

Rheological behaviour of submicron mullite–carbon nanofiber suspensions for Atmospheric Plasma Spraying coatings

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Abstract

Mullite is widely used as a structural material for applications like thermal and environmental barriers coatings. For some of these applications, thermal spray is a suitable technique due to its fast production time and versatility. This makes mullite a very interesting coating material for thermal spray industry. In the present work, the viability to produce coatings by thermal spray using mullite–CNFs agglomerated powders is analyzed. The stability of aqueous mullite and mullite–CNFs suspensions was studied in terms of zeta potential and rheological behaviour of concentrated slurries. Slurries were optimized in terms of dispersant concentration and solid content. The optimized suspensions were used for the granules preparation by spray drying technology. The obtained granules were characterized through the determination of particle size distribution and shape factor by field emission scanning electron microscopy and laser scattering. These granules were used to form the coatings by Atmospheric Plasma Spraying which were characterized by evaluating the composition, structure, shape, and thickness.

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1. Introduction

There is a broad body of work dealing with the design and fabrication of aluminosilicate-based materials (within the Al_2O_3 – SiO_2 system) which have received great attention due to their high potential as catalyst supports, catalysts for industrial processes, substrates for electronic devices, and coatings in advanced materials applications. In particular, among the various aluminosilicate phases, mullite ($3\text{Al}_2\text{O}_3$ – 2SiO_2) has been found to possess many of the properties – such as low thermal expansion and conductivity, low dielectric constant, excellent creep resistance, robust chemical and thermal stability, good high-temperature strength and oxidation resistance – that are required for ceramics for high temperature engineering and electronic packaging applications, or to be used as a matrix for advanced composites.^{1–4} The enhanced properties achieved for mullite-based coatings make mullite to be a brave candidate to

improve materials by proper surface engineering control. However, the main limitation of mullite coatings onto metals comes from the relatively high tendency to cracks formation.⁵ The spontaneous cracking can be associated to two main reasons. The first one is the higher thermal expansion coefficient of the metal substrate. The second one is related to the intrinsically brittle nature of mullite.

Reinforced mullite materials have been largely studied in the past, by introducing either oxides (Al_2O_3 , ZrO_2 , etc.) or non oxides (Si_3N_4 , SiC , etc.).^{6,7} In this way, the current development of carbon nanofibers (CNFs) have supposed a revolution in the design of reinforcing mechanisms for ceramics, and opened new paths to improve mechanical properties in ceramics.⁸ CNFs are carbon materials that present a cylindrical shape similar as carbon nanotubes (CNTs) but with differential structural and textural characteristics, diameters in the range of 100–200 nm, length up to 500 μm and bundles with a diameter less than 1800 μm . One positive characteristic of CNFs with respect to CNTs is the easier production technology existing for the former.⁹ However, unmodified CNFs are subjected to aggregation into packed ropes or entangled networks due to the strong inter-fiber van der Waals attraction,^{10–12} which acts as

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an obstacle to applications and constitutes the main concern of this study. Different approaches have been proposed to improve the dispersion of CNFs into ceramic matrices such as the use of ultrasounds in diluted suspensions, milling techniques, functionalization by chemical routes, colloidal processing or sol–gel technology.^{13–16} In addition to aggregation problems, CNFs themselves cannot be sprayed by Thermal Spray techniques because they will be burn-out at the spraying temperature.

In the present work, owing to the difficulty to prepare mullite by sol–gel techniques, a micron-sized mullite powder has been selected as starting material. This powder has been attrition milled down to a submicronic size. It is well known that submicron powders could not be directly applied for thermal spraying process due to their poor fluidity and low packing density. Therefore, the preparation of a suitable feedstock containing submicron sized particles for thermal spray requires their controlled agglomeration by spray drying methods and, in some cases, the consolidation of such agglomerates by a thermal treatment, this being the so-called reconstitution process.^{17–19} The aim of the present work was to prepare homogeneous CNFs-reinforced mullite suspensions suitable for granules production in order to obtain coating using Atmospheric Plasma Spraying (APS). For this purpose, milled mullite powders were mixed with CNFs to obtain concentrated aqueous suspensions, whose stability was optimized in terms of their rheological behaviour. Optimization from slurries was made by adjustment of additive content and solid content. Agglomerates from CNFs-mullite suspension were produced by spray-drying and used for coating production by APS technique.

2. Experimental

2.1. Starting raw materials

Mullite powder (MUBS) with nominal stoichiometry $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$, and composition 0.1 wt.% Fe_2O_3 , 0.1 wt.% CaO and 0.4 wt.% NaO was used in this study. This powder was obtained by electric arc and was supplied by Elfusa Trading SL (Málaga, Spain). Mullite powder was attrition milled in water using alumina jar and alumina balls of 5 mm in diameter. After milling, powder was dried for 6 h at 80 °C till constant weight.

CNFs were supplied by Grupo Antolin Ingeniería (Burgos, Spain). CNFs were manufactured by vapour phase growth,²⁰ through decomposition of hydrocarbons in the gas phase in the presence of catalytic particles of nickel. Grupo Antolin Ingeniería has a patent about the product as GANFs carbon nanofibers. From manufacturing process, nickel impurities are observed in final CNFs.

2.2. Colloidal behaviour

The colloidal stability of aqueous suspensions of mullite and CNFs was studied by measuring the zeta potential as a function of pH and deflocculant content using a Zetasizer NanoZS instrument (Malvern, UK), based on the laser Doppler velocimetry technique. HCl and KOH were used to change the pH, and KCl 10^{-2} M was used as an inert electrolyte. An ammonium

salt of Polyacrylic Acid (PAA) was used as a deflocculant with additions of 0.5, 0.8, 1.0, and 1.5 wt.% (on a dry solids basis). No binders were added to avoid the presence of undesired organic compounds in the final structure of the granules, as this can be deleterious or can complicate further processing steps. Suspensions for zeta potential measurements were prepared to a powder concentration of 10^{-1} g/l and sonicated for 1 min with an ultrasound (US) probe (UP 400S, Dr. Hielscher GmbH, Germany) in order to prevent agglomeration. Measurements were performed on the fresh, as-prepared suspensions.

2.3. Rheological study

Concentrated milled mullite suspensions with solids loadings of 30 and 40 vol.% and different contents of PAA (0.8, 1.0 and 1.5 wt.%) were prepared. Concentrated suspensions of milled mullite/CNFs mixtures were also prepared with solids loadings of 30 and 40 vol.% and different contents of PAA (0.8, 1.0 and 1.5 wt.%) using relative contents of CNFs of 1 and 2 wt.% with respect to mullite mass. The rheological behaviour of all prepared slurries was performed with a rheometer (Haake RS50, Thermo, Germany) operated at controlled shear rate (CR) conditions. The sensor system consisted on a double-cone rotor (with a cone diameter of 60 mm and cone angle of 2°) and a stationary plate, this system being surrounded by a cylindrical wall. The chamber is protected with a solvent trap to reduce evaporation phenomena. Measurements were performed by increasing the shear rate from 0 to 1000 s^{-1} in 5 min, maintaining at 1000 s^{-1} for 2 min and returning to 0 in 5 min. Temperature was maintained constant at 25 °C.

2.4. Granulation

Optimized suspensions were spray dried by a NIRO Mobile Minor atomizer (GEA NIRO, USA) to obtain granules. This atomizer evaporated a maximum volume of 7 kg water/h. Suspension was sprayed by a pneumatic nozzle placed at the bottom of the apparatus. A nozzle diameter of 2 mm and air pressure of 0.8 bars were chosen as spray drying parameters.

2.5. Thermal spray

As-obtained agglomerates were thermal sprayed using an APS A-3000S system with an F4 plasma torch (Sulzer Metco, Germany) with a theoretical plasma temperature of 11,000 °C, spraying distance of 120 mm and cooling at room temperature. Five layers were deposited on a 3061 steel substrate. Spraying conditions were 35:13 l/min Ar:H₂ gas ratio with 4.5 l Ar/min gas flow, at 120 mm and an intensity of 600 A.

2.6. Characterization techniques

The physico-chemical characterization of initial and milled mullite powder was performed by measuring the particle size distribution, by using the laser diffraction (LD) technique (LS 13320, Beckman Coulter, USA), and the specific surface area, which was determined using the single-point BET method

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