SLURRY SPRAYED THERMAL
BARRIER COATINGS FOR
AEROSPACE APPLICATIONS

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CHAPTER 4

CHARACTERISATION OF THERMO-MECANICAL PROPERTIES OF TBC
4.1 Introduction

The integrity and durability requirements for Thermal Barrier Coatings (TBCs) to be used in high temperature applications are increasing rapidly. This raises the need for a comprehensive assessment of the thermo-mechanical properties and longevity of the TBCs. Full-scale test setups have been utilised in several previous studies [Braun, Leyens and Fröhlich, 2005; Brunner, Rose, Mühlbauer and Abhari, 2008], however, the cost of full-scale testing is usually very high, and such testing cannot provide detailed information on the ‘weakest link’ in the coating performance or manufacturing procedure.

The use of small-scale laboratory studies offers a cheap alternative to full-scale testing. These studies can be aimed at a comparative assessment of TBCs or provide a broad range of thermo-mechanical data, which can be linked to the weakest link in the manufacturing process or application performance. This, in its turn, can be used to improve the operating performance of TBCs to increase its reliability and longevity. In addition, the laboratory studies are normally more versatile and far less time-consuming than full-scale testings.

This chapter is focused on the development of a set of experimental methods for quantitative assessment of various mechanical and thermal characteristics of thin coatings. In particular, these experimental methods aim to provide quantitative characteristics of the porosity level, microhardness, adhesion strength, thermal conductivity and thermal cycling resistance. All these testing methods are based on the well-known physical or mechanical principles and utilise standard or specifically designed testing equipment.

The obtained experimental information was utilised in the previous Chapter to extend the Wet Powder Spray (WPS) and develop a new manufacturing procedure which is able to fabricate a good quality TBC including Functionally Graded Thermal Barrier Coatings (FG–TBCs). The testing methods presented in this Chapter also serve as a tool for a comparative assessment of the quality of the coatings manufactured with
other various coating deposition techniques including those currently recommended as an industrial standard for manufacturing TBCs.

This chapter provides a brief outline of the experimental methods and focuses on the quantitative and comparative assessments of the thermo-mechanical properties of the mono-layered and multi-layered coatings fabricated with the Slurry Spray Technique (SST). The analysis of the experimental results clearly demonstrated that many of the mechanical properties and characteristics of the thermal barrier coatings fabricated with SST, including durability and integrity characteristics, are comparable to TBCs manufactured with the standard coating deposition techniques. The latter provides confidence in the future development and implementation of the new SST for a wide range of industrial applications.
Chapter 4  Characterisation of Thermo-Mechanical Properties of TBC

4.2 Porosity Measurements

4.2.1 Introduction

The level of coating porosity is an essential characteristic for the determination of the quality of TBCs. It influences various physical properties of the coating such as the thermal conductivity, thermal expansion coefficient as well as other important characteristics, including fracture resistance and the ability to withstand aggressive environment (corrosion) [Khor and Gu, 2000]. Normally, a higher level of porosity reduces the thermal conductivity of the TBCs, which is critical for thermal protection of metallic load-bearing structures when subjected to high temperatures [Rogé, Fahr, Giguère and McRae, 2003; Sumirat, Ando and Shimamura, 2006]. High levels of porosity (above 10 – 15%) can also significantly degrade the integrity of the TBC and decrease the adhesion strength between the coating and substrate [Portinha, Teixeira, Carneiro, Martins, Costa, Vassen and Stoever, 2005]. In this section, the method for the porosity measurements is briefly described followed by the experimental results related to the effect of the sintering parameters on the porosity level of the coating. These results were utilised in the development of the Slurry Spray Technique (SST) and determination of the optimal fabrication parameters as described in Chapter 3.

4.2.2 Experimental Setup and Procedure

The examination of the TBC surface porosity abides by ASM standard E2109-01, Test Methods for Determining Area Percentage Porosity in Thermal Sprayed Coatings. As described in the standard, the testing method utilises equipment which is able to produce microscopic images with inverted pictures in order to distinguish the pore areas as well as its dimensions.

The microstructure and porosity of the TBCs were examined by the candidate using
Chapter 4  Characterisation of Thermo-Mechanical Properties of TBC

the Philips XL 20, Scanning Electron Microscope (SEM), which is shown in Figure 4.1. The Philips XL 20 SEM has a maximum resolution of 4 nm and a maximum magnification of 50,000.

Figure 4.1: Philips XL20 Scanning Electron Microscope (SEM)

The experimental procedure consisted of two main stages (Specimen Preparation and SEM Examinations), which are briefly outlined below.

**Specimen Preparation**

For SEM imaging, specimens were required to be electrically conductive at the surface and grounded. Metal specimens require minimal preparation for SEM imaging, as they are highly conductive. Coating material such as ceramics; may cause scanning faults, due to the non-conductive nature of the ceramics-based material. However, there are various ultra thin coatings that are electrically conductive, which are used to cover the ceramics specimens for SEM examinations. The coating material can be, for example, carbon, carbon/gold and platinum. The platinum coatings have a much finer structure than the gold coating, and is used for high resolution imaging (up to 10 000 x). However, the samples are unable to be applied evenly with the platinum coating due to the dimensions of the specimen. As a
result, the platinum coatings were inapplicable for the SEM examinations as they would disturb the image scans. Therefore, for the proposed porosity measurements of TBCs, the carbon coatings were applied by the Adelaide Microscopy.

**SEM Examinations**

The porosity test method is based upon ASTM standard E2109-01. The experimental examinations utilised ‘Porosity Figures’ from the Standard. The images from this Standard were used for the quantitative assessment of the level of porosity from various locations of the surface of the TBCs specimens by matching the SEM images with the ‘Porosity Figures’ given in the Standard, which can be seen in Appendix C. A typical SEM image used for investigation of the cross sectional porosity of the TBCs is shown in Figure 4.2.

![Figure 4.2: Cross section of a spalled coating](image-url)
The procedure included the following steps:

- Bring the examination chamber of the SEM to atmospheric pressure.
- Place the prepared specimen into SEM chamber and secure into position with Allan key pressurise the chamber.
- Observe the image of specimen on the viewing screen or video monitor.
- Select the magnification, this allows the resolution of the voids and pores, which fills the screen with the entire coating thickness.
- Compare the SEM images with the figures from ASTM E2109-01, as shown in Appendix A. The SEM image and the figures should have approximately the same size with a minimum image area of 9 x 11 cm.
- Record the value of the porosity on the figures that best matches the SEM image. If image does not match a figure, it may be rounded to the nearest whole number between the figures values. The following limitations are adopted in the assessment of the porosity level:
  - If SEM image exhibits less than 0.5 % porosity, it is reported as < 0.5. These fields should be considered zero when computing the average area percentage porosity for the specimen.
  - If SEM image exhibits more than 15 % porosity, the SEM image is recorded outside the range along with a numerical value representing the operator’s estimation of the percentage porosity.
  - Once examinations have been completed, the final step: depressurise the chamber and removing the specimen from the chamber.
4.2.3 Results and Discussion

The following set of experiments was conducted to investigate the influence of the sintering parameters on the level of porosity of the fabricated TBCs. As explained in Chapter 2 and Chapter 3, the sintering methods are, probably, one of the major factors affecting the level of porosity.

Specimens for the porosity measurements of TBC represented a mono-layered ceramic (ZrO$_2$) coating deposited on 50 x 50 x 3 mm Nickel plate substrate. The basic sintering parameters, such as the method of sintering and sintering time were set different for different groups of specimens (with 3 specimens in a group). The specimens from each group were investigated using SEM as described in the previous section to study the effect of the sintering parameters on the porosity along the coating surface. Along with these studies, the sintering parameters were examined, which led to the highest quality of the coating with the lowest level of porosity and absence of structural defects (cracking and spallation).

The summary of the experimental studies on porosity measurements is given in Table 4.1. The corresponding SEM images of the surface of the fabricated specimens from various groups are shown in Figure 4.3 to Figure 4.5. These figures provide microscopic images with various magnifications ranging from 20 to 500 times.

<table>
<thead>
<tr>
<th>Specimen Group</th>
<th>Parameters of sintering stage</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sintering Method</td>
</tr>
<tr>
<td>1</td>
<td>Automated sintering platform</td>
</tr>
<tr>
<td>2</td>
<td>Automated sintering platform</td>
</tr>
<tr>
<td>3</td>
<td>Automated sintering platform</td>
</tr>
<tr>
<td>4</td>
<td>Oven</td>
</tr>
</tbody>
</table>
Low magnitude SEM examinations

SEM images of the TBCs, at 20 times magnification, are shown in Figure 4.3, where the difference in the quality of the coating between the various groups of specimens can be clearly observed. These differences include the various levels of visible porosity and different levels of structural defects such as cracking, and spallation.

SEM images of a typical specimen from group 1, showed no cracking on the coating surface, as seen in Figure 4.3a. Despite the absence of cracking on the coating surface, a significant amount of coating porosity can be observed from these images.

In SEM images of a typical specimen from group 2 (with the sintering time reduced from 30 minutes to 20 minutes), notable cracks were observed along the coating surface as shown in Figure 4.3b. However, the porosity of the coating surface was significantly lower than for specimens from group one.

With the specimens from group 3, the height of the oxy acetylene torch was reduced from 300 mm to 150 mm, which led to a higher heat flux and higher sintering temperatures. SEM images of a typical specimen for this group show that the coating spallation takes place as well as formation of large discontinuity areas on the coating surface. The coating surface represents a sporadic and porous surface, as seen in Figure 4.3c.

Specimens from group 4 were sintered utilising a high temperature oven instead of the automated sintering rig. The SEM images show a smooth continuous surface coating, with low levels of porosity, as shown in Figure 4.3d. No cracking or spallation was observed for all specimens from this group.

From the examination of the low magnification SEM images, it was concluded that, as expected, the sintering parameters have a substantial effect on the porosity and overall quality of the coating. A more detailed SEM image examination with a greater magnification was conducted in the following section to further investigate the porosity and micro-structure of the deposited coatings.
Porosity Size Examination

The SEM images of 100 times magnification are shown in Figure 4.4. At this magnification, a considerably higher level of microstructural features of the coating surface can be observed and the qualitative assessment of the characteristic pore size of the fabricated TBCs can be conducted. Before the quantitative assessments, it can be noted from the SEM images shown in Figure 4.4 that the second group of specimens has the lowest porosity level, while the largest level of porosity corresponds to group three.

For group 1 the typical pore size normally ranges from 36.0 to 58.8 μm, with an
average pore length of around 46 μm. For specimens from group 2, the pore size ranges from 53.6 μm to 58.8 μm with an average size around 56 μm. This group of samples is also characterised by significant cracking defects as discussed above.

Specimens from group 3 failed during the fabrication stages, with spallation of the entire coating surface, as shown in Figure 4.4c. The specimen coating spallation was accompanied with the formation of large-size pores ranging from 115 μm to 152 μm, with an average pore length of 134 μm.

Specimens from group 4 have the smallest size of pores, as observed in Figure 4.4d. The pore size of the coating ranges from 22.6 to 34.6 μm with an average pore length of approximately 29 μm.
Porosity characterisation

The plane view SEM images of the coatings at 500 times magnification are shown in Figure 4.5. These high magnification images were used to determine the level of surface porosity of the TBCs fabricated with SST.

SEM images of a typical specimen from group 1 are shown in Figure 4.5a. From the examination of the specimens, the TBC showed good coalescence between the coating powder particles. However, a moderate level of coating porosity was observed, with a value obtained from the figures and assessed in accordance to the Standard, of approximately 2%.
SEM images of the surface of a typical specimen from group 2, as shown in Figure 4.5b, where the specimens were observed to exhibit low levels of coalescence between the ceramics particles. This was attributed to insufficient supply of thermal energy to the surface to promote the coalescence between ceramics particles similar to what was found in [Liu, Gao, Tao, Zhou and Luo, 2009]. As a result, the specimens from group 2 had a significantly higher level of porosity than the specimens from group 1. Using the same standard method, the level of porosity was found to be higher than 3%.

The high magnification SEM images of a typical specimen from group 3 are shown in Figure 4.5c. As mentioned above, the surface of these specimens was covered with large crack-like defects and large pores, which led to extremely high porosity levels assessed to be above 12%. One of the reasons for such poor quality could be a very high variation of the sintering temperature distribution within the specimen. In accordance with the flame appearance, the surface temperature of the specimen was estimated to be around 1500°C in the central region of the torch, while at the periphery regions just only 1000°C. For such differences in temperatures, the generation of very high thermal gradients as well as high thermal and residual stresses, this could lead to material phase changes in localised areas of the coating. Presumably, these thermal and residual stresses led to the extensive cracking and spallation phenomena similar to those observed in [Rogé, Fahr, Giguère and McRae, 2003], and, finally, to the complete failure of the coating as shown in Figure 4-5c.

The high magnification SEM image of a typical specimen from group 4 is shown in Figure 4.5d. The ceramic coating had a good coalescence between the coating powder particles, in comparison with the previously examined groups of specimens. This high level of coalescence achieved during sintering in a high temperature oven produced a relatively low level of porosity, which was assessed, based on the Standard described above, to be less than 0.5%.
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Figure 4.5: Plane surface SEM images of Specimen 1, 2, 3 and 4 for 500x magnification

**Distribution of Coating Surface Porosity**

The SEM images examined in the previous section, determined the coating porosity for the various areas of the specimens. However, due to different temperature and thermal flux conditions, to have homogeneous properties, including the level of porosity, across the whole coating area of the specimen are not possible. Therefore, this present section is focused on micro-examinations of the distribution of coating porosity. The following investigations are related to group four producing the best (lowest porosity and absent of structural defects) quality of the coating. The locations...
of a specimen from group 4, where the coating porosity was examined, are shown in Figure 4.6.

Figure 4.6: Points for surface porosity determination

The results of the investigation of the porosity distribution throughout the coating surface are shown in Figure 4.7. Examination of the SEM images shows the highest level of coating porosity (0.5%) was found in the centre of the specimen (point 1). For the remaining areas (points 2 -5) on the surface of the specimen, the level of coating porosity ranged from 0.1% to 0.3%.

The main factor responsible for variation of the porosity over the surface area of the specimen was the variation of the sintering temperature and pressure. The variation in the coating porosity can be seen in Figure 4.7. Other contributing factors to the spatial variation include the overlapping of deposited powder layers, as well as formation of pockets of trapped air during the powder spraying procedure, as described in Chapter 3.
Figure 4.7: Porosity distribution across coating
4.2.4 Summary

The examinations of the porosity of TBC specimens demonstrated that the sintering method has a large influence on porosity level and mechanical damage formation in coatings during the fabrication stage. The lowest level of coating porosity and minimal mechanical damage was achieved with the use of the high temperature oven. The porosity level had small amounts of variation across the specimen surface which did not exceed 0.5%. Such values of porosity, can be considered as satisfactory if compared with the typical values of this TBC quality characteristic achieved with other fabricating techniques such as High Velocity Oxy Fuel and Atmospheric Plasma Spray (where the porosity level varies from 0.1% to 0.5%).

An application of the oxy-acetylene torch as a thermal source for the sintering of the coating powder, generally led to a higher porosity and more significant levels of mechanical damage of the TBC. Typical values of the porosity obtained with oxy torch utilisation ranged from 2% to 12%, which are significantly higher than those obtained with the high temperature oven. In addition, the oxy torch sintering method produced a large variation of the porosity over the specimen surface, which was explained by the large differences of temperature during the sintering stage. Therefore, the development and improvement of the quality of SST was mainly focused on the use of the high temperature oven as a sintering method for SST.
4.3 Microhardness Measurements

4.3.1 Introduction

Ductile material microhardness can be linked to the resistance of the material to the development of plastic deformation [Degarmo, Black and Kohser, 2003]. For brittle materials such as ceramics, the hardness measurements normally associated with resistance to the initiation and propagation of micro-cracks, as the plasticity mechanisms are negligible at micro-indentation [Zhou, Li, He, Wang and Sun, 2008]. Therefore, microhardness examinations are of a particular importance for TBCs as this mechanical characteristic can be strongly linked to the fracture toughness and fracture resistance of the coatings to the initiation and propagation of brittle cracks. The microhardness of thin coatings is usually determined from static tests where indentation loads do not normally exceed 10 N. The most common types of microhardness tests are Vickers and Knoop methods. Vickers microhardness method uses a diamond shaped pyramid indenter, while Knoop’s technique utilises an elongated diamond pyramid shaped indenter. The following section overviews both Vickers and Knoop microhardness indentation techniques.

Vickers microhardness value is given by the equation

\[
HV = \frac{2F \sin(\theta)}{L^2},
\]

where the Vickers microhardness value, \(HV\) [N/mm²], is determined by the loading force, \(F\) [N], the average length of the diagonals, \(L_1\) and \(L_2\) [mm], and angle between opposite ends of the diamond (136°), as shown in Figure 4.8.

Therefore, the above equation prescribes the microhardness value of the coatings based upon the loading parameters \(F\), shape of the indenter tip \(\theta\) and the characteristics of the surface area of the impression.
The Knoop microhardness is given by the following equation

$$KHN = \frac{F}{CL^2},$$

where Knoop microhardness value, $KHN$ [N/mm$^2$], is determined by the loading force, $F$ [N], the measured length of the diagonals, $L$ [mm], and the constant related to the residual area of indentation, $C = 0.07028$, as shown in Figure 4.9.

Equation (4.2) determines the microhardness value of the coatings based upon only the loading parameters ($F$) and the length of the residual and the surface area of the residual cavity.

The Knoop indentation method is particularly useful for narrow shaped specimens or when the microhardness tests need to be closely spaced or conducted at the edge of the specimen. With these limitations, Knoop test method is unable to be used for TBCs microhardness testing. At the same time, Vickers microhardness tests are more commonly used in industry, providing comparative data which is considerably less
sensitive to surface conditions [Gatto, 2006; Hvizdos, Jonsson, Anglada, Anné and Van Der Biest, 2007]. Therefore, the Vickers approach was selected by the candidate for microhardness examinations of TBCs.

![Knoop hardness indenter for micro hardness testing](image)

Figure 4.9: Knoop hardness indenter for micro hardness testing

The microhardness examinations were conducted for mono-layered as well as for multi-layered TBCs fabricated with the Slurry Spray Technique (SST).

### 4.3.2 Experimental Setup and Procedure

The Vickers microhardness indenter utilised in the experiments was a LECO LM 700AT situated at the University of Adelaide. The microhardness indenter has a load ranging from 1 to 1000 gm, with the loading time varying from 5 to 99 seconds. For microhardness investigations of TBCs fabricated with SST, the load varied from 0.1 N to 0.3 N as shown in Figure 4.10. This range of loads was selected to avoid the effect of the microstructure of the coatings [Berndt and Lenling, 2004]. The loading
time was set at 5 seconds in accordance with ASTM standards. The Vickers microhardness tests also complied with the industry accepted ASTM standard E384-09, Standard Test methods for Micro indentation Hardness of Materials.

![A Vickers microhardness machine](image)

Figure 4.10: A Vickers microhardness machine

A single test measurement was averaged over a minimum of 5 indentation results to provide a valid value of the microhardness. The locations of indentation were at least 5 times the diagonal distance apart to exclude the effect of residual stresses left from the previous tests [Limarga, Widjaja and Yip, 2005; Zhou, Li, He, Wang and Sun, 2008].

The following table summarises the composition of the TBCs fabricated with the Slurry Based TBCs used for microhardness examinations. As mentioned earlier 5 tests for each specimen were conducted to obtain a valid result.
Table 4.2: TBCs fabricated using the SST for microhardness investigations

<table>
<thead>
<tr>
<th>No. Layers</th>
<th>Section</th>
<th>ZrO₂ (%)</th>
<th>Ni (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single</td>
<td>Top</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>Double</td>
<td>Top</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>Base</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>Top</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>Triple</td>
<td>Mid</td>
<td>75</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>Base</td>
<td>50</td>
<td>50</td>
</tr>
</tbody>
</table>

4.3.3 Results and Discussion

The results of investigations into the Vickers microhardness of the coating are presented in the following figures and tables. Figure 4.11 shows the results of the examination of the microhardness for a single, double and triple layered TBCs plotted against the coating thickness. Table 4.3 contains average values of the microhardness of the tested specimens, and average thickness of the coating. The microhardness of the triple layered TBCs has an average microhardness value of 14.5 HV. For double layered TBCs, the average microhardness is 16.1 HV and for single layered TBCs, the average microhardness is 19.0 HV.
From the results shown in Table 4.3, the microhardness of the TBCs was observed to decrease with the increase of the coating thickness. The largest microhardness value (19 HV) was measured for single layered coatings, which were closely followed by double (16.1 HV) and triple layered coatings (14.5 HV).

Table 4.3: The average of Vickers microhardness values for single, double and triple layered slurry based TBCs.

<table>
<thead>
<tr>
<th>No. Layers</th>
<th>Microhardness, HV$_{100}$</th>
<th>Coating Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average</td>
<td>Standard Deviation (2σ)</td>
</tr>
<tr>
<td>Single</td>
<td>19.0</td>
<td>9.0</td>
</tr>
<tr>
<td>Double</td>
<td>16.1</td>
<td>4.1</td>
</tr>
<tr>
<td>Triple</td>
<td>14.5</td>
<td>4.4</td>
</tr>
</tbody>
</table>
Higher residual stresses in single layered TBCs are one of the possible reasons for the higher microhardness observed in the micro-examinations for this type of coatings [Zhou, Li, He, Wang and Sun, 2008]. Graded coatings normally provide smoother variation of thermal and mechanical properties, which resulted in considerably lower levels of residual (and induced thermal) stresses.

For comparison purposes, Figure 4.12 presents the typical range of microhardness of TBCs fabricated with the Electron Beam Physical Vapour Deposition (EB–PVD) and Atmospheric Plasma Spray (APS) techniques [Lima, Kucuk and Berndt, 2001; He, Li, Zhou, Dai and Sun, 2007; Zhou, Li, He, Wang and Sun, 2008]. The TBCs fabricated with EB–PVD and APS had disk shaped specimens with the same composition of 8 wt. % of Y₂O₃ (Yttria Stabiliser) and 92 wt. % of ZrO₂ (Zirconia) for the topcoat with a layer of a NiCrAlY bond coat placed between the TBCs and titanium alloy substrate.

![Figure 4.12: Vickers microhardness of coating fabricated by EB–PVD, APS and SST. Error bars represent standard deviation (2σ) for all experiments](image_url)

The Vickers microhardness of APS, EB–PVD and SST are 547, 406 and 18 HV respectively. From the comparison of the SST to the values found in literature, it is evident that the TBCs fabricated with the SST have a significantly lower
microhardness than EB–PVD and APS based coatings. The microhardness of TBCs fabricated with the APS is normally lower than EB–PVD; however, the typical values are still comparable. Similar to EB–PVD, APS technology was able to produce a high quality coating; however, the costs of fabrication of these coatings were significantly higher in comparison with SST.

The conducted microhardness results clearly demonstrate that the SST can not be used in applications where the microhardness of the coating is an important issue for adequate performance and operation. These include various conditions when the coating can be in contact with other solid parts or particles, or be subjected to a long-term exposure of a heating flow. However, in many other applications where high hardness is not required, the SST can still be considered as a cheap alternative to the above considered fabricating techniques.

The low microhardness of TBCs fabricated with SST can be partially attributed to the relatively high porosity and low temperatures used by the oxy-torch and oven for the sintering method, as well as the relatively short sintering times used to produce the coatings. The thermal energy supplied from these sources was not sufficient for the formation of a dense coating and the promotion of particle diffusion between the coating and the substrate, which can help achieve a high adhesive strength between the coating and substrate.

### 4.3.4 Summary

In this section the Vickers microhardness of TBCs fabricated with the SST was examined. It was shown that the microhardness result decreases with the increase of the TBCs thickness and the maximum value was for a single layered coating of 19 HV.

The typical values of microhardness of TBCs fabricated with SST were almost of an order of magnitude lower than the values for the similar coatings fabricated with other deposition techniques. This was explained by the relatively low sintering
temperatures and sintering times used for SST in comparison with other fabricating techniques.

These microhardness results clearly demonstrate that the SST at the current stage of development cannot be used in applications and situations where the microhardness of the coating is one of the prerequisites for the normal operation of the structural component. These include various conditions where the coating can be in contact with other solid parts or particles. However, in other applications where a high microhardness is not required, the SST can still be considered as a low cost alternative to the aforementioned fabricating techniques.


### 4.4 Adhesion Strength

#### 4.4.1 Introduction

The adhesion strength of Thermal Barrier Coatings (TBCs) provides a good indication of the resilience of the coating to delamination and spallation during operation. The adhesion strength quantifies the strength of cohesion within the coating material as well as the strength of bonding between the coating and substrate.

A Functionally Graded Thermal Barrier Coating (FG-TBC) consists of several layers with varied compositions of metal and ceramic. The adhesion strength between each coating layer as well as at the metal interface can be dissimilar and significantly affect the overall adhesive strength of TBCs [Markocsan, Nylen, Wigren, Li and Tricoire, 2009]. Defects such as cracking, flaking and delamination may occur throughout the fabrication process, for example, during the sintering stage. These defects can also deteriorate the overall adhesive strength and lead to the failure of the coating [Thiery, Poulain, Dupeux and Braccini, 2009]. The adhesion strength of TBC is greatly influenced by the substrate surface characteristics such as surface roughness and surface contaminates. The adhesion strength is also affected by coating fabrication parameters and environmental conditions, such as humidity; and temperature [Lima and Guilemany, 2007; Waki and Kobayashi, 2008].

A quantitative assessment of the adhesive strength is normally based on the tensile adhesion pull-off test and the following equation

\[
\sigma_{\text{adh}} = \frac{F_f}{A_{\text{TBC}}} \quad (4.3)
\]

where the adhesion strength, \( \sigma_{\text{adh}} \) [MPa], is determined by the nominal failure area, \( A_{\text{TBC}} \) [m\(^2\)], and the applied tensile load in the direction normal to the interface surface, where it reaches maximum value just before the failure occurs, \( F_f \) [N].
Shear adhesive strength is determined using a similar equation:

\[
\tau_{adh} = \frac{T_f}{A_{TBC}}
\]  \hspace{1cm} (4.4)

where the shear force, \( T_f \) [N], is applied in the parallel direction to the interface surface.

In this section, the effect of various fabricating parameters on the pull-off adhesion strength of TBCs manufactured with the Slurry Spray Technique (SST) will be presented and discussed in some detail.

In the tensile adhesion pull-off test method the coated substrate adhered to a sand blasted uncoated counterpart, is tested in tension using either a universal test machine (as described in ASTM C633) or a portable testing device (the full procedure is described in ASTM D4541). The test is completed when the coating fails and no longer able to resist to the tensile loading. The adhesion strength is determined from the maximum value of the tensile load that is recorded during the tests. Typically, the coating failure can occur between the TBC layers, the substrate, and the bottom layer or at the counter part as shown in Figure 4.13, [Martena, Botto, Fino, Sabbadini, Gola and Badini, 2006]. Those failures in the adhesive layer or between the counterpart and substrate are considered as an unsuccessful test and new counterpart materials or adhesive should be used to obtain a valid value for the adhesive strength of TBCs.
A number of factors may affect the measured property of the TBC from such tests. The first and the most important factor is the misalignment issues with the tensile cell and the coating base, due to, for example, the variation of the coating thickness. The misalignment can create a bending moment leading to an additional stress on the side of the specimen, which produces a significant error in adhesion strength measurements. Other possible sources for the measurement errors are the change in the environmental conditions, such as temperature and humidity. It is well known that these factors could influence the adhesive strength in various situations [Bergant and Grum, 2009].

A portable pull-off adhesion device was selected as an inexpensive and flexible way to conduct the experimental study. The main advantage of the portable device (PosiTest pull-off tester) used in the adhesion tests is the self aligning actuator, eliminating errors from misalignment, which is the major issue in such tests. The other advantage is the limiter embedded to the control system. It controls the rate of
loading, which makes tests repeatable and minimises errors during the experimental study. The use of such devices for the adhesive strength measurements as well as the experimental procedures is guided by ASTM standard D4541.

### 4.4.2 Experimental Setup and Procedure

Adhesion tests were undertaken by the candidate using a PosiTest pull-off adhesion tester from the DeFelsko Corporation. This test apparatus abides to the various international standards including the ASTM D4541. The pull-off adhesion tester consists of an actuator connected to a hydraulic pump and digital display. A metal dolly is adhered to the coating surface, using a standard adhesives recommended by ASTM D4541. The metal dolly is then connected to a pressurised actuator, which generates tensile load for the adhesive strength test. In accordance with the manufacturer specification, the error in the determination of the adhesive with this test equipment is normally limited by one percent. The PosiTest pull-off adhesion tester, with self aligning actuator, dollies and hydraulic pump is shown in Figure 4.14.
Adhesion strength tests were conducted for a range of TBCs with various metal and ceramic compositions, to investigate the effects of the adhesion strength on the fabricated coating. The thickness of TBCs ranged between 100 - 150 μm. Table 4.4 details the compositions of the mono-layered coatings, which were subjected to the adhesion strength tests.
### Table 4.4: Testing regime for base layered TBCs

<table>
<thead>
<tr>
<th>Specimen Group</th>
<th>ZrO$_2$ (%)</th>
<th>Ni (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25</td>
<td>75</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>3</td>
<td>45</td>
<td>55</td>
</tr>
<tr>
<td>4</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>6</td>
<td>66</td>
<td>33</td>
</tr>
<tr>
<td>7</td>
<td>100</td>
<td>0</td>
</tr>
</tbody>
</table>

#### 4.4.3 Results and Discussion

Figure 4.15 shows the adhesion strength of mono-layered coating fabricated with SST with the percentage of the ceramic fraction in the composition of the TBC. The graph also shows that TBC with a volume fraction of ceramics (% ZrO$_2$) less than 40% wt., has virtually zero adhesive strength. For such TBC compositions, the specimens normally have large manufacturing defects between the coating and substrate. Therefore, during the adhesion strength tests, a very small force is required to fully separate the load bearing area and the failed specimen.
Figure 4.15: Adhesion strength of base layered TBC against Zirconia content. Error bars represent standard deviation ($2\alpha$) for all experiments.

The numerical values of the adhesion strength for each specimen group are presented in Table 4.5. The adhesion strength increases almost linearly and reaches the maximum value of 8.9 MPa for specimen group 4, which have composition of equal weight fraction of ceramics and metal powder. After the maximum adhesive strength, is closely followed by a gradual decrease in the adhesion, as the ceramic content of the coating increases. Specimen group 5 and 6 were found to have an adhesive strength of 8.2 and 7.2 MPa, respectively.
Table 4.5: Base layered adhesion strength for various coating compositions

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Adhesion Strength, MPa</th>
<th>Standard Deviation (2σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>1.9</td>
</tr>
<tr>
<td>4</td>
<td>8.9</td>
<td>1.3</td>
</tr>
<tr>
<td>5</td>
<td>8.2</td>
<td>2.2</td>
</tr>
<tr>
<td>6</td>
<td>7.2</td>
<td>2.5</td>
</tr>
<tr>
<td>7</td>
<td>4.2</td>
<td>1.4</td>
</tr>
</tbody>
</table>

The examination of the adhesion strength of the base layered coatings for various slurry compositions provided data on the relationship between the adhesive strength and the coating composition. This allowed the determination of the optimal coating composition to achieve the maximum adhesive strength. Furthermore, comparisons were conducted between the adhesive strength of mono-layered TBCs with corresponding values published in literature for other manufacturing techniques. These include the Atmospheric Plasma Spray (APS) technique, which is currently commonly used in many industrial applications.

A comparison of the adhesive strength of mono-layered TBCs manufactured using SST and APS [Lima and Trevisan, 1999], is shown in Figure 4.16. The thickness of the coatings fabricated by APS ranged from 100 to 250 µm and 50 to 150 µm for the specimens fabricated with the SST. Two compositions of the mono-layered coatings were examined for the adhesion strength, the first: 50% ZrO₂ – 50% Ni, and the second: 75% ZrO₂ – 25% Ni, and three specimens tested for each of the compositions.
Results of the average adhesion strength and standard deviation is shown in Table 4.6. From the test results, it is found that the average adhesion strength of the specimens fabricated with APS is 15.8 MPa for the specimens with 50 % of ZrO₂ and 14.5 MPa for the specimens with 75 % of ZrO₂. For the specimens fabricated with SST the values of adhesion strength for the mentioned coating compositions were 6.5 MPa and 8.9 MPa, respectively.

As expected, the adhesion strength of TBC fabricated with APS is higher than for coatings fabricated with the SST. The APS is able to generate very high temperatures, which is high enough to melt the ceramic powder, where the coating is formed as a result of the solidification process. This produces a strong bond between the coating and the substrate. The sintering process of the SST takes place under considerably lower temperatures and coating formation is controlled by the bonding energy and surface diffusion mechanisms. These mechanisms normally provide a lower adhesion strength in comparison with the bonds created by the particle diffusion process [Bernard-Granger, Monchalin and Guizard, 2008]. However, the values of the adhesive strength for the SST, especially, for the composition with lower concentration of the ceramic, are comparable.
Table 4.6: Base layered adhesion strength for various coating compositions and techniques

<table>
<thead>
<tr>
<th>Technique</th>
<th>Composition</th>
<th>Adhesion Strength, MPa</th>
<th>Standard Deviation (2σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>APS</td>
<td>ZrO₂ (%)</td>
<td>Ni (%)</td>
<td></td>
</tr>
<tr>
<td>75</td>
<td>25</td>
<td>14.5</td>
<td>6.9</td>
</tr>
<tr>
<td>50</td>
<td>50</td>
<td>15.8</td>
<td>5.9</td>
</tr>
<tr>
<td>SST</td>
<td>ZrO₂ (%)</td>
<td>Ni (%)</td>
<td></td>
</tr>
<tr>
<td>75</td>
<td>25</td>
<td>6.5</td>
<td>2.0</td>
</tr>
<tr>
<td>50</td>
<td>50</td>
<td>8.9</td>
<td>1.3</td>
</tr>
</tbody>
</table>

The previous results are related to the adhesive strength of the mono-layered base coatings. The results determined the optimal composition of SST to fabricate TBCs, leading to the maximum adhesive strength, as seen previously in Figure 4.15. The typical values of the adhesive strength of the mono-layered coatings of the SST and APS, were found to be quite close. For example, the adhesive strength of SST specimens lies within the standard deviation range of the adhesive values of APS specimens (Figure 4.16). Below, similar investigations were conducted for the multi-layered coatings.

A comparison of the adhesion strength of mono-layered and multi-layered coatings fabricated with SST are shown in Table 4.7. From this Table, the adhesive strength for multi-layered coatings was shown to be higher than for the single layered coatings. The same situation is typical for TBCs fabricated with other manufacturing techniques; which will be shown further on. This difference in the adhesive strength can be explained by the lower level of the residual stresses and the mechanical damage in graded coatings in comparison with the mono-layered TBCs. In general, the manufacturing defects are considered to be one of the major factors leading to reduction of strength characteristics for almost all mechanical systems [Kepets, Lu and Dowling, 2007].
Table 4.7: Comparison of adhesion strength for mono-layered and multi-layered TBCs produced by SST

<table>
<thead>
<tr>
<th>Layer No.</th>
<th>Composition</th>
<th>Adhesion Strength, MPa</th>
<th>Standard Deviation (2σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single 50</td>
<td>50</td>
<td>8.9</td>
<td>1.3</td>
</tr>
<tr>
<td>Single 100</td>
<td>0</td>
<td>4.2</td>
<td>1.4</td>
</tr>
<tr>
<td>Double 75</td>
<td>25</td>
<td>11.3</td>
<td>2.5</td>
</tr>
</tbody>
</table>

Figure 4.17 displays the adhesion strength of TBCs fabricated with SST, APS and Low Velocity Oxy Flame (LVOF) for multi-layered coatings [Lima and Guilemany, 2007; Bergant and Grum, 2009]. Coatings fabricated with APS and LVOF have a coating thickness of approximately 400 μm. Thicknesses of the graded coating specimens fabricated with SST were approximately 150 μm. The coating thickness of the SST was limited by the technological restrictions of the fabricating technique, as the attempts to produce a thicker coating normally led to cracking of the coating surface during evaporation and spallation failure during the sintering stage.

TBCs produced with APS have the highest average adhesion strength (22 MPa), which is followed by LVOF (18.2 MPa) and lastly, the SST (11.3 MPa). Despite that the average values for SST are approximately 100% lower than the APS and 40% lower than the LVOF, the adhesive strength for mono-layered and multi-layered coatings can be considered as acceptable; especially when the comparative costs of the fabrication of the coatings are taken into consideration.
In addition the standard deviation of the adhesive strength of TBCs fabricated with SST is a great deal lower than for specimens fabricated with the above mentioned manufacturing techniques, as shown in Table 4.8.

Table 4.8: Adhesion strength for a range of techniques.

<table>
<thead>
<tr>
<th>Technique</th>
<th>Adhesion Strength, MPa</th>
<th>Standard Deviation (2(\sigma))</th>
</tr>
</thead>
<tbody>
<tr>
<td>APS</td>
<td>22</td>
<td>5.2</td>
</tr>
<tr>
<td>LVOF</td>
<td>18.2</td>
<td>4.3</td>
</tr>
<tr>
<td>SST</td>
<td>11.3</td>
<td>2.5</td>
</tr>
</tbody>
</table>
4.4.4 Summary

Results presented in this section demonstrate that the adhesive strength of TBC is greatly affected by the composition of the coating. TBC with coating compositions below 40% ceramic have virtually zero adhesion strength, as the fabrication process normally leads to large interfacial defects. An increase of the ceramic component in the composition initially led to a sharp increase in the adhesive strength. With further increases of the specific weight of the ceramic in the slurry solution leading to the decrease of adhesive strength values. The optimum composition (50% ceramic and 50% metal powder) for the maximum adhesion strength for mono-layered coatings was determined (approximately 9 MPa). A comparison with the corresponding values sourced from literature demonstrated that the obtained adhesive strength of TBC fabricated by SST were generally lower than for other fabrication techniques.

A similar situation resulted for multi-layered coatings. The maximum adhesion strength of the multilayered coating fabricated by SST was determined to be approximately 11 MPa, which was found to be lower than the corresponding values for the TBC fabricated with other manufacturing techniques. Despite the lower values of adhesive strength for multi-layered coatings fabricated with SST, the results can be considered satisfactory for many industrial applications.
4.5 Thermal Conductivity

4.5.1 Introduction

The primary purpose of Thermal Barrier Coatings (TBCs) is to reduce the overall thermal conductivity of a structural component (normally plate or shell) subjected to a high thermal flux or temperatures. This, in turn, reduces the operating temperatures and protects the metallic load-bearing components, which usually has a relatively high conductivity from failures associated with the degradation of the strength or stiffness properties at elevated temperatures.

The thermal conductivity of a material is the measure of its ability to conduct heat. It is normally defined by the following equation

\[ k = -q \frac{\Delta x}{\Delta T}, \]  
(4.5)

where the thermal conductivity, \( k \) [W/mK], is determined by the thermal heat flux, \( q \) [W/m²], the gauge distance, \( \Delta x \) [m], and the temperature difference across this distance, \( \Delta T \) [K].

Traditional techniques are not always applicable to the measurement of the thermal conductivity for thin films or coatings, therefore, few methods have been developed in the past, specifically for TBC [Zhu and Miller, 2000]. A brief overview and summary of these test methods will be presented below.

The first method for determining thermal conductivity utilises a high heat flux generated from a laser, producing a high thermal gradient through the coating surface. In this method the test specimens are generally limited in size, with the maximum diameter of approximately 25 mm or smaller. The limited diameter size is attributed to the application of the laser as a heat source for large specimens results in significant errors associated with non-uniform heating of such specimens [Zhu and Miller, 2000]. The heat flow passes through the specimen and the temperature difference is measured by standard thermocouples. From these temperature
measurements, a thermal gradient can be calculated and used to determine the thermal conductivity of the coating in accordance with Equation (4.5).

Another method for measuring thermal conductivity utilises the following procedure. A coated specimen is placed between two round plates of the same diameter as the specimen, with the plates being well insulated to prevent heat losses. The plates and the specimen are equipped with thermocouples at various intervals to determine the temperatures of the specimen and plates at different locations. The heat flux is applied through the top plate, which is subjected to the heating. Thermal conductivity is determined from the measured temperature difference at different locations utilising the same equation (4.5). The equipment and methodology of this method is standardised and described in the ASTM Standard E 1530-04, Standard Test Method for Evaluating the Resistance to Thermal Transmission of Materials by the Guarded Heat Flow Meter Technique.

This latter measurement principle was selected by the candidate for the development of a test rig and measurements of thermal conductivity on the TBCs based of the simplicity and relatively low cost of test equipment needed for the evaluation of thermal conductivity of TBCs.

### 4.5.2 Experimental Setup and Procedure

A custom-made rig was designed by the candidate and supervised the construction of the apparatus for thermal conductivity investigations as seen in Figure 4.18. Final design of the thermal conductivity test rig was based on the ASTM standard, E 1530-04: Guarded Heat Flow Meter Technique. The test specimens were fabricated with Slurry Spray Technique (SST) in the shape of 80 mm diameter disks of 3 mm thickness. The diameter of the specimens was the same as the diameter of the plates. Details of fabrication of the standard TBC specimens were outlined in the Chapter 3.
An induction heater fixed on the upper plate, as shown in Figure 4.19, was used to generate the thermal flux, which passes through the whole thermal system. The induction heater has a control panel, which allows setting the desired temperature for the experimentations. In order to maintain a constant temperature at the lower end of the system, which is needed for the determination of thermal conductivity, the bottom surface of the second plate is kept at a constant temperature using flowing tab water to support this constant temperature.
The thermal conductivity rig was isolated with a fibreglass insulation wrapping to minimise heat losses to the environment. Heating plates were fabricated from brass, which possesses a very high thermal conductivity in comparison to ceramics and other materials, allowing the achievement of highly accurate thermal conductivity measurements. A heating coil, connected to an induction heater, was positioned on top of the heating plate, generating the necessary heat flow through the whole system, as shown in Figure 4.20.

![Figure 4.20: Thermal conductivity test rig setup](image)

Constant heat flow passing through the system is controlled by the input power of the induction heater and the temperature of the running water. The slot of the thermocouples on the upper and lower plates is machined with high level of tolerances in order to minimise possible errors in the experimental results.

The experimental procedure for determining thermal conductivity of the coating was based on the discrete representation of Equation (4.5):
where the unknown thermal conductivity, $k \, [\text{W/mK}]$, is determined by the thermal energy, $Q \, [\text{W}]$, the cross sectional area of the specimen, $A \, [\text{m}^2]$, the distance between the thermocouples, $\Delta x \, [\text{m}]$, and the difference in the measured temperatures between the thermocouples, $\Delta T \, [\text{°C}]$.

In the absence of thermal loses, the heat flux is constant at any cross section of the test rig. Therefore, at the steady state conditions the temperature was used along with the known information from literature on thermal conductivity of the brass plate to determine the thermal conductivity of the specimen. The overall thermal conductivity of the specimen is presented by the following equation [Mills, 1999],

$$\frac{l_o}{k_o} = \frac{l_i}{k_i} + \frac{l_c}{k_c}, \quad (4.7)$$

where the thermal conductivity of the coating, $k_c \, [\text{W/mK}]$, was determined with the thermal conductivity of the uncoated Nickel plate, $k_i \, [\text{W/mK}]$ and the overall thermal conductivity, $k_o \, [\text{W/mK}]$. From previous equations, the overall thermal conductivity of the thermal coating can be determined as

$$k_c = \frac{l_c}{l_o \frac{l_i}{k_i} - l_i k_c}, \quad (4.8)$$

where $l_c \, [\text{m}]$ the coating thickness, $l_i$ is the thickness of the substrate and $l_o$ is the overall thickness of the specimen.
4.5.3 Results and Discussion

The experimental study was conducted for a range of TBC specimens with various coating compositions fabricated with the SST. Details of these compositions are presented in Table 4.9 and were used to examine the relationship between the thermal conductivity of TBC with the different percentage of ceramic content.

Table 4.9: Composition of specimens for thermal conductivity tests

<table>
<thead>
<tr>
<th>No. Layers</th>
<th>Composition</th>
<th>Section</th>
<th>ZrO₂ (%)</th>
<th>Ni (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single</td>
<td>Top</td>
<td>100</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>Double</td>
<td>Top</td>
<td>100</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Base</td>
<td>50</td>
<td>50</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Top</td>
<td>100</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>Triple</td>
<td>Mid</td>
<td>75</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Base</td>
<td>50</td>
<td>50</td>
<td></td>
</tr>
</tbody>
</table>

In order to validate the experimental rig and thermal conductivity measurements, a number of specimens made of materials with well known and stable thermal properties were tested. The values presented in Table 4.10 show the results of the testing the thermal conductivity of Stainless Steel 304 and Nickel 200, with the published data.

Table 4.10: Reference condition specifications of the thermal conductivity

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Thermal Conductivity</th>
<th>Thermal Conductivity</th>
<th>Standard</th>
</tr>
</thead>
</table>
The results of testing thermal conductivity using the thermal conductivity rig showed good agreement with the published data [Sweet, Roth and Moss, 1987]. The obtained experimental values were within the range of the standard deviation of the published results. The following section investigates the thermal conductivity of TBCs fabricated using the SST for single, double and triple layered coatings.

Figure 4.21 shows the thermal conductivity of mono-layered and multi-layered coatings as a function of the coating thickness. Table 4.11 shows the averaged values of the thermal conductivity and coating thicknesses for mono-layered and multi-layered TBCs.

<table>
<thead>
<tr>
<th>No. Layers</th>
<th>Average</th>
<th>Standard Deviation (2σ)</th>
<th>Average</th>
<th>Standard Deviation (2σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single</td>
<td>0.9</td>
<td>0.2</td>
<td>58.4</td>
<td>18.4</td>
</tr>
<tr>
<td>Double</td>
<td>2.6</td>
<td>2.3</td>
<td>63.6</td>
<td>8.4</td>
</tr>
<tr>
<td>Triple</td>
<td>11.6</td>
<td>4.7</td>
<td>68.6</td>
<td>8.0</td>
</tr>
</tbody>
</table>

The thermal conductivity of the single-layered TBC had the lowest value and was not affected by the thickness of the coating layer, as one can normally expect. This is due
to the mono-layered TBC being composed from pure ceramics while the graded coating contains a significant portion of the metal constituency with a relatively high thermal conductivity. It was observed that the thermal conductivity of the graded TBC increases with the thickness of the coating, as shown in Figure 4.21. A similar tendency was observed for the thermal conductivity measurements of TBCs fabricated with other fabricating techniques as well. For example, experimental studies on TBCs fabricated by EB–PVD demonstrated a significant difference in the thermal conductivity of thin and thick coatings having the same composition. The thermal conductivity of 300 μm thick TBCs was approximately 40% higher in comparison with relatively thin 50 μm coatings [Rätzer-Scheibe, Schulz and Krell, 2006]. One of the explanations for this difference is the effect of the microstructure on the overall thermal conductivity and the by difficulties in providing consistent fabricating conditions for thin and thick coatings.

![Figure 4.21: Thermal conductivity for TBCs fabricated using the SST](image)

Figure 4.21: Thermal conductivity for TBCs fabricated using the SST
Mathematical Modelling of Material Properties

The following section presents results of mathematical modelling of the thermal conductivity of single, double and triple layered coatings. The mathematical models used for theoretical modelling are the Rules of Mixture (RoM) [Liu, 1997], and a Micromechanical Model (MM) based on Mori-Tanaka theory [Brinson and Lin, 1998]. In accordance with the RoM the thermal conductivity of a TBC layer is

\[ k_o = k_1 V_1 + k_2 V_2 \]  \hspace{1cm} (4.9)

where the apparent thermal conductivity, \( k_o \) [W/mK], is determined by the volume fraction of ceramic, \( V_1 \), and the volume fraction of the metal, \( V_2 \), the thermal conductivity of the ceramics, \( k_1 \) [W/mK] and the thermal conductivity of metal \( k_2 \) [W/mK], respectively.

The following equation presents the apparent thermal conductivity of a coating layer, using micromechanical modelling. The equation for the overall thermal conductivity of a two-phase composite can be derived from [Wakashima and Tsukamoto, 1992] and is given by

\[ k_o = \frac{V_2 k_1(k_2 - k_1)}{k_1 + \frac{1}{3}(1 - V_2)(k_2 - k_1)} + k_1, \]  \hspace{1cm} (4.10)

where the notations are the same as in the previous equation (4.9).

After determining the thermal conductivity of each coating layers, the overall thermal conductivity of the TBC can be found in accordance with the following equation, similar to Equation (4.7):

\[ \frac{l_o}{k_o} = \sum_{n=1}^{N} \frac{l_n}{k_{o,n}} \]  \hspace{1cm} (4.11)

which in the case of two layers, can be reduced to equation (4.7). In the last equation, \( N \) is the number of layers and \( l_n \) and \( k_{o,n} \) are the thickness and the overall thermal conductivity of \( n \)-th layer found from RoM or MM theories.

The following table presents the theoretical and experimental values of the thermal
Table 4.12: The Thermal Conductivity of Single, Double and Triple layered Slurry Based TBCs and coating thickness.

<table>
<thead>
<tr>
<th>Layers</th>
<th>Experimental Values, W/mK</th>
<th>Micromechanical Predictions, W/mK</th>
<th>Rule of Mixtures, W/mK</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single</td>
<td>0.9</td>
<td>0.9</td>
<td>0.9</td>
</tr>
<tr>
<td>Double</td>
<td>2.6</td>
<td>1.4</td>
<td>1.8</td>
</tr>
<tr>
<td>Triple</td>
<td>11.2</td>
<td>1.5</td>
<td>2.5</td>
</tr>
</tbody>
</table>

The experimental values for the single layered coatings were well within the range of the experimental values of thermal conductivities reported in the literature, from 0.8 to 1.5 W/mK [Ma, Wu, Roth, Gell and Jordan, 2006; Schulz, Rätzer-Scheibe, Saruhan and Renteria, 2007], showed good agreement with the values of determined by the mathematical models.

In the table above, the double layered coatings were found to have an apparent thermal conductivity of 2.6, 1.4 and 1.8 W/mK for the experimental and the mathematical values, respectively. The experimental values for the double layered coatings, showed relatively good agreement with the mathematical approximations using MM and RoM.

The thermal conductivity values of the triple layered coatings were found to be 11.6, 1.5 and 2.5 W/mK for the experimental, micromechanical (MM) and rule of mixture (RoM) mathematical models, respectively. The experimental results significantly exceeded the values determined numerically by the MM and RoM. The factors which affected the theoretical values for the thermal conductivity of the mono-layered and multi-layered coatings were the manufacturing defects as well as the TBC microstructure [Bordeaux, Saint Jacques and Moreau, 1991], which are currently...
completely ignored in the mathematical modelling.

A comparison of the thermal conductivity of mono-layered and multi-layered coatings fabricated with various manufacturing techniques is shown in Figure 4.22. The comparison includes the experimental results obtained for mono-layered TBC samples fabricated with Atmospheric Plasma Spray (APS), Electron Beam Physical Vapour Deposition (EB–PVD) as well as the SST.

The specimens fabricated with APS had the top coat layer with 8 wt. % of $Y_2O_3$–ZrO$_2$ (Yttria Stabilised Zirconia) and the NiCrAlY bond coat layer of 50 $\mu$m thickness. These layers were deposited on a Nickel alloy CMSX-4 substrate. The coating thicknesses ranged from 250 to 300 $\mu$m and were deposited on to a square plate specimen with dimensions of 12.7x12.7x3 mm.

The TBCs fabricated with EB–PVD had 7 wt. % $Y_2O_3$–ZrO$_2$ (Yttria Stabilised Zirconia) for the top coat with a NiCrAlY bond coat. The coats were deposited on a Nickel alloy CMSX-4 substrate, similar to the previous case. The dimensions of the test specimens were different, with coatings of a 200 $\mu$m thickness being deposited on round plate specimens with 10 mm diameter and 3 mm thickness.

The TBC specimens fabricated with the SST described previously, with the coating compositions shown in Table 4.9, were chosen to have coating thicknesses as close as possible to that fabricated with APS and EB–PVD, in order to compare the different fabricating techniques.

Figure 4.22 shows the experimental results for the thermal conductivity of the mono-layered TBCs fabricated with APS, EB–PVD and SST [Ma, Wu, Roth, Gell and Jordan, 2006; Schulz, Rätzer-Scheibe, Saruhan and Renteria, 2007]. TBCs fabricated with the APS technique had an average thermal conductivity of 0.9 W/mK, while EB–PVD had an average thermal conductivity of 1.5 W/mK, and the specimens fabricated with SST had an average thermal conductivity of 1 W/mK. The thermal conductivity for coatings fabricated with the SST was slightly higher than the APS specimens and much lower than the specimens fabricated with EB–PVD.
Figure 4.22: Thermal conductivity for mono-layer TBCs fabricated by APS, EB-PVD and SST. Error bars represent standard deviation (2\(\sigma\)) for all experiments.

Figure 4.23 presents experimental results for the apparent thermal conductivity of the multi-layered TBCs fabricated with APS and SST [Khor and Gu, 2000]. TBCs fabricated with APS had an average thermal conductivity of 3.0 W/mK, and specimens fabricated with SST had an average thermal conductivity of 2.6 W/mK. The lower measured thermal conductivity in the SST in comparison to APS could be explained by a higher level of defects and higher porosity of the coatings fabricated with SST as discussed in section 3.2. Past studies have shown that the thermal conductivity is dependent on the porosity level of the coating, [Sumirat, Ando and Shimamura, 2006].
Figure 4.23: Thermal conductivity for multi-layered TBCs fabricated by APS and SST. Error bars represent standard deviation (2σ) for all experiments.
4.5.4 Summary

In this section one of the most important physical properties, thermal conductivity, of TBC fabricated with the Slurry Spray based Technique, were investigated. With the guidance of the appropriate ASTM standard, a custom made rig was designed and constructed for these investigations. Validation tests demonstrated a good agreement between the measurements and published experimental results for known materials. Single, double and triple layered coatings were fabricated with the SST and tested with the thermal conductivity test rig. As expected the mono-layered coatings had the lowest thermal conductivity with no coating thickness effect on the thermal conductivity values. Quite opposite, multi-layer graded coatings had relatively higher thermal conductivity and experimental results showed a significant dependence of its values from the coating thickness.

Mathematical modelling of the thermal conductivity of the TBC was conducted using the Rule of Mixture approach and Micro-Mechanical modelling. Experimental results and mathematical models, with the exception of the single layered coating, were found to differ significantly, especially for triple layered coatings. Differences between the theoretical and experimental values of the thermal conductivities of the double and triple layered coatings were attributed to the effects of the coating microstructure, such as porosity and phase micro-structures, as well as manufacturing defects, which were always present during any manufacturing process. These effects are currently fully ignored in the mathematical models.

Further, the present experimental results were compared with the published results, which were selected to match as close as possible the composition and structure of the specimens used for the comparisons. From this comparison, it can be concluded that the values of the thermal conductivity of coatings fabricated with SST is similar or even lower than coatings fabricated with other traditional and well established techniques such as APS and EB–PVD.
4.6 Thermal Cycling

4.6.1 Introduction

Thermal Barrier Coatings (TBCs) are widely used in power generation and aerospace applications to protect components, such as blades and vanes, from harsh high temperature environments during service [Pichon, Lacoste, Barreteau and Glass, 2006]. These applications assume that the component protected with TBC undergoes a cyclic loading with the total number of cycles ranging from few cycles (disposable hypersonic components) to many thousand cycles (turbines and engines) [Takeuchi and Kokini, 1994; Kokini, Takeuchi and Choules, 1996; Ahmaniemi, Vuoristo, Mäntylä, Gualco, Bonadei and Di Maggio, 2005].

In this section, the thermal cycling behaviour of Functionally Graded Thermal barrier Coatings (FG–TBCs) specimens fabricated with the Slurry Spray Technique (SST) was investigated. The investigations focused on the low cycle fatigue region, as the primary focus of this particular technique is to be applied in aerospace application such as in hypersonic vehicles, where the total number of cycles is very limited. In this section TBC fabricated by SST was tested in a high temperature furnace and underwent a low number of thermal cycling. After thermal cycling the specimens were taken for examination of thermal damage to the coating by means of Scanning Electron Microscopy (SEM). Consecutive micro-examinations demonstrated the development of the mechanical damage and provided a qualitative assessment of the resistance of coating to the thermal cycling loading.

4.6.2 Experimental Setup and Procedure

Mono-layered and multi-layered coatings fabricated with the Slurry Spray Technique (SST), with 150 μm thickness, were used for the experimental study. The substrate material was Nickel 200. Specimens were cut in the form of a rectangular plate with
50 x 50 x 3 mm dimensions. Compositions of the tested specimens are shown in Table 4.13. Five specimens of each coating composition were fabricated. Different sintering methods (high temperature oven and oxy acetylene torch) were used in the fabrication of the test specimens. The sintering method was varied in order to understand the difference in the performance of the TBCs fabricated with these sintering parameters.

Table 4.13: Compositions of specimens for thermal cycling

<table>
<thead>
<tr>
<th>Test</th>
<th>Sintering Method</th>
<th>No. Layers</th>
<th>Composition</th>
<th>ZrO₂ (%)</th>
<th>Ni (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Oxy-Acetylene</td>
<td>Single</td>
<td>Top</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>Oxy-Acetylene</td>
<td>Double</td>
<td>Base</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Top</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>Oxy-Acetylene</td>
<td>Triple</td>
<td>Mid</td>
<td>75</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Base</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>4</td>
<td>Oven</td>
<td>Single</td>
<td>Top</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>Oven</td>
<td>Double</td>
<td>Top</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Base</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>6</td>
<td>Oven</td>
<td>Triple</td>
<td>Mid</td>
<td>75</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Base</td>
<td>50</td>
<td>50</td>
</tr>
</tbody>
</table>

During thermal cycling the included specimens were placed in the preheated oven and kept at 1000°C temperature for approximately 30 minutes, and in the following stage, were cooled to the ambient temperature for approximately 30 minutes. The fabricated specimens were subjected up to 30 thermal cycles, where the criterion for the failure of the coating was adopted as more than 20% of the spalled region of the
surface of the coating [Li, Sun, Hu and Guan, 2007].

![High temperature furnace](image)

**Figure 4.24: High temperature furnace**

Experimental procedure for the thermal cycling tests is as follows:

- Preheat high temperature furnace to 1000°C
- Once temperature has been reached, place first batch specimens in furnace
- After 30 minutes has elapsed remove specimens from oven and allow cooling for 30 minutes.
- Place second batch of specimens in oven, while first batch is cooling.
- Repeat steps 3 – 4 until failure of the coating.
- Record number of cycles for coating failures
4.6.3 Results and Discussion

Thermal cycling testing was conducted by the candidate to simulate the conditions similar to real life application of TBCs. A total number of 36 specimens of various compositions given in Table 4.13 were subjected to the thermal cycling tests. Results of the thermal cycling tests demonstrated that a large portion of all failures of the TBC took place during the first few cycles. If the failure does not occur during the initial stages of the thermal cycling, then integrity of the coating is preserved throughout the thermal cycling tests.

![Figure 4.25: Probability of survival against the number of thermal cycles for oven and oxy torch sintered TBCs.](image)

Figure 4.25 shows the portion of non-failed specimens after thermal cycling of TBCs fabricated with oven and oxy acetylene sintering methods. It was observed that TBCs sintered with the high temperature oven, in comparison with the oxy acetylene torch yielded considerably less coating failures during the thermal cycling tests.

Experimental results obtained 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 a constant heat flow and uniform temperature distribution a good coalescence between particles can be achieved, reducing the induced thermal stresses during the thermal cycling. Secondly, applying a slow cooling rate after sintering can effectively reduce the residual strain and stress, which is normally associated with rapid cooling.

Figure 4.26 shows the portion of non-failed specimen due to the thermal cycling for mono-layer and multi-layer TBCs. From this figure, it can be observed that the multi-layered TBCs provide a considerably better resistance to thermal cycling, where 70% of multi-layered specimens survived the thermal cycling against only 20% of mono-layered TBC specimens.

As expected, Functionally Grading leads to a significantly better resistance to the thermal cycling and provides greater integrity characteristics due to a considerable reduction in thermal stresses caused by mismatch in the material properties. To demonstrate the reduction in the mismatch of material properties which can be achieved with FG-TBCs, in the following section a micromechanical model is used to evaluate the Coefficient of Thermal Expansion (CTE) of graded multi-layered
TBCs. This thermo-mechanical characteristic (CTE) is considered to be the main factor contributing to the generation of thermal stresses as well as the thermal failure of coatings [Kakuda, Limarga, Bennett and Clarke, 2009].

**Mathematical Modelling of the CTE Mismatch**

The following section presents results of micro-mechanical modelling of the CTE for the graded coatings. The determination of the CTE is based upon the mathematical models Rule of Mixtures (RoM) [Liu, 1997] and Micromechanical Models (MM) based upon Mori-Tanaka theory and equations developed in [Wakashima and Tsukamoto, 1992]. In accordance with the RoM the CTE of a TBC layer is

$$\alpha_o = \alpha_1 V_1 + \alpha_2 V_2$$  \hspace{1cm} (4.12)

where the apparent CTE, \(\alpha_o [1/°C]\), is determined by the volume fraction of ceramic, \(V_1\), and the volume fraction of the metal, \(V_2\), the CTE of the ceramics, \(\alpha_1 [W/mK]\) and the CTE of metal \(\alpha_2 [W/mK]\), respectively.

The CTE for two-phase composite can be found as

$$\alpha_o = \left(\frac{1}{K_o} - \frac{1}{K_1}\right)\left(\alpha_2 - \alpha_1\right) + \alpha_1,$$  \hspace{1cm} (4.13)

where the overall CTE, \(\alpha_o [1/°C]\) is determined by the CTE of the ceramic and metal powders, \(\alpha_1 [1/°C]\) and \(\alpha_2 [1/°C]\), the ceramic bulk modulus, \(K_1 [GPa]\) and the metal bulk modulus \(K_2 [GPa]\).

The overall bulk modulus, \(K_o [GPa]\) can be found as

$$K_o = \frac{V_2 K_1 a (K_2 - K_1)}{(1 - V_2)K_2 + V_2 K_1 a} + K_1,$$  \hspace{1cm} (4.14)

where \(V_2\) is the volume fraction of the metal powder, and the remaining notations are the same as in the previous Equation (4.12). The constant, \(a\), is determined by Equation (4.15), where the \(K [GPa]\) and \(\mu [GPa]\) are the bulk modulus and shear
modulus of the materials, where subscripts 1 and 2 refer to the ceramics and metal powders. The bulk and shear modulus is determined by a function of the modulus of elasticity and the Poisson’s ratio.

\[ a = \frac{K_2(3K_1 + 4\mu_1)}{K_1(3K_2 + 4\mu_2)}, \quad (4.15) \]

Table 4.14 presents the theoretical values for the difference of CTE between the layers of the mono-layered and multi-layered coatings. The mathematical models used to determine the CTE, were observed to be comparable for simple and advanced methods.

The difference between the CTE of the single layered coating interface, were determined to be 5.5 \( \times 10^{-6} \) 1/°C for both the mathematical models, MM and RoM.

The difference in CTE between the double layered coating interfaces were determined to be 3.4, 2.1 \( \times 10^{-6} \) 1/°C and 2.8, 2.8 \( \times 10^{-6} \) 1/°C for the mathematical models RoM and MM, respectively. The values observed for the difference in CTE for double layered coatings, were found to be significantly lower than the values in single layered coatings.

The triple layer coating had lower levels of CTE between the coating interfaces, with 1.9, 0.8, 2.9 \( \times 10^{-6} \) 1/°C and 1.4, 1.4, 2.8 \( \times 10^{-6} \) 1/°C for RoM and MM respectively. The difference in the CTE was approximated to be more evenly distributed throughout the layers of the coating in comparison to the single and double layered coatings.
Table 4.14: The CTE of the mono-layer and multi-layered TBC, determined by mathematical models

<table>
<thead>
<tr>
<th>No. Layer</th>
<th>Interface</th>
<th>Difference in CTE, ((x \times 10^{-6}) , 1/\degree C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>RoM (4.12)</td>
</tr>
<tr>
<td>Single</td>
<td>Top Layer- Substrate</td>
<td>5.5</td>
</tr>
<tr>
<td>Double</td>
<td>Top Layer- Mid Layer</td>
<td>2.8</td>
</tr>
<tr>
<td></td>
<td>Mid Layer - Substrate</td>
<td>2.8</td>
</tr>
<tr>
<td></td>
<td>Top Layer- Mid Layer</td>
<td>1.4</td>
</tr>
<tr>
<td>Triple</td>
<td>Mid Layer- Base layered</td>
<td>1.4</td>
</tr>
<tr>
<td></td>
<td>Base layered- Substrate</td>
<td>2.8</td>
</tr>
</tbody>
</table>

With the introduction of the multi-layered coatings, mathematical models have demonstrated that the difference in the CTE between the interfaces of the coating layers and substrate has significantly been reduced. The reduction in the CTE lowers the residual and thermal stresses experienced by the TBC. The lower levels of the thermal stresses normally increase the survival rates, which correlates well with the experimental study conducted.
SEM Images

The following figures (4.27 - 4.29) show SEM images of a multi-layered TBC specimens at various stages throughout thermal cycling. The images show the surface of the TBC and a progressive development of mechanical damage, such as voids and cracks.

Figure 4.27 shows the surface of a TBCs specimen before thermal cycling. The SEM image shows the relatively high quality surface of the coating with a relatively low level of porosity and minimal evidence of cracking along the surface. Figure 4.28 shows the surface of the coating after 10 thermal cycles. A number of cracks initiated during thermal cycling can be observed, with the development of larger pores. Figure 4.29 shows the TBCs after 20 cycles, with the coating surface undergoing visible transformations accompanied with the formation of cracks and enlargement of pores. It was expected that further thermal cycling, which was beyond the scope of the present study, will lead to more pronounced deterioration, and finally, the overall failure of the coating.

Figure 4.27: 0 thermal cycles
Figure 4.28: 10 thermal cycles

Figure 4.29: 20 thermal cycles
4.6.4 Summary

Thermal cycling represents one of the typical loading conditions normally occurring during the life of a TBC. The total number of cycles experienced by TBCs depends on the particular application of TBCs. Since the current research targets potential hypersonic applications, where the number of thermal cycles is limited, the TBCs fabricated with SST were subjected to a low number of thermal cycles.

The study conducted, aimed to assess the ability of TBCs fabricated by SST to withstand limited thermal cycling regimes. A range of specimens was fabricated to investigate the effect of the composition and sintering method on resistance to the thermal cycling. As expected, the oven-based sintering technique and multi-layered graded coating composition provided a considerably greater survivability, which was attributed to the lower levels of thermal stresses.

It is interesting to note, that the majority of failures took place during the first few cycles. This can be explained by the various manufacturing defects, which probably led to coating failure in the first few cycles. Therefore, manufacturing control is necessary if the technique is to be adapted for industrial applications.

SEM images taken at various stages of thermal cycling demonstrated the development and accumulation of the mechanical damage during the thermal cycling tests. Despite that the coating did not fail within the 30 thermal cycles, it was expected that further thermal cycling would result in failure of the coating. Thermal fatigue investigations will require significantly longer time, however this was not included into the scope of the current research.
4.7 Conclusion

This chapter was devoted to the investigation of the thermo-mechanical properties of TBCs fabricated with the new SST. These thermo-mechanical properties were utilised for the following purposes:

- The development and optimisation of the fabricating parameters of TBC as described in Chapter 3,

- A comparative assessment of the TBCs fabricated with SST with current commercially available manufacturing methods, such as Electron Beam - Physical Vapour Deposition (EB-PVD) and Atmospheric Plasma Spray (APS), and

- The use these physical properties in further numerical studies of TBC aimed on the study of the performance of TBCs under realistic thermal and loading conditions as it will be described in the next Chapter.

The conducted thermo-mechanical studies showed the following results, which are described below.

**Porosity Study:** Examination of the porosity of the TBC specimens demonstrated that the sintering method has a strong influence on the porosity and mechanical damage formation in ceramics based coating during fabrication. The level of porosity achieved with the high-temperature oven sintering technique was about 0.1% approaching the porosity level values for commercially available manufacturing methods. Therefore, the development of the quality of SST focused on the use of the high temperature oven as a sintering method.

**Microhardness study:** The microhardness of the TBCs was found to decrease with the coating thicknesses. The following comparison of the microhardness of TBCs fabricated with EB-PVD and APS showed that typical values of the microhardness of TBCs fabricated with SST were significantly lower than their counterparts. The obtained microhardness results clearly demonstrated that TBC fabricated with SST
cannot be used in applications and situations where the microhardness of the coating is one of the prerequisites. However, in many other applications where high hardness is not required, the SST can be considered as a low cost alternative to the standard fabricating techniques.

**Adhesion strength study:** Investigation into the adhesive strength, demonstrated that the TBC was greatly affected by the composition of the coating. The optimum composition for the single layered (pure ceramics) and multi-layered coatings providing the maximum adhesion strength was determined experimentally. A comparison between the adhesion strength of the TBCs fabricated with SST and other fabricating techniques demonstrated that the adhesive strengths for all methods of fabrication is comparable.

**Thermal conductivity study:** The investigation of the thermal conductivity of mono-layered and multi-layered coatings fabricated with SST demonstrated that, as expected, the mono-layered coatings had the lowest thermal conductivity with no effect of the coating thickness on the thermal conductivity values. In contrast, the multi-layered coating had a relatively higher thermal conductivity as the layers (with the exception of the top layer) were fabricated from a mixture of metal and ceramics and metal powders. A comparison of the experimental results with micro-mechanical modelling demonstrated that mathematical models are not able to predict the material properties of TBC. As discussed above, the effect of the micro-structure, which is largely ignored by the current mathematical models, has a strong effect on the thermo-mechanical properties which is completely ignored in the mathematical modelling.

Further, a comparison with published experimental values of the thermal conductivity for the single and multi-layered coatings was conducted. It was found that the thermal conductivity of coatings fabricated with SST was similar or even lower than the corresponding values for coatings fabricated with other techniques such as APS and EB–PVD. The standard deviation was also lower in the case of the TBC fabricated with SST.
Chapter 4  Characterisation of Thermo-Mechanical Properties of TBC

Thermal Cycling study: The thermal cycling of TBC specimens was conducted to assess overall ability of TBC fabricated by SST to resist to the cycling loading. The effect of coating composition and the sintering method on cycling resistance was also investigated in the conducted thermal cycling tests. As expected, the oven-based sintering technique and graded composition provided a considerably greater survivability to thermal cycling in comparison to the oxy acetylene sintering method and mono-layered coatings, respectively. Higher levels of specimen survivability of the multi-layered coatings, was attributed to the considerably lower levels of the thermal and residual stresses experienced by the graded structure. This result was also in a good agreement with micromechanical models, which demonstrated that the thermal stresses would decrease with the number of coating layers.

The thermo-mechanical testing demonstrated that the TBCs fabricated with SST can be used in high temperature applications where high micro-hardness is not required. All other thermo-mechanical parameters of TBCs fabricated with SST were comparable to the corresponding parameters of TBCs fabricated with other manufacturing techniques.