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Alumina-zirconia coatings obtained by suspension plasma spraying from highly concentrated aqueous suspensions

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ABSTRACT

Suspension plasma spraying (SPS) deposition represents an innovative technique to produce coatings that exhibit improved properties. However, the key to obtain coatings with superior functional properties relies on the investigation of the suspensions as starting materials. For this reason, the present work deals with the suspension preparation for SPS process and its influence on the resulting coatings.
Laboratory-prepared 60/40 wt% alumina-zirconia suspensions were concentrated to avoid energy loss and were then successfully deposited by SPS technique. The liquid used was water instead of ethanol due to economical, environmental and safety reasons. The preparation of the suspension plays an important role in SPS process since stable and well-dispersed water suspensions are difficult to obtain. For this reason, colloidal behaviour characterisation of the starting particles as well as rheological optimisation of the feedstock suspensions was addressed in this research.
Suspensions with different solid loadings (up to 30 vol.% or 72 wt%) were deposited using several spraying distances. All coatings displayed a bimodal microstructure consisting in partially melted zones surrounded by a fully melted matrix. α-Al_2O_3 and t′-ZrO_2 constituted the main crystalline phases, but differences in the microstructure and properties of the coatings were observed. From these results, some relations between starting suspension and spraying parameters with coating characteristics were found. Thus the optimal spraying distance becomes shorter when the suspension solid loading increases.

1. Introduction

Yttria-partially stabilised zirconia (YSZ) are currently used for the fabrication of thermal barrier coatings (TBC) because of their low thermal conductivity and good mechanical properties at high temperature. However, YSZ undergoes phase degradation after long in-service times at relatively high temperatures, limiting the operational temperature of the coatings and their lifetime [1]. One possible solution addressed in previous works consists in adding alumina to zirconia stabilised with different yttria contents. This addition enhances the thermodynamic stabilisation and improves the mechanical properties and thermal fatigue [2,3].

Atmospheric plasma spraying (APS) represents one of the most suitable techniques used to obtain TBCs as a consequence of the good mechanical and thermal performance of the coatings obtained as well as an easy scaling-up of the technique [4]. It consists in the injection of a powder into a plasma flow where this powder is later subjected to melting and then accelerated until it impacts on the substrate. The main problem of the APS technique lies in the starting powder preparation because the feedstock must meet several requirements (an agglomerate apparent specific mass higher than 1700 kg/m^3 or a mean agglomerate size coarser than 60 μm). Furthermore, their production is costly [5,6]. One alternative to conventional APS technique, known as suspension plasma spraying (SPS), consists in spraying fine-particle suspensions instead of coarse powders. SPS coatings display smaller microstructural defects, which can lead to improved properties. In the case of TBCs, SPS technique also allows the reduction of the thermal conductivity due to a higher porosity made up of finer voids [7,8].

The SPS process has been intensively studied in the last years. However, more research in this technique is necessary to tackle its main problems. One of them deals with the preparation of suitable feedstock suspensions so that they could be industrially scalable. Suspensions must be well-dispersed and stable in order to avoid particle agglomeration or sedimentation. Besides suspension viscosity cannot be too high for the suspension to properly flow through the injector. However it is interesting to note that solids loading of the suspension should be as high as possible so as to improve the deposition efficiency [9,10].

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Concerning the suspension liquid the use of water instead of organic solvents would be preferred due to economical, environmental, and safety reasons. Nevertheless, most of the previous research about SPS deposition employs organic liquids (ethanol) due to their lower vaporisation enthalpy. Furthermore, when water is used suspension stabilisation becomes more difficult and therefore, a previous colloidal behaviour characterisation should be a mandate in these cases.

On the other hand, suspension solid loading is usually low for reducing the power necessary to evaporate the solvent and to melt the solid [5]. However, Vicent et al. demonstrated that power requirements become lower when solid loading is increased in the case of water suspension, in opposition to what occurs in ethanol suspensions [11]. Therefore, high solid loading suspension deposition is convenient in terms of energy and efficiency, so that thicker coatings can develop with less spraying passes. However increasing solids content leads to higher viscosity suspensions as a consequence of the exponential growth of viscosity with volume fraction of solids [12]. Moreover, some authors point out that coating properties are strongly dependent on suspension characteristics, finding a relationship between solid loading and coating properties [13–15]. Because of the above achieving all feedstock suspension requirements (no agglomeration, long-term stability and low viscosity) with high solid content suspensions as well as assessing the impact of suspension characteristics on coating microstructure and properties is certainly a challenge.

Apart from increasing the solid loading, the possibility of depositing a eutectic composition can also reduce the power requirements. In the case of the alumina/zirconia system, the eutectic composition is 58/42 wt%. However, no equilibrium phases form in plasma processes owing to rapid melting inside the plasma plume as well as fast cooling on the substrate surface. Some authors have found a pseudo-eutectic point for a composition of 60/40 wt% to Al_2O_3/ZrO_2, where the temperature to melt the solid inside the plasma plume is minimum [3,16]. Hence, a reduction of plasma enthalpy requirements is achieved by depositing this pseudo-eutectic composition. In previous works, Al_2O_3/ZrO_2 coatings by plasma spraying from powders were successfully developed [2,16]. However, Al_2O_3/ZrO_2 coatings by SPS have been less addressed. Besides when SPS coatings were researched the corresponding feedstocks suspensions were too diluted (30 wt%) for industrial scale [3,17]. For this reason, the aim of the present work is to develop a thermal barrier coating by SPS technique from aqueous Al_2O_3/YSZ suspensions with low power consumption by using concentrated suspensions of a eutectic composition.

2. Experimental

2.1. Materials

Two commercial submicron-sized powders were used as starting materials: (1) an α-Al_2O_3 (Condea-Ceralox HPA-0.5, Sasol, USA) with a mean particle size of 0.35 μm and a specific surface area of 9.5 m^2/g; and (2) a Yttria-tetragonal zirconia polycrystal (TZ-3YS, Tosoh Co., Japan) with a Y_2O_3 ratio of 3 mol% to stabilise the tetragonal phase, mean particle size of 0.4 μm and specific surface area of 6.8 m^2/g. In addition, a commercial salt (DURAMAX™ D-3005, Rohm & Haas/Dow Chemicals, USA) of polyacrylic acid-based polyelectrolyte (PAA), with 35 wt% active matter, was used as dispersant in order to achieve the colloidal stability of the powders in water. This dispersant has been used in other previous works with alumina and zirconia powders [18,19].

2.2. Suspension preparation and characterisation

Suspensions were prepared adding first 60 wt% Al_2O_3 particles and then 40 wt% Y-TZP particles to the dispersing medium (optimal quantity of dispersant into water). In order to breakdown any present agglomerates, the suspensions were dispersed with a sonication probe (UP 400S, Dr Hielscher GmbH, Germany). Suspensions with different solid loadings were prepared.

The colloidal stability of Al_2O_3 and Y-TZP powders in aqueous suspensions was studied by zeta potential measurements as a function of dispersant content and pH. The equipment used to measure the zeta potential is based on the laser Doppler velocimetry technique (Zetasizer NanoZS, Malvern, UK). In order to perform the measurements, diluted suspensions with a 0.01% solid content and 0.01 M KCl as electrolyte were prepared. The pH values were determined with a pH-meter (Titrino DMS 716, Metrohm, Switzerland) and they were adjusted with HCl and KOH solutions. Zeta potential measurements are suitable to establish the optimal amount of dispersant in the suspensions [12,18].

The rheological behaviour of all suspensions was determined using a rotational rheometer (Bohlin CVO 120, Malvern Instruments, Great Britain) operating at controlled shear rate (CR) by loading the shear rate from 0 to 1000 s^{-1} in 5 min, maintaining it at 1000 s^{-1} for 1 min and downloading from 1000 to 0 s^{-1} in 5 min. The measurements were carried out at 25 °C using a double-plate system. The ageing effect of the suspensions was also assessed by determining the rheological behaviour at 1 and 7 days after suspension preparation. During this time, suspensions were maintained agitated in a low speed orbital shaker [20].

2.3. Coating deposition

Coatings were obtained by suspension plasma spraying technique (SPS). A plasma torch (F4-MB, Oerlikon Metco, Germany) operated by an industrial robot (IRB 1400, ABB, Switzerland) was adapted in order to spray suspensions by mechanical injection assisted by air pressure with a maximum pressure of 6 bar. More details about SPS adaptation have been described in a previous work [21]. In this study, suspensions inside a vessel were agitated by a magnetic stirrer to avoid possible sedimentation and a viscosity rise in the case of thixotropic suspensions. Furthermore, two injectors with different diameters (150 and 200 μm) were utilised depending on the suspension viscosity. Suspensions with different solid loadings (from 10 vol.% up to 30 vol.%) were deposited using different spraying distances in order to study the influence of starting suspension characteristics, as well as spraying parameters.

Suspensions were deposited on an austenitic cylindrical stainless steel (AISI 304) substrate with a diameter of 25 mm. Prior to deposition, the substrates were grit-blasted with corundum at a pressure of 4.2 bar and cleaned with ethanol to remove any remaining dust or grease from the surface. The substrates were preheated between 300 °C and 350 °C with the plasma torch to enhance coating adhesion. The spraying parameters used in this work are specified in Table 1.

2.4. Coating characterisation

First, polished coating cross-section microstructures were observed by FEG-SEM (S-4800, Hitachi, Japan). Coating thickness was measured, as well as porosity and cracks quantified, by image analysis from 10

Table 1
Main spraying parameters of Al_2O_3/Y-TZP coatings.

	Suspension solid loading (vol.%)	
	10; 15; 20	15
Ar flow rate (slpm[a])	37	
H_2 flow rate (slpm[a])	8	
Intensity (A)	700	
Linear speed (m/s)	1	
Feedstock flow rate (ml/min)	27	
Number of spraying passes	4	
Injector diameter (mm)	0.15	0.2
Spraying distance (mm)	30; 40; 50	50; 60

[a] slpm: standard litre per minute.