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Manufacturing and Plasma Wind Tunnel Testing of UHTC Coated Components for Hypersonic Applications

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Abstract

Ceramic coatings are widely used as thermal barrier or as oxidation barrier, in many industrial applications. The use of UHTC is mandatory when dealing with hypersonic vehicles characterized by high thermal flux in oxidizing environment.

Ultra-high temperature ceramics (UHTCs) such as ZrB_2 and HfB_2 have been identified as potential candidates for operating in harsh conditions and their resistance to ablation. However, their poor fracture toughness and thermal shock resistance strongly limit their applicability. On the contrary, metallic refractory alloys have good thermo-mechanical properties but suffer from poor oxidation resistance.

The combination of metallic substrates with plasma spray coating of UHTC can potentially couple the high oxidation resistance of UHTCs to the better thermo-mechanical properties of substrates.

CIRA and RINA-CSM is developing and improving the features and the capabilities of the UHTC coating by plasma spray on metallic substrates. A wide campaign for characterization and selection of ceramic coatings, substrates and spraying techniques were performed.

Arc-jet test campaigns on small samples are carried out in the 2MW facility GHIBLI. Large scale metallic cone coated with UHTC have been tested in the 70MW SCIROCCO facility, to characterize the behavior of the proposed materials combinations under representative conditions using also representative geometries and size.

The paper shows all the preparatory activities and the results obtained in PWT tests on UHTC coating.

Keywords: *UHTC Coating, Metallic Substrates, Hypersonic Application, PWT Testing.*

Nomenclature

Latin

BSE	Backscattered Electron
CIRA	Italian Aerospace Research Centre
PWT	Plasma Wind Tunnel
RINA-CSM	Rina Consulting – Centro Sviluppo Materiali Spa
SE	Secondary Electron
SEM	Scanning Electron Microscope
UHTC	Ultra-High Temperature Ceramics

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1. Introduction

Since 2000, in the framework of the national aerospace research program (PRORA-SHS) and within various other National and European programs, CIRA has studied, developed, and tested monolithic UHTCs and, in cooperation with RINA-CSM, UHTC coatings on different high temperature structural materials. Small winglets and nose made in UHTC or UHTC coated were designed and manufactured for in-flight qualification.

UHTC coatings technology is for sure one of the candidates to solve the critical issue of sharp leading edges of hypersonic vehicles, where the high thermal loads are coupled with highly reactive plasma environment.

As for all type of coatings main criticality are the good adhesion and the thermal expansion mismatch between the coating itself and its substrate. In order to improve the adhesion of the coating, to better match different thermal expansions, and in order to provide an additional oxidation barrier too, some interlayers are used between the ceramic coating and the bulk substrate.

CIRA and RC-CSM are conducting a wide campaign of characterization and selection of ceramic coatings, substrates and spraying techniques. The campaign started with the selection of a set of substrate materials, a set of interlayer materials, and a set of ceramic materials. The chosen substrate materials are high temperature alloys already used in the aerospace field, and refractory metals that are more innovative in this field. In particular, for the second set of materials, the ambitious objective is to be able to use, in oxidizing environments, materials that have been usually discarded because, even if characterized by a very high melting point, they show a poor oxidation resistance.

The coatings for the selection campaign have been selected with the same main objectives: protect the bulk material and guarantee a good adhesion. The campaign foresees a large number of spraying activities, aimed to tune up the technique.

The first coated specimens were used for the first evaluations and pull tests. Once the coating procedure is well tuned, for each materials stack, the coating was applied to conical specimens, much more representative of the final aerospace applications. In this second phase, the spraying technique was tuned in order to have a good coating on the sharp tip of the cones.

Once the best combination of substrate-interlayer-coating were identified, the second step phase started with realization of small coupons with flat faced cylinder, sharp cones and wedges and blunt cylinders to be tested in the CIRA 2MW plasma wind tunnel facility GHIBLI, to test the coupons in a real hypersonic environment characterized by high temperatures and reduced pressures. During this phase, in parallel, some trial depositions on CMC substrates were executed and considering the results, small samples to be tested in GHIBLI were realized and tested.

The last step consisted in the realization of large test articles in the order of tens of centimeters to be tested in the 70 MW plasma wind tunnel facility.

2. Test Article Manufacturing

2.1. Screening Phase

Test samples were realized using two kind of materials: a Ni based superalloy (Material A) and a refractory metal (Material B). The samples were coated by plasma spraying with two kind of ceramic coatings: an oxide, thermally insulating coating (Coating 1) and an UHTC (Ultra High Temperature Ceramic) coating (Coating 2). To improve the adhesion between substrate and coating, a metallic intermediate layer was deposited by plasma spraying.

For the deposition, the CAPS (Controlled Atmosphere Plasma Spraying) facility available at RC-CSM was used.

Two different geometries of samples were realized, as a function of the specific test to be carried out on them:

a) Disc shaped samples, 25.4 mm of diameter and 2 mm of thickness.

b) Cone shaped samples, 30 mm of base diameter and 30 mm di highness. The tip of the samples was rounded with a 1 mm radius, which was maintained after the coating deposition. Some coated and uncoated samples are shown in Figure 1.

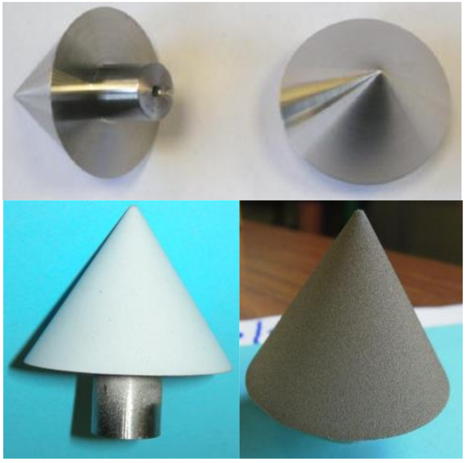

Fig 1. Conical samples before (top) and after coatings (bottom)

2.2. Small Scale Relevant Environment Test Phase

Test samples were realized using a Tungsten Alloy as substrates. The samples were coated by plasma spraying with the selected UHTC (Ultra High Temperature Ceramic) coating (see figure 2). In both case the same metallic intermediate layer was deposited:

Metallic substrates samples were realized with the following shapes:

a) Cone shaped samples, 30 mm of base diameter and 30 mm di highness. The tip of the samples was rounded with about 1 mm radius

b) Blunt Hemicylinders samples, 20 mm edge of squared base and 10 mm of cylinder radius;

c) Sharp Wedges, 30 mm of height, 10° semiapex angle and about 1mm of radius at the tip.

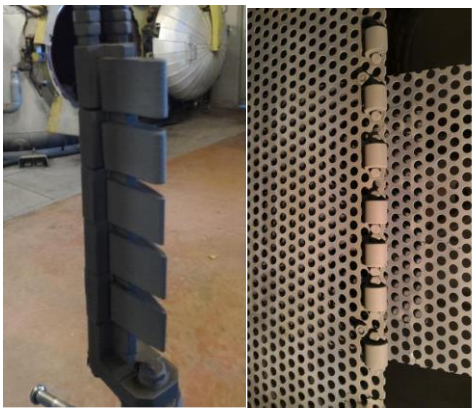

Fig 2. Sharp wedge and blunt cylinders after coatings

2.3. Large Scale Relevant Environment Test Phase

For the large scale a sharp cone has been considered representative of nose tip of hypersonic vehicles. Geometry was realized with Tungsten Alloy substrate. Long time was spent to optimize deposition on large test article.

During deposition on sharp cone it was observed the onset of a crack with subsequent grow. After dye penetrant test, it was decided to reduce the length of the cone and to continue the coating deposition achieving satisfactory results. Figure 3 show test article picture after coating.

Fig 3. Metallic substrate Sharp cone and wedge and CMC substrate wedge after coatings

3. TEST METHODS AND FACILITIES

3.1. Pull Test

On the disc shaped samples, pull tests were carried out according to the ASTM C633-01, in order to verify the adhesion of the coating system (constituted by the intermediate layer and the UHTC top layer) onto the specific substrate (Material A or B).

The test setup is shown in Figure 4. The substrate/coating system was glued between two steel cylinders, which were pulled apart, measuring the applied load.

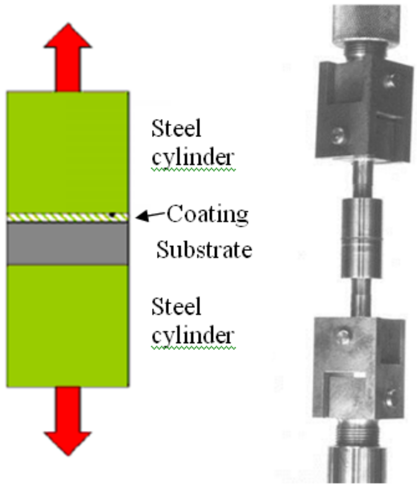

Fig 4. Schematics and realization of the pull test setup.

3.2. Plasma Torch Test

Conical samples were installed on a ceramic sample holder and a plasma stream was directed on the tip. The stream was generated by a F4 plasma torch, produced by Sulzer Metco (Switzerland).

Ar and H_2 were used as plasma gases. The torch was installed in the same CAPS apparatus used for the coating deposition.

The distance between the nozzle of the plasma torch and the tip of the conical sample was varied to obtain the target temperature on the tip. The sample temperature was measured by contemporary using an IR thermo camera and a pyrometer. Figure 5 shows the setup for the test.

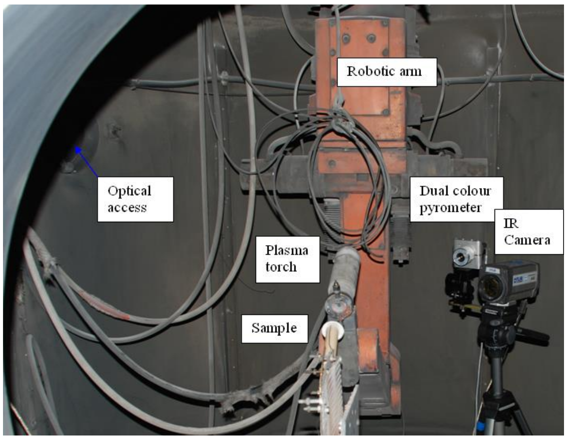

Fig 5. Plasma torch test setup

3.3. Plasma Wind Tunnel Test

Preliminary tests on small scale samples were conducted using the Ghibli facility. GHIBLI is a hypersonic high enthalpy arc heated continuous facility for the development of experiments on models of small size. It is particularly well suited for TPS material samples characterization. The hot flow is generated in a 2 MW segmented arc heater. During each test, the arc-heater was ignited and the amperage and gas flow rates were set. The temperature of the sample was recorded using dual color pyrometers. For the large-scale test article the SCIROCCO 70 MW facility was used. Both facilities are available at the Italian Aerospace Research Centre.

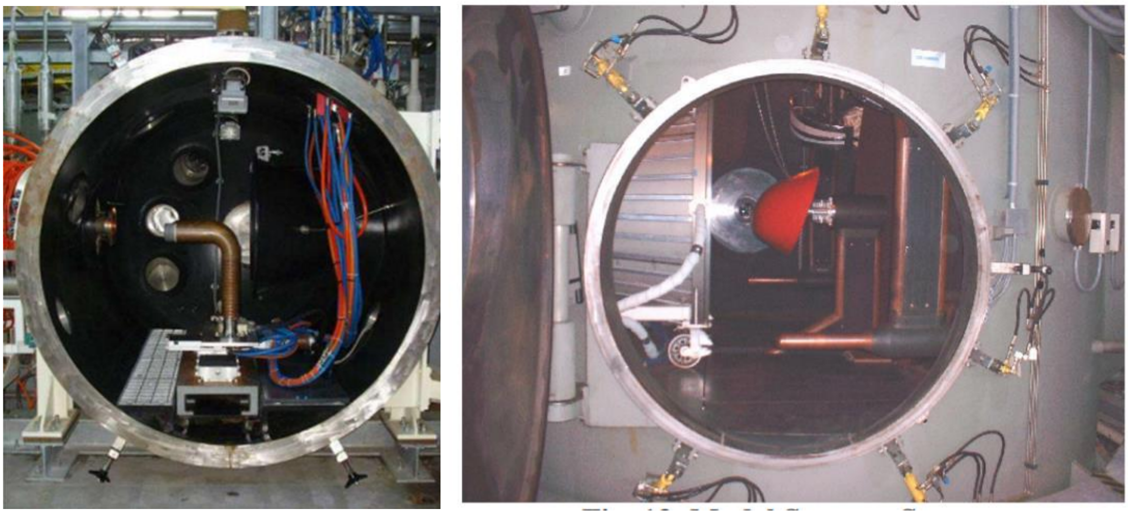

Fig 6. GHIBLI and SCIROCCO Test Chamber

4. RESULTS AND ANALYSIS

4.1. Pull Tests Results

Figure 7 reports the results obtained in the pull test. For each substrate/coating system three samples have been tested. Figure 8 shows a few samples after the pull tests.

The obtained values are typical for plasma sprayed ceramic coatings deposited onto metallic substrates.

In some cases, the adhesion of the coating was so good that during the pull test the glue failed earlier than the coating.

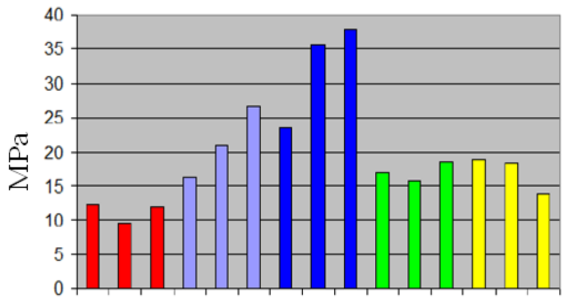

Fig 7. Pull tests results on five different coating/substrate systems.

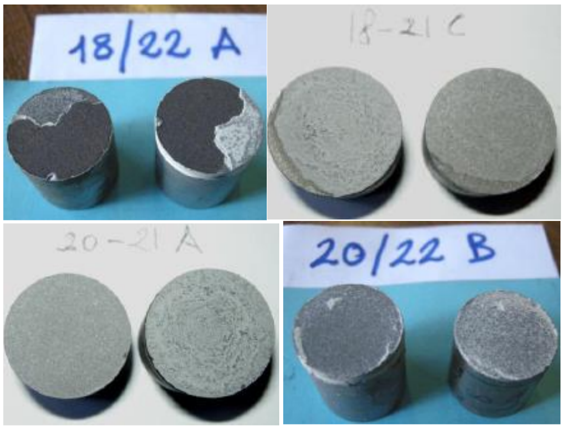

Fig 8. Samples after the pull tests

4.2. Plasma Torch Test Results

Material A samples (Nickel based alloy)

In a first test the sample was heated up to 840°C for 360 seconds. Apparently, the sample sustained the load, but once inspected after the test a crack was visible on coating.

A second nickel/coating system was tested, but the results were even more negative; after 300s at 950°C the coating felled in pieces. In both the cases, the damage occurred during the cooling phase.

Material B samples (refractory metal)

A sample coated with UHTC was exposed to different temperature levels and exposure time. The final test was 400s long and the specimen was exposed to about 1700°C. Figure 9 shows the measurements of the dual color pyrometer.

The images reported in Figure 10 show the comparison of a coated untested sample, a coated tested sample, and an uncoated tested sample. It is evident the erosion occurred on the uncoated sample, even if exposed to the plasma for a much shorter time

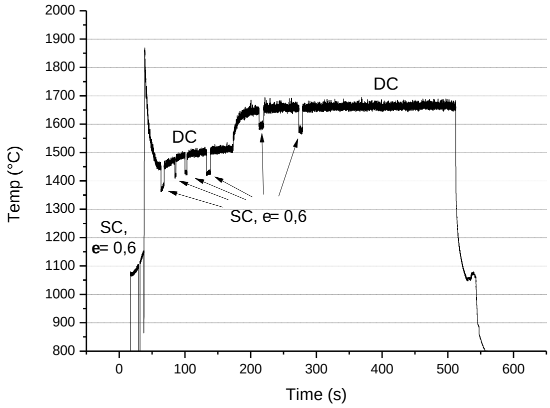

Fig 9. Pyrometer measurement during the final test executed on the first UHTC coated refractory metal sample.

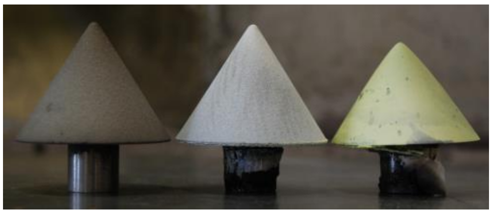

Fig 10. Conical samples uncoated and coated with different coatings

4.3. Small Scale Plasma Wind Tunnel Test Results

Four samples were exposed to plasma flow. Samples were subjected both to temperatures lower than the maximum working temperature of the coating, i.e. 1750° C, corresponding to the melting temperature of the SiO_2 layer which is formed during hot oxidation of the UHTC material and exceeding this limit.

The thermal history of the sample identified as nr. 5 is shown in Figure 11, where the readings of three pyrometers pointed at different areas of the sample are shown. As can be seen, the maximum temperature reached was close to 1700°C.

Figure 12 shows a section of the sample. The coating appears intact, not cracked and of constant thickness. Even at this magnification it is possible to observe on the surface of the sample the presence of roundish regions of dark color.

Figures 13 and figure 14 show the same coating region observed respectively backscattered and with secondary electrons. The comparison between the two micrographs allows a more detailed analysis. The dark spots observed near the surface are consisting of bubbles surrounded by material with a glassy appearance, probably SiO2. In these areas are present bubbles generated by the oxidation of Si, partly as SiO2, still solid at 1700°C, and partly as SiO gas. The latter expanding deform the vitreous SiO2. In the observed area, the Mo layer appears to be broken into large blocks, and a similar structure can be also observed in the lower layer of the UHTC coating. The presence of SiO2 is confirmed also by the EDS analyzes. Si is present throughout the sample; traces of Si can be found also in the interlayer due to a diffusion of Si caused by high temperatures. While the concentration of Si in the surface layer is very high, the Si is absent in a strip close to the surface. This result is due to the oxidation of SiC and the consequent migration of the Si to the surface. Hence also the spongy appearance of the layer.

As for comparison results of PWT test on the sample identified as 7 is reported. The sample was subjected to the temperature history shown in Figure 15. In this case it was reached a temperature of 1800°C. Looking at the graph it is clear that the temperature has a flex a 1700 ° C. The fact that, with the same incident flow, the temperature increases with positive derivative can be attributed to the triggering of an active oxidation phase, in particular of Si. This has led to a collapse of the lining on the side parts of the sample.

The SEM analysis of undamaged section areas showed that, despite the active oxidation of the surface, the central area of the coating appears much less damaged than the previous sample. In particular, the interlayer appears intact and free of oxidation, as shown in Figure 16, which shows one section of the sample observed at the backscattered electrons and at the secondary electrons.

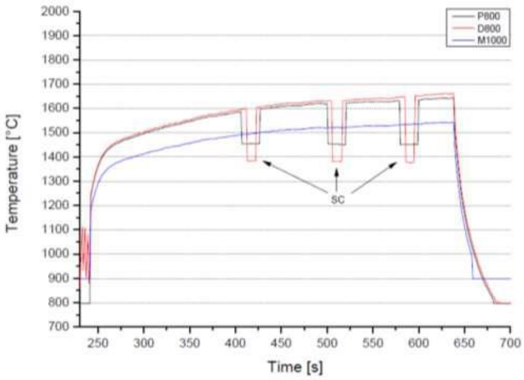

Fig 11. Pyrometer measurement during the PWT test executed on the sample 5

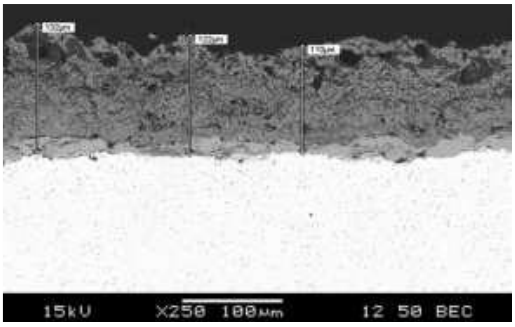

Fig 12. SEM-BSE images of mid section of the sample 5

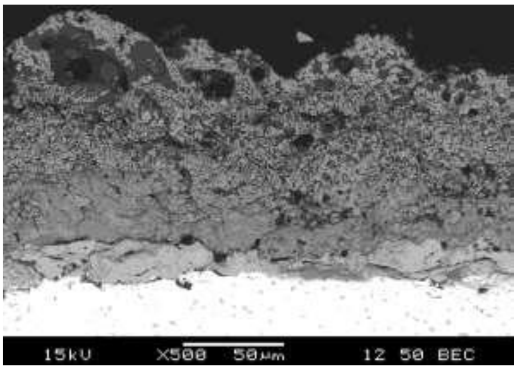

Fig 13. SEM-BSE image of mid section of the sample 5

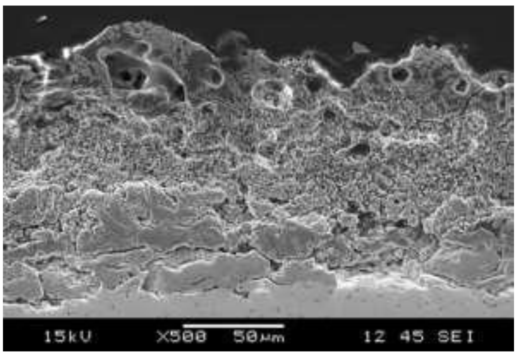

Fig 14. SEM-SE image of mid section of the sample 5

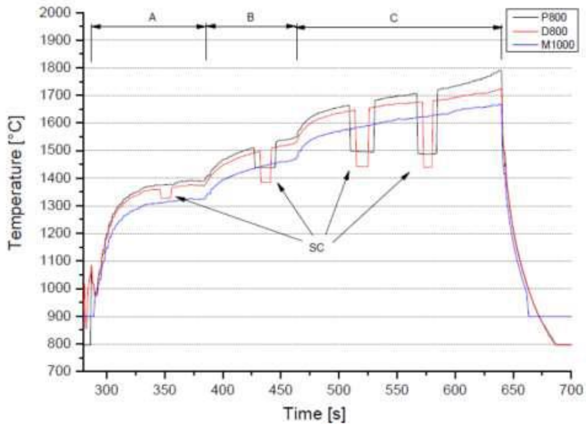

Fig 15. Pyrometer measurement during the PWT test executed on the sample 7

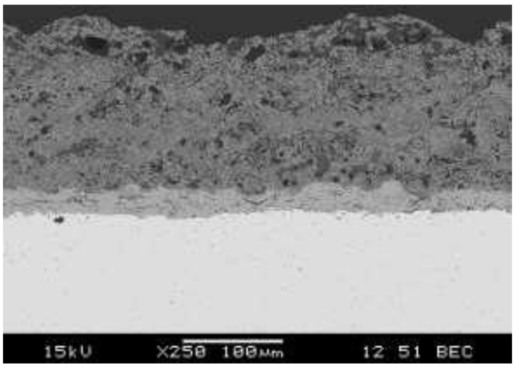

Fig 16. SEM - SE image of mid section of the sample 7

4.4. Large Scale Plasma Wind Tunnel Test Results

The tests were performed at CIRA Plasma Wind Tunnel "SCIROCCO" facility. The cone was inserted into steel adapter and joined on one of the calibration probe arms. Figure 17 shows integrated test articles. Pyrometers and IR cameras were used for temperature evaluation on the test articles surface. Furthermore, four type k thermocouples were installed inside the cone at different distance from the tip. Mandatory information was the verification that tip allow to withstand temperature beyond 1500°C for at least one test.

Fig 17. Integrated test article

Two tests were performed at the same conditions. Unfortunately in both cases small tilting (pitch down and yaw) of the cone due to large deformation of steel flange interfacing the cone with support was responsible for losing the pyrometer pointing. Thanks to the calibrated IR camera the surface temperatures were recorded (see Figure 18).

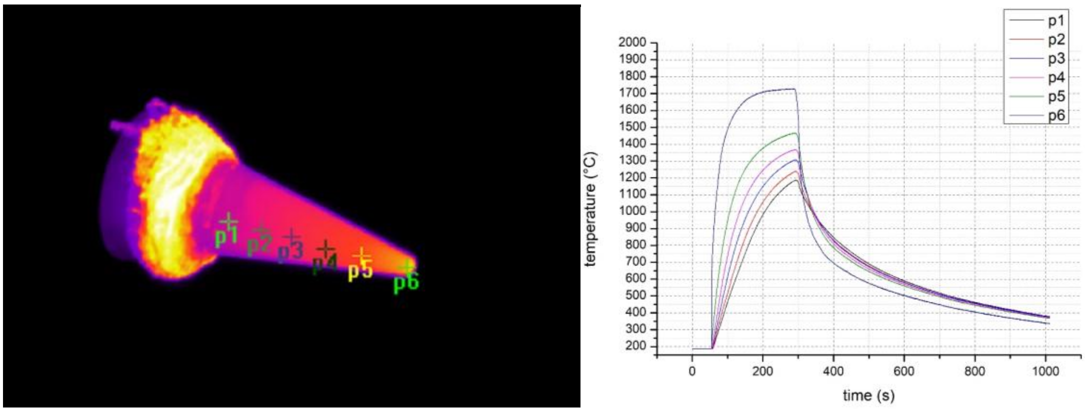

Fig 18. IR image and Measured (calibrated)Temperature

After two test the blant cone exhibit only a white discoloring on the tip steel keeping good adhesion and geometrical features as shown in figure 19.

Fig 19. Post-tests Blunt come appearance

5. CONCLUSION

The capabilities of a UHTC coating by plasma spray on metallic substrate was investigated. A wide campaign for characterization and selection of ceramic coatings, substrates and spraying techniques were performed. A suitable metallic interlayer was found improving the coating behavior in terms of adhesion and thermal stability. After a preliminary screening by pull tests and plasma torch test the best metallic substrate/interface coating compositions were identified. A wide small scale PWT test campaign were put in place on different shape specimens to investigate the system behavior in relevant environment. Large scale test on the best combination of substrate/coating with the developed interlayer demonstrate the capability of the system to withstand the required conditions. As a next step, further development of UHTC coating on CMC substrates will be investigated with the aim to enhance oxidation resistance.

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