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Criteria of microstructural assessment of the conventional and new TBC layers

G. Moskal*
Department of Materials Science, The Silesian University of Technology, ul. Krasińskiego 8, 40-019 Katowice, Poland
* Corresponding author: E-mail address: grzegorz.moskal@polsl.pl

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ABSTRACT

Purpose: The purpose of this paper is to present principles and criteria of microstructural assessment of thermal barrier coatings (TBCs). The conventional layers are described in expert papers, whereas there is no literature on new type of thermal barrier coatings on the basis of $RE_2Zr_2O_7$ compounds

Design/methodology/approach: The study was carried out on the TBC layers, sprayed by using the conventional powders on the basis of yttria stabilized zirconium (YSZ) oxides and by using the powders of pyrochlore structure. Industrial powder type $Gd_2Zr_2O_7$ was sprayed as a representative of a new group of materials to be sprayed with the APS method.

Findings: The carried out analysis allowed to compare guidelines and criteria, which were used to assess the conventional TBC layer and their reference to barrier layers of a new type. The carried out study proved that heretofore used criteria of assessment for the TBC layers by using the conventional powders are sufficient to assess layers of a new type.

Research limitations/implications: The carried out study suggests necessity to verify the received results in the case of the TBC layers, sprayed by using the powders of pyrochlore structures of other types.

Practical implications: The received results show the possibility of using the heretofore criteria of microstructural assessment of the TBC layers as sufficient to get information on quality of the TBC layers of a new type.

Originality/value: The information concerning basic principles in assessment of microstructural layers of a new type, which are sprayed with the APS method on high temperature creep resisting alloys, is an original value presented in this paper.

Keywords: Metallic alloys; Thin & thick coatings; TBC; Microstructure assessment

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MATERIALS

1. Characteristics of basic assessment criteria of the TBC layers

Thermal barrier coatings (TBCs) are used to sustain the highest temperature on the surface of high temperature superalloy substrates. TBCs have been widely used in hot-section metal components in gas turbines either to increase the inlet temperature with a consequent improvement to the efficiency or to reduce the requirements for the cooling air. Ni-based superalloys have usually been used with thermal barrier coating (TBC) for vanes and blades in gas turbines and jet engines. Generally, the TBC applied in gas turbines is made up of two components: a bond coat created by the vacuum or low pressure plasma-sprayed MCrAlY (M = Ni, Co) and a top coat of yttria and partially stabilized zirconium produced by the atmospheric plasma spraying or electron beam-physical vapour deposition (EB-PVD) [1-7]. A typical superalloy/TBC system consists of plasma-sprayed zirconium-yttrium ceramic layer with a nickel-chromium-aluminium-yttrium bond coat on a substrate made of nickel-based superalloy. These superalloy/TBC systems can be applied in both aerospace and land-based gas turbine engines. In automotive use, the piston head for diesel engine is coated to enhance lifetime and performance as far as fuel demand reduction and power improvement are concerned. These coatings, nevertheless, exhibit relatively short lifetime, which is a result of the applied material and thermal contrast between the coating and the base metal [8-12].

Metallographic study of the coatings sprayed thermally, including the thermal barrier coatings (TBCs), is the basic source of information, which enables to assess qualitatively the ceramic layers and interlayers. However, the metallographic procedures are very difficult to control and any incorrect preparation of samples may result in receiving the results that may be essentially different from a real state. In order to provide correct execution of metallographic procedures, it is necessary to define factors, which influence on quality of a final result. The following are included in those factors: [13-15]:

- training of workers on the required preparation procedure of ceramic layers of TBC type;
- working out and selection of the proper preparation procedure, considering equipment and conditions of observations;
- defining the structural factors as the basis for qualitative assessment of coatings.

As the TBCs are multilayer systems made up of a substrate, undercoat i.e. *„bondcoat"* and outer ceramic layer i.e. *„topcoat"*, different criteria will be taken for their qualitative assessment [16,17].

Introduction of new types of ceramic materials i.e. "pyrochlores" as the top coat is an additional challenge. From a constitutive point of view, not much is changed. The TBC layers of a new type are still made up of an outer ceramic layer and undercoat layer, and substrate material is not changed either. Nevertheless, verification of the heretofore used qualitative assessment criteria of barrier coatings is required.

The basic criterion in assessment of TBCs, irrespective of a type of sprayed ceramic powders, is thickness of individual zones of a layer system (Fig.1). However, it is to be noted that thickness of a ceramic layer and undercoat depends on application of a particular coating.

This parameter is usually assessed at relatively low magnification approx. 100x. If a coating (undercoat and a ceramic layer) is homogeneous in consideration of thickness and scatter of measured values from a mean value is not big (this quantity is defined for each type of coatings), then only a mean value of the received thickness should be given. However, in the case when differences are considerable (above the standard value), then a range of thicknesses or max and min values and dominating thickness of a layer should be given. The typical thickness of undercoat layer is approx. 100μm, and thickness of a ceramic layer is approx. 300μm, though in the case of a TTBC (*thick thermal barrier coating*) type layer can be approx. 2 to 3 mm.

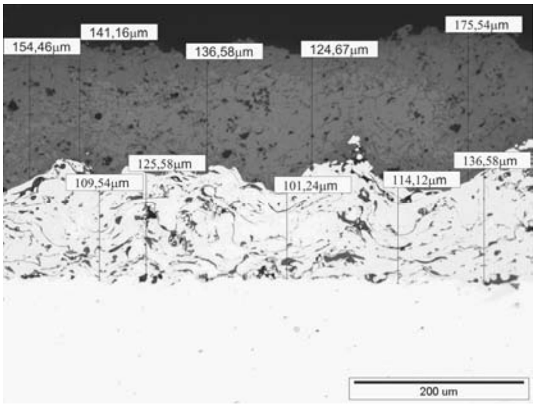

Fig. 1. TBC with $Gd_2Zr_2O_7$ top ceramic coat and NiCoCrAlY bond coat

In the case of ceramic layers of gradient nature, additionally thicknesses of individual sublayers are defined for a stroke gradient. Principles to assess thicknesses of those zones are similar to those described previously. Homogeneity of the received sublayer decides in this case as well. An example structure of a TBC type layer with a gradient of a chemical composition in the form of the YSZ and MCrAlY sublayers is presented in Fig. 2.

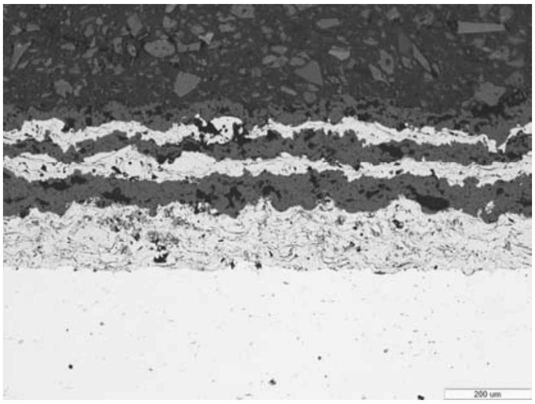

Fig. 2. Microstructure of TBCs with stroke gradient of chemical composition in a ceramic layer

However, at smooth change of the parameter, which the gradient relates to e.g. also to a chemical composition or porosity, the areas of predominant concentration of one of components can be the subject of assessment. An example microstructure of such a layer is shown in Fig. 3, where the outer thin YSZ layer, YSZ/MCrAlY transition zone and MCrAlY type undercoat can be seen.

The matter is relatively simple in the case of layers of considerably different colour, as in the above case, however, when ceramic layers of similar colour are sprayed, then a visual determination of thickness by light microscopy methods is not so simple. An example microstructure of the TBC with a gradient change of a ceramic component from 100% YSZ oxide at the NiCoCrAlY layer to 100% $Gd_2Zr_2O_7$ oxide is shown in Fig. 4.

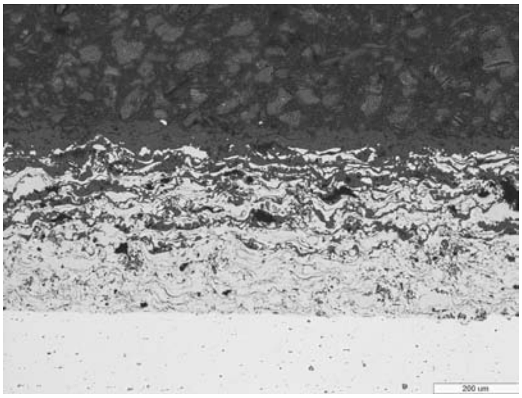

Fig. 3. Microstructure of TBCs with smooth gradient in chemical composition (YSZ + NiCoCrAlY).

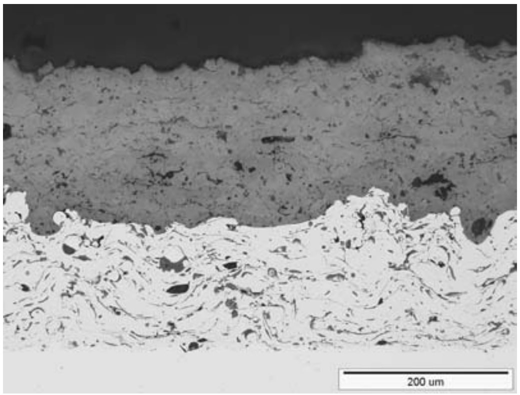

Fig. 4. Microstructure of TBCs with smooth gradient in chemical composition in the ceramic layer (YSZ – $Gd_2Zr_2O_7$)

Then the use of the BSE scanning microscopy technique or mapping is possible, which makes it possible to determine univocally layout of chemical elements, in this case zirconium and gadolinium. An example microstructure of the layer received with this method is shown in Fig. 5.

The next complicating element, which seems to be simple, i.e. measurement of layer thickness, is connected with application of gradient or modified (which can also be gradient ones) layers of MCrAlY type and diffusive layers, whose application in the case of the new TBC types is obvious. However, in this case depth of „penetration" by a modifying component and/or aluminium content as a function of distance from surface is definitely more important criterion – what practically decides of phase composition of an undercoat layer. An example microstructure of an undercoat layer of MCrAlY type, additionally subjected to diffusion aluminizing (aluminium modified adhesive layer with smooth gradient in chemical composition) is shown in Fig. 6.

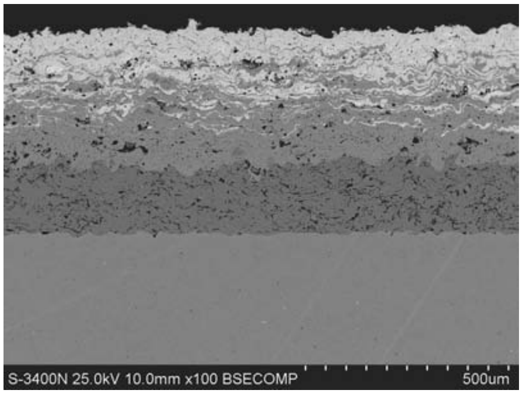

Fig. 5. Microstructure of TBCs with smooth gradient in chemical composition in the ceramic layer (YSZ–$Gd_2Zr_2O_7$) –SEM-BSE image

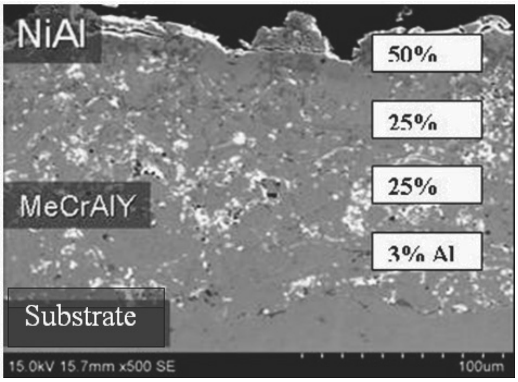

Fig. 6. Microstructure of NiCoCrAlY undercoat layer after gas aluminizing process and results of microanalysis of chemical composition

The visible changes in content of aluminium determine a changeable phase composition of an undercoat from the NiAl phase to the Ni_3Al-NiAl mixture. In the case of using an additional thermal treatment for the layers of MCrAlY type or for diffusive layers, an additional zone of carbides rich in alloy components may appear and this zone also plays an important role in ensuring high temperature creep resistance of undercoat layer, and that has direct influence onto chemical composition of the TGO zone and generally on stability of the whole barrier layer (Fig. 7).

Definition of geometric parameters of TBCs is only the first step to determine quality of the layers sprayed with the APS method. The next problems concern an area of a microstructural analysis. However, the parameters and criteria of assessment are different for the undercoat layer of MCrAlY type and different ones for an outer ceramic layer.

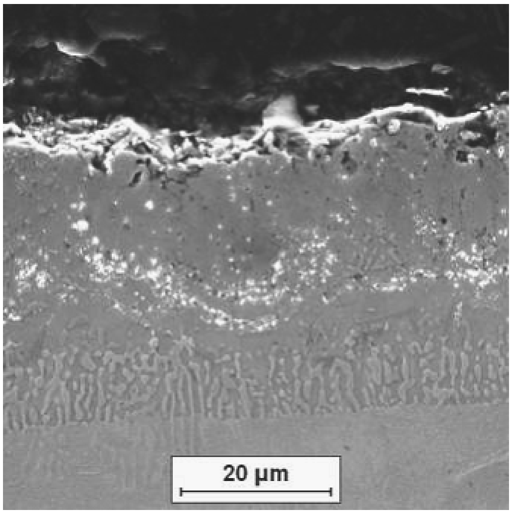

Fig. 7. Microstructure of the NiCoCrAlY undercoat layer after aluminizing process with a diffusive zone

For interlayers, which are usually the NiCoCrAlY or NiCrAlY powders sprayed with the VPS method, the requirements are connected with the following assessment criteria:

- Quality of joint between a metal substrate and interlayer, where the main purpose is to determine presence of contamination on the tested surface of distribution. A type of occurring contamination may have a form of oxides of substrate material, very fine particles of abradants (Fig. 8), residues of covering after previous cleaning operations. On this area there should not be any oxides coming from a covering material. The assessment for presence of distribution areas (delamination) of interlayers into the substrate (e.g. as a result of action of mechanical or thermal stresses) or no coherence between interlayers and substrate as a result of missing conditions to get adhesive joint, what is usually a result of insufficient roughness of substrate surface. (Fig. 9) and also morphology of distribution area in the form of „straight lines", should also be carried out. Finding any distribution areas (separation) of an interlayer into the substrate results in rejection of such a coat.

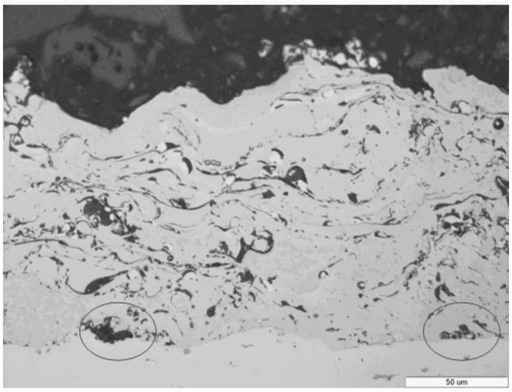

Fig. 8. Microstructure of the NiCoCrAlY undercoat layer with visible oxides on the distribution area

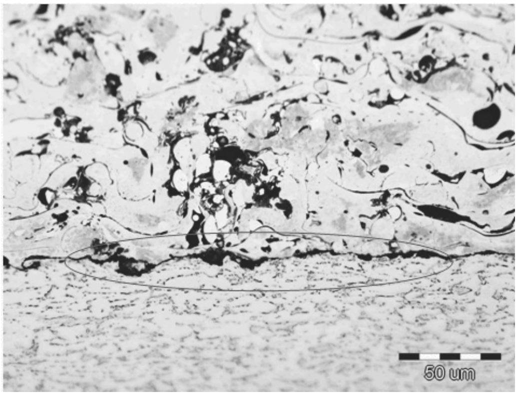

Fig. 9. Microstructure of the NiCoCrAlY undercoat layer with visible lack of coherence between a substrate and layer

- Presence of cracks - in the case of this assessment it is important to distinguish cracks which are a result of mechanical damages and those ones, whose source is in a structure itself. The cracks, which propagate along or are parallel to the substrate surface, are usually cracks of mechanical origin. And the cracks, which are perpendicular to the metal surface, are sometimes a result of substrate deflection or thermal stresses. Micro-cracks and shrinkage cracks are typical cracks connected with the interlayer microstructure. Presence of cracks in the interlayer results in rejection of such an interlayer.
- Resence of oxides - observation for presence of oxides with needle or globular morphology should be carried out. A location of their occurrence should also be determined. The assessment criteria depend on the layer type and spraying method. The assessment is visual on the basis of the guidelines included in specifications for a particular type of coatings. A typical appearance of oxides in the form of dark bands can be seen in Fig. 8.
- Porosity – this parameter is usually determined at magnification of 100 up to 500x, depending on the needs. A value of porosity is estimated visually, by using a line grid or by using a comparative method with the data included in specifications. One should also draw attention to all pores of big sizes. Conditions of acceptance for porosity are also presented in the specifications for a particular type of a substrate layer and can be e.g. up to approx. 15%. The view of pores received with the SEM TOPO method is shown in Fig. 10.
- Globular precipitations – are characterized as not melted particles of powder (Fig. 10). Usually they occur together with oxides and pores on their edges. The study of those particles is carried out, at magnification of approx. 500×, by estimating a percentage concentration of globular particles in a coating. Conditions of coating acceptance for presence of globular particles are presented in the specifications, but a diameter of a particle is a decisive criterion. For large globular precipitations, rejection of a coating may occur in the extreme case.
- Integrity of interlayers – is analysed, at magnification of approx. 100x, to determine total quality of a coating. In this case, one should pay attention to areas of loose contact between a substrate and coating, which is most often connected with the presence of globular precipitation and

pores, occurring separately or together. A homogeneous interlayer is required and it should not show the effects described above.

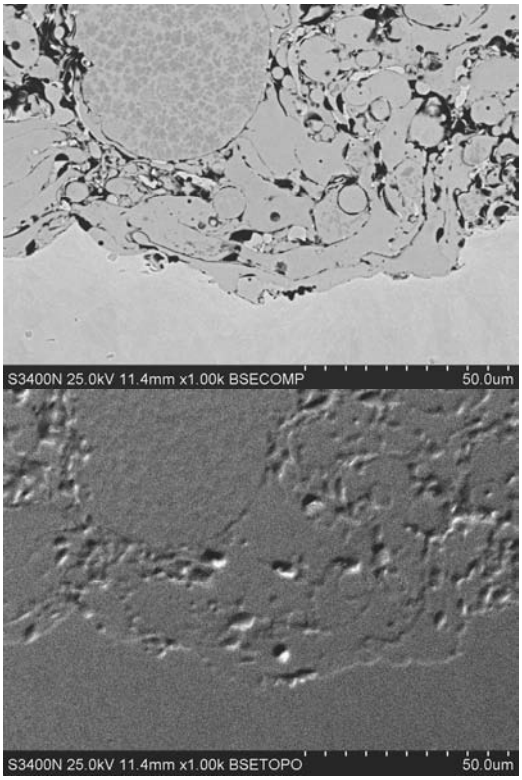

Fig. 10. Microstructure of the NiCoCrAlY undercoat layer with visible globular particles and porosity

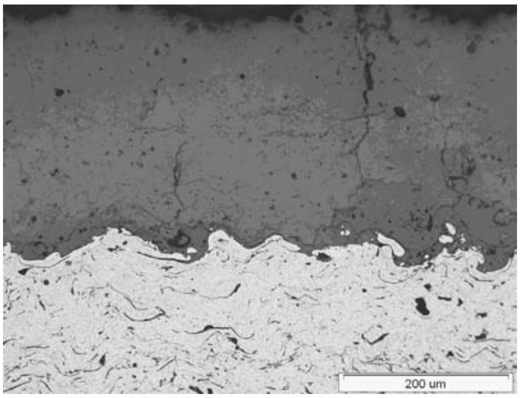

Fig. 11. Microstructure of the NiCoCrAlY undercoat layer with a ceramic layer and acceptable state of roughness

- Roughness of an interlayer surface – acceptance conditions involve comparison with the requirements included in the specifications for a particular type of a layer and if a state of an interlayer surface is in the predominant range close to a criterion state, then this interlayer is acceptable. In the case of sprayed layers under the APS ceramic layers, a relatively large development of area is required (Fig. 11), contrary to the ceramic layers of EB-PVD type.

Porosity, cracks and their segmentation are the most important criterion parameters in a scope of microstructural requirements for the outer ceramic layer. Those parameters are the most essential according to physical and chemical properties of the TBCs, in particular assessment of thermal conduction. In the data included in the guidelines for individual types of conventional powders used for spraying of the TBCs with the APS method, there is detailed information on type and distribution of porosity and cracks. The information is a reference point for the layers sprayed by using the powders of new type.

Basic questions for the assessment of artefacts, including mainly porosity and micro-cracks in ceramic layers, are connected with correct preparation of metallographic micro- sections. Just as in the case of suitability of microstructural assessment criteria of the conventional TBCs, it is necessary to verify metallographic procedures for preparation of micro-sections for the new types TBCs. One should remember of unusual sensitivity of ceramic layers for a way of cutting, mounting and temporal parameters of grinding and polishing processes, which may result in considerable differences in values received during measurements of porosity. Verification of those methods is presented in an item, where the standard procedures were used in order to obtain metallographic micro-sections of new types of TBCs - $Gd_2Zr_2O_7$ [18].

Microstructure of a ceramic layer is analysed at magnification of approx. 200x. Porosity, random orientated micro-cracks and segmentation of cracks are analysed. The porosity assessment of microstructure of the conventional TBCs (YSZ) and layers of new types ($Gd_2Zr_2O_7$) is shown in Fig. 12 and 13, for conventional and new types of powders respectively. Microstructure of a ceramic layer with macro- and micro-cracks for conventional coatings and new types of coatings is shown in Fig. 14.

- An acceptable level of porosity requires the predominant state of a ceramic layer to be similar to the layers presented in the specifications. Layout and sizes of pores has then basic meaning. It is required that the areas with homogeneously arranged fine pores and possible isolated areas with a bigger number of them are predominant. Single big pores are also acceptable. All structures not meeting requirements are rejected. As far as quantitative and qualitative assessments of porosity, by using a comparative method or an automatic analysis of an image is simple, characteristics of cracks, in particular its quantitative approach for vertical and horizontal cracks, is not so obvious [19].
- Metallic contaminations (Fig. 15) are subject to study at magnification of a ceramic layer of approx. 200x to reveal presence of metallic components. The acceptance condition for metallic contaminations is that their quantity should be lower than requirements presented in the specifications for a particular layer by using visual assessment.
- Globular precipitations are observed and assessed at magnification of 200x or more to show not melted ceramic particles. The assessment conditions are similar to those used for interlayer assessment.

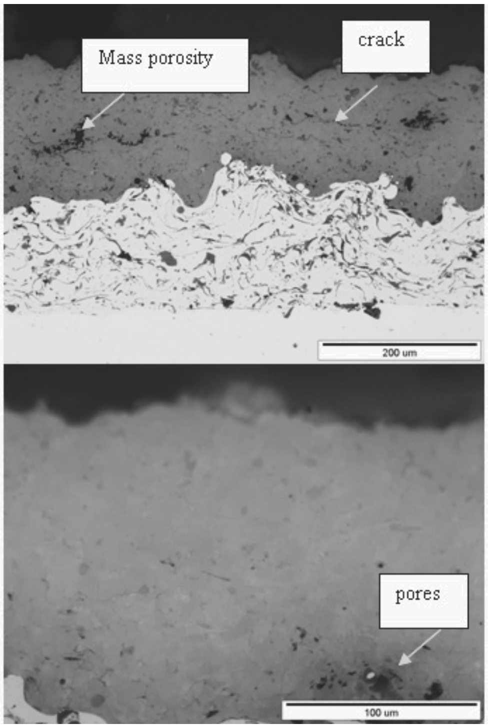

Fig. 13. Microstructure of the $Gd_2Zr_2O_7$ layer with visible fine pores and mass porosity and cracks

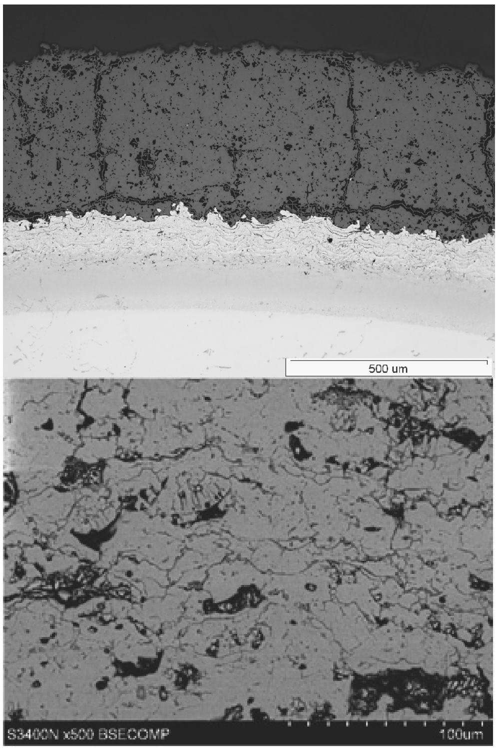

Fig. 14. Microstructure of the YSZ ceramic layer with micro-cracks and $Gd_2Zr_2O_7$ with micro-cracks

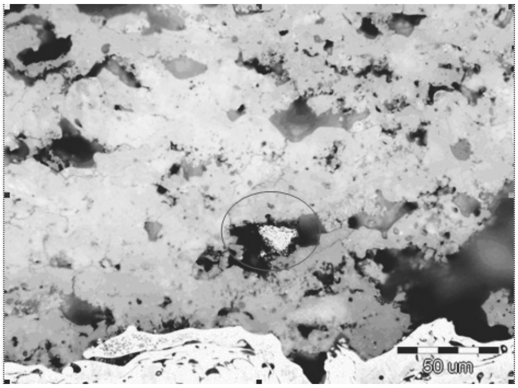

Fig. 15. Microstructure of the YSZ ceramic layer with visible fine metallic contaminations

2. Summary

- The analysis of results of the study, carried out on barrier coatings sprayed by using the APS method on a high temperature creep resisting alloy, showed that heretofore used criteria for microstructural assessment of the TBCs on the basis of the yttria stabilized zirconium (YSZ) oxides are also suitable for coating layers of new types on the basis of the $RE_2Zr_2O_7$ compounds.
- Microstructural assessment of the undercoat layer, where all heretofore used parameters and assessment criteria are sufficient to verify quality of the interlayers is an undisputed question.
- A question, which requires verification, is roughness of an interlayer for adhesiveness of new types of ceramic layers.
- Assessment parameters of new types of outer ceramic layer should be the same as in the case of conventional thermal barriers on the basis of zirconium oxide.
- Porosity and layout and sizes of pores are the most important criterion of assessment. An automatic analysis of image should be used for this purpose, which gives a possibility of statistic data processing [20].
- The second essential assessment criterion is characteristics of coating architecture in a macro- and microscope scale, which together with quantitative description of porosity enables, among other things, to make a quantitative description of thermal diffusion process in the TBCs.

Acknowledgements

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