COLLEGE of ENGINEERING
MIAMI

TECHNICAL REPORT

Metal Powder Characterization of Sample A (17-PH Stainless Steel)

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MAE 531

I. Objective

Metal powder characterization is crucial to the additive manufacturing industry. When feedstock arrives at a laboratory or a plant, the quality of metal needs to be validated to print parts safely and consistently. To identify metal powder, a test sequence must be performed with precise laboratory equipment. Under different tests, metals and metal alloys exemplify several defining characteristics such as visual, powder flow, and chemical composition. These properties affect printing and part outcomes including tensile strength, print specifications, and part finish. In this report, a full test sequence for metal powder will be performed to identify a sample's composition and assess the printability of the metal powder.

II. Background

A. Purpose

In the context of MAE 531: Introduction to Scientific and Engineering Foundations of Additive Manufacturing, an emphasis on the importance of material selection, specifically metallic alloys, provides the basis for additive manufacturing. By performing different tests to identify the qualities of the metallic powder, students will gain a better understanding of the relationship between material properties and additive manufacturing. Furthermore, they will strengthen their ability to work with technical laboratory equipment. Students of MAE531 receive an unidentified sample of metal powder and must use specific tests to conclude the metal powder composition and printability.

B. Tests Performed

Tests Performed	Characteristics Identified
Visual Characteristics	
Surface Inspection	Geometry and Surface condition of powder particles, Agglomeration, Uniformity of distribution, Foreign contamination, Segregation, Maximum and Minimum diameter of powder particles
Porosity and Contamination	Porosity, Contamination of powder core
Microstructure	Geometry of powder under high magnification
Powder Flow Characteristics	
Hall Flow Study	Speed of flow through an Orifice diameter of 0.1" or 2.5mm
Density Characteristics	
Apparent Density	Bulk density (mass per unit volume of loose packed powder)
Tap Density	Compressed density (mass per volume of compact packed powder)
Chemical Characteristics	
Energy Dispersive X-Ray	Chemical composition by Weight%
Particle Size Analysis	
MicroTrac Particle Size Analysis	Particle size statistics including distribution, average, and standard deviation

C. Reasoning for Tests

Every test in the sequence provides a descriptive characteristic that produces a well-rounded conclusion to the unidentified material and its printability.

Visual characteristic testing such as Surface Inspection tests provide identification of material flaws that could affect quality of the print. For example, if the material has irregular shaping, the printing of the part will be inconsistent and include holes from uneven surfaces. In addition to geometry, porosity affects consistent printing because the holes in the metal powder contribute to irregularities in the part. The high magnification of the metal powder using the Scan Electron Microscope enables a high-resolution image of the metallic powder microstructure to be captured.

Powder flow characteristic testing is required to predict the flow through a nozzle. The Hall Flow Study is standardized to ASTM B213 requiring that a powder consistently tests across laboratories. Standardization applies to many additive manufacturing areas especially with metals since the metal must flow consistently to produce a reliable part.

Density characteristic testing such as the Apparent and Tap Density tests provide information about the density of the powder before and after compaction, respectively. Density describes the volume per mass unit of which can compare to different metal alloys. This will be used to strengthen what the powder consists of.

Chemical characteristic testing provided by the Energy Dispersive X-Ray identifies chemical composition. This provides an elemental analysis of the powder and leads to a conclusion of the specific metal alloy.

Particle size characteristic testing identifies and distributes particle size. The ideal distribution should be aligned to the gaussian curve, and the addition of peaks or skewed results could mean different elements or alloys.

III. Experimental Procedure

Surface Inspection

1. Pour metal sample A into a plastic tray to cover the surface of the bottom of the tray in a LABCONCO Glove Box. To ensure proper use of the glove box, refer to LABCONCO’s operation manual.

2. Prepare Keyence VHX-5000 by referring to the Johnson & Johnson Collaborative Laboratory Keyence VHX-5000 Standard Operation Procedure using a 300x lens.
3. Place metal sample under lens and capture photos.
4. Apply rough measurements of particle diameter and save.

Porosity

1. Prepare acrylic sample of metal sample A with the Struers CitoPress-5 by referring to CitoPress-5 SOP written by Johnson & Johnson Collaborative Laboratory.
2. Grind sample down to create cross section of acrylic sample by referring to Struers LaboForce-100 SOP by Johnson & Johnson Collaborative Laboratory.
3. Prepare Keyence VHX-5000 by referring to the Johnson & Johnson Collaborative Laboratory Keyence VHX-5000 Standard Operation Procedure using a 1000x magnification.
4. Place metal sample under lens and capture photos.
5. Save photos.

High Magnification Observation

1. Start the Zeiss EVO 60 by referring to the manual.
2. Prepare sample on carbon tape and create vacuum within chamber.
3. Perform scan electron microscopy to a scale of magnification of 76x referencing to the magnitude of Polaroid 545.
4. Capture image and save photo.

Hall Flow Study

1. Refer to procedure section of ASTM B213.
2. Record flow time

Apparent Density (Bulk Density)

1. Refer to procedure section of ASTM B703.
2. Record mass of powder
3. Calculate Apparent Density using Formula 1

$$\text{Bulk density}(\rho_b) = \frac{\text{Weight of microcapsules(g)(M)}}{\text{Bulk volume(ml)}(V_b)} \quad (1)$$

Tap Density (True/Tapped Density)

1. Set up AS-100 Tap Density Machine by referring to the Johnson & Johnson Collaborative Laboratory AS-100 Tap Density Machine Standard Operation Procedure.
2. Refer to ASTM B527 for procedure and specifications of Tap Density testing.
3. Record mass of powder and final volume
4. Calculate Tap Density using Formula 2

$$\text{True/Tapped density}(\rho_t) = \frac{\text{Weight of microcapsules(g)(M)}}{\text{Tapped volume(ml)}(V_t)} \quad (2)$$

Energy Dispersive X-Ray

1. Start the Zeiss EVO 60 by referring to the manual.
2. Prepare sample on carbon tape and create vacuum within chamber.
3. Perform scan electron microscopy to a scale of magnification of 76x referencing to the magnitude of Polaroid 545.
4. Perform EDX examination for chemical composition of metal sample.
5. Record data and save.

Particle Size Analysis

1. Start the Microtrac S3500 Particle Analyzer by referring to the Standard Operation Procedure written by Johnson & Johnson Collaborative Laboratory
2. Adjust parameters of MicroTrac program for metallic samples.
3. Run Microtrac sample measuring according to the Standard Operation Procedure written by Johnson & Johnson Collaborative Laboratory.
4. Save and export results.

IV. Experimental Results

Visual Characteristics

The initial images of the Sample A surface inspection revealed several geometrical characteristics. The only apparent cosmetic defect was elongation. There were a large range of sizes with a minimum diameter of 14 µm and a maximum diameter of 71 µm recorded. No agglomeration, segregation, or foreign contamination (e.g. dust) identified in the images at 300x magnification.

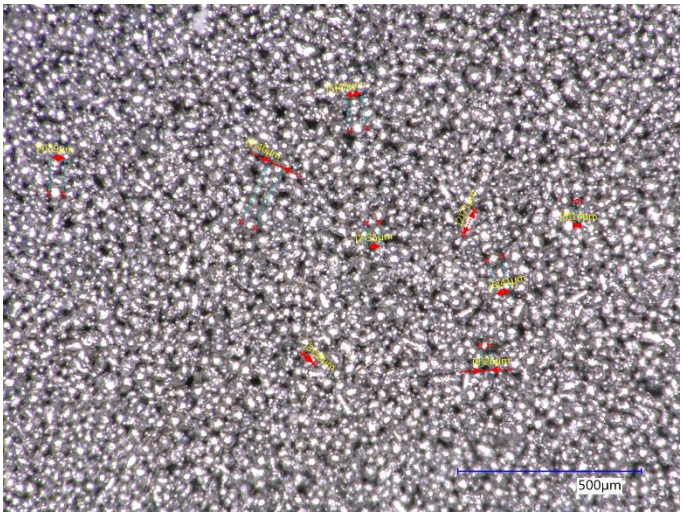


Figure 1
Keyence VHX-5000 300X Image with Measurements [µm]

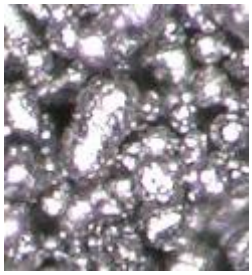


Figure 2
Keyence VHX-5000 300X
Elongation Example

High porosity and large grain boundaries identified in the images of the cross-sectional metal sample acrylic disk due to the geometric variation and lines of particles. There was no contamination of the metal particles since the core had a uniform appearance other than porous pockets in the images at 1000x magnification.

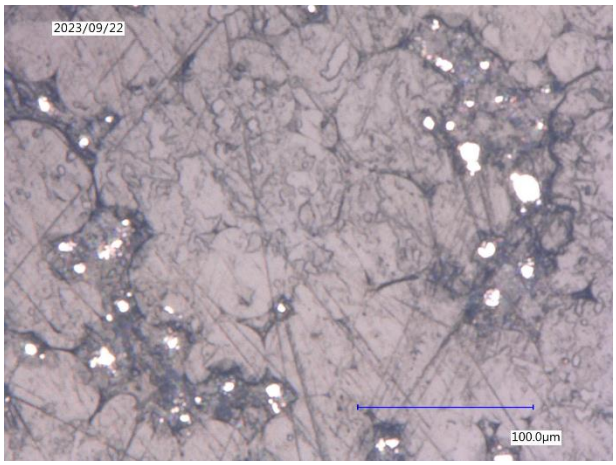


Figure 3
Keyence VHX-5000 1000X Image of Sample A Cross Section

High magnification images obtained by the scan electron microscope (SEM) confirmed geometric defects identified in the previous inspections. Different microstructure defects were classified such as elongation, agglomeration, satellites, and open porosity.

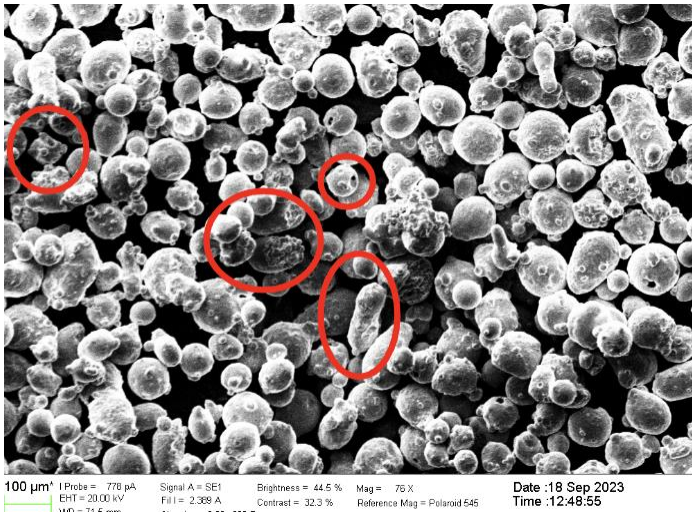


Figure 4
Zeiss EVO 60 SEM Image of Sample A Microstructure

Quantitative Characteristics

Apparent Density (Table 1)

Properties	**Values**
Mass of Container (g)	114.77
Mass of Container and Powder (g)	209.00
Mass of Powder (g)	64.23
Volume of Container (g)	25
Apparent Density (g/cm^3)	2.57

Tap Density (Table 2)

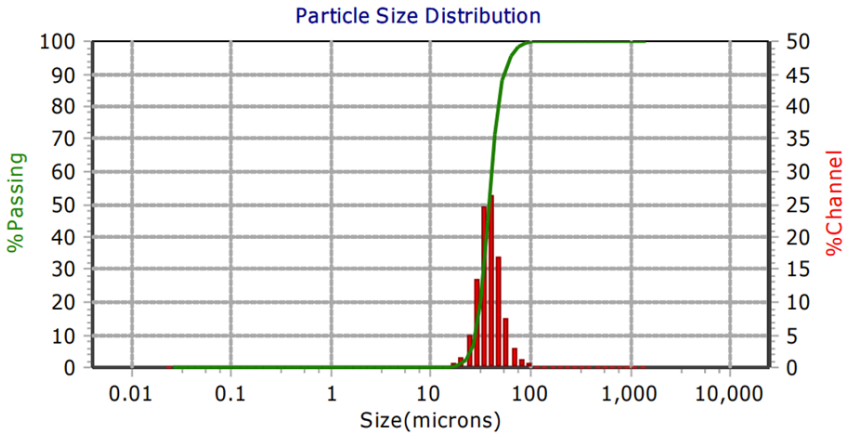
Properties	**Trial 1**	**Trial 2**
Mass of Powder (g)	99.99	99.99
Volume of Container (g)	22	22
Tap Density (g/cm^3)	4.55	4.55

Hall Flow (Table 3)

Properties	**Trial 1**	**Trial 2**	**Trial 3**
Mass of Powder (g)	50.12	50.12	50.12
Time Flowing (s)	34.94	33.03	27.65

Particle Size Analysis (Table 4)

Statistics	**Run 1**	**Run 2**	**Run 3**
Average Diameter (µm)	37.27	38.23	38.75
Standard Deviation (µm)	10.06	9.90	9.80

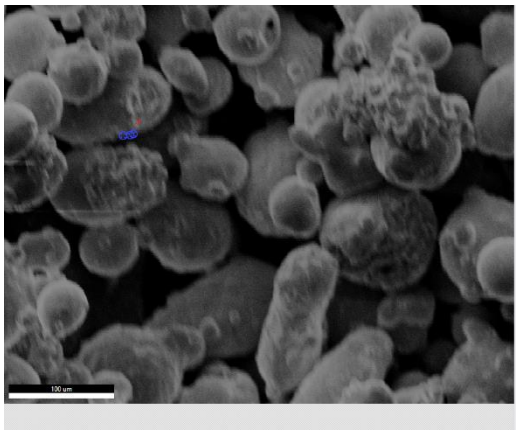


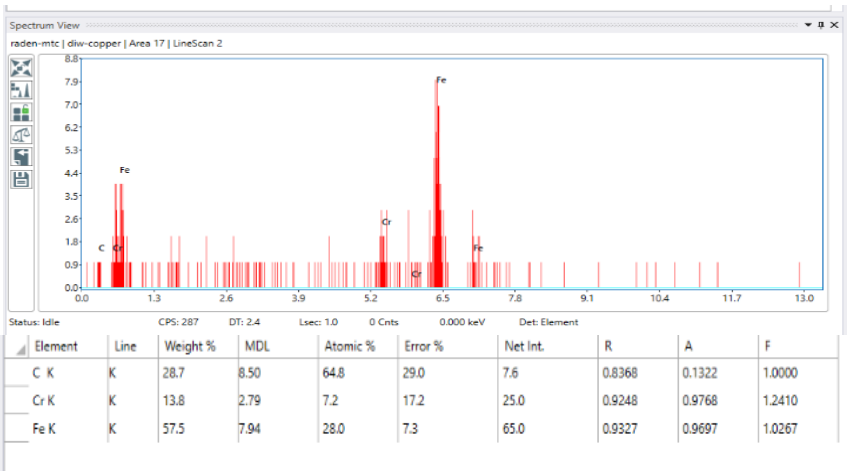
Percentiles	
%Tile	Size(um)
10.00	27.63
20.00	31.05
30.00	33.56
40.00	35.88
50.00	38.23
60.00	40.73
70.00	43.63
80.00	47.55
90.00	54.02
95.00	60.93

Figure 5
MicroTrac Particle Size Distribution of Sample A

Chemical Composition

Energy Dispersive X-Ray





Element	Line	Weight %	MDL	Atomic %	Error %	Net Int.	R	A	F
C K	K	28.7	8.50	64.8	29.0	7.6	0.8368	0.1322	1.0000
Cr K	K	13.8	2.79	7.2	17.2	25.0	0.9248	0.9768	1.2410
Fe K	K	57.5	7.94	28.0	7.3	65.0	0.9327	0.9697	1.0267

Figure 6
Zeiss EVO 60
Energy Dispersive X-Ray Chemical Composition Result

V. Analysis of Results

Based on the results of the metal characterization tests, two metals align with the characteristics of Sample A — 17-4 PH Stainless Steel Powder and 440C Stainless Steel Powder.

440C SS and 17-4 PH SS are considered due to the similarity of chemical composition to Sample A. Fe, Cr, and C were identified in the EDX of the unknown metal powder. Due to limited variation in samples and error in carbon tape preparation, C represented 28.7%Weight. This is a relatively high percentage compared to the C %Weight makeup of 440C and 17-4 PH Stainless Steel Powder. To account for the error of C %Weight, only elemental presence is considered in the identification of metal. Cr and Fe are present in both stainless steels. The chemical composition as evidence of a specific metal alloy is not entirely conclusive. Correct carbon tape preparation and more samples would have provided a more conclusive chemical composition.

The Hall Flow of Sample A resulted in no flowability. Due to the sticky quality of the powder, there was no consistent flow of material. The data of this test cannot support a strong conclusion on the powder. Humidity is attributed to inconsistent flow, and it is recommended to heat powder before flow to reduce moisture.

The Apparent Density of Sample A was 2.57 g/cc and Tap Density was 4.545 g/cc. Sample A and 17-4 PH Stainless Steel have a 1.18%Error of Apparent Density which is significantly less than the 11.38%Error comparing Sample A to 440C Stainless Steel. The Tap Density error between 17-4 PH Stainless Steel and 440C Stainless Steel is 3.29% when compared to Sample A. Because of smaller error between the Apparent Density expected values of 17-4 PH Stainless Steel and Sample A, the data supports 17-4 PH as the unknown metal sample.

Sample A is not recommended to be used for 3D printing. Although the chemical composition analysis leads to printable stainless steels for laser powder bed fusion, the visual

characteristics of the powder will cause deformities in printed parts. Inspection with increasing magnification identified several geometrical flaws such as elongation, open porosity, satellites, and agglomeration lead to uneven flow and inconsistent prints. The cross section of the suspended powder revealed porous pockets inside of the metal particles.

The MicroTrac reported a gaussian distribution of particle size. The average particle diameter after 3 trials was 38.08 µm with a standard deviation of 9.92 µm. There is a high %Error for 17-4 PH SS and 440C SS particle diameter compared to Sample A's average diameter. 17-4 PH SS had a 62.94%Error and 440C SS had a 41.56%Error. Because of the even distribution of particle size, above average particle diameter, and poor visual characteristics of the metal powder, it is plausible that the metal was recycled. Most likely, after a Laser Powder Bed Fusion print and prepared for the next cycle. This process ensures even distribution of powder but cannot eliminate defects and abnormal geometry in the metal powder.

%Error for Apparent Density, Tap Density, and Particle Diameter comparing Sample A to 17-4PH SS and 440C SS (Table 5)

Alloy	**Apparent Density [g/cc]**	**Tap Density [g/cc]**	**Particle Diameter [µm]**
Sample A	2.57	4.545	38.08
17-4 PH SS	2.54	4.7	23.37
17-4 PH SS %Error	1.18%	3.29%	62.94%
440C Stainless Steel	2.9	4.4	26.9
440C SS %Error	11.38%	3.29%	41.56%

The results of this analysis align with expectation of a stainless steel powder conclusion. The specific stainless steel alloy is unclear because of the errors due to novice experience using lab equipment and unaccounted moisture of powder. To establish a confident selection

of stainless-steel, it is recommended to repeat the qualitative and chemical characteristic testing of Sample A.

VI. Conclusion

The testing sequence to characterize metal powder is crucial to understanding metallic properties and the applications to additive manufacturing. MAE 531: Introduction to Scientific and Engineering Foundations of Additive Manufacturing, highlights the importance of material selection, specifically metallic alloys. By performing different tests to identify the qualities of metallic powder, students will gain practical knowledge with laboratory equipment and how to analyze a metal powder. Through comprehensive testing and analysis, Sample A is concluded to have potential matches with 17-4 PH Stainless Steel Powder and 440C Stainless Steel Powder.

The presence of Fe and Cr alluded to stainless steels. However, due to carbon tape preparation errors, a large %Weight of C skewed EDX results. The Hall Flow test indicated limited flowability which can be attributed to humidity and moisture within Sample A. Apparent and Tap Density comparisons of %Error suggested that Sample A is a closer match to 17-4 PH Stainless Steel.

After visual analysis, 3D printing with Sample A is not suggested. Sample A displayed visual flaws and irregularities. The large particle diameter suggested possible recycling after printing since there was an even distribution of size, but sieving cannot reduce all issues like porosity.

Further tests are recommended to ensure reproducibility of results. Since students were novices with subject material and lab equipment, the knowledge gained throughout the process can be applied to the future. Two recommendations would be mounting the carbon tape correctly in the SEM and heating powder to remove moisture which ensures proper Hall Flow testing.

Material characterization is the foundation of additive manufacturing. Every setting from nozzle size to temperature of print bed relies on the specifications of the material used. This analysis applies to current problems in the additive manufacturing industry and prepares students of MAE531 for future opportunities.

Appendix

1. **Elemental Composition Comparison by Avg %Weight**

Alloy	**Elemental Composition Comparison by Avg %Weight**									
	Fe	**Cr**	**Cu**	**C**	**Si**	**Mn**	**Ni**	**Co**	**Mo**	**Nb**
Sample A	**57.5**	**13.8**	**-**	**28.7**	**-**	**-**	**-**	**-**	**-**	**-**
17-4 PH SS	**Bal.**	**16.25**	**4.00**	**0.07**	**<1.0**	**<1.0**	**4**	**-**	**<0.5**	**0.3**
440C SS	**Bal.**	**17**	**-**	**1.05**	**<1.0**	**<1.0**	**<0.6**	**-**	**<0.75**	**-**

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