The Role of Powder Size Distribution on the Flowability of Ceramic Thermal Spray Powders

Michaela Remešová[1,*], Christian Hebenstreit[2], Serhii Tkachenko[1], Karl Körner[2], Vendula Bednaříková[1], Richard Trache[2] and Ladislav Čelko[1]

[1]CEITEC – Central European Institute of Technology, Brno University of Technology, Purkyňova 123, 612 00 Brno, Czech Republic
[2]Treibacher Industrie AG, Auer-von-Welsbach-Strasse 1, 9330 Althofen, Austria

Abstract. Thermal spray technologies require powders with sufficient flowability to ensure stable feeding and reliable injection into the spray plume. However, ceramic powders, particularly those in the fine particle size range, exhibit different flow behaviour compared to metal powders due to lower density, higher interparticle friction resulting from surface morpholgy, stronger electrostatic charging, and sensitivity to ambient moisture. This contribution investigates the flowability of agglomerated and sintered prototype yttrium-stabilised zirconia (YSZ) powder and evaluates the suitability of commonly used powder metallurgy methods for thermal-spraying applications. The YSZ ceramic powder was sieved into five particle size fractions ranging from below 25 µm to above 75 µm. Powder flowability was evaluated using Hall, Gustavsson, and Carney flowmeters, Carr indices, angle of repose measurements, and GranDrum rheometry. Practical flowability was additionally verified using an industrial powder feeder for thermal spraying. The results showed that powder flowability generally improved with increasing particle size. Powders below 35 µm were not flowable through Hall and Gustavsson cups, while the Carney cup enabled flow measurement for all fractions except those below 25 µm. However, all fractions exhibited stable feeding behaviour in an industrial powder feeder. Moisture significantly increased the cohesivity of fractions with medium particle sizes.

1. INTRODUCTION

For almost five decades, thermal spray technologies have been successfully used to convert solid powder feedstocks into functional coatings [1]. However, these powders are available on the market only in a relatively limited range of chemical compositions, which directly determines their current industrial application. To enable thermal spray technologies to respond sustainably to current challenges and opportunities related to raw materials while expanding into new areas of application, it is necessary to develop new materials or alternatives to existing ones that are well-suited to the spraying process.

The ideal particle size range for powders used in thermal spray technologies is typically between 25 and 150 µm [1,2], often within a narrower particle size distribution. Powder particles larger than 150 µm are difficult to melt in the spray plume, whereas particles smaller than 25 µm tend to agglomerate and accumulate within thermal spray system hardware. Consequently, commercial powders classified by manufacturers for thermal spray applications predominantly belong to this particle size range and, with only a few exceptions, fulfil the requirement for sufficient flowability.

Powder flowability represents one of the most critical parameters in thermal spraying because powders are commonly transported over medium or long distances from powder feeders, typically located outside the spray booth, to powder injectors mounted on plasma spray guns. These powders are subsequently injected into the plume under a compressed inert gas atmosphere. Powder flowability is influenced by numerous single-particle characteristics and interparticle interactions, including material density, particle shape, mechanical interactions, electrostatic forces, and reactivity with the surrounding environment, among many other less significant factors [1–3]. In general, powders with particle sizes below 25 µm are easier to feed when they are metallic or cermet materials, primarily due to their higher density, easier spheroidisation, lower electrostatic charging caused by friction, and lower atmospheric reactivity compared to ceramic powders.

Over time, a variety of methods have been developed and standardised for the characterisation of powder flow properties. Among the most commonly used techniques in powder metallurgy are the Carr indices [2–4], including angle of repose, aerated and tapped bulk densities, compressibility, cohesion, and uniformity. Additional commonly applied methods include particle size distribution analysis, powder rheometry, and powder

* Corresponding author: michaela.remesova@ceitec.vutbr.cz

charge analysis, all of which are among the most popular in the field of powder metallurgy [4–6].

The contribution focuses on the flowability of an agglomerated and sintered yttrium-stabilised zirconia (YSZ) ceramic powder and evaluates the reliability of commonly used powder metallurgy methods for the characterisation of thermal spray powders.

2. MATERIALS AND METHODS

2.1. Preparation of Ceramic Powder

An agglomerated and sintered prototype YSZ powder (Treibacher Industrie AG, Austria) was sieved using an AS200 Control sieve shaker (Retsch GmbH, Germany) into six thermal spray powder fractions: below 25 µm, 25–35 µm, 35–45 µm, 45–75 µm, above 75 µm. The powders were measured both in the as-received state and after heat treatment in a laboratory drying oven (SLW-53-W STD, POL-EKO Aparatura, Poland) at 200 °C for 8 h prior to measurement. Each fraction was examined both from the surface and in cross-section by means of scanning electron microscopy (SEM; Lyra3, Tescan, Czechia). The powder morphology was observed and documented in the secondary electron (SE) mode, while the cross-sectional micrographs were acquired in backscattered electron (BSE) mode. Cross-sections of the powder metallographic samples were prepared using standard metallographic procedures, including cold mounting, wet grinding, and polishing using diamond suspensions.

2.2. Laboratory Testing of Powder Properties

The Hall [7], Carney [8], and Gustavsson [9] flowmeters, different in funnel cone angle and calibrated orifice diameter (Figure 1), were used to evaluate the flowability of each prepared powder fraction. The flow time required for the entire standardised powder volume to pass through the flowmeter orifice was recorded. In addition, the angle of repose was measured using light projection recording, and the obtained data were subsequently analysed using ImageJ software (v1.54t, NIH, USA). The aerated bulk density (ABD) was determined using a standardised volume container and a precision laboratory balance (PA214CM/2, OHAUS Europe GmbH, Switzerland). Subsequently, a graduated cylinder was filled with the same volume of aerated powder and subjected to 50 taps to determine the powder packed bulk density (PBD). Based on ABD and PBD values, the compressibility Eq. (1) and Hausner ratio Eq. (2) were calculated:

$$\text{Compressibility [\%]} = \frac{\text{PBD} - \text{ABD}}{\text{PBD}} \times 100 \tag{1}$$

$$\text{HR} = \frac{\text{PBD}}{\text{ABD}} \tag{2}$$

Additionally, a new rheometer type GranuDrum (GranuTools, Belgium) was used for rotational powder cohesion testing. Forward rotational speeds in the range of 2 to 32 rpm were applied. The rheometer software, based on the integrated image analysis, provided cohesive index values of the ceramic powder.

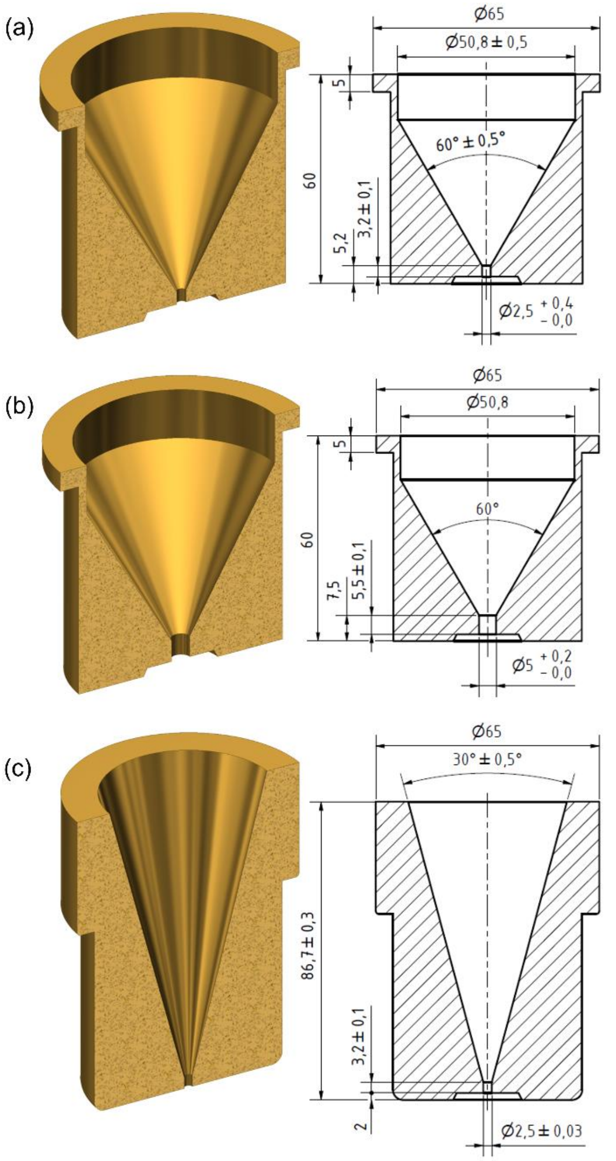

Figure 1. Schematics of the (a) Hall, (b) Carney, and (c) Gustavsson flowmeters.

2.3. Powder Feeder Testing of Powder Flowability

The flowability of the individual powder fractions was further tested directly using the industrially widespread thermal spray powder feeder PF 2/1LC (GTV GmbH, Germany) in order to verify the laboratory measurement results.

3. RESULTS AND DISCUSSION

The prototype YSZ ceramic powder exhibits a predominantly spherical morphology and, as apparent from its microstructure (see Figure 2), consists of individual particles that are sintered together. Typically, in every particle fraction, besides the particles of the specific sieved fraction, small satellite particles attached to their surface are also visible.

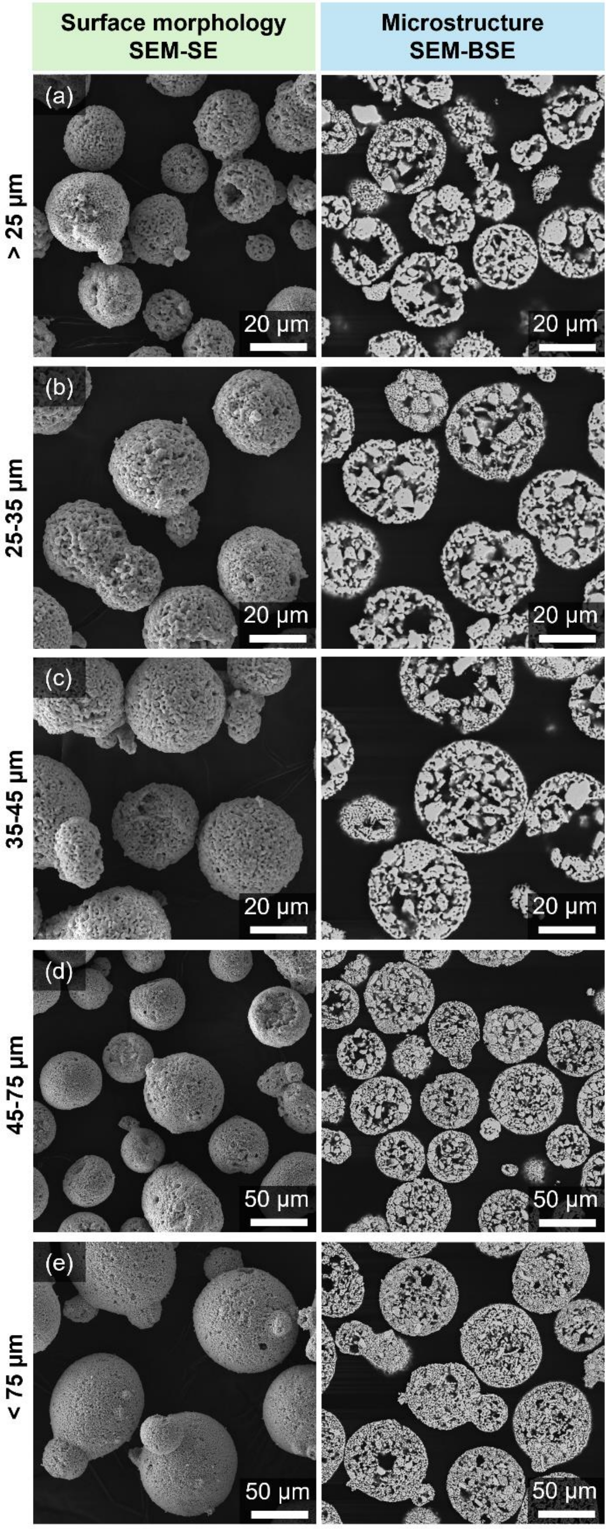

Figure 2. Surface morphology (SEM-SE, left) and cross-sectional microstructure (SEM-BSE, right) micrographs of the YSZ powder fractions: (a) below 25 µm, (b) 25–35 µm, (c) 35–45 µm, (d) 45–75 µm, and (e) above 75 µm.

The Carr indices provided confusing and not fully relevant results for the measured properties of the thermal spray powder. As can be seen in Figure 3, powder fractions with particle sizes below 35 µm were not flowable through the Hall and Gustavsson cups due to the small orifice diameter. Surprisingly, after several taps, the ultrafine powder with particle size below 25 µm started to flow through the Gustavsson cup, likely due to its lower cone angle. Nevertheless, since initial tapping was required to initiate flow, the measurement was considered invalid. In the case of the Carney cup, powder flow was observed for almost all prepared size fractions except for the fraction below 25 µm. The results generally confirmed easier, faster powder flow with increasing particle size.

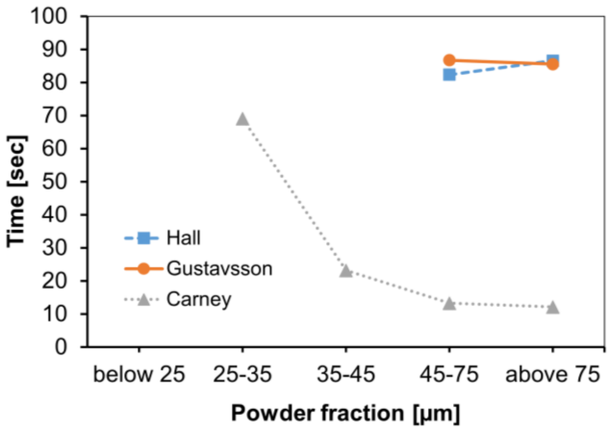

Figure 3. Powder flowability of all size fractions measured with different flowmeters (Hall–blue square, Gustavsson–orange dot, Carney–grey triangle).

The angle of repose ranged from 31° to 36°. Based on aerated and tapped bulk density measurements across all powder size fractions (Figure 4), the Carr Index was calculated, varying from 79–81%, suggesting good flowability. However, the Hausner ratio (4.6–5.2) indicates highly cohesive, non-flowing behaviour. Overall, the Carr indices suggest that for ceramic thermal spray powders, only the Carney cup provides limited and non-conclusive information on flowability.

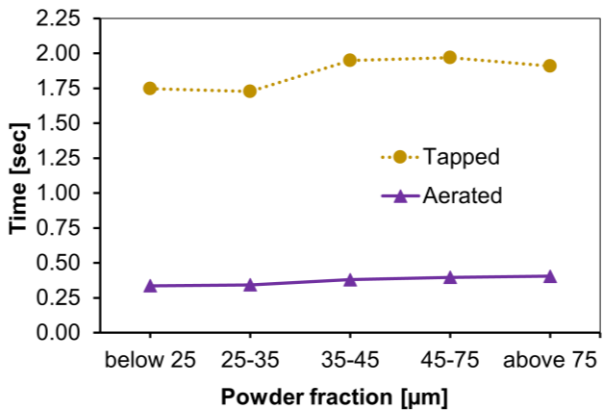

Figure 4. Comparison of aerated (purple triangle) and tapped (yellow dot) bulk density across powder size fractions.

The cohesive index values (Figure 5) measured using a GranuDrum rheometer [10] suggest acceptable flowability of the YSZ powders across all investigated size fractions. The lowest values of cohesivity index, corresponding to the best flowability, were observed for the fraction above 75 µm. The highest cohesivity was found for the 25–35 µm and 35–45 µm fractions. In these size ranges, the highest moisture contribution was also observed, and the cohesivity index decreased significantly after prior powder pre-heating. This pre-heating effect was less pronounced for fractions below 25 µm and above 75 µm.

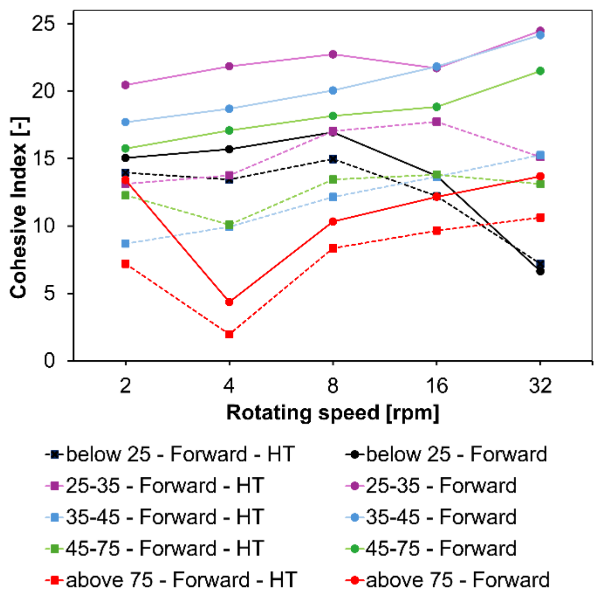

Figure 5. Cohesive Index (CI) across all particle size fractions, including powder, before (dot) and after heat treatment (HT, square).

For powder flow validation, the thermal spray powder feeder was used within a relatively short process window, typically applied by the thermal spray technology operator for powder feeding into the plume. The results showed excellent flowability for all powder sieve ranges, including the fraction below 25 µm, which was not observed to be flowable in the Carney cup flowmeter.

4. SUMMARY

The flowability of thermally sprayable ceramic powders decreases with a decreasing particle size compared to that of metals or cermets, where powder density can be considered the main critical parameter affecting powder flow.

The flowability of ceramic powders can be improved by modifying particle morphology, where particle spheroidization reduces mechanical interlocking, and by increasing particle size, resulting in a higher particle weight.

Electrostatic charge becomes more important for smaller particles below 25 µm, where particles tend to agglomerate.

Powder pre-heating prior to spraying, or heating the powder feeder vessel during spraying, significantly improves YSZ powder flowability by reducing moisture content in the particle size range of 25–75 µm.

Further investigation of powder flow mechanisms based on the initial powder compaction state and powder aeration prior to testing and thermal spraying is needed.

The Carr Index, Hausner Ratio, and avalanche angle measurement methods do not clearly describe the flow behaviour of YSZ ceramic thermal spray powders. Among the tested methods, only the Carney cup flowmeter provides information on guaranteed powder flow, although the results are still not entirely conclusive. Therefore, a reliable and rapid test method for ceramic thermal spray powder flowability still needs to be developed.

Acknowledgement

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