

Selected Abstracts of Thermal Spray Literature

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Applications

Biomaterials and Bioactive Materials

Behavior of plasma-sprayed hydroxyapatite coatings onto carbon/carbon composites in simulated body fluid. Two types of hydroxyapatite (HA) coatings onto carbon/carbon composite (C/C composites) substrates, deposited by plasma spraying technique, were immersed in a simulated body fluid (SBF) in order to determine their behavior in conditions similar to the human blood plasma. Calcium ion concentration, pH value, microstructure, and phase compositions were analyzed. Results demonstrated that both the crystal Ca-P phases or the amorphous HA do dissolve slightly, and the dissolution of CaO phases in SBF was evident after 1 day of soaking. The calcium-ion concentration was decreased and the pH value of SBF was increased with the increasing of the immersing time. The precipitation was mainly composed of HA, which was verified by X-ray diffraction (XRD) and electron-probe microanalyzer.

Keywords: body fluids, calcium, carbon composites, coatings, concentration (process), hydroxyapatite, hydroxyapatite coating, plasma spraying, x ray diffraction analysis, simulated body fluid

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Grit blasting of Ti-6Al-4V alloy: Optimization and its effect on adhesion strength of plasma-sprayed hydroxyapatite coatings. The effect of grit blasting parameters on the surface roughness of Ti-6Al-4V alloy as the substrate for plasma-sprayed hydroxyapatite (HA) coatings was examined using the factorial and Taguchi designs of experiments. In this study, two grit materials (Al_2O_3 and SiO_2) each at two sizes, and two types of blasting systems (pressure and suction) were used. An equivalent surface roughness of $3.51 \mu m$ was obtained in three optimum conditions. The results of the Taguchi designed experiments were analyzed using signal to noise ratio. The tensile bonding strength of HA coatings deposited on the roughened substrates at the three different optimum conditions was measured by the standard adhesion test (ISO 13779-4). As the crystallinity of the coating at the interface, evaluated by the XRD analysis, reduced the bonding strength of the coatings was increased. These findings suggest that the substrate surface topography significantly influences the properties of the coating at the interface.

Keywords: aluminum alloys, blasting systems, bond strength (materials), grit blasting, hydroxyapatite, optimization, plasma-sprayed hydroxyapatite (HA) coatings, plasma spraying, silica, surface roughness, Taguchi designs, Taguchi methods, titanium alloys, x-ray diffraction

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Influence of particle temperature and velocity on the microstructure and mechanical behavior of high velocity oxy-fuel (HVOF)-sprayed nanostructured titania coatings. Nanostructured titania feedstock powders were deposited via high velocity oxy-fuel (HVOF) spraying onto Ti-6Al-4V substrates. Using in-flight particle diagnostics, different particle temperatures and velocities were employed in order to reveal their effects on microstructure and mechanical properties of the coatings. A series of linear dependencies were observed involving processing conditions (i.e., in-flight particle temperature and velocity) and characteristics of the resulting coating microstructural features and properties, such as, phase composition, Vickers microhardness and the deflection of Almen strips (residual stress). High-bond strength values were observed when compared with other ceramic thermal spray coatings available in the literature. This study provides different levels of information on the processing of nanostructured ceramic powders via HVOF spraying and opens possibilities for development and application of HVOF-sprayed nanostructured titania coatings in the biomedical field and other disciplines, where superior mechanical behavior is required.

Keywords: bond strength (chemical), high velocity oxy-fuel (HVOF), in-flight particle diagnostics, linear dependencies, microhardness, microstructure, nanostructured titania, protective coatings, thermal spraying, velocity measurement

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Nano-hydroxyapatite coatings on titanium substrates. Finite element analysis of process and experimental plasma thermal sprayed coatings. The aim of this study was to create a nano-structured coating using Plasma Thermal Spraying (PTS). This process consists in introducing pre-agglomerated nanosized particles in a high-temperature and high-velocity gas jet and projected them onto the substrate to form, layer by layer, a nanostructured coating. In order to retain nanometer grain sizes in the deposited coating through specific PTS technologies, a thermal field and velocity distribution in the plasma jet are analytically calculated. A finite element analysis is employed to calculate the thermal field evolution inside the agglomerated particles and the thermal induced internal stress distribution is determined. The parameters determined by the theoretical analysis are used for experimental coatings. The average crystallite size of nano-hydroxyapatite powder was 90 nm. After deposit via Plasma Thermal Spraying (PTS) process and followed by a 2 h heat treatment to reduce amorphous fraction, the experimental deposited coating shows that it retains the nanometer crystallite sizes. The substructure of nanocrystals was evaluated at about 120 nm in size. Such a nanocoating may play the role of nucleation site to bone, allowing a faster stabilization of the implant.

Keywords: agglomeration, amorphous fraction, finite element method, hydroxyapatite, nanostructured coating, nanostructured materials, plasma thermal spraying (PTS), thermal spraying, titanium alloys

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The design and manufacture of biomedical surfaces. Surfaces are the primary place of contact between a biomaterial and its host organism. Typically, prostheses have to fulfill demanding structural and mechanical requirements, yet the material best for those functions may be bio-incompatible. Surface treatment or coating provides a means to overcome that problem, which means both integration within the host physiology and stabilization with respect to corrosion and wear. The adsorption of biomacromolecules is pivotal for biocompatibility. The impossibility of keeping proteins away from most implants means that very careful consideration has to be given to this aspect, and both prevention (for bloodstream implants) and promotion (for bone replacement and repair) occur with equal importance. This paper also considers the metrology of relevant physical and chemical aspects of surfaces.

Keywords: adsorption, biomaterials, bloodstream implants, bone replacement, chemical aspects, corrosion, host organism, physiology, problem solving, stabilization, surface treatment

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Zirconium oxide coating improves implant osseointegration in vivo. Objectives: Zirconium is widely used as material for prosthetic devices because of its good mechanical and chemical properties. When exposed to oxygen, zirconium becomes zirconium oxide (ZO, chemically ZrO_2) which is biocompatible. ZO can be also prepared as a colloidal suspension and then used to coat surfaces. Zirconium oxide coating (ZOC) can potentially have specific biologic effects. Methods: The effect of ZOC on bone throughout an in vivo study using dental implants covered with ZOC and then inserted in rabbit tibia was tested in this study. Results: The histologic analysis demonstrated that (1) bone growth is more evident around ZOC fixtures than in controls and (2) a more mature bone is present in the peri-implant ZOC surface than in controls. Significance: ZOC can enhance implant osseointegration.

Keywords: animal model, bioactivity, biocompatibility, coating techniques, dental prostheses, fixture, implants (surgical), osseointegration, oxygen, zirconia

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Thermal Barrier Coatings

Advanced thermal gradient mechanical fatigue testing of CMSX-4 with an oxidation protection coating. Frequently, turbine blades are cooled internally, which generates thermal gradients over the blade wall and, consequently, multi-axial stresses in addition to stresses due to centrifugal forces. In order to study these conditions, a new thermal gradient mechanical fatigue (TGMF) testing equipment with a lamp furnace has been developed. The advantages of this furnace are high power (16 lamps at 1000 W), controlled thermal gradients, high heating and cooling rates, and lamp lives exceeding 8000 thermal cycles. The studied material system was the single-crystalline superalloy CMSX-4 with a NiPtAl oxidation protection coating. Different TGMF tests with a maximal surface temperature of 1050 °C, mechanical loads up to 400 MPa, and cycle numbers up to 9000 resulted in microstructural changes and defects, reflecting in each case the particular temperature, thermal gradient, and local stresses. For example, phase evolution of the metal coatings and rafting of the γ/γ' substrate morphology was investigated. Furthermore, cracks at the inner specimen surface and at substrate pores were detected. The observed morphology and defects were related to the applied thermo-mechanical loads using finite element calculations.

Keywords: cracks, fatigue testing, morphology, oxidation, protective coatings stress analysis, superalloys, thermal gradients, thermomechanical fatigue/cycling, thermomechanical loads, turbomachine blades, turbine blades-protection coating

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Crack growth in a coated gas turbine superalloy under thermo-mechanical fatigue. The out-of-phase thermo-mechanical fatigue (TMF) behavior of the single crystal superalloy CMSX-4 was studied in the uncoated and in the low-pressure plasma sprayed (LPPS) coated condition. In addition, growth measurements of in-depth coating cracks were performed. Cyclic hardening of the substrate and superimposed multiple crack initiation/propagation within the coating leads to cyclic softening that consumes a significant portion of TMF lifetime. A three-step crack propagation mode of the coated material is described. Special attention is paid to the role of the inter-diffusion zone, since coating cracks are decelerated in the inter-diffusion zone before penetrating into the substrate.

Keywords: coated materials, coating cracks, crack initiation, fatigue crack propagation, gas turbines, inter diffusion zones, single crystal nickel base superalloy, single crystals, superalloys, thermo mechanical fatigues

A. Jung and A. Schnell, Alstom (Switzerland) Ltd., Materials and Chemistry, CH-5401 Baden, Switzerland. Cited: *Int. J. of Fatigue*, 2008, Feb, **30**(2), p 286-291. ISSN 0142-1123.

Cyclic-oxidation behavior of thermal-barrier coatings exposed to NaCl vapor. A Ni-24Cr-6Al-0.7Y (NiCrAlY) coating was deposited on a nickel-base superalloy by low-pressure plasma spraying, and the top coating, ZrO₂ partially stabilized with Y₂O₃ (7.5 wt.%), was deposited on the NiCrAlY coating by air-plasma spraying. The cyclic-oxidation behavior of the NiCrAlY + YSZ coating exposed to NaCl vapor was investigated under atmospheric pressure at 1050 °C, 1100 °C low cyclic life, and 1150 °C. The cyclic-oxidation life of the NiCrAlY + YSZ coating in the presence of NaCl vapor was shortened compared with that in air. The higher the temperature is, the shorter the cyclic oxidation life. The oxide scale formed at the interface between the bond coat and the ceramic layer after exposure to NaCl vapor consisted of voluminous and non-protective NiO, Al₂O₃ and NiCr₂O₄ spinel. The failure of the TBC exposed to NaCl vapor occurs within the top coat and close to the YSZ/thermal growth oxide interface. The failure mechanism has been discussed based on the experimental results and thermodynamics.

Keywords: atmospheric pressure, coatings, oxidation, plasma spraying, sodium chloride, thermal growth, thermodynamics, vapors

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Damage evolution during thermo-mechanical fatigue of a coated monocrystalline nickel-base superalloy. To prevent hot gas corrosion nickel-base turbine blades used in today's jet engines, they are protected with so called Pt-modified β -NiAl coatings which are also subjected to thermo-mechanical loading during service. The effects of thermo-mechanical fatigue (TMF) loading on the coating as well as TMF life are the subjects of this paper. Microstructural investigations before, during and after thermo-mechanical fatigue treatment using atomic force and scanning electron microscopy on the monocrystalline nickel-base alloy PWA1484 were conducted to reveal the dominating failure mechanisms.

Keywords: atomic force microscopy, crack initiation, crystalline materials, fatigue of materials, gas turbines, jet engines, nickel alloys, nickel base superalloys, oxidation resistant coatings, platinum aluminide, scanning electron microscopy, superalloys, thermo mechanical fatigues

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Degradation of yttria-stabilized zirconia thermal barrier coatings by vanadium pentoxide, phosphorus pentoxide, and sodium sulfate. The presence of vanadium, phosphorus, and sodium impurities in petcoke and coal/petcoke blends used in integrated gasification combined cycle (IGCC) plants warrants a clear understanding of high-temperature material degradation for the development of fuel-flexible gas turbines. In this study, degradation reactions of free-standing air plasma-sprayed (APS) yttria-stabilized zirconia (YSZ) in contact with vanadium pentoxide (V₂O₅), phosphorus pentoxide (P₂O₅), and sodium sulfate (Na₂SO₄) were investigated at temperatures up to 1200 °C. Phase transformations and microstructural development were examined using X-ray diffraction, scanning electron microscopy, and transmission electron microscopy. Molten V₂O₅ reacted with solid YSZ to form zirconium pyrovanadate (ZrV₂O₇) at temperatures below 747 °C. However, at temperatures above 747 °C, molten V₂O₅ reacted with YSZ to form yttrium vanadate (YVO₄). The formation of YVO₄ led to the depletion of the Y₂O₃ stabilizer and deleterious transformation to the monoclinic ZrO₂ phase. In addition, studies on YSZ degradation by Na₂SO₄ and a Na₂SO₄ + V₂O₅ mixture (50–50 mol.%) showed that Na₂SO₄ itself had no effect on the degradation of YSZ. However, in the presence of V₂O₅ at high temperatures, Na₂SO₄ forms vanadate compounds having a lower melting point such as sodium metavanadate (610 °C), which was found to degrade YSZ by the formation of YVO₄ at a relatively lower temperature of 700 °C. P₂O₅ was found to react with APS YSZ by the formation of zirconium pyrophosphate (ZrP₂O₇) at all the temperatures studied. At temperatures as low as 200 °C and as high as 1200 °C, molten P₂O₅ was observed to react with solid YSZ to yield ZrP₂O₇, which led to the depletion of ZrO₂ in YSZ (i.e., enrichment of Y₂O₃ in t prime-YSZ) that promoted the formation of the fluorite-cubic ZrO₂ phase.

Keywords: degradation, gas turbines, integrated gasification combined cycle (IGCC), phosphorus compounds, scanning electron microscopy, sodium sulfate, thermal barrier coatings, transmission electron microscopy, vanadium compounds, vanadium pentoxide, x-ray diffraction, yttria stabilized zirconia

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Effect of using a piston with a thermal barrier layer in a spark ignition engine. In this study, in order to decrease cold start HC emission at idle and the wide open throttle (WOT) conditions, a thermal barrier layer (TBL) was deposited on the top surface of a piston. The engine was tested at three compression ratios of 8-2, 7.2 and 6.2 and for two different conditions for each compression ratio. At the first stage, cold start HC emission was measured for the first 180 s. Second, the performance and exhaust characteristics were measured at WOT. Cold start HC emission in the engine with TBL decreased compared to the standard engine at the compression ratios tested. While observing an increase in brake power up to 3% and 5% at the compression ratio of 7.2 and 6.2 respectively, the brake power decreased up to 6% at the compression ratio of 8.2 compared to that of the standard engine. Maximum decreases in HC emissions of the engine with TBL piston at WOT were found to be 43-18%, 47-05% and 32-12% at the compression ratios of 8-2, 7.2 and 6.2 respectively.

Keywords: brake power, combustion knock, compression ratio (machinery), internal combustion engines, pistons, thermal barrier coatings, wide open throttle

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Engineering nanostructured thermal spray coatings: Process-property-performance relationships of ceramic based materials. Nanostructured powders were deposited using thermal spraying to produce coatings having internal features of nanosized dimensions. Several ceramic based materials were studied, including WC-12 wt.%Co, TiO₂, hydroxyapatite, Al₂O₃-3 wt.%TiO₂ and yttria stabilized zirconia. The effect of the thermal spray conditions on the microstructure, phase composition, properties and performance was investigated. Key nanostructural features of the coatings were identified and their potential benefit in contributing to enhanced behavior explored. Issues relating to design strategies and process control for engineering these types of coatings with performance characteristics tailored for targeted applications are discussed.

Keywords: hydroxyapatite, microstructure, nanostructured coatings, nanostructured materials, nanostructured powders, phase composition, sprayed coatings, process-property-performance relationships, thermal spraying yttria stabilized zirconia

B.R. Marple and R.S. Lima, Industrial Materials Institute, National Research Council of Canada Boucherville, QC J4B 6Y4, Canada. Cited: *Adv. Appl. Ceram.*, 2007, Oct, **106**(5), p 265-275. ISSN 1743-6753.

Fatigue cracks in a thermal barrier coating system on a superalloy in multiaxial thermomechanical testing. Thermal barrier coatings (TBC) are applied on cooled gas turbine components. Between the heated and cooled surfaces a thermal gradient develops, resulting for constraint components in high multiaxial stresses, which may exceed stresses due to mechanical loading. In order to investigate the damage behavior of TBC systems under close to reality conditions, thermal mechanical fatigue tests with controlled thermal gradients (TGMF-tests) were performed on coated tubular specimens. The specimen substrate was made from directionally solidified nickel base superalloy IN100 DS, and the coating system comprised a NiCoCrAlY bond coat and a ceramic top coat from partially stabilized zirconia. The stress distribution over the specimen wall, which was generated in the course of one TGMF cycle, has been analysed by linear elastic Finite Element calculations. In TGMF testing specific damages occurred underneath the adherent ceramic top coat, evolving into fatigue cracks, which propagated primarily in the metallic bond coat parallel to the surface. In length sections the crack shape in this stage resembles a "smiley". During further cycling the crack path deviated to the interface between BC and top coat, enhancing spallation. The "smiley"-crack patterns, which only occurred in TGMF but not in isothermal low cycle fatigue or thermal fatigue, are discussed with respect to the cyclic local stress distribution.

Keywords: crack initiation, fatigue crack propagation, fatigue of materials, finite element method, gas turbines, local stress distribution, multiaxial stresses, nickel, stress analysis, superalloys, thermal barrier coatings, thermal gradients, thermomechanical testing, thermomechanical fatigue/cycling, thermo-mechanical treatment

M. Bartsch, B. Baufeld, S. Dalkilic, L. Chernova, and M. Heinzlmann, German Aerospace Center (DLR), 51147 Koln, Germany. Cited: *Int. J. Fatigue*, 2008, Feb, **30**(2), p 211-218. ISSN 0142-1123.

Fatigue damage of a thermal barrier coated Ni-base superalloy. High temperature force controlled fatigue testing of thermal barrier coated (TBC), bond coated only and bare Superni C263 superalloy were conducted in air. Results reveal that the endurance limits for the TBC and bond coated substrate were substantially higher than that of the base alloy, while the opposite was found for high stress, low cyclic life times. It appears that the increase in endurance limit for the TBC and bond coated superalloy is due to load shifting to the bond coat, interdiffusion of Al from coating to substrate and the premature failure for these two materials is possibly due to high stress crack imitation/growth in the TBC/bond coat layers. The mode of fracture in the substrate at very high fatigue stress was intergranular whereas that at low stress was transgranular. Spallation of the ceramic layer was evident at very high fatigue stress and also at low fatigue stress where the TBC composite specimen failed after 5,400,107 cycles during fatigue testing at 1073 K in air, due to a continuous alumina scale growth at the top coat (TBC)/bond coat interface.

Keywords: alumina, base alloy, crack propagation, bond coat interface, fatigue damage, fatigue testing, nickel, high temperature force, load shifting, low cyclic life, superalloys, thermal barrier coatings

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Gradient complex protective coatings for single-crystal turbine blades of high-heat gas turbine engines. Complex diffusion-condensation protective coatings characterized by gradient distribution of alloying elements over the thickness due to formation of a diffusion barrier layer on the surface of blades followed by deposition of condensation alloyed layers based on the Ni-Co-Cr-Al-Y system and an external layer based on a NiAl alloyed β_2 -phase and a $ZrO_2 \cdot Y_2O_3$ ceramics are presented. A complex gradient coating possessing unique protective properties at $t = 1100-1200$ °C for single-crystal blades from alloy ZhS36VI for advanced gas turbine engines with gas temperature of 1550 °C at the inlet to the turbine is described.

Keywords: condensation, diffusion, gas turbine engines, gas turbines, gradient complex heat engines, nickel alloys, protective coatings, single crystals, turbine blades

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Investigations of thermomechanical fatigue for optimization of design and production process solutions for gas-turbine engine parts. A technique for thermomechanical fatigue of gas-turbine engine parts is discussed. It is shown that induction heating at a frequency of about 400 kHz is suitable for tests of parts with thermal barrier coatings (TBC). For thermocyclic tests at conditions when the radiant component of total thermal flow is significant, a rig with gas-flame heating has been developed. Some results of the tests are presented.

Keywords: gas dynamic heating, gas-flame heating, gas-turbine engine part, gas turbines, high-frequency heating, induction heating, optimization, TBC,

thermal barrier coatings, thermal cycling, thermal fatigue, thermomechanical fatigue, thermocyclic tests

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Investigations on vacuum-evaporated CSZ thin films for thermal barrier applications. Ceria-stabilized zirconia (CSZ) thin films have been developed over Ni-base alloy substrate by vacuum evaporation method using an electron beam. X-ray diffraction (XRD) analysis of the film heat treated at different temperatures reveals monoclinic phase stabilization. Transmission measurements of the films annealed at different temperatures, in the wavelength region 300–1100 nm, indicate that the band gap energy of the films lies between 3.6 and 3.8 eV. Refractive index of the films was found to increase with the increase in the annealing temperature. Micro hardness of the films increases nonlinearly with the increase in annealing temperature, indicating an improvement in the hardness of the films. Thermal conductivity studies of the CSZ-coated substrate show a nonlinear decrease with the increase in annealing temperature. Surface investigations of the CSZ films confirm an increase in grain size and a decrease in surface roughness with the increase in annealing temperature of the films.

Keywords: annealing temperature, cerium compounds, energy gap, film heat, phase stabilization, thermal barrier coatings, thermal conductivity, thin films, phase stabilization, vacuum evaporation, Vickers hardness number, x-ray diffraction, zirconia

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Monitoring system for thermal barrier coatings with RF radar measurement. A monitoring principle able to identify damage on the thermal barrier coating of gas turbine blades and vanes is presented. The principle is applicable to in-service turbine monitoring. It is based on the resonant quarter wavelength effect of thin layers, which causes phase shift and attenuation of the reflected signal. The reflectivity is derived theoretically and evaluated by laboratory measurements. It was evident that especially the phase information is adequate to distinguish coated areas from those where coating has already been spalled off.

Keywords: gas turbine blades, gas turbines, thermal barrier coatings, phase shift, RF radar measurement, radar measurement, radar reflection, reflected signal, thin layers, turbine monitoring

A. Schicht, M. Willsch, T. Bosselmann, and K. Huber, Friedrich-Alexander University Erlangen-Nuremberg, Department of Microwave Engineering and High Frequency Technology, Erlangen 91058, Germany. Cited: *Electronics Lett.*, **43**(24), p 1357-1359. ISSN 0013-5194.

On the compatibility of single crystal superalloys with a thermal barrier coating system. The compatibility of three Co-containing prototype single crystal nickel-based superalloys with a thermal barrier coating (TBC) system is examined. These contain 2.1, 8.4 and 12.6 at.% Co; the concentrations of Al, Cr, Ta, W, Re, Hf are identical and chosen to be representative of advanced grades of these alloys. The TBC consists of an yttria-stabilized zirconia (YSZ) layer formed by electron beam physical vapor deposition (EB-PVD) and a bond coat made by electrodeposited platinum with a subsequent interdiffusion heat treatment—a so-called "platinum-diffused" bond coat. The resistance to spallation of the TBC system is degraded as the Co content of the substrate increases. Wavelength-dispersive X-ray analysis and secondary ion mass spectrometry indicate that quantities of Co are present in the thermally grown oxide (TGO) by the time that failure occurs, this effect being most pronounced when the Co content of the substrate is high; the TGO is then more wavy and convoluted. The bond coat consists exclusively of the γ_2 and γ_1 prime phases, with the balance shifting towards γ_2 with increasing thermal exposure; the loss of Al from the bond coat due to TGO formation means that the TGO is eventually in contact with the γ_2 phase solely, which is enriched in Co.

Keywords: electrodeposition, electron beams, heat treatment, mass spectrometry, nickel-based single crystal superalloys, physical vapor deposition, single crystals, superalloys, thermal barrier coatings, thermally grown oxide, x-ray analysis, yttria stabilized zirconia

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Open porous metallic foams with thermal barrier coatings and cooling hole array for high temperature turbine applications. Open porous and high temperature resistant Ni-base structures are developed for the requirements of an effusion cooling. A metallic foam, that is produced by the Slip Reaction Foam Sintering (SRFS)-process, a powder metallurgical process, is used as the open porous structure. To withstand the high temperatures in the combustor of a gas turbine of up to 1520 °C the samples are covered with a Thermal Barrier Coating (TBC) using thermal spraying processes, which hermetically seals the open porous structures. Laser drilling is used to form blind holes through the TBC into the metallic foam in order to establish connections to a number of pores, which

make the mass flow of the cooling fluid possible. Therefore, the pores must not be clogged by molten material during laser drilling. The aim of this paper is to point out the general feasibility of the production steps of the open porous multi-layer component made out of the open porous foam and the thermal barrier coating, which is then opened by laser drilling. First design suggestions are given for the foam pore structure to enable the application of the TBC and for the dimension of the laser drilled holes.

Keywords: cooling, cooling fluid, drilling, high temperature effects, high temperature resistsants, laser drilling, thermal barrier coatings, thermal spraying, turbines

S. Angel, E. Ratte, W. Bleck, K. Bobzin, E. Lugscheider, R. Nickel, K. Richardt, N. Bagcivan, K. Walther, E.W. Kreutz, I. Kelbassa, and R. Poprawe, Surface Engineering Institute, IOT, RWTH University of Aachen, 52066 Aachen, Germany. Cited: *High Temp. Mat. Process.*, 11(3), p 321-343. ISSN 1093-3611.

Phase stability of thermal barrier oxides: A comparative study of Y and Yb additions. The maximum operating temperature of conventional thermal barrier coatings based on yttria-stabilized zirconia is ultimately limited by de-stabilization of the "non-transformable" t' phase, rendering it susceptible to the monoclinic transformation upon cooling. Investigations into alternative thermal barrier oxide compositions suggest that Yb offers superior t' phase stability compared with Y, Sc and larger rare earth cations at the same concentration. The present study sheds light on this behavior by comparing the microstructure evolution of specimens with 7.6% and 11.4% $MO_{1.5}$ ($M=Y$ or Yb) heat treated at 1450 °C for times up to 512 h. X-ray diffractometry and transmission electron microscopy revealed that the onset of partitioning occurs at short times but then the compositions of the phases evolve slowly over time until sufficient stabilizer is depleted from the t' phase to render it transformable. Substitution of Yb for Y delays the onset of monoclinic formation. Differences in the transformation behavior of the Y and Yb rich phases on cooling provide new insight and suggest refinements to the current thermodynamic models for the binary $ZrO_2MO_{1.5}$ systems are needed.

Keywords: mathematical models, microstructural evolution, monoclinic formation, phase stability, positive ions, substitution reactions, thermal barrier coatings, thermal barrier oxides, thermodynamics, yttria stabilized zirconia

J.M. Cairney, N.R. Rebollo, M. Ruhle, and C.G. Levi, Materials Department, 1361D Engineering II, University of California, Santa Barbara, CA 93106-5050. Cited: *Int. J. Mater. Res.*, 2007, Dec, 98(12), p 1177-1187. ISSN 1862-5282.

Study of failure of EB-PVD thermal barrier coating upon near- α ; titanium alloy. The cracking failure of a conventional thermal barrier coating (TBC), consisting of a near- α ; titanium substrate, a NiCoCrAlY bond coat (BC), and a 8 wt.% yttria-stabilized zirconia ceramic layer deposited by electron beam-physical vapor deposition (EB-PVD) method, was studied by cyclic furnace testing and isothermal exposure. The scanning electron microscope, electron probe microanalysis, and microhardness indentation were used to probe the failure mechanism. It is found that due to the mismatch of the coefficient of thermal expansion, the as-deposited BC is suffered the long-term tensile creeping at room temperature. During the high-temperature exposure, the TBC locally rumples, bringing in-plane tensile stress at the shoulders, and out-of-plane tensile stress at the peak of the rumped BC, where primal cracks are originated. During the cooling period, the ridges of substrate pulled by the local rumppling of the BC blocks the contracting of the BC, originating new cracks in planar BC, and aggravating the original cracks. Furthermore, the oxidation products pushed into the BC and the 8YSZ enlarges the TBC and cracks the substrate along the weakest diffused grain boundaries. The cracking failure related to the diffusion of the BC to the substrate is also discussed.

Keywords: crack propagation, electron beam-physical vapor deposition (EB-PVD) method, electron probe microanalysis, failure mechanisms, failure modes, microhardness, physical vapor deposition, thermal barrier coatings, titanium alloys, titanium substrates

B. He, F. Li, H. Zhou, Y. Dai, and B. Sun, State Key Laboratory of Metal Matrix Composites, School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai 200240, China. Cited: *J. Mater. Sci.*, 2008, Feb, 43(3), p 839-846. ISSN 0022-2461.

Thermal fatigue life of thermal barrier coatings. Two thermal barrier coatings (TBCs), the plasma sprayed (PS) and electron beam-physical vapor deposition (EB-PVD) coatings were selected to analyze the fatigue life. Firstly, the oxidation and thermal fatigue experiments were carried out, which were domestic spraying technology. The failure mechanisms of oxidation and thermal fatigue of TBCs were considered together. The weight of thermally grown oxide (TGO) was measured during oxidation experiments and modeling. Thermal fatigue experiments were induced by thermal mismatched stress and thermal fatigue with holding time. Secondly, based on the experiment data, the life prediction model of thermal fatigue of TBCs was set up for PS and EB-PVD coatings. Thirdly, the method of obtaining the parameters of the life prediction model was given. Finally, using the life prediction model, the life of TBCs was predicted under the designed load case. The life prediction model was verified.

Keywords: electron beams, life prediction, oxidation, physical vapor deposition, plasma spraying, thermal barrier coatings, thermal fatigue, thermal mismatched stress, thermally grown oxide (TGO)

Q. Hongyu, L. Rui, Y. Xiaoguang, and Z. Lizhu, School of Jet Propulsion, Beihang University, Beijing, 100083, China. Cited: *J. Rare Earths*, 2007, June, 25(Suppl. 2), p 370-374. ISSN 1002-0721.

Toughening of nontransformable t' prime-YSZ by addition of titania. Substitution of TiO_2 for ZrO_2 into single-phase tetragonal t' 7YSZ has been shown to induce a twofold increase in toughness. This enhancement has been achieved by increasing the tetragonality of the unit cell, upon substituting Ti^{4+} for the larger Zr^{4+} cation. The observed behavior is consistent with ferroelastic toughening, but direct evidence of the mechanism is lacking. Adding TiO_2 to 7YSZ has the additional benefit that it diminishes the transformability of the depleted tetragonal form: specifically, no monoclinic phase was observed even after equilibration at 1600 °C, followed by low-temperature annealing. The combination of properties bodes well for potential application as a thermal barrier coating in gas turbine engines.

Keywords: annealing, ferroelastic toughening, gas turbines, low temperature effects, monoclinic phase, phase equilibria, positive ions, substitution reactions, tetragonal forms, thermal barrier coatings, titanium dioxide, toughness

T.A. Schaedler, R.M. Leckie, S. Kramer, A.G. Evans, and C.G. Levi, Materials Department, University of California, Santa Barbara, CA 93106-5050. Cited: *J. Am. Ceram. Soc.*, 2007, Dec, 90(12), p 3896-3901. ISSN 0002-7820.

Cold Spray

Effect of standoff distance on coating deposition characteristics in cold spraying. In this study, the effect of standoff distance on coating deposition characteristics in cold spraying in cold spraying was investigated by the experiment and numerical simulation of particle acceleration. Al, Ti and Cu powders of different sizes were used as feedstocks. It was found that the deposition efficiency was decreased with the increase of standoff distance from 10 mm to 110 mm for both Al and Ti powders used in this study. However, for Cu powders, the maximum deposition efficiency was obtained at the standoff distance of 30 mm, and then the deposition efficiency decreased with further increasing the standoff distance to 110 mm. The standoff distance had a little effect on coating microstructure and microhardness for these three powders. Both the strain-hardening effect of the deposited particles and the shot-peening effect of the rebounded particles take the roles in coating hardness. It was also found that the surface of substrate or previously deposited coating could be exposed to a relatively high gas temperature at a short standoff distance.

Keywords: aluminum coatings, cold spraying, computer simulation, copper coatings, deposition, deposition efficiency, feedstocks, microhardness, particle accelerators, powder metals, shot peening, sprayed coatings, standoff distance, strain hardening, titanium coatings

W.-Y. Li, C. Zhang, X.P. Guo, G. Zhang, H.L. Liao, C.J. Li, and C. Coddet, LERMP, Université de Technologie de Belfort-Montbéliard, Site de Sevenans Cedex, France. Cited: *Mater. Des.*, 29(2), p 297-304. ISSN 0261-3069.

Microstructure, microhardness and dry friction behavior of cold-sprayed tin bronze coatings. In this paper, two types of tin bronze coatings (Cu-6 wt.%Sn and Cu-8 wt.%Sn) were prepared by cold spray process. The as-sprayed coatings were subjected to a vacuum heat treatment at 600 °C for 3 h. The coating microstructure, microhardness and tribological performance were characterized. The effects of the tin content and the vacuum heat treatment on the microstructure, microhardness and tribological behavior of the coatings were investigated. It is found that the as-sprayed CuSn6 (As6) and CuSn8 (As8) coatings exhibit practically an identical porosity. Meanwhile, As8 presents a higher microhardness than As6. In addition, the increase of the tin content in the powder feedstock leads to a lower wear rate. After a heat treatment, coating porosities are significantly reduced. However, the coating hardness is significantly decreased and the coating presents a much decreased wear resistance. For the as-sprayed coatings, such factors as ploughing and particle delamination could determine the sliding process. The heat treatment results in a distinct modification of the tribological behavior. For the annealed coatings, the adhesion, between the coating and the counterpart, could play a dominant role in the sliding process.

Keywords: coatings, dry friction, dry sliding wear, feedstocks, heat treatment, microhardness, microstructure, porosity, tin bronze, tribology, vacuum heat treatment

X. Guo, G. Zhang, W.-Y. Li, L. Dembinski, Y. Gao, H. Liao, and C. Coddet, Laboratoire d'Etudes et de Recherches sur les Matériaux, les Procédés et les Surfaces, Université de Technologie de Belfort-Montbéliard, 90010 Belfort Cedex, France. Cited: *Appl. Surf. Sci.*, 2007, Dec 30, 254(5), p 1482-1488. ISSN 0169-4332.

Measurement Methods

Analysis of a "barb test" for measuring the mixed-mode delamination toughness of coatings. The durability of thermal barrier coatings (TBCs) employed in most turbine engines is limited by delamination, both within the

coating and at the coating-substrate interface. A test used for evaluating the mixed-mode toughness pertinent to such delaminations has been fully-analyzed by determining the steady-state energy release rate, the mode mixity and the critical length for buckling. An analytic solution based on beam theory establishes basic estimates and scaling relationships. Selected finite-element calculations have been used to affirm the fidelity of the results. The energy release rate attains steady-state, but the phase angle changes throughout because of a small (but significant) bending moment induced at the supports. The coating buckles at a critical delamination length, whereupon the energy release rate increases rapidly and violates steady state. The solutions are used to re-interpret measurements reported in the literature for an electron beam physically deposited TBC.

Keywords: analytic solution, coating buckles, coating techniques, delamination, electron beams, energy release rate, interface toughness, steady states, substrates, thermal barrier coatings, toughness, turbines

Y.-F. Liu, Y. Kagawa, and A.G. Evans, Research Center for Advanced Science and Technology, The University of Tokyo, Meguro-ku, Tokyo, 153-8409, Japan. Cited: *Acta Mater.*, 2008, Jan, **56**(1), p 43-49. ISSN 1359-6454.

Common coating thickness measurement methods. Some of the most common coating thickness measurement methods are discussed. The Coulometric Measurement method is one such method that states that a metal coating is depleted from its metallic, or non-metallic substrate material, using an electrolyte and a controlled supply of electric current. Magnetic induction method is another coating thickness measurement method that states that the coating thickness is computed from the probe signal through the probe characteristic. The magnetic induction method is suitable for measuring coatings on ferrous metals, including electroplated coatings of zinc, chromium, and copper. The magnetic method requires that a permanent magnet integrated in the measurement probe, generates a constant magnetic field, whose strength is influenced by the distance between the probe and the substrate material.

Keywords: coating techniques, Coulometric Measurement methods, constant magnetic fields, electric currents, electrolytes, electromagnetic induction, electroplating, ferrous metals, magnetic fields, probe signals, thickness measurement

Anon, Cited: *Finishing Today*, 2007, Nov, **83**(11), p 71-73. ISSN 1073-4651.

Coriolis erosion testing approach for relatively thick coatings. To meet the challenge of wear and corrosion attack in various applications, coating has been an effective protection approach for the work surface of engineering components, including slurry pump wet-end parts (e.g., impellers, liners, and shells). The coated wear parts that handle flowing slurry are exposed to both sliding and impact erosion along with possible corrosion factors. In such erosive wear conditions, relatively thick coatings including overlays may be needed to extend service life of the wear components. Different wear testing methods have been used to evaluate the tribological properties of coating materials under both dry and wet wear conditions. Previous studies have shown that Coriolis erosion testing is a valuable and effective approach to study erosive wear behaviors of materials in flowing slurry conditions such as within a centrifugal slurry pump system. Although extensive research can be found on wear of hard coating materials, coating erosive wear research conducted using Coriolis wear testing methods is very limited. This paper demonstrates that Coriolis erosion testing can be applicable for both sliding and impact wear on relatively thick coatings (typically, 250 μm or thicker). By adjusting test parameters within the Coriolis erosion testing system, such as flow rate (or solids particle velocity), solids type, size, and concentration, and impact angle, various types of coatings can be tested for different erosive wear conditions. Through such Coriolis erosion testing, erosion rate and tribological characteristics of coatings can be determined and evaluated. The coating examples used in this study include NiCrSiB, NiWCrSiB, WC-NiCrSiB, and WC-Co-Cr coatings produced with flame-spray, sintering and/or high-velocity-oxygen-fuel (HVOF) and high-velocity-air-fuel (HVOF) processes. Some related factors have also been discussed.

Keywords: coatings, coating materials, Coriolis erosion, erosion, parameter estimation, slurries, slurry erosion, thick coatings, tribology, velocity measurement, wear of materials

H.H. Tian and G.R. Addie, GIW Industries, Inc., Grovetown, GA 30813. Cited: *J. ASTM Int.*, 2007, Oct, **4**(9).

Modern methods for investigating functional surfaces of advanced materials by mechanical contact testing. Modern methods for determining the hardness, Young's modulus, elastic recovery, adhesive/cohesive strength, friction coefficient, and wear resistance of thin films, coatings, multilayer materials, and bulk materials are considered. The experimental data obtained in instrumented indentation, instrumented scratching, and tribological tests of nanostructured and quasicrystalline coatings and composite materials are analyzed. It is noted that the elastic recovery of a number of advanced materials is higher than the elastic recovery of metal alloys by a factor of 2-3. The coefficients of sliding friction of sintered samples and thin films containing Al-Cu-Fe quasicrystals are found to be relatively low. An increase in the fraction of quasicrystalline particles to 30% in composites with an aluminum matrix leads to an increase in the wear resistance.

Keywords: coatings, elastic moduli, elastic recovery, friction coefficient, hardness, instrumented indentation, mechanical contact testing, nanostructured materials, quasicrystals, surface analysis, thin films, tribology, wear resistance

M.I. Petrzlik and E.A. Levashov, State Technological University Moscow Institute of Steel and Alloys, Moscow, 119049, Russian Federation. Cited: *Crystallogr. Rep.*, 2007, Nov, **52**(6), p 966-974. ISSN 1063-7745.

Microstructure

Microstructure characteristics of Al₂O₃-13 wt.%TiO₂ coating plasma spray deposited with nanocrystalline powders. Nanostructured Al₂O₃-13 wt.%TiO₂ coating was fabricated by plasma spray with nanocrystalline powders and the microstructures of the feedstock and the coating were characterized by means of XRD, SEM and TEM. It was found that three forms of substructure existed in the coating: one evolving from the unmelted part of the feedstock and showing a round-shaped morphology; one resembling the liquid-phase-sintered structure consisting of the flattened partially melted region and fully melted region; another being of the particulate-reinforced-solid solution type with fine particles distributed in the matrix. The TEM analysis revealed that partially melted α -Al₂O₃ particles were in the size range of 20-70 nm and were embedded in the TiO₂-rich matrix. The mechanism of the substructure formation was also explained in terms of the melting and flattening behavior of the powders during plasma spray processing.

Keywords: feedstocks, microstructure, nanocrystalline powders, nanostructured coatings, plasma spraying, round-shaped morphology, scanning electron microscopy, titanium dioxide

J. Zhang, J. He, Y. Dong, X. Li, and D. Yan, School of Materials Science and Engineering, Hebei University of Technology, Tianjin, 300130, China. Cited: *J. Mater. Process. Technol.*, 2008, Feb 1, **197**(1-3), p 31-35. ISSN 0924-0136.

Photocatalytic performance and microstructure of thermal-sprayed nanostructured TiO₂ coatings. Titanium dioxide coatings were deposited by atmospheric plasma spraying (APS) with the use of agglomerated P25 powders and different spraying parameters (e.g. power) to determine their influence on the microstructure and photocatalytic performance of the coatings. The microstructure of as-sprayed TiO₂ coatings was characterized by scanning electron microscope (SEM), transmission electron microscope (TEM) and X-ray diffraction (XRD). The photocatalytic performance was evaluated by using methylene blue (MB) aqueous solution. The results showed that the power and flow of the secondary plasma gas have an important influence on the microstructure and on the anatase content of the TiO₂ coatings. Porosity is also a key factor in determining the photocatalytic performance of the TiO₂ coatings.

Keywords: inorganic coatings, methylene blue (MB), microstructure, nanostructured materials, photocatalysis, scanning electron microscopy, thermal spraying, titanium dioxide, transmission electron microscopy, photocatalytic performance, secondary plasma gas, x-ray diffraction

Z. Yi, J. Liu, W. Wei, J. Wang, and S.W. Lee, Shanghai Institute of Ceramics, Chinese Academy of Science, Shanghai, 200050, China. Cited: *Ceramics Int.*, 2008, March, **34**(2), p 351-357. ISSN 0272-8842.

Modeling

Mechanical Modeling

Bending and free vibration response of layered functionally graded beams: A theoretical model and its experimental validation. A third order zigzag theory based model for layered functionally graded beams in conjunction with the modified rule of mixtures (MROM) for effective modulus of elasticity is validated through experiments for static and free vibration response. Two systems, Al/SiC and Ni/Al₂O₃, fabricated using powder metallurgy and thermal spraying techniques respectively, are considered for the validation. The theoretical predictions for the layered beams with the ceramic content varying from 0% to 40% are compared with the experimental data for the static deflection under simply-supported and cantilever boundary conditions, and for the natural frequencies under cantilever and clamped-clamped boundary conditions. The predictions using the MROM are found to be in close agreement with the experiments for both systems, whereas the linear rule of mixtures based property estimates lead to highly erroneous results. The effect of number of layers on the accuracy of the theoretical model is discussed. The accuracy of the predicted results gives confidence on the values of stress to strain transfer ratio used in the MROM for the two systems in the layered fabrication context, and also demonstrates the capability of the zigzag theory in accurately modeling the mechanics of such beams.

Keywords: beams and girders, bending strength, deflection (structures), elastic moduli, finite element method, free vibration, functionally graded materials, modified rule of mixtures, vibrations (mechanical), zigzag theory

S. Kapuria, M. Bhattacharyya, and A.N. Kumar, Applied Mechanics Department, I.I.T. Delhi, New Delhi, India. Cited: *Comp. Struct.*, 2008, Feb, **82**(3), p 390-402. ISSN 0263-8223.

Mechanisms of elastodynamic erosion of electron-beam thermal barrier coatings. The elastodynamic response of a columnar thermal barrier coating to normal impact by a spherical particle is explored using an explicit finite element method. The transient stress state involves a tensile component at the edge of each column, and this may lead to erosion. The contact problem can be simplified to the impact of a single column by an inclined wedge. The peak tensile stress is determined as a function of geometry of column and level of friction between wedge and columns. The initiation of a crack from a pre-existing flaw within the column is explored. The implications of the various calculations are discussed for erosion of thermal barrier coatings.

Keywords: dynamic loads, electron beams, erosion, finite element method, fracture mechanics, elastodynamic erosion, electron beam thermal barrier coatings, stress analysis, thermal barrier coatings, transient stress

A. Zisis and N.A. Fleck, Cambridge University, Engineering Department, Cambridge, CB2 1PZ, UK. Cited: *Int. J. Mater. Res.*, 2007, Dec, **98**(12), p 1196-1202. ISSN 1862-5282.

Process Modeling

Atomization of viscous and non-Newtonian liquids by a coaxial, high-speed gas jet. Experiments and droplet size modeling. This paper describes a collaborative theoretical and experimental research effort to investigate both the atomization dynamics of non-Newtonian liquids as well as the performance of coaxial atomizers utilized in pharmaceutical tablet coating. In pharmaceutically relevant applications, the coating solutions being atomized are typically complex, non-Newtonian fluids which may contain polymers, surfactants and large concentrations of insoluble solids in suspension. The goal of this investigation was to improve the understanding of the physical mechanism that leads to atomization of viscous and non-Newtonian fluids and to produce a validated theoretical model capable of making quantitative predictions of atomizer performance in pharmaceutical tablet coaters. The Rayleigh-Taylor model developed by Varga et al. has been extended to viscous and non-Newtonian fluids starting with the general dispersion relation obtained by Joseph et al. The theoretical model is validated using droplet diameter data collected with a Phase Doppler Particle Analyzer for six fluids of increasing rheological complexity. The primary output from the model is the Sauter Mean Diameter of the atomized droplet distribution, which is shown to compare favorably with experimental data. Critical model parameters and plans for additional research are also identified.

Keywords: atomization, atomizers, jets, mathematical models, non-Newtonian, Phase Doppler Particle Analyzer, viscosity

A. Aliseda, E.J. Hopfinger, J.C. Lasheras, D.M. Kremer, A. Berchielli, and E.K. Connolly, Oral Products Center of Emphasis, Pfizer, Inc., Global Research and Development Groton, CT 06340. Cited: *Int. J. Multiphase Flow*, 2008, Feb, **34**(2), p 161-175. ISSN 0301-9322.

Modeling of plasma spraying process to manufacture hybrid materials. A component, which has an optimized combination of different materials in its different portions for a specific application, is considered as the component made of a multiphase perfect material. To fabricate such components, a hybrid layered manufacturing process was proposed and applies spraying, engraving, and refinishing technologies, among which the spraying step is the key technology for generating a coating of hybrid materials with their required volume fractions in every unit volume. To manufacture such a coating, it is important to study the spraying characteristics. This paper intends to establish the behavior model of plasma spraying, to implement the virtual manufacturing according to the behavior model, to analyze the volume fraction error of material constituents and to optimize the related technological parameters to eliminate the volume fraction error, thus providing the reliable basis for future real manufacturing.

Keywords: behavior simulation, coatings, computer simulation, hybrid materials, hybrid layered manufacturing, multiphase perfect material, plasma spraying, rapid prototyping, virtual prototyping, virtual manufacturing, volume fraction

F. Wang, K.-Z. Chen, and X.-A. Feng, Department of Mechanical Engineering, The University of Hong Kong, Hong Kong. Cited: *CAD Comput. Aided Des.*, 2007, Dec, **39**(12), p 1120-1133. ISSN 0010-4485.

Post Processing

Improvement of mechanical properties of alumina and zirconia plasma sprayed coatings induced by laser post-treatment. Alumina and stabilized zirconia were plasma sprayed in air using a water-stabilized plasma torch. Nd-YAG laser was then used for additional treatment of the plasma sprayed coatings. The laser was maintained in a quasi-continual regime and defocused from the surface to increase the treated area. Energy density was varied together with the laser scanning velocity to ensure variance in thermal history of the treated surfaces. Microhardness, surface roughness and slurry abrasion resistance (SAR) were measured before and after the laser treatment. Results

vary in dependence on the laser treatment parameters. When the laser treatment resulted in substantial changes of the structure (up to a complete re-melting of the surface), enhancement of all measured properties was proven. It is demonstrated that the change of the mechanical properties can be correlated with the optical properties of the coating materials at the laser wavelength. Microstructural aspects of the laser treatment are discussed as well, especially at the boundary between the laser-annealed layer and the basic coating. It is pointed out that laser remelting done by the use of a high energy density can cause presence of cracks, although the wear resistance as well as microhardness of the coatings is improved by this way. A cast ceramic material Eucor with the composition $ZrO_2-Al_2O_3-SiO_2$ was treated by a diode laser utilizing the knowledge found on plasma coatings. The degree of improvement of the wear resistance, microhardness and homogeneity of microstructure was similar as in the case of plasma coatings.

Keywords: alumina, ceramic materials, laser applications, laser post-treatment, laser scanning velocity, microstructure, plasma coatings, plasma spraying, plasma torches, semiconductor lasers, slurry abrasion resistance (SAR), surface roughness, wear resistance, zirconia

P. Ctibor, L. Kraus, J. Tuominen, P. Vuoristo, and P. Chraska, Institute of Plasma Physics, Academy of Sciences of the Czech Republic, 180 00 Prague 8, Czech Republic. Cited: *Ceram-Silikaty*, **51**(4), p 181-189. ISSN 0862-5468.

Processing

Characteristics and catalytic properties of Pd/SiO₂ synthesized by one-step flame spray pyrolysis in liquid-phase hydrogenation of 1-heptyne. In this study, Pd/SiO₂ catalysts with 0.5–10 wt.% Pd loadings were prepared by one-step flame spray pyrolysis (FSP) and characterized by N₂ physisorption, X-ray diffraction (XRD), transmission electron microscopy (TEM), CO chemisorption, and X-ray photoelectron spectroscopy (XPS). The average cluster/particles size of Pd as revealed by TEM were ca. 0.5–3 nm. The turnover frequencies (TOFs) of the flame-made catalysts decreased from 66.2 to 4.3 per s as Pd loading increased from 0.5 to 10 wt.%, suggesting that the catalytic activity was dependent on Pd particle/cluster size. However, there were no appreciable influences on 1-heptyne selectivity. The flame-made Pd/SiO₂ showed better properties than the conventional prepared catalysts. Their advantages are not only the presence of large pores that facilitates diffusion of the reactants and products, but also the high-catalytic activity of as-synthesized catalysts so that further pretreatment is not necessary.

Keywords: catalyst activity, flame spray pyrolysis, heptyne hydrogenation, hydrogenation, liquid phase hydrogenation, nanoparticles, palladium, pyrolysis, silica, synthesis (chemical), transmission electron microscopy, x-ray diffraction

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Double precursor solution coating approach for low-temperature sintering of Pb (Mg_{1/3}Nb_{2/3})O_{3.0.63}PbTiO_{3.0.37} solids. Lead-based piezoelectric ceramics typically require sintering temperatures higher than 1000 °C at which significant lead loss can occur. Here, we report a double precursor solution coating (PSC) method for fabricating low-temperature sinterable polycrystalline [Pb(Mg_{1/3}Nb_{2/3}O₃)_{0.63}[PbTiO₃]_{0.37} (PMN-PT) ceramics. In this method, submicrometer crystalline PMN powder was first obtained by dispersing Mg(OH)₂-coated Nb₂O₅ particles in a lead acetate/ethylene glycol solution (first PSC), followed by calcination at 800 °C. The crystalline PMN powder was subsequently suspended in a PT precursor solution containing lead acetate and titanium isopropoxide in ethylene glycol to form the PMN-PT precursor powder (second PSC) that could be sintered at a temperature as low as 900 °C. The resultant d₃₃ for samples sintered at 900, 1000, and 1100 °C for 2 h were 600, 620, and 700 pm/V, respectively, comparable with the known value. We attributed the low sintering temperature to the reactive sintering nature of the present PMN-PT precursor powder. The reaction between the nanosize PT and the submicrometer-size PMN occurred roughly in the same temperature range as the densification, 850–900 °C, thereby significantly accelerating the sintering process. The present PSC technique is very general and should be readily applicable to other multicomponent systems.

Keywords: calcination, coating techniques, ethylene glycol, low temperature effects, piezoelectric ceramics, precursor solution coating (PSC), sintering, sintering temperature, titanium isopropoxide

H. Luo, W.Y. Shih and W.-H. Shih, Department of Materials Science and Engineering, Drexel University, Philadelphia, PA 19104. Cited: *J. Am. Ceram. Soc.*, 2007, Dec, **90**(12), p 3825-3829. ISSN 0002-7820.

In situ carbon nanotube reinforcements in a plasma-sprayed aluminum oxide nanocomposite coating. Carbon nanotubes (CNT) are potential reinforcements for toughening the ceramic matrix. The critical issue of

avoiding CNT agglomeration and introducing CNT-matrix anchoring has challenged many researchers to improve the mechanical properties of the CNT reinforced nanocomposite. In the current work, dispersed CNTs are grown on Al_2O_3 powder particles in situ by the catalytic chemical vapor deposition (CCVD) technique. Consequently, 0.5 wt.% CNT-reinforced Al_2O_3 particles were successfully plasma sprayed to obtain a 400 μm thick coating on the steel substrate. In situ CNTs grown on Al_2O_3 shows a promising enhancement in hardness and fracture toughness of the plasma-sprayed coating attributed to the existence of strong metallurgical bonding between Al_2O_3 particles and CNTs. In addition, CNT tentacles have imparted multi-directional reinforcement in securing the Al_2O_3 splats. High-resolution transmission electron microscopy shows interfacial fusion between Al_2O_3 and CNT and the formation of Y-junction nanotubes.

Keywords: agglomeration, aluminum, carbon nanotubes, catalytic chemical vapor deposition (CCVD) technique, ceramic matrix composites, chemical vapor deposition, nanocomposites, oxides, plasma spraying, powder particles, steel substrate, toughening

K. Balani, T. Zhang, A. Karakoti, W.Z. Li, S. Seal, and A. Agarwal, Mechanical and Materials Engineering Department, Florida International University, Miami, FL 33174. Cited: *Acta Mater.*, 2008, Feb, **56**(3), p 571-579. ISSN 1359-6454.

Study of injection angle and carrier gas flow rate effects on particles in-flight characteristics in plasma spray process: Modeling and experiments. This paper investigates the influence of particle injection angle on particle in-flight behaviors and characteristics at different primary and carrier gas flow rates through an integrated modeling and experimental approach. Particle in-flight status such as temperature, velocity, size and their distribution are analyzed to examine particle's melting status before impact. Results from the experiments and numerical simulations both show that, when carrier gas flow rate is fixed, a small injection angle favors the particle melting and flattening. This behavior is independent of primary and secondary gas flow rates, spray distance and carrier gas flow rate. When both carrier gas flow and injection angle vary, a high carrier gas flow rate and a small injection angle are recommended for high particle temperature and velocity.

Keywords: air plasma spray, carrier gas flow, computer simulation, flow of gases, flow rate, injection angle, melting, optimization, particle melting, plasma spraying, thermal effects, velocity measurement

W. Zhang, L.L. Zheng, H. Zhang, and S. Sampath, Department of Mechanical Engineering, Stony Brook University, Stony Brook, NY 11790. Cited: *Plasma Chem. Plasma Process.*, 2007, Dec, **27**(6), p 701-716. ISSN 0272-4324.

Supersonic laser spray of aluminum alloy on a ceramic substrate. Applying a ceramic coating onto a metallic substrate to improve its wear resistance or corrosion resistance has attracted the interest of many researchers during decades. However, only few works explore the possibility to apply a metallic layer onto a ceramic material. This work presents a novel technique to coat ceramic materials with metals: the supersonic laser spraying. In this technique a laser beam is focused on the surface of the precursor metal in such a way that the metal is transformed to the liquid state in the beam-metal interaction zone. A supersonic jet expels the molten material and propels it to the surface of the ceramic substrate. In this study, we present the preliminary results obtained using the supersonic laser spray to coat a commercial cordierite ceramic plate with an Al-Cu alloy using a 3.5 kW CO_2 laser and a supersonic jet of Argon. Coatings were characterized by scanning electron microscopy (SEM) and interferometric profilometry.

Keywords: aluminum alloys, argon, beam-metal interaction zone, ceramic coatings, ceramic substrate, corrosion resistance, interferometry, scanning electron microscopy, supersonic aerodynamics, supersonic laser spray

A. Riveiro, F. Lusquinos, R. Comesana, F. Quintero, and J. Pou, Applied Physics Department, ETS Ingenieros Industriales, University of Vigo, 36310 Vigo, Spain. Cited: *Appl. Surf. Sci.*, 2007, Dec 15, **254**(4), p 926-929. ISSN 0169-4332.

Toward the achievement of substrate melting and controlled solidification in thermal spraying. The substrate is usually kept at a distant location in traditional thermal spraying, and substrate melting, which can improve splat adhesion usually does not happen. By moving the substrate close to the plasma flame and attaching a temperature control device to the backside of the substrate, as well as by additional heating from the molten droplets, substrate melting may occur and directional splat solidification becomes possible. In this proposed design, the substrate temperature is controlled by spray distance, flame temperature and initial substrate temperature. The variations of particle in-flight characteristics and contact interface temperature on spray distance are investigated. Optimal operating conditions are determined.

Keywords: crystal growth, melting, plasma flame, plasma spraying, solidification, splat solidification, substrate melting, substrates, temperature control, temperature control device

W. Zhang, G.H. Wei, H. Zhang, L.L. Zheng, D.O. Welch, and S. Sampath, Department of Mechanical Engineering, State University of New York at Stony Brook, Stony Brook, NY 11794. Cited: *Plasma Chem. Plasma Process.*, 2007, Dec, **27**(6), p 717-736. ISSN 0272-4324.

Properties

Corrosion

Advanced military corrosion sensing technology. BAE Systems is developing an advanced corrosion sensing technology called Sentinel corrosion sensor that has the capability of generating cost reduction in military maintenance cost. The Sentinel is based on thin metallic films prepared by physical vapor deposition. The crucial phase towards this development is the ability to tailor the deposition parameters and subsequent heat treatment of the films to produce a material that will show similar corrosion behavior to the bulk alloys used in aircraft structure. The sensor is designed simulating the part of the vehicle being monitored and the structure is mimicked using thin layers of alloy with the same protective coating. Currently, the Sentinel corrosion sensor is used in conjunction with environmental monitoring sensors and is being tested on the F35 Lightning II Joint Strike Fighter program.

Keywords: alloys, bulk alloys, corrosion, corrosion protection, corrosion sensing technology, corrosion sensor, heat treatment, metallic films, physical vapor deposition, sensors

M. Hull, Cited: *Mater. Technol.*, 2007, Sept, **22**(3), p 186-187. ISSN 1066-7857.

Capability of thermodynamic calculation in the development of alloys for deposition of corrosion-protection coatings via thermal spraying. The capability of thermodynamic calculations for the development of materials for corrosion protection of steels via thermal spraying is illustrated in several practical examples. Although the thermodynamic calculations are usually performed for the equilibrium state, they can yield important information even about fast chemical reactions that are far from the equilibrium conditions. The relevance and reliability of thermodynamic calculations can be improved significantly if their results are complemented by chemical and microstructural analyses. In this contribution, details on the melting and alloying processes in technically relevant nickel-based alloys were obtained from the combination of the thermodynamic calculations, differential thermal analysis, local chemical analysis using scanning electron microscopy with energy dispersive spectroscopy of characteristic X-rays and X-ray diffraction analysis. Furthermore, the results of the thermodynamic calculations performed on nickel-based alloys clarified the role of individual chemical elements dissolved in the alloys for the corrosion resistance of the alloys and thus they contributed to the improvement of the chemical stability of these alloys during the chemical reaction with gaseous substances containing chlorine.

Keywords: chemical stability, energy dispersive spectroscopy, microstructural analyses, nickel alloys, protective coatings, reliability theory, temperature measurement, thermal spraying, thermodynamic calculations, thermodynamic properties, x-ray diffraction analysis

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Zinc-aluminum-coatings as corrosion protection for steel. An analysis was made to investigate the influence of the alloying of sprayed coatings on the corrosion performance in climates of practical relevance and to evaluate the optimized alloy compositions. Furthermore, the mechanism of corrosion and coating formation was evaluated. The protective effect increased up to an aluminum content of 15 and 22 wt.%, respectively, with corrosion rates below 1 μm per year. Electrochemical investigations found a distinctive decrease in the cathodic reaction together with an accumulation of aluminum in percent and a depletion of zinc. Due to the increasing concentration of aluminum within the layer and the correlated presence of inhibitive corrosion products, the cathodic reaction and therefore the corrosion of the sprayed zinc-aluminum coatings can be increasingly eliminated.

Keywords: aluminum content, coating formation, corrosion inhibitors, corrosion performance, corrosion protection, protective coatings, reaction kinetics, steel metallurgy, zinc alloys

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Lubrication

PM304 coating on a Ni-based superalloy rod for high temperature lubrication. PM304 coating on a Ni-based superalloy rod for high temperature lubrication has been prepared by high-energy ball milling and powder metallurgy techniques. The composition of the PM304 coating is the same as that of PS304 coating, but the microstructure is quite different. The microstructure of PM304 coating is fine and dense; the size of self-lubricating particles in the coating is very small. Self-lubricating Cr_2O_3 particles are about 100 nm, BaF_2CaF_2 particles about 1 μm , Ag particles below 5 μm , while BaF_2/CaF_2 and Ag particles precipitated from NiCr matrix are less than 50 nm. The fine

and dense microstructure results in increased tensile strength and crack growth resistance of PM304 coating. The mean tensile strength is about 46 MPa.

Keywords: ball milling, composite coatings, crack growth resistance, crack propagation, lubrication, microstructure, Ni based superalloy, nickel alloys, powder metallurgy, sintering, superalloys

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Optical

Investigation of in-flight particle characteristics and microstructural effects on optical properties of YSZ plasma-sprayed coatings. The purpose of this study is to investigate the microstructural effects of yttria-stabilized zirconia (YSZ) plasma-sprayed coatings on their optical properties in order to improve the thermal performance of these coatings that are often used as thermal barrier coatings (TBCs). Four coatings with significant microstructural differences have been manufactured. A preliminary investigation of in-flight particle characteristics has been performed to select the spray conditions. This investigation used two complementary particle diagnostic tools: the DPV2000 system and a particle collection device. Both hemispherical transmittance and reflectance spectra of the coatings have been investigated over the 0.25–10 m wavelength range. The Gouesbet-Maheu four-flux model has been implemented to calculate the absorption and scattering coefficients. Their variations are discussed in terms of microstructural effects.

Keywords: coatings, in-flight particle diagnostics, microstructural effects, microstructure, plasma spraying, thermal barrier coatings, thermoanalysis, yttria stabilized zirconia, transmittance

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Oxidation

The oxide scale formation and evolution on detonation gun sprayed NiCrAlY coatings during isothermal oxidation. NiCrAlY coatings were prepared by the newly-developed detonation gun spray process. The oxide scale formation and evolution on these coatings during isothermal oxidation in air at 1100 °C were investigated. It was found that semi-molten particles, particle debris and pores, are present in the surface layer of the as-sprayed coating. During 100 h oxidation, the particle debris and some semi-molten particles gradually change into oxide mixture consisting of spinel, chromia and nickel oxides. However, after removal of the surface layer of the coating by a grinding treatment, a dense and single-layer Cr-Al₂O₃ scale forms on the surface of the coating during the oxidation. The mechanisms governing the oxide scale formation and evolution are discussed in terms of atomic diffusion and thermodynamic stability. In addition, thermogravimetric analysis showed that the oxidation rate of the ground NiCrAlY coating at 1100 °C is much lower than that of the as-sprayed one. The residual stress in thermally grown oxide scales was investigated using photo-stimulated luminescence spectroscopy.

Keywords: detonation, detonation gun spray, growth (materials), nickel compounds, oxidation, protective coatings, semi-molten particles, spraying, thermally grown oxides

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