Archives of Materials Science and Engineering

Volume 39
Issue 1
September 2009
Pages 53-60

International Scientific Journal
published monthly by the
World Academy of Materials
and Manufacturing Engineering

Metallographic preparation of the conventional and new TBC layers

G. Moskal*, B. Witala, A. Rozmysłowska
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

Received 12..02.2009; published in revised form 01.09.2009

ABSTRACT

Purpose: Verification of up-to-now used metallographic preparation of the TBC coating thermal barriers and adaptation of them to layers of new types, based on new ceramic compounds, sprayed on conventional high temperature creep resisting alloys by the APS method, is a purpose of this paper. New types of used ceramic powders are so called pyrochlores of a general formula $RE_2Zr_2O_7$.

Design/methodology/approach: A scope of investigations comprised realization of a process of preparation of metallographic micro-sections, beginning from a cutting moment, through mounting, grinding and polishing. A standard method of preparation of micro-sections, typical for conventional layers was used and microstructural observation, from a point of view of presence of artefacts of mechanical origin was carried out.

Findings: The carried out analysis allowed to compare methods of preparation of micro-sections and principles of preparation, used to assess the conventional TBC layers and relation them to barrier layers of new types. The carried out investigations showed that up-to-now used methods and procedures for the TBC layers, got by usage of conventional powders, are sufficient for layers of new types.

Research limitations/implications: The carried out investigations suggest a necessity to verify the got results in a case of the TBC layers, sprayed by use of powders of pyrochlore structure of another type.

Practical implications: The got results show a possibility to use up-to-now metallographic procedures for the TBC layers of new types.

Originality/value: Information, concerning basic principles of microstructural assessment of layers of new types, sprayed by the APS method on high temperature creep resisting alloys, is an original value.

Keywords: Metallic alloys; Thin & thick coatings; TBC; Sample preparation

Reference to this paper should be given in the following way:

G. Moskal, B. Witala, A. Rozmysłowska, Metallographic preparation of the conventional and new TBC layers, Archives of Materials Science and Engineering 39/1 (2009) 53-60.

TECHNICAL PAPER

1. Characteristic of basic stages in preparation 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 yttrium and partially stabilized zirconia 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 the result of the applied material and thermal contrast between the coating and the base metal [8-12].

Metallographic investigations of coatings, thermally sprayed, including coating barrier layers type TBC (*thermal barrier coating*), are basic sources of several pieces of information, which enables to assess qualitatively the ceramic layers and interlayers. However metallographic procedures are very difficult to be controlled and in an effect of faulty preparation of samples, the got results can be essentially different from a real state. A necessity to prepare specimens without artefacts is obvious. Reasons of artefact origin and methods of prevention of these faults are described in details on monographs and handbooks [13-15]. In order to provide correct realization of metallographic procedures, it is necessary to define factors, which have influence on quality of a final effect. The following aspects are included to these factors:

- training of workers in a required scope of preparation procedure of ceramic layers type TBC;
- working out and selection of proper preparation procedure, considering equipment and conditions of observations;
- defining the structural factors, which are grounds for quality assessment of coverings.

Techniques of metallographic thermal barriers are, unfortunately, different in many cases in various laboratories. In consequences, it leads to meaning differences in results. Independently of a kind of a used outer ceramic layer, preparation of metallographic micro-sections consists of the following stages [16-19]:

Cutting

It is the first of operations, which should be realized in order to prepare samples to further investigations. Usually, samples are cut out from a bigger element, however this place should be representative and this aspect is essential. A method, depending on simultaneous spraying the „exemplary" sample e.g. flat one of small sizes, what limits a number of cuttings or at very small sizes of it, such samples are excluded at all. Time and costs limit this way.

Generating of numerous defects in a layer, mainly in a form of cracks, is an essential fault of this cutting process. It can be avoided by cutting out a sample in such a way that a side, in a state so called „as sprayed", is a subject of further preparation. Otherwise, it is necessary to remove defects in a ceramic layer by use of rough and final grinding. Correct grinding will enable to discover not defected areas. An exemplary structure of the TBC layer with visible defects, as a result of a cutting process is shown on Fig. 1.

There are several kinds of devices, used to cut elements with ceramic coverings i. e. a manual abrasive saw, automatic abrasive-cutting machines , low-rotary diamond precise saws or bigger high-rotary precise cut-off machines. To get a cutting surface in a highly smooth and flat form, free of overheating and showing minimal deformation is an ideal effect. In order to get results of cutting, which are the most close to this state, it is necessary to consider influence of individual parameters of a cutting process.

Thickness of disk is the first of them. Usually, disks of the least possible thickness (< 0.1 in) should be used, because it provides a minimal contact surface with surface of a sample, what in consequence reduces overheating and a degree of surface deformation. Guidelines, issued by the Company Buehler [3] suggest applying a precise cut-off machine with a diamond disk or with ultra thin disk made of aluminium oxide.

To locate a sample, which should be fixed and positioned in such a way that defects of a coating are minimized, is the second factor. To locate a sample in such way that it has got least possible cutting surface, is the most advantageous location whereas one should consider that a ceramic layer at this location is charged with compressive stresses during cutting, what is one of the most important requirements because it prevents a delamination process of a ceramic layer. In a case of curve samples or of changeable shape, covering should be located in such a way that a cutting process begins from convex surface. In each of these cases, a distribution area between substrate – layer is required to be perpendicular to a cutting direction, what is favourable in pressing down a layer to substrate.

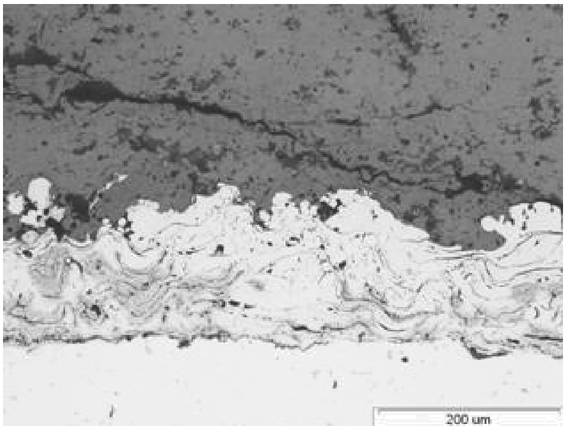


Fig. 1. Microstructure of the TBC layer with visible horizontal cracks above interlayers

The matter is more complicated in a case of cutting of elements or degraded samples with visible macro-scope cracks and delamination effects (Fig. 2). Then, cutting of such samples can cause residues of a ceramic layer, which are to be removed off and it can make the planned microstructural investigations be difficult.

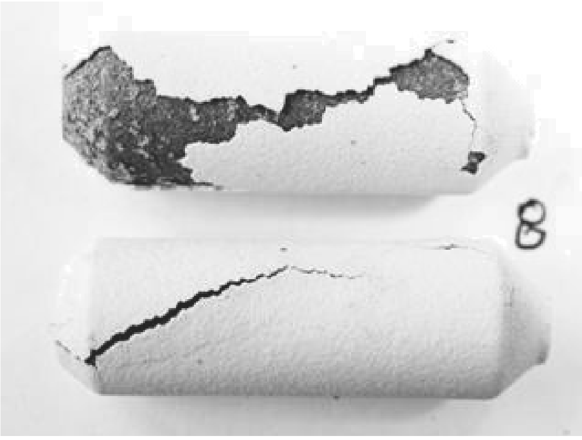


Fig. 2. Microstructure of layers type TBC of stroke gradient of chemical composition in a ceramic layer

Preliminary mounting in cold of the whole sample in a form (e.g. PVC pipe of suitable length and diameter) is an effective solution, what enables to protect surface during cutting process (Fig. 3) and relatively small defects of a layer microstructure (Fig. 4).

Cooling of a sample during a cutting process is the third factor, what enables to compensate emitting heat. In consequence, inclination to microstructural changes is reduced, scale is dropped-off and cracks are generated.

Selection of a cutting method is the next question. Manual cutting is not a repeatable method and it depends very much on operator's experience. Also automatic abrasive cutting, at constant charge, does not warrant getting a high quality of surface. Automatic abrasive cutting, with a constant feed rotational speed (with no charge) is the most effective method from a point of view of quality of got micro-sections.

Fig. 3. Microscope view of samples with barriers type TBC, after having been tested by cyclic oxidation and preliminary mounted in cold

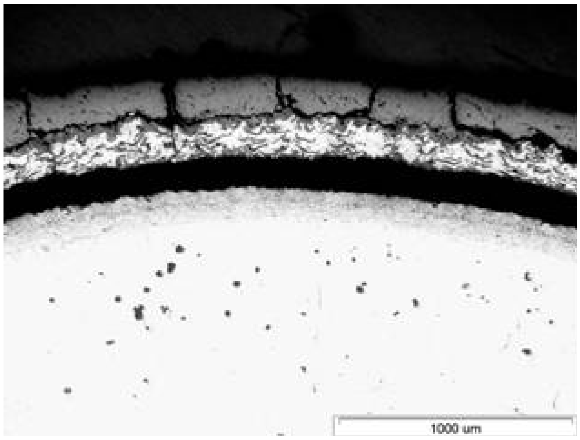


Fig. 4. View of the LM microstructure of the TBC sample after tests of cyclic oxidation resistance and preliminary mounting

In abrasive methods, disks comprise aluminium oxide or silicon carbide with additives of phenol resins. Cutting of hard materials requires using disks, relatively soft. Use of methods of precise cutting is especially an effective method to get a good quality of surface. In low-rotary machines, diamond disks or CBN are used.

Devices of such types can entirely be automatized with full control of force, charge and rotational speed of cutting. They enable to cut fast and repeatable and quality of got surface eliminates necessity to grind roughly. In more complicated cases i. e. when a layer type TBC is on both sides of an element or on round inner surface, it is very difficult to provide conditions pursuant to guidelines i.e. e.g. a state of compressive stresses. To apply cutting by means of water nozzles at pressure approx. 380MPa with added carnet abradants, is recommended. Electric spark cutting is the second method.

Rotational speed and cutting force are the last factors, which should be considered in a case of cutting of samples with the TBC layers. This parameter is especially important at manual cutting, while an operator, controlling pressure and rotational speed, is able to get assumed cutting quality. In a case of too intensive process, local overheating of surface and dropping off of a layer can occur.

Guidelines, issued by the Buehler Company [15] suggest using, in this case the following directions. Samples with deposited TBC coatings should be cut perpendicularly to their axes by use of a precise diamond cut-off machine with a diamond disk or ultra thin disk of aluminium oxide. These samples should be fixed and positioned in such a way that defects of coating are minimized. In a case of cutting of porous coating, to use protection for the coating in a form of vacuum impregnation with epoxy resin, is recommended.

The following parameters are recommended:

- Saw - ISOMET®2000
- Disk – diamond 15HC or of aluminium oxide (thin)
- Rotational speed – 2500 rpm or 4000 rpm
- Charge 600 gram or 500 gram
- Lubricant – ISOCUT®Plus (dissolved in water).

Similar guidelines are introduced by the Struers Company [20]. Exemplary photos of the TBC layer, conventional and of a new type, with a ceramic surface type $Gd_2Zr_2O_7$, are shown adequately on Fig. 5 a and b. A sample, after having been cut off and a metallographic micro-section itself are visible on this photo. Effects of mechanical cracks, typical for incorrect process of cutting, as on Fig. 1, are not observed on a micro-section. The same, on a sample before mounting, „edge” effects are not observed, which could be related to cutting of a ceramic layer.

Mounting

Mounting has to protect covering against outside factors and protect edges in a case of grinding and polishing. There are mentioned two types of mounting: mounting in hot (pressure mounting) and mounting in cold.

Mounting in hot is realized by use of epoxy powders and/or phenol powders in temperature 150°C and pressure approx. 30MPa. Duration time of the process is approx. 10-15 minutes. These parameters are very attractive from a point of view of laboratory needs. However, a method with application of higher temperature and pressure is not the best solution for ceramic layers, which are thermally sprayed. It occurs usually in a case of porous layers, where penetration through resins of a ceramic layer is too small, relating to epoxy resins of less viscosity, which are used in mounting in cold. In effect, grinded samples with a ceramic layer of less penetration degree through resin undergo easily cracking. Additionally, during mounting process, pressure and temperature generate cracks, what causes ceramic layers type TBC not to be suitable for mounting by this method. An exemplary conventional layer and of new type TBC with cracks, got as a result of mounting in hot are shown in Fig. 6 a and b.

a) b)

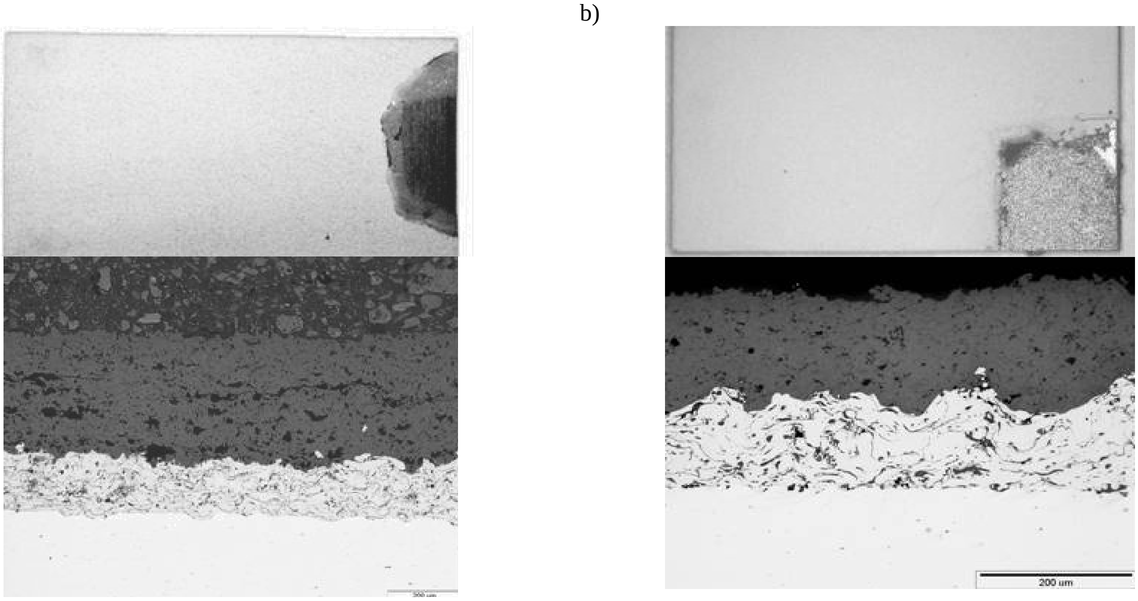


Fig. 5. View of microstructure of the TBC layer with an outer ceramic layer a) type YSZ, b) type $Gd_2Zr_2O_7$,after cutting pursuant to guidelines issued by the Buehler Company

a) b)

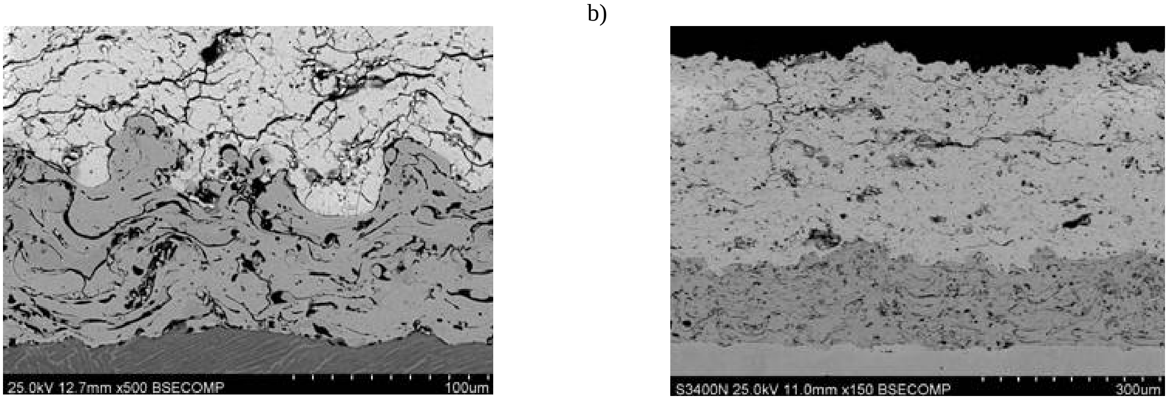


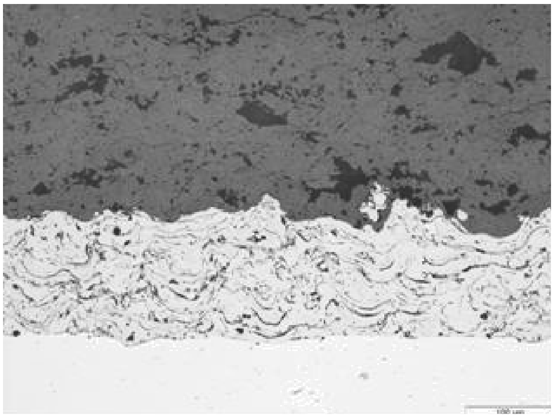
Fig. 6. View of microstructure of the LM sample after mounting in hot a) with TBC (YSZ), b) with TBC ($Gd_2Zr_2O_7$)

Mounting in cold means a notion of protection of a sample in mixture of epoxy and acrylic and in polyesters. The process is realized in temperature close to ambient temperature. Selection of compounds, possible to be used for coatings, which are sprayed thermally, is quite wide. Speed of congealing, hardness, viscosity and susceptibility to shrinkage are, in this case, basic criteria. Epoxy resins, which are characterized by optimal range of required properties.

Mounting in cold can be carried out in vacuum. In a moment of removing of air, liquid epoxy resin impregnates cracks and pores in a ceramic layer. Depth of penetration depends on viscosity of resins and is bigger in a case of slowly congealing resins.

A new type of resins of short time of congealing (below 2 hours in temperature 70-80°C) and of low viscosity, which appeared on market, what provides to get big depth of impregnation, is a good solution of this problem. Transparency of epoxy resins is an additional advantage, what enables precisely to detect artefacts and distinguish e.g. pores from oxides and delamination, by adding dyestuff or fluorescent substances.

a)



b)

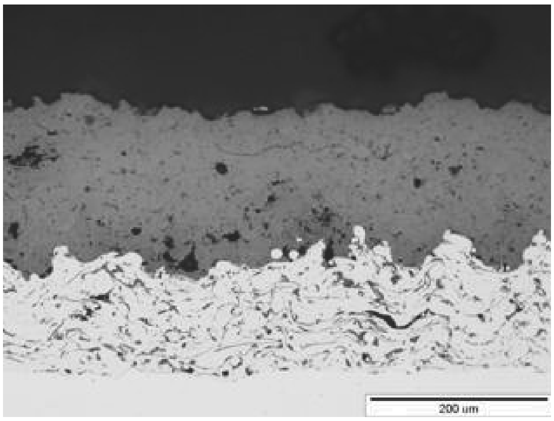


Fig. 7. View of microstructure of the LM sample: a) with TBC (YSZ) b) with TBC ($Gd_2Zr_2O_7$), after mounting pursuant to a procedure, issued by the Buehler Company

Guidelines, issued by the Buehler Company [17] suggest using, in this case, the following agents EPO-KWICK®, Epoxide or EPO-THIN®. Each of samples should be placed in a vacuum chamber of an impregnation system. Then, the whole is filled with resin and it remains in this state for approx. 5 – 10 minutes in order to impregnate porosity in an open coating.

An exemplary microstructure the conventional TBC layer and of a new one, after having applied a procedure issued by the Buehler Company, are show in Fig. 7 a and b.

Rough grinding

This stage has a basic meaning for final quality of got micro-sections. Then, removing the outside deformed and defected layer of material, what occurred as a result of cutting. It is approx. 0,006in in a case of cutting with thick disks. Usually, abrasive papers on base of SiC, Al_2O_3 and ZrO_2 are used. Carbide papers are used in a wide range of gradation from 60 to 1200, and oxide papers are used for rough grinding in gradation 60 to 120. In order to get satisfying quality of surface, grinding with papers of more and more gradation is used. No standardization in a case of SiC papers is a basic problem. It makes differences in quality of surface of treated samples with layers, what is an effect of a different method in production of these papers and in consequence a different orientation of abradant particles to substrate. It has a basic importance for mechanics of abrasion process. It appears with different abrasion rotation. In a case of wide areas of elements and large quantity of material to be removed e.g. to get micro-sections from a turbine vane, grinding with papers is inconvenient, so grinding with stones on a base of aluminium oxide is acceptable. Usually, this stage of preliminary preparation of surface is made on stone of gradation 60 to 80 at rotation approx. 1400 rpm. This tape of treatment can cause additional defects in a sprayed layer, so it is necessary to grind on coarse grained SiC papers.

Final grinding and polishing

In this stage of preparation, abrasive materials of very fine granularity (sub-micron size), remove only small quantity of material. This stage is not able to remove defects and deformations, which originated in sooner stages. However, there is no consensus, when grinding finishes and polishing begins. As far as rough grinding and final grinding is a multi-stage process, then polishing is generally one-stage operation, depending on use of cloth (felt) with addition of very fine particles of diamond (approx. 1μm). However, this method is long-lasting and in effect can lead to surface rounding and other artefacts, which are Essentials from a point of view of microstructural assessment (Fig. 8).

The Buehler Company proposes the following solutions. Usually, samples with thermal barriers undergo preliminary grinding with the SiC papers of gradation 120 or 180. Diamond disks 45μm (ULTRA+PREP™) can be also used. Correct selection of abrasive agent enables to avoid sensible defects of a thermal barrier. Then, ultrasonic washing should be applied. However, it is not commonly accepted. Washing time should be minimal in order to avoid defects. An exemplary microstructure of the TBC layers of a new type, got pursuant to these guidelines, is shown in Fig. 9.

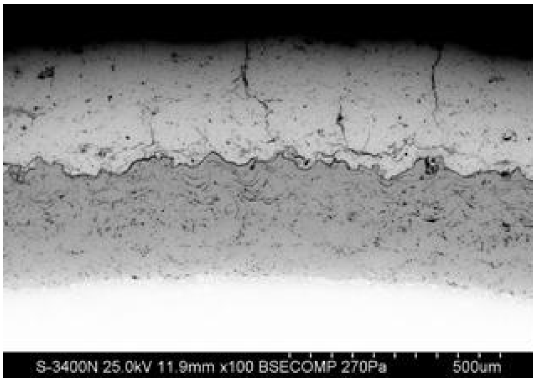


Fig. 8. View of the TBC structure after too long polishing. Use of SiC as a polishing material is minimized, and diamond paste of decreasing itself the gradation degrees is recommended. It is also multi-stage process

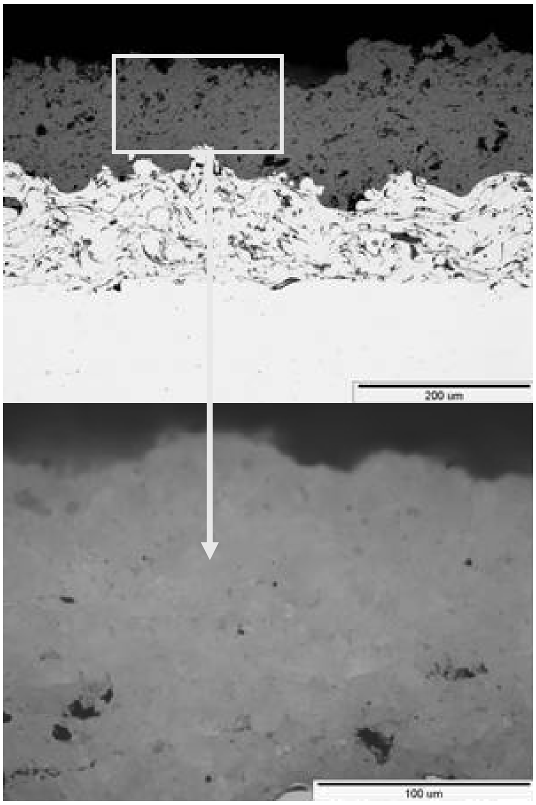


Fig. 9. View of microstructure of the TBC layer with outer ceramic layer type $Gd_2Zr_2O_7$ after final grinding, pursuant to guidelines issued by the Buehler Company

In item [16], polishing and final grinding are defined as a process of grinding with use of coarse-grained diamond pastel of gradation 6 to 15μm, considering together with grinding disks or hard polishing clothes. Grinding disks are made of rigid metal disks with a composite strap on surface, which is not an abrasive material. Diamond particles, during a grinding process, deposit themselves on composite substrate, and thanks to it a process of final grinding is realized.

A last stage of preparation of metallographic micro-sections of samples, covered with thermally sprayed layers, has considerable importance for quality of them and possibilities to assess artefacts. A polishing process has to remove any remaining defects on treated surface and other defects after sooner actions.

Polishing can be defined as actions on treated surface by means of fine grained diamond pastes of 1, 3 or 6 mm or sub-micron particles of abrasive material (SiO_2, Al_2O_3) in co-operation with polishing clothes. In modern polishing methods, more than one step is applied. A view of the TBC layers of a new type, after having finished the whole procedure, recommended by the Buehler and Struers Companies, are shown in Fig. 10 a and b.

2. Summary

- The carried out analysis of results of microstructural investigations on samples, which are covered with ceramic barrier layer type TBC showed that the applied metallographic preparation has a meaning influence on correct appearance of artefacts and structural elements, which are subjects of criterion assessment in quality assessment of the TBC layers.
- Investigations on microstructure of the TBC layers of new types (outer ceramic layer – $Gd_2Zr_2O_7$) showed that a new kind of a used material was also characterized by strong „sensitivity" on applied procedures and conventional layers type YSZ as well.
- Correct cutting out the samples from bigger elements with obeyed principles, presented in expert systems, has meaning importance, as in a case of conventional layers.
- From a point of view of quality of got micro-sections, it is essential to maintain parameters of a process, which are related to mounting of samples. Mounting in cold is recommended, as for conventional materials.
- Application of worked out procedures to prepare metallographic micro-sections for conventional layers type TBC, is sufficient also for the TBC layers of new types.

Acknowledgements

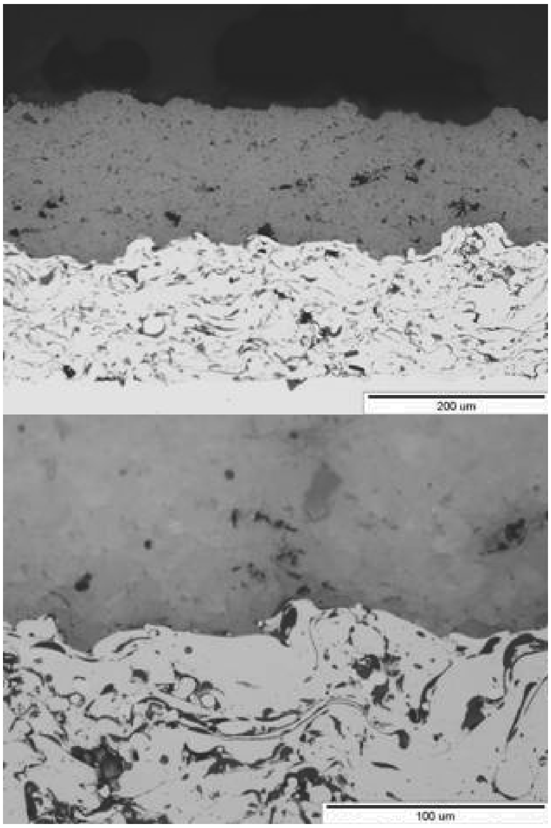
This work was supported by the Polish Ministry of Education and Science under the research project No PBZ-KBN-100/T08/2003.

References

[1] J.T. DeMasi-Marcin, D.K. Gupta, Protective coatings in the gas turbine engine, Surface and Coating Technology 68/69 (1994) 1-9.

[2] J. Wigren, L. Pejryd, Thermal barrier coatings-why, how, where and where to, thermal spray: meeting the challenges of the 21st century, in: C. Coddet (Ed.), Proceedings of the 15th International Thermal Spray Conference, ASM International, 1998, 1531–1542

a) b)



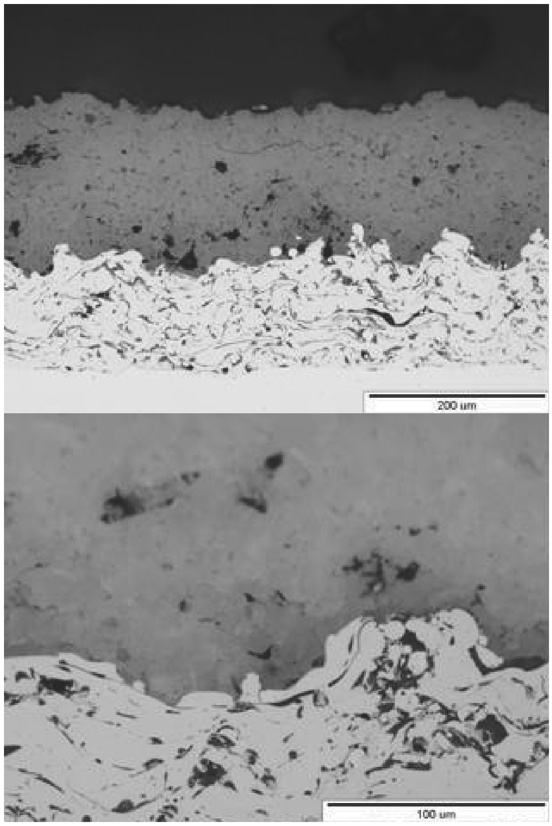


Fig. 10. View of microstructure of the TBC layer with outer ceramic layer, a) type $Gd_2Zr_2O_7$, b) layer type $Gd_2Zr_2O_7$, having been prepared pursuant to guidelines issued by the Buehler Company

[3] M. Konter, M. Thumann, Materials and manufacturing of advanced industrial gas turbine components, Journal of Materials Processing Technology 92-117 (2001) 386-390.

[4] D. Stover, C. Funke, Directions of the development of thermal barrier coatings in energy applications, Journal of Materials Processing Technology 92-93 (1999) 195-202.

[5] J.F. Li, H.L. Liao, C.X. Ding, C. Coddet, Optimizing the plasma spray process parameters of yttria stabilized zirconia coatings using a uniform design of experiments, Journal of Materials Processing Technology 160 (2005) 34–42.

[6] Ashok Kumar Ray, Characterization of bond coat in a thermal barrier coated superalloy used in combustor liners of aero engines, Materials Characterization 57 (2006) 199–209.

[7] W.A. Nelson and R.M. Orenstein, TBC experience in land-based gas turbines, Journal of Thermal Spray Technology 6 (1997) 176-180.

[8] Evans AG, Mumm DR, Hutchinson JW, Meier GH, Pettit FS., Mechanisms controlling the durability of thermal barrier coatings, Progress in Materials Science 46 (2001) 505–53.

[9] L. Swadźba, G. Moskal, B. Mendala, T. Gancarczyk, Characterization of APS TBC system during isothermal oxidation at 1100°C, Archives of Materials Science and Engineering 28/12 (2007) 757-764.

[10] G. Moskal, Effect of TBC on oxidation behaviour of γ-TiAl based alloy, Journal of Achievements in Materials and Manufacturing Engineering 22/2 (2007) 31-34.

[11] G. Moskal, L. Swadźba, T. Rzychoń, Measurement of residual stress in plasma-sprayed TBC with a gradient of porosity and chemical composition, Journal of Achievements in Materials and Manufacturing Engineering 21/2 (2007) 31-34.

[12] L. Swadźba, G. Moskal, B. Mendala, T. Gancarczyk, Characterization of air plasma sprayed TBC coating during isothermal oxidation at 1100ºC, Journal of Achievements in Materials and Manufacturing Engineering 21/2 (2007) 81-84.

[13] L. Bjerregaard, K. Geels, B. Ottesen, M. Rückert, Metalog Guide, Struers A/S, Rodovre, Denmark, III eds, Metallografic Guide, Richard Larsen A/S, Denmark, 2001 (www.struers.com; www. prospecta.pl).

[14] D. Cebula, J. Widerman, Metallographic Investigations – preparation and observation methods, Office Gamma, Warsaw, 1999.

[15] Buehler's Technical Information Guide & Preparation Methods, Buehler Ltd, Illinois, USA, (2000).

[16] J.P. Sauer, D.G. Puerta, G. Blann, Accepted practices of thermal spray technology, Journal of Thermal Spray Technology 14 (2005) 131.

[17] J.P. Sauer, D.G. Puerta, G. Blann, Accepted practices of thermal spray technology, Journal of Thermal Spray Technology 14 (2005) 450.
[18] J.P. Sauer, D.G. Puerta, G. Blann, Accepted practices of thermal spray technology, Journal of Thermal Spray Technology 15 (2006) 31.
[19] J.P. Sauer, D.G. Puerta, G. Blann, Accepted practices of thermal spray technology, Journal of Thermal Spray Technology 15 (2006) 174.
[20] K. Gels, The True Microstructure of Metals, Struers Journal of Metalography 35 (2000) 5-13.