SLURRY SPRAYED THERMAL BARRIER COATINGS FOR AEROSPACE APPLICATIONS

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CHAPTER 5

SCALE TESTS OF TBCs USING HIGH TEMPERATURE BURNER
5.1 Introduction

Various mechanical and thermo-mechanical testings, such as those described in the previous Chapter, cannot fully characterise and predict the performance of TBCs under real conditions. These tests are mostly aimed for a comparative assessment of thermo-mechanical characteristics of TBC. The conclusive information about the reliability and performance of TBCs can only be obtained from the tests in which the thermo-mechanical loading closely follows to the expected operating conditions. As a result, much work has been done on the development of experimental procedures capable to simulate TBC operating thermal and loading conditions. In the beginning of this Chapter, a brief overview of these procedures is presented.

Trunova et al. [2008], used a high temperature infrared furnace, powered by a 6 KW lamp to investigate a ZrO$_2$/NiCrAlY coating deposited on a CMSX-4 substrate and fabricated with Atmospheric Plasma Spray (APS). The thermal shock experiments were conducted with 300 µm specimens, which were subjected to temperatures of up to 1050°C. The exposure times varied from 2 minutes to 96 hours, which simulated the operating cycle of power generation gas turbines of an aircraft.

Bolcavage et al. [2004], utilised a burner rig to create high temperature and thermal stress gradients along the TBC surface similar to those found in a gas turbine engine in an aircraft. A high running cost of the experiments forced these researchers to look for other available options such as oxygen-propylene torches to supply the heat to the specimen surface. In the described above experiments the temperatures reached up to 1400°C.

A ZrO$_2$/NiCrAlY coating layers were tested in a scaled rocket combustion engine, located at the NASA Lewis Research Centre [Nesbitt, 1990]. The fabricated specimens of 100 – 125 µm thickness fabricated with APS were subjected to gas temperatures of up to 1400°C, with an exposure time of approximately 1.2 s in the hydrogen rich environment.

Carbon/Carbon composite combustion chambers for the Japanese Space Shuttle
HOPE [Wakamatsu, Saito, Ono, Ishia, Matsuzaki, Hamamura, Sohoda and Kude, 1997], with a 140 μm thick ceramic coating of thick Silicon Carbide/Carbon Functionally Graded Thermal Barrier Coatings (FG–TBCs) fabricated by Chemical Vapour Deposition (CVD) were tested in a full scale combustion chamber. The tests underwent firing cycles of 55 seconds with subsequent quenching by liquid nitrogen. A similar approach was adopted to investigate the FG–TBCs of thrust chambers of an orbital manoeuvring system [Moriya, Kuroda, Sato, Tadano, Moro and Nino, 1999]. The ZrO₂/Ni coatings were fabricated with APS, and subjected to firing tests with exposure times of up to 550 seconds.

In this project a new approach for scale testings of TBC is developed, which is based on the use of a high temperature flat flame burner. The use of the flat burner significantly reduced the cost of testing in comparison with the similar concept of testing by [Bolcavage, Feuerstein, Foster and Moore, 2004], as described above. The experimental rig was designed by the candidate and supervised the construction, which was to evaluate the integrity of coatings fabricated with SST and provide information on the performance and reliability of these coatings. The specimens were subjected to micro-examinations before and after these tests to evaluate the effect of the high heat fluxes on the integrity of the coatings. The investigations were supported by the Finite Element Analysis (FEA), simulating the thermal conditions of the scale flat flame burner tests as well as the real thermal loading. The virtual tests by means of FE analysis were conducted to investigate, in detail, the temperature and thermal stress distribution through the thickness of TBCs.
5.2 Experimental Setup and Procedures

The experimental setup utilises a flat flame high temperature burner, burner attachment and data acquisition equipment, which are shown in Figure 5.1. The following section will briefly describe the flat flame burner designed to produce the realistic high temperature and temperature flux, experimental rig and instrumentation used to conduct and record information from the scale tests.

Figure 5.1: Experimental Setup of High Temperature Burner Apparatus
5.2.1 Flat Flame Burner

The experimental rig utilised a flat flame high temperature burner designed and fabricated by the Chemical Engineering Department, University of Adelaide in 2002 [Medwell, Chan, Kalt, Zeyad, Dally and Nathan, 2010]. The flat flame burner is fabricated from Hastelloy honeycomb, brass plates and stainless steel tubing hypodermic tubing to carry the fuel streams. With the flat flame burner, natural gas (Ch₄), oxygen (O) and hydrogen (H) are used as fuel and oxidiser, respectively. The top view of the flat flame high temperature burner is shown in Figure 5.2.

![Figure 5.2: Top view flat flame high temperature burner](image)

The high temperature burner produces flat flames with temperatures from 1000°C up to 2400°C (depending on the gas composition), which are stable over these wide range of temperatures. The flat flame burner is also able to produce a highly uniform temperature distribution and velocity profiles with no temporal fluctuations and with excellent flame reproducibility. These characteristics of the high temperature flat burner are prerequisite for the high temperature tests of TBCs, as these characteristics of the flat flame burner allow control of the thermal loading conditions with a high level of accuracy.

The high temperature flat flame burner generates a uniform thermal flux with values
ranging from 20 kW to 80 kW, depending on the gas composition. The burner generates a high temperature gradient across the coating thickness, similar to the heat fluxes found for a typical rocket combustor or leading edge of a hypersonic vehicle [Nesbitt, 2000].

### 5.2.2 High Temperature Burner Attachment

CAD drawings of the high temperature burner attachment for testing TBC specimens are shown in Figure 5.3. The design allows the specimen to be easily fixed, changed and tested with the high temperatures produced by the burner. The specimen is placed into the specimen holder (top) with the TBC surface being exposed to the thermal flux from the flat flame burner. The specimen holder (base) is coupled with the specimen holder (top) as shown in Figure 5.3. The specimen holder (base) and (top) were fabricated from Inconel 601; while the high temperature burner attachment was fabricated from a combination of Inconel 601 plate and Stainless Steel 316 square tubing.
To minimise the heat transfer between the TBC specimen and other metal components of the rig, a cloth insulation made of ceramics was applied to isolate the sides of the specimen which otherwise would be in contact with the other metal parts of the rig. The insulation was utilised to prevent heat transfer from metal components of the high temperature burner attachment, and allow the simulation of the one-dimensional thermal flux through the coating thickness, uniform over the whole surface area of the specimen, which excludes edge effects on the temperature measurements. The burner attachment with a TBC specimen is shown in Figure 5.4.
5.2.3 Instrumentation

The rig was equipped with two type K thermocouples (Ø ~ 1.0 mm) to measure the temperatures of the rear surface of the TBC specimen and one type R thermocouple (Ø ~ 0.7 mm) to measure the flame temperature. The thermocouples with insulated transmitters where connected to the data acquisition equipment, USB-1208FS, which was calibrated using standard procedures. The temperature readings were recorded with 1 Hz frequency and processed with standard data analysis software.

5.2.4 Thermocouple Calibration

In order to determine the flame temperature, the values recorded from the type R thermocouple were adjusted to take into account the effects of the radiation flux. The calibration was conducted with the help of software package GASEQ for calculating the gas properties in conjunction with ABB AP-Calc 9 for determining the flow rates of the natural gas and oxygen. A special procedure based on the energy balance was used to determine the actual temperature of the flame. Details of this procedure can be found in [Medwell, Chan, Kalt, Zeyad, Dally and Nathan, 2009]. The flat flame temperature used in the scale test ranged from 1400, 1700 and 1900°C.
5.2.5 Specimen Preparation

TBC specimens with dimensions of 50 x 25 x 3 mm were fabricated from Nickel 200 (UNS N02200). The fabricating parameters of the TBCs specimens were the same as those used for thermo - mechanical tests described in Chapter 4, where Table 5.1 describes the compositions of the TBC specimen fabricated with SST. Three specimens of each composition were fabricated for the current investigations. These specimens were subjected to the thermal loading with burner flame temperatures of 1400°C, 1700°C and 1900°C, respectively.

Table 5.1: Specimens for high Temperature burner Investigations of TBCs fabricated using the Slurry Spray Technique

<table>
<thead>
<tr>
<th>No. Layers</th>
<th>Composition</th>
<th>Coating Thickness, μm</th>
<th>Exposure time, minutes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Section</td>
<td>ZrO₂ (%)</td>
<td>Ni (%)</td>
</tr>
<tr>
<td>Double</td>
<td>Top</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>Base</td>
<td>50</td>
<td>50</td>
<td></td>
</tr>
<tr>
<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>
</tr>
<tr>
<td>Base</td>
<td>50</td>
<td>50</td>
<td></td>
</tr>
</tbody>
</table>
5.3 Results of high temperature tests

This section presents the results of the scale tests of the TBC specimens fabricated with SST subjected to realistic thermal loading, which was simulated by means of the high temperature rig. The results include the temperature response of uncoated and coated specimens subjected to various flame temperatures as well as micro-examination of the integrity of the coatings before and after testing.

5.3.1 Temperature Response of TBC Specimens

The temperature variation of the rear surface of TBC specimens with uncoated, double and triple layered coating surfaces subjected to flame temperatures of 1400°C can be seen in Figure 5.5. The temperature difference between the uncoated, double and triple layered coatings is rather small due to low thermal capacity of the specimen (substrate). Again, these tests were aimed to subject the TBC to the realistic temperatures and thermal fluxes and investigate the effect of these on the integrity of the coatings, while the efficiency of the coatings will be investigated at the virtual modelling of TBC behaviour by means of FEA.
Figure 5.5: Rear surface temperature of the specimen against exposure time for flame temperature of 1400°C

Figure 5.6 shows the rear surface of TBC specimens with uncoated, double and triple layered coating surfaces subjected to flame temperatures of 1700°C.

Figure 5.6: Rear surface temperature of the specimen against exposure time for flame temperature of 1700°C
The graph of temperature variation of the rear surface of TBC specimens with uncoated, double coated and triple layered coated surfaces subjected to flame temperatures of 1900°C, is shown in Figure 5.7.

![Graph showing temperature variation](image)

Figure 5.7: Rear surface temperature of the specimen against exposure time for flame temperature of 1900°C

### 5.3.2 Effect of Coating Thickness on Rear Surface Temperature

The results presented in the current section, examine the effect of coating thickness on rear surface temperatures. Figure 5.8 shows a graph of the average rear surface temperature of double layered, triple layered TBC plotted against the coating thickness for flame temperatures of 1400°C. From this figure, a clear trend can be observed that, and as expected, the temperature of the rear surface decreases with the increase of the coating thickness. From Figure 5.8 it is also can be seen that the temperatures at the rear surface of the specimens with double layered and triple layered coatings have similar values and trends.
Similar dependences were observed during the tests for flame temperatures of 1700°C as it can be seen in Figure 5.9, where the rear surface temperature decreased with the increasing coating thickness.
However, this tendency disappears at higher flame temperatures. Figure 5.10 shows the graph of the rear surface temperature of double layered and triple layered TBC, plotted against coating thickness, for flame temperatures of 1900°C. In contrast to previous results, the rear surface temperatures of the double layered coating specimens were noticeably larger in comparison to the triple layered coating specimens.

Figure 5.10: Rear surface temperature against coating thickness of double and triple layered coatings for 1900°C

5.3.3 Integrity of TBCs under Realistic Thermal Loading

This section examines the integrity of coated surfaces exposed to the flame temperatures as described before. Before high temperature tests and immediately after the testing, TBC specimens were subjected to micro-examinations with the Digital Single Lens Reflex (DSLR) camera, Canon EOS 5D Mark I, with a Canon EF 100mm f/2.8 L IS USM macro lens.
Chapter 5  Scale Tests of TBCs using High Temperature Burner

**Flat Flame Temperature - 1400°C**

Pictures of double layered and triple layered coating surfaces before and after the experimentation are shown in Figure 5.11 and Figure 5.12. Throughout the experiments, no coating damage was observed for the double and triple layered coatings.

![Figure 5.11: Images of double layered coating surface before and after experiments, for flame temperature of 1400°C](image)

a. Before  
b. After

Figure 5.11: Images of double layered coating surface before and after experiments, for flame temperature of 1400°C
Figure 5.12: Images of triple layered coating surface before and after experiments, for flame temperature of 1400°C
Flat Flame Temperature - 1700°C

The double layered and triple layered coating surfaces before and after the experimentation are shown in Figure 5.13 and Figure 5.14. Like the previous images, no coating damage was observed for the double and triple layered coatings.

Figure 5.13: Images of double layered coating surface for flame temperature of 1700°C
Figure 5.14: Images of triple layered coating surface for flame temperature of 1700°C
Flat Flame Temperature - 1900°C

Figure 5.15 and Figure 5.16 show images of the coating surface of the double and triple layered TBC before and after the experimentation. Throughout the experiments, structural failure by delamination and / or spallation was observed for both double and triple layered coatings. The delamination and spallation for the double layered coating was observed during the testing, while the triple layered coating was observed to fail by delamination, which occurred after the experiments had been completed.

Figure 5.15: Images of double layered coating surface for flame temperature of 1900°C
Figure 5.16: Images of triple layered coating surface before and after experiments, for flame temperature of 1900°C
5.4 Virtual testing of TBCs using Finite Element Analysis

The following section discusses a numerical model developed to examine the performance of TBC system with a high heat flux, utilising Finite Element Method (FEM) and software package ANSYS 12.0. The purpose of these tests was simulation of the realistic temperatures and thermal fluxes to test the performance and efficiency of TBC fabricated with SST. The FE model was constructed based on solid brick elements. Materials properties prescribed for the layers and substrate were taken from the previous Chapter 2 and are summarised in the next section. In these virtual tests the exposed surface of the model was subjected to a sudden temperature change of 1600°C and the back surface of the model kept at a constant temperature of 25°C simulating the effect of the whole structure having a high thermal capacity. Then, a thermal-stress transient analysis was conducted.

5.4.1 Material Properties

Material properties of the Zirconia/Nickel powder and the Nickel substrate used in the Finite Element (FE) simulations were determined in Chapter 4, with the remaining material properties taken from literature [Cao, Vassen and Stoever, 2004]. Properties of the individual layers were determined from Micromechanical (MM) equations used in Wakashima et al. [1992] and Rule of Mixtures (RoM). Thermo-mechanical properties of Zirconia and Nickel are displayed in Table 5.2, with the material properties of the individual layers which were determined by the MM and RoM are outlined in Table 5.3.
Table 5.2: Thermo-mechanical properties of Zirconia and Nickel

<table>
<thead>
<tr>
<th>Material Property</th>
<th>Zirconia</th>
<th>Nickel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elastic Modulus, GPa</td>
<td>53</td>
<td>207</td>
</tr>
<tr>
<td>Poisson's Ratio</td>
<td>0.25</td>
<td>0.312</td>
</tr>
<tr>
<td>Bulk Modulus, GPa</td>
<td>35.3</td>
<td>183.5</td>
</tr>
<tr>
<td>Thermal Conductivity, W/mK</td>
<td>2</td>
<td>72</td>
</tr>
<tr>
<td>Shear Modulus, GPa</td>
<td>21.2</td>
<td>78.9</td>
</tr>
<tr>
<td>CTE, (x 10^{-6}) 1/°C</td>
<td>7.2</td>
<td>1.3</td>
</tr>
</tbody>
</table>

Table 5.3: Thermo-mechanical properties of each individual layer of TBC system using MM and RoM

<table>
<thead>
<tr>
<th>Layer</th>
<th>Single</th>
<th>Double</th>
<th>Triple</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Top</td>
<td>Base</td>
<td>Top</td>
</tr>
<tr>
<td>Thermal Conductivity, W/mK</td>
<td>2</td>
<td>7.12</td>
<td>2</td>
</tr>
<tr>
<td>CTE, (x 10^{-6}) 1/°C</td>
<td>7.20</td>
<td>1.06</td>
<td>7.20</td>
</tr>
<tr>
<td>Density, kg/m³</td>
<td>6037</td>
<td>7459</td>
<td>6037</td>
</tr>
<tr>
<td>Specific Heat, J/mK</td>
<td>656</td>
<td>558</td>
<td>656</td>
</tr>
<tr>
<td>Elastic Modulus, GPa</td>
<td>53</td>
<td>130</td>
<td>53</td>
</tr>
<tr>
<td>Poisson's Ratio</td>
<td>0.25</td>
<td>0.28</td>
<td>0.25</td>
</tr>
</tbody>
</table>

5.4.2 Selected Results

Figure 5.17 present simulation results of the effect of the variation of the coating thickness on the average temperature of the substrate. These results correspond to a single layered coating, the coating thickness ranged from uncoated (0), 50, 100, 200 and 400 μm. With the increase of thickness, a substantial decrease in the average temperature of the substrate (Nickel plate) can be observed. Even the use of thin 50
μm coating leads to the decrease of the averaged temperature by approximately 50%. The coating fabricated with SST normally has thickness between 120 to 170 μm. At these thicknesses it is observed that the average substrate temperature can be decreased from 850°C to approximately 200°C. Such temperatures usually do not represent any threat to the load bearing structures as well as to other structural components made of metals or alloys.

The following results of virtual testing (Figure 5.18) show the distribution of thermal stresses across the coating thickness, with varying gradation. These results clearly demonstrate that the single layered coating experience much higher tensile stresses close to the interface between the coating and the substrate in comparison to the double and triple layered coatings. The difference between the maximum tensile stresses for the single and triple layered coating is approximately 50%. For this reason, it is not surprising that the multi-layered coatings have proven to perform considerably better than their single layered counterparts (as shown in Chapter 4, section 4.6). The same observation has been made in the current scale tests discussed.
above. In general, the results of the virtual modelling have demonstrated that TBCs fabricated with the SST can be efficiently applied as a thermal protection of load bearing structures subjected to very high temperatures and temperature gradients.

Figure 5.18: Thermal stress distribution in the x-direction through TBC over time for varying coating thickness
5.5 Summary and Conclusions

A new testing method for TBCs was developed based on the application of a high temperature flat flame burner. With this design concept, the temperature and thermal flux in the experiments can be controlled by the fuel mixture, which can be further evaluated based on thermal heat energy balance. The operating temperatures of the rig could reach 1900°C, which covers the operating temperature diapason of TBC aimed to work in the conditions corresponding to a hypersonic flight.

With the developed experimental test rig, a number of graded coatings were tested to evaluate the integrity of TBC under realistic temperatures and thermal fluxes. The outcomes of the experiments were encouraging. For example, the multi-layered coatings were able to survive in temperatures of up to 1700°C. For higher flame temperatures of 1900°C, the coatings did not respond well, with the double layered coatings failing by spallation and delamination during the tests. However, the triple layered coatings survived the entire duration of the tests, with the spallation failure occurring after the tests had been conducted and during the cooling stage. The conducted tests have demonstrated that the triple layered coating exhibits a good and adequate performance and can be used as a thermal protection in the conditions described above.

Further, virtual tests by means of Finite Element Analysis (FEA) were conducted to further investigate the performance and the effectiveness of TBC. This study demonstrated that TBCs fabricated with SST can significantly reduce the operating temperatures of the load bearing structure. With the introduction of graded coatings, the thermal stresses experienced through out the TBC were significantly decreased. Together with the physical testings conducted in this Chapter, and the virtual testing, provide confidence that with future development of the technique it can be effectively used in practical applications, specifically in the aerospace industry, where there is a need to cover large curved surfaces and a strong demand for a cheap alternative to the current techniques.
CHAPTER 6

SUMMARY AND CONCLUSION
In this research project, a simple and cost effective technique for fabricating TBCs including Functionally Graded Thermal Barrier Coatings (FG-TBCs) was developed. The technique, called the Slurry Spray Technique (SST), represents an extension of the Wet Powder Spray (WPS) technique. The later manufacturing technique was able to produce homogeneous ceramics coatings; however the quality of these coatings was poor and not appropriate for most of the industrial applications. The development of the new fabricating technique included a new way of the depositing the ceramic powder, investigation of alternative methods for sintering and a pressure stamping stage to stimulate the sintering. Each of these technological stages was extensively investigated experimentally and the optimum parameters have been identified from various scale testings. Finally, the quality of the TBCs fabricated with SST was significantly improved and scale tests conducted under realistic conditions confirmed such that TBCs can potentially be used in some hypersonic applications, in which large and curved surfaces have to be protected from high temperatures and temperature fluxes. The primary objective of the research, which is to develop a cheap alternative to the current manufacturing techniques, was therefore achieved.

Below major achievements of the research project are summarised by each chapter:

In the first chapter, the problem of development of a cheap alternative to the current fabricating technologies was discussed.

In the second chapter the advantages and disadvantages of the existing manufacturing technologies for fabricating TBC were discussed. It was noted that all these current technologies are quite expensive, need a well qualified personnel and often are not applicable to cover large and curved surfaces. Based on the literature review, a suitable base (Zirconia) for the development of a new fabricating technique was selected. Zirconia has an excellent sinterability, low thermal conductivity, low Coefficient of Thermal Expansion, which is critical for the purpose of the current study. In this chapter the typical conditions for a hypersonic flight were reviewed as it is expected that the primary use of the new technique for fabricating TBCs will be for hypersonic vehicles.
The third chapter describes the development of the new technique which was largely based on the analysis of the previous works, consideration of the mechanics and physics behind the various technological stages and thermo-mechanical and scale testing conducted in the following chapters. Effects of the variation of various technological parameters on the quality of the coating were studied using micro-examinations. Finally, the optimum parameters for fabricating single and multi-layered coatings were determined. The new technique includes new features in comparison with the standard WPS technique, which led to a dramatic improvement of the quality of the deposited coating.

The fourth chapter discusses the various thermo-mechanical testing of TBCs fabricated with SST which were conducted to comprehensively estimate and analyse the physical properties of the deposited coatings as well as the effect of various technological parameters (method of sintering, concentration of the constituents, coating thickness). From these tests it was found that the TBCs fabricated with SST have relatively low micro-hardness which makes them inapplicable in situations where high micro-hardness is prerequisite. These include, for example, some high temperature applications associated with mechanical contact with other solid bodies or with abrasive particles. However, in the applications where high micro-hardness is not required the fabricated coatings had comparable thermal and mechanical properties to that which were obtained with the standard fabricating techniques. In particular, the thermal conductivity was even lower than the values for the corresponding coatings fabricated with Electron Beam Physical Vapour Deposition (EB–PVD) techniques. Another important conclusion was made from thermal cyclic loading results, which showed the need for non-destructive defect detection methods to inspect the coatings prior their use.

In the fifth chapter a new method for scale tests of TBCs under realistic conditions was developed and utilised for investigations. The new method was based on the use of a high temperature flat burner, which allows achievement of controllable and reproducible high temperatures and thermal fluxes. Tests conducted with the flat burner demonstrated that the graded TBCs fabricated with SST are able to survive
realistic thermal conditions, which correspond to hypersonic applications. It has to be noted that only triple layered coatings were able to survive realistic tests. The scale tests were supported with a modelling approach which was based on numerical transient analysis with Finite Element software package ANSYS. The numerical simulations confirmed that the coatings can significantly reduce the operating temperatures of the load bearing structures. In addition, the simulations have also demonstrated that the reason for survival of triple layered coatings is the significant reduction of the maximum tensile stresses in the coating, which are generated due to the mismatch in material properties of the coating and the substrate. The functional grading allows the reduction of thermal and residual stresses, which, in its turn, leads to improved reliability survivability of the TBCs.

The low microhardness results from the thermo-mechanical investigations, referred to in Chapter 4, had indicated that the TBC fabricated with the SST coating was not applicable to high temperature aerospace applications. However, the high temperature burner results have shown that the coating fabricated by the SST, was able to be subjected to extreme temperatures (of up to 1700°C) and survive the duration of the tests. It is therefore concluded the TBCs produced by the SST are able to be used in limited aerospace applications, where physical contact between components is not required.

All results obtained in this research project indicate that the developed techniques can be applied to real structures. That the technique under development is approximately one hundred times cheaper (to cover the same area of the structure with thermal protection) than the standard techniques, this indicates the new technique can be considered as a cost effective alternative to the current technologies.

Future work could involve further improvements to achieve higher quality of coatings, for example, by the application of a base layer bond coating, such as NiCrAlY. The introduction of this bond coat would decrease the effect of Thermally Grown Oxides (TGO) on the coating life. TGO have been known for a long time to substantially affect on the life of a TBC. Further full scale tests are also required to
confirm the integrity and reliability of such coatings in real conditions.