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Rheological properties of two stainless steel 316L powders for additive manufacturing

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Abstract. This study measures the rheological properties of two stainless steel 316L powders which are used for the powder-bed-fusion based additive manufacturing process. The purpose is to evaluate the newly acquired powder in comparison with the used and recycled powder, so that both powders can be mixed with each other to supplement the powder usage. The powder rheology properties, such as dynamic property, bulk property, and shear property, are tested and compared. The results and analysis confirm the compatibility of powder mixing.

1. Introduction

The Additive Manufacturing (AM) is a process of joining materials to make objects from 3D CAD model, usually layer upon layer [1]. This technology has been widely used for metal part production. The commonly used metal AM process consists of selective laser melting (SLM), laser cusing, laser engineered net shaping (LENS), electron beam melting (EBM), etc. The pre-alloy powders, such as stainless steel, cobalt chromium, titanium alloy, etc., can be directly used by these metal AM processes to produce functional parts, in contrast to the conventional molding or powder metallurgy process. In particular, the SLM and EBM processes, which are two popular powder-bed-fusion (PBF) based AM processes, have been extensively employed for the metal part fabrication. The characteristics of powder for PBF are the key for the process performance and part quality [2]. Many studies have pointed out the importance of understanding the powder effects on the PBF process, with the purpose of ensuring the production stability using AM. Otherwise, a profound understanding to the powder will help identify the powder quality so that the users can determine whether or not to keep using or discard the recycled powder, in order to maximize the material usage.

Some standardized testing methods have been applied to the PBF powders, such as the measurements of tap density, particle size distribution (PSD), flow through an orifice, powder morphology, and angle of repose (AOR). The testing results provide some insights about powder performance in the PBF process in correlation with the powder characteristics. Gu et al. [3] measured the AOR and PSD of 3 types of Ti-6Al-4V powders from different suppliers and concluded that the flowability is significantly influenced by the fine particle inclusions. Also, the thermal conductivity of powder bed is higher because of the fine particles. Tang et al. [4] studied the PSD, apparent density, tap density, flowability, and particle morphology of reused Ti-6Al-4V powder of EBM process. It is found that the reused powder exhibits improved flowability and modified PSD and particle morphology. Spierings et al. [5, 6] tested the properties of parts produced using stainless steel 316L powders with three different PSD and pointed out PSD should be optimized for better results.



Slotwinski et al. [7] characterized the size and shape of virgin powder and recycled powder, using X-ray computed tomography, optical microscopy, and scanning electron microscopy. The particle size and shape have a critical impact on forming agglomerates and irregular porous surface of metal AM parts [8]. Spierings et al. [9] also reviewed different powder flow measurement techniques for PBF based AM processes. However, researchers also realized that it is challenging to interconnect the properties of the individual powder particle with bulk powder behavior, in-process performance and finally part property because some powder particle properties can have contrary effects on the next-level properties [2].

Recently, powder rheometer has been of interest to be employed for the powder characterization. Strondl et al. [10] used a powder rheometer to determine various powder flow and powder bed properties. The study concluded that powder rheology is a useful method to determine differences in flow properties of powders used in AM. Lyckfeldt et al. [11] also demonstrated the versatility of powder rheometer on characterizing the properties of metal powders and correlated the powder cohesion, compressibility, aeration effect, and the yield stress at shear. Clayton et al. [12] use some case studies to illustrate how powder rheology supports optimization and lifecycle management of powders for AM. These studies exhibit promising potentials of utilizing powder rheology measurement to characterize metal powders for PBF process. Hence, in this study, the rheological properties of two stainless steel 316L powders are measured and compared. One powder is used and recycled for a couple of times. The other powder (virgin powder) is newly acquired for supplement the deficiency of the first powder. In order to identify the difference and achieve a repeatable performance of powders for metal AM, rheological parameters of both powders, such as stability index, specific energy, aeration ratio, etc., are measured and discussed.

2. Materials and Experiment

Two stainless steel powders were measured for this study. The used and recycled powder FS 316L came with the Farsoon FS271M SLM additive manufacturing machine which was procured from Farsoon High Tech Co. Ltd. The virgin powder MetcoAddTM 316L-A was ordered from Oerlikon AM, which is a metal powder supplier for AM. The chemical compositions and PSD of the two powders were provided by the powder supplier, as shown in Table 1 and 2. Both powders were produced by the gas atomized method and have a spherical morphology, with the apparent density larger than 4 g/cm³.

Table 1. Chemical composition of stainless steel 316L powders.

| | Weight Percent (wt. %) | | | | | | |
|---------------|------------------------|------|------|-----|-------|-------|-------|
| | Fe | Cr | Ni | Mo | Mn | C | Other |
| FS 316L | Balance | 18.0 | 12.0 | 2.0 | < 1.0 | <0.03 | <1.0 |
| MetcoAdd 316L | Balance | 17.4 | 11.2 | 2.6 | 1.5 | <0.02 | <1.0 |

Table 2. Particle size distribution of stainless steel 316L powders.

| | D10* | D50 | D90 |
|---------------|------|-----|-----|
| FS 316L | 22 | 34 | 52 |
| MetcoAdd 316L | 19 | 30 | 46 |

*Units: μm

The powder rheology measurements, i.e. dynamic property, bulk property, and shear property were conducted using a *Freeman* FT4 Powder Rheometer. As for the dynamic property, the rheometer uses a precision blade (impeller) to move downwards, with a certain revolving speed, through the powder to establish a precise flow pattern. The powder particles start to interact, or flow relative to one another. The resistance experienced by the blade represents the difficulty of this relative particle movement, or the bulk flow properties. The vertical force on the base and the torque on the blade are measured. The bulk property is determined by applying a normal force to the powder bed using a vented piston which allows entrained air to escape. Shear property provides an indication of how

easily a powder will move from a static condition into dynamic flow. The test applies a known normal stress to the powder, followed by a rotational torque to determine the point of failure.

3. Results and Discussion

The chemical composition of both powders shows a similar elemental inclusion, which conforms to the standardization of the stainless steel 316L alloy. The PSD indicates that the average size of FS 316L powder is a little larger than the MetcoAdd 316L powder. The powder with a small particle size might present a decreased flowability and a higher specific surface area, causing agglomeration [13]. This is the major concern of mixing both powders to supplement the powders usage for AM. The following sections compare and discuss the rheological measurement of both powders.

3.1. Dynamic Property

The dynamic property test consists of dynamic flow test, aeration test, and consolidation test. The torque and axial force on the blade are measured and the resistance to flow is calculated and illustrated as a flow energy. The results of dynamic flow test are shown in Figure 1, including basic flowability energy (BFE), specific energy (SE), stability index (SI), and flow rate index (FRI). The BFE represents the energy required to displace a powder during non-gravitational forced flow, i.e. the powder's resistance to flow in a constrained environment. MetcoAdd 316L powder has a higher BFE which indicates the powder is resistant to the forced flow. The SE measures the energy required for gravity induced flow (resistance to flow in an unconstrained environment). The marginally higher SE value of MetcoAdd 316L powder indicates an increased level of mechanical interlocking and friction. The measurement results of BFE and SE imply the insensitivity of the MetcoAdd 316L powder subjected to powder dispensing of AM process. Powder particles need a higher stimulating energy to flow across the powder bed. The SI is a measure of the physical stability of the material as a function of repeat tests. MetcoAdd 316L powder appears to be physically stable with an SI value close to 1. The higher SI value of FS 316L powder suggests this powder is more sensitive to be handled and processed. The FRI Measures the response of the powder to change flow rates, especially relevant for mixing applications and variable feed rate environments. The FS 316L powder shows a little higher FRI value, indicating a slight sensitivity to changes in flow rate.

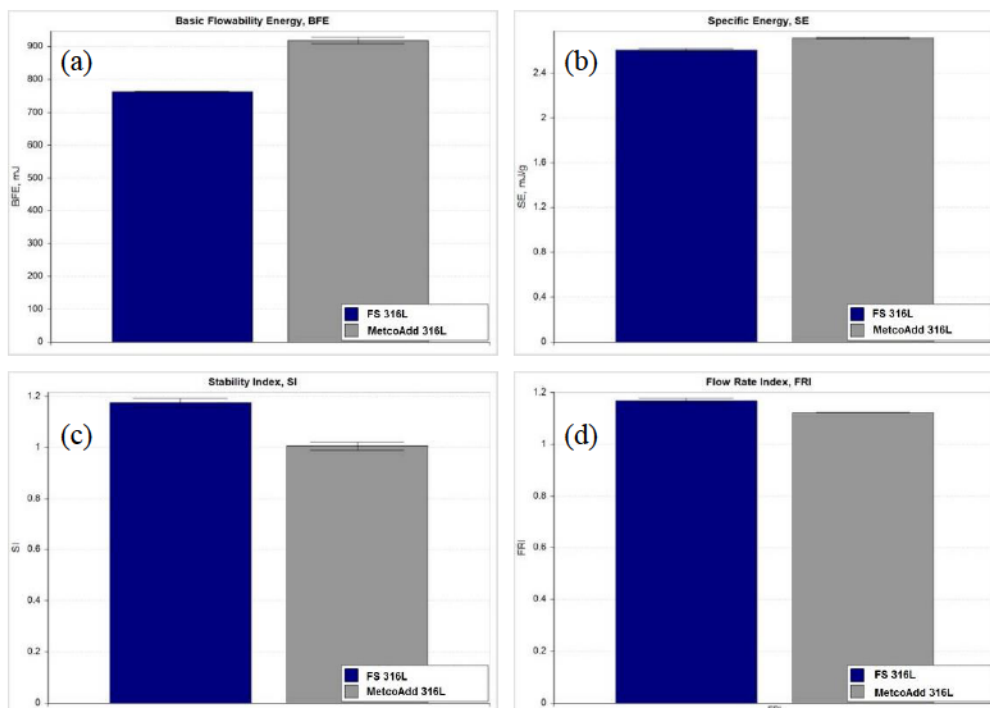


Figure 1. Results of dynamic flow test. (a) basic flowability energy; (b) specific energy; (c) stability index; (d) flow rate index

The aeration test measures the changes in the flow properties due to the introduction of air into the sample. It identifies how powder behaves during mixing, pneumatic conveying and fluidization. It ultimately provides an indication of absolute cohesive strength. As shown in Figure 2, the FS 316L powder presents a higher value of aeration ratio. Thus, this powder is easily to reach a fluidized state at a lower air velocity in comparison with the MetcoAdd 316L powder. The difference of powders to aeration may not be influential to PBF base AM process. But for the LENS process, it could be critical [14]. The consolidation test measures the impact of tapping on the flow properties of a powder. High consolidated energy (CE) value can be indicative of some potential problems during processing or transportation due to consolidation. Figure 3 shows a higher CE value of the MetcoAdd 316L powder. Its particles are more sensitive to vibrational forces and are liable to be packed resulting in increased resistance to flow.

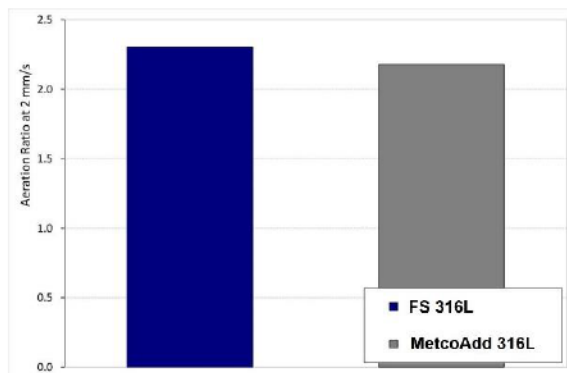


Figure 2. Result of aeration test

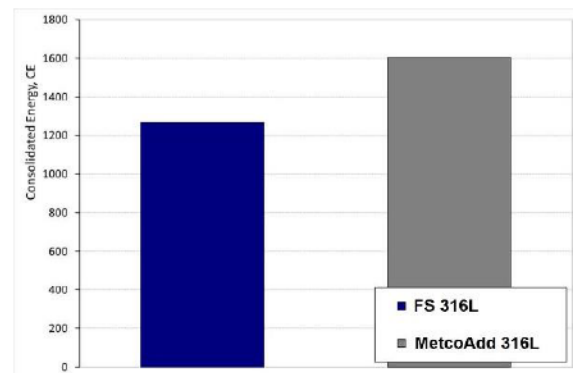


Figure 3. Result of consolidation test

3.2. Bulk Property

Permeability test and compressibility test were conducted to reveal the bulk property of both powders. The permeability test measures the air pressure required to maintain a constant air flow through the powder bed under an increasing normal stress. Generally, a higher normal stress results in a higher Pressure Drop (PD) as the permeability is reduced due to particles becoming more closely packed. MetcoAdd 316L powder exhibits a higher PD value, as shown in Figure 4, indicating a decreased permeability. Low permeability is not desired in the discharging or filling process of metal AM processes. Compressibility is simply a bulk property measurement that reflects the ability of the powder to become consolidated when subjected to a normal stress. It is not a direct measure of flowability but can be used to understand the behavior of volume change when being handled in the powder dispensing process. The FS 316L powder and MetcoAdd 316L powder show a comparable compressibility value (Figure 5). Hence, both powders have similar levels of particle packing efficiency.

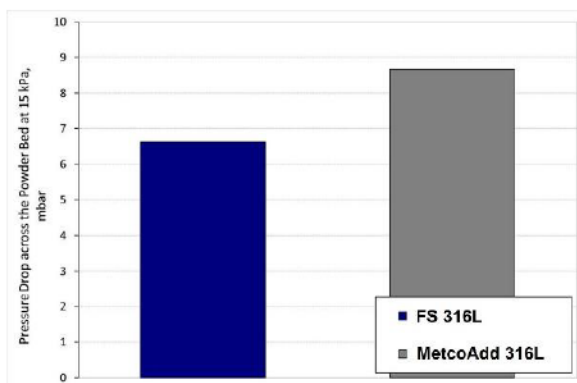


Figure 4. Result of permeability test

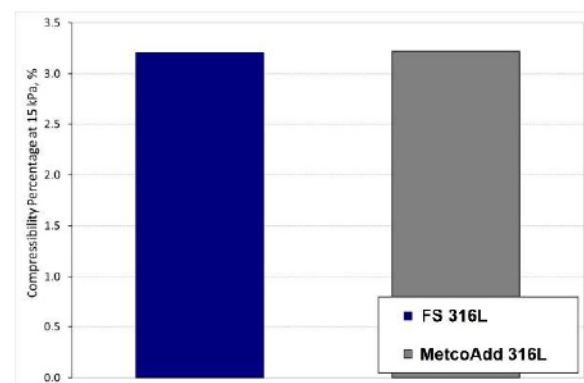


Figure 5. Result of compressibility test

3.3. Shear Property

Shear property measurement includes shear cell test and wall friction test. The shear cell test is intended to determine the shear stress required to initiate flow in a pre-consolidated powder. This provides an indication of how easily a powder will move from a static condition into dynamic flow. Figure 6 shows the result of shear cell test. It is noted that the two powders have comparable shear stress values, indicating a similar resistance to the onset of flow at this level of consolidation. The wall friction test is similar to the shear cell test. Instead of a shear cell, a disc made of a known material is forced to shear against the powder bed surface, rather than the powder being forced to shear against itself. Varied normal stresses are applied to the disc during the test. As shown in Figure 7, the wall friction angle values are also comparable. Hence, both 316L powders exhibit similar shear properties.

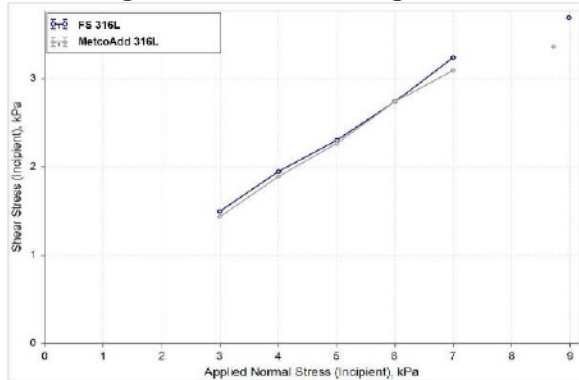


Figure 6. Result of shear cell test

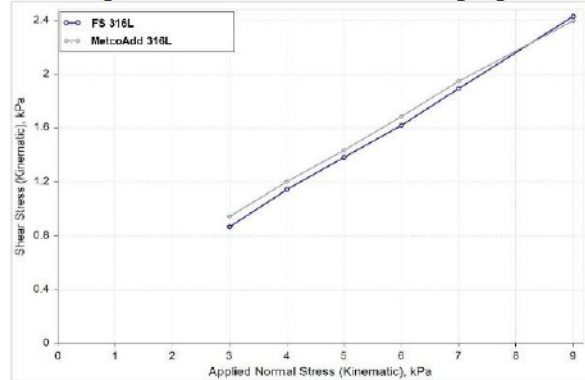


Figure 7. Result of wall friction test

4. Summary

Two stainless steel powders were measured and compared in this study regarding their rheological properties for PBF based AM process. The dynamic property, bulk property, and shear property of both powders were tested and discussed. The dynamic flow test indicates a distinguishable difference of powder flowability. But it is believed that the flow of virgin powder would be improved due to the loss of small particle after several production cycles. Other testing results indicate comparable results of two powders. Overall, it is essential to employ the powder rheology to measure the powder attributes. The testing results confirm that the newly acquired MetcoAdd 316L powder (virgin powder) is compatible to the used FS 316L powder when mixing them.

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