FRACTURE TOUGHNESS OF RARE-EARTH STABILISED ZIRCONIA THERMAL BARRIER MATERIALS: EFFECT OF PHASE TRANSFORMATIONS

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Yttria-stabilized zirconia (YSZ) containing ~8 mol% YO₁.₅ with t’ phase is the most successful material for thermal barrier coatings (TBC’s). This is partly because of its higher fracture toughness compared to other materials. For instance, cubic zirconia with higher yttria content has lower thermal conductivity but also lower toughness than t’ YSZ. Similar effect is seen in rare-earth stabilized zirconia. The high toughness of t’ is believed to originate in ferroelasticity. However, t’ is prone to destabilization via cubic phase formation during thermal exposure, and possible formation of monoclinic phase during cooling. The question addressed in this work is: what happens to fracture toughness when t’ phase undergoes phase transformations? The compositions selected include the conventional YSZ and zirconia with rare-earth stabilizers (Yb, Gd and Nd). The total stabilizer content has been fixed at 8 mol% REO₁.₅. Dense compacts sintered at 1250°C were used for facilitating fracture toughness measurements and composition control. Phase transformations were studied by thermal exposure up to 192 h at 1250°C. Indentation fracture toughness was monitored concurrent to phase transformations. Phases were identified by X-ray diffraction (XRD) and Raman spectroscopy. The initial fracture toughness of t’ YSZ was 90 Jm⁻². The highest initial toughness of 117 Jm⁻² was seen in Gd-stabilized zirconia which also showed the finest crystallite size of 44 nm and highest c/a ratio of 1.014. During thermal exposure at 1250°C, the fracture toughness of YSZ decreased to 28 Jm⁻² in the first 96 h, and then increased to 71 Jm⁻² after 192 h. The initial degradation of fracture toughness is likely due to coarsening of the microstructure parallel to cubic phase precipitation. The subsequent increase in the toughness is likely due to the formation of monoclinic phase around the crack-tip. Raman spectroscopy has substantiated these observations. A co-doped composition containing Yb+Gd+Nd was resistant to phase transformations and its toughness steadily increased from 110 to 304 Jm⁻² during the same thermal exposure as YSZ. The results have been correlated with TBC stability and durability issues.
EFFECT OF MOISTURE ON THE COMPLIANCE OF THERMAL BARRIER COATINGS

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Mechanical compliance is one of the key characteristics of thermal barrier coatings (TBCs) which enables them to withstand high thermal stresses generated from thermal mismatch between coatings and its underneath materials. It has long been accepted that microstructural defects embedded in TBCs are responsible for their compliance. Our recent observations have revealed that the moisture present in the environment is capable of altering the coating compliance by introducing changes in physical properties of these defects. In this study we discuss a tool and a methodology using bilayer curvature measurements to quantify the coating compliance, and, next, the method is applied to observe the subtle changes in the coating compliance due to moisture variance.
IMPROVED OXIDATION RESISTANCE OF A NOVEL NICOCRALY COATING FABRICATED BY PLASMA-ACTIVATED EB-PVD

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MCrAlY (M=Ni, Co or Ni+Co) overlay coatings are commonly employed on superalloys or as bond coats (BCs) for zirconia based thermal barrier coating (TBC) systems. The protection offered by such coatings against high-temperature oxidation and hot corrosion, relies on the ability to produce and maintain a continuous and adherent thermally grown oxide (TGO) on its surface. In this study, a novel duplex NiCoCrAlY coating was fabricated by plasma activated EB-PVD (PA EB-PVD). Within the coating, beta-NiAl phase precipitated as small lamellae (less than 1 micrometer in thickness), predominantly oriented perpendicular to the coating surface. The high-density phase boundaries of gamma/beta provided the rapid diffusion route for the migration Al, thus promoting the formation of a dense alpha-alumina scale on the coating surface. The oxidation results at 1373 K show that the scaling rate of the novel coating is almost one order of magnitude lower than that of the conventional EB-PVD coating with similar composition.
STRESS INFLUENCE ON HIGH TEMPERATURE OXIDE SCALE GROWTH: EXPERIMENTAL INVESTIGATION ON THE AM1/NiAlPt/EBPVD YSZ SYSTEM

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Thermal barrier coating (TBC) systems are now widely used by OEM (Original Engine Maker) in the design of high pressure cooled turbine blades and vanes of turboreactors in order to achieve higher operating temperature. As a result, better engine efficiency, lower specific fuel consumption and lower NOx emissions are obtained. However, in service damage mechanisms of those coatings are still misunderstood and numerous processes may interact such as rumpling and/or phase transformation of the bond coat (BC), sintering of the top coat (TC)… Since a majority of the damage processes of those coatings concentrate in the vicinity of the thermally grown oxide (TGO) scale at the TC/BC interface, more or less complex lifing methodologies for TBC coating systems have been developed. These methods are always based on the computation of the TGO growth whose evolution is only considered as temperature dependent. However, little work has been done to evaluate precisely the effect of the mechanical loading on the oxide scale growth in these systems [1, 2].

In this study, low stress creep tests have been performed on the AM1/NiAlPt/EBPVD YSZ system in the 1000°C-1200°C temperature range and for durations as long as 700h. Doubly tapered specimens were used allowing the analysis of different stress states for a given creep condition. Oxide thickness evolutions have been characterized as a function of time and temperature for different stress levels using scanning electron microscopy and subsequent image analysis. These creep tests have been compared to pure isothermal oxidation tests on circular discs performed to establish the “mechanical stress free” oxide thickness evolution (i.e. without any applied stress). This presentation will discuss the results of these series of experiments in an aim to clearly identify the effect of the mechanical loading on the TGO growth, especially in the early stages of oxidation where the TGO growth stresses are sufficiently low compared to the applied stress.

Thermal barrier coatings (TBCs) serve for the thermal protection of hot section components in gas turbines to improve their life-time and thermal efficiency. With increasing operating temperature of advanced gas turbines, spallation failure of TBCs becomes more and more important, since loss of the TBC causes exposure of the metallic blade material to hot gases at temperatures far beyond the operation limit of advanced Ni-superalloys. Thus, the understanding of spallation failure and underlying damage mechanisms is a key issue for further TBC development and a requirement for the full integration of TBCs into component design.

The aim of the present study is to identify the lifetime and damage mechanisms of air plasma-sprayed TBCs during thermal cycling using microstructural and acoustic emission analysis. A TBC system consisting of air plasma sprayed partially stabilized zirconia ZrO2-7-8wt. %Y2O3 with NiCoCrAlY bond coat (BC) on CMSX-4 substrate was subjected to thermal cycling in the temperature range from 60 to 1050°C with different dwell times at maximal temperature. Additionally, the effect of the minimum cycling temperature on the lifetime was studied in tests with 2 hours high temperature dwell time.

The microcrack evolution observed suggests that the lifetime of APS TBSs is governed by the kinetics of crack formation, growth and linking of individual cracks. The degradation of the TBC system for a certain dwell time is affected by the concurrent contribution of bond coat oxidation and of crack growth related degradation processes driven by TGO growth and thermal mismatch stresses. Increasing the high temperature dwell time during thermal cycling led to increase of time-at-temperature life and to decrease of cycles to failure, indicating that the cyclic damage has more detrimental effect on the TBC lifetime under cycling with longer high temperature dwell times.

The in situ acoustic emission analysis during thermal cycling showed that delamination cracking mainly occurs during cooling in the low temperature part of the cycle, when the thermal-expansion mismatch stresses at the BC/TBC interface are maximal and cannot be compensated by plastic deformation of the BC (T< ductile-to-brittle transition temperature (DBTT)).

These observations are in good agreement with the thermal cycling lifetimes observed in experiments with different minimal temperature. Increasing of minimal temperature from 60°C to 350°C resulted in almost two times higher time-at-temperature lifetime of TBC. Finally, TBC lifetime after cycling in temperature range from 500 to 1050°C reached the same value as after isothermal exposure. Thus no influence of the cycling degradation processes on the TBC lifetime during thermal cycling above the DBTT was observed.
Segmentation cracks can enhance the thermal cycle life of the atmospheric plasma spray (APS) thermal barrier coatings (TBCs). The solid angular and hollow powders can prepare this modified coating easily, but evident branch cracks can form simultaneously which can supply a heat reflection interface and form the source to crack growth. The ceramic top layer material used in this investigation comprises two agglomerated nanocrystalline ZrO2-7%Y2O3 powders (P1 with high impurity content, P2 with low impurity content) with melt shell and high apparent density (about 2.0g/cm3). The common and segmented thermal barrier coatings (TBCs) whose thickness were about 0.30mm deposited by APS, and the microstructure and properties of these powders and coatings were analyzed in this work. The experimental results show that YSZ material P2 have better high temperature phase structure stability after long-term heat treatments at 1400°C. Minute quantity branch cracks occurred in the segmented coating from the P1 and P2, and this phenomenon was not similar to the segmented coating from solid angular and hollow powders reported in the literature, that resulted from the change of the spraying powder’s characteristic. The segmented coating had higher bond strength(>40MPa), furnace cycle life (1100°C,1708h) and little lower heat-insulating property, hot corrosion resistance than the common coating (about 15% porosity by micrograph, 1468h cycle life) from P2. And the results were related to that vertical cracks enhanced the strain tolerance and supplied the passage to the hot corrosion medium (sea water and kerosene’s impurity from the experimental environment). The common and segmented coating from P2 had long furnace cycle life than the segmented coating from P1(cycle life of 838h), and that show the decrease of impurity content in YSZ material would help to improve the coating's thermal cycle life.
THERMAL CONDUCTIVITIES OF ZIRCONIA-CERIA-YTTRIA SOLID SOLUTIONS

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Thermal conductivities of [(ZrO2)1-x(CeO2)x]0.92(Y2O3)0.08 (0 ≤ x ≤ 1) solid solutions were studied by a laser flash method. The incorporation of ZrO2 and CeO2 in the solid solutions decreases the thermal conductivity compared with their end members yttria stabilized zirconia and yttria doped ceria (YSZ and YDC). The lowest thermal conductivity is obtained at the compositions of x = 0.3~ 0.5, which is around 10% lower than YSZ and 33% lower than YDC at 500 ºC. The thermal conductivities of the solid solutions in ZrO2-rich region are almost temperature-independent, and show marginal composition dependence. On the contrary, the thermal conductivities of the solid solutions in CeO2-rich region decrease with increasing temperature, and show obvious composition dependence. The temperature and the composition dependence are discussed in terms of a scattering coefficient and the defect ordering.
Yttria-stabilized zirconia (YSZ) thermal barrier coatings (TBCs) are currently used in jet engines, allowing for higher operating temperatures, and greater efficiency. However, increasing temperatures make engines more susceptible to attack by ingested airborne sand particles that melt and form calcium-magnesium-aluminosilicate (CMAS) glass, which can lead to loss of strain tolerance and subsequent TBC failure. New air plasma sprayed TBCs containing Al2O3 and TiO2 in solid solution in YSZ have been shown to mitigate CMAS attack by partial penetration followed by crystallization of the glass. Understanding how these partially penetrated TBCs would fare in jet engines is currently limited. To better simulate jet engine-like conditions, thermal gradient cycling was performed on a burner rig and compared to mechanical models. Results from isothermal and thermal gradient cycling of conventional YSZ and CMAS-resistant coatings with and without applied CMAS will be presented.
TECHNOLOGY AND THE DEVELOPMENT OF ADVANCED THERMAL BARRIER COATINGS

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Dynamic development of civil aviation and requirements of environment protection determine development of advanced turbofan engines with lower level of fuel consumption. This effect can be obtained through increase of engine service temperature, which is connected with development of advanced materials for turbine blades and new advanced thermal barrier coatings. As a typical thermal barrier coatings obtained with EB-PVD method with columnar microstructure are used for protection of turbine blade surface. Diffusion aluminide and platinum-modified aluminide coatings or MeCrAlY coatings obtained by PVD method are used as bond-coat, which has corrosion resistant properties. Research and Development Laboratory for Aerospace Materials conducts development work on creating modern and advanced TBC coatings, which are formed on (casted in our Lab) monocrystalline blades. Development of bond-coats is connected with application of CVD technology for diffusion aluminide coatings. BPXPro 325S IonBond equipment is used for this purpose. Currently, a research is being conducted on development of aluminide zirconium- and hafnium-modified coatings with a using of external high-temperature generators. Moreover, a research on using the electroplated platinum and palladium for obtaining of aluminide coatings modified by those elements is being conducted. Described coatings will be used as bond-coats for thermal barrier coatings deposited by EB-PVD i LPPS- Thin Film methods. EB-PVD SmartCoater device is prototype system of ALD, which enables obtaining of ceramic coatings on elements created for research purposes. The development of deposition of coatings with a “zigzag” microstructure and with gradient of structure with a use of new ceramic (for example perychlore-type) materials is planned. There will be also a research on manufacturing technology of ingots for this kind of coatings conducted. LPPS Thin Film technology developed by Sulzer Metco allows creating ceramic coatings for TBC coatings with complex chemical composition and EB-PVD-like structure, obtained during Plasma Spray Physical Vapour Deposition, which enables evaporation of ceramic material. Moreover, it is possible to apply the described system to obtain the MeCrAlY bond-coats with a use of classical LPPS Thin Film methods. Apart from coating deposition, the research on isothermal, cyclic oxidation and hot corrosion resistance as well as mechanical properties is being conducted.
LIFETIME MODELING OF COAL FLY ASH INFILTRATED THERMAL BARRIER COATINGS IN CYCLIC THERMAL GRADIENT TESTING

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Thermal barrier coatings are used to protect metal components in gas turbine engines from the extreme temperatures and combustion environment. With the increasing cost of natural gas, power-generating turbines are looking towards synthetic gases, which have the potential of hosting numerous impurities and high water vapor content. The effects of water vapor and one such impurity, coal fly ash, are examined on conventional yttria-stabilized zirconia coatings, as well as gadolinium zirconate coatings in a thermal gradient burner rig. The time to spallation is measured for the coatings with and without water vapor and ash impurity deposition, and the results are framed in the context of existing mechanical models. These models are then adapted to more accurately take into account the cyclic nature and time-dependent properties of the coatings and deposition. The result is a novel model that accurately predicts failure based on material properties, sintering rate, thermally grown oxide growth rate, and thermal history.
Due to their inner hollow structure, the hollow micro-nano-powders have many special physical and chemical properties and are more suitable for the preparation of coatings. In this paper, the preparation process of hollow YSZ micro-nano-powders used for thermal spraying was investigated and the properties of their coatings were further validated. The result shows that the hollow micro-nano-powders, composed of the center hole and the dense micro-nano outer wall, could be gained from the original nano-powders by spray drying, follow-up heat treating and plasma densification process. Meanwhile, in the same atmospheric plasma spraying conditions, the thermal barrier coatings prepared by such hollow micro-nano-powders performed better in thermal insulation properties.
EXPERIMENTAL TESTS FOR MEASURING INTERFACE FRACTURE TOUGHNESS OF THERMAL BARRIER COATINGS

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The increase of aircraft engine performances is tightly related to the increasing combustion gas temperature. Therefore, the aeronautic turbine blades are exposed to extreme thermo-mechanical stresses and oxidation phenomena. In order to limit their exposure to high temperatures and increase the thermal fatigue life, a thermal barrier coating (TBC) is deposited onto their surfaces. However, due to internal as well as external stresses, this ceramic protection spalls, leaving the bare metal exposed to a high temperature environment, and then leading quickly to blade degradation. Our work focuses on getting essential data in order to build a physically-based model, able to assess thermal barrier spalling on turbine blades. An energetic approach has been chosen. Several mechanical tests have been developed to characterize the interface toughness of thermal barrier coatings, as a function of the ageing state of the system. The first developed one is a modified four-point bending test. This test is commonly used and widespread for characterizing thin film adhesions. However, it provides a delamination of the thermal barrier coating, only under near-mode I cracking conditions, whereas it is generally believed that the delamination of TBC on turbine blades occurs under near-mode II conditions. A second experimental facility has been developed for measuring delamination toughness with the objective to be close to mode II conditions. This novel test is called four points end notched flexure, and is derived from the standard test ENF. Under this particular cracking condition, the thermal barrier coating is believed to be submitted to roughly the same condition as on the turbine blade. Such a test is therefore well adapted to investigate what is happening during crack propagation. In order to reproduce the temperature cycles experienced by the blade in service, our samples are preliminary aged by heating them N times at high temperature. Then, with thermal cycles at different temperatures and different durations, we can determine the evolution of the interface toughness as a function of the thermal history and the delamination mode. The first results, obtained using these two experimental facilities, on a YPSZ EBPDV ceramic top coat deposited on an AM1 Ni-based superalloy with a (Ni,Pt)Al bond coat, will be presented and discussed.
State-of-the art thermal barrier coatings (TBCs) based on the yttria-stabilized zirconia (YSZ) system are subject to a maximum use temperature due to their dependence on a “non-transformable” metastable tetragonal phase, t'-YSZ. Upon long-term exposure at high temperature this metastable phase eventually decomposes into a yttria-rich cubic phase and a yttria-lean tetragonal phase, the latter susceptible to the deleterious monoclinic transformation upon cooling. However, recent work has revealed that t'-YSZ decomposes at a small fraction of the time necessary for the monoclinic phase to evolve. The resulting microstructure consists of a coherent array of yttria-lean and yttria-rich lamellae, which are constrained from transforming to the monoclinic phase. On this basis, the nature of t'-YSZ has been redefined to encompass this modulated architecture and the influence of the microstructure on the phase evolution path in YSZ TBCs has been reassessed. Specifically, the evolving microstructures for EBPVD and APS TBCs have been characterized via both x-ray diffraction and transmission electron microscopy and compared using a Holloman-Jaffe type parameter to further elucidate the mechanisms leading to complete destabilization and predominance of the monoclinic phase. The factors emerging from this comparison include homogeneity of the initial microstructure both in size and composition.
Failure mechanisms in thermal barrier coatings (TBCs) often involve the propagation of delamination cracks through the ceramic layer. Mode I toughness measurements on air plasma-sprayed, dense, vertically cracked (DVC) 7-8YSZ TBCs revealed toughness values of $\Gamma_{Ic} \sim 350\ J/m^2$. These are much higher than values obtained on dense specimens of the same material by other means, motivating an analysis of the test itself and the possible mechanisms responsible for the toughness elevation. On the experimental side, digital image correlation was used to provide local displacement data along the entire length of the cantilever, including at the load point, and to reveal the location of the crack front. In analyzing the experiment, the compliant foundation of the cantilever (due to the presence of the TBC) and shear effects at the crack tip must be included. Finite element analysis of the DCB specimen introduces a finite layer (with reduced stiffness) between the beams to create a physical manifestation of the compliant foundation construction. The model produces results that are consistent with those from experiment; it is thus possible to calculate an energy release rate solely with measured parameters, known material properties and the displacement data. Additional experiments on a variety of air plasma-sprayed coatings show the evolution of the toughness and the possible contributions of multiple toughening mechanisms, including ferroelastic domain switching, crack bridging and pull-out.
This work presents the development and application of suitable measuring methods to determine the elastic and fracture mechanical parameters of plasma-sprayed TBCs. Special attention was paid to the suitability of the procedures to an industrial environment (simple preparation, reproducibility, use of standard measuring techniques) and the direct applicability for production coatings. The methods developed supply reliable values of the modulus of elasticity, the fracture toughness and the strain energy release rate, as could be confirmed by measurements of model materials with well-known properties. By applying these procedures to TBC samples of different degrees of thermal aging (freely sintered at 1000°C - 1300°C) a data set was generated, with whose assistance TBC characteristics under near-operating conditions can be predicted.
ANALYTICAL TOOLS FOR INVESTIGATION OF EX-SERVICE THERMAL BARRIER COATINGS

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Assessment of ex-service parts is important for power generation industry; giving the opportunity to correlate part conditions to specific operating conditions like fuel used, local atmospheric conditions, operating regime, temperature load. It also allows checking and validating thermal as well as mechanical models used for designing and assessing mechanical integrity of gas turbine parts. One of the most valuable parameter for the part assessment is the knowledge of the local thermal condition; an information that is not trivial to obtain during engine operation or determine accurately by modeling. In Alstom, a method has been developed, allowing determination of an ex-service part average surface temperature. The method has been validated by comparing it to thermal models and to base metal temperature mapping, showing a good correlation with both types of data. Data will show how this method can be applied for assessment of ex-service combustor liner condition and how the measured TBC surface temperature can be correlated with other coating properties.
Some materials of aluminate type with relative low thermal expansion coefficients have been discussed for TBC application since several years. Examples of those are the group of Rare-earth doped hexaaluminates owing magnetoplumbite structure or YAG owing garnet structure. These aluminates tend to be in a partly amorphous state when deposited by means of thermal spraying. Re-crystallization takes place at elevated temperatures and segmentation cracks can form which might induce increased strain tolerance.

Thermal gradient cycling tests and SEM inspections indicate that the incorporation of incompletely molten particles into the coating plays an important role to the mechanical stability and thermal cycling lifetime. In this study the role of grain size and plasma spraying parameters on the microstructure evolution after heat treatment has been studied e.g. by means of DPV measurements, XRD and materialographic analysis. These results will be also discussed with respect of the lifetime of double layered TBCs utilizing YAG or LaLiAl$_{11}$O$_{18.5}$ as a topcoat onto a standard YSZ layer in thermal gradient tests.
THERMOGRAPHIC ONLINE MONITORING OF FAILURE EVOLUTION OF THERMAL BARRIER COATINGS IN GAS BURNER THERMAL CYCLING RIG ENVIRONMENT

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One major shortcoming of thermal barrier coatings applied to gas turbine components is the spallation of the ceramic coating under mechanical stress developing during thermal cycling environments. In order to study the evolution of failure and the expectancy of lifetime under realistic conditions cycling burner rig tests are a well established matter of choice. In the same way the technique of infrared (IR) thermography has been widely proofed to provide insight to microscopic crack formation and localization of hidden delaminations, respectively. The technique can be utilized to record the evolution of microscopic and macroscopic defects in advance to the apparent failure. Indirectly, this knowledge allows to verify and to improve lifetime models.

The aim of this study is to expand the use of IR testing as a rugged in-situ monitoring tool for combustion driven cycling rigs and to provide spatial resolved information on thermal load and failure evolution of the TBC in these tests. For a successful application to an experiment using a gas fired and air cooled burner rig it is necessary to overcome some limitations which are mainly due to the high level of interfering signals under these experimental conditions. For the IR thermography the evaluation of data recorded from transient states can be used to eliminate variations due to the inhomogeneous combustion heating.
Bond coats on Ni-base alloys are used in aero engines and stationary gas turbines and undergo thermal cycling during their lifetime. Thus, the composition changes due to diffusion between the bond coat and the substrate and to oxidation and hot corrosion. Important for the system besides the microstructural changes that will occur under thermal loading, are also the mechanical properties. In this work, several bond coats applied on PWA 1484 and IN 100 Ni-based superalloys are investigated before and after at 1100°C in ambient air. The mechanical properties as measured by nanoindentation are correlated with their chemical composition focusing on nickel, aluminum and chromium. Since the main phase of the investigated bond coats is usually beta-NiAl, which is very brittle at low temperatures, its ductility should be increased by varying the composition. Aluminum and chromium form many intermetallic phases over the whole composition range. This leads to a great variety of phases which influence the mechanical properties of the bond coat differently. Phase identification was carried out with x-ray diffractometry, energy dispersive x-ray analysis and electron backscatter diffraction. Nanoindentation was used to investigate the local mechanical properties of the phases with employing a Nanoindenter XP from Agilent. Indenting measurements in each phase separately provides information for hardness as well as Young’s modulus. Comparison of the ternary phase diagram of Ni-Al-Cr with the obtained data from the methods mentioned above allows linking hardness and Young’s modulus with the chemical composition of the bond coats.
IN-SITU TENSILE TESTING AND RESIDUAL STRESS CHARACTERIZATION OF NIAL BOND COATS USED ON NICKEL BASED SUPERALLOYS

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Bond coats on turbine blades are exposed to thermal and mechanical loads. Additional most coatings are pre-stressed by internal stresses which arise usually from the coating process or thermal cycling. In order to estimate the stress limits of the coatings system, the applied stresses and the residual stresses have to be known. Tensile testing is a widely-used method to evaluate the mechanical properties of materials. Characterizing coatings in this manner is usually done by testing the coating on a substrate which results in evaluation of both materials. In this work micro tensile experiments are performed on freestanding aluminum bond coats. Dog bone shaped specimens with a thickness of 100 µm are prepared from the bond coat and the substrate using electrical discharges machining (EDM) which are then polished to the final thickness. The samples are mounted on a micro tensile testing machine which can be placed in a scanning electron microscope (SEM) allowing the observation of the specimen during deformation. From images taken during deformation the strain is calculated using digital image correlation (DIC). With this approach the elastic and plastic behavior of the coating itself can be evaluated similar to macroscopic testing. The superalloy substrate shows a hardening behavior after yielding and after about 15 % strain the sample failed in shear fracture. Results of the tensile tests on the more brittle bond coats will be shown in this presentation. The residual stress was characterized by a FIB milling method introducing rectangular slits of 4 µm by 20 µm in the surface of the coating. The resulting relaxation of the surrounding material is measured using digital image correlation. Based on the relaxation the residual stresses are calculated and compared to finite element models. This procedure offers the possibility to measure residual stresses on a small scale and for a variety of materials which are hard to characterize with other methods. In case of the bond coats compression stresses in the order of 0.5 GPa are found.
The refinement, protection or functionalization of surfaces by versatile, economic and environmentally-friendly Physical Vapor Deposition (PVD) technologies plays an increasing role in modern manufacturing. Specifically, electron beam (EB) sources can beneficially be used as heating sources for materials evaporation because they provide the highest coating rates for industrial-scale processes. FEP has developed powerful plasma sources which can be combined with EB evaporation thus providing ionization and excitation of the vapor and, eventually, reactive gas species. This is the key to growing well adhering layers with controlled morphology and composition at high rates. The potential of plasma-activated high-rate deposition of yttria-stabilized zirconia (YSZ), a material typically used for thermal barrier coatings, was studied using a hollow cathode arc plasma source and an axial EB gun. The influence of plasma parameters on layer microstructure and layer properties was investigated and results will be presented. It could be validated that more dense YSZ layers can be deposited with plasma-activation in contrast to the typically columnar microstructure of coatings deposited by EB evaporation without plasma. A strong influence of plasma activation on the layer microstructure and texture was found at a deposition rate in the range of 20 to 80 nm/s. The challenging process conditions typical for plasma-activated high-rate EB-PVD have triggered substantial equipment innovations concerning the EB guns, the high-voltage power supplies and the control systems, too. As an enabling tool for advanced coating processes, FEP has developed a new class of high-power (up to 300 kW) EB modules. They excel by improved dynamic pressure decoupling stages between EB gun and process chamber (max coating pressure of 50 Pa), enhanced acceleration voltage (up to 80 kV), implementation of high-speed deflection systems (up to 100 kHz), availability of high-dynamic mid-frequency power supplies (arc recovery time in the 1-10 ms range), and advanced concepts for close-loop control of the beam deflection patterns. Another field of work at FEP is dedicated to the development of high-power axial EB guns which are favorable in cost, of compact design and easy to maintain. Contrary to industrially established systems, plasma-stimulated electron emission from cold or discharge-heated solid cathodes is used here for beam generation. Beam sources of this type can be utilized as economic but versatile annealing or preheating tools as required, e.g., in turbine blade coaters.
Ion plasma deposition (IPD) has been used to fabricate Hf-containing β-phase bond coats with varying Pt and Pd concentrations. The oxidation and rumpling behavior of these bond coats were investigated under cyclic and isothermal conditions and compared with state-of-the-art platinum aluminide coatings. Pt and Pd additions were found to increase the spallation resistance while the presence of Hf was not found to inhibit rumpling. Additionally, the relationship between the rate of rumpling and oxide growth has been analyzed and will be discussed.
Calcium magnesium alumino-silicate (CMAS) deposits on thermal barrier coatings (TBCs) melt above ~1200°C, whereupon they infiltrate and crystallize within the pores of the structure, stiffening the penetrated layer and leading to a loss of strain tolerance. This loss of compliance can lead to coating delamination when the strains generated during thermal cycling produce sufficiently high stresses in the affected coating. To determine the thermal conditions under which TBC delamination will occur, a novel laser thermal gradient test (LGT) was developed that allows control of the thermal gradient across the TBC as well as the thermal history. Both 7YSZ and gadolinium zirconate (GZO) TBCs, with and without CMAS deposits, were subjected to the LGT. In the absence of CMAS, no microstructural degradation was observed in either the 7YSZ or GZO TBCs. When loaded with CMAS, delamination cracks near the TBC-substrate interface were observed when cooled slowing. However, these cracks did not induce coating exfoliation. When rapidly cooled, an extensive network of delamination cracks grew in both GZO and 7YSZ which resulted in coating exfoliation.