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Processing and characterisation of plasma sprayed oxides: Microstructure, phases and residual stress

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ABSTRACT

Ceramic coatings of alumina, titania, chromia, alumina-13 wt% titania and zirconia powder were deposited on steel substrate using atmospheric plasma spraying technique. The coatings were characterized for their cross-section, surface morphology, phase composition, indentation fracture toughness, elastic modulus and residual stresses. The nanoscopic features of some coatings were captured using transmission electron microscopy. The coating porosity was found to depend on the melting point of the powder and also the powder size. Equi-axed grains were found to grow in the plane of the coating. All coatings showed tensile surface residual stress. The magnitude of this stress was found to depend on the elastic modulus of the coating material and coating defect density.

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1. Introduction

Atmospheric plasma spraying is a very versatile process for fabrication of thick coatings. The working principle of this process involves injection of particles into a plasma created by ionising gases in an electric arc and then accelerating them towards the substrate at a high velocity. As the consumable material is heated in flight, it transforms to a plastic, fully-molten or semi molten state before reaching the substrate. The particles, i.e., the precursor powders, impinge on the substrate surface with a high impact energy, flatten and form thin platelets known as splats [1–4]. These splats adhere to each other and to the substrate, to form a lamellar structure upon deposition of successive layers. It is difficult to deposit ceramics directly on steel substrates owing to the difference in coefficient of thermal expansion (CTE) between the metallic substrate and the ceramic top coat [5,6]. In such cases, a metallic bond coat is applied first on the metallic substrate followed by the ceramic top coat.

The purpose of ceramic coating deposition is to endow the metallic substrate with a property that it does not have. Depending on the composition, these coatings are resistance to wear, heat and chemical attack. Examples of such oxide coatings are alumina, titania, zirconia, chromia, alumina-titania, etc. Alumina and alumina-titania coatings are suitable for wear applications owing to their high hardness and chemical stability, even at elevated temperature [7–10]. Titania has reasonably high hardness and cohesive strength. It is used to control abrasive wear, erosion and fretting wear of engineering components like pump seal, shaft bearing sleeve [11,12]. Plasma sprayed zirconia coating is extensively used as a thermal barrier (TBC) and wear resistance coating [13,14]. Chromia is a well-known corrosion resistant coating material [15]. This coating is used in IC engines, pumps, and printing rolls. Chromia coating has also been used effectively to protect piston ring surfaces [16,17].

Plasma sprayed coatings have several limitations. First, such coatings usually harbour residual stresses. Residual stress poses a problem in plasma sprayed coatings when its magnitude goes above the coating's adhesive strength. This leads to the failure of the coating either by flaking off from the substrate or cracking [18,19]. Residual stress plays a key role in determining the durability of the coating since it has adverse effects on strength, resistance to thermal shock, fatigue, erosion etc. [20].

The present work was undertaken to study the effects of the parameters of spray process, i.e., particle size and power variation and chemistry of ceramic powders on the coating properties. The effect of variation in particle size and arc power was studied for alumina powders of two size fractions (standard and fine) with respect to the changes in cross sections, hardness, porosity, phase transformation and magnitudes of residual stresses of the coatings. Next, the investigation was extended to other plasma sprayed oxide ceramic coatings of industrial significance. An extensive literature survey revealed little information on residual stresses of certain coatings like chromia, titania and alumina-titania. Further, it was observed that few published reports are available on the physical properties, microstructures and phases of chromia and titania coatings. In addition, the data available on oxide coatings are scattered across the literature and very few comprehensive reports involving all oxides are available. Hence, it was felt appropriate to carry out a comprehensive study of the primary properties of oxide coatings deposited and characterized using the same set of equipment and instruments.

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2. Materials and methods

Test coupons of dimension 10 × 10 × 100 mm were sliced from AISI 1060 bright steel square bars. They were grit blasted inside a grit blasting chamber (Sandstorm, Bangalore, India) using alumina grits of mesh size 24 at 100 psi (0.7 MPa) blasting pressure with a standoff distance of 100 mm. The average roughness (R_a) of the grit blasted surface was 6.1 ± 0.3 μm. The roughened samples were ultrasonically cleaned for 10 min in a 2-propanol solution. Ceramic oxide coating was deposited immediately after cleaning process. Six ceramic top coat and two bond coat powders were used in this study. Particle size range, chemical composition, synthesis method and source for each ceramic oxide are listed in the Table 1. The NiCrAlY bond coat was used for YSZ top coat only. Coatings were deposited using a Sulzer-Metco 9 MB plasmatron mounted on a CNC X-Y table. Prior to coating deposition, the grit blasted substrates were preheated to a temperature of 150–200 °C. This temperature was monitored using a non-contact type pyrometer. Then a layer of bond coat of thickness 80–100 μm was deposited on the steel substrate. This was followed by a top coat of thickness 300–600 μm. The plasma spraying parameters are listed in Table 2. The feedstock ceramic powder was injected into the jet through a vertical nozzle (θ = 90°, nozzle diameter = 1.85 mm). The spray parameters and carrier gas flow rate were selected from the list of parameters recommended by the powder manufacturer (Sulzer Metco user manual).

For the microstructural investigation, samples of dimensions 10 × 10 × 10 mm were sliced from the sprayed sample using a low speed diamond saw (150 low speed diamond, MTI Corp.). These samples were subsequently hot mounted and polished metallographically. For powder morphology and cross sectional observation, a scanning electron microscope (SEM) (Zeiss EVO 18, Germany) and an optical microscope (Zeiss Axio Vert.A1, Germany) were used. Investigation of phases was undertaken using a high resolution X-ray diffractometer (PANALYTICAL PW 3050/60 X'Pert, Netherlands) generating Cu Kα radiation. The coating hardness was measured using a hardness tester (LECO LM700, USA) and an average of ten hardness readings were considered for each coating. Porosity of the top coat was measured from the images of the cross sections using AXIOVISION image analysis software. Surface roughness of the coating was measured using a contact type surface roughness tester (TAYLOR-HOBSON Surtronic 3+, AMETEK Inc., UK). The sampling length for roughness evaluation was set to 4 mm. The coatings were also studied under a transmission electron microscope (TEM). For TEM sample, a 3 mm slice was removed from the coating by a low speed saw and polished using fine grade polishing papers to a thickness of 100 μm. Next the samples were ion milled using an ion beam milling system (Gatan 691, USA), in order to reduce the sample thickness to 20 μm approximately. TEM observation was made under a JEOL JEM-2100 high resolution transmission electron microscope. Indentations were also made on the sample cross sections under a load of 1.962 N (≈200 gf), using a micro hardness tester (MVH-S-AUTO, Omnitech, India). The purpose of these indentations was to generate cracks for the measurement of indentation fracture toughness. These cracks and imprints were observed under optical microscope (Zeiss Axio Vert.A1, Germany). The cracks and diagonal lengths were measured using AXIO VISION (Zeiss, Germany) and Image J (NIH, USA) software. Depth sensing indentation were performed using an instrumented hardness tester (Micro Combi Tester (MCT), CSM instruments, Switzerland) equipped with a Vickers indenter. The indentation measurements were undertaken under a maximum load of 2000 mN, loading and unloading rate of 4000 mN/min and with a dwell time of 15 s [21]. Indentation fracture toughness (K_{IC}) values were calculated using the following expression proposed by Evans and Charles [22]

$$K_{IC} = 0.15k\left(\frac{c}{a}\right)^{-3/2}\frac{H\sqrt{a}}{\phi} \quad (1)$$

where k = correction factor 3.2 for ceramics, ϕ = constraint factor ≈3, H = Vickers hardness (MPa), a = half of Vickers diagonal length measured in the direction of crack (m), c = sum of length of the crack and half of the Vickers diagonal in the direction of the crack (m). This method can be used very effectively for comparison of toughness of brittle ceramic coatings having similar microstructure and order of toughness [23]. The calculated values are in good agreement with already reported results. The elastic moduli of the coatings were calculated from the slope of the unloading part of load-displacement curve [24]. Residual stresses were measured using a PW1710 Philips X-ray diffractometer (Philips, Netherlands) equipped with Co target. Samples of size 10 mm × 10 mm were cut off from coated test coupons. Measurements were done in seven tilt angles in 0° to ±45° range. Uniaxial residual stress magnitudes were determined along the plasma gun movement direction. The signal detection unit was managed using a Philips X'pert Data Collector software for a given range of 2θ values (2θ is the angle between source and diffracted X-ray beam), chosen to encompass the Co-Kα doublet for these specific plane (e.g. for Al_2O_3 coating, the corresponding 2θ range is 108° to 112° respectively).

3. Results and discussion

3.1. Powder morphology and phases

Fig. 1(i)–(ii) shows the secondary electron images of fine alumina and alumina powders. Both powders were produced by fusing and crushing technique and hence have angular and chunky morphology. Fig. 2(i)–(ii) also shows the X-ray diffractions of both fine alumina and coarse alumina powders. X-ray diffraction of the powder shows peaks from a stable hexagonal α-phase only.

Conventional alumina-13 wt% titania feedstock powder was also produced using crushing and fusing technique. The particle size range of the powder is - 45 + 5 μm (Table 1). It is clear from Fig. 1(iii) that, they are angular and blocky in nature. Fig. 2(iii) shows the X-ray diffraction for conventional alumina-13 wt% titania feedstock powders. The two constituents, alumina and titania exist in α-phase and anatase form, respectively.

The secondary electron image of the titania particles (Fig. 1(iv)), shows the fused and crushed powder particles. It has a shape similar to that of the alumina powders. X-ray diffraction peaks of titania shown in Fig. 2(iv) confirms the presence of the rutile phase along with peaks from other phases, namely, anatase, brookite and magneli.

Table 1
Ceramic powders used coating.

Sl no	Powder name	Formula	Size (microns)	Morphology	Source
1	Fine alumina	Al_2O_3	−31 + 3.9	Crushed	Sulzer Metco, Westbury, NY, USA
2	Alumina	Al_2O_3	−45 + 15	Crushed	
3	Alumina–titania	Al_2O_3-13 wt% TiO_2	−45 + 5	Clad	
4	Titania	TiO_2	−88 + 7.8	Crushed	
5	Chromia	Cr_2O_3	−125 + 11	Crushed	
6	Yttria stabilized zirconia	YSZ	−75 + 45	Spheroidal	
7	Nickel-aluminum	Ni 95, Al 5	−90 + 45	Clad	
8	Ni-Cr-Al-Y	NiCrAlY	−106 + 53	Spheroidal	

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