

# The behavior of multilayer ceramic protections at quick thermal shock

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**Abstract:** *Protective layers of “hot parts” of the turbo engines as well as co-generative systems of energy industry are exposed to a combination of wear factors which may act together at high values. The main goal of the paper is the behavior of some advanced layers, duplex and triplex, multifunctional, ceramics in relation to the most complex wear factor and disturbing as well, the quick thermal shock.*

*The quick thermal shock test installation designed and constructed by the INCAS covers the domain of some high gradients of heating/cooling and is currently integrated in a network of European infrastructure that evaluates the properties of functional layers for turbo engines.*

*Micro-structure inter- and intra- facial changes gradually induced in ceramic structures are highlighted and on this basis their ranking and selection for application on physical parts are established.*

**Key Words:** *turbo engines, wear factors, quick thermal shock, thermal fatigue, TBC*

## 1. INTRODUCTION

Gas turbine generators work at mechanical and thermal superior limits, plus the corrosive effects of chemical fuels. The temperature in commercial aircraft turbines can reach 1500°C [1]. Knowing the materials behavior at high heating and cooling speeds is very useful in managing extreme operational conditions that occur during flight such as stopping of the engine in flight, bailed landing, etc and also in metallurgical industry or in high power machinery operation.

From all the wear factors that work simultaneously on the “hot parts” (blades, adjustable nozzles, burning chamber, diffuser) of the turbo engines – temperatures above 1500°C, quick thermal shock, pyrolyze particle erosion to speeds above Mach 3, corrosion, adhesion, etc.- the thermal factor is the most disturbing.[2]

In the case of “hot parts” of turbo engines, temperatures vary depending on flight operation rules on taking off, landing, intermediate cruising, engine stopping in flight, bailed landing, etc. The increases in thermal efforts for short duration can have considerable value

and lead to plastic deformation of the material.

The use of protective systems in the case of “hot parts” of turbo engines is absolutely necessary in view of the operating at high temperatures.

## 2. EXPERIMENTS

Taking into account the extreme operational conditions of the “hot parts” of the turbo engine appeared to be necessary to study the material behavior at high rate speeds of heating-cooling, at quick thermal shock.

### 2.1. Materials

Multilayer samples composed of stainless steel support, bonding layer MeCrAlY and ceramic thermal barrier coating (TBC) layer have been used in experiments.

### 2.2. Methods and instrumentation

To assess structural changes due to thermal shock, thermal barrier coatings were tested at quick thermal shock and then investigated by electron microscopy.

Layers of protection were obtained by successively depositing the bonding layer and ceramic layer by air plasma jet method on a METCO -type installation.

It should be noted that there is only two ISO standards referring to tests for TBC materials. ISO 13123-Metallic and other inorganic coatings-Test method of cyclic heating for thermal-barrier coatings under temperature gradient and ISO 14188 N985-Metallic and other inorganic coatings-Test methods for measuring thermal cycle resistance and resistance to thermal shock for thermal barrier coatings. [10,11]

Generally the manufacturers as well as the materials users have both created their own equipment. [3]

The parts from the aerospace industry, space shuttles, “hot parts” of the turbo engines, parts of metallurgical industry, turbine blades from power industry, etc. are subjected to hard heating- cooling cycles within a few tens of seconds.

Below is presented the QTS2 installation, designed and built by the authors for testing materials in extreme conditions of heating-cooling rates. (Fig.1)



Fig. 1 QTS2-Installation for material testing in extreme thermal conditions

Functional parameters of QTS2 installation are: testing materials up to 1500°C, variable heating and quick cooling speeds of the specimen up to 70°C/s, operating in automatic cycle,

monitoring functional parameters, continuous measurement of temperature specimen at heating and cooling, Lab View data acquisition system, view oven heating curve, heating curve, cooling curve of specimen. [9]

### 2.3 Thermal shock resistance test

Thermal shock resistance test aims to reveal micro structural changes of samples tested. Thermal shock test is completed when macroscopic exfoliation damage appears on more than 25% of the TBC surface coating [11].

The thermal cycling has been performed at a temperature of 1200°C.

There were 50 tests for this temperature cycling. There was tested 1 specimen, numbered N14.

The oven is heated at the test cycling temperature. The sample is moved from the environment temperature into the oven.

The heating speed of the specimen is variable depending on the specimen size, type of material, single layer or multilayer.

The specimen is moved from inside the oven to the cooling area where is cooled till about 40°C.

The quick thermal test shock were carried out with the following parameters: measured average specimen heating speed in the first 5 seconds 149°C/s; measured average specimen cooling speed in the first 4 seconds 70.41°C/s; cooling time – 60 s; maintain time in oven-5 min; test duration-6 min; cooling air maximum pressure- 8, 7 bar; cooling air minimum pressure-7.13 bar. Size specimen: 30 x 50 x 1.7 mm.

Images of the specimen N14, before and after thermal shock are presented in Fig. 2.

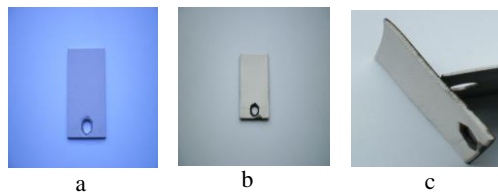


Fig. 2 Specimen N14 before thermal shock test and after test shock at 1200°C  
2a - before thermal shock test  
2b and 2c - after thermal shock test at 1200°C

Fig. 3 shows the graphic of thermal shock test at temperatures of 1200°C and [Fig. 4 presents a Lab View captured image for tests number 15, 16 and 17, specimen N14. The data were obtained with Lab View software program.

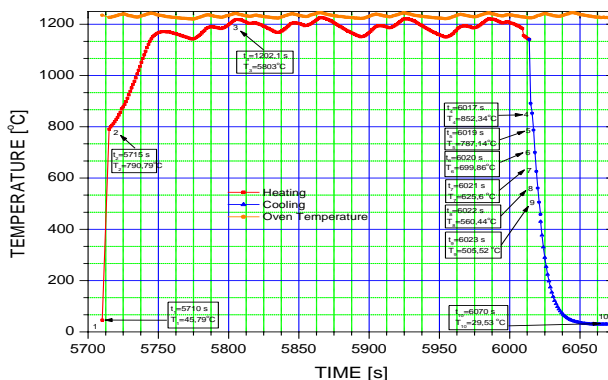


Fig. 3 Graphic thermal shock test N14 specimen after 50 tests at 1200°C

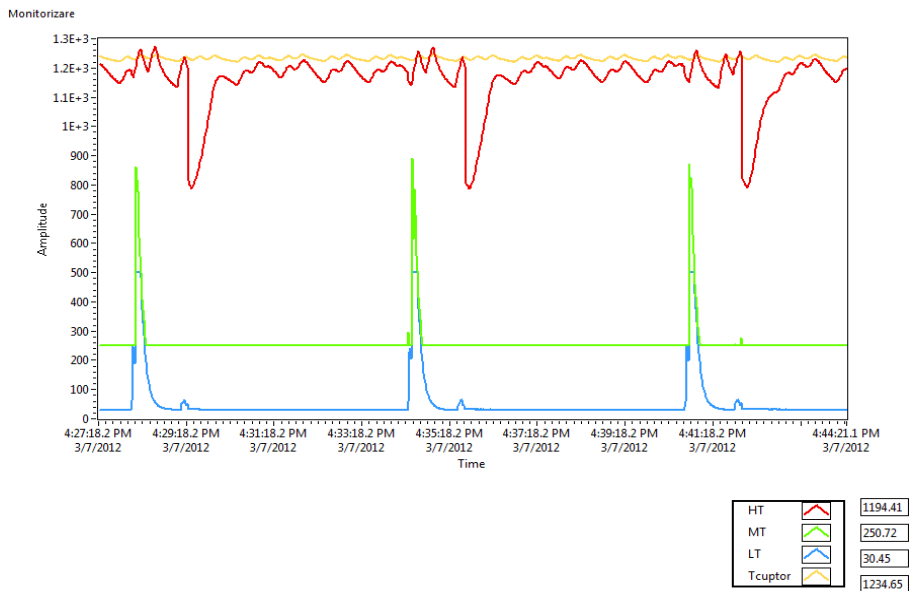


Fig. 4 Lab View captured image of quick thermal test shock, N14 specimen at 1200°C (tests 15, 16 and 17)

### 3. MICROSCOPIC INVESTIGATIONS

Micro structural investigations were made by a scanning electronic microscope SEM. It was made a comparative study of the layers deposited both before and after successive testing of specimens at thermal shock.

The work has been carried out by means of investigations of electronic microscopy on nanometric ceramic multilayer structures such as Me/MeCrAlY/ ZrO<sub>2</sub>•Y<sub>2</sub>O<sub>3</sub>•Al<sub>2</sub>O<sub>3</sub> before and post heat treatment- quick thermal shock, 50 cycles at 1200°C. We mention that nanometric powder ZrO<sub>2</sub>•Y<sub>2</sub>O<sub>3</sub>•25%Al<sub>2</sub>O<sub>3</sub> was achieved by The National R&D Institute for Non-ferrous and Rare Metals -INMR Bucharest [8].

The testing parameters of specimens within the program, correspond to the work temperature indicated by the known European temperatures rules of 1200<sup>0</sup> C and they are superiors in the fields of heating-cooling gradients [3].

The testing conditions put into evidence the characteristics of multilayer structures in relation to their sensitivity to quick thermal shock due to the difference between the expansion coefficient of metallic support and ceramic structure.

Investigations were performed by a high resolution scanning electron microscope (HRSEM) equipped with a field emission gun (FEG) and an energy-dispersive detector (EDS) of EDAX type.

The specimens utilized were noted N14, E2.

The Support material is stainless steel, coated with nanostructured ceramic layer of ZrO<sub>2</sub> • Y<sub>2</sub>O<sub>3</sub> • Al<sub>2</sub>O<sub>3</sub> (E2) before and after (N14) thermal shock test.

The work was carried out by the micro structural analysis associated with qualitative and quantitative microanalyses of X rays (EDS.).

Analysis of the basic materials for specimen support, stainless steel, indicate the average value for 4 fields :Si=1,58%;Mo=5,32%;Cr= 23,56%;Fe= 63.5%;Ni=6.25%.

The bonding layer, especially after thermal shock treatment is non-uniform, with frequent oxide particles (fig. 5).

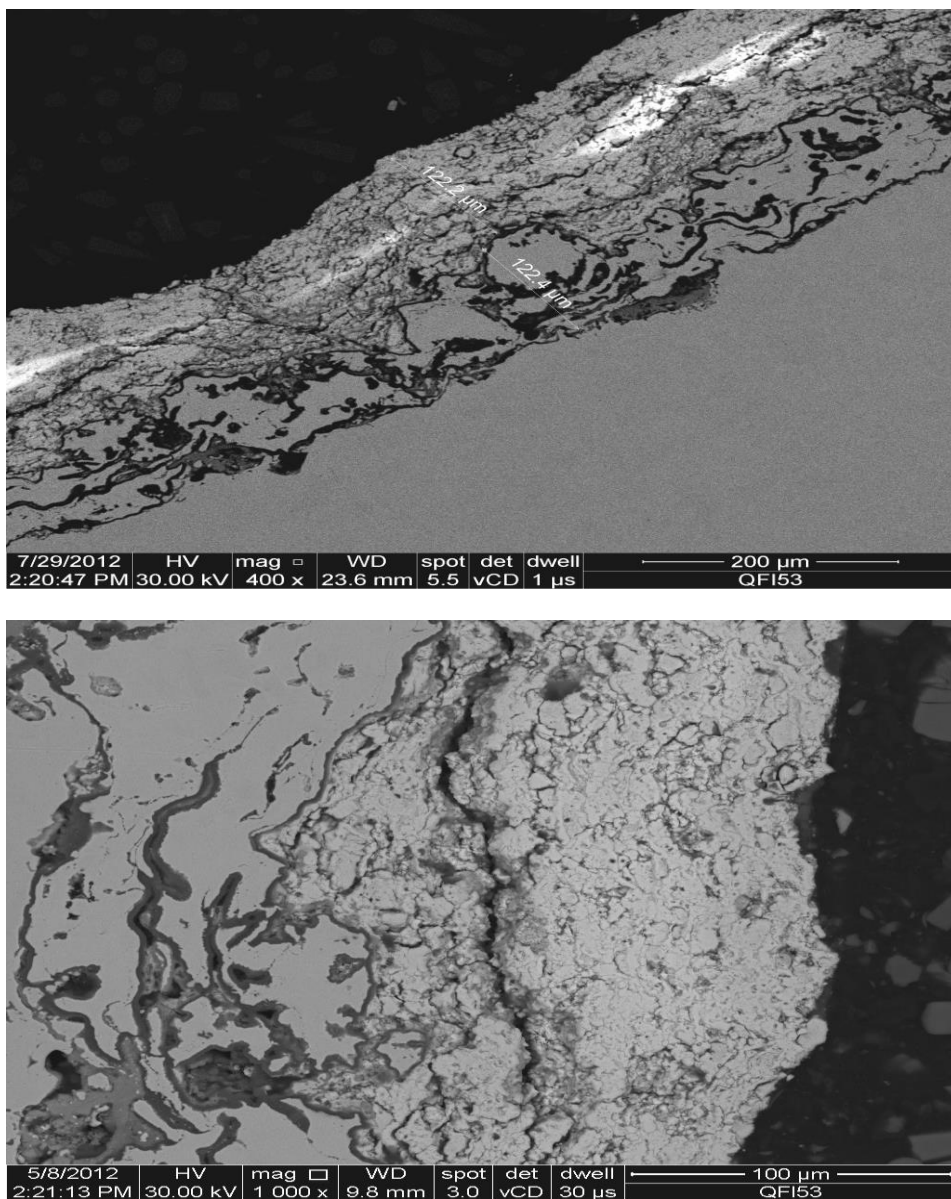


Fig. 5 Back scattering electron images, highlighting the general appearance of the layers deposited on the sample N14 (x400, x1000).

After testing non compacted volume of ceramic powder alternated with the compacted one appear in the ceramic layer.

In the ceramic layer post thermal treatment one can see also, the presence of micro cracks and cracks in the adhesion zone, bonding layer-ceramic layer, having the size of about 20-50  $\mu\text{m}$  (fig. 5).

For the specimen under post thermal shock treatment in the bonding zone the analysis of distribution images EDS indicates a quasi-uniform repartition of Ni and Cr and a less uniform one of Al appearing as oxides.

In the ceramic layer oxides of Zr and Y are uniformly distributed. (fig. 6)

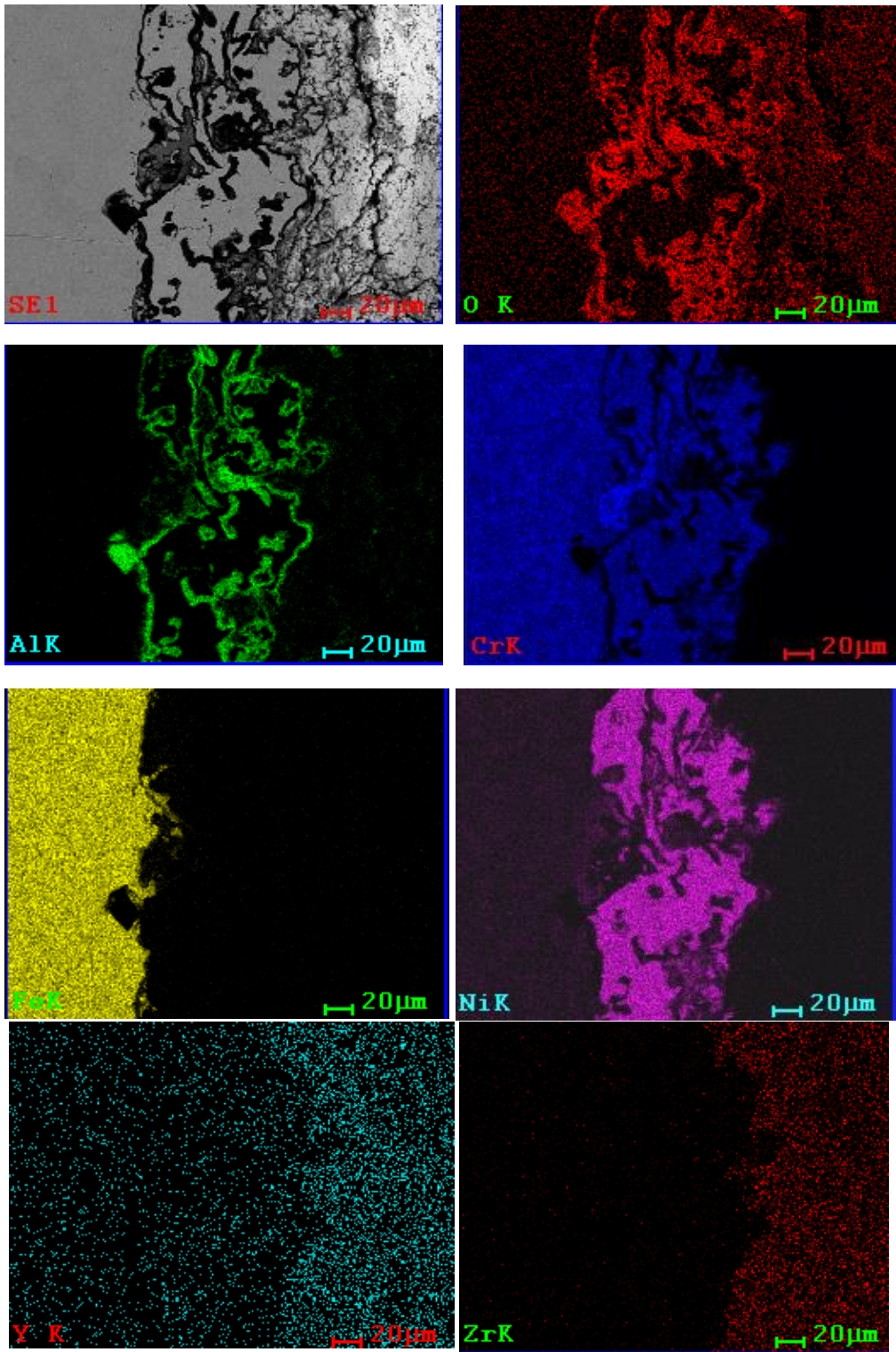


Fig. 6 Images of back scattered electrons and surface distribution of the characteristic X-ray relative intensity for the detected elements in the N14 specimen

In the context of the analysis of all the EDS for non-thermal shock treatment specimen, we remark the supplementary concentration of Al at the interface bonding layer-ceramic layer and Zr in the near of bonding layer, compared with thermal shock treatment EDS images.

The analysis of untreated specimen images indicates a relatively uniform thickness of bonding and ceramic layers, homogeneity and appropriate adhesion within the corresponding technology APS-air plasma spray for a new type of elaborated ceramic powder  $ZrO_2 \cdot Y_2O_3 \cdot Al_2O_3$ .

The testing in thermal shock conditions that correspond to functional conditions of turbo engines shows the appearance of micro and macro structures and morphology changes of ceramic layers which are consistent with mechanical and physical characteristics of utilized ceramic materials.

#### 4. CONCLUSIONS

1. The “hot parts” of the turbo engines but also those of the co-generative systems from power industry are subject to factors of wear-corrosive, erosive, adhesive, by thermal fatigue, which act simultaneously at high values.
2. The thermal shock acts most disturbing on the endurance of TBC-Thermal Barrier Coating.
3. In order to evaluate the behavior of materials under extreme conditions of the turbo engines the thermal shock tests of the elaborated materials, were made with an original installation conceived and achieved by the authors of this paper. The test parameters as per QTS2 installations were the following: average heating rate specimen for first 5 seconds –  $149^\circ\text{C/s}$  and maximum cooling rate specimen –  $87.29^\circ\text{C/s}$ .
4. The quick thermal shock testing on QTS2 installation, conceived and realized by INCAD, allowed the hierarchy of the elaborated materials.
5. The quick thermal test shock parameters increase induces macro and micro structural changes of the TBC layers-porosity, developing networks of reticular cracks, oriented mainly horizontally.
6. After the thermal shock the electron microscopy study reveals the formation of complex oxides layers, with nano or micron thickness at the interface bonding /ceramic layers, TGO-Thermal Oxide Growth – due to migration of reactive elements from the bonding layer and their subsequent oxidation.
7. TGO layer grows, due to increasing thermal shock parameters amounts, (temperature, and heating-cooling speed) and may represent the fundamental cause which initiates the delaminating of the ceramic coating of the turbo engine and finally its deterioration.
8. In post thermal treatment of the ceramic layer there are alternated compacted and non-compacted zones.

#### REFERENCES

- [1] Edwin Adelpour, *Thermal Barrier Coating for Gas Turbine Engine*, NASA/TM—1999-209453, October 1999 39/12 (1987) 28-31.
- [2] B. A. Pint, I. G. Wright, W. J. Brindley, Evaluation of TBC Systems on Novel Substrates, *Journal of Thermal Spray Technology* 9/2, 198-203, 2000.
- [3] Ann Bolcavage, Albert Feuerstein, John Foster, and Peter Moore, Thermal Shock Testing of Thermal Barrier Coating/Bondcoat Systems, *Journal of Materials Engineering and Performance*, Volume 13, No. 4, pp. 389 – 397, August 2004.

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- [4] V. Manoliu, Gh. Ionescu, A. Stefan, C. Valcu, *Principle regarding the thermal test of materials in extreme conditions*, 15<sup>th</sup> IFHTS&SMT20 Congress, Vienna, 24 – 29 September 2006.
- [5] V. Manoliu, A. Stefan, Gh. Ionescu, S. Gaman, I. Dinca, A. Stan, *Nanostructured multilayer behavior at quick thermal shock*, The Materials Science and Engineering Congress, Nurnberg 1-4 September, 2008.
- [6] V. Manoliu, Gh. Ionescu, A. Stefan, C. Serghie, S. Ilina, *Quick Thermal Test Shock Installation - QTS1*, *INCAS BULLETIN*, (online) ISSN 2247–4528, (print) ISSN 2066–8201, ISSN–L 2066–8201, <http://bulletin.incas.ro>, Volume 1, Number 2, pp. 116-121, 2009.
- [7] V. Manoliu, Gh. Ionescu, C. Vilcu, *Method for testing materials in thermal extreme conditions*, International Conference AFASES, Brasov, 27-29 May, 2010.
- [8] S. Ilina, Gh. Ionescu, V. Manoliu, R. R. Piticescu, *Nanostructured zirconia layers as thermal barrier coatings*, *INCAS BULLETIN*, INCAS – National Institute for Aerospace Research “Elie Carafoli”, Bucharest, Romania, (online) ISSN 2247–4528, (print) ISSN 2066–8201, ISSN–L 2066–8201, <http://bulletin.incas.ro>, Vol. 3, Number 3, pp. 63-69, 2011.
- [9] Gheorghe Ionescu, Sorin Dimitriu, Victor Manoliu, Sorina Ilina, Constantin Valcu, *Wear factors acting on aviation turbo engines*, The 13<sup>th</sup> International Conference “SCIENTIFIC RESEARCH AND EDUCATION IN THE AIR FORCE” AFASES, Brasov, 26-28 May, 2011.
- [10] ISO 13123-Metallic and other inorganic coatings-Test method of cyclic heating for thermal-barrier coatings under temperature gradient.
- [11] ISO 14188 N985-Metallic and other inorganic coatings-Test methods for measuring thermal cycle resistance and resistance to thermal shock for thermal barrier coatings.