower thermal stress distribution across the thickness; minimisation of stress concentrations at interface corners; and an increase in bonding strength. There are many fabricating methods for depositing ceramics or other coating materials on a metal substrate which have been developed over the past three decades [5]. All fabricating techniques can be categorised in three main groups: bulk processes, flame spray techniques and deposition techniques; each technique differing from each other greatly, in terms of physical principal used, cost and simplicity. However, the main obstacle in the widespread application of these techniques is a relatively high cost of manufacture and equipment. Moreover, many of these techniques are not applicable to cover large or curved areas. All these drawbacks form the main motivation for the current developments.

First, the new developed technique will be briefly outlined. The technique is also suitable for producing the FG-Coating. Examples of the FG-Coatings will be given in the paper. Experimental results on thermal cycling, adhesion strength, investigation of microstructure and effect of various manufacturing parameters on the quality, fracture and durability of the coating will be discussed. The paper will be concluded with a summary of major outcomes of the current experimental study and suggestions on future work.

## **SLURRY SPRAY TECHNIQUE**

The Slurry Spray technique for manufacturing TBC's utilises traditional wet powder spraying methods to deposit sinterable coating materials onto target substrates to produce a functional coating [6]. The process involves suspending the coating material within a fluid to form a slurry mixture that can be applied to a surface using common gravity fed spray guns. Successive layers are then sprayed onto the inconel substrate and dried using varying slurry compositions. The optimal thickness of the layers to deter surface cracking during the drying process is approximately 100  $\mu$ m (which can be seen in Fig. 1) and the drying time is approximately an hour, depending on ambient conditions. After the desirable number of layers of the TBC is deposited the multi-layered coating is loaded in a compression chamber to form a densified layer before being sintered with an acetylene torch or furnace. The applied pressure varies depending on the number of coating layers, typically between 10 and 40 MPa. Details of this technique can be found in [7] and [8].



Figure 1. Cross-section of a monolayered coating

TBCs produced using the Slurry Spray Technique have many advantages compared to other fabrication methods in terms of increased simplicity, reduced fabrication costs, potential application to a range of coating materials and the ability to coat complex surface geometries. The Slurry Spray technique utilises unsophisticated equipment and techniques in simple progressive stages to create the final coating. The process has the potential to be adopted in an automated environment but can also be utilised for manual applications.

The Slurry Spray technique can be applied to a range of TBC materials, provided that the materials can be prepared as a powder capable of being sintered at temperatures below the melting point of the substrate. As well as being an established TBC material, zirconia powders are readily suited for the Slurry Spray Technique. Zirconia powders are required to be either partially or fully stabilised using a metal oxide addition to prevent cracking within the TBC after sintering due to shrinkage associated with structural phase changes during cooling [9].

The technique also addresses limitations associated with other TBC methods in terms of the complexity of the surface geometries that can be coated and also the total coating area. The spray and sintering streams can be easily manipulated and controlled to coat surfaces out of direct line of sight, such as internal bend sections of pipes, and is limited only to areas that can be accessed by the spray and sintering streams. The process does not require isolated or evacuated environments and can be used to coat large areas in a more cost effective manner in comparison to other coating methods

## EXAMPLES OF SLURRY BASED THERMAL BARRIER COATINGS

Below we describe several examples of TBC fabricated using the developed manufacturing technique, which can be seen in Fig. 2.



(a) MonoLayer Coating (b) FG – Multilayered Coating

Figure 2. Cross-sectional view of a Slurry Sprayed TBC's

An example of a monolayer coating, fabricated utilising the Slurry Spray Technique, can be seen in Fig. 2(a). The composition of the monolayered coating is 50% ZrO<sub>2</sub> –

50% Ni, which can be distinguished by the different grain structure. The nickel substrate can be seen on the left hand side of Fig. 2(a, b), and the epoxy resin on the right hand side on the image; the epoxy resin was used to set the TBC specimens for SEM investigations. In Fig. 2(b) an example of a Functionally Graded (FG) – Multilayered Coating can be seen. This coating consists of 2 layers, with the initial layer of the FG-TBC composition consisting of 50%  $ZrO_2 - 50\%$  Ni, and the top layer of the FG-TBC consisting of 100%  $ZrO_2 - 0\%$  Ni. In Fig. 2(b), the 50%  $ZrO_2 - 50\%$  Ni layer is more pronounced than the monolayer coating seen in Fig. 2(a), this is due to the mechanical densification of the coating during the fabrication of the FG-TBC [10].

## **EXPERIMENTAL RESULTS**

#### Adhesion Test

Experiments were conducted for different compositions of the TBC components (zirconia and nickel). The purpose of these tests was to investigate the effect of the composition versus the adhesive strength of the fabricated coating. The outcomes of the experimental investigation of the monolayered coatings are presented in Fig. 3.



Figure 3. Adhesion strength of TBC versus zirconia contents

From Fig. 3 it can be seen that TBC with volume fraction of zirconia less than 40% has zero adhesive strength and generally spalls or delaminates during and after the sintering process. An example of coating delamination is shown in Fig. 3 above.

The adhesion strength increases almost linearly and peaks with the maximum adhesion strength of 9 MPa at composition of 50% ZrO<sub>2</sub> – 50% Ni. However the percentage of the ceramic/nickel composition of the base layer is restricted by certain constraints. These constraints include the mismatch of thermal expansion properties, a source of thermal stress, and hence lead to the spallation of the coating during the stages of fabrication. Moreover if the content of slurry mixture exceeds 50% ZrO<sub>2</sub> a flocculated slurry is produced, introducing surface irregularities during application of the coating. These surface irregularities will create problems such as uneven pressure application during pressure stamping stage of the fabrication technique. This will introduce stress concentrations within the surface of the coating, which will eventually lead to cracking and spallation of the TBC during the sintering process. The outcomes of the testing the graded coatings as well as the comparison of the adhesive strength are presented in Fig. 4.



Figure 4. Adhesion strength range for various types of coatings

Adhesion tests of the adhesion strength of the various TBC compositions were carried out with the PosiTest pull off adhesion tester and results showed that FG-TBC (2 layers) were a 100% improvement from the monolayered TBC with 100% of  $ZrO_2$  and 25% improvement from the monolayered TBC with 50%  $ZrO_2 - 50\%$  Ni (Fig. 4). The maximum adhesion strength obtained through the experiment, for the FG-Coating was approximately 11 MPa. However in comparison with existing coating techniques such as the flame spray method, with adhesion strength of approximately 21 MPa, the adhesion strength of the FG-TBC produced is 50% lower than the flame spray method [11].

## **Thermal Cycling Test**

The purpose of the thermal cycling tests were to give an indication of thermal fatigue behaviour of the slurry based TBC [12]. The maximum temperature reached was 900°C, with a 30 minute heating/cooling cycle. Monolayered TBC with 100% of  $ZrO_2$  and graded coating with two layers of 100%  $ZrO_2 - 0\%$  Ni and 50%  $ZrO_2 - 50\%$  Ni were subjected to the thermal cycling.

The experimental study showed that the majority of the failures occur during the first few cycles (see Fig. 5 and 6). If the coating survived the first 4-5 thermal cycles, the coating integrity is preserved throughout the following thermal cycling. Fig. 5 shows the ratio of the surviving samples to the total number of the tested samples for different sintering methods and Fig. 6 demonstrates that the FG-Coating is normally much more durable and better resistant to the thermal cycling. The effect of the first few cycles can be explained by the manufacturing defects, which lead to the almost immediate failure of the coating. It is clear from the obtained results that the oven sintering; similar to what has been found from the adhesive tests. The FG-Coating has much lower probability of failure during the thermal cycling. This can be explained by the lower mismatch in material properties and the lower level of thermal stresses during the sintering of the TBC.



Figure 5. Ratio of the survived TBC to the total number of tested samples for oven and torch sintering



Figure 6. Ratio of the survived TBC to the total number of tested samples for functionally graded and monolayered coatings

The results obtained through the experiment coincide with theoretical analysis presented in literature reviews where TBCs are expected to perform better in real life application if manufactured in a controlled sintering and cooling environment. Firstly, by applying constant heat flow, uniform heat expansion, and ideal boundary grain growth between particles is achieved thus reducing thermal stress induced during the thermal expansion process. Secondly, a slow cooling rate after sintering effectively reduces the strain and stress associated with rapid cooling. Thirdly, introduction of FG-Coating induces a temperature gradient across the coating hence minimising thermal mismatch due to cooling and the resulting residual stresses.



(c)

Figure 7. Microstructure of FG-TBC after fabrication (a), 10 thermal cycles (b) and 20 thermalcycles (c), magnitification – 500x.

From SEM images taken after the fabrication, 10 and 20 thermal cycles (see Fig. 7), it can be seen that the increase of porosity with the number of thermal cycles and formation of crack damage, eventually propagates through the thickness and lead to the failure of the coating.

## CONCLUSION

This paper presents results of an experimental study on the mechanical properties and fracture of TBCs fabricated using a new method based upon the Slurry based TBC technique. The main advantages of this technique are the low costs and the ability to cover large and curved surfaces, which are critical for a number of important practical applications. This technique allows the fabrication of the multilayered FG-Coatings, which can significantly reduce the thermal stresses and, and as a rule, have higher durability and lower failure rates in comparison with monolayered TBCs.

The test results demonstrate a satisfactory adhesive strength of the coating, which is comparable with the adhesive strength of other coatings, fabricated using traditional techniques such as the Flame Spray method. The outcomes of the experimental study also showed that the functionally graded coatings fabricated using the Slurry Spray technique are able to survive low-cycle thermal excursions, when the temperature increases up to 1000°C. Further work will focus on real-life applications, such as high temperature burner tests and leading edge of scramjet propulsion systems.

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