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Study on the flow properties of Ti-6Al-4V powders prepared by radio-frequency plasma spheroidization

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ABSTRACT

Spherical Ti-6Al-4V powders were prepared using radio-frequency plasma spheroidization. A laser particle size analyser, a scanning electron microscope, an X-ray diffractometer and a Freeman FT4 powder rheometer were used to analyse the granulometric parameters, micro-morphologies, phase constitutions and flow properties of the raw and the spheroidized powders, respectively. The spheroidized powders exhibited an almost 100% degree of sphericity, smooth surfaces, favourable dispersion and narrow particle size distribution under appropriate plasma technological parameters. The average particle size of the spheroidized powders increased slightly as compared with that of the raw powders. In addition, the spheroidized powders exhibited higher conditioned bulk density and improved flow properties (including the dynamic flow properties, aeration, compressibility, permeability and shear properties) as compared with those of the raw powders.

1. Introduction

Three-dimensional (3D) printing, also known as rapid prototyping or additive manufacturing, is a technology used to directly manufacture 3D objects layer by layer from digital models [1,2]. Selective laser melting (SLM) is a 3D printing technique that uses a laser beam as the energy source to rapidly melt metal powders. The molten metal pool then rapidly cools down, and the consolidated material starts to form the product. SLM requires metallic powders with a small average particle size, high degree of sphericity, good flowability and reasonable particle size distribution [3].

Ti-6Al-4V has received increasing attention for 3D printing applications in the aerospace, automobile and biomedical fields because of its low density, high specific strength, superior corrosion resistance, excellent biocompatibility and mechanical properties. Several manufacturing methods can be used to prepare Ti-6Al-4V spherical powders (SP), such as gas atomising, rotating electrode processing and radio-frequency (RF) plasma spheroidization [4–6]. However, the spheroidized powders (SP) prepared using RF plasma spheroidization exhibit a higher degree of sphericity, better flowability and more reasonable particle size distribution than those prepared using the other techniques.

Gu et al. [7] and Mapar et al. [8] reported that the flow properties of powders greatly affect the quality of 3D-processed parts. The flow properties of powders define the extent to which the powders are packed together when a new layer of powders is covered on the previously formed solid layer. Which have a significant effect on the density and particle distribution in the powder bed, thereby determining the density, dimensional precision, surface roughness and mechanical properties of 3D-printed products [9,10].

The flow properties of powders can be used to efficiently distinguish certain macroscopic powder properties that cannot be measured using traditional methods. At the beginning of 3D printing, the development of a comprehensive powder database would be helpful for the selection and filtration of raw powders (RP). It is also useful for the optimisation and adjustment of the technology for the study of SP. Therefore, the evaluation of the characteristics of SP for 3D laser printing and clarification of the evolution mechanism of powder flowability will have practical significance and research value [11–13].

Generally, powder morphology is investigated using scanning electron microscopy (SEM), and the particle size and size distribution of the powder are identified using a laser micron sizer. The apparent density of a powder is typically measured using a Hall flowmeter, and the apparent viscosity of metal slurries is determined using a rotational viscometer [14]. The flowability of a pow-

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der has been previously determined by measuring the Hall flow speed and angle of repose [15]; however, the evolution mechanism of the flow properties of the investigated powders could not be well explained by these two parameters. Hence, in this study, a powder flow properties analyser (Freeman FT4 powder rheometer) [16] was employed to analyse the evolution mechanism of the flow properties of powders, including the dynamic nature and shear strength of the powder as well as bulk properties such as the conditioned bulk density, aeration, compressibility and permeability.

In the work, we analysed the micro-morphologies and the particle size distribution of the RP and the SP of Ti-6Al-4V and further studied the evolution mechanism of the flow properties of these powders.

2. Experiments

SP of Ti-6Al-4V was prepared using a RF plasma spheroidization system (Tekna, with a maximum 40-kW high-frequency output using a PN-35 induction plasma torch). The optimal technological parameters (feeding rate of 80 g·min^{-1} and carrier gas flow rate of 6 L·min^{-1}) were determined based on the degree of sphericity and spheroidization rate of the SP of Ti-6Al-4V.

The particle size distributions of the RP and the SP of Ti-6Al-4V were recorded using a laser particle size analyzer (Fritsch Gmbh, Analysette22 MicroTec plus). The morphologies of powders were observed using a field emission scanning electron microscope (FESEM, JEOL, JSM-7800 F). The X-ray diffraction (XRD) patterns of powders were recorded using an X-ray diffractometer (Fangyuan, DX-2700). The contents of elements in Ti-6Al-4V powders were analyzed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES, Thermo Scientific iCAP6300). The oxygen content of powders was measured using a gas analyzer (Leco TCH600). The flow parameters (dynamic flow properties, compressibility, aeration, permeability and shear properties, etc.) of powders were tested using a powder flowability analyser (Freeman FT4 powder rheometer). Flow parameters and their definitions as shown in Table 1.

The schematic diagram of the FT4 powder rheometer is presented in Fig. 1. After being weighed, the powder sample is loaded into a cylindrical vessel. A twisted blade is forced to pass through the powder column along a helical path. The blade motion imposed forces causing the deformation and flow of the powder that are continuously measured to calculate the flow energy essential for the powder to flow. Different flow patterns are obtained by adjusting the starting height, final height, helical path angle, axial and rotational speeds and moving direction (downwards or upwards, clockwise or anti-clockwise) of the blade. More details can be found in Refs. [17,18].

3. Results and discussion

3.1. Morphology, phase constitution, composition and particle size

Fig. 2 shows the SEM morphologies of the RP and the SP of Ti-6Al-4V and presents the EDS analysis of the SP of Ti-6Al-4V. The RP was irregular with noticeable edges and corners as shown in Fig. 2(a). After the plasma spheroidization, the SP of Ti-6Al-4V had smooth surfaces and favourable dispersion as observed in Fig. 2(b) and (c). They became regular and spherical, and the degree of sphericity was nearly 100%. During the RF plasma spheroidization, the RP of Ti-6Al-4V passed through a high-temperature plasma region and rapidly absorbed the heat and melted. The irregular edges of the RP melted preferentially and condensed into spherical shapes under the effect of surface tension. Further, the powders entered the cooling chamber, and SP was formed after quenching [19].

Fig. 3 presents the XRD patterns of the RP and the SP of Ti-6Al-4V. The diffraction peaks of the RP and the SP appeared at the same diffraction angle, indicating that the plasma spheroidization process did not affect the phase constitution of the SP [20].

Table 2 lists the chemical compositions and oxygen contents of the RP and the SP of Ti-6Al-4V. After the plasma spheroidization, the Al content decreased, while the V, Fe and C contents increased. These changes may be attributed to the sublimation of few Al atoms changing the chemical composition of the Ti-6Al-4V powders because of its relatively low vaporization point. Some spherical nanoparticles were observed on the surface of the SP of Ti-6Al-4V as shown in Fig. 2(b) and (c), because the RP with smaller particle size easily absorbed more energy and formed nanoparticles during the plasma spheroidization. These nanoparticles had larger specific surface energies, thus resulting in higher oxygen content achieved. Therefore, to control the oxygen content of the SP, it is

Table 1
Flow parameters and their definitions or descriptions.

Nomenclature	Units	Definition or description
Basic Flowability Energy (*BFE*)	mJ	The energy needed to displace a conditioned powder sample during downwards testing. In this study, the helix angle is negative 5°, the diameter of blade is 23.5 mm, the blade tip speed is 100 mm·s^{-1}, the diameter of vessel is 25 ml except the shear test using a 50 mm diameter. The basic flowability energy is determined by the seventh test
Stability Index (*SI*)		The factor by which the measured flow energy changes during repeated testing or processing. It is equal to the ratio of flow energy of the seventh test to the first test
Flow Rate Index (*FRI*)		The factor by which the flow energy is changed when the blade tip speed is reduced by a factor of 10. It is equal to the ratio of flow energy of the eleventh test to the eighth test
Specific Energy (*SE*)	mJ·g^{-1}	The energy per gram needed to displace conditioned powder during upwards testing. It represents a measure of how the powder will flow in a relatively low stress environment
Conditioned Bulk Density (*CBD*)	g·ml^{-1}	Bulk density of a conditioned powder sample
Aerated Energy (AE_n)	mJ	The energy needed to displace powder sample while being aerated at n mm·s^{-1}
Compressibility (CPS_m)	%	The percentage change in volume after compression while apply a normal stress of m kPa to powder sample. It indicates the ability of cohesiveness of the powder
Pressure Drop (PD_m)	mBar	The decreased pressure while passing air through the powder at a speed of 2 mm·s^{-1} while apply a normal stress of m kPa to powder
Cohesion (*C*)	kPa	Shear strength at zero normal stress
Major Principal Stress (*MPS*)	kPa	Major consolidation stress given by Mohr stress circle of steady state flow
Unconfined Yield Strength (*UYS*)	kPa	A previously consolidated cylinder of powder could withstand before plastic deformation
Flow Function (*FF*)		The ratio of the major principal stress to the unconfined yield strength