

Selected Abstracts of Thermal Spray Literature

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Coatings for Biological Systems

Effects of technological parameters on the microstructure of laser remelted hydroxyapatite (HA) coatings. In this paper, the influence of different laser power and scanning speed on the microstructure of laser remelted as-sprayed hydroxyapatite (HA) coatings was studied and the optimum technological parameters were obtained. The morphologies, elements, and phase analysis of both sprayed and remelted coatings were examined by means of electron probe microanalysis, x-ray diffraction, and so on. The results show that the plasma sprayed coatings could be improved by laser remelting, and the optimum technological parameters are that the laser power is 600 W and the scanning speed is 11.2 mm/s. In the technological condition, the remelted coating that has compact columnar and cellular dendritic crystal consists of HA, α -TCP, CaO, and TiO₂ phases, and the Ca/P ratio of the coating is the most approximate to that of HA. When increasing the laser power and slowing the scanning speed, the structure of the coating will become much coarser and the Ca/P ratio will deviate more from that of HA. On the contrary, if the laser power is reduced and the scanning speed is accelerated, the influence of technological parameters of laser remelting on the sprayed coating will be weakened.

Keywords: dendritic crystal, electron probe microanalysis, hydroxyapatite, laser remelted hydroxyapatite (HA) coatings, lasers, optimum technological parameters, plasma spraying, remelting, scanning, scanning speed, x-ray diffraction

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Nanostructural bioactive gradient coating fabricated by computer-controlled plasma spraying technology. The poor mechanical property of hydroxyapatite was the major problem for load bearing and implant coating in clinical applications. To overcome this weakness, a bioactive gradient coating with a special design composition of hydroxyapatite (HA), ZrO₂, Ti, bioglass was developed. This 120 μ m coating with an upper layer of 30-50 μ m porous HA produced by computer-controlled plasma spraying that maintained the energy level of the plasma to ensure proper melting of powder. The crystal size of the coating was 18.6 to 26.2 nm. Transformation of t-ZrO₂ to m-ZrO₂ reduced the thermal stress that weakened the coating and lowered interfacial strength of the coating and metal substrate. Thermal stress of sprayed coating was 16.4 MPa, which was much smaller than the sample without thermal treatment of 67.1 MPa. Interfacial strength between the coating and metal substrate was 53 MPa, which is much higher than conventional hydroxyapatite coating. Based on XRD analysis crystallinity of HA approached 98%. Therefore, high-temperature treatment improved long-term stability of the coating through improved crystallinity of hydroxyapatite and reduced other impure calcium phosphate phase.

Keywords: computer control, crystal structure, gradient coatings, hydroxyapatite, load bearing, melting, metal substrates, nanostructured materials, plasma spraying, powders, sprayed coatings, thermal stress

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XPS and bioactivity study of the bisphosphonate pamidronate adsorbed onto plasma sprayed hydroxyapatite coatings. This paper reports the use of x-ray photoelectron spectroscopy (XPS) to investigate bisphosphonate (BP) adsorption onto plasma sprayed hydroxyapatite (HA) coatings commonly used for orthopedic implants. BPs exhibit high binding affinity for the calcium present in HA and hence can be adsorbed onto HA-coated implants to exploit their beneficial properties for improved bone growth at the implant interface. A rigorous XPS analysis of pamidronate, a commonly used nitrogenous BP, adsorbed onto plasma sprayed HA-coated cobalt-chromium substrates has been carried out, aimed at: (a) confirming the adsorption of this BP onto HA, (b) studying the BP diffusion profile in the HA coating by employing the technique of XPS depth profiling, and (c) confirming the bioactivity of the adsorbed BP. XPS spectra of plasma sprayed HA-coated discs exposed to a 10 mM aqueous BP solution (pamidronate) for periods of 1, 2, and 24 h showed nitrogen and phosphorous photoelectron signals corresponding to the

BP, confirming its adsorption onto the HA substrate. XPS depth profiling of the 2 h BP-exposed HA discs showed penetration of the BP into the HA matrix to depths of at least 260 nm. The bioactivity of the adsorbed BP was confirmed by the observed inhibition of osteoclast (bone resorbing) cell activity. In comparison to the HA sample, the HA sample with adsorbed BP exhibited a 25-fold decrease in primary osteoclast cells.

Keywords: adsorption, binding affinity, bisphosphonate, cells, hydroxyapatite, orthopedic implants, phosphorus compounds, plasma spraying, x-ray photoelectron spectroscopy

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Computer-Based Design

Development of an Internet system for composite design and thermophysical property prediction. A thermophysical property prediction system for composite materials was developed and is available via Internet access. This system offers users a platform to design new composite materials and to predict properties such as density, specific heat, thermal conductivity, and thermal diffusivity based on the properties of the component materials and the composite structure. The system is composed of a knowledge base, a materials database, and a simulation system. Two simulation methods, an analytical method and a finite element method, are available to calculate the thermal conductivities of the composites. This system has been successfully used to predict the thermal conductivities of SiC whisker-reinforced aluminum alloy matrix composites, thermal sprayed ZrO₂ thermal barrier coatings, and some other composites. The reliability and effectiveness of this system have proven to be good.

Keywords: composite material design, composite materials, computer simulation, database systems, Internet, materials database, property prediction, specific heat, thermal conductivity, thermal diffusion, thermodynamic properties

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Corrosion and Oxidation

Corrosion behavior and thermal conductivity of plasma sprayed AlN/Al₂O₃ coating. Plasma sprayed alumina coating has demonstrated great potential in improving the loading capacity and service life of engineering equipments, while the low thermal conductivity limits its application when high heat exchange efficiency is required. In this paper AlN with different proportion was added into the feedstock powder of Al₂O₃, and a series of AlN/Al₂O₃ composite coatings was deposited on mild steel substrate by plasma spray. Its mechanical properties were studied and thermal conductivity was measured by transient plane source (TPS) method. XRD and EDS analysis revealed that most of the Al₂O₃ in composite coating underwent phase transformation from a phase to metastable γ phase during plasma spraying, and the mass fraction of AlN was decreased comparing with the chemical composition of feedstock powders. With the increase of AlN proportion, the microhardness reduced from 847 HV of pure Al₂O₃ coating to 685 HV when the mass fraction of AlN was 0.47, accompanied by bond strength decreasing from 27.4 to 21.3 MPa, and the corrosion rate decreased by half. The addition of AlN resulted in the increase of thermal conductivity by several times comparing with the original Al₂O₃ coating.

Keywords: alumina coatings, aluminum nitride, energy dispersive spectroscopy, feedstock powder, heat exchangers, plasma spraying, powders, protective coatings, service life, steel substrates, thermal conductivity, transient plane source, x-ray diffraction

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Corrosion characteristics of newly developed structural DMR-1700 steel and comparison with different steels for industrial applications. Corrosion characteristics of different steels in industrial environment were studied systematically to understand the protective nature of oxide scales that form on their surfaces with a view to explore the possibility of using a newly developed ultrahigh-strength steel, DMR-1700, for fabrication of components to use in industrial applications. Further, the studies related to pitting and crevice corrosion resistance of a variety of steels under industrial environmental conditions have been carried out for comparison purposes. The surface morphologies of corroded steels were observed by scanning electron microscope (SEM) in order to understand the nature of corrosion. The corrosion mechanism of steel components that fail under industrial environmental conditions has been discussed. Based on the results obtained with different techniques, the newly developed DMR-1700 ultrahigh-strength low-alloy steel in association with successfully developed high-performance protective coating has been recommended for manufacture of components to use in industrial systems. This steel helps in improving the efficiency of the systems significantly by eliminating failures during service.

Keywords: alloys, industrial applications, morphology, pitting, protective coatings, scanning electron microscopy, service life, steel corrosion, steel structures, surfaces, ultrahigh-strength steel

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Finite element simulations of microvoid growth due to selective oxidation in binary alloys. Selective oxidation-induced void growth is observed in thermal barrier coating systems used in gas turbines. These voids occur at the interface between the bond coat (BC) and the thermally grown oxide (TGO) layer. In this paper, the authors develop the modeling framework to simulate microvoid growth due to coupled diffusion and creeping in binary alloys. They have implemented the modeling framework into an existing finite element program. The developed modeling framework and program is used to simulate microvoid growth driven by selective oxidation in a binary β -NiAl alloy. Axisymmetric void growth caused by the combined action of interdiffusion and creeping is simulated. The sharpness of the void and direction of creeping are considered as parameters in this study. The simulations show that the voids dilate without any change in shape when creeping is equally likely in all the directions (isotropic). Void growth patterns similar to those observed in experiments are predicted when the creeping is restricted to occur only along the radial and tangential directions. A hemispherical void grows faster compared to a sharp void. The sharpness increases in the case of a sharp void and could lead to interactions with the neighboring voids leading to spallation of the TGO layer as observed in experiments.

Keywords: binary alloys, computer simulation, coupled diffusions, finite element method, finite element programs, gas turbines, interfaces (materials), mathematical models, microvoid growth, oxidation, thermal barrier coatings, thermally grown oxide layers

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Structure and oxidation resistance of plasma sprayed Ni-Si coatings on carbon steel. Ni-Si coatings consisting of mainly NiSi_2 and NiSi were deposited on a carbon steel by air plasma spraying. Isothermal oxidation tests of the carbon steel substrates with the Ni-Si coatings at 500 to 800 °C have been carried out. The result indicated that a protective SiO_2 -based oxide scale was formed on the surface of the coatings after oxidation. On the other hand, during oxidation, phase transformation occurred among the NiSi_2 , NiSi, and Ni_2Si phases constructing the Ni-Si coatings. This was caused by the extraction of silicon from the silicides and the reformation of silicides at the silicide/Si-blocks interface. Above 700 °C, the outward diffusion of iron and carbon became very fast and consequently decarburization happened at the coating/substrate interface, which induced the formation of pores in the substrates near the interface. In addition, grain-boundary oxidation of Cr in the steel substrate was observed above 700 °C.

Keywords: carbon steel, chromium, coatings, deposition, diffusion, extraction, grain boundaries, high-temperature oxidation, interfaces (materials), interfacial reaction, iron, isotherms, nickel alloys, oxidation resistance, phase transitions, plasma sprayed Ni-Si coatings, plasma spraying, silicon, structure of coating layer, substrates

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Electrochemical Applications and Studies

Characterization of atmospheric plasma sprayed Sc_2O_3 -ZrO₂ electrolyte coating. Sc_2O_3 -stabilized ZrO₂ (ScSZ) coating in free-standing planar shape was prepared by atmospheric plasma spraying (APS) using ScSZ powder consisting of 10 mol% Sc_2O_3 . ScSZ powder was prepared by solid phase reaction followed by sintering and screening processes. The coating microstructure was characterized using x-ray diffraction and scanning electron microscopy. The electrical conductivity of ScSZ coating was measured using both direct-current (d.c.) and alternating-current (AC) approaches. The electrical conductivity of the as-sprayed ScSZ coating was about 0.07 S/cm at 1000 °C. It is one-fourth that of the sintered one. This result is due to the lamellar structure feature with the limited interface bonding that dominates the electrical conductivity of APS coatings. The activation energy of the as-sprayed coating was 1.31 eV at temperature range less than 750 °C and 0.97 eV at above 750 °C. The change of activation energy indicates that the ion transportation dominants are changed with temperature. This fact is due to higher activation energy of the grain boundary than that of intragrain.

Keywords: activation energy, atmospheric plasma spraying, coatings, electric conductivity, electrolytes, ion transportation, microstructure, plasma spraying, scandium compounds, scanning electron microscopy, shapes, sintering, solid phase, zirconia

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Characterization of the microstructure and electrical conductivity of plasma sprayed $\text{La}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$ coating. Lanthanum strontium cobalt oxide (LSCO) has been employed as cathode and intercollector materials for solid oxide fuel cells (SOFCs). In the present study, the $\text{La}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$ layer was deposited by atmospheric plasma spray (APS). The effect of heat treatment on microstructure and electrical conductivity of the LSCO deposits was examined. The electrical conductivity of the deposits along the lamellar direction was measured by a four-probe dc approach. The microstructure of the deposit was characterized by scanning electron microscopy and x-ray diffraction analysis. The results showed that the electrical conductivity of plasma sprayed LSCO coating was lower than that of sintered bulk material with the same composition. The minor metastable phase in the as-sprayed coating reduced the electrical conductivity. The annealing treatment at 1000 °C led to the elimination of the metastable phase and improved the electrical conductivity. The lamellar structure features with the limited interface bonding limits the electrical conductivity.

Keywords: atmospheric plasma spray, cathodes, characterization, coated materials, electric conductivity, heat treatment, lamellar structure, lanthanum compounds, lanthanum strontium cobalt oxide, metastable phase, microstructure, plasma spraying, scanning electron microscopy, solid oxide fuel cells

C.-X. Li, M. Gao, C.-J. Li, W. Zhou, G.-J. Yang, and Y.-Y. Wang, State Key Laboratory for Mechanical Behavior of Materials, School of Materials Science and Engineering, Xi'an Jiaotong University, Xi'an, Shaanxi 710049, China. Cited: *Mater. Trans.*, 2006, July, **47**(7), p 1654-1657. ISSN 1345-9678.

Effect of powder structure on microstructure and electrical properties of plasma-sprayed 4.5 mol% YSZ coating. Electrolyte coatings by atmospheric plasma spraying were prepared by 4.5 mol% yttria-stabilized zirconia (YSZ) powders manufactured by agglomerate-sintered (A-S) and fusing-crashed (F-C) processes. Microstructure of the powders and the coatings were characterized using scanning electron microscopy. The electrical conductivity of the coatings was investigated using both impedance spectroscopy and direct-current methods. The results showed that the electrical conductivity of coating prepared with A-S powder was lower than that with F-C powder. It was found from the impedance analysis that both the grain and grain-boundary resistances were large in the coating formed by A-S powder. This fact resulted from deposition of partially melting of spray particles. A model was proposed to explain the effect of powder structure and melting state on the coating microstructure and properties.

Keywords: agglomeration, coatings, deposition, electric conductivity, electric properties, electrolyte coating, electrolytes, grain boundaries, impedance analysis, impedance spectroscopy, melting, microstructure, plasma spray, plasma spraying, powder morphology, powders, scanning electron microscopy, sintering, solid oxide fuel cells, spectroscopic analysis

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Electrochemical and structural characterization of heat treated Cr₃C₂-NiCr coatings. The influence of heat treatments on the electrochemical behavior of thermal spray Cr₃C₂-NiCr coatings prepared by high-velocity oxyfuel (HVOF) was studied in NaCl solution, at 25 °C, using open-circuit potential (E_{OC}) and electrochemical impedance spectroscopy (EIS) measurements. Coating characterization was performed before and after the heat treatments and electrochemical tests by scanning electron microscopy, x-ray diffraction, and Auger electron spectroscopy. In addition to the changes in the original powder composition occurring during HVOF process, heat treatment performed at 450 °C caused no significant changes in electrochemical response compared with untreated sample, and at 760 °C the main difference was the formation of a thin and defective layer of Cr₂O₃ at the coating surface, which increased the total impedance at the first day of immersion. Higher influence on the electrochemical was noted for samples treated at 880 °C, which also showed higher E_{OC} and total impedance, and lower corrosion current. This behavior was interpreted considering the formation of a chromium oxide layer on the coating surface, dissolution and decomposition of smaller carbide particles, and their surface enrichment with Cr due to C diffusion and dissolution into the matrix, and possible Ni, Cr, and Fe diffusion to coating/substrate interface.

Keywords: Auger electron spectroscopy, chromium compounds, electrochemical corrosion, electrochemistry, electrostatic coatings, heat treating furnaces, high-velocity oxyfuel, open-circuit potential, scanning electron microscopy, solutions, spectroscopy, substrate interfaces, thermal spray, velocity measurement, x-ray diffraction

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Influence of YSZ electrolyte thickness on the characteristics of plasma sprayed cermet supported tubular solid oxide fuel cell (SOFC). Novel Ni-Al₂O₃ cermet-supported tubular SOFC cell was fabricated by thermal spraying. Flame sprayed Al₂O₃-Ni cermet coating played dual roles of a support tube and an anode current collector. Y₂O₃-stabilized ZrO₂ (YSZ) electrolyte was deposited by atmospheric plasma spraying (APS) to aim at reducing manufacturing cost. The gas tightness of APS YSZ coating was achieved by postdensification process. The influence of YSZ coating thickness on the performance of solid oxide fuel cell (SOFC) test cell was investigated in order to optimize YSZ thickness in terms of open circuit voltage of the cell and YSZ ohmic loss. It was found that the reduction of YSZ thickness from 100 to 40 μm led to the increase of the maximum output power density from 0.47 to 0.76 W/cm² at 1000 °C. Using an APS 4.5 YSZ coating of about 40 μm as the electrolyte, the test cell presented a maximum power output density of more than 0.88 W/cm² at 1030 °C. The results indicate that SOFCs with thin YSZ electrolyte require more effective cathode and anode to improve performance.

Keywords: atmospheric plasma spraying, cermets, coatings, electrolyte thickness, electrolytes, geothermal springs, plasma spraying, solid oxide fuel cells, tubular cermet supporter, yttria-stabilized zirconia, zirconium compounds

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Microstructure and environmental functionalities of TiO₂-supported photocatalysts obtained by suspension plasma spraying. This paper deals with the elaboration of titanium dioxide coatings, designed for photocatalytic applications, obtained by a nonconventional method of deposition, suspension plasma spraying (SPS), which is an alternative of the atmospheric plasma spraying (APS) in which the material feedstock is a suspension of the material powder to be sprayed. TiO₂ P25 powder (Degussa AG) mechanically dispersed in distilled water and/or ethanol was injected in argon-hydrogen and argon-hydrogen-helium plasma under atmospheric conditions. Scanning electron microscopy and x-ray diffraction were performed to study the morphology and the crystalline phases of the titania coatings. Surface features were investigated by Fourier transform infrared (FT-IR) and x-ray photoelectron spectroscopy (XPS). Photocatalytic efficiency of the elaborated samples was tested in an environmental test chamber setup and evaluated from the conversion rate of nitrogen oxides. The results showed that the coating obtained from the injection of an alcoholic suspension contained 23% of anatase ratio and ensured a very low photocatalytic decomposition of nitrogen oxides. In contrast, the injection of an aqueous suspension into the plasma permitted to obtain deposits where the anatase phase and the crystallites size were preserved. Also, a conversion rate of the pollutants of about 40%, slightly better compared to that of the initial P25 powder (around 32%) was noticed. This slightly higher efficiency was

correlated with a cleaning of the particle surfaces when crossing the plasma and a higher hydroxylation of the coating surface.

Keywords: anatase ratio, atmospheric plasma spraying, Fourier transform infrared spectroscopy, hydroxylation, material feedstock, microstructure, nitrogen oxides, photocatalysis, plasma applications, scanning electron microscopy, spraying, suspension plasma spraying, titanium dioxide, x-ray diffraction, x-ray photoelectron spectroscopy

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Performance of thermal cells and batteries made with plasma sprayed cathodes and anodes. Cathodes for thermally activated ("thermal") batteries based on CoS₂ and LiCl-LiBr-LiF electrolyte and FeS₂ (pyrite) and LiCl-KCl eutectic were prepared by thermal spraying catholyte mixtures onto graphite-paper substrates. Composite separator-cathode deposits were also prepared in the same manner by sequential thermal spraying of LiCl-KCl-based separator material onto a pyrite-cathode substrate. These materials were then tested in single cells over a temperature range of 400 to 600 °C and in five-cell and 15-cell batteries. A limited number of battery tests were conducted with the separator-cathode composites and plasma sprayed Li(Si) anodes—the first report of an all-plasma-sprayed thermal battery. Thermal spraying offers distinct advantages over conventional pressed-powder parts for fabrication of thin electrodes for short-life thermal batteries. The plasma sprayed electrodes have lower impedances than the corresponding pressed-powder parts due to improved particle-particle contact.

Keywords: anodes, cathodes, electric batteries, electrodes, electrolytes, LiCl-KCl, LiCl-LiBr-LiF, mixtures, plasma spray, plasma spraying, thermal batteries

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Phase formation during deposition of TiO₂ coatings through high-velocity oxyfuel spraying. The crystalline structure of TiO₂ coating is of significant importance for controlling its property and performance, such as photocatalytic activity. The aim of this study is to examine the phase formation mechanisms during high-velocity oxyfuel (HVOF) spraying of TiO₂ coating. TiO₂ coatings were deposited under different spray conditions using both anatase powder and rutile powder as feedstocks. The results showed that the anatase content in the coatings was increased with the increase of fuel gas flow when using the rutile powder as feedstock. A TiO₂ coating of anatase content up to 35% can be obtained by well-melted particles. The high content of anatase phase possibly resulted from rapid solidification and cooling process of the particles. The anatase content in the TiO₂ coating deposited with an anatase powder in partially melted state reached 55% to 65%. The coating deposited by well-melted anatase powder contained the same anatase content as that by rutile powder. A model was proposed to explain the phase formation within the coatings deposited through HVOF spraying.

Keywords: catalyst activity, coatings, enzymes, high-velocity oxyfuels, phase formation, phase transitions, photocatalysis, photocatalytic activity, powders, rutile, spraying, titanium oxides

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Plasma erosion properties of ceramic coating prepared by plasma spraying. Application of plasma sprayed ceramic coatings with a high purity of more than 99.9% has been sharply increasing in semiconductor and liquid-crystal display (LCD) production equipments in the last few years. The size of the equipments becomes larger with increasing Si wafer size and the LCD size that promotes the replacement of conventional techniques, such as alumite film and bulk ceramics, to plasma spray coatings, where the high durability against the plasma erosion (anti-plasma-erosion resistance) is required. However, as far as we know, no systematic studies on the plasma-erosion properties are reported. In this work, durability of plasma sprayed alumina and yttria coatings against CF₄/O₂ plasma are investigated by reactive ion etching (RIE) system and are compared to that of the conventional techniques. The erosion mechanism and the effect of the powder properties are discussed through the microstructural analysis.

Keywords: alumina, ceramic coatings, liquid crystal displays, microstructure, plasma sprayed ceramic coatings, plasma spraying, plasma-erosion properties, reactive ion etching, semiconductor

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Preliminary study of performance of dye-sensitized solar cell of nano-TiO₂ coating deposited by vacuum cold spraying. The control of nanoporous microstructure of TiO₂ coating in the dye-sensitized solar cells (DSCs) through preparation methods influences significantly the performance of the cells. To investigate the effect of microstructure of TiO₂ deposit on the performance of the cells, in the present study, vacuum cold spray (VCS) process is employed to deposit nanocrystalline TiO₂ coatings on an ITO conductive glass substrate. TiO₂ deposits were produced using nanosized particles of 25 nm in diameter and composite powder composed of polyethylene glycol (PEG) and 25 nm TiO₂ particles. The deposition characteristics and the microstructure of the coating are characterized by scanning electron microscopy, x-ray diffraction analysis. The performance of the cell is tested under illumination of a metal halide lamp with a radiation intensity of 600 W/m². It was found that a dense TiO₂ coating with retention of crystal structure of powder can be deposited by vacuum cold spraying directly using nanosized TiO₂ powder. A cell with TiO₂ coating of 3.8 μm thick deposited directly by 25 nm powders yielded a short current density of 90.0 A/m² and conversion efficiency of 5.1%, which was comparable with that of the wet doctor blading TiO₂ coating. Using a composite powder of TiO₂ with PEG, a porous TiO₂ coating with a thickness up to several tens of micrometers can be deposited by vacuum cold spraying. A DSC cell of TiO₂ coating deposited using a composite powder containing 41.2% PEG presented a higher short current density of 145.0 A/m², and conversion efficiency of 7.1%.

Keywords: dyeing, dye-sensitized solar cell, halide lamp, nanostructured materials, nano-TiO₂ coating, polyethylene glycols, protective coatings, scanning electron microscopy, solar cells, titanium dioxide, vacuum cold spraying, vacuum-deposited coatings

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Material Synthesis

Ceramic foams directly-coated with flame-made V₂O₅/TiO₂ for synthesis of phthalic anhydride. Flame-made airborne V₂O₅/TiO₂ nanoparticles were deposited directly onto mullite foam supports to create ready-to-use catalysts for the *o*-xylene conversion to phthalic anhydride. These particles containing 10% (w/w) V₂O₅ were created by combustion of liquid precursor sprays and characterized by transmission electron microscopy, nitrogen adsorption, x-ray diffraction (XRD), temperature-programmed reduction (TPR), and Raman spectroscopy. The specific surface area, anatase content, and dominantly monomeric vanadia species on titania were thermally stable up to 450 °C. Catalyst structure was controlled in situ during deposition by the particle-laden gas flow rate through the foam, resulting in homogeneous to patchy V₂O₅/TiO₂ coatings. The catalytic activity and selectivity were affected by both coating texture and particle morphology. These flame coated foams showed superior catalytic activity and selectivity at high conversions than classic, wet-made V₂O₅/TiO₂ catalysts.

Keywords: carboxylic acids, catalyst activity, ceramic foams, deposition, direct deposition, flame-spray pyrolysis, nanostructured materials, partial oxidation, Raman spectroscopy, synthesis (chemical), temperature-programmed reduction, titanium oxides, vanadium compounds, x-ray diffraction analysis

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Coatings of metastable ceramics deposited by solution-precursor plasma spray: I. binary ZrO₂-Al₂O₃ system. We show that the solution-precursor plasma spray (SPPS) method can be used to deposit ~75% dense coatings of metastable ceramics in the ZrO₂-10 mol%Al₂O₃ binary system. The microstructure of the resulting SPPS coatings contains a rich variety of features and phases, as determined using x-ray diffraction, transmission electron microscopy, and energy dispersive x-ray spectroscopy. The SPPS coatings are predominantly nanostructured (grain size ≤100 nm), and made of tetragonal phase of ZrO₂ (*t*-ZrO₂) with Al³⁺ in solid solution. The Al³⁺ solute appears to stabilize partially the *t*-ZrO₂. These coatings also contain some grains of monoclinic (*m*-ZrO₂) and orthorhombic (*o*-ZrO₂) phases, and Al-rich grain-boundary amorphous phase. Crystalline Al₂O₃ phases are absent in the as-sprayed SPPS coatings. However, after a prolonged heat treatment (1400 °C, 30 h), αAl₂O₃ grains precipitate out in the coating, and the *t*-ZrO₂ grains coarsen and transform to *m*-ZrO₂.

Keywords: ceramic materials, energy dispersive spectroscopy, grains precipitate, metastable ceramics, nanostructured materials, plasma spraying,

solution-precursor plasma spray, sprayed coatings, tetragonal phase, transmission electron microscopy, x-ray diffraction analysis

A.L. Vasiliev, N.P. Padture, and X. Ma, Department of Materials Science and Engineering, Ohio State University, Columbus, OH 43210. Cited: *Acta Mater.*, 2006, Oct, 54(18), p 4913-4920. ISSN 1359-6454.

Coatings of metastable ceramics deposited by solution-precursor plasma spray: II. ternary ZrO₂-Y₂O₃-Al₂O₃ system. The solution-precursor plasma spray process has been used to deposit ~75% dense, metastable coatings in the following ternary systems (mol%): (a) 10Al₂O₃-86.4ZrO₂-3.6Y₂O₃ (10AlZrY) and (b) 20Al₂O₃-76.4ZrO₂-3.6Y₂O₃ (20AlZrY). The microstructures of these as-sprayed coatings consist of a rich variety of features and phases, as determined using x-ray diffraction, transmission electron microscopy, energy dispersive x-ray spectroscopy, and electron energy loss spectroscopy. The nanostructures (10 to 40 nm) in both coatings are primarily ZrO₂ in tetragonal (*t*-ZrO₂) form, with some cubic (*c*-ZrO₂) phase present. Crystalline Al₂O₃ phases are absent in the nanostructured regions of these coatings, and the chemical compositions of these regions are close to the respective nominal compositions of the coatings. These results show clearly that Al³⁺, in addition to Y³⁺, is in solid solution with ZrO₂, leading to the stabilization of *t*-ZrO₂ and *c*-ZrO₂. The submicrometer regions in these coatings, in addition to *t*-ZrO₂, contain small amounts of crystalline Al₂O₃ phases. Both coatings contain small amounts of spherical grains of orthorhombic ZrO₂ (*o*-ZrO₂). A heat treatment of 1400 °C for 30 h is insufficient to effect complete *t* → *m* transformation in ZrO₂. Heat treatment at 1500 °C (30 h) results in the precipitation of αAl₂O₃ and a new tetragonal form of Y₂O₃ in both coatings. This is accompanied by Al and Y depletion in and coarsening of the surrounding ZrO₂ grains, and complete *t* → *m* transformation.

Keywords: ceramic materials, electron energy loss spectroscopy, heat treatment, metastable ceramics, microstructure, nanostructured materials, plasma spraying, solid solutions, solution-precursor plasma spray, spherical grains, sprayed coatings, submicrometer regions, ternary systems, transmission electron microscopy, x-ray diffraction analysis, yttrium compounds, zirconia

A.L. Vasiliev and N.P. Padture, Department of Materials Science and Engineering, Ohio State University, Columbus, OH 43210. Cited: *Acta Mater.*, 2006, Oct, 54(18), p 4921-4928. ISSN 1359-6454.

Electron microscopy and EXAFS studies on oxide-supported gold-silver nanoparticles prepared by flame spray pyrolysis. Gold and gold-silver nanoparticles prepared by flame spray pyrolysis (FSP) were characterized by electron microscopy, in situ x-ray absorption spectroscopy (XANES and EXAFS), x-ray diffraction (XRD), and their catalytic activity in CO oxidation. Within this one-step flame-synthesis procedure, precursor solutions of dimethyl gold(III) acetylacetonate and silver(I) benzoate together with the corresponding precursor of the silica, iron oxide, or titania support, were sprayed and combusted. In order to prepare small metal particles, a low noble metal loading was required. A loading of 0.1 to 1 wt.% of Au and Ag resulted in 1 to 6 nm particles. The size of the noble metal particles increased with higher loadings of gold and particularly silver. Both scanning transmission electron microscopy (STEM) combined with energy dispersive x-ray spectroscopy (EDXS) and x-ray absorption spectroscopy (XAS) studies proved the formation of mixed Au-Ag particles. In case of 1%Au-1%Ag/SiO₂, TEM combined with electron spectroscopic imaging (ESI) using an imaging filter could be used in addition to prove the presence of silver and gold in the same noble metal particle. CO oxidation in the presence of hydrogen was chosen as a test reaction sensitive to small gold particles. Both the influence of the particle size and the alloying of gold and silver were reflected in the CO oxidation activity.

Keywords: electron microscopy, flame spray pyrolysis, flame spraying, gold alloys, gold-silver alloys, in situ processing, nanostructured materials, oxidation, pyrolysis, scanning electron microscopy, scanning transmission electron microscopy, transmission electron microscopy, x-ray diffraction

S. Hannemann, J.-D. Grunwaldt, F. Krumeich, P. Kappen, and A. Baiker, Institute of Chemical and Bioengineering, Swiss Federal Institute of Technology, ETH Honggerberg, CH-8093 Zurich, Switzerland. Cited: *Appl. Surf. Sci.*, 2006, Sept 15, 252(22), p 7862-7873. ISSN 0169-4332.

Flame-derived Pt/Ba/Ce_xZr_{1-x}O₂: Influence of support on thermal deterioration and behavior as NO_x storage-reduction catalysts. Pt/Ba catalysts for NO_x storage-reduction (NSR) supported on ceria/zirconia were prepared by two-nozzle flame spray pyrolysis. Emphasis was placed on the effect of the support composition on the thermal deterioration and the related behavior during NO_x storage and reduction. The materials were characterized by x-ray diffraction (XRD), nitrogen adsorption (BET), electron microscopy (TEM), and temperature-programmed decomposition (TPD). The as-prepared material consisted of intimately mixed agglomerates of BaCO₃ and Ce_xZr_{1-x}O₂ particles. Low thermal stability of BaCO₃ resulted in high NO_x storage capacity. The support composition (Ce_xZr_{1-x}O₂) strongly affected the NO_x

reduction activity of Pt. Higher Ce content favored the formation of stable Pt oxides exhibiting lower reduction activity. Thermal deterioration was investigated in different atmospheres. At 800 °C, BaCO₃ transformed into inactive Ba zirconate and Ba cerate. At high Ce content, BaCO₃ was reformed when exposed to CO₂ at high temperatures, recovering its high NO_x storage capacity. During the high-temperature treatment, however, Pt strongly deactivated, resulting in a loss of the catalyst NO_x reduction activity.

Keywords: adsorption, barium carbonate, barium cerate, barium zirconate, catalyst activity, flame synthesis, high-temperature applications, nitrogen oxides, NO_x storage reduction, platinum, pyrolysis, temperature programmed desorption, thermal deterioration, transmission electron microscopy, x-ray diffraction, zirconia

R. Strobel, F. Krumeich, S.E. Pratsinis, and A. Baiker, Institute for Chemical and Bioengineering, Department of Chemistry and Applied Biosciences, ETH Zurich, CH-8093 Zurich, Switzerland. Cited: *J. Catal.*, 2006, Oct 25, **243**(2), p 229-238. ISSN 0021-9517.

Flame-made Pt-Ba/Al₂O₃ catalysts: structural properties and behavior in lean-NO_x storage reduction. A series of Pt-Ba/Al₂O₃ catalysts with Ba loadings in the range of 4.5 to 33 wt.% was prepared using a two-nozzle flame spray pyrolysis method. The structural properties and activities of these catalysts in NO_x storage-reduction (NSR) were compared to corresponding standard Pt-Ba/Al₂O₃ catalysts prepared conventionally via wet impregnation. The catalysts were characterized by XRD, TEM, ESI, DRIFT, TG-MS, TPRD, and gas adsorption. Calcined flame-derived catalysts were composed of agglomerated spherical nanoparticles of alumina and Pt/barium carbonate of low thermal stability (LT-BaCO₃), whereas barium carbonate of high stability (HT-BaCO₃) was absent. In contrast, in the corresponding conventionally prepared catalysts, the Ba-containing phases were composed of BaO, LT-BaCO₃, and HT-BaCO₃. DRIFTS measurements of CO adsorption revealed distinct differences in the distribution of CO chemisorbing sites on Pt particles depending on the preparation method applied. The CO:Pt ratio varied between 0.8 and 0.7 depending on the Ba loading and the preparation method. Decreased Pt dispersion at higher Ba loadings was observed for the wet-impregnated catalysts, whereas Pt dispersion increased only slightly at higher Ba loadings for the flame-made catalysts. The structural differences of flame-made and conventionally prepared catalysts affected strongly their NO_x storage efficiency. Investigation of the behavior of the catalysts in NSR showed that the larger Ba/Al₂O₃ interface existing in conventionally prepared catalysts results in a higher NO_x storage efficiency of these catalysts at Ba loadings up to ~17 wt.%, whereas the absence of the HT-BaCO₃ phase in flame-made catalysts led to improved NO_x storage potential at higher Ba loadings.

Keywords: alumina, barium carbonate, barium nitrate, catalysts, mass spectrometry, platinum, pulse thermal analysis, pyrolysis, structure (composition), temperature programmed desorption, temperature programmed reaction-desorption, thermogravimetric analysis, transmission electron microscopy, x-ray diffraction analysis

M. Piacentini, R. Strobel, M. Maciejewski, S.E. Pratsinis, and A. Baiker, Institute for Chemical and Bioengineering, Department of Chemistry and Applied Bioscience, ETH Zurich H-8093 Zurich, Switzerland. Cited: *J. Catal.*, 2006, Oct 1, **243**(1), p 43-56. ISSN 0021-9517.

Microstructural evolution in spray formed and melt-spun Al₈₅Nd₅Ni₁₀ bulk hybrid composites. Microstructure evolutions in the spray-formed and melt-spun Al₈₅Nd₅Ni₁₀ alloys were studied. The spray forming process has shown to be capable of directly producing a bulk scale Al₈₅Nd₅Ni₁₀ hybrid composite consisting of amorphous and nanostructured phases without the need of using an amorphous precursor. The spray formed overspray flakes were partially amorphous, and the amorphous phase came from the undercooled liquid droplets upon deposition. The as-spray-formed deposit was completely crystallized due to the devitrification of the retained amorphous phase to nanoscale secondary crystals upon deposition. In the deposit and flake, the primary crystals were microscaled Al₆NdNi₂, and the secondary crystals devitrified from the amorphous phase during spray forming process were nanoscaled Al₃Ni and Al₆NdNi₂, dispersed in nanoscaled α Al matrix. The completely devitrified melt-spun ribbon composite consisted of secondary crystals Al₃Ni, Al₁Nd₃ and Al₆NdNi₂ with nanoscale sizes, dispersed in the nanoscaled α Al matrix. Deformation twins were observed in the primary Al₆NdNi₂ crystal in the as-spray-formed flake and deposit, but were not found in the secondary Al₆NdNi₂ crystals in the completely devitrified ribbon.

Keywords: amorphous precursor, composite materials, crystallization, crystals, intermetallics, liquid droplets, melt spinning, microstructure, microstructure evolutions, nanostructured materials, rapid solidification, spray forming, transmission electron microscopy, twinning

M.L.T. Guo, C.Y.A. Tsao, J.C. Huang, and J.S.C. Jang, Department of Materials Science and Engineering, National Cheng Kung University, 70101 Tainan, Taiwan. Cited: *Intermetallics*, 2006, Aug/Sept, **14**(8-9), p 1069-1074. ISSN 0966-9795.

Nano- α -Al₂O₃ by liquid-feed flame spray pyrolysis. The use of liquid-feed flame spray pyrolysis (LF-FSP) to convert nano-*t*-aluminas into dispersible 30 to 80 nm α -Al₂O₃ powders was investigated. The 1 to 10 wt.% nano-*t*-alumina powders were milled with alumina media in ethanol for 24 h, then ultrasonicated for 30 min with a 500 W titanium horn. The dispersion was aerosolized with O₂ and combusted at temperatures near 1600 °C. The resultant powders exhibited XRD powder patterns that indicate a high degree of conversion to mixtures of θ - and α -Al₂O₃, with the gibbsite-derived powders having the lowest conversions at roughly 50%. The initial presence of θ - and traces of α -Al₂O₃ in the LF-FSP *t*-aluminas shows the higher α conversion for these powders.

Keywords: Al₂O₃, alcohols, alumina, aluminum compounds, derived powders, liquid feed flame spray pyrolysis, nanostructured materials, powder metals, pyrolysis, titanium compounds, ultrasonication, x-ray powder diffraction

R.M. Laine, J.C. Marchal, H.P. Sun, and X.Q. Pan, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI 48109-2136. Cited: *Nature Mater.*, 2006, Sept 3, **5**(9), p 710-712. ISSN 1476-1122.

Nano α -Fe/epoxy resin composite absorber coatings fabricated by thermal spraying technique. In this paper, the low-temperature high-velocity air fuel (LTHVAF) spraying technique was applied to prepare the α -Fe/epoxy resin nanocomposite coatings. The composite powders were mixed with different mass fractions, and the microstructure and reflectivity coefficient of coatings were tested. The results show that the microstructure of coatings is dense and low porosity; nanometal particles are dispersed in the coatings. The coatings are closely combined with substrate. In these coatings, the volume fraction calculated with density, component distribution, properties of metal particle, and coating thickness can affect the microwave absorption ability of the coatings. The reflectance coefficient of 70 mass% nano α -Fe/epoxy resin composite coatings is lower than others. In these nanocomposite absorber coatings, the relationship of the reflectivity coefficient and the coating structure were constructed with permittivity, permeability, and thickness. The optimal mass fraction of absorber coatings is about 0.3. It is analyzed with self-bonding strength and reflectivity coefficient. This means that the change of the coating structure affects the performance of the nanocomposite coatings.

Keywords: composite materials, epoxy resins, mechanical permeability, microwave absorber coatings, nanocomposite coatings, nanostructured materials, permittivity, protective coatings, reflection, self-bonding strength, thermal spraying

X. Yuan, H. Wang, G. Hou, L. Jiang, and C. Yao, Mechanical Electric Engineering, Xi'an Research Institute of Hi-Tech, Hongqing, Xi'an, Shaanxi 710025, China. Cited: *IEEE Trans. Magn.*, 2006, Sept, **42**(9), p 2115-2120. ISSN 0018-9464.

Preparation and evaluation of ordinary attritor-milled Ti-Al powders and corresponding thermal sprayed coatings. Ordinary attritor milling of elemental metallic powders under atmospheric condition was utilized to prepare desirable amount of powders for thermal spraying. The effect of different BPR (ball-to-powder weight ratio) has been investigated in terms of nitridation during milling. To investigate the effect of heat treatment on the formation of dispersed phases, heat treatment to the powder was performed as well. Titanium aluminide coatings with carbonitride-dispersed phases were successfully fabricated by low-pressure plasma spraying. The hardness and specific wear of the coatings prepared by the powders with different milling conditions was measured so as to investigate the effect of the content of dispersed titanium-base carbonitride phases. Experimental results show that the formation of dispersed carbonitride phases depends strongly on milling condition, irrespective of heat treated powders or thermal sprayed coatings, and directly affects the mechanical properties of the coatings. Compared with the phase composition of heated powders and corresponding thermal sprayed coatings, it seems that the temperature of processing the MA powders is also a decisive factor on the phase formation, especially carbonitride phases and oxide phase.

Keywords: hardness, heat treatment, low-pressure plasma spraying, milling condition, milling (machining), ordinary attritor mill, phase composition, plasma spraying, powder metals, specific wear, sprayed coatings, TiAl coatings, titanium compounds, titanium-base carbonitride, wear of materials

M. Fukumoto, M. Nie, and T. Yasui, Department of Production Systems Engineering, Toyohashi University of Technology, Toyohashi 441-8580, Japan. Cited: *Mater. Trans.*, 2006, July, **47**(7), p 1717-1722. ISSN 1345-9678.

Preparation by flame spray pyrolysis of ABO_{3 Δ} catalysts for the flameless combustion of methane. Perovskitic mixed oxides prepared through flame spray pyrolysis possess a good stability in high-temperature application, namely, the catalytic flameless combustion of methane. Some preparation operating parameters are here analyzed, such as O₂ pressure drop along the spraying nozzle, O₂ discharge velocity and flow rate. These parameters have been correlated with specific surface area, activity, and durability of the prepared samples, as well as with flame temperature, varied by using different fuel mixtures. It was found that specific surface area

increases with increasing O₂ velocity and flow rate and with decreasing the combustion enthalpy of the solvent mixture. This reflects on both activity and durability of the catalyst.

Keywords: catalysts, catalytic flameless combustion of methane, combustion, combustion enthalpy, enthalpy, flame spray pyrolysis, flame spraying, high-temperature effects, methane, perovskite, perovskitic mixed oxides, pyrolysis
G.L. Chiarello, I. Rossetti, P. Lopinto, G. Migliavacca, and L. Forni, Dip. di Chimica Fisica ed Elettrochimica, Università degli Studi di Milano, I-20133 Milano, Italy. Cited: *Catal. Today*, 2006, Oct 15, **117**(4), p 549-553. ISSN 0920-5861.

Protective magnesia coating on Y₂O₃:Eu phosphor powders. Protective magnesia coating on Y₂O₃:Eu phosphor powders is formed by a layer-by-layer (LbL) method in aqueous solutions. The phosphor powders are first coated with a negatively charged, anionic polyelectrolyte of ammonium salt of poly(acrylic acid) (PAA-NH₄), on which a second-layer, positively charged magnesium hydroxide coating is then deposited by precipitation and heterocoagulation. A uniform and multiple Mg(OH)₂/PAA-NH₄ bilayer coating on the phosphor powder is prepared by repeating the above coating processes. Protective magnesia coating on the phosphor powders, which is evidenced by insignificant degradation in optical properties after an extended period of electron bombardment, is formed by calcining.

Keywords: anionic polyelectrolytes, electron bombardment, electron irradiation, magnesia, magnesium hydroxide, phosphor powders, phosphors, polyelectrolytes, precipitation (chemical), protective coatings, yttrium compounds
C.-C. Chung and J.-H. Jean, Department of Materials Science and Engineering, National Tsing Hua University Hsinchu, Taiwan, Taiwan. Cited: *J. Am. Ceram. Soc.*, 2006, Sept, **89**(9), p 2726-2730. ISSN 0002-7820.

Sintering behavior of in situ cobalt oxide doped cerium-gadolinium oxide prepared by flame spray pyrolysis. Ce_{0.9}Gd_{0.1}O_{1.95} (CGO10) and in situ cobalt oxide doped CGO10 were prepared by pilot-scale flame spray synthesis, yielding powders with an average particle size of 40 nm. Cobalt oxide was shown to be a very effective sintering aid for CGO10 and lowered the maximum sintering temperature from 1450 to about 1200 °C. Sintering studies revealed that in situ cobalt oxide doped CGO10 exhibited a temperature of maximum shrinkage rate of 880 °C for a dopant concentration of 1 mol% CoO_{1-x}, whereas for conventionally cobalt oxide doped CGO10, this temperature was 914 °C. This decrease is believed to be a result of a more homogeneous dopant distribution of the in situ cobalt oxide doped CGO nanopowders as compared with the powders in which the doping was introduced as nitrates.

Keywords: cerium compounds, cerium-gadolinium oxide, cobalt compounds, cobalt oxide, dopant distribution, doping (additives), flame spraying, nanopowders, nitrates, particle size analysis, pyrolysis, shrinkage, sintering, synthesis (chemical)

E. Jud, L. Gauckler, S. Halim, and W. Stark, ETH Zurich, Department of Materials Science, CH-8093 Zurich, Switzerland. Cited: *J. Am. Ceram. Soc.*, 2006, Sept, **89**(9), p 2970-2973. ISSN 0002-7820.

Spray pyrolysis of electrolyte interlayers for vacuum plasma sprayed SOFC. The effects of gadolinia-doped ceria (CGO, Ce_{0.8}Gd_{0.2}O_{1.9-x}) and yttria-doped zirconia (8YSZ, Zr_{0.92}Y_{0.08}O_{2-x}) interlayers prepared by spray pyrolysis between vacuum plasma sprayed 8YSZ electrolytes (8YSZ-VPS) and screen-printed (La_{0.8} Sr_{0.2})_{0.98}MnO₃ cathodes (LSM) on the power output of solid oxide fuel cells (SOFC) are investigated. Amorphous thin films are deposited and then converted to nanocrystalline electrolyte-cathode interlayers during the first heat-up cycle of a SOFC to the operating temperature. CGO thin films between the YSZ plasma sprayed electrolyte and the LSM cathode increased the power output by more than 20% compared to cells without interlayers, whereas YSZ films degraded the power output of cells. It is assumed that CGO improves the charge transfer at the electrolyte-cathode interface and that the CGO layer prevents the formation of undesirable insulation of La-zirconate at the interface 8YSZ/LSM.

Keywords: amorphous thin films, cathodes, electrolytes, gadolinium compounds, nanocrystalline electrolyte-cathode interlayer, nanostructured materials, plasma sprayed electrolytes, pyrolysis, solid oxide fuel cells, thin films, vacuum plasma spray

A.O. Stoermer, J.L.M. Rupp, and L.J. Gauckler, BMW Group, Future Powertrain Technology Projects, 80788 Munich, Germany. Cited: *Solid State Ionics*, 2006, Oct 15, **177**(19-25 SPEC. ISS.), p 2075-2079. ISSN 0167-2738.

Synthesis and characterization of mixed-metal oxide nanopowders along the CoO_x-Al₂O₃ tie line using liquid-feed flame spray pyrolysis. We report here the use of liquid-feed flame spray pyrolysis (LF-FSP) to produce a series of nanopowders along the CoO_x-Al₂O₃ tie line. The process is a general aerosol combustion synthesis route to a wide range of lightly agglomerated oxide nanopowders. The materials reported here were produced by aerosolizing ethanol solutions of alumatrane [Al(OCH₂CH₂)₃N]

and a cobalt precursor, made by reacting Co(NO₃)₂·6H₂O crystals with propionic acid. The compositions of the as-produced nanopowders were controlled by selecting the appropriate ratios of the precursors. Nine samples with compositions (CoO)_y(Al₂O₃)_{1-y}, Y = 0 to 1 along the CoO_x-Al₂O₃ tie line were prepared and studied. The resulting nanopowders were characterized by x-ray fluorescence, BET, scanning electron microscopy, high-resolution transmission electron micrographs, x-ray diffraction (XRD), thermogravimetric analysis (TGA), and FTIR. The powders typically consist of single-crystal particles less than or equal 40 nm diameter and specific surface areas (SSAs) of 20-60 m²/g. XRD studies show a gradual change in powder patterns from δ-Al₂O₃ to Co₃O₄. The cobalt aluminate spinel phase is observed at stoichiometries (21 and 37 mol%) not seen in published phase diagrams, likely because LF-FSP processing involves a quench of ≥1000 °C in microseconds frequently leading to kinetic rather than thermodynamic products. Likewise, the appearance of Co₃O₄ rather than CoO as the end member in the tie line is thought to be a consequence of the process conditions. TGA studies combined with diffuse reflectance FTIR spectroscopic studies indicate that both physi- and chemisorbed H₂O are the principal surface species present in the as-processed nanopowders. The only sample that differs is Co₃O₄, which has some carbonate species present that are detected and confirmed by a sharp mass loss event at ~250 °C. The thermal behavior of the high cobalt content samples differs greatly from the low cobalt content samples. The latter behave like most LF-FSP-derived nanopowders exhibiting typical 1 to 4% mass losses over the 1400 °C range due mostly to loss of water and some CO₂. The high cobalt content samples exhibit a sharp mass loss event that can be attributed to the decomposition of Co₃O₄ to CoO.

Keywords: agglomeration, alumina, cobalt compounds, ethanol, Fourier transform infrared spectroscopy, metal oxides, nanopowders, nanostructured materials, propionic acid, pyrolysis, synthesis (chemical), thermogravimetric analysis, x-ray diffraction, x-ray fluorescence

J. Azurdia, J. Marchal, and R.M. Laine, Departments of Materials Science and Engineering, Macromolecular Science and Engineering Center, University of Michigan, Ann Arbor, MI 48109-2136. Cited: *J. Am. Ceram. Soc.*, 2006, Sept, **89**(9), p 2749-2756. ISSN 0002-7820.

TiO-base coatings prepared by plasma spraying in air of Ti + C mixtures. This work presents an original way for preparing TiO-base coating by thermal spraying. As titanium monoxide is oxidized by the mere trace of oxygen contained in hydrogen, it is obvious that plasma spraying of TiO powder does not lead easily to the formation of a titanium monoxide coating. However, thermodynamic calculations show that the conditions necessary for the preparation of TiO can be reached, at the titanium melting temperature (T_m = 1933 K), when oxygen reacts with liquid metal in the presence of excess carbon. These results have led to experiments in which TiO-base coatings have been prepared by spraying a stoichiometric mixture of graphite and titanium grains onto cast iron in air. In optimal conditions, a gastight hard coating (1000 ± 15 HV_{0.05}) of a TiO-base phase (composition: TiO_{0.81±0.06}Co_{0.04±0.02}) has been obtained.

Keywords: cast iron, gastight hard coating, graphite, liquid metals, melting, oxidation, phase composition, plasma spraying, stoichiometry, thermal spraying, thermodynamics, titanium monoxide coating, titanium oxides

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Titania and titania-silver nanoparticle deposits made by liquid flame spray and their functionality as photocatalyst for organic and biofilm removal. Titania and titania-silver nanoparticle deposits were made by liquid flame spray technique, in which the liquid precursor is injected into a high-temperature flame, where it will evaporate and nucleate to nanosize particles. One-step and two-step methods were used for preparation of titania-silver deposits. The amount of silver added was 1 wt.%. The deposits were collected in the flame zone on steel and glass surfaces and were analyzed by TEM, EDS, XPS, and SAXS. The titania deposits consisted of porous nanosized titania agglomerates of primary particles (~10 nm). With silver addition, small spherical silver metal particles (~2 nm) were detected on the agglomerates. An increase in the photocatalytic activity was verified by stearic acid decomposition and biofilm removal using *Deinococcus geothermalis* as the model organism.

Keywords: agglomeration, biofilm removal, biofilms, decomposition, energy-dispersive spectroscopy, liquid flame spray, nanostructured materials, photocatalysis, photocatalytic activities, titania-silver deposits, titanium alloys, titanium deposits, transmission electron microscopy, x-ray photoelectron spectroscopy

H. Keskinen, J.M. Makela, M. Aromaa, J. Keskinen, S. Areva, C.V. Teixeira, J.B. Rosenholm, V. Pore, M. Ritala, M. Leskela, M. Raulio, M.S. Salkinoja-Salonen, E. Levanen, and T. Mantyla, Institute of Physics, Tampere University of Technology, FIN-33101 Tampere, Finland. Cited: *Catal. Lett.*, 2006, Nov, **111**(3-4), p 127-132. ISSN 1011-372X.

Mechanical Behavior and Characterization

The bond strength of Al-Si coating on mild steel by kinetic spraying deposition. Kinetic spraying (or cold gas dynamic spraying) works by accelerating small solid particles to supersonic velocities and then impacting them onto a substrate. These high-impact velocities and low particle temperatures are the principal attributes of kinetic spraying technology. However, only recently has the interfacial behavior of this technology, due to particle/substrate impaction, become well understood. In order to investigate the particle/substrate bond behavior, Al-Si feedstock was deposited onto mild steel, over a range of particle velocities; next, their respective coating bond strengths were measured by the stud pull coating adherence test. The effects of the particle velocity and the substrate surface roughness on the coating bond strength were presented, and a model of the particle/substrate bond generation was discussed in an effort to estimate the bond strength.

Keywords: acceleration, aluminum, bond strength, carbon steel, coatings, cold gas dynamic spraying, deposition, high-velocity impact, kinetic spraying, silicon, spraying, velocity control

J. Wu, J. Yang, H. Fang, S. Yoon, and C. Lee, State Key Lab of Advanced Welding Production Technology, Harbin Institute of Technology, Harbin, Heilongjiang 150001, China. Cited: *Appl. Surf. Sci.*, 2006, Sept 15, 252(22), p 7809-7814. ISSN 0169-4332.

Ceramic coatings: Effect of deposition method on damping and modulus of elasticity for yttria-stabilized zirconia. We have studied the mechanical damping and elastic stiffness of ceramic coatings, of yttria-stabilized zirconia, deposited by air plasma spraying (APS) or electron beam-physical vapor deposition (EB-PVD) on Inconel beams. The loss factor and Young's modulus of the coating have been estimated as a function of strain amplitude with the aid of a numerical analysis. It has been found that the coating produced by APS has higher damping, lower elastic stiffness, and stronger amplitude dependence than that produced by EB-PVD. These features are correlated with the microstructure examined by scanning electron microscopy.

Keywords: air plasma spraying, amplitude dependence, ceramic coatings, crystal microstructure, damping, elastic moduli, elastic stiffness, electron beam-physical vapor deposition, numerical analysis, physical vapor deposition, plasma spraying, scanning electron microscopy, stiffness, yttria-stabilized zirconia, yttrium compounds, zirconia

S. Patsias, N. Tassini, and K. Lambrinou, Department of Mechanical Engineering, University of Sheffield, Sheffield, S1 3JD, U.K. Cited: *Mater. Sci. Eng. A*, 2006, Dec 20, 442(1-2 SPEC. ISS.), p 504-508. ISSN 0921-5093.

Ceramic coatings: A phenomenological modeling for damping behavior related to microstructural features. Recent research has shown that both stiffness and damping of ceramic coatings exhibit different nonlinearities. These properties strongly depend on the microstructure, which is characterized by heterogeneous sets of elastic elements with mesoscopic sizes and shapes, as in nonlinear mesoscopic elastic materials. To predict the damping properties of this class of materials, we have implemented a phenomenological model that characterizes their elastic properties. The model is capable of reproducing the basic features of the observed damping behavior for zirconia coatings prepared by air plasma spraying and electron beam-physical vapor deposition.

Keywords: ceramic coatings, damping, elastic elements, elasticity, mathematical models, nonlinear mesoscopic elastic materials, stiffness

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Characterization and erosion behavior of plasma sprayed NiCrAlY and Ni-20Cr coatings on an Fe-based superalloy. Degradation of materials due to solid particle erosion is encountered in a variety of engineering industries, either at room temperature or elevated temperatures. Nickel-base coatings are commonly used in applications where wear resistance, combined with oxidation or hot corrosion resistance, is required. In the present work, NiCrAlY and Ni-20Cr metallic coatings were deposited on an iron-base superalloy by a shrouded plasma spray process. The coatings were characterized by scanning electron microscopy, optical microscopy, microhardness testing, and x-ray diffractometry. Erosion studies were conducted using an air-jet erosion test rig at a velocity of 40 m/s⁻¹ and impingement angles of 30° and 90°. Scanning electron microscopy was used to analyze the eroded surfaces. Three-dimensional surface roughness profiles of the eroded samples were taken using a Veeco Optical Profilometer. NiCrAlY coatings had slightly lower average porosity and lower microhardness as compared to Ni-20Cr coatings. The observed erosion rate of the NiCrAlY coatings, however, was lower than that of the Ni-20Cr coatings at both 30° and 90° impingement angles. Ni-20Cr coating had shown higher erosion rate at 90° impingement angle than that at 30°, whereas the effect of impingement angle

on the erosion rate is negligible for plasma sprayed NiCrAlY coating. The higher bond strength of NiCrAlY coating might be one of the major contributing factors for lower erosion rate of NiCrAlY coating as compared to Ni-20Cr coating under the tested conditions. Erosion mechanisms of plasma sprayed coatings are discussed.

Keywords: erosion, erosion mechanism, microhardness, oxidation resistance, plasma spray coatings, plasma spraying, porosity, scanning electron microscopy, solid particle erosion, sprayed coatings, surface roughness, tribology, wear of materials, wear resistance

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Composite damping ceramic coatings by polymer impregnation. The interest in the use of thermal barrier ceramic coatings as damping treatments has recently arisen since they were shown to provide sufficient additional damping. Ceramic coatings can be applied mainly by either air plasma spraying (APS) or electron beam-physical vapor deposition (EB-PVD). APS was used to deposit the specimens studied here. Previous research has shown that it is possible to enhance their damping effectiveness by tailoring the deposition parameters. This paper presents further work aimed at improving their damping effectiveness significantly by introducing a second material in the ceramic coating, thus creating a composite system. This paper covers the experimental findings of impregnating a ceramic damping coating with a number of commercially available polymeric materials. The most promising case of a type of polyurethane provided more than 40% improvement and was selected for further tests at various temperatures, ranging from -40 to 150°C. Furthermore, the damping and stiffness of ceramic coatings present amplitude-dependent behavior, which was also taken into consideration when evaluating the composite system. Finally, the paper concludes with a discussion of these promising findings as well as planned research into understanding the physical mechanisms dissipating energy in this composite material.

Keywords: air plasma spraying, ceramic coatings, composite coating, damping, electron beam-physical vapor deposition, electron beams, energy dissipation, impregnation, physical vapor deposition, plasma spraying, polymer impregnation and damping ceramic coatings

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Development of lateral compression method of circular tube thin coating for mechanical properties of plasma sprayed CoNiCrAlY. CoNiCrAlY coatings have been developed to protect gas turbine blades from oxidation and corrosion at high temperature. However, the mechanical properties of the thin CoNiCrAlY coatings used in actual turbine system were unknown, because there was not a proper measurement method of thin-sprayed coatings. Lateral compression test for the circular tube coatings of plasma sprayed thin coating was newly developed. In considering the deformation of the tubes, the elastic contact with a flat plate jig and a tube surface was taken into account as well as the bending of the curved beam of the tube. Young's moduli of well-known materials obtained from the lateral compression tests agreed well with the true values. CoNiCrAlY tube specimens independent of substrates were manufactured by dissolving out the substrates by nitric acid. The thickness of coatings were selected from 150 to 700 μm. The effects of spraying particle size, spraying atmosphere, and thermal treatment on mechanical properties, such as Young's modulus and bending strength, of CoNiCrAlY were examined. It was found that Young's modulus and a fracture stress increased with an increase of the coating thickness. Young's modulus was sensitive to the spraying powder size and increased with a decrease of the size. It was found that spraying with small spraying powder was the most effective in increasing the Young's modulus. It was also found that Young's modulus was governed by the porosity if thickness and process were given. It was found that thermal treatment was the most effective in increasing the bending strength.

Keywords: cobalt compounds, CoNiCrAlY, deformation, elastic moduli, fracture, fracture stress, high-temperature effects, lateral compression, plasma spraying, protective coatings

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Effect of spray distance on the mechanical properties of plasma sprayed Ni-45Cr coatings. Ni-45Cr coating was plasma sprayed at different spray distances in order to clarify the influence of spray distance on the mechanical properties. The mechanical properties of Ni-45Cr coatings include the fracture toughness, elastic modulus, and Poisson's ratio parallel to lamellar plane. The fracture toughness of plasma sprayed Ni-45Cr coating in terms of critical strain energy release rate (G_{IC}) was investigated using a tapered double cantilever beam approach. The elastic modulus and Poisson's ratio parallel to lamellar

plane, were obtained by measuring the relation of stress and strain using strain gages. It was found that the fracture toughness of plasma sprayed Ni-45Cr coatings was not significantly influenced by spray distance up to 190 mm and further increase in spray distance to 210 mm resulted in an evident decrease in the fracture toughness of the coatings. Moreover, spray distance showed minor effect on the elastic modulus and Poisson's ratio parallel to lamellar plane of Ni-45Cr coating. The examination of fractured surface revealed that the fracture toughness of plasma sprayed Ni-45Cr coatings is related with the lamellar bonding.

Keywords: cantilever beams, elastic moduli, fracture toughness, lamellar bonding, nickel chromium, plasma spraying, Poisson ratio, strain energy, strain gages, thermal barrier coatings

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Fabrication and evaluation of D-gun sprayed WC-Co coating with self-lubricating property. A WC-Co coating with self-lubricating property was deposited by detonation gun (D-gun) process, using a commercial WC-Co powder doped with a MoS₂-Ni powder, under a proper spray condition. It is proved that the MoS₂ composition in the feed powder was kept, which is attributed to the protection of Ni around it, and its content is a little higher in the resulting coating. Evaluation on sliding wear property indicates that the MoS₂ composition plays an important role in lowering both coefficient of friction and wear rate for the resulting coating, which is confirmed by observations on wear track, as well as X-ray photoelectron spectroscopy (XPS) results on worn surface. It suggests that the deposition of WC-Co coating with self-lubricating property by D-gun spray is feasible by controlling lubricant powder and spray conditions, which can exhibit higher sliding wear resistance.

Keywords: coefficient of friction, detonation gun, friction, self-lubricating composites, sliding wear property, sprayed coatings, tungsten carbide, wear of materials, wear rate, wear resistance

H. Du, C. Sun, W.G. Hua, Y.S. Zhang, Z. Han, T.G. Wang, J. Gong, and S.W. Lee, Division of Surface Engineering of Materials, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China. Cited: *Tribol. Lett.*, 2006, Sept, **23**(3), p 261-266. ISSN 1023-8883.

High-temperature gas nitridation of thermally sprayed coatings of Ti and Ti containing NiAl5Mo. Titanium and a mixture of 1:1 weight ratio titanium and bond coat (Ni-5 wt.%Al-5 wt.%Mo) were thermally sprayed on mild steel disks that were subsequently nitrided in 10 cm³/s ammonia at 1200 K to form titanium nitride(s). The coatings were analyzed using XRD and EDX to determine phases and elements contained in the phases. Knoop hardness (HK) values of the Ti and the mixture coatings at 25 μm depth with 1200 K nitridation are 1216.2 ± 112.5 and 1124.9 ± 221.8 kg/mm², respectively. Pin-on-disk wear rates of the coatings with 1200 K nitridation are three orders of magnitude higher than those of the tungsten carbide pin.

Keywords: carbon steel, high-temperature applications, high-temperature gas nitridation, Knoop hardness, mild steel disks, nitriding, sprayed coatings, thermally sprayed coatings, titanium, titanium compounds, titanium nitride, tungsten carbide

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Microstructure and tribological properties of plasma-sprayed nanostructured sulfide coating. The friction and wear properties of plasma sprayed nanostructured FeS coating were investigated on an MHK-500 friction and wear tester under both oil lubrication and dry friction condition. The microstructure, worn surface morphology, and phase composition of the coating were characterized by scanning electron microscopy (SEM) and x-ray diffraction (XRD). It was found that the coating was mainly composed of FeS, a small quantity of Fe_{1-x}S and oxide were also found. The coating was formed by small particles of 50-100 nm in size. The thickness of the coating is approximately 150 μm. The friction-reduction and wear-resistance properties of plasma sprayed nanostructured FeS coating were superior to that of GCr15 steel substrate. Especially under oil lubrication condition, the friction coefficient of nanostructured FeS coating was 50% of that of GCr15 steel, the wear scar widths of the coating were also reduced to nearly 50% of that of GCr15 steel under high load. The failure of the coating was mainly attributed to plastic deformation under both oil lubrication and dry friction condition.

Keywords: characterization, coatings, composition, dry friction, friction, friction reduction, microstructure, morphology, nanostructured iron sulfide, nanostructured materials, oil lubrication, plasma spraying, sulfur compounds, thickness, wear of materials, wear resistance properties

Y. Xu, Y. Guan, Z. Zheng, and X. Tong, College of Engineering, China Agricultural University, Beijing 100083, China. Cited: *J. Mater. Sci. Technol.*, 2006, Sept, **22**(5), p 589-593. ISSN 1005-0302.

Sliding wear characteristics of plasma sprayed 8%Y₂O₃-Zr coating for postspray heat treatment. The purpose of this study is to investigate the wear characteristics of yttria-stabilized zirconia coating as a function of effect on postspray heat treatment. The authors are concerned that the residual stress is due to during cooling from a high deposition temperature. They focus on the tribological wear performance as a magnitude of residual stress. The effect of residual stress as postspray heat treatment temperature is discussed. The plasma sprayed 8%Y₂O₃-zirconia coating is studied to know the relationship between phase transformations and wear properties after postspray heat treatment. Wear tests are carried out with ball-on-disk type at 50, 70, and 90 N at room temperature. The transformation of phase and magnitude of residual stress are measured by x-ray diffraction method (XRD). Tribological characteristics and wear mechanism for postspray heat treatment is observed by SEM.

Keywords: deposition, deposition temperature, heat treatment, oxides, phase transitions, plasma spraying, postspray heat treatment, sprayed coatings, tribological characteristics, tribology, wear of materials, x-ray diffraction analysis, yttrium compounds

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Surface modification of Cr₃C₂-NiCr cermet coatings by direct diode laser. Thermal spraying has emerged as an important tool of increasingly sophisticated surface engineering technology, and it is being used widely to repair and surface modification in metallic parts. The Cr₃C₂-NiCr sprayed coatings are frequently used as wear-resistant coatings against abrasion and erosion at high temperature up to 1173 K, and in corrosive environments. Hardness and microstructure of Cr₃C₂-NiCr cermet coatings fused by direct diode laser process was compared with that formed by high-velocity oxygen fuel spraying (HVOF) process. The effect of beam characteristics (power density, power, scanning speed, etc.) was examined on the surface modification of sprayed coatings. In this study, we treated Cr₃C₂-25%NiCr cermet coatings by laser irradiation process and examined its hardness compared with that formed by HVOF process. Consequently, the average hardness of laser-treated Cr₃C₂-25%NiCr cermet coatings has been found out to be higher than that of HVOF coatings. Laser remelting improved markedly the wear resistance of HVOF sprayed Cr₃C₂-25%NiCr cermet coatings.

Keywords: cermet coatings, chromium, Cr₃C₂NiCr cermet, direct diode laser, erosion, hardness, high-velocity oxygen fuel spraying, irradiation, microstructure, nickel compounds, semiconductor lasers, surface engineering, thermal effects, wear resistance

J. Morimoto, Y. Sasaki, S. Fukuhara, N. Abe, and M. Tukamoto, Faculty of Science and Technology, Kinki University, Osaka, 577-8502, Japan. Cited: *Vacuum*, 2006, Sept 7, **80**(11-12), p 1400-1405. ISSN 0042-207X.

Tensile and high-cycle fatigue properties of spray formed Al10.8Zn2.9Mg1.9Cu alloys after two-stage aging treatment. Based on the investigation of the tensile properties of spray formed ultrahigh-strength Al10.8Zn2.9Mg1.9Cu alloys, the high-cycle fatigue properties under different theoretical stress concentration factors were investigated, the fatigue fracture surfaces and microstructures were observed, and the fatigue mechanism was discussed. The results indicate that the ultimate tensile strength of spray formed Al10.8Zn2.9Mg1.9Cu alloys can reach up to 730 to 740 MPa, and the elongation is about 8 to 10% under the condition of two-stage aging treatment. For the stress ratio is 0.1, the maximum stress for 10⁷ cycles is more than 400 and 120 MPa, when the theoretical stress concentration factor is 1 and 3, respectively.

Keywords: aging of materials, Al10.8Zn2.9Mg1.9Cu alloy, aluminum alloys, copper alloys, crack initiation, fatigue of materials, fracture, high-cycle fatigue properties, magnesium alloys, microstructure, spray forming, spraying, stress concentration, tensile properties, tensile strength, two-stage aging treatment, ultrahigh-strength aluminum alloy, zinc alloys

Z.-X. Wang, Y.-A. Zhang, B.-H. Zhu, H.-W. Liu, F. Wang, and B.-Q. Xiong, State Key Laboratory for Fabrication and Processing of Nonferrous Metals, General Research Institute for Nonferrous Metals, Beijing 100088, China. Cited: *Trans. Nonferrous Met. Soc. China (English Edition)*, 2006, Aug, **16**(4), p 808-812. ISSN 1003-6326.

Wear behavior of low-pressure plasma sprayed AlCuFe quasi-crystalline coating on titanium alloy. The wear-resistant AlCuFe quasi-crystalline coating was fabricated on substrate of titanium alloy by low-pressure plasma spraying (LPPS) method. The LPPS AlCuFe quasi-crystalline coating has a rapidly solidified lamellar microstructure consisting of mainly icosahedral phase and small amount of cubic phase peaks. The results showed that AlCuFe quasi-crystalline coating improved the wear resistance of titanium-base alloys under dry sliding wear test conditions. The excellent wear

resistance may be attributed to the high hardness of AlCuFe quasi-crystal and the formation of the tribofilm.

Keywords: AlCuFe quasi-crystalline coating, aluminum compounds, coatings, low-pressure plasma spraying, microstructure, plasma spraying, quasi-crystals, titanium alloys, tribofilm, wear of materials, wear resistance

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Wear behavior of thermally sprayed coatings under different loading conditions. Wear behavior of three kinds of thermally sprayed coatings with similar hardness have been investigated under steady-state and dynamic loading tests. The steady-state loading tests were conducted on a reciprocating sliding device, and the dynamic loading tests were conducted with a single-pendulum scratching device. Experimental results show that the wear mechanisms of the coatings under steady-state sliding friction testing are microcutting and microplowing, whereas the material losses under the dynamic impact scratch testing are mainly due to split cutting and fracture. Tribo-oxidization in the sliding process was found to have an influence on the wear behaviors of the thermally sprayed coatings. The results also indicated that wear resistance of thermally sprayed coatings can be correlated to hardness, plasticity, toughness, and cohesion. As far as the coatings of similar hardness were concerned, the wear resistance under steady-state loading was mainly due to the cohesion of the laminar structure of the coatings and the wear resistance under dynamic loading was mainly due to the toughness and deformation compatibility of the coatings.

Keywords: dynamic loads, friction, materials science, reciprocating sliding device, single-pendulum scratching, sprayed coatings, testing, thermodynamic properties, wear mechanisms, wear of materials, wear-resistant coating

D.L. Duan, S. Li, X.H. Duan, and S.Z. Li, State Key Laboratory for Corrosion and Protection, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China. Cited: *Tribol. Trans.*, 2005, Jan/March, **48**(1), p 45-50. ISSN 1040-2004.

Process Analysis and Modeling

Critical velocities for high-speed particle deposition in kinetic spraying. During kinetic spraying (or cold gas dynamic spraying), small solid particle can be deposited onto the substrate by a supersonic velocity impaction. A critical velocity of flying particle was found for the minimum deposition speed. Generally, it was thought that higher impact velocity caused higher deposition efficiency. However, some different phenomena were observed during spraying processing in laboratory. In this study, individual particle impact tests have been carried out to probe the effect of impact velocity. Instead of deposition efficiency (DE) of coatings, ratio of bonds, which was defined as the number fraction of attached particles to total impact particles (craters + bonds) in unit area of impact surface, was measured in our test. A maximum velocity for particle deposition was observed, because of which a high fraction of rebounded particles occurred at a high impact velocity. A model considering the adhesion and rebound energy was built up to estimate the particle/substrate interaction. The particle deposition behavior was a result of the competition between adhesion and rebound energies during the impact process. Only when the adhesion energy was higher than the rebound energy, the impacting particles could be attached onto the substrate.

Keywords: adhesion, coatings, cold gas dynamic spraying, deposition efficiency, individual particle impact, kinetic spraying, reaction kinetics, spraying, velocity measurement

J. Wu, H. Fang, S. Yoon, C. Lee, and H. Kim, Kinetic Spray Laboratory (NRL), Division of Materials Science and Engineering, Hanyang University, Seongdong-Ku, Seoul 133-791, Seoul, South Korea. Cited: *Mater. Trans.*, 2006, July, **47**(7), p 1723-1727. ISSN 1345-9678.

Deposition behaviors of solid phases in liquid-solid two-phase particles in high-velocity oxyfuel spraying. Three types of composite coatings were deposited by high-velocity oxyfuel (HVOF) spraying using metal-clad WC-18Co and SiC-50Co cermet powders, and sintered-crushed Al_2O_3 -75Ni composite powders. Liquid-solid two-phase state of the sprayed particles with two different structures was achieved prior to their impingement on the substrate. The effects of particle structure and HVOF spraying parameters on volume fraction of the solid phase were investigated. The distribution of ceramic particle size inside coatings was also clarified. Scanning electron microscopy was employed to characterize the microstructure of HVOF sprayed cermet coatings. The volume fraction of solid particles and the distribution of particle size in the three types of coatings were estimated using a quantitatively metallographic approach from the microstructure of the

coatings. The volume fraction of the solid phase in the coating made from Al_2O_3 -Ni powders was increased with increase in the fuel gas flow. The maximum volume fraction of the Al_2O_3 phases reached about 64% of the original powder. On the other hand, the volume fraction of solid phase in the clad cermet coatings was decreased with increasing the fuel gas flow. The volume fraction of the solid phase dropped significantly from 72.2% in the original WC-18Co particle to 8.5% in the coating. The limited retention of the solid phase from spray particle to subsequent coating is due to rebounding off of large solid particle upon impacting.

Keywords: cermet coatings, coatings, composite coatings, deposition, high-velocity oxyfuel, impingement, scanning electron microscopy, sintering, solid-liquid two-phase state, spraying, velocity measurement, volume fraction

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On high-velocity impact of microsized metallic particles in cold spraying. In this study, a systematic examination of particle deformation behavior in cold spraying was conducted for Cu particle using both the Lagrangian and Arbitrary Lagrangian Eulerian (ALE) methods. It is found that the meshing size in modeling by Lagrangian method influences significantly the localized shear instability at interface areas. With refining the meshing size the onset velocity for interface shear instability decreases. The extrapolation of these results yields a reasonable critical velocity comparable to the actual one in cold spray practice. The results indicate that both the flattening ratio and compression ratio of the deformed particles increase with the increase in particle velocity, which are in good agreement with the experiment results. The ALE method provides a suitable way to examine the particle deformation in cold spraying. Moreover, the numerical results also show that there exists the similarity for the deformation of particles of different diameters.

Keywords: arbitrary Lagrangian Eulerian (ALE), cold spraying, computer simulation, copper, high-velocity impact, interfaces (materials), Lagrange multipliers, metallic particles, numerical analysis, particle deformation, shear deformation, shear instability, spraying

W.-Y. Li, H. Liao, C.-J. Li, G. Li, C. Coddet, and X. Wang, LERMP, Université de Technologie de Belfort-Montbéliard, 90010 Belfort Cedex, France. Cited: *Appl. Surf. Sci.*, 2006, Dec 30, **253**(5), p 2852-2862. ISSN 0169-4332.

In-flight formation and characterization of nickel aluminide powders in a DC thermal plasma jet. The strength of some intermetallics increases with temperature instead of exhibiting a decrease; thus, they are ideally suited for high-temperature applications. The formation of intermetallic compound during the spray process leads to metallurgical bonding due to the high exothermicity of the formation reaction. In this paper, formation of nickel aluminide in a thermal plasma jet from ball-milled nickel-aluminum powders is reported. Commercially available nickel and aluminum powders were mixed in the appropriate amount and injected into a thermal plasma jet produced by an atmospheric plasma torch. During flight, the formation of nickel aluminide takes place in plasma jet. Powders were collected at two different collection distances (40 and 80 cm) for three power levels (10, 15, and 20 kW), different plasma gas flow rates (15 and 20 lpm), and different powder feed rate (7 and 14 g/min). The powders are characterized using SEM, optical micrograph, TG/DTA, and XRD analysis. The effect of plasma parameters on the formation have been studied and reported. The formation is better at higher power levels, low powder feed rate, low plasma gas flow rate, and longer collection distances.

Keywords: intermetallics, nickel aluminide, nickel powder metallurgy, plasma applications, plasma spraying, powder metallurgy, scanning electron microscopy, spheroidization, thermal plasma jets

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Influence of impact parameters of zirconia droplets on splat formation and morphology in plasma spraying. In this study, the effects of the impact parameters, namely, the diameter d_0 , velocity V_0 , and temperature T_0 , of an impacting droplet of yttria-stabilized zirconia (YSZ) on splat morphology have been investigated systematically under plasma spraying conditions. In particular, fully molten droplets of 30 to 90 μm in d_0 that impact on a preheated quartz glass substrate at V_0 of 10 to 70 ms have been examined via hybrid plasma spraying. The degree of flattening of final splat morphology was found to be predicted by the relationship $= 0.43 \text{ Re}^{13}$, where Re is the Reynolds number. The dimensionless spreading time of droplets, $t_s^* = t_s / V_0 d_0$, was distributed around 2.7, where t_s is the spreading time of the droplet. The ideal maximum spread factor derived from the splat height was approximately proportional to Re^{14} . The latter two findings suggest that the analytical model developed by Pasandideh-Fard (*Phys. Fluids*, 1996, **8**, 650) can be applied to the droplet impact in plasma spraying especially for the case

of YSZ. In addition, the thermal contact resistance of disk-shaped splats decreased with the increase of V_0 within the range of 10^{-5} to 10^{-6} m²·KW.

Keywords: glass, morphology, parameter estimation, plasma spraying, quartz, Reynolds number, splat morphology, yttria-stabilized zirconia, zirconia, zirconia droplets

K. Shinoda, T. Koseki, and T. Yoshida, Department of Materials Engineering, Graduate School of Engineering, University of Tokyo, Bunkyo-ku, Tokyo 113-8656, Japan. Cited: *J. Appl. Phys.*, 2006, **100**(7), ISSN 0021-8979.

In situ densification of Ti coatings by the warm spray (two-stage HVOF) process. Coating of titanium is one of the surface modification techniques attractive for corrosion protection in various industrial and medical applications. In situ densification of titanium coatings fabricated by the warm spray (two-stage high-velocity oxyfuel, HVOF) process was investigated for the purpose of obtaining impermeability together with minimum oxidation level. Ceramic beads of ZrO₂-SiO₂ were mixed with titanium feedstock powder. By the peening effect of the ceramic particles, the density of the resultant titanium-matrix coatings could be improved, and the minimum coating porosity obtained in this study was 0.65 vol.%.

Keywords: coating porosity, coatings, corrosion protection, densification, oxidation, peening effect, porosity, shot peening, spraying, titanium, velocity measurement, warm spray

J. Kawakita, S. Kuroda, S. Krebs, and H. Katanoda, Thermal Spray Group, Materials Engineering Laboratory, National Institute for Materials Science, Tsukuba 305-0047, Japan. Cited: *Mater. Trans.*, 2006, July, **47**(7), p 1631-1637. ISSN 1345-9678.

Limits of gaseous detonation spraying. The maximum possible values of adhesion and cohesion are calculated for a number of powder materials, substrate materials, sizes of installation barrels, and compositions of explosive gas mixtures. These data give a clear idea about the areas of applicability of gaseous detonation spraying.

Keywords: adhesion, composition, detonation, gas mixtures, installation barrels, mixtures, powders, spraying, substrate materials, substrates

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Simulation of solid particle impact behavior for spray processes. Several thermal spray processes developed recently are characterized by relatively low temperature and higher velocity of sprayed particles. They include cold spray, high-velocity oxyfuel spray, and warm spray, in which majority or all the sprayed particles are in solid phase when impinging onto the substrate surface. Therefore, in order to understand the coating formation process of such processes, detailed knowledge concerning impact phenomena of a solid particle is essential. It is generally accepted that there exists a minimum velocity beyond which a particle adheres to the substrate and this velocity is called the "critical velocity." How the critical velocity depends on various materials and process variables is not fully understood yet. In this study, analysis of a metal particle impacting onto a metal substrate was carried out by using a dynamic finite element code (ABAQUS). Effects of a substrate and a particle temperature on the critical velocity were numerically studied. Also, effects of thermal conduction on the simulation results were discussed. It was found that critical velocity decreases with (a) higher stiffness of the substrate, (b) higher particle temperature, and (c) greater particle size.

Keywords: cold spray, computer simulation, critical velocity, high-velocity oxyfuel spray, low-temperature effects, particle size analysis, solid particle impact, sprayed coatings, spraying, thermal conductivity, thermal sprayed coating, velocity measurement, warm spray

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Thermoactive coating as a means for controlled action on the temperature field of an infinite solid body with a spherical heating source. A mathematical model of the process of formation of a temperature field in an infinite isotropic solid body containing a spherical heating source with a thermally thin thermoactive coating of its surface has been proposed. The obtained analytical solution of the corresponding problem of nonstationary heat conduction has been used for substantiation of the possibility of acting on the temperature field of the system under study in a controlled manner.

Keywords: heating, heating source, protective coatings, temperature distribution, temperature fields, thermal effects, thermoactive coating

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Spray Forming

Deformation behavior of an Al-Cu-Mg-Ti alloy obtained by spray forming and extrusion. An aluminum-base alloy, Al-Cu-Mg-Ti, was prepared via Osprey forming and extrusion. An elongated substructure toward the extrusion direction was observed after extrusion. Annealing treatments at temperatures up to 450 °C do not have an effect on such microstructure. Emergence of (sub)grains was observed at the highest annealing temperature of 520 °C, which is associated with the misorientation increase of the (sub)boundaries. Tensile tests at different temperatures and also temperature-change tests were performed at strain rates of 10^{-4} , 10^{-3} , and 10^{-2} s⁻¹. The analysis of data showed that the stress exponent coefficient increases from 5 at 500 °C to close to 14 at about 250 °C. The activation energy for deformation in both regimes was higher than that corresponding to aluminum self-diffusion. This creep behavior was associated with that of dispersion-strengthened aluminum alloys.

Keywords: aluminum alloys, annealing, deformation, deformation mechanisms, diffusion, mechanical behavior, metal extrusion, metal forming, microstructural characterization, microstructure, spraying, substructures

M. Eddahbi, F. Carreno, and O.A. Ruano, Departamento de Metalurgia Fisica, Centro Nacional de Investigaciones Metalurgicas, CSIC, 28040 Madrid, Spain. Cited: *Mater. Lett.*, 2006, Nov, **60**(27), p 3232-3237. ISSN 0167-577X.

Effect of additional elements on aging behavior of Al-Zn-Mg-Cu alloys by spray forming. The microstructure and aging behavior of spray formed Al-Zn-Mg-Cu alloys were investigated as a function of alloying element addition. It is revealed that the grains of the as-deposited alloys are refined with increasing Zn element, while the function of Ni addition is to reduce grain-boundary particles and eutectic in the as-extruded condition. Particles containing Mg and Zn are found to increase with Zn content increasing, while the role of Ni is to reduce both the number and size of these particles. After uniform heat treatment, parts of educts in grain boundary have melted and the grains have not grown up obviously. After heat extrusion, the microstructure becomes denser and there are many precipitated phases in cross section, while there are second phase arranging along extruded direction in longitudinal section. During artificial aging, the increment of Zn content produces not much effect on peak hardness, in addition to an accelerated overage softening. An addition of about 0.13%Ni, however, gives rise to not only improved peak hardness, but also an improvement of property stability at the aging temperature.

Keywords: addition reactions, additional elements, aging behavior, aging of materials, alloying, aluminum alloys, copper alloys, grain boundaries, grain growth, magnesium alloys, microstructure, nickel, property stability, spraying, zinc alloys, zinc content

L. Li, T.-T. Zhou, H.-X. Li, C.-Q. Chen, B.-Q. Xiong, and L.-K. Shi, School of Materials Science and Engineering, Beijing University of Aeronautics and Astronautics, Beijing 100083, China. Cited: *Trans. Nonferrous Met. Soc. China (English Edition)*, 2006, June, **16**(3), p 532-538. ISSN 1003-6326.

Gas tunnel type plasma spraying deposition and microstructure characterization of silicon carbide films for thermoelectric applications. Gas tunnel type plasma spraying deposition has been applied successfully to the deposition of the SiC films on stainless steel substrates. The microstructure and the surface morphology of the SiC films were characterized by means of x-ray diffraction (XRD) and scanning electron microscope (SEM). The control of the processing parameters such as powder feeding rate, composition of plasma working gases, spraying distance, and carrier gas flow rate allowed the deposition of dense, uniform, continuous, and high purity crystalline SiC films. The thickness of the SiC films varied from 3 to 10 μm. EDS analysis confirmed the presence of SiO₂ in the deposited SiC films.

Keywords: composition, gas tunnel plasma spraying, gas tunnels, microstructure, plasma spraying, scanning electron microscopy, silicon carbide, stainless steel, thermoelectric materials, thermoelectricity, x-ray diffraction

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Modeling the influence of the gas-to-melt ratio on the solid fraction of the surface in spray formed billets. In this paper, the relationship between the gas-to-melt ratio (GMR) and the solid fraction of an evolving billet surface is investigated numerically. The basis for the analysis is a recently developed integrated procedure for modeling the entire spray forming process. This model includes the atomization stage taking thermal coupling into consideration and the deposition of the droplets at the surface of the billet taking geometrical aspects such as shading into account. The coupling between these two models is accomplished by ensuring that the total droplet size distribution of the spray is the summation of "local" droplet size distributions along the *r*-axis of the spray cone. The criterion for a

successful process has been a predefined process window characterized by a desired solid fraction range at a certain distance from the atomizer. Inside this process window, the gas and melt flows have been varied and their influence on the solid fraction at the surface of the billet has been analyzed.

Keywords: atomization, billets (metal bars), computer simulation, deposition, drop formation, gas-to-melt ratio, mathematical models, melting, process window, spray forming, spraying, surface properties

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Substrate-Based Studies

Accelerated creep resistance of thermal barrier coated superalloy. This paper deals with accelerated creep properties of thermal barrier coated (TBC) Superni C263 alloy used as a candidate material in combustion chamber of aero engines. Results reveal that the life of the TBC composite under accelerated creep is substantially higher compared to that of the bare substrate. The mode of fracture in the substrate at very high stresses was transgranular, whereas that at low stresses was intergranular. Delamination of bond coat, oxidation of the substrate and spallation of the ceramic layer were evident at very high stress. It was evident that the substrate has negligible estimated rupture strength after 10,000 h of service exposure.

Keywords: combustion chambers, creep resistance, creep testing, delamination, fracture, intergranular, oxidation, rupture strength, spallation, stresses, superalloys, thermal barrier coatings

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Growth stresses in oxidized tubes under uniaxial and multiaxial oxidation strain. When metallic components operate in aggressive environments their service life is, in general, determined by the presence of a protective surface film that acts as a barrier to the reactants. Large residual stresses can result from the volume changes due to oxidation, the so-called growth stresses. These stresses may lead to film cracking or spalling or both. A viscoelastic model for the calculation of growth stresses in oxidizing tubes has been developed. It can deal with uniaxial and multiaxial oxidation strain tensors. Different oxidation modes such as surface and interface oxidation as well as duplex scale formation are treated. It appears that even relatively small lateral oxidation-strain components could have a considerable effect on the stress level in the tube. A simplified version of the model has been applied to simulate the geometrical changes of Zry tube sections exposed to air having reached the break-away regime. We think that lateral oxidation strains were mainly responsible for the observed diameter increase.

Keywords: elastic models, interfaces (materials), mathematical models, oxidation, oxidation strain tensors, oxidizing tubes, protective coatings, stresses, tensors

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On the high-temperature oxidation protection behavior of plasma sprayed Stellite 6 coatings. High-temperature oxidation resistance of the superalloys can be greatly increased by plasma sprayed coatings, and this is a growing industry of considerable economic importance. The purpose of these coatings is to form long-lasting oxidation protective scales. In the current investigation, Stellite 6 coatings were deposited on two Ni-base superalloys, Superni 601 and Superni 718, and one Fe-base superalloy, Superfer 800 H, by a shrouded plasma spray process. Oxidation studies were conducted on the coated superalloys in air at 900 °C under cyclic conditions for 50 cycles. Each cycle consisted of 1 h heating followed by 20 min of cooling in air. The thermogravimetric technique was used to approximate the kinetics of oxidation. All of the coated superalloys nearly followed the parabolic rate law of oxidation. X-ray diffraction (XRD), scanning electron microscopy/energy dispersive x-ray (SEM/EDAX), and electron probe microanalysis (EPMA) techniques were used to analyze the oxidation products. The Stellite 6 coating was found to be successful in maintaining its continuous surface contact with the superalloy substrates in all cases. The oxide scales formed on the oxidized-coated superalloys were found to be intact and spallation free. The main phases analyzed for the coated superalloys were oxides of cobalt and chromium and spinel of cobalt and chromium, which are suggested to be useful for developing oxidation resistance at high temperatures.

Keywords: cooling, energy dispersive spectroscopy, heating, high-temperature properties, microanalysis, oxidation resistance, plasma sprayed coatings,

plasma spraying, scanning electron microscopy, spallation, sprayed coatings, Stellite, surface contact, thermogravimetric analysis, x-ray diffraction analysis
H. Singh, D. Puri, S. Prakash, and V.V. Rama Rao, Mechanical Engineering Department, BBSB Engineering College, Fatehgarh Sahib-140407, India. Cited: *Metall. Mater. Trans. A: Phys. Metall. Mater. Sci.*, 2006, Oct, 37(10), p 3047-3056. ISSN 1073-5623.

Influence of substrate materials upon fabrication of aluminum nitride coatings by reactive RF plasma spraying. Influence of the substrate materials upon fabrication of aluminum nitride (AlN) coatings by reactive RF plasma spraying was investigated according to spraying onto various substrate materials. Aluminum particles were injected into Ar/N₂ plasma and were deposited onto carbon steel (S45C), aluminum alloy (A6063), and quartz substrates. In the case of using carbon steel substrate, fabrication of thick and dense Al-based Al/AlN composite coatings was possible. However, higher concentration of AlN phase in the coatings peeled off the coatings during cool down after spraying due to the difference in thermal expansion coefficient between AlN and carbon steel. Therefore, formation of Al interlayer was attempted between AlN layer and carbon steel substrate in order to decrease the thermal stress. As the result, Al interlayer included Al/AlN composite coating was fabricated onto a carbon steel substrate with control the N₂ flow rate in plasma gas during spraying. While fabrication of AlN layer onto Al interlayer was possible, it was concerned that formation of Fe-Al intermetallic compound at the interface between the coating and the substrate formed defects. Then not the Al interlayer but aluminum alloy was used as the substrate. However, the aluminum alloy substrate melted during spraying even the substrate was cooled by water-cooled substrate holder. Therefore, it is demanded that the substrate materials have a melting point that is much higher than aluminum. On the other hand, almost completely AlN coatings were fabricated using quartz substrate, which involves lower thermal expansion coefficient and higher melting point. Therefore, the characteristics of low thermal expansion coefficient and high melting point were required for substrate materials in order to fabricate AlN or AlN based Al/AlN composite coatings by reactive RF plasma spraying. Especially, quartz was useful for the substrate material.

Keywords: aluminum nitride, coated materials, fabrication, melting, nitride ceramics, particle beam injection, plasma spraying, quartz, radio frequency plasma, reactive plasma spraying, substrates, thermal expansion, thermal spray

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Substrate effect on the high-temperature oxidation behavior of a Pt-modified aluminide coating. Part I: Influence of the initial chemical composition of the coating surface. The effect of substrate composition on the oxidation behavior of the industrial NiPtAl coating RT22 was investigated by studying the isothermal and cyclic-oxidation behavior of this coating deposited on three different Ni-base superalloys (CMSX-4, SCB, and IN792). Isothermal tests were performed at 900, 1050, and 1150 °C for 100 h. Cyclic oxidation was studied at 900 °C with a holding time of 300 h for up to 52 cycles (i.e., 15,600 h at 900 °C). Thermogravimetric analysis (TGA), x-ray diffraction (XRD), and microstructural and analytical investigations using scanning-electron microscopy (SEM) and transmission-electron microscopy (TEM), both equipped with energy-dispersive X-ray spectroscopy (EDS) were performed to characterize the oxidation behavior of the systems studied. An effect of the superalloy substrate was observed and related to the initial chemical composition of the coating surface, which depends on the superalloy and the associated heat treatments. The effect of the alloying elements of the substrate is discussed. Particularly the influence of Ti and Ta that formed rutile-type oxides inducing oxide-scale cracking and spallation. The excellent resistance to cyclic oxidation of the coating systems studied at 900 °C was also demonstrated from very long duration tests of 15,600 h.

Keywords: alloying elements, composition, high-temperature effects, high-temperature oxidation, long-term cyclic oxidation, oxidation, platinum, platinum-modified aluminide coating, protective coatings, substrates, superalloys
N. Vialas and D. Monceau, CIRIMAT UMR 5085, ENSIACET-INPT, 31077, Toulouse Cedex 4, France. Cited: *Oxid. Met.*, 2006, Oct, 66(3-4), p 155-189. ISSN 0030-770X.

Surface Properties

Wetting behavior of plasma sprayed oxide coatings. Wetting behavior of several plasma sprayed oxide surfaces were characterized using contact angle measurements. Since surfaces contained pores and cracks, the evaluation of wetting angles led only to rough estimation of surface free

energies. In order to find out the effect of atmospheric contamination the wetting behavior of plasma-etched surfaces was followed as a function of time. It was found out that the sample preparation method had great influence on the contact angle of plasma sprayed oxide surfaces. The contact angle of plasma-etched surfaces increased when the surfaces were exposed to air. The probable reason for that was adsorption of low surface free energy contaminants to the sample surfaces.

Keywords: adsorption, atmospheric contamination, coating techniques, contact angle, contamination, impurities, oxides, plasma etching, plasma spraying, plasma-etched surfaces, surface free energy, surface properties, wetting angle

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Thermal Barrier Coating Systems

Comparison of microstructure and oxidation behavior of CoNiCrAlY bond coatings prepared by different thermal spray processes. To protect various gas turbine components against high temperature in the hot sections of power generation plants and aircraft engines, thermal barrier coatings (TBCs) have been developed and widely used. Conventional TBCs consist of a MCrAlY bond coating for oxidation resistance and a ceramic top coating for thermal insulation. High-quality coatings of MCrAlYs have been produced mostly by low-pressure plasma spraying, but other more economical processes are also used depending on the operating conditions of the component to be coated. In this study, CoNiCrAlY powders were deposited on Inconel 718 substrate with three types spraying system, that is, low-pressure plasma spraying, high-velocity oxyfuel spraying, and atmosphere plasma spraying. The specimens without top ceramic coating were isothermally tested for up to 100 h in air at 1373 K, and mass gain of the coatings was measured. Microstructure of the coating cross sections and the surface oxides were observed with SEM. Moreover, phase changes during the oxidation test were investigated with calculated phase diagrams for the CoNiCrAlY alloy.

Keywords: bond coatings, cobalt compounds, high-temperature oxidation, high-velocity oxyfuel spray, low-pressure plasma spray, microstructure, oxidation, oxidation resistance, plasma spraying, thermal barrier coatings, thermal insulation

M. Shibata, S. Kuroda, H. Murakami, M. Ode, M. Watanabe, and Y. Sakamoto, National Institute for Material Science, Tsukuba 305-0047, Japan. Cited: *Mater. Trans.*, 2006, July, **47**(7), p 1638-1642. ISSN 1345-9678.

Effect of beam angle on HAZ, recast, and oxide layer characteristics in laser drilling of TBC nickel superalloys. Industrial applications of laser drilling include the production of cooling holes at acute angles in certain parts of the aeroengine components. These parts are often covered with ceramic thermal barrier coatings (TBCs) to protect them from reaching excessive temperatures in hot engine environments. Acute angle TBC drilling brings three major simultaneous complications to the process. These are: (a) multilayer drilling, (b) nonsymmetrical geometry and melt ejection, and (c) increased depth of drilling. In a previous investigation by the authors, delamination of TBC was found as a main problem of angled drilling and mechanisms involved were studied. In the present study, implications of these difficulties on the hole quality is investigated through a comparative study of vertical and acute angle drilled holes. Characteristics of recast layer, heat-affected zone (HAZ), oxide layer, and TBC delamination are investigated. Variation of these metallurgical characteristics with the depth of the hole is evaluated. Results for vertical and inclined holes are compared. The extent of HAZ, recast layer, and oxide layer is seen to vary significantly with location and is found increasing with decreasing drilling angle to surface. Numerical simulation of pulsed laser heating of TBC Nimonic 263 was carried out for acute angle drilling with assist gas considerations. Results from the simulation suggested that the total heat transfer rate is higher on the leading edge side than the trailing edge of the heated region. Experimentally observed larger HAZ on leading edge side and larger recast layer on trailing edge side are explained by the analysis of heat flow characteristics obtained with the model.

Keywords: cooling, drilling, heat-affected zone, laser applications, laser beams, laser drilling, melt ejection, nickel, nonsymmetrical geometry, oxides, recast layer, superalloys, thermal barrier coatings

H.K. Sezer, L. Li, M. Schmidt, A.J. Pinkerton, B. Anderson, and P. Williams, Laser Processing Research Centre, School of Mechanical, Aerospace and Civil Engineering, The University of Manchester, Manchester, M60 1QD, U.K. Cited: *Int. J. Machine Tools Manuf.*, 2006, Dec, **46**(15), p 1972-1982. ISSN 0890-6955.

Effect of hollow spherical powder size distribution on porosity and segmentation cracks in thermal barrier coatings. The effect of characteristics of hollow spherical (HOSP) powders on porosity and

development of segmentation cracks in plasma sprayed thick thermal barrier coatings (TBCs) was investigated. Three powders with particle size ranges of 20 to 45, 53 to 75, and 90 to 120 μm were selected from a commercial HOSP powder feedstock for spraying the TBCs. The 20 to 45 μm powder has a higher deposition efficiency and a greater capability of producing segmented coatings than the other larger powders. Diagnostics of in-flight particles show that the average surface temperature and velocity of the particles sprayed from the fine powder is higher by 250 $^{\circ}\text{C}$ and 50 m/s compared with those sprayed from the 90 to 120 μm powder, respectively, due to its greater ratio of surface area to mass. The lower porosity of the coating sprayed from the fine powder is mainly attributed to the decreased volume of intersplat gaps and voids.

Keywords: cracks, deposition, hollow spherical powders, intersplat gaps, particles (particulate matter), plasma spraying, porosity, powder metals, segmented coatings, thermal barrier coatings

H.B. Guo, H. Murakami, and S. Kuroda, Department of Materials Science and Engineering, Beijing University of Aeronautics and Astronautics, Beijing 100083, China. Cited: *J. Am. Ceram. Soc.*, 2006, Dec, **89**(12), p 3797-3804. ISSN 0002-7820.

The effect of preoxidation atmosphere on oxidation behavior and thermal cycle life of thermal barrier coatings. The effects of oxygen partial pressure (p_{O_2}) of the preoxidation atmosphere on the growth of thermally grown oxide (TGO) and thermal cycle life of plasma sprayed thermal barrier coatings (TBCs) were investigated. The p_{O_2} of the preoxidation atmosphere was controlled by using a solid-state electrochemical oxygen pump system. The purity and microstructure of continuous Al_2O_3 layer formed on the bond coat during preoxidation at 1050 $^{\circ}\text{C}$ were highly influenced by the p_{O_2} of the atmosphere. The specimen preoxidized at 1050 $^{\circ}\text{C}$ under a p_{O_2} of 10^{-14} to 10^{-15} atm, which is around the dissolution pressure of $(\text{Co,Ni})(\text{Al,Cr})_2\text{O}_4$ spinel, showed the lowest growth rate of TGO and the longest thermal cycle life.

Keywords: oxidation, oxides, oxygen, oxygen partial pressure, partial pressure, preoxidation, preoxidation atmosphere, pressure effects, thermal barrier coatings, thermal cycle life, thermally grown oxide

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Evolution of surface morphology of thermomechanically cycled NiCoCrAlY bond coats. We investigate morphological surface instabilities on bond coat surfaces of thermal barrier coatings, induced due to thermo-mechanical loading. Experimental results of hollow circular cylindrical specimens, consisting of a directionally solidified superalloy (IN 100 DS) coated with a NiCoCrAlY bond coat, show that the morphological instabilities are strongly dependent on the load conditions. In particular, the morphological instabilities develop during thermal cycling with a thermal gradient over the cylinder wall, whereas the surface remains smooth for thermal cyclic conditions without a gradient. Furthermore, if a cyclic, axial tensile force is applied (synchronized with the thermal cycling), the morphological instabilities become aligned with the axial direction. We discuss a model, quantified by finite element simulations, capturing this behavior and elucidating the thermomechanical response.

Keywords: bond coat surfaces, computer simulation, elasticity, elastic-plastic material, finite element method, mathematical models, morphology, nickel alloys, oxidation, plasticity, superalloys, surfaces, thermal barrier coatings, thermal cycling, thermal gradients, thermomechanical testing

J. Shi, A.M. Karlsson, B. Baufeld, and M. Bartsch, University of Delaware, Newark, DE 19716-3140. Cited: *Mater. Sci. Eng. A*, 2006, Oct 25, **434**(1-2), p 39-52. ISSN 0921-5093.

Microstructural characterization and properties of $\text{ZrO}_2/\text{Al}_2\text{O}_3$ thermal barrier coatings by gas tunnel-type plasma spraying. Spraying condition plays an important role in the plasma sprayed coating process and affects the final properties of the coatings. Zirconia, alumina, and zirconia/alumina composite coatings were prepared on a stainless steel substrate (SUS304) by the gas tunnel-type plasma spraying. Effects of different alumina mixing ratios on the coating properties were investigated. The results indicated that the mixing ratio of powders and the traverse number of substrate had an influence on the hardness, porosity, and wear weight loss of composite coatings. The hardness increased while the porosity decreased with the increase in alumina mixing ratio. The porosity that was less than 10% and a hardness about $\text{HV} = 1400$ was obtained for the alumina coating. The adhesive strength and wear weight loss of the composite coatings were also clarified at different alumina mixing ratios.

Keywords: adhesion, adhesive strength, alumina, gas tunnel-type plasma spray, hardness, microstructure, plasma spraying, porosity, powders, stainless steel, substrates, thermal barrier coatings, wear of materials, wear weight loss, x-ray diffraction, zirconia, zirconia/alumina composite coating

G. Shanmugavelayutham, S. Yano, and A. Kobayashi, Joining and Welding Research Institute, Osaka University, Ibaraki, Osaka, 567-0047, Japan. Cited: *Vacuum*, 2006, Sept 7, **80**(11-12), p 1336-1340. ISSN 0042-207X.

Modification of thermal barrier coating architecture by in situ laser remelting. Ytria partially stabilized zirconia thermal barrier coatings (TBCs) are widely used to protect components of aero gas turbines against high heat fluxes and hence increase their properties by reducing their in-service temperature. However, these coatings degrade in service conditions. Therefore, manufacturing TBCs that present both low thermal conductivity and high lifetime is a real challenge. Engineering the coating architecture by an adapted process is a prerequisite to modify TBC characteristics. In this study, laser remelting was combined to thermal spraying in order to modify the TBC properties. In situ laser treatment (a) changes structure from lamellar to dendritic columnar; (b) generates a pore architecture less sensitive to sintering, maintaining the TBC thermal and mechanical properties during thermal treatments at high temperatures; (c) improves the thermal insulation properties of the TBC by decreasing its thermal conductivity of about 30%; (d) decreases its permeability permitting reduction of oxidation and corrosion phenomena of the underneath layers and substrate; (e) increases the resistance to isothermal shocks (with the possibility to double the number of cycles); (f) conducts to a metastable tetragonal phase more stable during thermal shocks; and (g) without modifying the elastic response of the deposit.

Keywords: corrosion, elastic response, heat resistance, heat shielding, heat treatment, laser applications, laser remelting, oxidation, plasma-laser hybrid process, porosity, remelting, thermal barrier coatings, thermal conductivity, thermal insulating materials, thermal shocks, zirconia

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Oxidation damage of APS thermal barrier coatings under high temperature. Thermal barrier coatings (TBCs) have received increased attention for advanced gas turbine engine application. The oxidation damage plays an important role of the failure under high temperature load. An Y_2O_3 partially stabilized ZrO_2 ceramic top coating was deposited a NiCrAlY bond coating by air plasma spray (APS). The substrate was directionally solidified superalloy (DZ40M). Isothermal oxidation has been performed at 700 and 1050 °C for 100 h. The oxidation results in three transformations of the weight, the interface, and the surface characters. All the specimens were characterized by highly precision balanced, scanning electron microscopy (SEM) in cross section, microscope with MFK2 visual measurement system on its surface. The curve of oxidation kinetics is obtained by weighting those specimens. The weight gain of the specimen under 700 °C is close to zero. Under 1050 °C, the weight gain is very distinct. On the other hand, the interface cracks occur and extend at the interface between the ceramic and bond coating by SEM method. The thermal growth oxide (TGO) gradually appears on the coating surface under high-temperature oxidation by MFK.2. Last but not least, those observations will provide some ideas and damage parameters for the nondestructive inspection (NDI) for TBC under thermal load.

Keywords: air plasma spray, bond coatings, ceramic coatings, high-temperature effects, interfaces (materials), loads (forces), oxidation, oxidation damage, reaction kinetics, thermal barrier coatings

Q. Hongyu, Y. Xiaoguang, L. Rui, and W. Hongliang, School of Jet Propulsion, Beihang University, Beijing 100083, China. Cited: *Key Eng. Mater.*, 2006, **324-325**, p 595-598. ISSN 1013-9826.

Residual stress and damage evolution in TBCs by optical method. In recent years, ruby fluorescence spectroscopy has been demonstrated as a powerful technique for monitoring residual stress evolution in the thermally grown oxide scale in thermal barrier coatings (TBC) systems. The measured residual stresses, in turn can be used to monitor evolution of damage in the coatings. Effective use of this technology for real-time damage monitoring requires the identification of strength in measured stresses that can be used as indicators of damage evolution. The present work focuses on studying the evolution of residual stresses in TBC systems during oxidation. The coatings are atmospheric plasma sprayed (APS); the residual stress were measured at different oxidation time and to identify critical features so as to be used as indicators of failure in TBCs.

Keywords: atmospheric plasma sprayed, coatings, failure (mechanical), fluorescence spectroscopy, residual stresses, thermal barrier coatings

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The thermal cyclic behavior of the TBCs with the MCrAlY coating method. The rotating components in the hot sections of land-based gas turbine are exposed to severe environment during several tens of thousands of hours at above 1100 °C operating temperature. To protect such components from high-temperature oxidation, an intermediate bond coat is applied, typical of a MCrAlY-type metal alloy. Various processing methods have been studied for bond coat deposition. This study is concerned with the thermal cyclic behavior of thermal barrier coatings. The MCrAlY bond coatings are deposited by a vacuum plasma, low vacuum plasma, and high-velocity oxygen fuel spray on a nickel-base superalloy (GTD-111). Thermal cyclic tests at 1100 °C in still air for various periods of time were used to evaluate the thermal cyclic resistance of the TBC coating with the various processing sprayed bond coating layer. The microstructure and morphology of as-sprayed and of thermal cycled coatings were characterized by scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS) and x-ray diffraction (XRD). The order of thermal cyclic resistance was YSZ-VPS bond coat, YSZ-LVPS, and YSZ-HVOF. The influence of bond coat spray methods on durability of TBCs is discussed.

Keywords: gas turbines, high-velocity oxyfuel, protective coatings, rotating machinery, scanning electron microscopy, superalloys, thermal barrier coating, thermal cyclic resistance, thermal cycling, thermooxidation, x-ray diffraction

J.-S. Jung, K.-B. Yoo, E.-H. Kim, C.-H. Jeon, and D.H. Kim, Power Generation Laboratory, KEPRI, Daejeon 305-380, South Korea. Cited: *Key Eng. Mater.*, 2006, **324-325**, p 631-634. ISSN 1013-9826.

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