Morphological and structural changes of ceramic powders during plasma spraying

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Abstract

Powders with spherical particles are utilized in various industries such as health, food, or in additive manufacturing due to their enhanced flow properties. Plasma spray spheroidization is an effective tool for manufacturing of such powders from angular or agglomerated powder feedstocks, namely thanks to its high feedstock throughputs. In this paper, the formation process of spherical particles in hot plasma, the in-flight behaviour of the particles, and several powder characterization methods are described. The collection chamber for powder spheroidization using the high-enthalpy hybrid water stabilized (WSP-H) plasma torch was designed and manufactured with emphasis on maximal collection efficiency. Successful spheroidization of Al_2O_3 powder was performed and the morphology, sphericity, flowability, particle size, and chemical and phase composition of resulting powders were observed.

Key words: Atmospheric plasma spraying; Spheroidization; Flowability; Phase composition; Image analysis.

Introduction

Commonly, the plasma torch is used for preparing coatings or structures providing novel surface properties such as increased thermal, wear, or corrosion resistivity, which are hardly, if at all achievable by conventional bulk materials. These unconventional materials are prepared from, e.g., powders or liquid feedstocks [1, 2]. When a feedstock is injected into a plasma jet, it is dragged by its flow and the solid material is melted. The droplets then hit the substrate and solidify in a form of so-called splats. However, when the substrate is not present, the molten particles solidify in air at a certain distance from the jet, as their temperature decreases below the melting point of the material. Due to the surface energy of the liquid droplets of the molten material, the single particles form spheres in the air, resulting in spherical solidified particles. This process is called spheroidization. In general, spherical powders are often required for various industrial processes, e.g., in health or food industry [3]. Also, for recently fast developing additive manufacturing, the spherical powders are required to ensure their smooth flowability as the powders often need to be spread evenly and melted layer by layer [4].

This work comprises of two tasks. In the first task, a new powder spheroidization chamber was developed for hybrid water/argon-stabilized plasma torch (WSP-H). The main objective was to achieve maximal powder collection efficiency, high temperature resistance during powder collection and easy manipulation, maintenance, and cleaning in order to avoid contamination of powders. In the second task, the developed chamber was used for spheroidization of Al_2O_3 powders. The collected powders were analysed in terms of particle size and morphology, flowability, phase composition and chemical composition.

Materials and methods

Designing the collecting chamber

Experiments were carried out with various designs of collector. The schematic of the initial chamber showing the principle of spheroidization using atmospheric plasma spraying is shown in Figure 1.

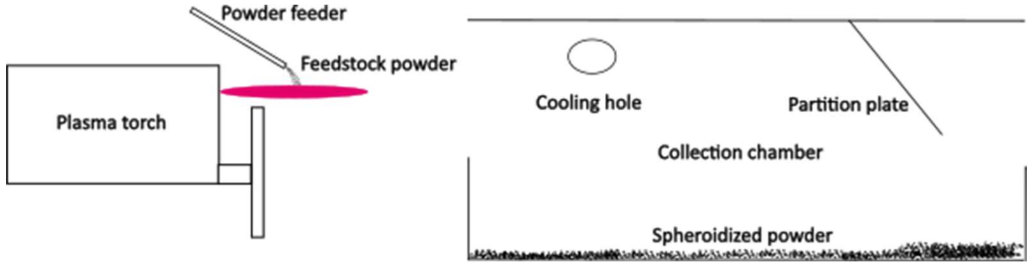

Figure 1: Schematic of the original spheroidization set-up used at Institute of Plasma Physics.

Such chamber was used in the initial spray collection experiment which was carried out in order to observe the benefits and shortcomings of the original collecting chamber, in particular the collection efficiency of the chamber. Several step-wise changes in the chamber design, such as sealing the cooling holes or adding a water cooling, were made in four iterations to achieve an optimal final design (Figure 2). With the knowledge of the amount of powder fed into the jet, the collection efficiency in percent was determined. Therefore, the progress in collection efficiency could be evaluated and the chamber designs could be compared to each other.

Figure 2: The final design of the collecting chamber rendered in Solid Edge 2022 software.

Spheroidization experiments

For spheroidization, WSP-H 500 (ProjectSoft HK a.s., Czechia) system was used. The feeding of powders was provided and controlled by two gravimetric feeders G4™ (Uniquecoat Technologies, LLC, USA). As feedstock material, Al_2O_3 Surprex AW24 (Fujimi, Japan) powder was used. Denomination of samples and the spraying parameters of spheroidization experiments are listed in Table 1. In case of A2x-SD150 experiment, the previously collected powder from A-SD150c experiment was used as a feedstock in order to evaluate the influence of repeated spheroidization of the powder.

Table 1: Spraying parameters of spheroidization experiments.

	A-SD150c	A2x-SD150
Material	Al_2O_3	Al_2O_3 (A-SD150c)
Granulometry [µm]	-75+38	~ (-75+38)
Feeding distance [mm]	45	45
Feeding rate [kg/h]	10	10
Powder collection area	Front and rear of the chamber	Front and rear of the chamber

Powders' observation and measurements

For microscopical observation, a few grams of each collected powder as well as the feedstock powders were embedded in low-viscosity epoxy resin (Epofix, Struers, Denmark). After the resin cured, the samples were grinded and polished using a semi-automated polisher Tegramin 25 (Struers, Denmark) with sandpaper and diamond suspensions according to the standard procedure used at Institute of Plasma Physics (IPP) for ceramic samples. Free surfaces of loose powders were also prepared for microscopic observation. Small amount of each powder was poured on adhesive conductive carbon tabs.

The observation of both free surfaces and polished cross-sections of powders was performed using an analytic high-resolution scanning electron microscope (SEM) Apreo 2 S LoVac (Thermo Fisher Scientific, Czechia) with Schottky field emission gun (FEG). Energy-dispersive X-ray spectroscopy (EDS) was performed using Ultim Max detector (Oxford Instruments, UK) for elemental mapping of a single particle.

The image analysis of circularity and percentage of non spheroidized particles was performed with ImageJ v.1.53k software (National Health Institute, USA). The particle size distribution was measured using laser particle size analyser Mastersizer 3000 (Malvern, UK) equipped with a wet cell. The flowability was measured according to ASTM B213 standard, using Hall flow meter with two funnels of different outlet orifice sizes, 2.5 mm, and 5 mm, to compare the flowability through different sized orifices. The evaluation of chemical and phase composition was carried out using X-ray diffraction (XRD) and X-ray fluorescence spectroscopy (XRF). The X-ray powder diffraction was performed with diffractometer D8 Discover (Bruker, Germany) with 1D detector LynxEye. For the energy-dispersive XRF, spectrometer S2 PUMA (Bruker, Germany) equipped with HighSense LE SDD detector was used.

Results and discussion

As a result of iterative modifications of the powder collection apparatus, 93 % of fed material was captured. Out of the captured material, 66 % was the powder and 27 % was unwanted coating on the partition plate. Therefore, only 7 % of Al_2O_3 evaporated or escaped from the chamber through the chimney and the chamber inlet.

The cross-sections of the angular AW24 feedstock powder as well as the processed powders can be seen in Figure 3. In the micrograph of the AW24 powder, the sharp-edged and mostly elongated particles can be seen, which reflects their anticipated production by crushing. In micrographs of A-SD150c powder, mostly spheroidized particles can be observed. However, approximately 10 % of the particles were not spheroidized (Figure 3). Any significant difference in the number of non-spheroidized particles between the powders collected in the front and the rear parts of the chamber was not observed.

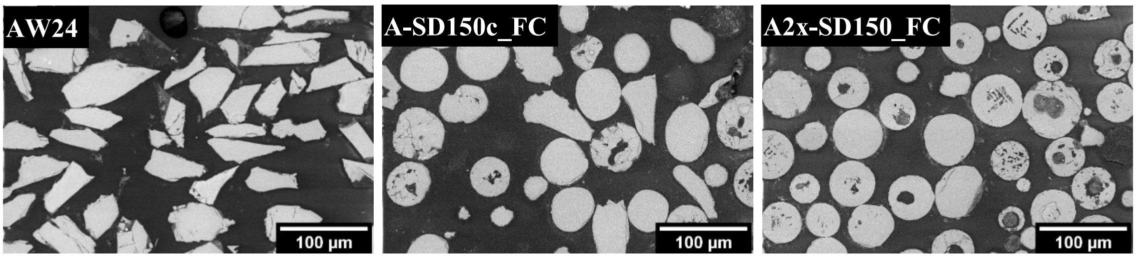

Figure 3: SEM micrographs of cross-sections of powders in BSE mode.

Ensemble results of the image analysis of the powders is shown in Figure 4. The amount of particles unaffected by the plasma jet in each sample is graphically represented together with the values of mean circularity. The values confirmed the successful spheroidization - mean circularity increased while the circularity scatter decreased after exposing the powders to the plasma jet. Moreover, the mean circularity further increased when the powder was exposed to the plasma for the second time (A2x-SD150 sample), effectively eliminating the unprocessed particles present in the powder after the first pass (A-SD150c sample).

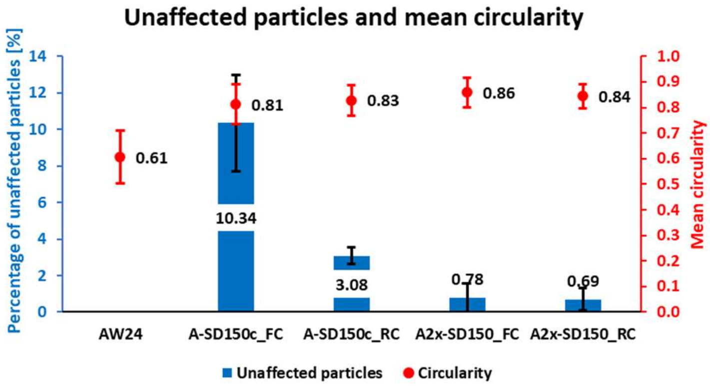

Figure 4: Percentage of particles unaffected by plasma jet and mean circularity values of the samples.

In Figure 5, the measured particle size distributions for each powder collected separately in the front (FC) and rear (RC) part of the collecting chamber are graphically represented. The measured values for A-SD150c and A2x-SD150 powders showed a significant decrease in particle size of coarser grains and overall narrowing of the distribution when compared to the original AW24 feedstock. The effect of repeated spheroidization process on particle size was virtually negligible, demonstrating that Al_2O_3 undergoes a very limited (if any) evaporation during plasma treatment.

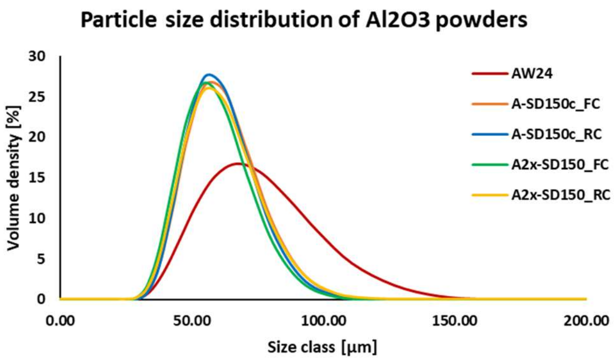

Figure 5: Particle size distribution curves of the Al_2O_3 powders.

The flowability of AW24 powder was immeasurable with Hall flowmeter as the powder got stuck in both funnels and could not flow through the orifice. After spheroidization, the flowability of powders was measurable and the measured values are shown in Figure 6. The flowability of the repeatedly spheroidized powder, A2x-SD150, increased slightly with respect to the A-SD150c powders and the increasing trend was confirmed by both funnel sizes. The influence of place of collection of the powder in the chamber on trend of flowability was not observed.

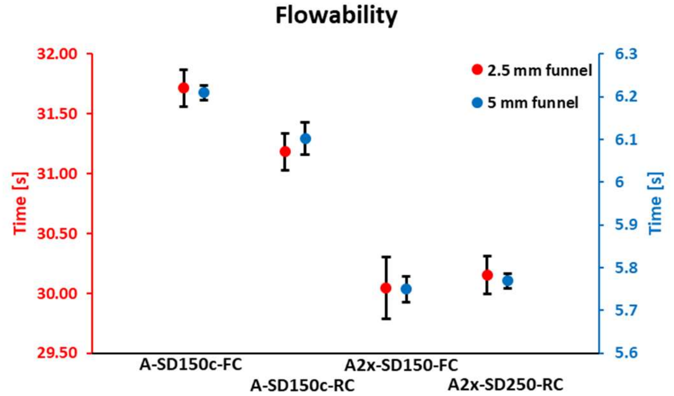

Figure 6: Flowability of the samples using two different funnels in Hall flowmeter.

The XRF chemical composition measurement showed the presence of more than 99 % of aluminium and negligible trace of other elements measurable by this technique. The results of XRD measurements showed that the AW24 feedstock powder was pure α-phase Al_2O_3 (corundum). This phase was predominant also after the spheroidization experiments. However, phase transformations of corundum into the θ phase and relatively rare δ phase Al_2O_3 occurred.

Conclusions

In this paper, the chamber suitable for spheroidization of powders with WSP-H plasma torch was designed. The main goal was to maximize the collecting efficiency. The collecting efficiency of 93 % of material, of which 66 % was the spheroidized powder and 27 % was the coating on the partition insert inside the chamber, was achieved after several improvements of the collecting chamber and the spraying parameters.

Two experiments with Al_2O_3 powder were performed, in which the AW24 powder was spheroidized and part of the spheroidized powder was used again as a feedstock for the second spheroidization experiment. It was shown that after the first spheroidization pass, about 90 % of the particles were spherical. The spheroidized powder exhibited significantly improved flowability. Phase composition of the feedstock was mostly preserved. The second spheroidization pass further increased the number of spherical particles to >99 %, slightly improved flowability and induced further α phase transformation to the metastable phases.

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