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Segregation of titanium powder with polydisperse size distribution: Spectral and correlation analyses

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ABSTRACT

Additive manufacturing with powder metallurgy is becoming a popular and cost effective process due to improvements in powder availability and the ability to produce a part close to the desired net shape. Because the particles have a variety of shapes and sizes, the flow of titanium powder may be hindered if it is not uniformly mixed, resulting in poor quality end products. Understanding how titanium powder flows, and how the flow is affected by various flow parameters is essential for the proper utilization in additive manufacturing. By studying the segregation of the distribution of many sizes of particles in titanium powder, proper flow conditions could be established in order to produce the highest quality end products as possible. The results of several analysis techniques indicate that the greatest mixing of industrial titanium powder occurs nearest the center of a rotating tumbler with the smallest diameter. This indicates that short flow distances are best if titanium parts with maximum homogeneity are sought.

1. Introduction

Powder metallurgy and additive manufacturing have become a widely used and cost-effective means of manufacturing where complicated part geometry can be produced with little material waste. Advantages compared to other production methods such as forging and machining include low energy requirements and high raw material utilization. Since the powder is introduced to a near net shape mold of the final product, the resulting product requires only minor machining [1]. There are essentially no waste products or scraps as long as the input material can reliably flow and remain undamaged during handling.

Powder metallurgy is particularly attractive when the parts are manufactured from titanium because of the expense of the raw resource metal. Titanium is desirable for its corrosion resistance, high temperature durability, and high strength to weight ratio [2]. Since the powder metallurgy process produces little waste and the machining of titanium is challenging, this form of manufacturing is cost effective and attractive to industry. The primary drawback, however, is the limitation of titanium powder to reliably flow as a uniformly distributed material.

Understanding how Ti powder flows, and how the flow is affected by various flow parameters is essential for its proper utilization. The powder considered in this research is produced using the Armstrong Process implemented by International Titanium Powder (ITP) subsidiary of Cristal Metals [1]. The raw powder output from the Armstrong Process is composed of particles of many sizes; ITP has implemented a proprietary milling technique which attempts to create more round and uniform particles by breaking off extremely small asperities and edges without substantially altering the particle size distribution [3]. A critical feature is that the powder is composed of a variance of shapes and sizes resulting in a high degree of poly-disperse particles. The lack of uniform consistency throughout the entire material hinders the automated powder metallurgy process, which requires the powder to move efficiently without any blockage or disruptions. Furthermore, the powder needs to be delivered uniformly mixed so that the produced parts are homogeneous and free of imperfections or voids which could cause poor quality end products that do not perform as intended. Understanding the flow characteristics of particular Ti powder size ranges under various conditions is essential to developing optimal manufacturing techniques, which was the emphasis of the previously reported study [3]. The focus of this subsequent investigation is the segregation of poly-disperse titanium powder mixtures, and how segregation is affected by flow rates and free surface energy.

Particle segregation occurs in granular flow when there is a difference in particle size, shape or density [4]. There are two major mechanisms that drive particle segregation within a flowing layer with dissimilar particles: percolation of smaller particles through the voids created by larger particles and buoyancy of less dense particles of similar size. Since all of the material is composed of Ti, this study considers only differences in particle size alone. Most segregation studies consider bidisperse mixtures of only two sizes [5–9]. For flow generated in a rotating tumbler, a free surface flowing layer forms at the dynamic angle of repose above a fixed bed that rotates with the perimeter walls of the tumbler. In most cases, steady flow creates a central core of small

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particles around which large particles congregate. The distribution of sizes in the Ti powder means that particles cannot perpetually reinforce the core, but will sometimes flow all the way to the edge of the tumbler. Similar features of stratification or sun bursts have appeared in bidisperse mixtures due to the kinematics of the flowing layer and a wave breaking mechanism [10]. This paper reports similar stratified results for Ti powders of large size distribution where flow is generated in a circular rotating tumbler.

Fig. 1 shows a single instance of the segregation where radial stripes are observed at the edges of the tumbler. The stripe feature is a result of the concentration of small and large particles reflecting light differently. The radial banding is quantified using image and frequency analyses (correlation and spectral methods) in order to characterize rotation rates and locations in the tumbler where powder is more likely to segregate. While paddle mixers [11,12] and double-cone blenders [13] offer mechanisms for improving granular homogeneity, the free surface flow within the rotating tumbler emulates the potential segregation during discharge from a mixer and delivery to the additive manufacturing device. Therefore, the results are very important in manufacturing where predictable, uniform properties are necessary in order to produce quality parts.

2. Experimental procedure and analysis methods

To generate a steady flowing layer through which size segregation could take place, a tumbler with a circular cross section was rotated about its central axis. The tumbler volume is filled to 50% with the powder, providing a free surface flow along the diameter of the cross-section. The flow is continuous at the dynamic angle of repose since there was always a supply of new material from the fixed bed rotating with the tumbler. The combination of different tumbler diameters and rotation rates are investigated but all are in the steady, continuous avalanching flow regime rather than in the more complex forms of unsteady cataracting flow (a curved free surface), and centrifuging flow [14,15]. The Froude number was used as a means to non-dimensionalize the tumbler variables

$$Fr = \frac{r\omega^2}{g} \tag{1}$$

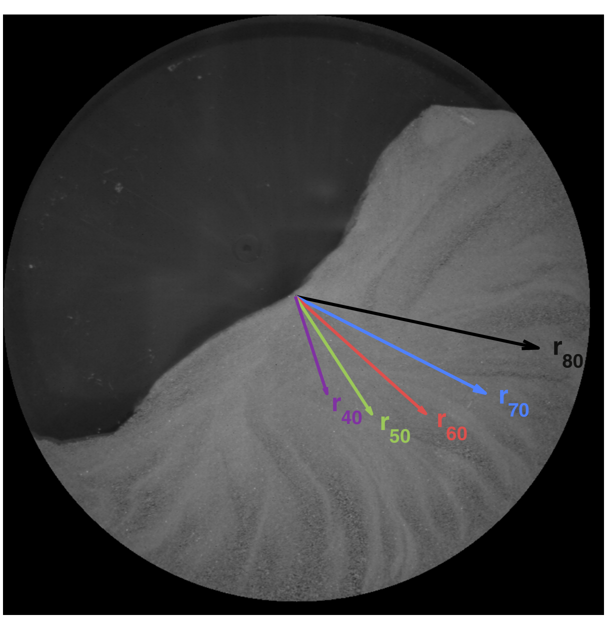

Fig. 1. Example of circular tumbler where lines of segregation are visible. Overlaying the image are dimensional lengths of radial percentages used in cross-correlation and auto-spectra analyses.

where r is tumbler radius, ω is the rotational speed, and g is the acceleration due to gravity. The titanium powder typically avalanches up to a Fr number of approximately 3×10^{-4} whereas Froude numbers greater than 5×10^{-3} began to curve the free surface like in the cataracting flow regime. Therefore, all experiments in this research were conducted in the range of $5 \times 10^{-4} \leq \mathrm{Fr} \leq 5 \times 10^{-3}$. The steady flow is not explicitly uniform along the entire length of the free surface as the material tends to generate a long run-out at the end of the flowing layer. This feature likely drives the curvature of the stratified radial bands observed in Fig. 1.

The tumbler is constructed of clear, static-dissipative acrylic end plates to observe the titanium powder motion. The presence of electrostatic forces is further reduced by cleaning the surfaces with anti-static spray which provides an additional safety measure to prevent sparks from igniting the fine titanium powder [16–19]. The tumbler is 12.7 mm thick and has a maximum radius of 17.8 cm. Concentric rings were designed as inserts for the tumbler allowing a total range of diameters from 15.24 to 35.56 cm in steps of 5.08 cm.

The mass fraction of ten sieved samples of ITP processed particles is shown in Fig. 2. While a carefully controlled distribution would enhance repeatability and reduce statistical variance, the implications of segregation in authentic industrial settings are preferred for this analysis. The distribution shows that 3% of the mass is larger than 500 μm, and that a nearly even distribution of particles is achieved between 75 and 500 μm. The milling process also generates a large number of fine particles less than 75 μm with a significant concentration, about 20%, of ultrafine particles that are less than 45 μm. The tumbler is filled to 50% volume fraction and then mounted to a shaft driven by a DC stepper motor. A constant input frequency provided by a function generator controls the input rotation rate for each trial.

The images of the powder within the tumbler are taken by a monochrome high speed camera (Gigaview by Southern Vision Systems). Uniform lighting was provided above and below the tumbler to illuminate the gray titanium powder and contrast it with the backplate made of black painted aluminum on which the tumbler is mounted. Images are taken as the tumbler rotates, and this is done for all the combinations of the ten Fr numbers and four tumbler diameters. Ten representative frames were taken from the complete set of images to complete the ensemble statistics. The images are separated in time such that all the powder volume has passed through the flowing layer.

Image processing software is used to analyze the intensity data from the images. A program was designed to convert the void space in the tumbler (black backplate) to a low intensity background which allows for better histogram equalization of the images and improved contrast. Next, the program takes a one degree pie-shaped slice of the image, separates the slice into five radial areas and sums the intensity values of the pixels within each area. The intensities as a function of θ and r are organized into vectors and the tumbler void space is subtracted, which

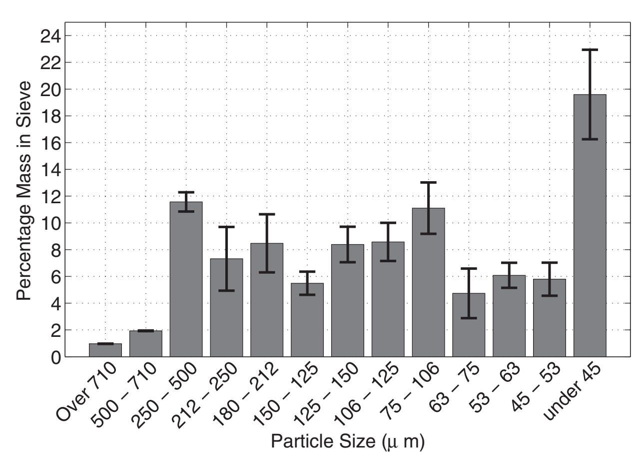

Fig. 2. Particle size distribution across the sieve stack.