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Characterization of titanium powder flow: A review on current status on flowability

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ABSTRACT

Particle size, shape, density, chemical composition and moisture content are essential characteristics that determine the ability of powder to flow. These factors are mainly affected by powder production technology. Various powder flowability measurements methods such as angle of repose, avalanche angle, compressibility index, flowmeters, bed expansion ratio, capillary tube and shear test were intensively reviewed. The shear test method was found to provide more flowability properties such as angle of internal friction, cohesion, flow function and kinematic wall of friction than other methods. However, studies in titanium powder flowability have only been conducted using dynamic angle of repose and flowmeter revealing an existing knowledge gap that calls for more research.

KEYWORDS

Titanium powder;
Characterization;
Flowability;
Additive manufacturing.

INTRODUCTION

Titanium can be alloyed with iron, aluminium, vanadium, molybdenum, among other elements, to produce strong lightweight alloys for aerospace (jet engines, missiles, and spacecraft), military, industrial process (chemicals and petro-chemicals, desalination plants, pulp, and paper), automotive, agri-food, medical prostheses, orthopedic implants, dental and endodontic instruments and files, dental implants, sporting goods, jewelry, mobile phones, and other applications[14,16,21].

The two most useful properties of titanium are corrosion resistance and the high strength-to-weight ratio. In its unalloyed condition, titanium is as strong as some steels, but 45% lighter. There are two allotropic forms and five naturally occurring isotopes of this element, ^{46}Ti through ^{50}Ti, with ^{48}Ti being the most abundant (73.8%)[14]. Titanium and its alloys are classified into 31 grades[4] with grades 1 to 4 being referred to as commercially pure (unalloyed) while the rest are alloys with different properties[12] such as ductility, strength, hardness, resistance to corrosion, electrical resistivity and creep resistance[14].

Wrought and cast titanium is available in the market in form of billet, bar, plate, sheet, strip, hollows, extrusions, wire, powder etc.[16]. Because of chipping and premature tool failure[16] conventional machining operation on titanium such as turning, milling, reaming, drilling, tapping, sawing and grinding are cumbersome and challenging[45]. This is caused by the tendency of tita-

nium metal to weld on the cutting tool as well as the presence of high interface temperatures and stresses between the tool and the work piece during machining[16]. Powder metallurgy and additive manufacturing processes of titanium powder are recommended[26], while utilizing the advantages of powder forming process[45].

Flowability is one of the essential physical characteristics considered during powder manufacturing process. Studies on flowability have only been conducted on pharmaceutical powders like Ibuprofen and other powders such as alumina, soda, limestone, cement, dolomite, aluminum oxide trihydrate, coal and clay[7,32]. Little research on flowability of titanium powder has been conducted.

This review paper focuses on characteristics of titanium powder flow in an additive manufacturing context. However, since many factors affect powder flow, the characteristics of commercially available powders will fall within the ambit of the paper.

Titanium in additive manufacturing

Additive Manufacturing (AM), previously referred to as rapid prototyping[17], is a process of joining materials layer upon layer to fabricate objects from 3D model data[33] in form of continuous slices[44]. Various types of AM processes[44] exist, namely,

- Fused deposition modelling (FDM): The feedstock material in molten or partially molten state is extruded through a heated nozzle and deposited in the mould.
- Laminated object modelling (LOM): It entails cutting material into layers using a laser and adding them together layer by layer to form a solid laminated block.
- Selective laser sintering (SLS): Powder is sintered using a laser.
- Stereolithography (SLA): Photosensitive resins are cured using a laser.
- 3D printing: Powder particles are bound together by a liquid binder injected by a inkjet printer cartridge.

Metallic powders such as titanium, titanium alloy, stainless steel, aluminium oxide, silicon carbide, bronze, nickel, cobalt, chromium[44] as well as non metallic powders such as plastics, wax, papers, glass, polymer among others have been used as feedstock materials[39] in AM processes.

AM process produces titanium parts with complex shapes, having net or near net shape which require limited finishing processes or none[5], thus only small amounts of material waste is produced[45]. These produced parts, also have good mechanical properties such as strength, near full density, porosity and stiff moduli of elasticity, etc., all of which are enhanced by sintering processes[15]. However, the drawbacks of AM process are limited maximum part size and low manufacturing speed.

Titanium powder production

Pure titanium powder is produced by the Armstrong Process[45] where titanium tetrachloride ($TiCl_4$) is injected into a flowing stream of liquid sodium (Na+) producing a two phase mixture of Ti powder and the by-product of sodium chloride (NaCl). Titanium alloy powder is produced mainly by either the blended elemental or the pre-alloying techniques, followed by powder consolidation.

Traditionally, titanium production is through the Kroll process which involves chlorination of TiO_2 ore at high temperatures in the presence of carbon and then reacting the resulting $TiCl_4$ with magnesium to produce titanium sponge[53]. In the blended elemental approach the sponge fines are blended together, then hot pressed isostatically and sintered to near full density[24].

Various processes are used in the production of prealloyed titanium powder such as titanium Ti_6Al_4V Grade 5[6], which include;

- The Hydride- De-Hydride (HDH) process where raw material in the form of solid scrap, billet or machined turnings are cleaned from impurities then hydrogenated to produce brittle material which is ground in the presence of inert argon gas[55].
- The Plasma Electrode Process (PREP) where Ti grade 5 alloy in the form of a rotating bar is arced with gas plasma[21]. As the bar rotates the molten metal is centrifugally flung off, cooled and collected[55].
- The Titanium Gas Atomisation (TGA) process where melting of titanium metal is done in a vacuum induction skull melted in a water cooled crucible[20]. The molten metal is collected and atomized with a

stream of high pressure inert gas[55].

- The Plasma Atomization (PA) process employs 3 inert gas plasma jets to atomise titanium wire to form powders[20,21].

All of the above production processes produce spherically shaped particles except for the HDH process that produces angular shaped particles. Spherically shaped particles can achieve full density[21], thus the parts produced have no porosity or void defects.

Titanium components production

Various powder consolidation methods are used to produce final additive manufacturing products, *inter alia*,

- Metal injection or powder injection moulding (MIM), where the metal powder is mixed with polymer binder to form the feed stock that is injected into the mould[21] after which the debinding[48] is done, followed by vacuum sintering.
- Direct powder rolling process (DPR), where powder produced by elemental processes is used in fabrication of both single and composite multilayered sheet and plates[26].
- Laser engineered net shaping (LENS) process, where a high power solid state laser is focused on to the metal to make a melt pool[2]. The metal powder is injected into the pool to increase the volume of material while the component is built up layer by layer[26].
- Hot isostatic pressing (HIP), where powders are consolidated close to their relative densities by using high temperature and pressure[5,21].
- Spark plasma sintering (SPS), which consolidates powders close to their maximum densities[25] by application of resistive heating and pressure[8].

CHARACTERIZATION OF TITANIUM AND TITANIUM ALLOY POWDERS

In Material Science, characterization is defined as the use of physical techniques to investigate the composition, internal structure and properties of materials[3]. Research has shown that for a manufacturing industry to be more efficient in its production operation it has to embark on the use of sound prototype components[26]. In this regard, titanium and titanium alloys are gaining more industrial applications because they have better properties, such as high fatigue strength, low density and good corrosion resistance, than stainless steel and cobalt chromium alloys[47].

Various physical parameters such as particle size distribution, shape, chemical composition, density and soundness among others are used to characterize titanium powder[19], however these parameters also affect its flowability[38].

Particle size distribution

Titanium powder production methods produce particles with various particle sizes i.e. BE 45-180μm, HDH 50-300μm, PREP 100-300μm, TGA 50-350μm and PA 0-200μm particle size ranges[40,41]. Grain size and shape affect the particle movement, permeability and angle of friction.

Particle size is measured in wet or dry conditions. In dry conditions methods such as sieving, optical microscopy, transmission electron microscope and scanning electron microscope are used, but these methods do not measure particles in solutions[42]. In wet conditions interactive force apparatus and various laser source methods such as dynamic light scattering and laser diffraction are used[49] even though the latter is not suitable for use in a high particle concentration solution[42]. The above size determination methods are discussed here under,

- Sieving method is carried out according to ASTM C136 where mesh screens of reducing sizes[41] and mechanical shaker are used. Particle size range of 45 -710μm can be determined by sieving[45] in either dry or wet conditions. In practice this method enables particles of various size ranges to be separated easily but its major drawback is that some powder sticks to the mesh holes preventing further separation[34]. TABLE 1 shows a mesh comparison for titanium powder currently being used by the manufacturers such as Timet Powder Metals.
- Optical microscopy methods have been used to study the structure of powder and metals[29], however they are limited in their use since they have small depth of focus, low magnification x 2000, their resolution depends on wavelength of light used and, in addition, the specimens have to be polished[29].
- Transmission electron microscopy has high magni-

TABLE 1 : Mesh size comparison chart[51]

Mesh	Microns	Inches	Millimeters	Netafim Disk Ring Color	Object
3	6730	0.2650	6.730		
4	4760	0.1870	4.760		Gravel starts at 4.75 mm
5	4000	0.1570	4.000		
6	3360	0.1320	3.360		
7	2830	0.1110	2.830		
8	2380	0.0937	2.380		
10	2000	0.0787	2.000		
12	1680	0.0661	1.680		
14	1410	0.0555	1.410		
16	1190	0.0469	1.190		Eye of a Needle = 1,230 microns
18	1000	0.0394	1.000		
20	841	0.0331	0.841		
25	707	0.0280	0.707		
28	700	0.0280	0.700		
30	595	0.0232	0.595		
35	500	0.0197	0.500		
40	420	0.0165	0.420	Blue	
45	354	0.0138	0.354		
50	297	0.0117	0.297		
60	250	0.0098	0.250		
70	210	0.0083	0.210		
80	177	0.0070	0.177	Yellow	
100	149	0.0059	0.149		
120	125	0.0049	0.125	Red	
140	105	0.0041	0.105	Black	
	100	0.00394	0.100		Beach sand (100 – 2,000 microns)
170	88	0.0035	0.088		
200	74	0.0029	0.074		Portland cement
	70	0.00276	0.070	Brown	Average Human Hair (70 – 100)/Grain of salt
230	63	0.0024	0.063		
	55	0.00217	0.055	Green	
270	53	0.0021	0.053		
	50	0.00197	0.500		Remove visible particles from liquid
325	44	0.0017	0.044		Silt (10 – 75)
	40	0.00157	0.040	Purple	Lower limit of visibility (Naked Eye)
400	37	0.0015	0.037		Plant pollen
(550)*	25	0.00099	0.025		White blood cells/level to achieve “optical clarity’ in a liquid
(625)	20	0.00079	0.020	Gray	
(1200)	12	0.0005	0.012		
(1250)	10	0.000394	0.010		Talcum powder/level to remove haze from liquid/fertilizer (10 – 1,000)/ mold spores (10 – 30 microns)
	7	0.000276	0.007		Red blood cells (8 – 12 microns)
(2500)	5	0.000197	0.005		Bacteria (0.5 – 20 microns)
(4800)	3	0.000118	0.003		
(5000)	2.5	0.000099	0.0025		Cigarette smoke & bacteria (Cocci) =2 microns
(12000)	1	0.0000394	0.001		Cryptosporidium (1 – 10 microns)

*** Mesh numbers in parentheses are too small to exist as actual screen sizes. They are only estimations and are included for reference**

fication of x 300,000. It is usually used to study the effects of heating, cooling, deformation and oxidation when fitted with the necessary accessories[29]. However, the equipment is fairly costly and it requires great care and skill to operate[29].

- Scanning Electron Microscope (SEM) is used to study surfaces of metal, plastic, ceramic and biological materials such as blood, tissue or live insects[29]. The specimens are usually prepared for non condutive materials by depositing a thin layer of metallic or carbon coating to avoid specimen charging. It has a large depth of focus of about 300 times that of optical microscope. The images are formed by scanning[29] and are analyzed by size digital programmed software.

Laser diffraction and scattering method uses principles of Fraunhofer diffraction and Mie scattering theory[50] to measure particle size distribution using particle diameter and diffraction or scattering pattern relationship[50]. The particle size distribution is determined from two different light intensity distributions in which one is generated by the particles while the other, is determined using theoretical scattered light intensity using either Fraunhofer diffraction or Mie scattering method for particles whose diameter are known[50]. This technique is based on the principle that particles passing through a laser beam will scatter the light rays at angles related to the particle sizes, where larger particles will scatter light at narrower angles and higher intensity than smaller particles[35]. The sizes obtained represent the volumetric diameter[35] of the particle which are represented inform of a cumulative frequency curve. Parameters D10, D50 and D90 are used to represent the particle size range diameters, with D10 representing the smallest, D50 the average size and D90 the maximum particle diameter[31,37]. However this technique assumes that particles are spherical in shape hence it is not suitable for non spherical[50] particles. In addition, elongated or agglomerated powders particles which are difficult to break by sonication are measured as one large particle leading to inaccurate particle size range readings[42].

- In the Interactive Force Apparatus (IFA) method[42] particle size distribution in the size range of 147-471nm are determined. The process involves suspending the particles in a functional fluid (aqueous, non aqueous solution or organic solvent) under magnetic or electric field thus determining the extent of particle dispersion and coagulation[42].

Recent studies show that particle size distribution is mostly being determined by laser diffraction method[6]. This is because of its ability to determine particle sizes less than 1μm and also gives more accurate results than earlier discussed methods. The common laser diffraction equipments in use are; Malvern master sizer analysette (Nanotec), BT-9300H or coultier Laser particle analyser fitted with sonication and diffoculant that breaks up agglomerates[15].

Particle's shape and size is described by three axes namely length (a-axis), width (b-axis) and thickness (c-axis). Length is taken to be the maximum caliper dimension while the thickness is the orthogonal dimensions to the a-axis or b -axis[49]. The ratio between the width and the length is referred to as aspect ratio (AR). AR gives information on the production process used to produce the particles and the transportation technique employed[35]. The two main methods used to measure particle axes are the manual technique and image analysis method. In the manual technique each particle is measured by hand with the help of a Danish box or a caliper, though the method is cumbersome[49]. In the image analysis method also referred to as digital sieving uses computers where particles images are analyzed by already programmed software that interprets quantitative data from digital images.

Particle shape or morphology

The shape of a particle, whether natural or crushed, normally has complex morphology and is not uniform[49]. Shape is described in three methods namely form, roundness and surface textures although, in some studies, form has been termed as shape without any regard to roundness and surface texture[49]. These methods are briefly described below;

- Form depends on relative dimensions of the three axes of a particle and is usually expressed in particle axial ratios and can be plotted on zingg or sneed and folk diagrams. These axial ratios are classified into four groups namely flat (disk shaped or oblate), spherical (equant), columnar (rod like or prolate) and flat and columnar (bladed)[49].

Review

- Roundness is the smoothness of a particle and it shows how the edges are curved (curvatures of the corners), usually defined by[49];

$$P = \frac{\Sigma r_n}{R}$$

where

P - Roundness.

r_n - Average radii that can be fitted into all corners.

R - Radius of the largest inscribed circle.

It can also be determined through high magnification observation by[45];

$$P = \frac{4A\pi}{K^2}$$

where

P - Roundness.

A - Area enclosed by the boundary of the particle.

K - Perimeter of the particle.

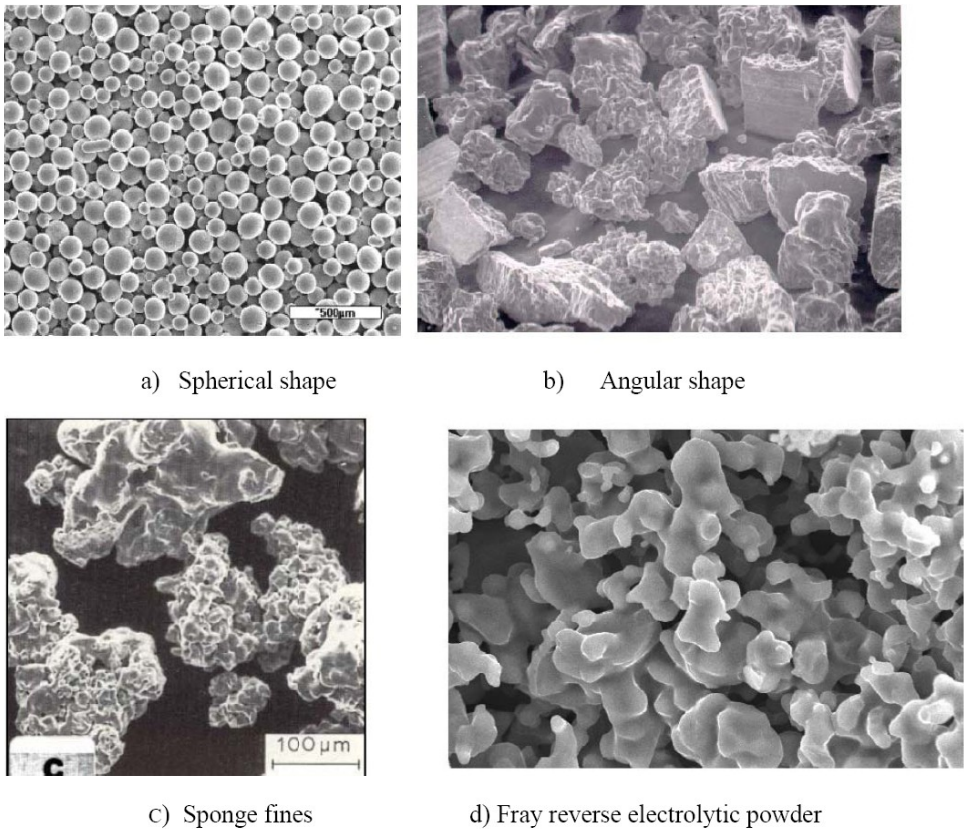
However in practice, particle roundness is done by comparing a particle with standard silhouette charts or photographic images[49].

- Surface texture shows features or the outer physical appearance of a particle. It gives an indirect estimation of intuitive qualities such as roughness, smoothness or bumpiness (irregularity of surface between corners). Surface texture gives information on transport mechanism and deposit setting of sediments[49].

Both spherical and angular shaped titanium powders are readily available in the market and have wide industrial applications, however, other shapes like sponge fines, flake and coral also exist. Figure 1 shows the most common particle shapes of Titanium powders available in the market. The particle shape is determined using SEM[45]. This type of microscope also shows other particle properties such as grain boundaries, size and chemical composition when fitted with Energy Dispersive Spectrometry (EDS)[45].

Spherical shaped particle powders have better packing density, good flowability and a smoother macroscopic surface[22]. In addition, they have uniform pore distribution, good air permeability and are suitable for thermal spraying since they produce symmetrical and homogeneous layers[22]. The sphericity of the particle ø is given by the ratio of surface area of the equivalent volume sphere to the particle surface area[35].

Angular particles also referred to as ground par-



a) Spherical shape b) Angular shape

c) Sponge fines d) Fray reverse electrolytic powder

Figure 1 : Titanium powder shapes[21]

ticles have irregular shapes. Their characteristics are low fluidity index, low bulk and tap density, high degree of compression and high angle of repose[22]. In this regard, they have poor flow characteristics and are unsuitable for moulding processes such as injection, sintering and hot pressing[22]. However, angular shaped particle powders are still being used to produce low tensile strength parts such as valves for production models, the Toyota Altezza family automobile, golf club heads and softball bats[21] despite poor flow properties.

The properties of angular particles are enhanced by spheroidization technology. This technology involves converting angular shaped particles to spherical shape[22]. It employs Particle Composite System (PCS) consisting of high speed rotary mixer fitted with blades in a ring shape chamber operated between 2000 to 8000 revolutions per minute and at a temperature of 45 to 75^0C for a minimum of 20 minutes[22]. When these particles are sintered they possess superior properties than those obtained from original spherical powder[22].

Chemical composition and crystal structure of titanium

The titanium metal is a dimorphic allotrope whose hexagonal alpha form changes into a body-centered cubic (lattice) β form at 882 °C (1,620 °F)[16]. The specific heat of the alpha form increases dramatically as it is heated to this transition temperature but then falls and remains fairly constant for the β form regardless of temperature. An additional omega phase exists, which is thermodynamically stable at high pressures, but is metastable at ambient pressures. This phase is usually hexagonal (ideal) or trigonal (distorted) and can be viewed as being due to a soft longitudinal acoustic phonon of the β phase causing collapse of (111) planes of atoms[32].

The formation of alpha and beta phases in the titanium alloy depends on the amount of alloying additions present. Alpha alloying elements stabilise the alpha phase[32] by increasing the beta transus temperature. These elements include aluminium, oxygen, nitrogen, carbon, gallium, germanium, lanthanum and cerium[32]. However, aluminium is commonly used as the alloying element up to 8% content due to its low density. The alpha phases have high modulus of elasticity, tensile and creep strength[16].

Alternatively, some alloying elements stabilise the beta phases by lowering the beta transus temperature[32]. The elements are divided into two groups namely beta isomorphous and beta euctoid. Beta isomorphous are completely soluble in titanium and they include elements such as molybdenum, vanadium, tantalum, rhenium and niobium[32]. However, rhenium and tantalum have high particle density hence they are not preferred for use.

Beta euctoid elements are partially soluble in titanium and form intermetallic compounds by euctoid decomposition of the beta phase. They include manganese, iron, chromium, cobalt, nickel, copper, silicon, hydrogen and silver elements[32]. Chromium, iron and silicon are commonly used. Hydrogen, on the other hand, causes embrittlement effect (formation of cracks) by precipitating into hydrides, thus, its content in titanium alloy is minimised. Beta phases are easy to fabricate both in hot and cold working because they have low modulus of elasticity and are corrosion resistant.

Tin, zirconium and Hafnium are considered as neutral elements since they do not have any effect on the transus temperature[32]. However when tin and zirconium dissolve in aluminium they cause hardening effect that stabilises the alpha phase.

Titanium alloys have been classified into four categories as commercially pure alloys, alpha and near alpha alloys, alpha-beta alloys and beta alloys[16]. They are briefly described here-under;

- The commercially pure alloys contain small amounts of oxygen and iron and are not heat treatable since they are single phase alloys[16]. They have excellent corrosion resistance but are low in strength.
- Alpha alloys are single phase alloys since they do not contain any beta alloying elements. Ti5Al2.5n is the only true alpha alloy. Alternatively, near alpha alloys have small amounts of beta phases up to 2 % in their matrix and therefore, are heat treatable[16]. They are good in weldability, notch toughness, corrosion and creep resistance.
- The alpha and beta or dual phase alloys contain both alpha and beta phases. They are heat treatable and have superior ductility and strength properties when compared to their individual phases[16]. Titanium grade 5 alloy falls into this group.
- The beta alloys have sufficient percentage of beta

stabilizing elements and small amounts of alpha phases making them heat treatable[16]. They are good in cold forming and offer high strength up to 700^{0}C.

The alpha and beta phases are determined crystallographically by X ray Diffraction (XRD) while the composition of alloys is determined by X ray fluorescence (XRF), however, oxygen, nitrogen and hydrogen contents are determined by combustion methods.

The overall density of titanium alloy is determined by the densities of the alloying element present. Beta stabilizers and neutral alloying elements have higher densities than alpha stabilizers[32].

The American Society for Testing and Materials (ASTM) chemical analysis standard for cast and wrought titanium powder (ASTM F1108 and F1472 respectively) by percentage mass is given in TABLE 2.

Density

Both bulk density and particle density are of importance in titanium powder characterisation. Particle density is the mass of a particle divided by its volume excluding open and closed pores and is not dependent on the degree of compaction[1]. Essentially, it is the density of the individual grains within the sample. If the grains or particles comprise several phases, the particle density is the average density of the phases present. The particle density is expressed as[56];

$$\rho_{particle} = \sum_{i=1}^{i=n} f_i \rho_i$$

where:

f_i = fraction of phase i in the particle

TABLE 2 : Titanium powder chemical analysis[4]

Element	Al	V	Fe	Si	O	C	N	H
Ti 1 max	-	-	0.21	n.a	0.15	0.08	0.05	0.013
Ti 1 typical	-	-	0.05	0.01	0.11	0.01	0.04	0.005
Ti 2 max	-	-	0.25	n.a	0.25	0.08	0.05	0.013
Ti 2 typical	-	-	0.03	n.a	0.19	0.02	0.04	0.005
Ti 5 max	5.5 – 6.5	3.4 -4.5	0.25	n.a	0.13	0.08	0.05	0.012
Ti 5 typical	5.9	3.9	0.19	n.a	0.12	0.01	0.01	0.004

ρ_i = density of phase i.

The ASTM standard gives the particle density of titanium grade 5 alloys Ti6Al4V as 4.43 g/cm^3 (Bolzoni *et al.*, 2012b). On the other hand bulk density is the ratio of the mass and the volume of a sample including open and closed pores and is dependent on the degree of compaction. The relationship between particle density and bulk density is given by[56];

$$\rho_{bulk} = (1 - n)\rho_{particle}$$

where:

n = porosity of the material or sample

$\rho_{particle}$ = density of the particles

ρ_{bulk} = density of the bulk material or sample

The porosity of the material (n) is the ratio of open and closed porosity which is determined by weight measurements prior to and after dipping a compacted sample in a highly volatile solvent like xylol for 36 hours[15].

Different values of bulk densities are obtained when the powder is measured in different states for instance when settled, packed or compacted[1] and, therefore, has no definite value for a particular powder[1]. Compaction involves morphological modification of the granules while packing involves rearranging the powder particles such that there is a reduction of interparticle voids without changing the original particle shape or dimensions[10]. Bulk density shows the product quality and it is a very important control parameter for volumetric packing machines and during the calibration of moisture content machines[11].

For powders, bulk density is determined by two major methods. One method is to measure the volume of a known mass of a powder sample by pouring it through a sieve into a graduated cylinder while the other is to measure the mass of known volume of powder that has been passed through a volumeter into a measuring cup or vessel[10]. Bulk density is also used to measure powder flowability using random loose packing and tapped bulk density equipment. There are two types of random loose packing densities namely aerated density and poured or apparent density[10].

In the aerated density measurement, the powder is left to settle down under the influence of gravity by

means of a vibrating sieve or under fluidization[10] while, in poured density measurement, the powder is poured into a container gently. From the two methods discussed above, the values of mass and volume obtained are used to calculate their respective densities. Powders that resist collapse after being poured gently into a vessel have strong structural strength (strong particle cohesiveness) hence have low bulk densities, while those that collapse easily are structurally weaker (free flowing) hence have high bulk densities[1].

In tapped bulk density measurement, the container having random loose packed powder samples is tapped either by vibrating or hitting over a given period of time until the powder bed stabilises at minimum volume[10]. A cohesive powder collapses easily after tapping while a weak or free flowing powder shows minimal consolidation[1] since it exists in a consolidated state naturally.

Particle density is measured using a manual or a gas Pcynometer, a vessel with precisely known volume, using Archimedes principle[30], whereas bulk density is determined using highly volatile xylol and a precision balance equipped with density determination kit[15].

Soundness

Soundness or stability is the ability of the powder to resist disintegration when the powder is stored, transported or while in use as it comes into contact with the environment, through the absorption of moisture and environmental gases such as oxygen, nitrogen and hydrogen. This phenomenon may lead to either reduction or increase in the particle sizes which affects the powder flow.

Powder particles are usually surrounded by fluids mostly air between the particles such that both the particle and the fluid are used in the computation of the bulk property. When the powder is stored or packed for a long period, the entrained fluid escapes causing strong chemical bonds to form between particles resulting in agglomeration (http// www.freemantech.co.uk). This causes the formation of unwanted build ups, cakes, bridges and lumps that leads to poor flow properties (http// www.freemantech.co.uk).

From the literature reviewed, it is only the soundness of cement powder that has been determined, using weight ratios. The procedure involves passing the powder through a sequence of sieves of decreasing mesh under vibration[45]. The quantity of material P_M in each sieve tray is measured using high precision weighing balance machine. After the exposure to the environment, the same process is repeated and new weight measurements P_N are obtained for each sieve. The powder soundness is then determined by the percentage change in weight as shown below[9];

$$\text{Degree of soundness } (\%) = \frac{P_M - P_N}{P_M} \times 100$$

Therefore, a soundness standard for titanium powder needs to be developed.

Flowability

In the design and operation of powder handling processes such as storage, discharge, transport and packing, powder flowability is one of the important factors to be considered. Powders should posses good flow properties. For example, when being transported from a silo or a hopper, powders should be able to flow easily to avoid the formation of large holes evolving from significant bridging effect that leads to inconsistent flow rates and segregation[31]. During pneumatic transportation the powder should be able to expand uniformly to avoid powder agglomeration and clumping when fluidized by a gas[31]. In addition a good flow characteristic is essential during mixing of powders to ensure uniform blending and homogenous mixture[31].

Powder flowability depends upon particles' properties such as particle size, shape (uniformity and roundness), density, attrition, surface coating, adhesion and agglomeration properties, compressibility, hardness, stiffness, strength, fracture toughness[38] and other external factors such as vibration, temperature, humidity, electrostatic charge, aeration, transportation, gravity forces, surface forces (static and dynamic friction) and storage time[35,38], hence the complexity of flowability models here under;

For a spherical shaped particles[35];

$$\rho_{\text{Particle}}\, d^3 g\varepsilon \geq (1+k)F_{H0} + kF_N$$

Where:

ρ_{particle} – Particle density.; d - Particle diameter (usually the smallest diameter D10 in laser diffraction method); ε – porosity of the material.; g – gravitational force.; k - materials contact conslidation constant; F_{HO} - Adhesion force between particles with no external normal force; F_N - Normal

force applied to the powder.

While for a non spherical shaped particles[35];

$$\rho_{Particle} d^3 g \varepsilon \emptyset^2 \geq c\pi(1+k)F_{H0} + kF_N$$

where:

$\emptyset$ – Particle sphericity

c – Proportionality constant

Powder particles starts to flow when the product of the parameters in left hand side (LHS) is equal or greater than the right hand side (RHS). The parameters in the LHS of these two flow models equation are easily measurable and significantly affect the powder flow rate. In this regard, any increase in particle density, size and material porosity will have a directly proportional effect on the powder flowability for spherical powders[35]. However, if particle density is taken as a constant (not easily variable), thus particle flowability will depend on the particle size and the porosity of material[35].

The difference between LHS of the two models is $\emptyset^2$ (the square of sphericity factor), however sphericity factor is a ratio less that unit, thus the above models confirms the free flowing nature of spherical shaped particles when compared to non spherical particles[35].

The above models where developed under the assumptions of ideal conditions such as there is a constant inter-particle distance between particles of the same powder, however in practice different production batches have different material properties thus it is recommended each batch to be tested separately[35] to determine its flowability.

In general, fine particles <100 μm are more cohesive and, therefore, less free-flowing than larger, denser particles[1] which are non cohesive and permeable. However, the flow properties of fine powders are improved by blending it with dry nano size material that acts as the flow additive[31] thus reducing the van der waals forces. When fine particles <10 μm are aerated and forced to flow, they behave like fluids and are able to flow through holes and round corners unlike the coarse particles that interlock upon each other hence resisting the flow (http// www.freemantech.co.uk).

Therefore, prediction of powder flow properties depends on the material flow properties, equipment processing characteristics, handling and processing conditions such as static and dynamic head, consolidation, aeration level, absorbed moisture and the electrostatic charge present. Some of the common problems encountered in powder processing industry are (http// www.freemantech.co.uk);

- Blockages on the hopper or bin discharge.
- Changes of powder density due to aeration.
- Weight variation caused by irregular powder dosing operation due to the entrained fluid.
- Agglomeration caused by particle coalescence.
- Particle attrition due to the wear and tear of the moving particles in a conveyor.
- Segregation caused by non cohesive powders having different bulk densities and particle size.
- Consolidation caused by high level of compaction in the silo or hopper during storage or transportation.
- Effects of moisture in the powder causing poor flow properties.
- Controlling of the fluidized powder which behaves as a fluid.
- Poor plant design that makes the powder to flow in an irregular pattern (first in, last out).

The powder flowability affects the quality of the final product. For instance in high speed drug tabletting, uneven flow causes capping and lamination defects[35] while in powder metallurgy products are produced with sections having imperfections and voids or porosity[45].

Several experimental methods for powder flowability measurement are available, and the choice of method depends on the specific property to be measured[31]. Some techniques determine the flow rate when the powder is in a static state (stored) while others determine flowability in a dynamic state (transported)[38]. Traditionally methods such as angle of repose, compressibility index and funnels have been used to measure powder flowability. With advancements in technology, avalanche angle and shear test methods have been developed although the latter has also been used in the silo and hopper design[18]. Currently, the most common methods in use are angle of repose, avalanche angle, compressive index flowmeters, bed expansion ratio, capillary tube and shear cell tests. The experimental results obtained from the above mentioned methods are usually modelled using computer software programs such as Matlab and student t test[13].

Angle of repose and avalanche angle

The angle of repose is, as shown in Figure 2, the angle at which a conical pile of powder maintains its stability. The angle of repose is related to the rate of powder flow as shown in TABLE 3. It is assumed that lower angles of repose materials are indicative of lower inter-particle forces and more flowable material. Materials with a high angle of repose may still flow uniformly, but require a greater amount of energy input to maintain steady motion. Decreasing the particle sizes of powders results in poorer flow properties[31], suggesting that finer powders have higher angles of repose.

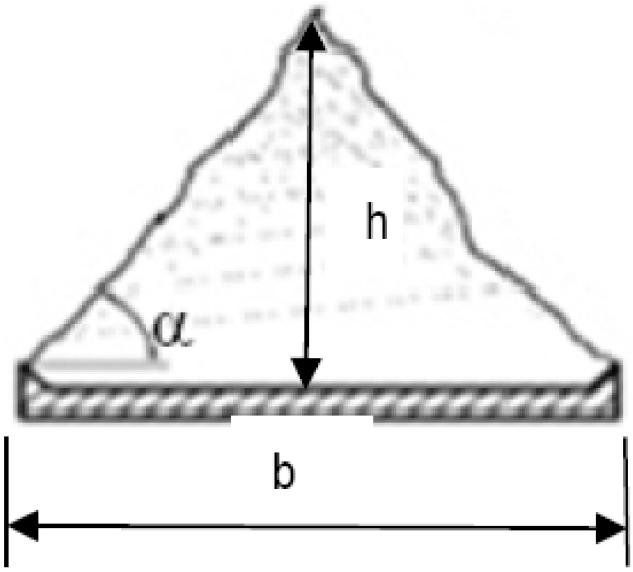


Figure 2 : Static angle of repose[36]

TABLE 3 : Flow property[36]

Flow property	Angle of repose (°)	Hausner ratio	Carr's Index
Excellent	25-30	1.00-1.11	5-15
Good	31-35	1.12-1.18	12-16
Fair	36-40	1.19-1.25	18-21
Passable	41-45	1.26-1.34	
Poor	46-55	1.35-1.45	>23
Very poor	56-65	1.46-1.59	
Very very poor	>66	>1.6	

Known methods of determining the angles of repose both in static and dynamic states are described briefly. In static angle of repose, powder is poured in a fixed height funnel above the flat base or the funnel is filled with powder then raised gradually to allow the powder to fall out[23]. Pictures of the pile are taken using a digital camera and analysed by an image analysis program, when the powder heap has build up until further addition of powder slides down the slope[13]. The angles measured on the right and left hand sides of the pile are averaged to get a single static angle of repose[13]. Alternatively the powder heap is formed on a circular support of diameter b and the heap height h is measured. The angle of repose α is then calculated from $\tan\alpha = \frac{2h}{b}$[23] as shown in Figure 2.

The dynamic angle of repose is measured when the powder is in motion in a tilting table or a circular rotating tumbler (a revolving cylinder). The powder forms a fixed bed that rotates with the supporting edges of the cylinder until it reaches a point that it can no longer support its weight making it to collapse[45]. The maximum angle attained before collapse is known as avalanche angle[31].

The dynamic angle of repose is developed when the fixed bed rises inducing a thin rapidly flowing layer of material where particle decelerate and return to the fixed bed[45]. Previous experiments have shown that Froude's number given by the following expression[45], $F_r = \frac{R\omega^2}{g}$, where R is the radius of the tumbler, ω is the rotational speed and g is the acceleration due to gravity, offers reliable prediction of powder flow[45]. To obtain a continuous flow of the thin layer powder the Froude number for titanium powder[45] should be between 5×10^{-4} and 5×10^{-3}. Data obtained from rotating drum tumbler experiment is non-dimensionalised by Froude number[45].

However, it is inappropriate to extend results obtained from static evaluation process to dynamic process and vice-versa. Since static technique determines powder stability properties (storage) whereas dynamic technique determines dynamic properties (transportation), therefore both static and dynamic processes should be carried out independently[38].

Compressibility index

The Hausner Ratio (HR) and Carr Index (CI) are determined using pour bulk density (untapped) and tapped density. The powder is gently loaded into a cylinder through a funnel and weighed to calculate Bulk Density (BD). To determine powder tapped density (TD) the cylinder is tapped or mechanically raised and

lowered at a set distance until a consistent volume is reached that corresponds to the maximum packing density of the material. The two ratios are determined as shown by the following expressions[35],

$$HR = \frac{TD}{BD} \text{ and } CI = \frac{TD - BD}{TD} \times 100\%$$

TABLE 3 shows HR and CI flow property values. The accuracy of the results depends on both the equipment sensitivity[13] and the operator's precision while taking the reading[23].

Flowmeters

Powder flow through orifice or a fixed funnel is determined by using either Hall flowmeter or Carney funnel equipment. As per ASTM B213 standard[36], Hall flowmeter has a funnel diameter of 0.1 inches and is used to measure flow rate and density of freely flowing metal powders. Carney funnel has funnel diameter of 0.2 inches and measures non free flowing metals[36]. The funnels used in both processes are similar and thus interchangeable. Therefore, to carry out the test only a density cup and one complete stand are required.

Bed expansion ratio (BER)

Powder is loaded into a clear acrylic vertical pipe to a certain depth then using a dry compressed air. At superficial velocity, the powder is fluidized from the bottom of the pipe, to make it homogenous[31]. The superficial gas velocity is reduced to a constant value allowing the bed to settle down, at which point the first reading is taken. Then the gas flow is cut off and the second reading is done. The ratios of the two readings are computed. For particle size below 30μm the BER should be between 1.4 to 1.5 in order to achieve sufficient fluidization, while for larger particles the tolerance of the BER is reduced to compensate for the reduced specific surface area of the particles[31].

Capillary tube

This is a simple method where the powder is discharged through a capillary tube. Its sensitivity is higher than other methods. However, if the powder has higher inter-particle adhesion forces, bridging occurs inside the capillary making it difficult to take any measurements even with the help of vibration[38].

Recently, a vibrator shear tube to measure flowability has been developed[38]. It has a piezoelectric vibrator, glass tube and a metallic bottom. The vibration is transferred directly to the particles in the narrow gap between the vibrating tube edge and the flat bottom surface[38]. The particles experience high shear forces that overcome friction and adhesion forces. Therefore the particles are able to pass through the gap as both static and dynamic measurements are taken, although errors due to interference occur.

Shear test

Shear test method on the powder is carried out using either direct or indirect methods. The operation principle used in the direct method equipment is either linear, ring or rotational while the indirect method uses axial cells. Powder rheometer shear testers are either linear or rotational, for instance FT4 powder rheometer is rotational, Schulze shear tester is a ring type while Jenike shear tester is linear. These types of shear testers are currently being used to determine flow properties in the industry[37]. The above methods use the same operation principle however Jenike shear tester is commonly used in the industry[37] because it obtains flow function used in predicting flowability of bulk material. A Mohr circle is constructed from different values of shear stress τ and normal stress σ obtained during pre shear (consolidation) and steady state flow in the shear cell (shear step) thus giving yield loci where flow properties such as angle of internal friction, cohesion, flow function and kinematic angle of wall friction are determined. The inverse of flow function is referred to as flow index FF_c that shows consolidated powder strength to be surmounted before flow occurs[13] .

The classification of powder flowability by flow index is shown in TABLE 4. From this table, a higher angle of internal friction signifies that the material flowability is (smooth) easy, steep slope and high cohesion values show that the flow of the powder is poor (hardest), while higher values of flow index shows that the flow is easier[13]. The major drawback of Jenike shear test is that it requires a certain level of expertise since the movement of shear stress is limited[38] and time consuming[13].

Discussion and summary

AM process is a preferred manufacturing method because it produces small amounts of waste. Addition-

TABLE 4 : Flow index[7]

Flowability	Hardened	Very cohesive	Cohesive	Easy flowing	Free flowing
Flow index (FF_c)	<1	>1to≤2	>2to≤4	>4 to ≤10	>10

ally, the parts produced have near net shapes, and in addition, their properties can be improved further by the sintering. Titanium powder particles produced by Blended Elemental (BE) approach are spongy- like in shape, while those produced by Pre-Alloyed (PA) are spherical except those produced by HDH method that are angular. Mostly spherical particles are used in AM process because they have superior flow characteristics, smooth macroscopic flow and are able to achieve near or full density[48]. To this end, there are spheroidization techniques that convert angular particle shape to spherical. However, the cost of production of BE and PA manufacturing processes is too high[21]. This has led to the development of other new technologies that rely on electrolytic reduction of titanium dioxide to titanium powder having fine homogeneous particles. Success in this technology will enable titanium powder to compete effectively in the market with other metal powders.

Titanium powder is consolidated by various methods. Laser engineered net shaping (LENS) and direct powder rolling (DPR) are used mainly for additive manufacturing to produce parts for medical implants and aerospace structures. In the metal injection moulding process a binder is mixed with the powder before being injected into the mould. However, it is not possible to remove all the binder during the debinding process thus introducing impurities in the powder. Further consolidation of the powder is done by hot isostatic pressing and spark plasma sintering. Hence the parts produced by this method are used in areas where tensile and impact properties are less important. To avoid contamination of the powder these processes are conducted in the presence of inert gas such as argon.

Titanium powder is classified either as commercially pure (CP) titanium or titanium alloy. The chemical composition (especially in terms of O, N, H contents) determines the powder grade. For CP titanium, oxygen level determines the powder grade as well as the tensile strength. By mass, grades 1 and 4 has oxygen level of 0.18% and 0.4% respectively, however as the oxygen level rises, so does the tensile strength. Oxygen and nitrogen regulate the tensile strength of the material by forming phase alloys, but they cause embrittlement when their levels are high (exceeding the limits shown in TABLE 2). High levels of hydrogen content in the alloy causes hydride to precipitate; hence the alloy product easily cracks when subjected to stress. The level of alloying elements present in the composition determines which type of phase will be formed whether alpha, beta or alpha and beta phases. However, since one of the advantages of the titanium alloy is low density, careful selection of alloying elements is required to maintain low density. In addition, an increase in powder density will also increase the cost of transporting the powder.

From the literature reviewed, particle density is determined by a pcynometer. Bulk density indicates the porosity levels and the way powder particles coalesce upon settling, consolidation or compaction. Materials with low density are more cohesive (not free flowing) because they have strong structural bonds between particles where else materials with high bulk density have weaker structural bonds hence they are free flowing[1].

Research has shown that particle size, shape, roundness and moisture content are the major factors that affect flowability. Spherical shape particles flow better than angular ones that are produced by grinding. High moisture content and particles sizes of < 100 μm (which tend to be round in shape), cause particles to have high cohesion due to the increase of van der waals forces between particles[13] and this leads to poor flow properties. Moreover, particle size influences flowability more than particle shape[45].

Flowability is also affected by the way the powder is packed, transported and stored which determines its soundness. In this regard, a soundness standard needs to be developed. In addition the quality of the product produced depends on the flow of powder to the mould. A uniform flow produces sound components while uneven flow produces components with defects such as imperfections and voids[45]. However, the powder processing industry is still being faced with powder handling and processing challenges which require solutions.

Hausner ratio and Carr's index use the same principle of compressibility to determine flowability. The lower the compressibility ratios, the better flow characteristics. However, the accuracy of the results de-

pends entirely on the operator, when using the powder and taking the readings (without errors), and the equipment sensitivity is usually ignored.

For the angle of repose, good powder flow properties occurs between 25° - 40° while angles >40° have poor flow properties as shown in TABLE 2. Both static and dynamic processes are conducted independently. Static process determines flow stability where else dynamic process determines both mass flow rate and particle flow[38]. This is because the two methods do not subject the powder to the same stress states[31].

Flowmeters require well trained operators and, in addition, they are unsuitable for flow measurement in high moisture content powder. Bed expansion ratio and capillary tube methods determine powder flow rate only, whereas shear test methods (Powder rheometer, Schulze and Jenike shear testers) determine various flowability properties such as angle of internal friction, cohesion, flow function and kinematic wall of friction which are also used in silo and hopper design. However, they are difficult to operate and time consuming.

The summary of these flow measurements methods is presented in TABLE 5. From the literature reviewed, Matlab and student t-test software programs have been used to model results obtained from the flowability measurement methods discussed in the preceding paragraphs.

Of all the literature reviewed, research on flowability on titanium powder has only been conducted using flowmeters and dynamic angle of repose. The literature reviewed did not indicate why the other methods of flowability were not used may be due to the notion that flowmeters give satisfactory results and are cost effective. Furthermore, there is no flowability test which has been universally accepted as being both reliable and easy to use. This leaves knowledge gaps, although it is not clear why this is so. Therefore this work recommends that the shear test method be experimentally investigated with respect to titanium powder.

TABLE 5 : Summary of flowability measurements methods

S/No.	Flow measurement method	Remarks
1.	Angle of repose and Avalanche	The two methods give results which are less accurate.
2.	Compressibility index	The accuracy of the results depend on the equipment sensitivity and the human operator
3.	Flowmeters	Results obtained are affected by the degree of moisture content, thus they are affected by the environmental conditions
4.	Bed expansion ratio	The process is difficult to control since fluidized powder behaves like fluid
5	Capillary tube	Frequent occurrence of powder blockage in the capillary tube during the test especially when testing small particles
6.	Shear test	This method gives more accurate results, although large amounts of sample material is required to carry out the test. Additionally, skilled operators are required to carry out the tests.

CONCLUSION

Flowability of powder depends on particle size, shape, density and moisture content. Static and dynamic processes are conducted independently and are used to determine flow stability and mass flow rate or particle flow, respectively. Various methods such as angle of repose, compressibility index, flowmeters, bed expansion ratio, capillary tube and shear test method are used to determine powder flowability, however shear test methods gives additional flowability properties such as angle of internal friction, cohesion, flow function and kinematic wall of friction which are also used in silo and hopper design.

Very little work has been done on titanium powder flowability. Infact, titanium powder flowability has only been studied using dynamic angle of repose and flowmeter revealing an existing knowledge gap. More research needs to be done in titanium powder flow preferably, using shear test method. Results obtained by shear test method could be factored into development of titanium powder soundness standard.

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