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# *PROCEEDINGS*



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# 25<sup>th</sup> IFHTSE CONGRESS PROCEEDINGS

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Chinese Heat Treatment Society

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## **Fabrication of Al matrix composites via cold spraying-process, structure and properties**

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**Abstract:** Carbon nanotube reinforced aluminum matrix composites (CNTs/Al) are regarded as the new generation lightweight structural materials for aerospace and automobile applications. The powder metallurgy is the most commonly used method for producing CNTs/Al composites. However, during the sintering process, extra heat source is added to consolidate the CNTs/Al powders, which may lead to grain growth and the formation of the unexpected  $Al_4C_3$  phase; Besides, the consolidation approaches for these powders like hot rolling and hot extrusion are not flexible to fabricate the component with complex shapes. Cold spray (CS) is a unique solid-state materials consolidation process which utilizes high velocity particles impinging upon a substrate to build up coatings and/or near-net shaped parts without the use of combustion fuel. Owing to its 'cold' feature, the deleterious effects of oxidation, phase transformation, decomposition, grain growth, and other problems can be minimized or eliminated. Such significant advantages make CS a promising technique for producing temperature-sensitive materials, like CNTs/Al composites. In this work, CNTs/Al composites with a nanolaminated structure were fabricated by mechanical alloying followed by CS. Microstructure characterization was carried out in terms of X-ray diffraction, Raman spectroscopy, scanning and transmission electron microscopy. It was revealed that the nanolaminated architecture, containing nanosized grains of around 160 nm in the average size, was properly created in the CNTs/Al composite powder by ball milling, which was then conserved in the cold sprayed component. Further, the harmful excessive interfacial reaction product  $Al_4C_3$  was not revealed and structural integrity of initially incorporated CNTs was well retained. Further, the effect of heat treatment on the microstructure and mechanical properties of the cold sprayed CNTs/Al composite was investigated. Heat treatments were performed under temperatures ranged from 350 °C to 500 °C for 2 h. Microstructural features including the precipitation of nano-sized Si particles, interfacial reaction, grain size evolution, and the interface between CNTs and Al matrix were characterized and related to their mechanical properties. It was found that after heat treatment, a large number of nano-sized Si particles precipitated interlayers, and  $Al_4C_3$  formed when the annealing temperature reached 450 °C. The micro-hardness of the CNTs/Al composite increased slightly, while that of unreinforced Al decreased due to the grain growth and a decrease in dislocation density. This result suggests that the addition of CNTs into Al matrix can hinder the grain growth and dislocation movements during the annealing process. Hence, CS technique is potential to open a new avenue for additive manufacturing of CNTs/Al composite with complex shape, which has been unachievable so far.

**Keywords:** cold spray, additive manufacturing, carbon nanotube reinforced aluminum matrix composite, ball milling, mechanical properties

## **Robust corrosion protection of magnesium alloys via in-situ microforging assisted cold spray**

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**Abstract:** Magnesium (Mg) alloys have wide applications in the aerospace, automotive and microelectronics industries due to their excellent properties including low density (one quarter of steel and only two thirds of aluminum), high specific strength, high damping property, high thermal conductivity and strong electromagnetic shielding ability. However, the relatively poor corrosion resistance of Mg alloys restricts their wider applications. Cold spray (CS) is a relatively new coating process in which the coating is deposited by accumulated supersonic speed (300-1200 m/s) impact of the microsized particles (5-50  $\mu\text{m}$ ) at solid state. The low processing temperature in CS avoids microstructural deterioration of the Mg alloy substrate and makes CS coating corrosion resistant metals a potential approach to improve the corrosion resistance of the Mg alloys. However, the connected poor bonded inter-particle boundaries in conventional cold sprayed coatings allow the penetration of the corrosive media into the coating and reach the substrate. This makes the CS Ni coated carbon steel reveal a corrosion behavior in-between bulk Ni and carbon steel substrate. In this work, an in-situ micro forging effect was introduced by mixing large sized micro-forging particles into the feedstock particles, so that the deposited layer will be hammered and deformed by the micro-forging particles and thus results in fully dense coating. In current study, Ni and 6061Al were selected as the coating materials on AZ31 Mg alloy. Corrosion protection performance of the in-situ micro-forging assisted CS coatings was evaluated by a series of electrochemical tests and long-term corrosion environment exposure test. It was found that the fully dense Ni and 6061Al coating can be achieved by the in-situ micro-forging assisted cold spray under the optimized forging intensity. Both Ni and 6061Al coated AZ31 Mg show comparable open cell potential and polarization curves with bulk Ni and 6061Al bulk materials, respectively suggesting excellent corrosive media barrier ability of the coatings. After 2000 h of immersion test and salt fog test, no corrosion products were detected at coating/substrate interface. By partially anodic oxidizing or micro-arc oxidizing of the 6061Al coating on AZ31 Mg substrate, a composite bilayer coating composed of ceramic top layer and 6061Al bottom layer was formed and notable improvement in corrosion resistance and wear resistance was simultaneously achieved. The corrosion rate and wear rate were further reduced by 1000 and 200 times as compared with the bare AZ31 Mg. The present work provides a novel strategy for robust corrosion protection of magnesium alloys.

**Keywords:** magnesium alloy, in-situ microforging assisted cold spray, robust corrosion protection

## Evolution of anisotropy of cold-sprayed Cu during post-spray heat treatment

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**Abstract:** Cold spray is an effective approach to additively manufacture ductile metals due to its high efficiency and excellent coating/deposit performance. In cold spraying, micro-sized particles (typically 10-50  $\mu\text{m}$ ) are fed into high-velocity gas flow and accelerated to velocities of 300-1200 m/s. The deposit was formed by the high velocity particle impact induced severe plastic deformation at fully solid state. Since the high velocity impact results in nonuniform plastic deformation at the interface between particle/particle or particle/substrate, the bonding is discrepant at the center and fringe of the interface. The discrepant bonding characteristic leads to an anisotropic behavior for the cold sprayed deposits in mechanical properties, electrical and thermal conductivities, especially in the directions paralleling or perpendicular to the particle deposition. The anisotropic manner may restrict the application of the deposits in some specific circumstances. It is known that post-heat-treatment is capable to heal up the unbonded interface through atomic diffusion. However, it is not clear that whether and how the post-heat-treatment will affect the anisotropic microstructure and performance in coating/deposit. To clarify the effect of post-spray heat treatment on the anisotropic microstructure and performance of the deposit, the Cu deposit prepared at a gas pressure of 5 MPa and a gas temperature 800 °C by using nitrogen was selected as the starting material. The Cu deposits were annealed at series of temperatures from 200 to 600 °C. The microstructures of both the as-sprayed and heat-treated samples were characterized by scanning electron microscopy (SEM). The tensile performance, micro-hardness, electrical and thermal conductivities of the as-sprayed and heat-treated samples prepared along both directions paralleling and perpendicular to the particle deposition were investigated. The results show that the number of the unbonded interparticle boundaries decreases with the increase of the post-heat-treatment temperature and the dwelling time. The tensile strength and micro-hardness of the samples decrease slightly after heat-treatment while the electrical and thermal conductivity of the samples increase significantly. The performance difference between both directions paralleling or perpendicular to the particle deposition after heat-treatment decreases.

**Keywords:** cold-sprayed Cu, post-spray heat treatment

## Influence of impinging angle, particle shape and tamping on splats deformation and interface bond of cold sprayed Ti particles

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**Abstract:** To understand the splats deformation and the interface bonding, the angular and spherical Ti particles were deposited on polished stainless steel and Ti6Al4V substrate using cold spray process with different impact angles. The nitrogen was used as accelerating and powder feeder gas with the pressure of 2.0 and 2.2 MPa, respectively. The gas temperature measured in gun chamber was kept  $300\pm 30$  °C and  $600\pm 30$  °C, respectively. The spray distance was 10 cm and the transverse speed of gun was 200 mm/s. The surface and cross sectional morphologies of splats as well as the interface were examined by SEM through cutting splats with the assistance of focus ion beam. It was revealed that the impinging angle, particle's shape and tamping possesses significant effect on splats deformation and the interface bonding. For spherical particles, in the impinge angle of  $90^\circ$ , the most splats exhibit symmetrical deformation. The splats present a hemispherical shape with some revers on splats rim. The bonding area on splats/substrate interface exhibits a ring band feature and is close to the splats periphery on gas temperature of  $300\pm 30$  °C, the bonding area enlarges when gas temperature increased to  $600\pm 30$  °C. In the impinging angle of  $60^\circ$  most splats exhibit un-symmetric deformation. The bonding region is liable to appear in rear area in the slide direction. Nevertheless, the particles can hardly embed the substrate when the impact angle at  $30^\circ$ . The bonded area of splats/substrate interface enlarges after tamping by impact of thereafter incident particles. Nevertheless, this enhancement is restricted when the impact of incident particle was interfered by other particles. For angular particles, the deformation of splats is complicated with the irregular jets appears on the interface. There also exist some grooves or corrugations on splats surface. The bonding area of splats/substrate interface in impact angle of  $90^\circ$  is larger than that in  $60^\circ$ . These above experimental results indicate that the increase the gas temperature can improve the splats deformation and interface bonding. The hammering of incident particles can enhance the deformation and interface bonding of splats.

**Keywords:** cold spray, Ti, impact deformation, bonding, tamping

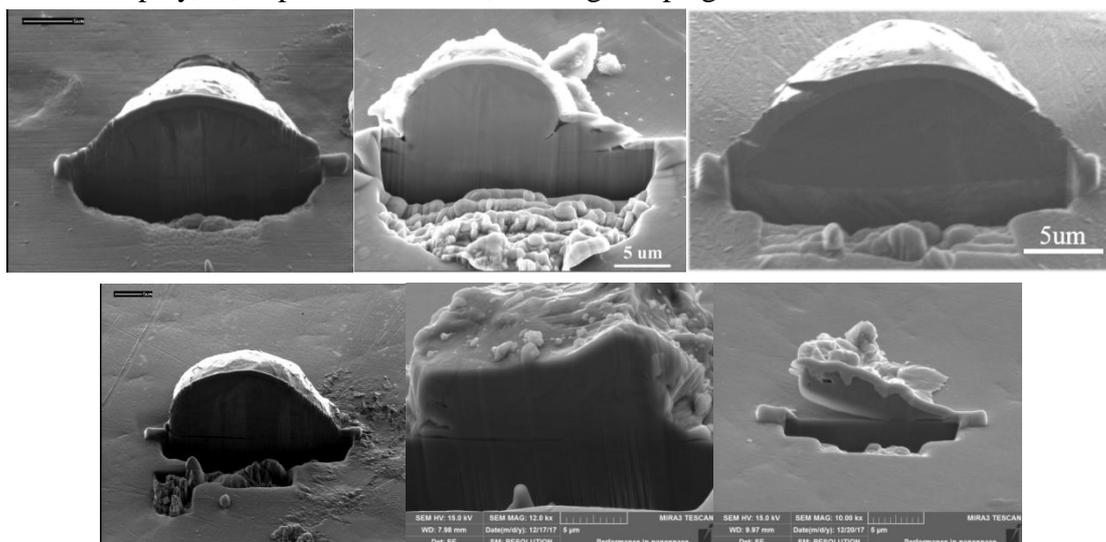


Fig.1

## Comparative investigation on the electrical properties of plasma sprayed

### TiO<sub>2</sub> and TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> coatings

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**Abstract:** Titanium dioxide shows broad prospect of applications in many environmental friendly and green energy occasions, owing to its n-type semi conductivity, good photo-catalytic activity along with high physical and chemical stability and good mechanical property. Recently, it was found that TiO<sub>2</sub> films or coatings were easier and more economy to be recycled than TiO<sub>2</sub> powders. Among all the methods of preparing coatings or films, plasma spray has been considered as one of the most promising ways to manufacture TiO<sub>2</sub> functional coatings for large scale application. The conductivity of plasma sprayed TiO<sub>2</sub> coatings was found very sensitive to the microstructure of the coatings as well as the phase transformation behaviors during the spraying process. Also the conductive TiO<sub>2</sub> coatings could become invalid along with great decrease in conductivity and damage in microstructure after a period of service at certain temperatures. The purpose of this comparative investigation is to find out the effect of Al<sub>2</sub>O<sub>3</sub> addition on the microstructure and electrical properties of plasma sprayed TiO<sub>2</sub>-based coatings, with better understanding of their failure mechanism after long time and high temperature services.

TiO<sub>2</sub> and mixed TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> powders with average particle size of 40-50 μm were used as feedstock for coating spraying. Both TiO<sub>2</sub> and TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> coatings were prepared with atmospheric plasma spray system (Metco 9M). The electrical properties were continuously measured through electrifying the coatings until a material failure occurred. X-ray diffraction (D8 Advance, Cu Kα1 radiation, 35 kV and 35 mA) was used to characterize the crystalline structure of the coatings. The topographic morphology of coatings was examined by scanning electron microscopy (Hitachi S-3400N and FEI Quanta 200) with EDS probe.

The results obtained show: a) the effective electrifying operations of TiO<sub>2</sub>-40%Al<sub>2</sub>O<sub>3</sub> coatings can remain longer time (over 46 hours at 400 °C, over 170 hours at 190 °C) than those of the pure TiO<sub>2</sub> (8 hours at 295 °C, 52 hours at 167 °C), demonstrating a positive effect of Al<sub>2</sub>O<sub>3</sub> addition on improving the high temperature resistance of plasma sprayed TiO<sub>2</sub> coatings. b) The characterization and analysis of failure microstructure of the coatings after long term electrifying operation indicate that three major microstructure origins in the coating failures can emerge from the arc crack caused by high current electrifying, the local hot spots leading to an inner short circuit within the coatings, and the re-oxidation of conductive phases during long term thermal cycle. The new information obtained in this investigation may be useful for improving the electrical properties of the TiO<sub>2</sub> based coatings and in retrofitting the plasma spraying process.

**Keywords:** electrical property, TiO<sub>2</sub> based coatings, microstructure failure

## Effect of heat-treated on the microstructure of micro-plasma sprayed hydroxyapatite coatings

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**Abstract:** Hydroxyapatite (HA) is widely used as surface coatings on biomedical implants because of its chemical composition similarity to calcified tissue. Atmospheric plasma spraying (APS) is known as a cost effective deposition method. However, the low crystallinity of APS deposited coating accelerates its dissolution in body fluid. Micro-plasma spraying (MPS) is an alternative plasma spray method using low plasma power ( $\leq 4$  kW). It is feasible to deposit coating on small-scale medical devices because of the concentrate plasma torch. The inflight particles is 60-80 mm/s during MPS, which is 1/3 of that for APS. Therefore the dwell time of HA particles within plasma is sufficient for well melting before deposition and thus improving the powder using efficiency. The compared with thin film techniques, MPS provided an economically possibility to obtain HA coating with sufficient structure. Many factors influencing the properties of HA coatings include their phase composition, crystallinity and surface topography. Another essential microstructure feature responsible for the cell adhesion behavior and mechanical properties of HA is crystallographic texture. Importantly, MPS can produced HA coatings with high crystallinity, and usually accompanied by crystallographic texture. It is a common preferential crystal orientation of HA in human teeth.

In this study, there representative HA coatings were deposited onto Ti-6Al-4V substrate by micro-plasma spraying: 1) high crystallinity (93.3%) coatings with porous structure, 2) high crystallinity coatings (86%) with columnar structure, 3) higher amorphous calcium phosphate (ACP, 50%) coatings with dense structure. The commercially available hydroxyapatite powder (Medicoat, M ägenwil, Switzerland) with particle sizes of 45-63  $\mu\text{m}$  was employed as a raw powder for depositing the coatings. The effect of the heat treatment on the crystallinity, texture and surface morphology of these coating is discussed by and field emission scanning electron microscopy (FESEM). The Ca/P ratio was determined by energy dispersive spectrometer (EDS). The effect of holding period on texture and surface morphology of HA coatings is also investigated. XRD analysis revealed that after treatment the HA peaks were sharper and the amorphous diffuse background was reduced. Tetra calcium phosphate (TTCP) and tri-calcium phosphate ( $\alpha$ -TCP) were also reduced by means of post heat treatment. Heat treatment HA coatings at 600 °C for period of 3 h leads to complete transfer of TTCP and amorphous phase in HA. The texture intensity of as-sprayed HA coatings was found to decreased after treatment. However, the texture intensity of HA coatings increased with the increase of the treatment times. SEM results showed that micro particles were formed in the coatings surface. They were generally 100-1300 nm. The micro particles that originally formed at the splat rim. The surface of the coating with more amorphous, by contrast, contains more micro particles. In addition, with a further increase of the holding period, the micro particles become coarse.

**Keywords:** hydroxyapatite, micro-plasma spraying, heat treatment, texture

## **Fabrication and characteristics of heating coatings by atmospheric plasma spraying**

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**Abstract:** Heating rollers have been widely used in the paper making, printing, film, and foil converting industries in the past few decades. Oil, water and steam heating rollers were used initially, and their market share is still above 90% even today. However, they can no longer fulfill the performance requirements due to the disadvantages such as lack of temperature uniformity, high energy consumption, and handling safety problems for the operators, and accordingly the high maintenance cost and downtime. In order to overcome the problems mentioned above, electrical heating rollers, which adopt ohmic heating or electromagnetic induction heating, were developed and have been used mostly for the upmarket industries. Despite the excellent performance, the complex roller structure and high maintenance cost have been the bottleneck in promoting the application of this technology. Thus, it is of crucial importance to develop a new heating method, for instance, the thermal spray technology, to fabricate the heating coating directly on the surface of the roller. In this study, a heating coating system composed of metallic bond coating, ceramic insulating coating and TiO<sub>2</sub> based ceramic conductive coating, was sprayed onto the gritblasted roller-shaped steel substrate in sequence by atmospheric plasma spraying. The microstructures and characteristics including coating phase composition, adhesion strength, insulating property of as-sprayed spinel coatings, and the electrothermal performance of conductive coating, were systematically investigated. According to the results, the existence of bond coating significantly improves the adhesion strength between the coating and substrate. Spinel coating has an excellent insulating performance especially at high temperature. TiO<sub>2</sub> based coating operates reliably around 400 °C and can even reach the highest temperature of 490 °C. Electrothermal cycle test indicates that the repeatability and controllability of the coating perform quite well within the experimental time. In general, the presence of deoxidization phase Magneli in the TiO<sub>2</sub> coatings is the main factor of conducting. However, the Magneli phase can be oxidized during service time and make the electrical resistivity of the coating increase, thus the service life and heating stability might be affected negatively. As an alternative solution, MoSi<sub>2</sub> based coating was fabricated as the conductive coating, and a thin protective ceramic coating was applied optionally on the top of the coating to provide necessary corrosion or wear performance. The newly developed coating can operate at 300 °C stably for a long time. However, with the increase of service temperature, the peeling and oxidation phenomenon occurred rapidly. To summarize, MoSi<sub>2</sub> based coating has the potential to be used as conductive coating, but intensive investigation is still needed.

**Keywords:** APS, microstructure, heating coating

## Microstructures, phase compositions and properties of warm sprayed

### HA/Ti composite coatings

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**Abstract:** Titanium and stainless steel alloy are widely used as biomedical implant materials due to their good mechanical and biochemical properties. Nevertheless, the problems of body's allergy and granuloma aroused by the release of a large number of metal ions influence their use as long-term implantation of the human body. Hydroxyapatite (HA), is considered as one kind of biomaterial used to coat metal implant due to their good bioactivity and biocompatibility. Commonly, Ti is added to HA to fabricate HA/Ti composite coatings for improving mechanical property of pure HA. Plasma spraying is usually employed to deposit HA and its composite coatings. However, the high temperature led to the decomposition of HA and the formation of CaO, TCP, TTCP which affects the performance of the plasma deposited HA coating. Compared with plasma spraying, warm spray has low spraying temperature. It can avoid the formation of detrimental phase and retain the structure of HA, compromise the mechanical and bio-properties of the coating by adjusting the spraying parameters.

To investigate the enhancement of Ti on HA coating, the pure hydroxyapatite (HA) powder and composite powders with 10, 30, 50 and 70 wt% titanium (Ti) were sprayed onto 316L stainless steel substrates by warm spray processes. The microstructures, phase compositions, chemical structures of the composite coatings were investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD) and fourier transform infrared spectrometer (FTIR), respectively. The Vickers micro-hardness and elastic modulus as well as fracture toughness were determined by indentation tests. The adhesion strength was determined by tensile tests. The vitro bioactivity of all the composite coatings was evaluated by immersing in simulated body fluid (SBF).

The experimental results show that the cross section of the HA/Ti composite coating presented typical lamellar structure composing of a large number of spherical/flat melted particles and the un-melted particles. The main phases of composite coating were HA and TiO<sub>2</sub>, in addition to a small amount of TiO. The bonding strength and the microhardness of the all composite coatings were higher than that of the pure HA coating. The apatites with the spherical and needle-like shape were formed on coatings' surface after immersed in simulated body fluid for 7 and 14 days respectively. The apatite appeared on the pure hydroxyapatite coating surface is the largest and densest and on 30wt%HA/Ti coating surface is the least. It can be concluded that the Ti particles added in HA is beneficial to improve the mechanical performance of composite coating, The warm deposited HA/Ti composite coating possesses well bioactivity and mechanical properties.

**Keywords:** warm spraying, HA/Ti composite coatings, microstructures and phase compositions, mechanical properties, vitro bioactivity

## Thickness measurement and interface morphology characterization of thermal barrier coatings based on terahertz time-domain spectroscopy

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**Abstract:** Thermal barrier coatings (TBCs) are applied to ensure the structural integrity of high-temperature components in gas-turbine engines. Various failure modes will occur during service and usually result in thinning and spalling of ceramic coating. Due to the complexity of service condition and difficulty of detecting, health monitoring of TBCs are absolutely vital and full of challenges. Terahertz nondestructive testing (NDT) technology, a novel method to measure the thickness and quality of TBCs, is proposed to tackle it. When terahertz waves are incident on TBCs, partial terahertz waves are reflected from the ceramic coating surface, while the remaining part transmits through the surface and is completely reflected at the interface between ceramic coating and metallic bondcoat. Partial reflected terahertz from the interface transmits through the ceramic coating surface into air and partial terahertz is reflected back into ceramic coating. Multiple reflections are repeated at the ceramic coating surface and interface. The propagation property could be used for nondestructive testing of TBCs. In this work, yttria-stabilized zirconia (YSZ) coatings were deposited by atmospheric plasma spraying (APS). Finite Difference Time Domain (FDTD, Lumerical Solutions Inc. Canada) was used to measure the single-layer and multilayer ceramic coating thickness with an angle of incidence of 0. Gaussian beam was chosen by FDTD Solutions to explore the propagation characteristics of terahertz waves in different thickness coatings. The simulation results showed that terahertz waves were effective in measuring coating thickness. Terahertz time-domain spectroscopy (THz-TDS) system of reflection mode was used to measure the ceramic coating thickness with an angle of incidence of 0 before and after erosion test. Refractive index and absorption coefficient of YSZ were obtained considering the interaction between the terahertz and TBCs. The obtained refractive index was  $4.98 \pm 0.09$  at the frequency range of 0.3-0.5 THz and the obtained absorption coefficient was  $32.96 \pm 1.82 \text{ cm}^{-1}$  by analyzing the amplitude attenuation. The ceramic coating thickness was calculated by the mathematical model on refractive index and time delay of reflection peaks. The ceramic coating thickness was  $405.68 \pm 11.33 \text{ }\mu\text{m}$  which obtained by scanning electron microscope observation. The thickness obtained by terahertz-domain spectroscopy was  $418.17 \pm 6.26 \text{ }\mu\text{m}$ . The relative error  $(d_{\text{THz}} - d_{\text{SEM}}) / d_{\text{SEM}}$  was 3.08% which proved the reliability of this method. The refractive index, when considering the surface roughness, was  $5.16 \pm 0.10$ . The average thickness was  $403.05 \pm 5.88 \text{ }\mu\text{m}$  considering the surface roughness. The relative error  $(d_{\text{THz}} - d_{\text{SEM}}) / d_{\text{SEM}}$  was 0.57% and the measurement accuracy was improved after considering the influence of surface roughness. The first surface reflection peak in time-domain spectrum moved backward with the increasing erosion. The thickness loss obtained by terahertz time-domain spectrum was compared with thickness loss obtained by electronic digital readout micrometer. The results showed that ceramic coating was thinning after erosion test and the interface morphology of TBCs was arc-shaped. Moreover, surface roughness was also an important factor to be considered in the process of interface morphology characterization. More accurate results with roughness were also given out. Accordingly, the thickness and interface morphology could be quickly determined by terahertz time-domain spectroscopy. Finally, it can be concluded that THz-TDS provides a new idea for nondestructive and non-contact on-line evaluation of the integrity of TBCs applied to gas-turbine engines.

**Keywords:** non-destructive, thermal barrier coatings, terahertz time-domain spectroscopy, ceramic coating thickness, interface morphology

## **Al-modified 7YSZ thermal barrier coating prepared by PS-PVD and its application prospect**

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**Abstract:** Plasma spray-physical vapor deposition (PS-PVD) is a novel coating process. Unlike conventional technologies, deposition can take place from solid, liquid and vapor state. This offers new opportunities to obtain advanced microstructures and thus to meet the growing demands for thermal barrier coatings (TBCs). PS-PVD, due to its electrical current up to 3000 A and plasma gas flow up to 200 slpm, an input power level of 180 kW, the plasma plume can expand to a length of more than 2000 m and 200-400 mm in diameter. The PS-PVD facility was shown in Fig.1(a) and its plasma jet was presented in Fig.1(b).

Recently, much attention has been paid to the development of PS-PVD. It has been reported that feather-like columnar structured 7YSZ coating can be prepared with high deposition rate and cost efficiency. The columnar structured YSZ coating has lower thermal conductivity (0.7-1.2 W/(m K)) than APS (0.8-1.7 W/(m K)) and EB-PVD coating (1.5-2.0 W/(m K)), due to its higher porosity (30%-50%) compared with APS (15%-25%) and EB-PVD coating (10%-20%). Besides, high porosity will lead to good strain tolerance performance resulting in good thermal cycle resistance (>5000 cycles, surface temperature kept at 1250 °C for 5 minutes, back temperature cooled by compressed air). However, in high engine operation temperature, the TBCs surface will be covered with a molten CMAS glass deposit, which will penetrate into the porous TBCs leading to the loss of strain tolerance and premature failure. CMAS attack should be solved before application in next-generation gas-turbine engines. For solving the urgent problem, a new approach that Al-modification was proposed. An Al film was deposited on TBCs surface by magnetron sputtering. A dense  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> overlay can be in situ synthesized on top of the 7YSZ coating through the reaction of Al and ZrO<sub>2</sub> after vacuum heat treatment. This method not only decreases the porosity of the top ceramic coating but also increases the CMAS corrosion resistance.

In this investigation, the feedstock material was agglomerated 7YSZ (M6700, Oerlikon Metco). Disks made of superalloy were used as substrates and the topside edge of the specimens was rounded. Prior to deposition of 7YSZ coating, the substrates were coated with a 150  $\mu$ m thick NiCoCrAlYTa bond coating (Amdry 386, Oerlikon Metco) by the PS-PVD. With the aim to improve the durability of PS-PVD TBC, the specimens coated with polished bond coating were heated up in the vacuum chamber by the plasma jet to generate  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> layer on the bond coating. After that, the feather-like columnar 7YSZ coating was prepared. Sequently, an 5  $\mu$ m thick Al film was deposited on TBCs surface, as shown in Fig.2(a). Fig.2(b) is the magnified image indicating dense interface between Al film and 7YSZ coating. The above Al-deposited 7YSZ TBCs samples.

The CMAS corrosion of as-sprayed and Al-modified 7YSZ coating was compared as following. Firstly, the CMAS powders were sprinkled on the surface of 7YSZ coating (0.1 g/mm<sup>2</sup>). Then, the TBCs samples were placed in a furnace kept at 1200 °C for 24 h followed by air cooling. Cross-sectional microstructure of both as-sprayed TBCs and Al-modified TBCs were analyzed, as shown in Fig.3(a-b). During CMAS corrosion test, the 7YSZ grain in as-sprayed coating would dissolve in the molten CMAS and separate from the oriented structure into small independent blocks, seen in Fig.3(a). As opposed to the Al-modified 7YSZ coating, the top of the Al-modified 7YSZ coating has no obvious change due to an  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> overlay which was in situ synthesized on the top of porous 7YSZ coating. The  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> overlay

has a good CMAS corrosion resistance and acts as a CMAS diffusion barrier. Apart from CMAS corrosion, thermal cycle is also an important performance in application. For a more realistic evaluation, turbine blade was used. The as-sprayed and Al-modified TBCs were prepared in the same blade, seen in Fig.4(a). After 30 thermal cycles (1100 °C, 10 minutes holding time per-cycle, water-quenching), the spallation area and crack density in Al-modified TBCs are lower than the Al-modified TBCs, seen in Fig.4(b) showing a greater thermal cycle property.

**Keywords:** PS-PVD, thermal barrier coatings

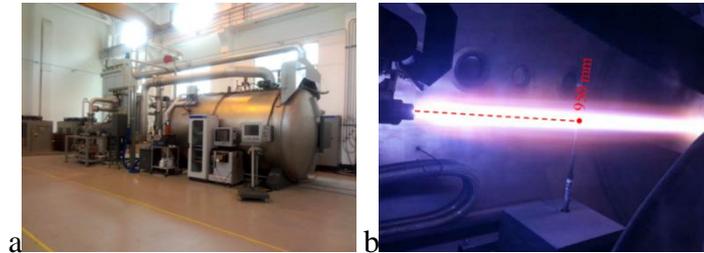


Fig.1 (a) PS-PVD facility at author's lab, (b) plasma jet in PS-PVD

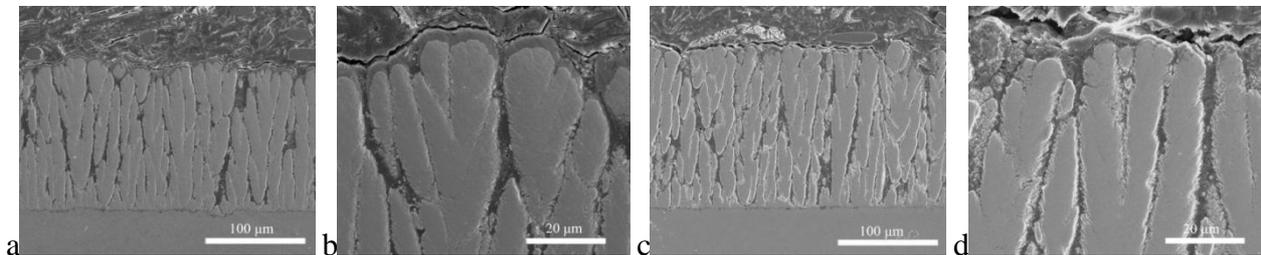


Fig.2 Top micrograph of TBC after CMAS corrosion, (a) as-sprayed TBC, (b) Al-modified TBC were carried out with vacuum heat treatment. The Al-modified 7YSZ TBCs are shown in Fig.2(c-d), which imply that the Al film has disappeared. Because the Al film had infiltrated into the porous 7YSZ coating and reacted with the  $ZrO_2$  forming  $\alpha-Al_2O_3$

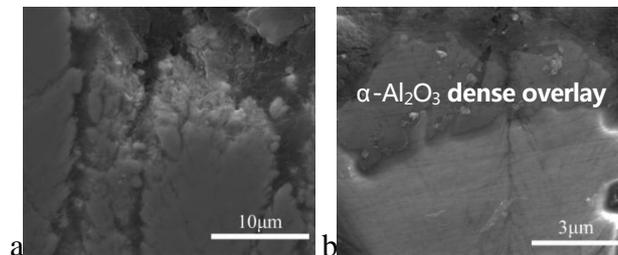


Fig.3 Top micrograph of TBC after CMAS corrosion, (a) as-sprayed TBC, (b) Al-modified TBC

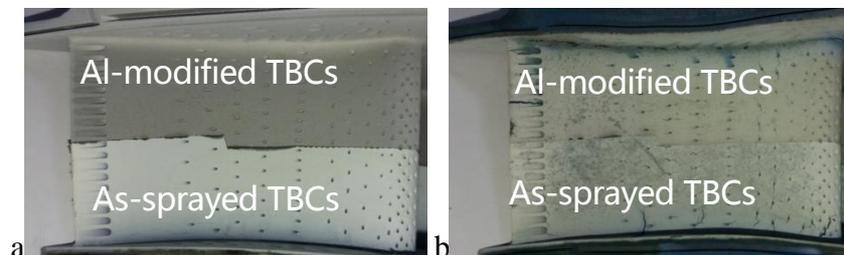


Fig.4 Water-quenching test of as-sprayed and Al-modified TBCs, (a) before testing, (b) after testing with 30 thermal cycles

## The evaluation of durability of plasma-sprayed thermal barrier coatings with double-layer bond coat

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**Abstract:** The thermal expansion mismatch between layers in plasma-sprayed thermal barrier coating system (TBCs) dramatically affects the durability of TBCs. In order to reduce the detrimental effect, the double-layer bond coat with gradient thermal expansion coefficient was attempted to be deposited in the case. The Co<sub>32</sub>Ni<sub>21</sub>Cr<sub>8</sub>Al<sub>0.5</sub>Y powder with a spherical shape was used to spray the bond coat. The bond coat consisted of porosity layer and oxidation layer was designed. The shrouded plasma spraying was employed to deposit the porosity layer on super alloy substrate, then the oxidation layer was sprayed on porosity layer via high-velocity oxygen fuel (HVOF). The whole thickness of bond coat was about 180 μm. Two bond coats with different thickness ratio of porosity layer to oxidation layer were prepared. The ratio of porosity layer to oxidation layer in first bond coat is about 1/2 (type A), correspondingly, the ratio of porosity layer to oxidation layer in second type bond coat is 1/1 (type B). The yttria-stabilized zirconia (YSZ) powder was used to deposit the ceramic topcoat through atmospheric plasma spraying (APS). The high temperature oxidation behaviors of these two bond coats were addressed, and the durability of TBCs were also evaluated via thermal cycling test. The high temperature oxidation were performed in atmosphere at 1000 °C for 5, 20, 50, 100, 200 and 500 h, respectively. The durability of TBCs was evaluated. The peak temperature of thermal cycling test was 1120 °C. One whole thermal cycling test was 30 min, including 26 min holding time and 4 min cooling time. The results show that the porosity layer of the bond coat has a typical laminar structure with little oxidation. The α-Al<sub>2</sub>O<sub>3</sub> based thermally grown oxide (TGO) generates in HVOF bond coat. Compared with the porosity layer, the oxidation layer has a lower thermal expansion coefficient because of generation of α-Al<sub>2</sub>O<sub>3</sub>. Therefore, the TBCs with a gradient thermal expansion coefficient bond coat were deposited. The α-Al<sub>2</sub>O<sub>3</sub> generated in HVOF bond coat grows with increasing oxidation time. The TGO growth rate is much different, depending on the bond coat microstructure. The oxidation resistance of bond coat named type A is dramatically higher than that of type B bond coat, i.e., the oxidation resistance of bond coat increases as the thickness of oxidation layer. Moreover, no recognized oxidation generates in porosity layer in these two bond coat. The lifetime of type B TBCs is approximately 48 cycles, which is comparable to the lifetime of TBCs with single bond coat indicated in previous report. In comparison, the thermal cyclic lifetime of type A TBCs is 58 cycles. Therefore, the durability of TBCs can be improved via design of bond coat with gradient thermal expansion coefficient. The reason that bond coat with a thinner HVOF layer has a lower durability is associated with the oxidation resistance, based on the oxidation behavior and cracking mode. Thus, the durability of TBCs may increase if a fraction of α-Al<sub>2</sub>O<sub>3</sub> is added into the powder during HVOF.

**Keywords:** double-layer, plasma-sprayed thermal barrier coating

## **Effect of particle size on the cracking behavior of plasma-sprayed YSZ coatings during thermal shock and erosion testing**

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**Abstract:** In the present study, yttria-stabilized zirconia (YSZ) coatings were deposited by atmospheric plasma spraying (APS) using feedstocks with different particle sizes. The effect of particle size on the cracking behavior of the coatings during thermal shock and erosion condition has been studied.

The particle size of feedstock powder affects the stacking morphology of the splats that forms the coating. Coatings fabricated from coarse YSZ powder show a relatively rough inter-lamellar surface. Due to the insufficient lamellar interface roughness, the cracking resistance of coatings fabricated from fine YSZ is worse than that from coarse one. Coatings prepared by fine powder are tend to form large cracks parallel to the substrate direction under indentation, while no cracking phenomenon was found in coatings prepared by coarse powder. Moreover, the porosity for the coatings made from the coarse powder is higher than the porosity of coatings made from fine one, which increasing the strain tolerance of the coatings made from the coarse powder. The orientation of the pores in the coatings prepared by different particle sizes is different. Coatings prepared by coarse YSZ powder tend to form almost the same number of horizontal and vertical pores, while coatings prepared by fine powder tend to form horizontal ones parallel to the direction of the substrate. Finite element simulation results revealed that the maximum stress shows a decreasing trend as the pore orientation changes from horizontal to vertical.

Due to the higher cracking resistance and better compliance, the coatings deposited from coarse particles show high thermal shock and a high thermal cycle life compared with coatings deposited from fine powders. During thermal cycling, the crack length of the coatings made from the fine powder increases much more than the crack length of the coatings made from the coarse powders, and an incubation period is observed for the coatings made from the coarse powder, which appears to postpone the onset of rapid cracking. During the erosion test, the cracking of coatings prepared by fine YSZ powder tends to take place from the lamellar or splat interfaces, whereas that in coatings prepared by coarse powder tends to occur inside the splat. The difference of crack propagation mode is an important factor that affects the cracking behavior of coatings. Coatings prepared by coarse powder show better erosion resistance than that by fine one due to its higher cracking resistance. The results of this investigation are a benefit to the design and integrity of TBCs.

**Keywords:** YSZ, particle size, cracking, thermal shocks, erosion

## Sintering mechanism of plasma-sprayed TBCs-from free-standing to constrained states

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**Abstract:** As two key performances, the thermal barrier effect and the resistance to spallation exposed to high temperature guide the material selection, structural tailoring and process optimization of thermal barrier coatings (TBCs). During thermal exposure, sintering would lead to performance degradation inevitably. Therefore, revealing structural evolution of the top coat is crucial to understand the failure phenomenon during thermal service, and to provide some fundamental supports for structure optimization. For TBCs, the structural evolution of the top coat would be affected alternately by the sintering and the substrate constraint. Consequently, the top coat would exhibit a dynamic structural evolution. However, the mechanism behind is still cannot well understood up to now, since most of the current investigations were focused on the individual effect of the strain or the sinter. The objective of this study is to reveal the sintering mechanism of plasma-sprayed TBCs from free-standing to constrained states. To begin with, a comprehensive understanding on sintering behavior of the free-standing coatings was obtained. Results show that the healing of 2D pores was aging stage-sensitive, resulting in a two-stage evolutionary trend of mechanical property. In detail, at the stage-I, a rapid healing induced by multi-point connection occurred at the narrow parts of the 2D pores. At the following stage-II, the residual wider parts of the 2D pores decreased the possibility of multi-point connection at their counter-surfaces. Consequently, the further healing would proceed primarily from two tips to center without multi-connection, resulting in a much lower sintering kinetic at this stage. Subsequently, the effect of substrate constraint on healing behavior of the 2D pores was considered. It is revealed that the overall evolution trends of microstructure and property can be divided into three stages affected by the substrate constraint. Moreover, the anisotropic healing of the 2D pores reported in free-standing coatings was enhanced significantly due to the additional stress in coatings resulting from constraint of substrate. This means that the healing of inter-splat pores became faster and severer. Following on, a structural model was developed to correlate the structural evolution and mechanical property. Results show that the model predictions are well consistent with the experimental data. Finally, an outlook on structural tailoring to retard the performance degradation of TBCs was proposed.

**Keywords:** substrate constraint, anisotropic healing kinetics, sintering; thermal barrier coatings, structural tailoring

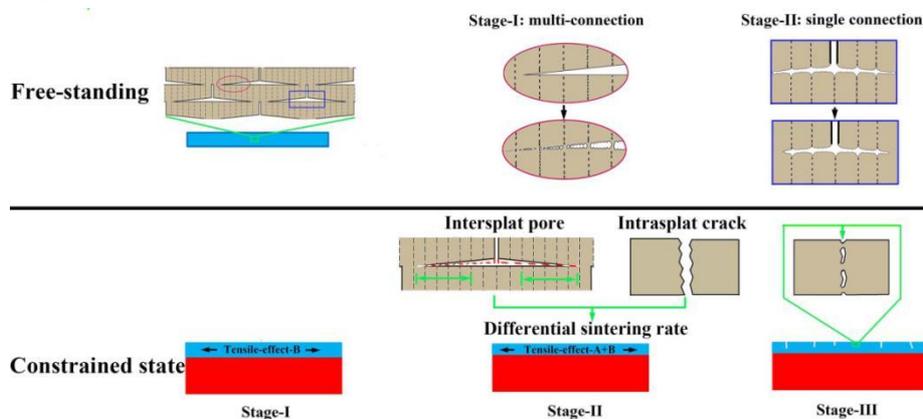


Fig.1 Schematic of sintering mechanism of TBCs: from free-standing to constrained states

## Structurally integrated damage tolerant coatings

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**Abstract:** Thermal spray coatings are used extensively for the protection and life extension of engineering components exposed to harsh wear and/or corrosion during service in aerospace, energy, and heavy machinery sectors. Cermet coatings applied via high-velocity thermal spray are used in aggressive wear situations almost always coupled with corrosive environments. In several instances (e.g., landing gear), coatings are considered as part of the structure requiring system-level considerations. In addition, spray based remanufacturing of worn components is also expanding with the advent of high velocity spray technology and here the integration of the restored and parent material from a structural point of view is of importance. Despite their widespread use, the technology has lacked generalized scientific principles for robust coating design, manufacturing, and performance analysis. Advances in process and in situ diagnostics have provided significant insights into the process–structure–property–performance correlations providing a framework-enhanced design. In this overview, critical aspects of materials, process, parametrics, and performance are discussed through exemplary studies on relevant compositions. The underlying connective theme is understanding and controlling residual stresses generation, which not only addresses process dynamics but also provides linkage for process-property relationship for both the system (e.g., fatigue) and the surface (wear and corrosion). The anisotropic microstructure also invokes the need for damage-tolerant material design to meet future goals.

This presentation will provide an overview of emerging concepts of structurally integrated coating design and structural remanufacturing of engineering components. In addition to traditional methods of coating evaluation, new methods of integrated characterization of coating and structure is contemplated. Using these principles approaches to enhancing applications will be presented.

**Keywords:** structurally integrated coating

## Tailoring porous structures of nano zinc oxide coatings by liquid plasma spraying for enhanced photocatalytic performances

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**Abstract:** It is known that zinc oxide is an n-type semiconductor with promising photocatalytic performances under ultraviolet light. Enlarging surface area keeps as one of the major research goals for ZnO for enhanced photochemical reaction rate. In the present study, we report a new technical route for constructing porous topographic morphology of nano ZnO coatings. The coatings were fabricated by plasma spraying the mixture of suspension and solution liquid precursors. The as-sprayed coatings in microns thickness show porous skeleton and fluffy top layer consisting of ultrafine ZnO grains. Photocatalytic degradation testing using 5 ppm methylene blue suggested promoted performances of the coatings by the special structural features. The hybrid-structured ZnO coatings present narrower band gap and higher degradation rate of dye than those deposited from single liquid feedstock. The results shed light on one-step easy fabrication of functional nanostructured coatings by employing hybrid liquids as starting feedstock for plasma spraying.

**Keywords:** nano zinc oxide, liquid plasma spray, hybrid structure, dye degradation

## Mechanical properties of in-situ micro-forging assisted cold spray additively manufactured AA6061 alloy

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**Abstract:** Cold spray is a solid-state material deposition technology which has recently been of great interest to be an additive manufacturing process to fabricate individual components or repair damaged components. Six series Al alloys are mainly alloyed with silicon and magnesium and show excellent combined performance, which makes them widely used as structural components in the aerospace and automotive industry. In this study, an in-situ micro-forging assisted cold spray process was developed to manufacture the AA6061 deposit by mixing large sized micro-forging particles into the spray powders. Microstructural and mechanical properties of cold sprayed AA6061 deposit were investigated. SEM observations reveal that no shot-peening particle was deposited into Al6061 deposit. By deliberate introducing the in-situ hammering effect to the CS process, a highly dense (porosity<0.3%) AA6061 deposit was prepared. The as-sprayed AA6061 deposit showed relatively higher ultimate strength (about 270 MPa) and microhardness (116.4 HV100) and lower ductility (about 0.5%) compared to tempered bulk counterpart due to significant finegrain strengthening and dislocation strengthening mechanisms. The dimple characteristics in the fractured specimens revealed that the inter-particle of in-situ shot-peening-assisted cold sprayed AA6061 presented well bonding. In order to further optimize the mechanical properties of AA6061 deposit, the solution and aging treated were investigated as a function of treatment time. After 30 min solution treated at 535 °C and 2 h aging treated at 175 °C, great improvement in ductility was achieved (about 8% elongation). Meanwhile, the ultimate strength was further increased to about 310 MPa which is comparable to the. The correlation between the detailed microstructure in terms of grain size, content and size of the Mg<sub>2</sub>Si precipitation and the mechanical properties was also systematically investigated.

**Keywords:** AA6061 alloy, mechanical properties

## Influence of the temperature on the microstructure and wear performance of HVOF sprayed bimodal WC-12Co coatings

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**Abstract:** The bimodal WC-12Co coatings were prepared using high-velocity oxygen-fuel (HVOF) spraying technique. The coatings were then heat treated for 4h at 350, 450 and 550 °C in air atmosphere. The compositions, microstructure, mechanical property and wear performance of the heat-treated bimodal WC-12Co coatings were systematically investigated. The results show that the bimodal WC-12Co coating was composed of WC, W<sub>2</sub>C, amorphous or nanocrystalline Co(W, C). After heat treatment, the WO<sub>3</sub> and CoWO<sub>4</sub> were detected at 450 °C and 550 °C. The pores were healed at 450 °C and the coating exhibited loose structure at 550 °C. After heat treatment, the hardness first increased and then decreased, the maximum hardness was 1261 HV when the coating was heat treated at 450 °C. The friction coefficient decreased due to the lubricating WO<sub>3</sub> and CoWO<sub>4</sub>, the wear rate first decreased and then increased with the increase of heat-treated temperature, the minimum value of  $3.95 \pm 0.42 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$  was obtained when the coating was heat treated at 450 °C for 4 h.

**Keywords:** HVOF, WC-Co

## **Suspension plasma spray fabrication of black titanium dioxide photocatalytic coatings with visible light absorption performances**

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**Abstract:** Due to their excellent photocatalytic performances, nano titanium dioxide particles and coatings have attracted extensive worldwide research efforts. The persistent challenges yet pertain to further enhancing the activity by a variety of modifications, among which tailoring the structure of titania already showed great promises. Here we report for the first time suspension plasma spray construction of black titanium dioxide coatings with porous microstructures and excellent photocatalytic performances. Controllable phase transformation from anatase to black titania was accomplished by adjusting the moving speed of plasma nozzle and spray power. The black titania comprising the mixture of anatase, rutile, metastable brookite, and  $Ti_2O_3$  exhibited remarkably enlarged exposed surface area. Further TEM characterization revealed that formation of the black titania is associated with grain growth of titania and tremendous lattice disorders, which likely offered the capability of visible light absorption. Examination of photocatalytic performances of the coatings showed enhanced absorption and degradation of methylene blue under visible light, which was predominately attributed to the narrowed band gap as compared to anatase structure.

**Keywords:** suspension plasma spray, black titanium dioxide, photocatalytic degradation, visible light catalysis

## **Development of a FeCrMnBC-based economical wear and corrosion resistant coating**

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**Abstract:** Main advantages of Fe-based coatings are their favourable lower costs as well as their good environmental behaviour compared to other established protective coatings. In this study, a highly economical coating system based on a FeCrMnBC alloy was developed, consisting only of economically favourable elements. The coating was produced using a spray powder with a size distribution of  $-90+45$   $\mu\text{m}$  by a three-cathode plasma generator TriplexProTM-210 and evaluated in terms of the microstructure, hardness, wear and corrosion resistance. It was found that the coating exhibited a dense lamellar microstructure and a microhardness value of more than 600 HV0.1. The coating was significantly more wear resistant compared with a plasma sprayed stainless steel coating of 316 L. Salt spray test confirmed that the corrosion behavior of the FeCrMnBC coating was comparable to that of a plasma sprayed 316 L coating. Due to the higher wear resistance, comparable corrosion behavior and lower material costs this new coating system can be used as more economical alternative or replacement to conventional stainless steel coatings to protect carbon steel as well as cast iron parts against wear and corrosion.

**Keywords:** Fe-based coating, plasma spraying, microstructure, wear, corrosion

## Optimization of spray parameters on the corrosion behavior of HVOF sprayed Fe-based amorphous coatings

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**Abstract:** Fe-based amorphous coatings were deposited on the substrate of Q235 steel by the high-velocity oxygen-fuel (HVOF) thermal spraying process. The effects of the selected parameters (spray distance, oxygen flow and kerosene flow) on the porosity and, in turn, the corrosion resistance of the coating were investigated by using the Taguchi method. Statistical tools such as design of experiments (DOE), signal-to-noise (S/N) ratio and analysis of variance (ANOVA) were used to optimize the spray parameters. The important sequence of spray parameters on the porosity of the coating is kerosene flow > spray distance > oxygen flow. The optimum spray parameters (OSP) were determined as 380 mm for the spray distance, 1840 scfh for the oxygen flow, and 6.8 gph for the kerosene flow. The potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) results showed that the Fe-based amorphous coating exhibits superior corrosion resistance and seems to be an alternative to hard chromium coating. Besides, the coating with lower porosity exhibits better corrosion resistance than the higher one.

**Keywords:** Fe-based coatings, amorphous, HVOF, Taguchi method

## Microstructure and wear behavior of Al<sub>0.6</sub>TiCrFeCoNi high entropy alloy coating produced by HVOF

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**Abstract:** High-entropy alloys (HEAs) are in the focus of research in recent decades due to the excellent properties such as high strength, high hardness and good wear resistance. Amongst these alloys, AlTiCrFeCoNi system HEAs attract great attention because of the potential application as lightweight materials. Al<sub>0.6</sub>TiCrFeCoNi high entropy alloy possesses a single BCC phase with a high hardness. Because of these properties, Al<sub>0.6</sub>TiCrFeCoNi high entropy alloy is a promising candidate material for wear-resistance application by means of thermal spray technology. In this study, the atomized Al<sub>0.6</sub>TiCrFeCoNi high-entropy-alloy powder was used for high-velocity-oxygen-fuel (HVOF) to prepare a dense coating on Q235 steel. The coating were deposited by a JP5000 HVOF spraying system with the spraying parameters of kerosene flow 6.8 GPH, oxygen flow 2200 SCFH, nitrogen Flow 26 SCFH, feed rate 5 r/min and spraying distance 380 mm. The microstructure and phase composition of the coating were investigated, indicating that both the powder and coating exhibited a bcc phase while some unmelted particles and pores were found in the coating. The wear behavior under different temperature conditions were measured in ball-on-disk with the force of 5 N, speed of 10 cm/s, distance of 500 m and a diameter of 6 mm Al<sub>2</sub>O<sub>3</sub> as the counter body. Meanwhile, the worn surface and cross-section morphologies as well as EDS analysis were conducted. The results showed that as the temperature increased from room temperature to 500 °C, the wear resistance reduced greatly. Furthermore, the wear mechanism of Al<sub>0.6</sub>TiCrFeCoNi coating at room temperature,  $T=300$  °C and  $T=500$  °C were discussed.

**Keywords:** high-entropy-alloy, wear behavior, HVOF

## Detonation spraying of the copper powder coated with graphene

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**Abstract:** The paper presents the results of work on manufacturing and testing of properties of Cu-G coatings sprayed with detonation method.

The detonation spraying is one of the methods of thermal spraying, enabling to manufacture the coatings which are characterised by high hardness, low porosity, good adherence and increased wear resistance. The essence of the denotation spraying is the utilization of the explosive combustion energy of a gaseous mixture to heat the particles of the powdered coating material and to give them the specified high kinetic energy. With use of a detonation method the copper powder coated with graphene–Graphene 3DIMP was thermally sprayed in order to manufacture a composite coating.

The basis of this modern composite material is graphene coated on copper powders, so called the 3DIMP Graphene which has been manufactured in the Institute of Precision Mechanics. Forming of the graphene structures on the surface of the copper powder comes into being as a result of the thermal-chemical processes taking place in the atmosphere of hydrocarbons in a fluid deposit. The copper powder together with graphene was subjected to the tests by means of an electron scanning microscope and a Raman spectrometer. In the Raman spectra the 2D and G bands characteristic for graphene spectra are clearly visible, which confirms its presence in the tested powders.

The fundamental advantage of the Cu-G composite coating obtained by detonation spray method compared with the Cu coating is the cohesion of the structure which results from the much fewer number of oxides and from the good connection of the grains among one another. There is a good connection of the coating with the substrate.

The pure copper powder is strongly oxidized during the process of spraying. A large number of oxides, which are included in the coating and are arranged in the bands between the copper grains, cause a significant decrease of the cohesion of the coating and make the connection with the substrate worse. During the spraying of the copper powder with graphene, the carbon structures protect the particles of the copper powder against the excessive oxidization and thanks to this there are much fewer oxides in this coating than in the sprayed Cu powder coating.

**Keywords:** detonation spraying, graphene, copper powder, thermal spraying

## Microstructure and properties of laser re-melting FeCoCrNiAl<sub>0.5</sub>Si<sub>x</sub>

### high-entropy alloy coatings

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**Abstract:** FeCoCrNiAl<sub>0.5</sub>Si<sub>x</sub> ( $x = 0.5, 1.0, 1.5,$  and  $2.0$ at%, respectively) high-entropy alloy (HEA) coatings were produced on Q235 steel via the air plasma spray and laser re-melting technique to investigate the effect of different Si concentrations on the microstructure and properties of HEA coatings. The results showed that the structure of BCC phase in the HEA coatings increased with the addition of Si. The average hardness of the HEA coatings also increased gradually. The maximum hardness reached at 1085 HV. The friction coefficient reached its lowest value about 0.25. The main wear mechanism was abrasive wear. The wear resistance of the HEA coatings was enhanced.

**Keywords:** air plasma spray, laser processing, high-entropy coatings, microstructure, wear behavior

## Surface nanocrystallization of AZ91D magnesium alloy by cold spraying shot peening

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**Abstract:** A new technology for surface self-nanocrystallization of AZ91D magnesium alloy induced by cold spraying shot peening (CSSP) was investigated, and effects of surface nanocrystallization by cold spraying shot peening and heat treatment at different temperatures were investigated. The microstructure evolution was conducted using X-ray diffraction (XRD) and field emission scanning electronic microscopy (FESEM). The hardness was measured by microhardness tester. The experimental results show that surface nanocrystallization of AZ91D obtained by CSSP would lead to the increase of microhardness. Low temperature heated below 280 °C for 2 hours do not change the property obviously. However, both the microstructure and microhardness vary greatly after heat treatment at 400 °C for 2 hours.

**Keywords:** surface nanocrystallization, AZ91D magnesium alloy, cold spraying shot peening, microstructure, microhardness, heat treatment

## Preparation and flowability of shell-core-structured graphite-gray cast iron powder for thermal spray application

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**Abstract:** The oxidation loss of the graphite in a plasma-sprayed gray cast iron particle in the plasma jet will go against the enhancement in the wear resistance of the resultant coating. Therefore, to reduce the oxidation loss of the graphite in the coating, the usage of a shell-core-structured powder is considered as an effective method, which can be used to deplete the oxygen around the particle and protect the graphite in the particle from oxidizing by forming a protective layer with carbon oxides around the particle. In this work, the shell-core-structured powder was fabricated by mechanical alloying method, which is carried out by ball-milling the mixed powders of submicron graphite particles and micron cast iron particles in a planetary ball mill. The effects of rotational speed, time, and weight ratio of ball-to-powder on the structure (surface area coverage) and the property (flowability) of the powders were investigated. The surface area coverage was measured by scanning electron microscopy coupled with image analysis. While the flowability of the powders was measured by the Hall flowmeter. The results showed that, with the increase of the rotational speed and time, repeated collisions of the graphite powders, cast iron powders, ball and vessel wall with each other result in deagglomeration and delamination of the graphite powders, which contribute to obtain high degree of the surface area coverage of coated powders. Furthermore, increased surface area coverage by graphite shell leads to a increase in the flowability of the graphite-cast iron powders for self-lubricated feature of graphite shell.

**Keywords:** gray cast iron powder, shell-core-structured, surface area coverage, flowability

## **A highly porous thermal barrier coating based on Gd<sub>2</sub>O<sub>3</sub>-Yb<sub>2</sub>O<sub>3</sub> Co-doped YSZ**

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**Abstract:** Co-doped zirconia based thermal barrier coatings (TBCs) have been found to possess lower thermal conductivity and higher sintering resistance at high temperatures than current state of the art ZrO<sub>2</sub>-(7-8)wt% Y<sub>2</sub>O<sub>3</sub>. In this study, a highly porous TBC based on Gd<sub>2</sub>O<sub>3</sub>-Yb<sub>2</sub>O<sub>3</sub> co-doped YSZ was produced using a special spray powder prepared by high energy ball milling with a three-cathode plasma generator TriplexProTM-210. The coating was characterized in terms of the microstructure, phase composition, hardness and thermal conductivity. The thermal shock behavior was evaluated using thermal cyclic test at  $T=1, 150$  °C. A free-standing coating was heat-treated in a furnace at  $T=1,150$  °C for  $t=500$  h to investigate the effect of sintering on the microstructure and properties of the coating. Results revealed that the coating in as-sprayed condition exhibited a highly porous microstructure with a porosity of  $p=39\%$ . The highly porous TBC presented a better thermal shock behaviour compared to a conventional TBC. After heat-treatment, the value of porosity amounted to  $p=26\%$ , representing a highly porous microstructure. Both the thermal conductivity and hardness of the coating significantly increased due to sintering. However, the coating exhibited low thermal conductivity values of  $\kappa < 1.1$  W/m.k at high temperatures.

**Keywords:** thermal barrier coating, high energy ball milling, thermal shock, behaviour, microstructure, thermal conductivity

## **A novel non-parabolic kinetic model for long term oxidation of MCrAlY bond coat**

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**Abstract:** Thermally grown oxides (TGO) on bond coat surface play a dominant role in determining thermal cyclic lifetime of thermal barrier coatings (TBCs), which can be used in hot section parts for both advanced aircraft engine and land-based gas turbine. Generally, the TGO growth is regarded to follow parabolic kinetics, and is proved by relatively short duration oxidation experiment. In this study, a novel non-parabolic oxidation kinetic model of MCrAlY bond coat was reported based on a systematically examination of long term oxidation. To qualitatively explain the abnormal growth phenomenon of TGO, the changes that occurred to TGO microstructure during the oxidation process were studied. Based on these observations, a modified model was developed to understand and quantitatively predict the non-parabolic oxidation and growth kinetics of TGO. This new oxidation kinetic model, which fits well with experimental results, provides a novel method to quantitatively predict the long-term growth behavior of TGO, and thereby benefits the development of longlife and highly reliable thermal barrier coatings.

**Keywords:** MCrAlY bond coat, non-parabolic kinetic model

## Thermal shock resistance of thermal barrier coatings with different shapes processed by laser

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**Abstract:** Inspired by the coupling phenomenon in biological system, an attempt to improve the thermal shock resistance of thermal barrier coatings (TBCs) with different shapes on the surface was made by using the laser remelting process. The NiCrAlY/ZrO<sub>2</sub>-7wt% Y<sub>2</sub>O<sub>3</sub> double-layer structured TBCs were prepared by air plasma spraying. The microstructure, microhardness and phase composition of the as-sprayed and laser-treated specimens with different shapes were examined. The thermal shock behaviors of laser-treated specimens with different shapes including ‘dot’, ‘striation’ and ‘grid’ were investigated. The results indicated that the dot-shaped specimen had the better thermal shock resistance than the as-sprayed specimen, while the striation-shaped and grid-shaped specimen had the worse thermal shock resistance than the as-sprayed specimen. The different shaped units were characterized by the dense columnar crystal structure and the high hardness. The TBCs’ failure was due to the oxide scale growth of bond coating and the thermal expansion mismatch stress during thermal shock cycles. The inhibition of the edge effect and the effects of thermal crack deflection are the main reasons for improving the thermal shock resistance of dot-shaped specimen. However, the residual stress of laser processing leads to the bad thermal shock resistance of striation-shaped and grid-shaped specimens.

**Keywords:** thermal spray, thermal barrier coatings, thermal shock resistance, laser

## Evaporation of small droplets in plasma spray-physical vapor deposition based on plasma heat transfer

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**Abstract:** In plasma spray-physical vapor deposition (PS-PVD) process, the state variation of molten particles in plasma jet is an important factor affecting the deposition unite type and final coating structure. In this work, the properties of the plasma jet are examined by optical emission spectrometry (OES), and the results reveal that the heat flux in free plasma jet is smaller than that in the plasma torch nozzle (as reported about 108 W/m<sup>2</sup>). However, the distance of molten ZrO<sub>2</sub> flying in the open plasma jet is much longer than that in the plasma torch nozzle, so the heating in the open plasma jet can’t be ignored. Thus, an evaporation model of small molten ZrO<sub>2</sub> is established by examining the heat and mass transfer process of molten ZrO<sub>2</sub>. The results of the evaporation model show that the molten ZrO<sub>2</sub> with a diameter less than 0.28 μm and an initial temperature of 3247 K can be completely evaporated within the axial distance of 450 mm by heat transfer.

**Keywords:** evaporation, heat ux, heat transfer, PS-PVD

## Hot corrosion behavior of NiCrB coating sprayed by wire arc thermal spraying

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**Abstract:** Waste-To-Energy (WTE) technique is used to dispose solid waste worldwide. However boiler tubes bursting frequently due to aggressive gas components including  $\text{Cl}_2$ ,  $\text{HCl}$ ,  $\text{S}$ , alkali metals, and heavy metals such as  $\text{Zn}$  and  $\text{Sn}$ . Thermally sprayed coating is widely used to protect tubes such as superheater and waterwall tubes in power generation plant and waste incineration plant. In the present paper, NiCr coatings with different content of element Boron (B) (0, 0.8, 1.6, 2.5, 3.5wt%) were designed and sprayed on boiler tube steel (15CrMoV) by Wire Arc Spraying technique. The deposited thickness of the coatings were around 500  $\mu\text{m}$ . The range of coatings porosity including crack, pore was measured at 1%-4% by software Image Pro Plus analyzing 20 optical microscope (OM) photographs. Some pores and lamella structures resulting from the nature of thermal spraying coating were clearly observed. Microstructures and phase components of coatings were characterized using OM, SEM-EDS, XRD, and oxygen content analysis. The pictures and XRD patterns showed rarely porosity and crack at coating/substrate interface and the element B has a powerful influence on decreasing oxides of NiCr coatings. The hot corrosion resistance of free-standing coatings, substrate 15CrMoV and the coated were exposed at 800 °C in a laboratory test. The parameter of mixture salt and atmosphere was  $\text{Na}_2\text{SO}_4+10\text{wt}\%$   $\text{NaCl}$  and air respectively. Hot corrosion kinetics were plotted by measuring the weight gain of the specimens at 10h intervals for a duration of 200 h. The weight gain of substrate increased sharply. On the contrary free-standing coatings had obviously lower level. SEM figures showed rarely cracks and lamellar corrosion products on top of coatings surface which elements are Ni, Cr, O detected by EDS. Furthermore, Cr-deplete zone was found beneath the surface of coatings. Compared to uncoated substrate, the coated had lower weight gain. In addition, little stripping and obvious oxides was found at center of coating/substrate interface. Main corrosion production including  $\text{Cr}_2\text{O}_3$ ,  $\text{NiCr}_2\text{O}_4$  and  $\text{NiO}$  was found on corroded coating surfaces by XRD. Moreover, coating with 3.5wt% B content had Boron oxides be detected on corroded surface. Coatings had lower weight gain owe to the oxides with Ni and Cr to against further deposits deterioration. Combining with hot corrosion kinetics, XRD, SEM and EDS, NiCr coating with 0.8wt% B had a lowest weight gain and formed a lamella corrosion product comprised with  $\text{Cr}_2\text{O}_3$  along with a small amount  $\text{NiCr}_2\text{O}_4$  and  $\text{NiO}$ . Element B probably had chemical reaction with oxygen that from air during the spraying prior to Ni and Cr. Given relatively low melting point, most of boron oxide may be turning into gas from liquid to be the ash and smoke and only a little amount left in coatings. Then relatively more amount element Ni and Cr without be oxidized preserved and formed the coatings. Starting from the coating/gas and coating/melting-salt interface, corrosion media diffused into coating through connected porosity and crack to deteriorate coating. Meanwhile element Cr could continuously diffused to form oxides an the interface between the corrosion product and the coating from inner coating.

**Keywords:** hot corrosion resistance, wire arc spraying, NiCrB coating, waste to energy

## Research on crack failure modes of thermal barrier coatings based on acoustic emission technique

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**Abstract:** The thermal barrier coatings is a typical brittle, heterogeneous, multilayer structure material. During the service process, it is mainly affected by the heat-force load, which will lead to premature peeling failure of the coating. The main failure modes is the cracking of the ceramic coating and the failure of the interface peeling. In general, the various failures in the thermal barrier coatings are mainly the accumulation of microcrack initiation, expansion and connectivity. In this paper, the failure process of thermal barrier coatings under tensile load is studied by using acoustic emission Technique combined with microscopic morphology observation, and the crack damage modes of thermal barrier coatings is identified. Firstly, using the acoustic emission characteristic parameter analysis method (such as the number of acoustic emission events, amplitude), the failure process of the thermal barrier coatings is divided into several different stages. Secondly, according to the observation of the microscopic morphology under different loads, it can be viewed directly. The relationship between the acoustic emission characteristic parameters and the crack damage failure information is established until the failure process of the thermal barrier coatings under tensile load. Finally, the acoustic emission waveform signal is transformed from the time domain to the fast Fourier transform (FFT). In the frequency domain, then compare the number of peaks of different waveforms and the frequency values of each peak, and then observe the damage pattern of the coating by microscopic morphology. The results show that the surface failure of the thermal barrier coatings is formed in the surface layer of the ceramic layer. As the matrix deforms, the surface crack rapidly expands to the interface of the ceramic layer/bonding layer, and the number of cracks gradually increases to saturation. It also gradually widens. When the surface crack propagates to the interface of the ceramic layer/bonding layer, the crack will grow and expand along the interface, causing the thermal barrier coatings to delamination. As the load increases, the ceramic layer/bonding layer The discontinuous interface cracks on the interface are connected to each other and eventually cause spalling of the ceramic layer; the parameter analysis of the detected acoustic emission signal is performed, and the failure process of the thermal barrier coatings is divided by the event counting and amplitude distribution of the acoustic emission signal. For the fourth order, the microscopic morphology observation shows that the first stage of failure is the elastic deformation stage of the matrix, the second stage is the surface crack initiation stage, the third stage is the interface crack growth stage, and the fourth stage is the interface crack large area spalling stage. The spectrum analysis shows that the frequency component of the substrate is around 0.025 MHz, and the frequency component of the surface crack is about 0.20-0.25 MHz.

**Keywords:** thermal barrier coatings, crack propagation, fast Fourier transform, failure modes

## **In situ synthesis of TiN reinforced Fe-based amorphous coating by reactive plasma spraying**

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**Abstract:** Titanium nitrides reinforced Fe-based amorphous coating has been successfully prepared by reactive plasma spraying technology. The phase composition, microstructure and microhardness of the composite coating were investigated in detail. The results indicate that TiN<sub>x</sub> phase is observed in the amorphous matrix and the coating is very dense with a low porosity of 1.35%. Moreover, the average microhardness of the coating is about 1586 HV0.1.

**Keywords:** in situ synthesis, Fe-based amorphous coating, reactive plasma spraying, titanium nitrides

## **The tribological and electrochemical behavior of HVOF sprayed WC-10Co-4Cr coatings**

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**Abstract:** The dry sliding wear properties of high-velocity oxygen-fuel (HVOF) sprayed WC-10Co-4Cr coatings against Al<sub>2</sub>O<sub>3</sub> at different temperatures were investigated using a pin-on-disk high-temperature tribometer. The corrosion characteristics of the coatings under different corrosive environments were also evaluated by electrochemical measurements. The evolution of the friction coefficient, wear characteristics and their mechanisms of the coatings were studied with the change of the temperature. With increasing test temperature, the coatings showed an increase in wear rate. Potentiodynamic polarization test and electrochemical impedance spectroscopy (EIS) test were carried out to compare the corrosion resistance of the coatings to that of hard chromium coating in NaOH and HCl solutions. The coating exhibited better corrosion resistance than the hard chromium coating in HCl solution. However, the corrosion performance of the coating was found inferior than the hard chromium coating in NaOH solution. The present study proposes a new understanding of the occurrence and the related mechanisms of the wear and corrosion of the cermet coatings.

**Keywords:** HVOF, WC-CoCr, wear, corrosion