JTTEE5 15:33-36
DOI: 10.1361/105996306X92550

Effects of Humidity on the Flow Characteristics of a Composite Plasma Spray Powder

Malcolm K. Stanford and Christopher DellaCorte

(Submitted April 18, 2005; in revised form October 26, 2005)

The effects of environmental humidity on the flow characteristics of a multicomponent (composite) plasma spray powder have been investigated. Angular and spherical BaF_2–CaF_2 powder was fabricated by comminution and by atomization, respectively. The fluorides were blended with nichrome, chromia, and silver powders to produce a composite plasma spray feedstock. The tap density, apparent density, and angle of repose were measured at 50% relative humidity (RH). The flow of the powder was studied from 2 to 100% RH. The results suggest that the feedstock flow is only slightly degraded with increasing humidity below 66% RH and is more affected above 66% RH. There was no flow above 90% RH except with narrower particle size distributions of the angular fluorides, which allowed flow up to 95% RH. These results offer guidance that enhances the commercial potential for this material system.

Keywords APS coatings, composite materials processing, feedstock production/preparation technology, powder flowability, relative humidity

1. Introduction

It is common practice to dry plasma spray powders to enhance their flow properties, yet little experimental data are available on this issue. To better understand the flow behavior of the National Aeronautics and Space Administration (NASA) PS304 powder feedstock versus the level of environmental humidity, flow tests were conducted in a controlled humidity environment. NASA PS304 plasma spray deposited coating was developed for friction and wear reduction at high temperatures (Ref 1, 2). The feedstock for this coating is a powder blend consisting of nichrome (80Ni-20Cr), chromia (Cr_2O_3), silver, and eutectic barium fluoride-calcium fluoride ($68BaF_2$-$32CaF_2$). Previous studies have shown that the flow characteristics of this powder blend are highly dependent upon the morphology (Ref 3) and size (Ref 4) of the BaF_2-CaF_2 constituent. As part of this study, the effects of BaF_2-CaF_2 on the cohesiveness of the studied powder blends were further investigated by measurement of the tap density, apparent density, and angle of repose. This paper also reports the relationship between environmental humidity and the flowability of NASA PS304 feedstock powder. The effects of humidity were determined by comparing powder flow characteristics at a constant temperature while varying relative humidity (RH). Background on the analysis of particle cohesion mechanisms and their test methods has been described in detail elsewhere (Ref 5, 6). The overall goal of this investigation was to better understand the effect of humidity on the flow characteristics of the feedstock with various BaF_2-CaF_2 particle sizes and morphologies to enhance the commercial potential of NASA PS304 coating.

Malcolm K. Stanford and **Christopher DellaCorte,** National Aeronautics and Space Administration, Glenn Research Center, Cleveland, Ohio 44135–3191. Contact e-mail: Malcolm.K.Stanford@nasa.gov.

2. Experimental Procedure

Commercially available nichrome (44-74 μm in size), chromia (30-44 μm), and silver (45-100 μm) powders were obtained for this investigation. These powders were described in greater

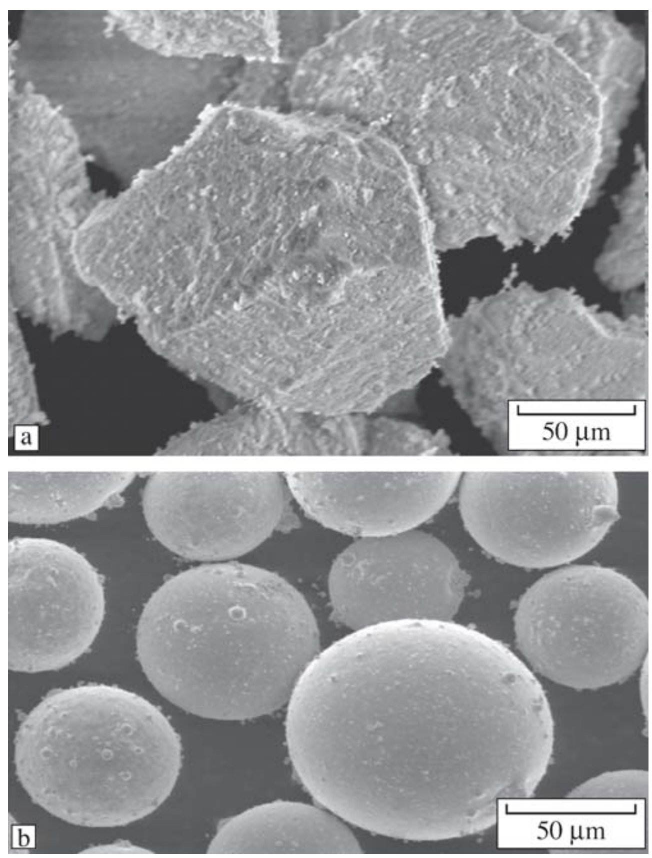

Fig. 1 (a) Angular and (b) spherical BaF_2-CaF_2 particles (original magnification 500×)

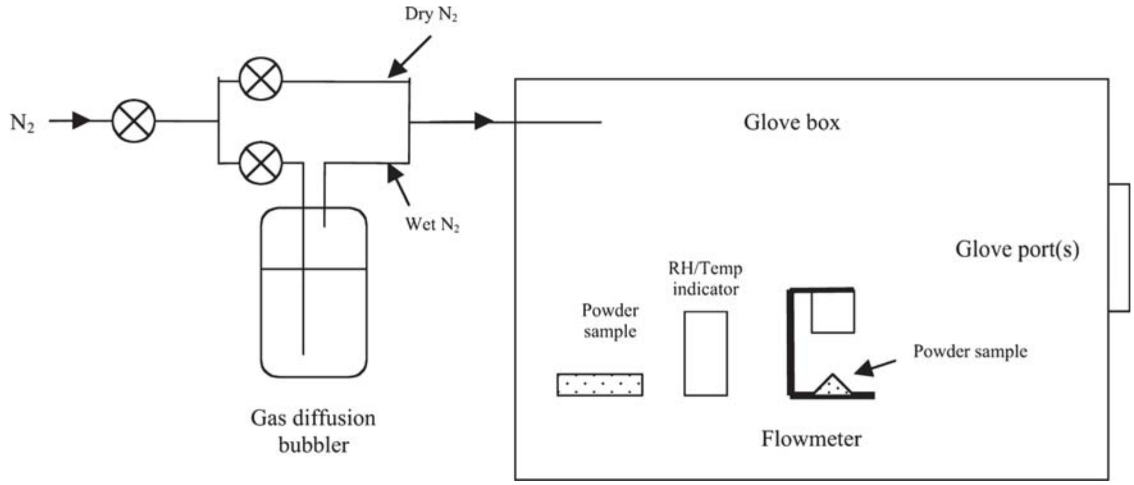

Fig. 2 Experimental setup for controlled humidity powder flow tests

detail previously (Ref 3) and were not modified in this study. The BaF_2-CaF_2 constituent was fabricated by two different techniques to obtain two distinct particle morphologies. The angular fluorides (Fig. 1a) were fabricated by comminution of the fused eutectic. Figure 1(b) shows spherical fluorides, which were fabricated by gas atomization. An equal particle size distribution by mass of −140 + 325 mesh (45-106 μm) powders was prepared of comminuted and of atomized BaF_2-CaF_2 powders, in addition to –140 + 170 mesh (90-106 μm) and –270 + 325 mesh (45-53 μm) comminuted fluorides.

Powder blends consisting of 60 g nichrome, 20 g chromia, 10 g silver, and 10 g each of the four BaF_2-CaF_2 types were prepared by mixing the constituents together in a 125 ml high-density polyethylene bottle until the powder was well blended. Each powder was then spread evenly on the bottom half of a glass petrie dish and placed in a vacuum oven for 12 h at 59 mm Hg (absolute pressure) and 205 °C. The powders were then covered with the top half of the petri dish and transferred immediately to a glove box purged with dry nitrogen gas. The powders were allowed to cool to room temperature (~22 °C) in the nitrogen atmosphere. To control RH in the glove box, the nitrogen gas inlet was connected to a gas diffusion bubbler containing deionized water as shown schematically in Fig. 2. The powder samples were exposed to the selected humidity level for 2 h prior to testing and all tests were performed at 50% RH unless otherwise specified.

The tap density of each powder specimen was measured according to ASTM B 527-93 (Ref 7). A 50 g sample of each powder blend was poured into a 25 ± 0.3 ml graduated cylinder with 0.5 cm^3 divisions. A commercially available tapping apparatus dropped the cylinder through a stroke of 3 mm at a frequency of approximately 250 taps per minute. The volume the powder occupied after 3000 taps V_t was then recorded to the nearest 0.5 cm^3. The tap density was then calculated as the mass of the powder divided by the volume it occupied, 50 g/V_t.

The apparent density was measured according to ASTM B 212-99 (Ref 8). Each powder blend was poured into a 25cm^3 brass cup using a Hall flowmeter funnel (Ref 7). When the powder filled the cup to overflowing, the funnel was swiveled about 90° to stop powder flow into the cup. A metallic spatula was used to level the powder to the top edge of the cup. The cup with the powder was then transferred to the scale. The mass of the powder m_a was recorded to the nearest 0.1 g. The apparent density was then calculated as the mass of the powder divided by the volumetric capacity of the cylinder, $m_a/25cm^3$. The reported tap density and apparent density values were the averages of three separate measurements.

Table 1 Selected physical properties of the studied powder blends

BaF_2-CaF_2 constituent size (and chape)	Tap density	Apparent density	Angle of repose
No fluorides (control)	4.35	3.04	36.4 ± 0.01
45-106 μm (angular)	4.00	2.87	39.4 ± 0.02
45-106 μm (spherical)	4.00	2.90	36.4 ± 0.03
45-53 μm (angular)	3.92	2.85	37.1 ± 0.02
90-106 μm (angular)	4.00	2.95	36.5 ± 0.003

The angle of repose was measured using a procedure slightly modified from ASTM C 1444-99 (Ref 9) to test smaller samples of powder. Approximately 100 g powder was loaded into a Hall funnel and then allowed to empty onto a flat base plate. Once the powder heap accumulated to the funnel opening, flow through the funnel ceased, and the diameter of the cone base was measured in four places using 0.0025 mm precision calipers. The average of these four measurements D_1 was recorded and this procedure was repeated for two additional readings D_2 and D_3.

The average cone diameter D_A is the average of the three readings D_1, D_2 and D_3. The angle of repose α is then given by:

$$\alpha = \tan^{-1}\left(\frac{2H}{D_A - d}\right)$$

where H is the height of the cone (17.8 mm) and d is the diameter of the funnel opening (5 mm). The angle of repose was reported to the nearest 0.1°.

Flow was tested according to ASTM B 213-97 (Ref 10). A digital stopwatch was used to measure the time it took for the entire 50 g sample to exit the funnel to the nearest 0.1 s. The average and standard deviation of 5 consecutive tests were reported.

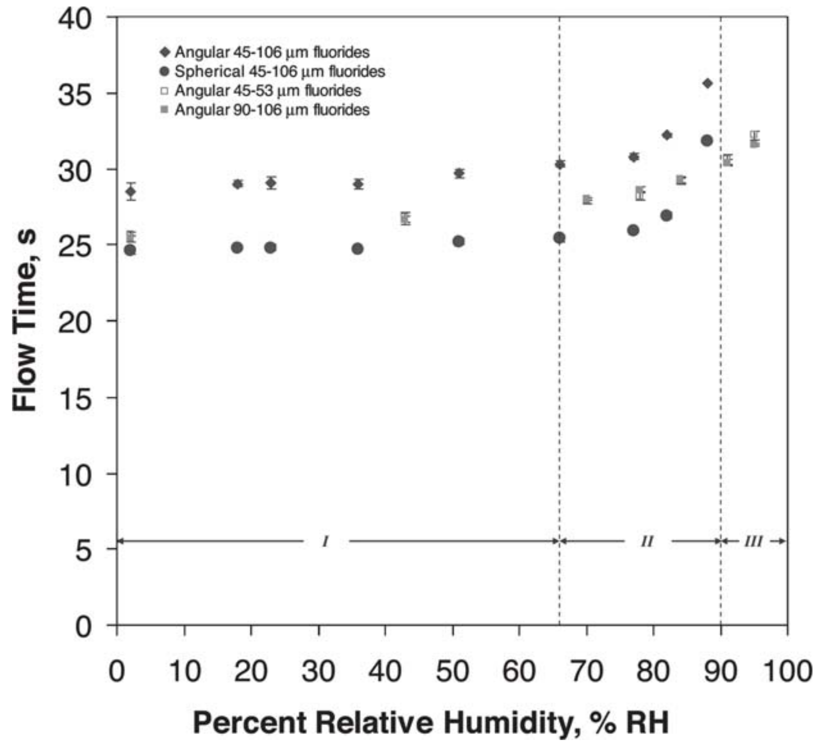

Fig. 3 Flow times of NASA PS304 feedstock powders with various BaF_2-CaF_2 particle types as a function of percent relative humidity

3. Results and Discussion

The density and angle of repose measurements for the studied powder blends are listed in Table 1. There is less than 4% difference between the densities of the studied powder blends containing BaF_2-CaF_2, which is insignificant. Because the density of the powder partially determines the volume a given powder sample will occupy, this result indicates that each of the studied 50 g powder samples will occupy essentially the same volume. Therefore, if particle-particle interactions are neglected and only the rule of mixtures is considered, each of the studied powder blends would have approximately the same flow time. Any observed differences in powder cohesiveness will be due to humidity as well as the influence of the BaF_2-CaF_2 particles. Given the angle of repose measurements (Table 1), the powder blends with 45-106 spherical and 45-53 μm, and 90-106 μm angular BaF_2-CaF_2 have cohesiveness at nearly the same level as the control powder. However, the powder blend containing 45-106 μm BaF_2-CaF_2 had an angle of repose approximately 9% higher than the control measurement. Because these measurements were taken at approximately 50% RH, this is an indication that BaF_2-CaF_2 particle shape has an effect on the cohesiveness of the powder blend.

The flow times of the powder blends with various BaF_2-CaF_2 particle types with respect to humidity are plotted in Fig. 3. Three regions are labeled on the figure to discuss changes in flow behavior. In Region I (0-66% RH), flow is only slightly degraded with increasing humidity. Humidity has a greater effect on flow from 66 to about 88% RH (Region II) and in Region III (>90% RH), flow was only observed with the feedstock employing narrower size distribution 45-53 μm and 90-106 μm angular BaF_2-CaF_2 powders. This is probably due to the reduced interparticle contact area that results from packing of particles with narrow particle size distributions (Ref 11). Powders with wider particle size distributions can pack more efficiently because the interstices of coarser particles can be filled with finer particles. The flow time increase with increasing relative humidity indicates an increase in interparticle cohesion. This behavior is similar to results reported by Peterson and Small for iron powders (Ref 12, 13). The fact that the error bars overlap on measurements for the powder blends containing 45-53 μm and 90-106 μm BaF_2-CaF_2 particles indicates that there is no significant difference in the flow behavior of these powders. However, there is a significant difference between the flow behaviors of powder blends with angular and spherical BaF_2-CaF_2 and between wide and narrow BaF_2-CaF_2 particle size distributions.

4. Conclusions

The objective of this investigation was to study the effects of environmental humidity on the flow rate of NASA PS304 feedstock under the influence of gravity. Based on the results, the following conclusions were made.

- Humidity has a minor effect on the flowability of the studied NASA PS304 feedstock powders from 2 to 66% RH.
- Above 66% RH, humidity had a more detrimental effect on flow of the powder, and above 88%, RH no flow was observed for feedstock containing either angular or spherical 45-106 μm fluorides.

- Feedstock flow could be obtained up to 95% RH using narrower size distributions of angular fluorides (45-53 μm or 90-106 μm).
- BaF_2-CaF_2 particle morphology and particle size distribution have a noticeable effect on feedstock cohesiveness.

References

1. C. DellaCorte, V. Lukaszewicz, M.J. Valco, K.C. Radil, and H. Heshmat, Performance and Durability of High Temperature Foil Air Bearings for Oil-Free Turbomachinery, *Tribol. Trans.,* 2000, 43 (4), p 744-780
2. J.A. Laskowski and C. DellaCorte, Friction, and Wear Characteristics of Candidate Foil Bearing Materials from 25 °C to 800 °C, *Lubr. Eng.,* 1996, 52, p 605-612
3. M.K. Stanford, C. DellaCorte, and D. Eylon, Effect of Particle Morphology on Flow Characteristics of a Composite Plasma Spray Powder, *J. Therm. Spray Technol.,* 2004, 13 (4), p 586-592
4. M.K. Stanford, C. DellaCorte, and D. Eylon, Particle Size Effects on Flow Characteristics of PS304 Plasma Spray Coating Feedstock Powder Blend, *Ceram. Eng. Sci. Proc.,* 2003, 24 (3), p 577-585
5. R.M. German, *Powder Metallurgy & Particulate Materials Processing*, Metal Powder Industries Federation, Princeton, NJ, 2005
6. M.K. Stanford, "Control of Interparticle Cohesion in PS304 Plasma Spray Deposited Solid Lubricant Coating Powder Feedstock," Ph.D. dissertation, University of Dayton, Dayton, OH, 2002
7. "Standard Test Method for Determination of Tap Density of Metallic Powders and Compounds," B 527-93, *Annual Book of ASTM Standards*, ASTM, 2001
8. "Standard Test Method for Determination of Apparent Density of Free-Flowing Metal Powders Using the Hall Flowmeter Funnel," B 212-99, *Annual Book of ASTM Standards*, ASTM, 2001
9. "Standard Test Method for Measurement of the Angle of Repose of Free-Flowing Mold Powders," C 1444-00, *Annual Book of ASTM Standards*, ASTM, 2001
10. "Standard Test Method for Flow Rate of Metal Powders," B 213-97, *Annual Book of ASTM Standards*, ASTM, 1998
11. D.J. Cumberland and R.J. Crawford, *The Packing of Particles*, Elsevier, Amsterdam, The Netherlands, 1987
12. J.E. Peterson and W.M. Small, Physical Behavior of Water-Atomized Iron Powders: Effects of Relative Humidity and Particle Size, *Int. J. Powder Metall.,* 1993, 29 (2), p 121-130
13. J.E. Peterson and W.M. Small, Evaluation of Metal Powders Using Arnold Density Meter and Hall Flowmeter, *Powder Metall.,* 1994, 37 (1), p 37-41