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The porosity assessment of thermal barrier coatings obtained by APS method

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ABSTRACT

Purpose: The article presents the outline of methods and range of microstructural assessment of ceramic coatings, using the example of thermal barrier coatings. The major structural parameters describing the quality of the barrier layers have been characterised as well as the problems related to the correct metallographic specimen preparation and the methodology of their assessment.
Design/methodology/approach: A procedure of porosity assessment, employing quantative metallographic principles and automatic image analysis has been propounded, together with types of quantitative parameters and methods of their application.
Findings: It was found that the application of the quantitative metallographic methods combined with automatic image analysis can form an effective tool of both quantitative and qualitative assessment such parameters of structural ceramic layers as porosity.
Research limitations/implications: This type of assessment enables obtaining more than just the absolute value of the porosity of the given area: it provides the means for determining a number of other quantitative parameters, e.g. the surface area of the pores, their elongation and shape together with the whole statistical analysis.
Practical implications: The application of scanning microscopy, especially observation techniques such as BSE, BSETOPO or BSE3D enables a precise differentiation of the areas constituting pores from other artefacts.
Originality/value: Description of procedure of porosity assessments in APS TBC system by use of the quantitative metallographic methods combined with automatic image analysis, and possibility of applications special techniques of scanning microscopy.
Keywords: Thin & thick coatings; TBC; Degradation; Oxidation

1. Introduction

During the last decade, research efforts were devoted to the development and manufacturing of ceramic TBCs on turbine parts because the traditional turbine materials have reached the limits of their temperature capabilities. Thermal barrier coatings (TBCs) are used to sustain the highest temperature at the surface in high temperature superalloy substrates. TBCs have been widery used in hot-section metal components in gas turbines either to increase the inlet temperature with a consequent improvement of the efficiency or to reduce the requirements for the cooling air [1–10]. The typical TBC used in gas turbines consists of a bond coat produced by the vacuum or low pressure plasma-sprayed MCrAlY (M = Ni, Co) and a top coat of yttria partially stabilized zirconia made by the atmospheric plasma spraying or electron beam-physical vapor deposition (EB-PVD) [11,12].

The porosity of TBC coatings is one of their cardinal properties. On one hand, it can be seen as a drawback as it opens ways of easy penetration for aggressive corrosion factors such as air or combustion gases. On the other hand, however, the presence of pores enables the compensation of the stress in the coating resulting from exploitation and temperature changes. The acurrate porosity assessment is a complex problem, difficult from the technical point of view due to both the preparation of the metalographic specimens preparation and the interpretation of the results. The major problem in the porosity assessment of thermal barriers (and other thermally sprayed coatings) is the proper preparation of metalographic specimens. Taking into account the fact that these coatings are 0,005 up to 1,5 mm thick, as well as their notable brittleness and the significant differences in the hardness of individual phases, one has to admit there is a serious hazard of "faking" the real metalographic structure and of artefacts, which, consequently, leads to inaccurate results.

The necessity of preparing artefact-free specimens according to the given rules is absolutely obvious. The causes of artefacts and the methods of prevention of their occurence are thoroughly described in monographs and handbooks [13-16]. The metalographic examination of thermally-sprayed coatings constitute a principal source of information providing the means for qualitative assessment of obtained ceramic layers.

The metallographic preparation techniques of thermal barriers in various laboratories are frequently diversified, which leads to substantial differences in the results. Buehler Ltd method is one of the utilised methods. Another one is based on the information included in Structure, Struers e-Journal of Materialography 2/2004.

As has already been mentioned in the introduction, each laboratory has, as a rule, its own procedures of metalographic specimens preparation for the samples containing thermal barriers. They may also be based on the suggestions presented above.

2. Experiments, methodology and materials

The procedure of the quantitative porosity assessment in the bond coat and in the ceramic layer employing quantitative materialography and image analysis has been shown schematically as follow:

The quantitative porosity assessment, appplying quatitative metalography and image analysis

- The sampling strategy: defining the places of metalographic specimen taking and the amount of specimens ensuring repeatable and reproducible test results,
- Image acquisition of the surface of the entire tested material (obtaining the image of 32 measurement fields using Olympus GX71 optical microscope; magnification 200x; the field of view of individual image 625 x 470 μm = 0,39 mm^2; the analysis area 20 mm x 470 μm),
- Pore image detection (image calibration, determination of the upper and the lower limit of the pore detection):
  - The porosity measurement on every measurement field (the analysis of quantity, size, shape and placement of the pores
  - the number of pores Aa pores – the surface proportion of pores [%],
  - the size of pores A – the average surface area of the plane section of the pores [μm^2], P – the average circumference of the plane section of the pores,
  - the shape of the pores: a non-measurement size coefficient, a non-measurement elongation coefficient,
  - the assessment of distribution diversification: the pore proportion variability coefficient on the individual fields of measurement.
- The analysis of pore placement diversification,
  - obtaining an image consisting of individual fields of view using Satge Manager module in AnalySIS programme,
  - the measurement of the proportional pore content in each mesh of the grid,
  - creation of pore surface proportion distribution in the entire metalographical specimen on the basis of the matrix of results using Surfer programme.
- Analysis of results.

The sample chosen for the porosity assessment was prepared by cutting and grinding with abrasive paper (gradation 200, 400, 600), followed by polishing on diament pastes according to the procedure recommended by Struers company for ceramic coatings. The images were registered in optic microscope Olympus GX-71, in the bright field, magnified 300x. The image acquisition of the structure of the studied layer was done with analySIS®. programme. Thanks to its „Stage Manager" module, managing the motorized stage of the microscope and its „MIA" („Multiple Image Alignment") module designed to perform the image editing task a picture of the coating sized 3102 x 934,3 μm (13601x4096x8 pixels) was obtained, consisting of 40 (4 lines, 10 columns) images of individual fields of view. The complex image, containing the major part of the studied coating, has saved the resolving power characteristic of the individual field of view. It was confirmed that the thickness of the investigated coating equaled 700 μm, consisting of 600 μm of the layer and 100μm of the bond coat. A measurement grid was placed on the image of the registered surface, dividing it into equal fields. This enabled thorough investigation of every fragment of the layer without the risk of re-analyzing the same area or omitting any fragment. The size of the mesh (150x100 μm) had been adjusted so as to perform the porosity assessment separately for the bond coat (whose average thickness roughly equaled 100 μm) and the ceramic layer. The measurements were taken on 25 bond coat fields and on 150 barrier layer analysis fields. To present the differentiation of the surface pore content on the cross-section graphically, the so-called "structural maps" were created, presenting the value of surface pore content in every mesh of the grid on the cross-section (Fig. 1). These were created with the help of Surfer®, a programme for topographic map construction.

When the grid had been placed on the layer image with AnalySIS programme, the morphological parameters were measured in individual areas. The porosity of the bond coat and the barrier layer were characterized with:

- the percentage of pore surface content AA on the surface of the whole sample [%],
- the percentage of pore surface content AA on the surface of the worst field of view [%],
- the pore surface content variability coefficient – the quotient of standard deviation of the pore surface content of the individual fields of view and the average content value expressed in percentage; the higher the value of this coefficient, the higher the diversity in the pore distribution.
- the number of pores contained in a single metalographic specimen NA [mm^{-2}],
- the surface pore plane section areas A, the circumference of the pores P, being the measurements of the pore size;
- the pore elongation indicator δ = Fy/Fx and non-measurement pore shape coefficient $\xi = 4\pi$ A/P 2 (it takes the value 1 for the circle),where: Fx, Fy – Ferret's diameters.

3. Results

The application of automatic image analysis enables the statistical treatment of the data as well, e.g. obtaining frequency distribution and the surface content of the pores as a function of the surface area, circumference, elongation and shape coefficient of their plane section.

The examples of distributions for ceramic layer and the bond coat are presented in Fig. 2.

For the sake of the repeatability, another, analogous series of porosity assessment has been conducted independently by a different person using Met-Ilo v. 9.04 quantitative analysis programme [17]. The obtained results were very similar, which indicates that the propounded procedure is correct.

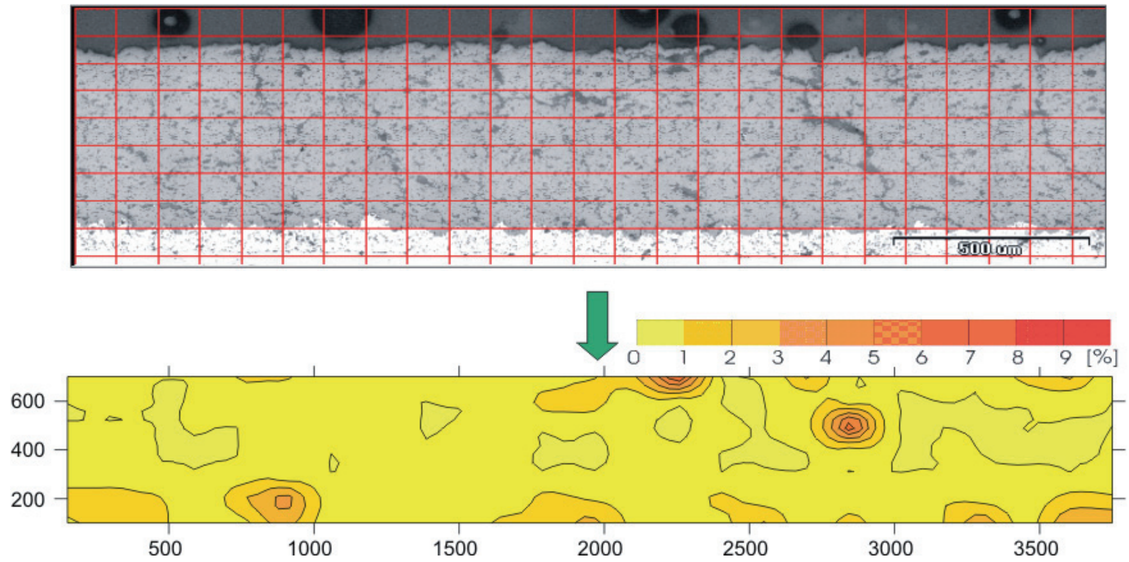

Fig. 1. The measurement grid placed on the coating image and the maps of the pore surface content percentage on the surface of the whole cross-section of the layer

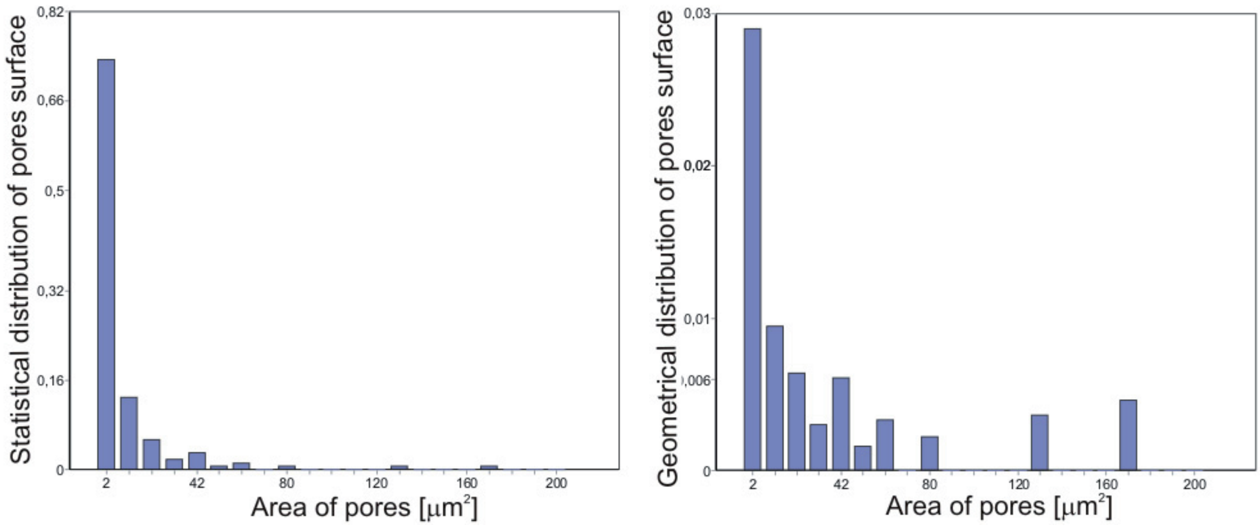

Fig. 2. The frequency distribution and the surface content of the pores distribution as a function of the surface area of their plane section for the ceramic layer

4. Conclusions

- For the sake of the accurate metallographic procedures realisation, a description of the factors influencing the final effect, i.e. a TBC ceramic layer preparation staff training; the elaboration and choice of correct preparation processes, taking into account the equipment and the conditions necessary for the observations; and, eventually, the determination of the structural factors constituting the basis for the qualitative assessment of the coatings.
- The application of the quantitative metalography methods combined with automatic image analysis can form n effective tool of both quantitative and qualitative assessment such parameters of structural ceramic layers as porosity.
- This type of assessment enables obtaining more than just the absolute value of the porosity of the given area: it provides the means for determining a number of other quantitative parameters, e.g. the surface area of the pores, their elongation and shape together with the whole statistical analysis.
- The structural maps showing the distribution of the proportion of the pore surface to the whole cross-section of the sample constitute a clear graphic interpretation of the study results. As well as this, they make it possible for the scientist to define the areas with the highest pore content in the sample.
- The application of scanning microscopy, especially observation techniques such as BSE, BSETOPO or BSE3D enables a precise differentiation of the areas constituting pores from other artefacts.

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