

Dynamic tap density as a powder characterisation technique for metal powders

Andri de Waal¹, Deborah Blaine^{1,*}, Francois Giliomee¹ and Melody Neaves¹

¹Stellenbosch University, Department of Mechanical and Mechatronic Engineering, Stellenbosch,

South Africa

*dcblaine@sun.ac.za

Abstract. Powder characterisation is critical for developing high-quality, consistent powder metallurgy processes and materials. Internationally standardised tests have been developed to measure specific powder characteristics that are relevant to specific powder metallurgy manufacturing techniques. Dynamic tap density is a new characterisation method for granular materials that has not yet been applied to metal powders. In this study, the dynamic tap density of different metal powders is measured and compared to standard powder characteristics. The results support the theoretical assumption that observing the dynamic tap density can give you further information on the powder characteristics and whether the powder will be suitable for a specific powder metallurgy process.

1 Introduction

Powder metallurgy (PM) encompasses a broad field of metal powder-based manufacturing techniques that include die-compaction, metal injection moulding (MIM) and additive manufacturing (AM) processes such as laser powder bed fusion (LPBF) and binder-jetting. PM offers unique manufacturing advantages as the shaping processes are typically near-net-shape, resulting in significant reduction in raw material waste and machining steps. PM can typically combine multiple formed and machined parts that require assembly into a single shaped part. An additional benefit is the ability to produce complex shapes from customised alloy compositions, made by elemental powder mixtures, especially for high-melting-point metals. Some PM processes, such as die-compaction, MIM and binder-jetting are comprised of separate forming and material solidification steps: the forming step produces a “green” shape where the powder particles are bonded together by a polymeric binder or by cold welding and interlocking of powder particles. In order to create solid metal bonds between the powder particles, the green part is then sintered. Sintering is a high temperature heat treatment process that activates solid state diffusion between powder particles, resulting in densification of the material and a solid part that may have some residual porosity (primarily arising from interparticle gaps that transform into pores along grain boundaries during sintering) [1, 2].

Die-compaction presses loose metal powder inside a die cavity between two punches to produce the required green (compacted but unsintered) shape. Wax lubricant is often mixed in with the metal powder to protect the tooling against wear and reduce green density gradients in die-compacted parts. The green parts are sintered below the metal powder melting temperature to allow solid state diffusion between powder particles to occur, thus

creating a solid structure of interconnected metal with some residual porosity (around 10-15 % typically). Die-compacted parts often require minor machining after sintering to add features such as undercuts or thread that cannot be easily shaped by the uniaxial compaction process [1, 2].

The MIM process involves combining metal powders with a binder (typically a thermoplastic polymer mixed with paraffin wax) to create a feedstock, which is heated and injected into a die under high pressures. Injection moulding is followed by debinding (removal of the polymeric binder) and sintering to produce the densified final part. The high-pressure injection moulding process ensures the part has a good surface finish and tight tolerances are achieved [1, 2].

AM technology, also known as three-dimensional (3D) printing, is a process where material is added to create a component layer by layer. LPBF is a specific type of AM technology where thin layers of powder are melted in the 2D cross-sectional shape of the 3D part using a laser or an electron beam. This process is repeated until the part is built up into the required near-net shape. LPBF, unlike die-compaction and MIM, does not require sintering to bond the powder particles and densify the final material.

Metal powders for use in PM are produced using a range of different production processes, such as gas atomisation, crushing and sieving, water atomisation, and plasma atomisation [1, 2]. Powder characteristics are significantly influenced by the powder production process [2, 3]. Table 1 illustrates some of the different powder production processes and the associated powder characteristics.

Table 1. Typical powder characteristics associated with some powder production processes.

Powder production process	Particle shape	Particle size range	Common materials	Cost (1 = Low, 4 = High)
Water atomisation	Irregular	< 500 µm	Fe	2
Gas atomisation	Spherical	< 500 µm	Ti, Al, Ni, Fe, Co	3
Plasma atomisation	Highly spherical	< 200 µm	Ti alloys	4
Crushing & sieving	Angular	45 – 500 µm	Ti alloys	1

To produce parts of consistently high quality, powders require specific characteristics relevant to each specific PM manufacturing technique. These characteristics include particle size distribution (PSD), apparent and tap density, angle of repose (AoR) and flow rate, the definition and measurement of which are specified by internationally standardised test methods [4]. Recent research on powder characterisation has focused on establishing suitable test methods, such as powder spreadability, for evolving PM processes, such as LPBF [5].

Dynamic tap density (DTD) is a test method that is currently used in the pharmaceutical industry for characterising non-metal powders [6]. It has yet to be evaluated for its suitability to metal powders and PM manufacturing. DTD parameters are calculated based on data collected using a powder characterisation method that measures the powder’s transient packing behaviour (settling) while it is being tapped. In this study, a set of five metal powders were characterised using a range of standardised powder characterisation tests, as well as by the DTD test. The powder spreadability is also assessed using a custom build spreadability rig [5]. The results are compared and used to evaluate DTD as a suitable powder characterisation method for identifying critical powder properties appropriate for individual PM manufacturing techniques.

2 Background

PM manufacturing techniques require specific powders with associated critical powder characteristics to ensure consistent high-quality products. For instance, the characteristics that make a metal powder suitable for die-compaction differ from those for a powder that is used for MIM or AM.

Powders that are suitable for die-compaction typically have a narrow PSD with particle sizes $<150\text{ }\mu\text{m}$. This is to ensure that the die cavity is filled uniformly so that the part has sufficient density and high dimensional precision [2]. Typically, irregularly shaped powders work well for die-compaction as the powders have sufficient flowability to flow freely into the die cavity during filling, but also to provide mechanical interlocking and cold-welding of powder particle surfaces during die-compaction. Ductile metals work well for die-compaction as the powder particles undergo plastic deformation due to the application of pressure. This allows the powders to form and maintain the shape of the part after ejection from the die. Powder made from brittle materials fracture and do not provide sufficient green strength for the compacted shape. Metals that are typically used for die-compaction are iron and ductile steel alloys, copper and copper alloys such as bronze [1, 2, 7].

Powders that are suitable for MIM typically have a narrow PSD, with particle sizes ranging from $1 - 40\text{ }\mu\text{m}$ [2]. While powders that are used for die-compaction are preferably irregularly shaped to allow interlocking of powder particles, MIM powders are preferably spherical with a narrower PSD to allow uniform flow, without powder-binder separation, of the fluid (heated) feedstock during the injection moulding process [1, 2]. The effect of the smaller PSD is that MIM parts typically have a better surface finish than die-compacted parts. The powder particles should also have a low oxidation rate and surface moisture. Typical MIM metals include stainless steel, Co-Cr alloys, and titanium alloys [1, 2, 7, 8].

LPBF typically uses spherical powders with a narrow PSD that spread easily and deposit uniform, densely packed powder layers over the part during builds. Particle sizes usually range between $15 - 45\text{ }\mu\text{m}$, similar in range to MIM powders. The powder should be highly spherical with few satellites to decrease interparticle electrostatic forces and thus reduce the risk of powder agglomeration during spreading.

The powder should have high flowability and high packing density to increase the powder layer's spreadability (the ability to homogeneously spread powder over a base plate). Metal powders ideal for AM include stainless steel, aluminium, titanium, copper, brass, gold, silver, and nickel alloys [7, 8].

3 Experimental analysis

Five metal powders were characterised using various test methods including DTD [6], apparent density, flowability and AoR [1], and spreadability [5]. The powders were produced using different processes, resulting in different powder morphology and sizes that make them suitable for different PM manufacturing techniques.

3.1 Materials

The five metal powders used for this study are presented in Table 2 with supplier information related to the powder grade, powder production process and particle size specification. The powder designation provided in the first column is used throughout the paper to refer to each powder. Two commercially pure titanium powders (CP Ti) were evaluated, one produced by plasma atomisation for AM, designated CP Ti (PA) [8] and the other produced from titanium sponge by a hydride-dehydride (crushing and sieving) process [9], designated CP Ti (HDH). The powder production process for the CP Ti (HDH) powder results in angular powder

particles that can be for a range of different PM manufacturing techniques. The Ti-6Al-4V Grade 5 titanium alloy powder is produced by plasma atomisation for AM [8]. The iron (Fe) powder is the ASC100.29[®] grade iron powder produced by water atomisation for die-compaction [10]. The Grade 718 Inconel[®] powder, designated IN718, is produced by plasma atomisation for AM, specifically LPBF [8].

Table 2. Metal powders used in the experimental analysis.

Powder designation	Grade, Supplier	Production process	Particle size specification (μm)
CP Ti (PA)	Grade 1, AP&C	Plasma atomised	15 – 45
CP Ti (HDH)	HDH Ti sponge, Global Titanium	Crushing and sieving (hydride-dehydride)	10 – 75
Ti-6Al-4V	Grade 5, AP&C	Plasma atomised	10 – 45
Fe	ASC100.29, Höganäs AB	Water atomised	20 – 180
IN718	Inconel 718, AP&C	Plasma atomised	15 – 53

3.2 Standard powder characterisation methodologies

The different powder characterisation methods used to evaluate the five different powders are the powder morphology by scanning electron microscopy, the particle size distribution by laser diffraction, apparent density, angle of repose and flowability by international standardised testing methods [4], and spreadability by a customised test method [5]. The details of these tests are described in the following sections.

3.2.1 Morphology

The morphology (shape and size) of the powder was identified by observing the powders with a Zeiss Merlin field emission scanning electron microscope (FE-SEM). Table 3 shows typical FE-SEM settings for observing powders.

Table 3. FE-SEM settings for powder morphology observation.

EHT (kV)	Current (pA)	Detector	Working Distance (mm)	Brightness (%)	Contrast (%)
2 - 3	100	InLens & SE2	± 5 – 6	± 50	± 30

3.2.2 Particle size distribution

The PSD was determined using the laser diffraction or light scattering technique with a Micromeritics Saturn DigiSizer 5200 V1.12 device. To determine the volume frequency of the powder particles, laser diffraction data was analysed according to Mie theory [1]. The respective refractive index used for each powder was 2.22 for the titanium-based powders, CP Ti (PA), CP Ti (HDH) and Ti-6Al-4V, and 2.28 for IN718. Powders were suspended in distilled water with a flow rate of 12 l/min and sonicated for 60 seconds before the measurement. The PSD test was repeated three times for each powder. The volume frequency percent at equivalent spherical particle diameter data are used to determine the particle size

distribution data. The D10, D50 and D90 particle sizes indicate the maximum particle size below which 10 %, 50 % and 90 %, respectively, of cumulative volume percentage of the PSD is contained. The PSD for the Fe powder was not measured using laser diffraction; the sieving analysis results as reported on the powder specification sheet were used to report the PSD for this powder [10].

3.2.3 Apparent density

The apparent density, ρ_A , is defined as the mass of loose (untapped) powder that fills a specified volume. It gives an indication of how densely the loose powder packs into a given volume. Either a Hall or Carney flow meter with a $25 \pm 0.03 \text{ cm}^3$ brass density cup and a A&D FX-1200i precision scale, with an 0.01 g resolution, was used. Separate standard test methods are applied for free-flowing [4: B212], using a Hall funnel, and non-free-flowing [4: B964], using a Carney funnel, powders.

3.2.4 Angle of repose

The standard test method for measuring the angle of repose (AoR) is the fixed funnel method [4: D6393]; however, in this study, calculation of the AoR value does not follow the standard procedure and utilises digital image analysis as this was deemed to be more accurate. The AoR measurement is used to classify the flowability of the powder from excellent or free flowing to very poor flow or non-flowing [4: D6393].

The AoR of the powder heap was determined using ImageJ [11] digital image analysis software. The digital image (for example, as shown in Figure 1a) is cropped and converted to a binarised image by manual adjustment using ImageJ's thresholding tool. A best fit line is applied to the coordinates of the outline of the powder heap using MATLAB®, as shown in Figure 1b. Each line's gradient, equivalent to h/r , is used to calculate the AoR, α , according to Equation (1).

$$AoR(\alpha) = \tan^{-1}\left(\frac{h}{r}\right) \tag{1}$$

The AoR is reported as the average of the two angles, α_1 and α_2 , as measured on opposite sides of the heap. Figure 1b illustrates the construction lines and angles used.

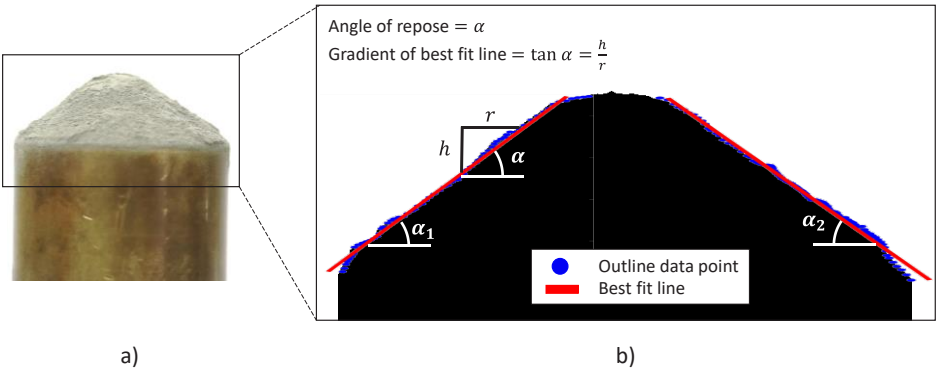


Fig. 1. a) Original digital image of powder heap and b) cropped and binarised image of the powder heap with outline data points and best fit line shown, used to calculate the AoR, α , from the gradients of the best fit lines.

3.2.5 Flow rate

The same equipment that is used to measure apparent density, Hall or Carney flowmeters, are used to measure powder flow rate. The Hall flowmeter has a 2.54 mm diameter orifice and is typically used to measure the flow rate of coarse and free-flowing powders, whereas the Carney flowmeter, with an orifice of 5.08 mm, is used to measure “no-flow” powders [4: B212, B964]. Flow rate is determined by recording the time it takes for 50 or 200 g of powder to flow through the funnel, for the Hall and Carney flowmeters, respectively, and reported in sec/50 g of powder.

3.2.6 Spreadability

Powder spreadability is characterised by two measurements: percentage coverage and spread density [5, 12]. These tests were conducted using a custom-built powder spreadability rig [5], shown in Figure 2, with a 100 × 100 mm² baseplate.

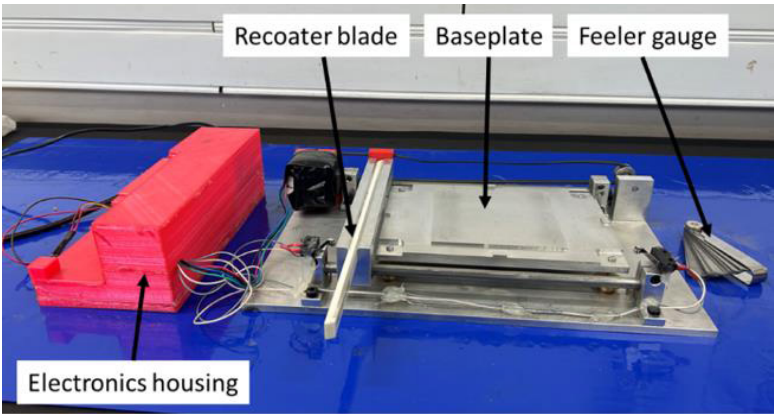


Fig. 2. Custom built spreadability rig.

The percentage coverage is the layer coverage of powder of a set mass, m , spread over a baseplate, where the mass is determined by:

$$m = \rho_A A t \quad (2)$$

In Equation (2), ρ_A is the apparent density of the powder, measured as described in Subsection 3.2.3. A is the area of the baseplate and t is the powder layer height set by positioning the recoater blade above the baseplate using a feeler gauge. The mass of powder was placed in a heap that is spread evenly across the width of the baseplate in front of the recoater blade, as shown in Figure 3a. The recoater blade then spreads the powder over the baseplate at a uniform recoating speed of 150 mm/sec. Thereafter, a digital image of the spread powder was taken using a camera mounted to a tripod (Canon EOS 750D with a 50 mm lens) directly over and perpendicular to the baseplate, as shown in Figure 3b.

As shown in Figure 3c, the area of interest in the image of the spread powder layer was cropped and converted to a binary image. Conversion of the grayscale image into a binary image was performed using the `imbinarize` MATLAB function which uses the widely accepted Otsu’s method for image thresholding [13]. The percentage coverage was calculated from the ratio of the number of white pixels to the total amount of pixels in the area of interest using MATLAB.

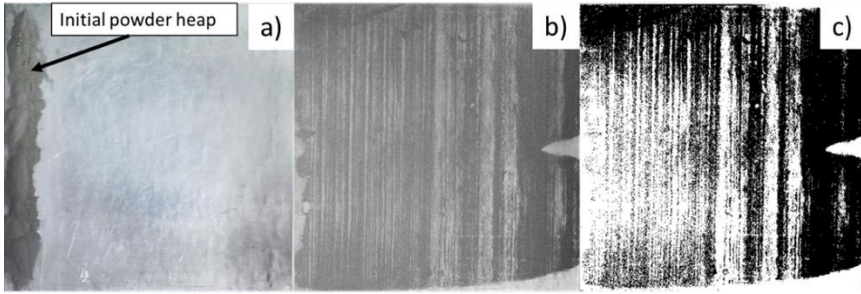


Fig. 3. Overhead digital camera images of powder spreadability test showing a) original powder heap before spreading, b) cropped image of the powder layer after spreading, and c) binarised image for calculation of percentage coverage.

Double the powder mass, m , that was calculated using Equation (2) was placed at the start of the baseplate to determine the spread density. A recoater blade spread powder at a set layer height and speed across the baseplate. Due to using an excess amount of powder, the baseplate should theoretically be fully covered by the powder after spreading has occurred. Four levelling points on the baseplate allow it to be easily removed from the rig. A brush was used to remove the excess powder that fell off the raised baseplate, as shown in Figure 4a.

The powder remaining on the baseplate, shown in Figure 4b, was weighed and then the baseplate mass was subtracted. The spread density, ρ_s , is calculated as:

$$\rho_s = \frac{m_s}{At} \tag{3}$$

where m_s is the mass of powder that remained on the build plate, and A and t are similarly defined as in Equation (2). It should be noted that the calculation of spread density implies that the spread powder mass, m_s , is evenly distributed throughout the expected spread powder volume, as estimated by the spread area and height, A and t . However, as can be seen from Figure 4, the powder does not necessarily spread evenly over the baseplate area. It is critical to consider both percent coverage in combination with spread density in order to gain meaningful insight into the characterisation of powder spreadability.

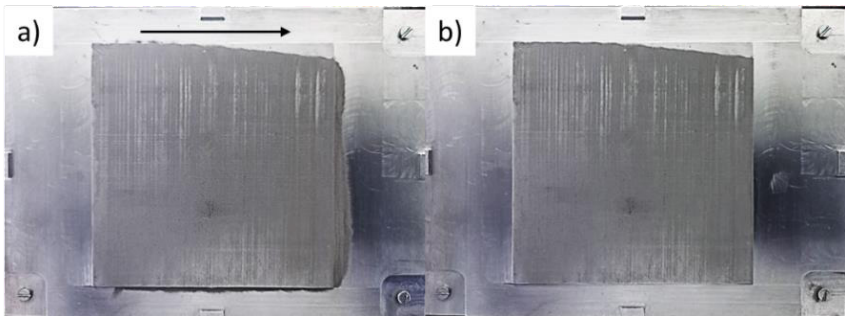


Fig. 4. Spread density analysis with a) excess powder and b) excess powder removed. The arrow indicates the direction of the recoater blade motion.

3.3 Dynamic tap density methodology

3.3.1 Setup and testing procedure

Dynamic tap density (DTD) refers to the packed density of a specified volume of powder after it has settled due to tapping. Figure 5 shows the DTD experimental setup. The apparatus operates by positioning a 100 ml graduated glass cylinder, filled with 100 g of powder, above a rotating cam that lifts and drops the cylinder by approximately 3 ± 0.2 mm per rotation. This test method is based on the standard tap density test [4: B527]. Using a plastic 3D-printed holder and four wingnut screws, the glass cylinder is securely mounted to prevent lateral movement and to clamp it to the rotating tapping cam.

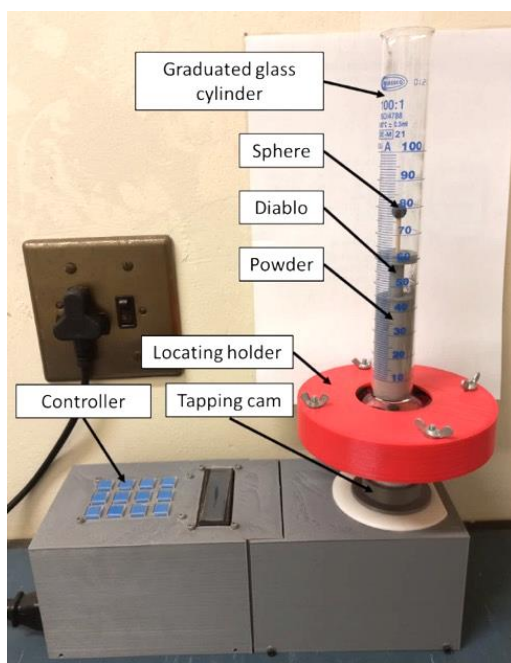


Fig. 5. DTD tester and experimental setup.

A 3D-printed, polymer 6.11 g diablo (weighted mass that fits within a graduated cylinder) rests on top of the powder. A rod with a tracking sphere at its end is attached to the top of the diablo. The diablo ensures that the top level of the powder column remains level throughout the tapping test. To ensure consistency, powder was poured into the cylinder using a 9 mm funnel.

The shape of the tracking device is spherical due to its symmetry when viewed from different angles. An EFS 18 – 55 mm lens on a Canon EOS 750D camera recording 50 fps was used to capture the tapping process by tracking the vertical movement of the tracking sphere. The camera was positioned on a tripod, 70 cm away from the sphere as this is the closest possible position to allow for the use of the optical zoom. The sphere was positioned at the highest cam level and the sphere is positioned at the top of the camera frame at the beginning of the test.

This allows for the entire tracking of the movement of the sphere through the frame during the test, taking into account the settling of the powder during the test (downward movement of the sphere). The DTD controller was used to set up the test for 250 taps per minute, and a stopwatch was used to record the time. Each test ran for 8 minutes as this was the minimum

amount of time required to measure any noticeable settling in the least settled powder (CP Ti (HDH)). After 8 minutes (2000 taps), the powder volume was measured from the cylinder.

3.3.2 Dynamic tap density tracking procedure

In order to record the dynamic motion of the powder column during tapping, motion of the sphere was recorded during the DTD test and analysed. The Moving Picture Expert Group (MP4) file from the Canon camera was converted to an Audio Video Interleaved (AVI) file. ImageJ was subsequently used to crop and convert this file to greyscale. Most of the graduated glass cylinder level indications were removed after the colour balance step, but the sphere shape was not altered. The parallax error was reduced by facing the camera head-on with respect to the graduated measurements of the glass cylinder.

An open-source tracking software plugin, Track Mate version 7.8.0 in ImageJ was used to track the sphere mounted on the diablo. The plugin identifies a spherical object and continuously produces the x and y frame coordinates of the sphere for each frame. Every spot in each frame of an equivalent size was detected based on the approximate sphere diameter in pixels. The linear assignment problem tracker was chosen, due to its ability to link spots from one frame to another.

An Extensible Markup Language (XML) file was created that recorded each x - and y -coordinates of the sphere for each frame. The results were exported to Microsoft Excel and sorted into three columns for frame number, x - and y - coordinates, respectively. To graph the instantaneous powder column volume against number of taps, only the frame number and y -coordinates were imported into MATLAB R2020b.

MATLAB was used to convert the frame number into number of taps, by multiplying the frame number by 0.02 (reciprocal of the frame rate, 50 fps) to get time elapsed, and multiplying time elapsed by the tapping rate (taps per second). The global scale was obtained by multiplying the y -coordinates by a pixel-to-mm conversion factor using the distance between the level indicators on the glass cylinder as a reference. Finally, an offset was added to the y -values to correlate the starting volume with that measured from the cylinder before the start of the test. Figure 6a shows both the upper and lower limits of the sphere (as it is bounced during the tapping process). In Figure 6b, a clear volume curve is displayed by removing the higher peaks as the sphere settles at the lower peaks. The initial volume, V_0 and volume after N^* taps, $V(N^*)$ are indicated on Figure 6b. For this study, $N^* = 2000$ taps, the point after which there is minimal change in volume of the powder column with additional tapping.

3.3.3 Parameters

The DTD test data set is comprised of six parameters that are calculated based on the powder column volume data per tap.

A summarised description of how each of these parameters is calculated and what each one represents in terms of powder compressibility and flowability follows, based on the detailed characterisation methodology provided by Traina *et al* [6].

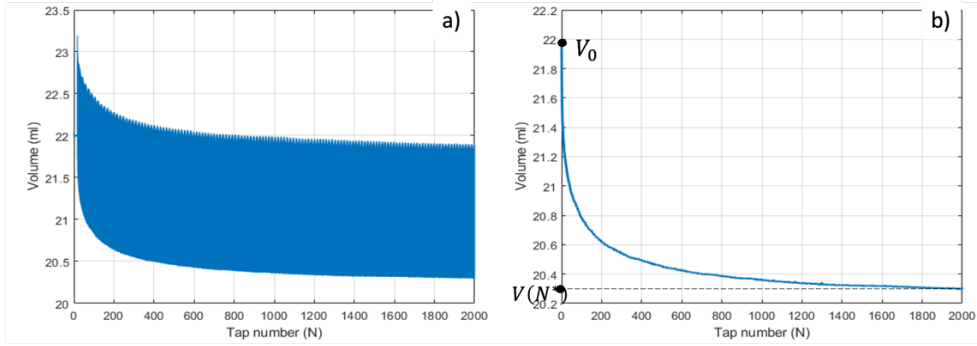


Fig. 6. Example of a DTD volume curve for IN718 powder a) before the upper peaks are removed and b) the processed graph after the peaks are removed showing the initial volume, V_0 , and the volume after the total number of taps, $V(N^*)$.

The **compaction ability** is characterised by the apparent packing fraction, $D(0)$, of the powder column before tapping, defined as the ratio of the apparent or bulk density of the loose powder before any tapping occurs, ρ_B , to the theoretical density of the solid material, ρ_{th} :

$$D(0) = \frac{\rho_B}{\rho_{th}} \quad (4)$$

Theoretically, it is equivalent to the relative apparent density, a term typically used in powder packing research [3]. However, it is important to note that the apparent density is measured according to a standard test method, as described in Subsection 3.2.3, while the apparent packing fraction is measured based on pouring 100 g of powder into the graduated cylinder, as described in Subsection 3.3.1. As such, ρ_B in Equation (4) is calculated from V_0 by multiplying it with the mass of powder used for each test. The compaction ability is inversely related to the flowability of the powder and gives an indication of how densely the powder is packed in the loose state, before tapping and settling [6].

The flow criterion is based on the Hausner ratio, H_R , that is defined as a ratio of tapped to apparent density [1]. According to standard tap density test methods, H_R is calculated after 500 taps [4: B527]. While some research relates Hausner ratio to powder flowability, using this metric for characterising powder flow has been criticised [1, 7]. Theoretically, the H_R increases with an increase in interparticle friction which in turn limits the flow of the powder.

For the DTD test, the **flow criterion** is calculated as the reciprocal of $H_R(N^*)$ and is determined from the DTD curve, an example of which is shown in Figure 6b, as the ratio of the tapped volume, $V_T(N^*)$ to the initial bulk volume V_0 of the powder:

$$1/H_R(N^*) = \frac{V_T(N^*)}{V_0} \quad (5)$$

According to the DTD interpretation of the flow criterion, a higher flow criterion value indicates better powder flowability.

The **granular relaxation index**, $\tau_{1/2}^D(N^*)$, is the ability of the powder system to tend towards its optimal packing state. It is calculated as

$$\tau_{1/2}^D(N^*) = [D(N^*) - D(0)] \times N_{1/2}^D \quad (6)$$

where N^* is a specified number of taps, $D(N^*)$ is the dynamic packing fraction after N^* taps, and $N_{1/2}^D$ is the number of taps required to be halfway between the initial volume, V_B and final volume, $V_T(N^*)$. The lower the granular relaxation index, the higher the ability of the powder to tend towards its optimal packing state.

The **ventilation ability** is characterised by the specific relative volume, $\bar{V}_{sp.rel}(N^*)$, defined as

$$\bar{V}_{sp.rel}(N^*) = \frac{V_T(N^*)}{V_{th}} \quad (7)$$

where V_{th} is the theoretical solid volume of the powder excluding all trapped air or voids as well as internal pores inside powder particles. V_{th} is inversely related to the theoretical density of the solid powder. The ventilation ability of the powder indicates its tendency to trap air in the loose state and is related to the volume fraction of trapped air in the powder that is released by tapping, after N^* taps. It is influenced by interparticle friction. Although Traina et al. [6] originally compares the initial ventilation ability, $\bar{V}_{sp.rel}(0)$, across powders, this tapped version, $\bar{V}_{sp.rel}(N^*)$, provides more insight into the dynamism of the powder. Furthermore, $\bar{V}_{sp.rel}(0)$ is already taken into account as it is the inverse of the compaction ability, $D(0)$.

The **de-aeration ability** is characterised by the variation in additional porosity, $\bar{\epsilon}_{add}(N^*)$, defined as

$$\bar{\epsilon}_{add}(N^*) = \frac{V_0 - V_T(N^*)}{V_0} \quad (8)$$

The de-aeration ability gives an indication of the ability of the powder to reduce its porosity by releasing trapped air during tapping. Releasing trapped air increases the packing density by filling the voids with powder particles, and so reduces the flowability of the powder.

The compaction ability, Hausner ratio, ventilation ability and de-aeration ability are related to the flowability of the powder and are considered to be compressibility indices [6].

The **de-aeration speed**, $v_{1/2}^\epsilon(N^*)$, is the speed at which air is removed from powder during tapping, indicating the powder settling rate, and is calculated as

$$v_{1/2}^\epsilon(N^*) = \frac{\bar{\epsilon}_{add}(N^*)}{N_{1/2}^\epsilon} \quad (9)$$

where the kinetic parameter $N_{1/2}^\epsilon$ is the number of taps it takes to reduce the variation in additional porosity, $\bar{\epsilon}_{add}(N^*)$, by half. Note that $N_{1/2}^\epsilon = N_{1/2}^D$.

Whereas the granular relaxation index gives an indication of the ability of a powder to settle into its optimal packing state, the de-aeration speed gives an indication of how quickly it reaches this state by releasing the additional porosity of the air trapped in the loose powder before tapping.

3.3.4 Radar graph representation

Traina *et al.* [6] recommends plotting DTD parameters in a specified polar order as a radar graph to assist in visually and consistently characterising the flow characteristics of each powder in a powder. Some of the parameters require normalisation so that all the parameters can be plotted on the radar graph between values of 0 to 1. The adjusted parameters are ventilation ability, granular relaxation index and de-aeration speed, and they

are normalised by the highest measured value of each respective parameter over a set of powders. Plotting starts with the compaction ability parameter in the 90° (North) position, followed by plotting the rest of the parameters at 60° intervals in a clockwise direction in the following order: flow criterion (unadjusted), granular relaxation index, ventilation ability, de-aeration ability (unadjusted), de-aeration speed, as illustrated in Figure 8. By following this method, the relative flow and packing behaviour of the powders in the test set can be compared, based on the centroid of the characteristic radar graph polygon for each powder. A powder with a centroid located towards to upper section of the graph (North) indicates a more free-flowing powder. The radar graph polygon is constructed by connecting the radar graph points with straight lines in a clockwise order, and then the centroid of the polygon is mathematically determined.

4 Results

4.1 Standard powder characterisation results

Powder characterisation data for the CP Ti (HDH) [9], CP Ti (PA) [5], and Ti-6Al-4V [5] powders were reported from previous related research, using the methods described in Section 3, unless otherwise stated. As the Fe powder is a standard powder that is produced under consistent quality, the supplier datasheet was used for powder characteristics [10].

4.1.1 Morphology

Figure 7 displays SEM images of the different powders. The SEM image of Fe [14], shown in Figure 7a, shows irregular powder particles that range from around 20 – 150 µm. This is typical for water-atomised powder designed for die-compaction. The CP Ti (HDH) powder, shown in Figure 7b, is angular with particles ranging from 10 – 60 µm, as is typical for crushed and sieved sponge powder. CP Ti (PA), Ti-6Al-4V and IN718 powders are highly spherical, as shown in Figure 7c, d and e, respectively. While the CP Ti (PA) and Ti-6Al-4V powders have similar particle sizes, ranging from around 10 – 40 µm, the IN718 powder has a larger particle size range, from around 10 – 50 µm.

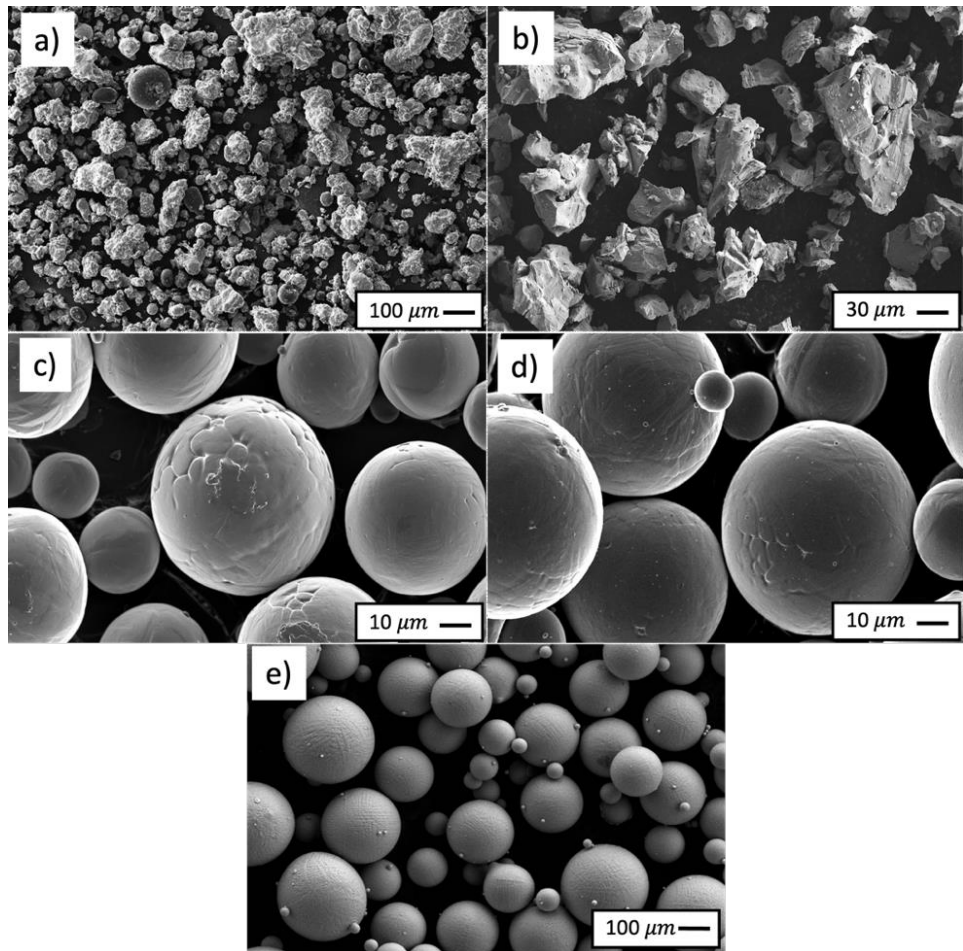


Fig. 7. SEM images showing the powder morphology of powders a) Fe [14, [CC BY 4.0](#)] b) CP Ti (HDH), c) CP Ti (PA) d) Ti-6Al-4V and e) IN718.

4.1.2 Particle size distribution

The PSD is reported as the cumulative volume distribution by equivalent spherical particle diameter. Table 4 presents the D10, D50 and D90 distribution points and Table 5 presents the sieve analysis for the Fe powder [10].

Table 4. The PSD of the powders determined using laser diffraction.

Powder	Cumulative volume particle size (μm)		
	D10	D50	D90
CP Ti (HDH)	11	32	53
CP Ti (PA)	20	33	44
Ti-6Al-4V	19	32	44
IN718	22	36	52

The Fe powder is notable more coarse (larger particle size) than the other powders. While mass fraction is not directly comparable to volume fraction, it is still clear that 7% by mass of the Fe powder is greater than 150 μm while 10% by volume of the other powders is above the 30 – 50 μm range. This implies that 90% by mass of the other powders are less than 50 μm , while only 23% by mass is smaller than 45 μm for the Fe powder. While the D50 particle size for the CP Ti (HDH), CP Ti (PA), Ti-6Al-4V and IN718 powders are comparable, ranging from 32 – 36 μm , the D10 – D90 spread of the PSDs varies for these powders. The CP Ti (HDH) powder has the widest PSD between these powders. The PSD for the CP Ti (PA) and Ti-6Al-4V powders are practically the same, and the narrowest of all the powders. The IN718 powder PSD is similar to these two powders, with a slightly wider PSD.

Table 5. The PSD of the Fe powder determined using sieving analysis [10].

Powder	Particle size (μm)	Mass fraction by percent (%)
Fe	+ 212	0
	+ 150	7
	– 45	23

4.1.3 Apparent density

The apparent densities of the powders are reported in Table 6, along with the theoretical densities of the metals [15] and the relative apparent densities, as calculated using Equation (4). The relative apparent density is the ratio of the apparent to theoretical density of the powder and gives an indication of how densely the loose powder is packed, where a value of unity would indicate a fully solid mass of metal with no trapped air, inter- or intraparticle voids in the powder.

The apparent density of CP Ti (HDH) and Fe powders were measured to compare against published results [9,10]. Typical apparent density for the Fe powder is 2.98 g/cm^3 , indicating that the measured value in this study is consistent with published data [10].

Table 6. The apparent, theoretical and relative apparent densities of the powders.

Powder	Apparent density (g/cm^3)	Theoretical density (g/cm^3)	Relative apparent density
CP Ti (HDH)	1.10 $\pm$ 0.01	4.51	0.24
CP Ti (PA)	2.56 $\pm$ 0.01	4.51	0.57
Ti-6Al-4V	2.44 $\pm$ 0.01	4.43	0.55
Fe	3.05 $\pm$ 0.02	7.87	0.39
IN718	4.80 $\pm$ 0.05	8.20	0.59

4.1.4 Angle of repose

The AoR measurements and the corresponding flow classification of the spherical powders [4: D6393] are reported in Table 7.

Table 7. The angle of repose and flow classification of the powders.

Powder	AoR (°)	Flow classification
CP Ti (HDH)	44.71 ± 1.48	Poor flow
CP Ti (PA)	28.20 ± 0.10	Free flowing
Ti-6Al-4V	39.20 ± 1.20	Fair
Fe	36.82 ± 0.55	Fair
IN718	31.10 ± 2.28	Free flowing

4.1.5 Flow rate

The flow rates of the powders are reported in Table 8. All powders flowed freely using the Hall flowmeter except for the CP Ti (HDH). However, it should be noted that the Ti-6Al-4V had to be measured immediately after being dried otherwise it would not flow using the Hall flowmeter. The CP Ti (HDH) did not flow, the standard [4: B213], indicates that if a powder does not flow using the Hall flowmeter, the Carney flowmeter should be used [4: B964]. The CP Ti (HDH) still did not flow using the Carney flowmeter. Data for the CP Ti (PA) powders was reported from previous research [5]. The published flow rate for the Fe powder is 25 s/50g, indicating that these results may be slower than typical, due to storage conditions [10].

Table 8. The flow rate of the powders.

Powder	Flow rate (s/50 g)
CP Ti (HDH)	Does not flow
CP Ti (PA)	31.8 ± 0.4
Ti-6Al-4V	57.2 ± 0.6
Fe	31.9 ± 0.1
IN718	14.7 ± 0.2

4.2 Powder spreadability

The powder spreadability results for percentage coverage and powder bed density are presented in Table 9 and Table 10, respectively. The spreadability was measured at different layer heights of 60, 80, 90,100 and 150 µm. Powder spreadability is a measure that is specifically applicable to powders suitable for LPBF. The Fe and CP Ti (HDH) powders are not designed for LPBF, as is apparent from their non-spherical particle shape, and as such their spreadability was not evaluated.

Table 9. Spreadability results measured in terms of percentage coverage.

Powders	Percentage coverage (%)				
Layer height (µm)	60	80	90	100	150
CP Ti (PA)	68.0 ± 1.1	83.0± 4.2	79.6 ± 3.2	75.9 ± 2.7	86.1 ± 3.8

Ti-6Al-4V	22.0 ± 1.5	23.0 ± 1.8	41.3 ± 2.6	57.5 ± 7.2	41.0 ± 1.6
IN718	58.5 ± 2.6	58.6 ± 2.2	58.9 ± 3.1	55.8 ± 5.9	53.6 ± 7.7

Table 10. Spreadability results measured in terms of powder bed density.

Powders	Powder bed density (g/cm³)				
Layer height (µm)	60	80	90	100	150
CP Ti (PA)	1.0 ± 0.7	1.5 ± 0.1	1.9 ± 0.1	2.1 ± 0.2	1.7 ± 0.1
Ti-6Al-4V	1.0 ± 0.2	1.3 ± 0.2	1.3 ± 0.2	1.4 ± 0.1	1.0 ± 0.2
IN718	3.4 ± 0.1	3.7 ± 0.1	3.6 ± 0.4	3.7 ± 0.1	2.2 ± 0.2

While the powder bed density is reported in g/cm³, it should be noted that these three powders all have different theoretical densities. In order to get an indication of the packing fraction for each powder, these values should be normalised by the theoretical density for each alloy to give relative density values as indicated in Table 11.

Table 11. Powder bed density normalised by each alloy’s theoretical density.

Powders	Normalised powder bed density (%)				
Layer height (µm)	60	80	90	100	150
CP Ti (PA)	22 ± 0.2	33 ± 0.0	42 ± 0.0	47 ± 0.0	38 ± 0.0
Ti-6Al-4V	22 ± 0.0	29 ± 0.0	29 ± 0.0	32 ± 0.0	22 ± 0.0
IN718	42 ± 0.0	45 ± 0.0	44 ± 0.0	45 ± 0.0	26 ± 0.0

The normalised powder bed density for the Ti-6Al-4V powder ranges from 22 % to 32 %, for the CP Ti (PA) powder from 22 % to 47 %, and for the IN718 powder from 27 % to 46 %. These are typical values, lower than the relative apparent density of each powder, see Table 8. This is an expected result, because the spread powder packs less dense then the loosely flown powder. There does not seem to be a consistent relationship between percentage coverage and powder bed density. All the powders appear to have the highest powder bed density at a 100 µm layer height. The spreadability results indicate that more research into this characterisation method is required.

4.3 Dynamic tap density results

The average DTD parameters for each powder, as measured for this set of powders, are presented in Table 12. The maximum value of the parameters that were normalised, in order to present them as varying between 0 and 1, are indicated by the † in Table 12. The DTD parameters for the powders evaluated in this study are presented as a radar graph for each metal powder, as shown in Figure 8. The centroid of each polygon within the radar graph is indicated by a “×” symbol.

Table 12. DTD parameter values for powder set. Parameters that are normalised for the radar graphs are indicated by †, with the maximum value that was used for normalisation indicated by †.

DTD parameter	Powder				
	CP Ti (HDH)	CP Ti (PA)	Ti-6Al-4V	Fe	IN718
Compaction ability, $D(0)$	0.24	0.57	0.57	0.39	0.56
Flow criterion, $1/H_R(N^*)$	0.76	0.92	0.90	0.85	0.92
†Granular relaxation index, $\tau_{1/2}^D(N^*)$	3.61†	2.11	0.89	2.53	1.24
†Ventilation ability, $\bar{V}_{sp,rel}(N^*)$	2.68†	1.61	1.60	2.08	1.64
De-aeration ability, $\bar{\epsilon}_{add}(N^*)$	0.24	0.09	0.10	0.15	0.08
†De-aeration speed, $v_{1/2}^\epsilon(N^*)$	0.010	0.0047	0.0098	0.010†	0.0036

5 Discussion

The DTD radar graph for the three spherical powders CP Ti (PA), Ti-6Al-4V and IN718, shown in Figure 8c, all have similar centroids, located in the upper half of the radar graph. The CP Ti (HDH) and Fe powders, radar graphs shown in Figure 8a and b, have radar graph centroids that are in the lower half of the radar graph, with the CP Ti (HDH) centroid located the lowest of all the powders. In comparison to the other powders, CP Ti (HDH) has low apparent packing fraction and flow criterion, and a high ventilation ability, granular relaxation index and de-aeration ability. These parameters combined to lower the centroid of the radar graph polygon for CP Ti (HDH) and implies that CP Ti (HDH), according to the DTD data, it is the least flowable, followed by the Fe powder.

Three spherical powders have very similar flow criterion, compaction ability, ventilation ability and de-aeration ability. The difference between these powders is captured in the DTD parameters: granular relaxation index and the de-aeration speed. Both of these parameters link to the tendency for these powders to rearrange themselves into an optimal packing state. The DTD polygon centroid location for the Ti-6Al-4V powder is higher than for the CP Ti (PA) and IN718, strongly influenced by the high de-aeration speed for the Ti-6Al-4V powder. According to DTD polygon centroid interpretation guidelines, Ti-6Al-4V has the best flowability and packing behaviour of all the powders.

The DTD results in general correlate with the standard powder characteristics of apparent density, AoR and flow rate results, presented in Tables 8, 9 and 10, respectively. The relative apparent density for each powder correlates to the $D(0)$ value for each powder, reported in Table 13. This indicates that the initial packing fraction for the DTD test, calculated using the V_0 reading from the DTD curve, is equivalent to the relative apparent density. The three spherical powders, CP Ti (PA), Ti-6Al-4V and IN718, have the highest packing fraction, all close to 0.6 of full density, while larger, non-spherical powders, CP Ti (HDH) and Fe, pack less densely, trapping more air between their particles.

This result confirms the hypothesis that loose spherical powders with a relatively narrow PSD approach the theoretical limit for random packing of spheres, 64% [2, 3].

The AoR results indicate that the three spherical powders, CP Ti (PA), Ti-6Al-4V and IN718, are free-flowing, which correlates with the position of their centroids on the DTD radar graphs.

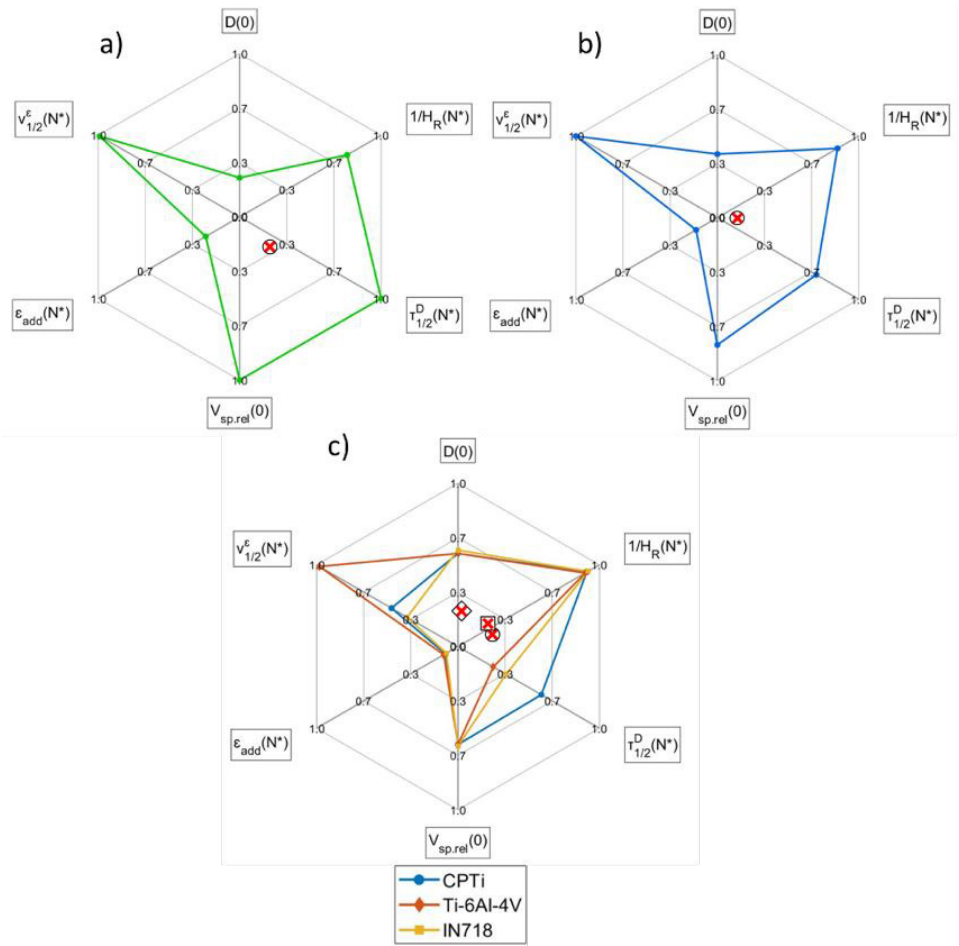


Fig. 8. Radar graph of dynamic tap density parameters of a) CP Ti (HDH), b) Fe and c) the spherical powders, CP Ti (PA), Ti-6Al-4V and IN718, indicated by the legend, respectively. The centroid of the radar graph polygon is indicated by an “x” symbol in each case.

The Ti-6Al-4V powder has the highest AoR and is classified as having passable flow properties. It also has the slowest flow rate of 57.2 s/50 g. The CP Ti (PA) powder has a faster flow rate than Ti-6Al-4V. Based on the AoR and flow rate results, the IN718 has the best flowability of all the powders, followed by the CP Ti (PA) and then Ti-6Al-4V. The highly cohesive nature of Ti-6Al-4V is reflected in the flow rate and AoR results, respectively. This could be related to the fact that Ti-6Al-4V is more sensitive to moisture uptake than CP Ti and Inconel and moisturised powder is not recommended for testing using the Hall flowmeter [16].

The spreadability results show that, for the three spherical powders, the percentage coverage and powder bed density is better for the CP Ti (PA) followed by IN718 and then Ti-6Al-4V.

Comparing the DTD parameters for these three powders shows that they have very similar compaction ability, flow criterion, ventilation ability and de-aeration ability. Differences between the powders are evident in the variation in granular relaxation index and de-aeration speed. The low granular relaxation index and high de-aeration speed of the CP Ti (PA) both

indicate that this powder settles into its optimal packing state more quickly than the Ti-6Al-4V or IN718 powders. This translates into the better spreadability result achieved for the CP Ti (PA).

The implication is that powders that are suitable for AM and LPBF, that have a narrow particle size distribution, are spherical and of similar size, may have similar apparent and tapped density, but it is the rate at which the powder particles are able to arrange themselves into an optimal packing state that influences their spreadability behaviour. This behaviour is not sufficiently captured by AoR or flow rate measurements.

Lastly, it is interesting to note that while the de-aeration speed of the angular CP Ti (HDH), irregularly-shaped Fe and spherical CP Ti (PA) powders is very similar, flow rate measurements for these powders are only similar for the Fe and CP Ti (PA) powders. The flow rate measurement for the CP Ti (HDH) powder indicates that it does not flow.

For this study, it is clear that no one powder characteristic or test is sufficient to fully describe the packing and flow behaviour of a powder. Powder characterisation tests should be used to create a broad picture of the flow behaviour and should be used to compare powders within a dataset. The DTD test polygon centroid gives a reasonable indication of powder flow and spreadability for LPBF, when used to compare similar powders.

6 Conclusion

This research is aimed at determining whether DTD testing is an effective characterisation method for metal powders, producing comparable results to standard powder characterisation methods. The results indicate that DTD testing should be used in conjunction with standard characterisation techniques in order to provide insight into the powder flow and packing behaviour, and that it is most effectively used as a comparative measure between similar powders. However, DTD has the potential to replace standard characterisation techniques for metal powders, especially for characterisation spreadability for LPBF. In particular, the DTD test measures the rate at which a powder rearranges itself into an optimal packing structure, which is important during the powder layer spreading process of LPBF. Future studies could focus on quantitative correlations between standard powder characteristics and DTD parameters to further enhance the reliability of DTD testing as a comprehensive powder characterisation technique.

This work is based on the research supported in part by the National Research Foundation of South Africa (Grant Number: 138432) and partial the DSI Collaborative Program in Additive Manufacturing (CPAM).

References

1. American Society of Metals (ASM) International, ASM Handbook Vol 7: Powder Metallurgy (2015)
2. R.M. German, Powder metallurgy and particulate materials processing (Metal Powder Industries Federation, 2005)
3. R.M. German, Particle packing characteristics (Metal Powder Industries Federation, 1989)
4. American Society for Testing and Materials (ASTM), ASTM Vol **02.05**: Metallic and Inorganic Coatings; Metal Powders and Metal Powder Products, Annual Book of ASTM Standards (2023)

5. B.S. Parker, Blending of powders for in-situ alloying of Ti-6Al-4V laser powder bed fusion, MEng Thesis, Stellenbosch University (2021)
6. K. Traina, R. Cloots, S. Bontempi, G. Lumay, N. Vanewalle, F. Boschini, Powder Technol. **235**, 842-852 (2013)
7. P. Muthuswamy, Lasers Manuf. Mater. Process **9**, 312–337 (2022)
8. AP&C [Online]. Available: <https://www.advancedpowders.com/powders>
9. H.L. Bosman, Influence of powder particle size distribution on processing of uniaxially compacted titanium and Ti-6Al-4V preforms, MEng Thesis, Stellenbosch University (2016)
10. Höganäs [Online]. Available: <https://www.hoganas.com/en/powder-technologies/pressing-sintering/products/iron-powders/>
11. C. Schneider, W. Rasband, K. Eliceiri, Nat. Methods **9**, 671–675 (2012)
12. L. Capozzi, A. Sivo, E. Bassini, J. Mater. Process. Technol. **308** (2022)
13. N. Otsu, IEEE Trans. Syst. Man Cybern. Syst. **9**, 62–66 (1979)
14. J. Wendel, S.K. Manchili, E. Hryha, L. Nyborg, Thermochim. Acta **692** (2020)
15. Material Properties. [Online]. Available: <https://material-properties.org>
16. L. Cordova, T. Bor, M. de Smit, M. Campos, T. Tinga, Measuring the spreadability of pre-treated and moisturised powders for laser powder bed fusion, University of Twente Drienerlolaan (2020)