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Thermal plasma spheroidization of aluminum oxide and characterization of the spheroidized alumina powder

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Abstract

Spheroidization of aluminum oxide powder was done by thermal plasma processing. The powder was injected into the plasma jet issuing out of a DC plasma torch. Trajectories of the particles in the plasma jet were seen using a high speed camera and then in-flight velocity and temperature of alumina particles were determined using a 'Spray Watch' system. Characterization of the spheroidized powder was done by Scanning electron microscopy (SEM) and X-ray powder diffraction (XRD). The results showed that increase of the plasma torch power leads to increase in the extent of spheroidization and conversion to γ-alumina. Results obtained showed that the process can be extended to synthesize free flowing alumina powder for thermal spray applications.

Keywords: In-flight particle temperature; Metastable alumina; Plasma processing; Spheroidization

1. Introduction

Ceramic and metal powders with spherical morphology are used for a variety of applications that require very good powder flow characteristics. Spheroidization of ceramic and metal powders is carried out by many processes [1–5] including thermal plasma processing. A plasma spheroidization process results in considerable improvement of the properties of the powder in multiple aspects like improving powder flow-ability and decreasing internal porosity that make it ideally suitable for plasma spray applications.

Thermal plasma synthesis has been successfully applied to generate spheroidized powders of a variety of metal and ceramic powders. High temperature and highly concentrated enthalpy available in the plasma jet can melt and vaporize virtually any refractory material [6]. The high quench rate favors homogeneous nucleation resulting in spheroidized particles. Particle injection is an important parameter that affects the trajectory of the particles in the plasma jet and therefore, the degree of spheroidization. The powder entry point and carrier gas flow should be properly chosen to ensure that all the particles injected into the plasma land at the central line of the plasma jet. Besides particle injection, the degree of spheroidization also depends on the plasma power, plasma gas flow rate and powder feed rate. In general, any parameter that enhances particle melting increases the extent of spheroidization [7–10]. For instance, as the input power increases, the extent of melting increases and therefore the spheroidization also increases.

Spheroidization of aluminum oxide powder in a thermal plasma jet is reported in the present work. Temperature and velocity of the alumina particles exiting the plasma jet were determined online. The spheroidized product was characterized by X-ray diffraction for phase analysis and scanning electron microscope for size and shape. The effect of plasma torch power on the shape, size and phase composition of the spheroidized powder was studied.

2. Plasma reactor and experimental methods

A 40 kW DC plasma processing system, developed in-house, was used for the experiment. The system uses a plasma torch consisting of a tungsten rod type cathode, 10 mm

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diameter with a conical tip and copper anode shaped in the form of a nozzle(7 mm diameter) to generate the plasma. Argon gas (30 SLPM) was used as the primary gas and N_2 (3 SLPM) was used as the secondary gas. Alumina powder (15–38 μm), stored in a turn-table type powder feeder, was injected through an internal injector located at the anode of the plasma torch (shown in Fig. 1) using argon (10 SLPM) as the carrier gas. The carrier gas flow was not changed based on the findings of Vardelle et al. [11], who observed that the mean trajectory of the particles in the size range of 5–45 μm zirconia particles and 5–22 μm chromium oxide powder particles did not change with the carrier gas flow rate for internal powder injection. Further, it was found that the velocity of small particles at the injector exit was nearly constant over a certain range of carrier gas flow rates. We have also observed during high speed photography of spray experiment that the powder trajectory did not show any practical deviation of the particles from the plasma jet axis. The plasma was initiated by a high frequency igniter and the plasma power was controlled by regulating the plasma gas flow and current.

In order to see the effect of plasma torch power on plasma spheroidization, experiments were carried out at different input power levels of 8–20 kW. The particles emerging out of the plasma torch nozzle were quenched in water kept in a flat bottomed stain less steel vessel at a standoff distance of 200 mm from the nozzle and then dried at 80 °C for an hour. Distance of 200 mm between the plasma nozzle and the water level was chosen to avoid splashing of water during the experiment

A high speed camera (CMOS Camera system: PCO 1200 h, PCO-TECH, Inc., United States) was used for analyzing fast processes in thermal plasma jets, and was used to track the trajectories of the particles emerging out of the plasma torch nozzle. The experimental set-up to view the trajectories of the particles consists of a high speed CCD camera, a photomultiplier tube (PMT), a digital storage oscilloscope and a PC incorporating Camware software including a driver for the high speed camera, an image processing software and digital signal processing software [12]. The images of powder laden plasma jet at various power levels in the range of 8 –18 kW were recorded using the high speed camera. Exposure time was kept 0.5 ms and the inter-frame time was 0.8 ms. CCD size of 670×411 pixels was selected so as to achieve the speed of 1300 frames per second (FPS).

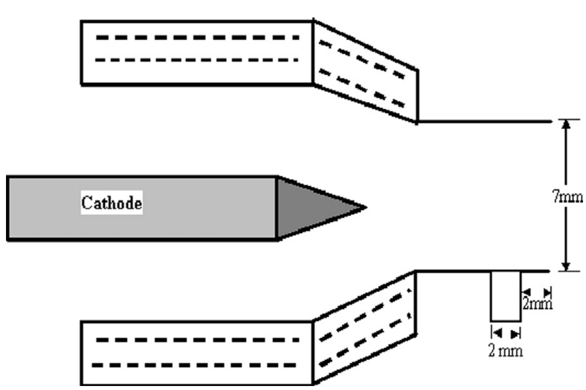

Fig. 1. Schematic of the plasma torch.

In order to determine the velocity and temperature of injected particles along the axial distance of plasma torch Oseir's (Oseir Ltd., Tampere, Finland) SprayWatch2i diagnostics system was used. The Spray Watch system measures the mean velocity and temperature of particles over a volume ensemble of 20 mm × 20 mm × 20 mm. Oseir's SprayWatch2i is an optical camera based system that images the plasma spray process and measures velocity and temperature of the particles as they exit the torch nozzle by the time of flight method and two-color pyrometry respectively. The particle trajectory was first obtained by high speed photography (as detailed in the foregoing paragraph) and then the SprayWatch2i diagnostics system was used to image the particle stream for a short duration of about 5–10 ms, which corresponds to 20–60 pixels on the CCD detector. The particle velocity is computed by the distance traveled by the particle during the time period of exposure using a special image-processing algorithm for detecting the particle traces in the image, measuring their lengths and angles and calculating the velocity of the particle. No measurement was possible up to 40 mm from the nozzle exit due to brightness of plasma jet, which eclipses the particles. Mean value and standard deviation of 25 readings (temperature and velocity) were reported.

The particles exiting the plasma jet were collected in water kept in a flat bottomed vessel of 200 mm from the torch nozzle and were characterized for phase and particle size and shape. Scanning electron microscopy (Zeiss EVO 40) was used to observe the morphologies of spheroidized powders at different input power. X-ray diffraction (Rigaku Miniflex II) was used to identify the phases formed in the spheroidized product. Ni-filtered Cu Kα radiation was used to record the diffraction pattern.

3. Experimental results and discussion

Based on the particles trajectories, the Spray Watch system was set to determine the average temperature and velocity of the particles at different locations. Fig. 2 shows a typical 'Spray Watch' computer screen showing the distribution of in-flight particle temperature, velocity, particle flux and their average. The experiment was carried out for 10 ms at various power levels and the particle velocity and temperature at two different locations were recorded. The average value of the velocity and temperature extracted from the velocity and temperature distribution curves obtained under each set of experimental conditions, at different axial locations from the nozzle exit for different plasma power levels, are shown in Tables 1 and 2. It is evident from the tables that at a given axial distance from the plasma torch nozzle, the particle velocity and temperature increase with the input power.

Results summarized in Tables 1 and 2 indicate that the particle velocity decreases with axial distance from the torch nozzle after 40 mm. A similar trend is also observed in the case of particle temperature. As the input power increases, the plasma temperature and velocity increase leading to enhanced