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# The effect of gas phase polydimethylsiloxane surface treatment of metallic aluminum particles: Surface characterization and flow behavior

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## ABSTRACT

Aluminum particles were exposed to gaseous polydimethylsiloxane (PDMS) to produce a hydrophobic surface coating for enhanced flow and fluidity. Surface retention of the intact PDMS was confirmed through infrared and X-ray photoelectron spectroscopy. Transmission electron microscopy was used to image cross-sections of the treated particles and energy dispersive spectroscopy element maps demonstrated the presence of a surface layer consisting of silicon and oxygen. Density measurements provided evidence for improvements in the Hausner ratio and Carr index of the PDMS-treated aluminum, indicating a reduction in inter-particulate cohesion through increased bulk density. Stability, compressibility, shear, aeration, and permeability of the particles were assessed by powder rheometer. The compressibility was reduced by approximately 32% following surface treatment, revealing a reduction in void space, while Mohr's circle analysis and shear testing determined that the extrapolated cohesion value was reduced by approximately 53% and the flow factor at 6 kPa was doubled. Aeration testing showed that the air velocity required to obtain a fluidized bed was on the order of 0.35 mm/s for the treated powder, whereas the raw powder could not be uniformly fluidized. PDMS may be a viable option for the large-scale treatment of aluminum powder for flow applications.

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## Introduction

Metallic aluminum powders are widely used as propellants, pigments, and fillers to impart enhanced mechanical and thermal properties (Bishay, Abd-El-Messieh, & Mansour, 2011; Mamunya, Davydenko, Pissis, & Lebedev, 2002). These applications take advantage of the metallic properties of aluminum, although the presence of the typical passivated surface layer can cause complications with regard to stability, compatibility, and dry powder flow. Most metals form a passivated oxide layer during exposure to ambient oxygen (Hunter & Fowle, 1956) and subsequent hydration of this layer can generate surface hydroxyl groups (Diggle & Ashok, 1976). These surface groups can be used as reactive sites to covalently attach surface coatings that improve the compatibility of the metal with other materials or introduce new properties. This technique is widely used in the pigment industry through employing

polymeric coatings to enhance the dispersion of metal powders in paints and plastics (Karlsson, Palmqvist, & Holmberg, 2006), provide corrosion protection (Montemor, 2014; Stratmann, Feser, & Leng, 1994), and increase colloid stabilization in organic media (Dobias, Qiu, & von Rybinski, 1999; Kuchibhatla, Karakoti, & Seal, 2005), all of which are predominantly liquid-phase applications. In some cases involving powder handling, however, a dry product is desired, such as during the delivery of bulk fillers and propellant powders. The associated flow behavior, a direct consequence of the powder surface properties, therefore becomes an important parameter in the design of off-loading equipment.

The flow behavior of powders is difficult to characterize because powders can behave differently depending on the discharge method, container materials, and method of fluidization. Powders that are considered free flowing in one application may exhibit poor flow under a different set of conditions. In gas fluidized applications, for example, stable delivery is dependent on the aeration and permeability properties of the powder. Generally, cohesive powders tend to have erratic flow in fluidizing environments because of the void volumes produced from air pressure

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differentials throughout the powder bed. These void spaces are also formed through particle–particle surface contacts or inter-particulate cohesion, which is common in fine powders where the effects of attractive forces between particles are greater than gravitational forces. Achieving reliable and consistent flow with fine powders can be challenges, although surface treatments can be used to reduce inter-particulate cohesion. As noted, metallic aluminum powders have surface reactive sites that make surface treatment an attractive option for improving the dry powder flow properties.

Hydrophobic alkoxy silane-based coatings produced through bulk solution depositions have been shown to enhance the flow and fluidization properties of metallic aluminum powders (Ludwig & Miller, 2015). Alkoxy silanes having more than one hydrolysable group have also been found to form oligomers and extended polymer networks in solution (Brochier Salon & Belgacem, 2010) because these multiple functional groups can react with one another, producing numerous short chain polymers that may subsequently react with the substrate surface. These cross-linked polymers are analogous to polysiloxanes or, more generically, silicones. Silicones have widespread commercial use in the pharmaceutical, food, and lubricant industries because they exhibit low reactivity and are non-toxic. They are produced in massive quantities each year and thus are ideal for large-scale operations. Polydimethylsiloxane (PDMS) is an example of a hydrophobic siloxane-based polymer. It is a liquid at room temperature and has the generic structure  $(\text{CH}_3)_2\text{SiO}[(\text{CH}_3)_2\text{SiO}]_n\text{Si}(\text{CH}_3)_3$ , where  $n$  can range from 0 to 5000. Despite the relatively low vapor pressure of this polymer, a processing temperature in the vicinity of 250 °C has been shown to deposit thin layers of PDMS on silica and copper powders (Kim et al., 2012; Park, Sim, Jeong, Seo, & Kim, 2012; Yuan et al., 2008). Previous applications of PDMS have used the polymer for coating purposes for its protective and electronic properties, whereas the work described herein employed PDMS for its hydrophobicity. This study was predicated on the theory that PDMS-treated aluminum particles would exhibit reduced cohesiveness, resulting in increased bulk density, enhanced flow behavior, and improved fluidity relative to untreated aluminum powder.

Typical alkoxy silane treatments are performed in bulk slurries, and these processes require special considerations with regard to handling the solvent because post-processing removal of the solvent can extend the processing time significantly. For this reason, the present work used a gas-phase method to reduce solvent waste and circumvent the technical challenges associated with solvents. The work presented in this paper is thought to provide valuable insights into the chemical properties and flow behavior resulting from a PDMS surface layer on fine aluminum particles.

## Materials and methods

### PDMS surface treatment

PDMS with terminal hydroxyl groups ( $M_n$  approximately 550, viscosity approximately 25 cSt; Aldrich, USA) and metallic aluminum powder ( $d_{50}$  approximately 20  $\mu\text{m}$ , Lot No. 12-3008; Valimet, USA) were used as received. Ultra-high purity nitrogen was employed as the fluidizing gas in conjunction with an inline oxygen/hydrocarbon trap and flow controller. A simple gas-phase deposition apparatus was fabricated in house using two porous gas distributor plates, a glass cylinder with a heating jacket, and a nitrogen gas flow line (flow velocity = 0.2 mm/s) connected to a PDMS reservoir. Prior to each deposition trial, aluminum powder (300 g) was loaded into the cylinder and, to improve the particle–gas-phase interactions. While in the apparatus, the powder was periodi-

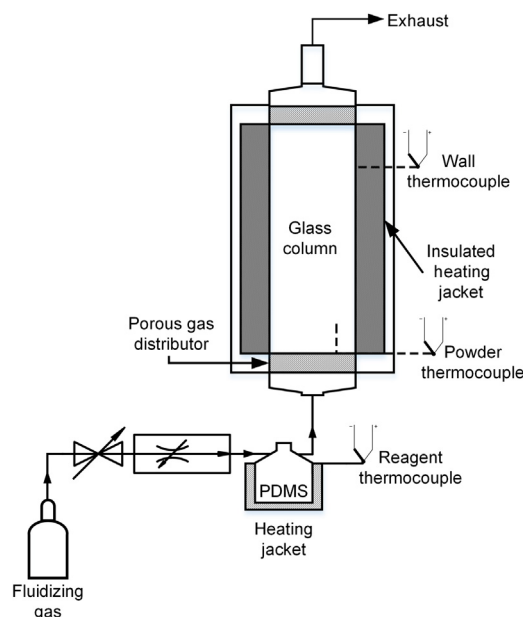


Fig. 1. Schematic of the polydimethylsiloxane (PDMS) gas deposition apparatus.

cally agitated/homogenized. The powder was heated to and held at approximately 100 °C for 1 h prior to treatment to remove physisorbed water and also maintained at this same temperature for the duration of the deposition process. The PDMS was heated to approximately 250 °C and the resulting vapors were mixed with the nitrogen gas stream over a period of approximately 4 h. A schematic of the deposition apparatus is shown in Fig. 1.

### Materials characterization

Diffuse reflectance Fourier transform infrared spectroscopy (DRIFTS) data were collected using a Fourier transform infrared spectrophotometer (Vertex V70, Bruker, Germany) equipped with a mercury–cadmium–telluride detector. Spectra were collected with a Praying Mantis™ diffuse reflectance accessory (Harrick, USA) equipped with  $\text{CaF}_2$  windows with a 900- $\text{cm}^{-1}$  cutoff. The reactor was purged with argon continuously through all measurements. The spectrum resolution was set to 6  $\text{cm}^{-1}$  and 400 scans were performed. Powdered potassium bromide (KBr) was used to acquire a reference spectrum and was dried to 350 °C for 30 min prior to use. Additional KBr spectra were collected to enable subtraction of atmospheric water contributions.

X-ray photoelectron spectroscopy (XPS) was performed using a Kratos Axis Ultra instrument (Shimadzu, Japan) with a monochromatic Al  $K\alpha$  X-ray source. Powdered samples were mounted on a silicon substrate using a double-sided tape (3M®). Analysis was performed at a constant system pressure of  $10^{-8}$  torr, employing a pass energy of 80 eV for the survey scans with a step size of 0.5 eV. High-resolution data were collected at 20 eV with a step size of 0.1 eV. Charge correction was employed using the C 1s peak at 284.8 eV as an internal standard.

Particles were imaged using environmental scanning electron microscopy (ESEM, Hitachi S300N, Japan) with a tungsten filament and 10 nm resolution. An Everhart–Thornley detector was employed to collect the secondary electron images. Samples were prepared by attaching a double-sided carbon tape on a standard scanning electron microscopy (SEM) stub and applying a small amount of powder (<5 mg) onto the tape surface, followed by dislodging any loose powder by tapping the stub. All powders were analyzed in high vacuum mode.

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