Essential characterization of metal powder for additive manufacturing

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Abstract. The aerospace industry is mostly focused on complex, low production volume as the components are often known with complicated internal geometries and dimensions. Heavy market competitions and restrictions in high manufacturing cost could be overcome with additive manufacturing (AM). AM has received notable attention in its ability to produce customized metal parts and exhibiting top-notch capabilities that would lead the AM industry in the future. Nonetheless, in producing trusted, high-quality products, characteristics of metal powders should be accounted so it would meet proper quality such as powder morphology, particle size distribution, porosity, and powder’s ability to flow. SEM, OM, laser diffraction and rheometer are suggested in studying the powder’s characteristics. All characterization methods found are used practically. These characteristics are important to assess AM quality. Comparison in different aerospace materials is studied to get the actual material combination in getting an optimum metallurgical bond between the substrate and molten pool of the powders. Therefore, characterization methods of 3D metal additive and characteristics of aerospace materials are evaluated and presented systematically.

Keywords: Additive manufacturing, 3D metal printing, characterizations, metal powders

1. Introduction

Additive manufacturing (AM) successes have received significant media attention in the media press in the last couple of years, where it involved in joining metal powders layer upon layer with no or minimal pre-processing and post-processing requirements [1-3] This approach can give industry new design flexibility, reduce energy use and shorten time to market when compared with conventional machining methods which are time-consuming and uneconomical [4-6]. One of the assuring AM technologies is direct laser metal deposition (DLMD) where it derived from micro-welding and laser technologies to generate molten pool and solidified after to form a complex structure. DLMD also has the ability to
repair existing worn-out parts that have not been repairable in the past, including both geometrical and structural restoration [7-13].

There are two main parameters for metal AM which are a type of input raw material and energy source used to form the desired part [14]. Input raw materials, as shown in Figure 1, play a significant role in producing a high-quality printed aerospace component and repairing existing component. For example, Ti-6Al-4V is commonly used as fans, compressor disc/blade and load-carrying components due to its strength and thermal stability at high-temperature environment [9] meanwhile SS316 has resistance towards corrosive environment [6]. It has been found that SS316 produced by AM have better yield strength (649.3 MPa) and hardness (235 HV) [16, 17]. Inconel 718 usually used in engine part due to excellent wear resistance and high tensile-creep rupture properties at temperature of 1300 ºF [18, 19]. However, these materials are hard to machine due to high work hardening rates make AM is favorable over machining process [20].

In general, DLMD in AM requires metal powder diameters in the range of 50 μm to 150 μm [21, 22]. Theoretically, the smaller and more spherical particle sizes are preferable because it is easy to infuse fine powder into a narrow stream. In ensuring the repeatable output of metal parts, these characteristics give a balance of good flowability, fair powder consumption efficiency, and smooth surface finish [23-27]. It is worth noting that the optimal particle size may also differ depending on the properties of the alloy. For example, light alloys such Al-Si -10Mg flow at best in a coarser shape, whereas dense alloys like Ti-6Al-4V work better in a more refined form [1,28]. However, the wide variety of powder manufacturers on the market has hampered the establishment of universal standards for powder properties [29]. Thus, determining the powder characteristic for metal-additive manufacturing is essential for industry to produce consistent parts with predictable properties. Otherwise, it may resulting in high degree of variation in process outcomes. Hence, characterizing raw metal powders for better output will be discussed.

2. Powder Characterization/Sampling
Consistent powder characteristics are crucial for repeatable manufacture so that printed component has desired mechanical properties. Thus, a deeper understanding and knowledge about powder characteristics and its impact on AM is crucial which affect the development, or quality of the printed parts [30]. In addition, powder sampling e.g., scoop sampling, chute splitting or spin rifling also must be done carefully to ensure the accuracy of the results. Furthermore, the effect of powder properties on
the performance and processing of the final component could be done by through the characterization of the mechanical, chemical and physical properties of a powder [31, 32].

2.1 Particle Density/Bulk Properties
Particle density is an essential powder characterization to estimate the built-in porosity or related to a powder’s ability to pack which concerning die packing and spreading mechanism during the deposition process. This property is measured based on ideal gas law derived from primitive density equation (mass/volume). In this method, the metal powder is placed in a pycnometer under controlled temperature (15-30 °C), and atmospheric pressure (0 – 0.7 MPa), type of gas (helium) [33, 34]. There are many ways to measure powder density; in particular, these methods include measurement of apparent density, tapped density, actual density, and powder-bed density. The apparent density simulates the loss of powder packing state following the ASTM B212-09 and ASTM B707-10 standards. It shows the correlation of a powders mass to freely fill a hollow space [35]. The tapped density indicates that the stress-packing state of self-powder is determined using B527 [35]. The actual density represents the bulk density of a powder using pycnometer as in ASTM B923 [35].

Thejane et al. [36, 37] indicated that the density of Ti-6Al-4V measured using pycnometer (4.0-4.42 g/m3) was close to the theoretical value (4.43 g/m3). Cordova et al. [21] found Inconel 718 has highest density (8.26 g/cm3), followed by Ti-6Al-4V (4.38 g/cm3), Scalmalloy (2.68 g/cm3) and Al-Si-10Mg (2.65 g/cm3), respectively [21]. The apparent and taped density of Inconel 718 virgin powder is ten times higher than reused IN718 powder but vice versa for true density [35].

2.2 Particle Size Distribution (PSD)
It is important to accurately characterize particle size distribution (PSD) in delivering high quality of AM product defined by \( (D_{v90}-D_{v10})/D_{v50} \), where \( D_v \) is diameter of volume-equivalent sphere and \( V_p \) is the volume of particles. Through PSD, the powders ability to flow, spread and packing during deposition process can be illustrated using histogram. Using smaller particle size is likely induce coagulation inside nozzle subsequently affect the mass flow. There are several methods used to determine PSD of the metal powders such as dry sieving (DS) and laser diffraction (LD). For DS, ASTM B214-07 or ASTM E161-00 is needed. The standard suggests sieve need to be arranged in consecutive order between 5 µm to 1 mm and agitated for 15 minutes. Compared to DS, LD provides real-time monitoring to facilitate the distribution of particle size during production and, at the same time, provides immediate feedback to improve the production process [37]. In LD, at wider angles, small particles scatter weakerly while large particles scatter light strongly at smaller angles [37].

However, there are some limitations of this method in which it assumed that all powders are in spherical structure, although irregular structures are presented [31,38] which give "ghosts" diffractions interpretation [39]. It has been found that defect formation can be reduced when the PSD of metal powder is within the range of 20µm to 50µm [22]. Author [54] in their finding, indicated that Inconel 625 with 60µm - 80µm provide excellent mechanical strength [35].

2.3 Particle Morphology/Surface Roughness
The morphology of metal powders and uniformity (e.g., spherical, angular, dendritic, dendritic, dish-shaped, circular, or quasi-quantitatively as established in ASTM B243-11) are crucial elements in determining powder flowability in AM [40,41] besides packing density [21]. It can be estimated using \( S_S/S_P \) in which \( S_P \) is the surface area of a given volume while \( S_S \) is equivalent surface area. However, these shapes are highly dependent on the type of powder processing method [42]. For example, the atomization process yields a smooth and spherical shape but exhibit leaf-like and irregular structure under precipitation or comminution [42]. Ideally, spherical particles provide better densification than non-spherical particles [43, 56]. There are several methods to determine the size and morphology of
particle properties as such adsorption, sieve, laser, and microscopy analyses [44]. Barret [51] offers a comprehensive review in expressing dimensional parameters for metal powders as depicted in Table 1.

Table 1: Dimensional parameters to characterize particle morphology [51]

| No | Formula | Description                        | Range     |
|----|---------|------------------------------------|-----------|
| 1  | $\frac{L + i}{2S}$ | Flatness index                      | 1 to $\infty$ |
| 2  | $\frac{i \cdot S}{L \cdot i}$ | Ordinate and Abscissa for plot to evaluate shape | 0 to 1 |
| 3  | $\frac{S}{L \cdot 100}$ | Elongation                          | 0 to 100  |
| 4  | $\frac{L}{S \cdot 100}$ | Flatness                            | 0 to 100  |
| 5  | $\frac{L}{S}$              | Flatness                            | 0 to 1    |

$L = \text{long axis}, S = \text{short axis}, i = \text{intermediate axis}$

Undeniably, different microscopy methods have been extensively used among researchers to study surface morphology. As much useful as the method is, there is a setback too. The number of particles analysed in each image is limited to 100 particles or lesser [44]. Chen et al. [45] investigated the characteristics of 15Cr-13Mo-Y using the scanning electron microscope (SEM) and metallographic microscope. Cleary and Sawley investigated powders’ aspect ratios discharge from hoppers have affected the mass flow rates [46]. The flow rate decreases when the powder shape changes from equiaxed to elongated [46].

2.4 Chemical Composition

In practice, raw metal powders used in AM are not 100% pure and might contain other unwanted materials. Thus, to ensure fidelity of powders require (i) micro-analysis methods such as energy dispersive spectroscopy (EDS), (ii) surface-analysis methods e.g., atomic emission spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), secondary ion mass spectrometry (SIMS), and (iii) bulk-analysis methods e.g., X-ray diffraction (XRD) and X-ray fluorescence (XRF) by identifying the chemical composition of metal powders. The presence of unwanted phases or secondary phases significantly affects the diffusion process during micro-welding, affecting the printed part's mechanical properties [42]. For example, a small amount of oxygen and nitrogen gases has negatively influenced AM product's strength and ductility [42].

2.5 Porosity

The porosity of the powders either in interconnected or particles’ surface is unwanted characteristic that need to be avoided. It has been established that powders' porosity might be transported to the melt pool hence lowering the mechanical properties of deposited structures or printed parts [47]. This characteristic is highly dependent on type of powder processing. For example, although atomization process able to produce uniform-spherical particles however it induced higher porosity than precipitation method due to entrapped gas/air inside the particles. The most appropriate approach in visualizing the metallic powder's porosity is using X-ray computed tomography (XCT) [48, 49]. However, metallography techniques (viewed under optical microscope) can still be used [36]. Author [35] used the optical microscopy (OM) technique to observe the powders' internal porosity, defects, and cross-sections [26].
2.6 Flowability/Rheology
The metal powder's flowability significantly affects the smoothness of the powder deposition and equable powder feeding in AM systems [39] in which ultimately affect the production rate and packing ability. This property is very much affected by particle size and distribution. It has been portrayed that powder with less than 10μm diameter has poor flowability than large particles due to higher surface fiction or cohesive force between the particles [39]. In addition, smaller particle size may also result in agglomeration. Among standardized methods to measure metal powder's flowability or rheological behavior (e.g., dynamic flow properties) is using FTF Freeman rheometer and Hall & Gustavson flowmeter based on ASTM B213-11 and ASTM B964-09, respectively. However, in term of sensitivity and accuracy, rheometer provides a finer evaluation of powder rheological behavior than flow meter [50].

2.7 Thermal Analysis / Conductivity
Thermal properties of metal powders are also extremely important to AM. It measured the ability of metal powders to absorb and transfer heat (thermophysical property) during deposition process [52]. Usually, differential scanning calorimetry (DSC) and laser flash experiment are utilized to determine the thermal characteristic (thermal conductivity, K) and calculating the kinetic parameters of metal powders. Other methods as such photoacoustic, crenel heating excitation and photopyroelectric techniques are also employed [53-56]. This property, K can be mathematically represented by $\frac{Z^2 p C_p}{a T_{max}}$ (where z is distance from powder surface to thermocouple junction, $\rho$ is bulk density and $C_p$ is specific heat capacity at time at maximum voltage ($T_{max}$) will elucidate the heat-dissipation effect during consolidation process [53]. However, this equation is valid when $\frac{Z}{a} > 3.87$ [54]. However, thermal conductivity for mixed powder with different particles size are varies.

3. Conclusion
To ensure the integrity of the printed products in AM, feedstock or metal powders' characteristic needs to be understood. Density, particle size distribution, morphology, chemical composition, porosity, and flowability are essential characteristics in ensuring the process's workability. Density will determine the spreading and packing density during deposition meanwhile particle size distribution determined the flow rate of the process. Smaller particle size induced coagulation. Other properties as such porosity need to be minimized since it affects strength of the printed part. Flowability is highly dependent on the particle morphology and density of metal powders. Although it may seems the characterization of metal powders are readily applicable however, standardizing methods used might be more challenging.

Acknowledgment
The authors would like to express their gratitude to the Universiti Teknologi Malaysia (UTM) for financial support under UTMICONIC research grant (Vot no. 09G59). The framework of the work was established by Mazarina MR to meet study objective related with characterization of metallic powders based on the state of the art and industrial partners from GKN Aerospace needs. The authors declare not conflict of interest.

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