Characterisation of Ti6Al4V (ELI) Powder Used by the South African Collaborative Program in Additive Manufacturing

by

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Abstract

The South African Additive Manufacturing Strategy recommends research and development towards "Qualified AM parts for medical and aerospace" as one of the national priorities. In response to this, the national Collaborative Program in Additive Manufacturing includes a sub-programme on "Qualification of Additive Manufacturing of Ti6Al4V for Medical Implants and Aerospace Parts". This sub-programme entails comprehensive systematic research aimed at establishing a database of material and process data needed for qualification of the AM powders, AM processes and post-process treatments used for medical and aerospace applications.

The paper describes the approach taken to characterise the Ti6Al4V (ELI) powder in the as received state and after exposure during repeated AM build cycles, by determining the physical and chemical properties of powder used in two selective laser melting (SLM) machines and a laser engineered net shaping (LENS) machine. Properties of the powder that were determined include the particle size, particle size distribution, morphology, oxygen and nitrogen gas content, and elemental composition. Characterisation techniques employed are Scanning Electron Microscopy, Laser Diffraction, Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), and Inert Gas Fusion. The results of the analyses on as-received powder and powder after exposure to numerous build cycles in the different AM machines are presented and discussed.

1. INTRODUCTION

Industrial applications of Additive Manufacturing (AM) range from aerospace to dentistry and medical implants. The South African Additive Manufacturing Strategy, developed with funding support by the Department of Science and Technology, recommends research and development towards “Qualification of AM parts for medical and aerospace” as one of the national priorities. In response to this, the national Collaborative Program in Additive Manufacturing was established. It includes a sub-programme on “Qualification of Additive Manufacturing of Ti6Al4V for Medical Implants and Aerospace Parts”. This sub-programme entails comprehensive systematic research aimed at establishing a database of material and process data needed for qualification of the AM powders, AM processes and post-process treatments used by the collaborators for medical and aerospace applications [1].

According to Slotwinski and his team [2], application of existing standardised powder methods for AM powders ensures the repeatable manufacture of metal parts with reliable properties [2]. This paper describes the approach taken to characterise the Ti6Al4V (ELI) powder in the as received state and after exposure during repeated AM build cycles, by determining the physical and chemical properties of powder used locally in two selective laser melting (SLM) machines and a laser engineered net shaping (LENS) machine. Knowing the characteristics of the powder used by all collaborators in the Collaborative Program in Additive Manufacturing is a necessary part of the larger qualification process. The powders used by the collaborators were purchased from the same supplier (TLS Technik GmbH) and were characterised according to the ASTM F 3049 – 14 standard [3].

2. CHARACTERISATION PROCEDURE

The following institutions collaborated in the study [1]:

- Central University of Technology (CUT) used an EOS M280 DMLS system capable of manufacturing metal parts by selective laser melting of Ti6Al4V (ELI) powder in the size range of <40 µm.
- Stellenbosch University (SU) performed selective laser melting of Ti6Al4V (ELI) with a Concept Laser M2 system. The system was designed to process the powder in an argon atmosphere at all times and used powder of particle range 25 – 55 µm.
- National Laser Centre (NLC) operated a powder blown Optomec 850R laser engineered net shaping (LENS) system. The system used Ti6Al4V (ELI) powder of particle size ranging between 40-100 µm.

The applicability of existing standardized powder measurement methods for AM powders, and the techniques utilised to measure chemical composition, morphology and size distribution of metal powders are described.

2.1. Powder sampling

The ASTM 3049-14 standard [3] provides a standard guide for characterising metal powder for AM including its chemical composition, morphology and size distribution. The powder as received from the supplier was analysed to determine potential variability in the properties of powder with differing size ranges taken from the same production batch and the influence of reusing powders after numerous build cycles. As powder reuse times increased, the properties of the powder were expected to be affected. The powder samples were characterised in the laboratories of CSIR and NECSA in South Africa.

Samples from as-received Ti6Al4V (ELI) powder supplied to CUT, SU and NLC, all from the same production batch, were taken for analysis. The NLC samples were taken from the sieved powder since the powder was not reused for more than one cycle. Samples of reused un-sieved Ti6Al4V (ELI) powder from CUT and SU, as well as sieved Ti6Al4V (ELI) from NLC were also taken for analyses to determine the changes in powder properties due to recycling of the powder. Representative samples were taken from a powder batch by using a sampler based on the Keystone Sampler [4] shown in Figure 1.

Figure 1: The Keystone-based Sampler designed by CUT

The Keystone Sampler is a long tubular device with a sample chamber along its length which can be opened and closed by turning the inner tube. It can be placed in multiple representative locations to extract powder samples, for example it can extract powder samples from the bottom, centre and top of a powder container. The sampling procedure used complied with ASTM B215-10 [4].

In Table 1, all the powders investigated in this study are listed. The new powder was analysed as received from the supplier and in all three cases it came from the same batch supplied by TLS Technik GmbH. The reused powders were used in the different additive manufacturing systems for several cycles.

Table 1: Powder samples investigated

Name	Manufacturer	Batch	Condition
Ti6Al4V (ELI) DMLS EOSINT M280	TLS Technik GmbH	1004763	New powder
Ti6Al4V (ELI) DMLS EOSINT M280	TLS Technik GmbH	1004763	Reused powder Cycle 11
Ti6Al4V (ELI) Concept Laser M2	TLS Technik GmbH	1004763	New powder
Ti6Al4V (ELI) Concept Laser M2	TLS Technik GmbH	1004763	Reused powder Cycle 3
Ti6Al4V (ELI) Optomec 850R LENS	TLS Technik GmbH	1004763	New powder
Ti6Al4V (ELI) Optomec 850R LENS	TLS Technik GmbH	1004763	Reused powder Cycle 1

2.1.1. CUT new and reused powder

The CUT system is equipped with a 200 watt fibre laser and operates in a protective argon atmosphere. Samples from three identical canisters of as-received new Ti6Al4V (ELI) powder were taken for analysis. For the CUT EOS M280 system the as-received powder was also dried at a temperature of 80 ºC for two hours and sieved before dispatching it into the dispensing container of the machine for the first build. A 100g sample of new powder was taken from each canister as received from the supplier for determination of the physical and chemical properties. The initial fabrication started with 33.6 kg of virgin powder from 14 canisters each weighing 2.5 kg. Two samples (50g each) from the 2nd and 4th use cycle were taken for chemical analysis. The machine's powder container capacity allowed for 10 times of reuse (10 cycles) of the powder in AM build operations without top-up with new powder. The new and reused powder were mixed and three samples (top, middle and bottom from the collected container) were taken after the 11th cycle for analysis. The samples were characterised for chemical composition, morphology and particle size distribution of the powder.

2.1.2. SU new and reused powder

The SU system was designed to meet the material storage and processing requirements of powders under an argon atmosphere at all times. The unused powder can be returned to the to the storage chamber at the end of a production cycle, providing efficient powder recycling and ensuring that the operator has no contact with the powder and the powder has no contact with the air. Samples (50 g each) of the new powder from three different containers were taken for analysis. A set of three builds were performed and after each build was completed the powder was submitted to a sieving process to remove large particles. Three samples (mass of 50g), one after each cycle were collected during the process and taken for analysis. The new and reused powder samples were characterised for physical and chemical characteristics of the powder.

2.1.3. NLC new and used powder

The NLC LENS machine uses powder-blown technology to build fully dense complicated prototypes directly from the CAD using a high powered laser beam. Five samples (sample mass of 50g) of new Ti6Al4V (ELI) powder from different canisters were taken for analysis. The powder was used for building only once and the residual powder was collected by placing a foil under a substrate during the process. Two samples (mass of 50g) from the used powder were taken to determine any changes in properties with regard to the new powder. The new and used powder samples were characterised to detect chemical and physical changes of the powder.

2.2. Ti6Al4V (ELI) characterisation techniques

In this paper the new and reused Ti6Al4V (ELI) powder were characterised through standardised powder measurement methods for AM powders as specified in ASTM F3049-14 [3]. The nitrogen and oxygen contents in the powder were analysed using an ELTRA OHN 2000 inert gas combustion analyser. The elemental composition analysis was performed using an ARCOS Spectro ICP-OES instrument. The particle size distribution (PSD) was determined by a Microtrac SI/S3500 instrument. The morphology of the powder was determined using a JEOL JSM-6510 scanning electron microscope (SEM). Images generated from the sample were displayed in secondary electron (SE) mode, which provided the morphology and the surface appearance of the powder.

Table 2 summarizes the techniques used to characterise the powder samples in this study.

Table 2: Techniques / Methods used for powder characterisation

Characteristic	Method/Technique	Instrument
Elemental composition	Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-EOS)	SPECTRO ARCOS
Gas content	Inert Gas Fusion	ELTRA OHN 2000
Morphology	Scanning Electron Microscopy (SEM)	JEOL JSM-6510
Size distribution	Laser Scattering	MICROTRAC SI/S3500

3. RESULTS AND DISCUSSION

Metal powders used in AM should be spherical and have a particle size distribution that is designed to ensure good flowability and packing behaviour, such that the final manufactured part has good mechanical properties and is fully dense. Other important properties include chemical composition, morphology and size distribution of Ti6Al4V (ELI) powder. Here the chemical composition of the powder samples is presented first, followed by the morphology and the particle size distribution. Differences detected between new powder and after it has been recycled in the AM processes are discussed.

Note: In the following sections the term “canister” refers to a sample from a canister, with sample number 1 coming from Canister 1.

3.1. Chemical composition

The chemical compositions of all samples tested are listed in Tables 3 to 8 showing variations from three different new powders as received from the same supplier and powder recycled in different AM processes for several cycles. The aim in this part of the study, was to compare the results found for the different AM powders and confirm that they actually came from the same batch. It should be noted that the NLC powder had only been used once and two samples were taken from the used powder for analysis. The results of the new powder in this paper were compared to the ASTM F3001 standard [6] as well as the TLS Technik GmbH actual values shown in the tables.

3.1.1. CUT New and reused powder

From Table 3 it is clear that the composition of the new CUT powder complies with the ASTM F3001 standard [6]. The measured Ti, Al, V and Fe concentrations also compare well with the analysis results of TLS Technik GmbH. However, the oxygen and nitrogen concentrations measured for the different canisters are consistently higher than the TLS results, although still within the ASTM F3001 specification. These differences between the TLS results and the current results will have to be further investigated.

Table 3: Chemical composition of CUT new Ti6Al4V (ELI) powder

Specification	Chemical composition (weight %)					
	Al	V	Fe	N	O	Ti
ASTM F3001 Ti6Al4V (ELI)	5.5-6.5	3.5-4.5	0.25	0.05	0.13	89
TLS Technik GmbH	6.34	3.94	0.25	0.006	0.082	89
Canister 1	5.94	3.90	0.17	0.018	0.11	90
Canister 2	5.93	3.85	0.16	0.019	0.11	90
Canister 3	5.98	3.87	0.16	0.020	0.12	90
Canister 4	6.04	3.92	0.17	0.015	0.12	90
Canister 5	6.06	3.90	0.17	0.022	0.11	90

In Table 4 the compositions of the reused CUT Ti6Al4V (ELI) powder after 2,4,7 and 11 cycles are shown. Small changes of the Al and Fe contents of the powder versus the number of reuse cycles were observed. Variations of the O and N contents were insignificant when compared to new powder results, which means the inert argon atmosphere within the build chamber successfully prevented an increase of the gas contents. The results of the reused powder were still within specification and this indicates that the powder can be reused more than 11 times in the CUT EOS M280 system to build AM parts.

Table 4: Chemical composition of CUT reused Ti6Al4V (ELI) powder

Specification	Chemical composition (weight %)					
	Al	V	Fe	N	O	Ti
ASTM F3001 Ti6Al4V (ELI)	5.5-6.5	3.5-4.5	0.25	0.05	0.13	89
Cycle 2	6.15	3.97	0.16	0.018	0.11	90
Cycle 4	6.09	3.91	0.16	0.021	0.12	90
Cycle 7	5.89	4.07	0.23	0.015	0.11	90
Cycle 11	5.85	3.95	0.23	0.014	0.12	90

3.1.2. SU new and reused powder

The SU new powder results complied with the ASTM F3001 standard specification and differ slightly from the TLS Technik values (see Table 5). The variation of chemical composition from different canisters were insignificant when comparing to the ASTM F3001 standard specification but slightly differ from TLS Technik.

Table 5: Chemical composition of SU new Ti6Al4V (ELI) powder

Specification	Chemical composition (weight %)					
	Al	V	Fe	N	O	Ti
ASTM F3001 Ti6Al4V (ELI)	5.5-6.5	3.5-4.5	0.25	0.05	0.13	89
TLS Technik GmbH	6.34	3.94	0.25	0.006	0.082	89
Canister 1	6.04	3.83	0.16	0.010	0.079	90
Canister 2	6.07	3.81	0.16	0.078	0.078	90
Canister 3	6.06	3.84	0.16	0.05	0.078	90

The SU powder was reused three times in the concept Laser M2 system and results are displayed in Table 6. There are no significant changes on after the third reused of the powder. The SU powder can be reused in the concept Laser M2 system more than three time to build AM parts.

Table 6: Chemical composition of SU reused Ti6Al4V (ELI) powder

Specification	Chemical composition (weight %)					
	Al	V	Fe	N	O	Ti
ASTM F3001 Ti6Al4V (ELI)	5.5-6.5	3.5-4.5	0.25	0.05	0.13	89
Cycle 1	6.07	3.88	0.17	0.049	0.082	90
Cycle 2	6.21	3.89	0.18	0.010	0.092	90
Cycle 3	5.96	3.84	0.17	0.011	0.096	90

3.1.3. NLC new and reused powder

The analysis results of the NLC new powder in Table 7 show that the compositions of the samples from Canisters 1 to 5. The values shows no significance changes when comparing with ASTM F3001 specification and differ significantly from TLS Technik values but the composition complied with the specification.

Table 7: Chemical composition of NLC new Ti6Al4V (ELI) powder

Specification	Chemical composition (weight %)					
	Al	V	Fe	N	O	Ti
ASTM F3001 Ti6Al4V (ELI)	5.5-6.5	3.5-4.5	0.25	0.05	0.13	89
TLS Technik GmbH	6.34	3.94	0.25	0.006	0.078	89
Canister 1	6.70	4.42	0.31	0.012	0.058	89
Canister 2	6.53	4.33	0.30	0.010	0.057	89
Canister 3	5.85	3.94	0.24	0.014	0.061	90
Canister 4	5.95	4.03	0.25	0.093	0.056	90
Canister 5	5.39	3.57	0.09	0.084	0.058	91

The NLC powder was used once in the LENS system and two powder samples were taken, denoted as Cycle 1a and 1b shown in Table 8. No significant changes from the new powder were observed and the compositions were still within ASTM F3001 specification, given the accuracy of the measurements.

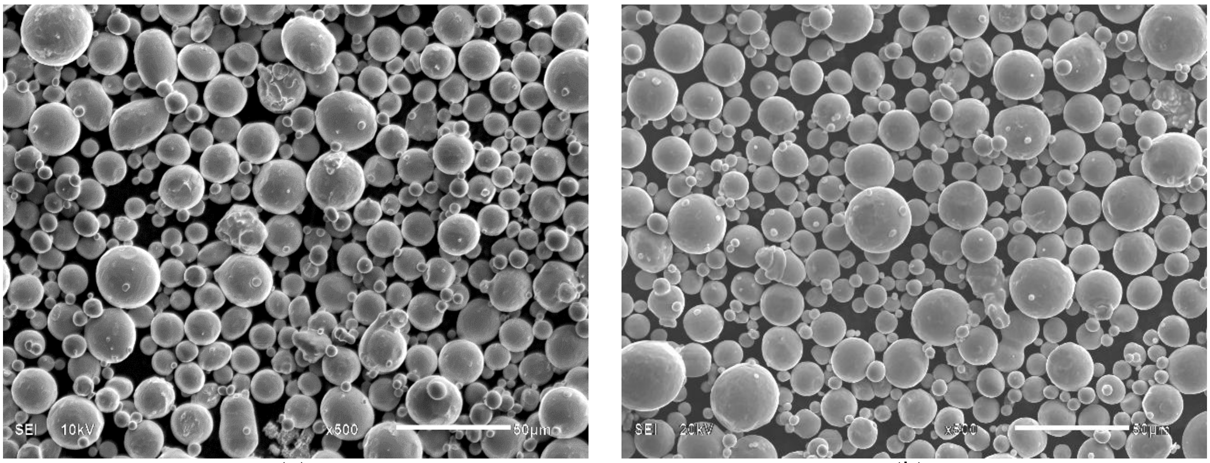
Table 8: Chemical composition of NLC reused Ti6Al4V (ELI) powder

Specification	Chemical composition (weight %)					
	Al	V	Fe	N	O	Ti
ASTM F3001 Ti6Al4V (ELI)	5.5-6.5	3.5-4.5	0.25	0.05	0.13	89
Cycle 1a	6.15	4.03	0.25	0.012	0.058	90
Cycle 1b	6.59	4.33	0.31	0.022	0.073	89

3.2. Particle Morphology

The shape of the powder particles can influence powder behaviour and the compaction during laser deposition processes. Samples from the new and reused powder from CUT, SU and NLC, were investigated through SEM using a consistent imaging scheme to enable comparison between the powders. Figures 2 to 4 show representative SEM micrographs for the Ti6Al4V (ELI) powders supplied by TLS Technik GmbH with different size distributions of <40 µm (CUT), 25 – 55 µm (SU), and 40 - 100 µm (NLC), all from the same batch of powder.

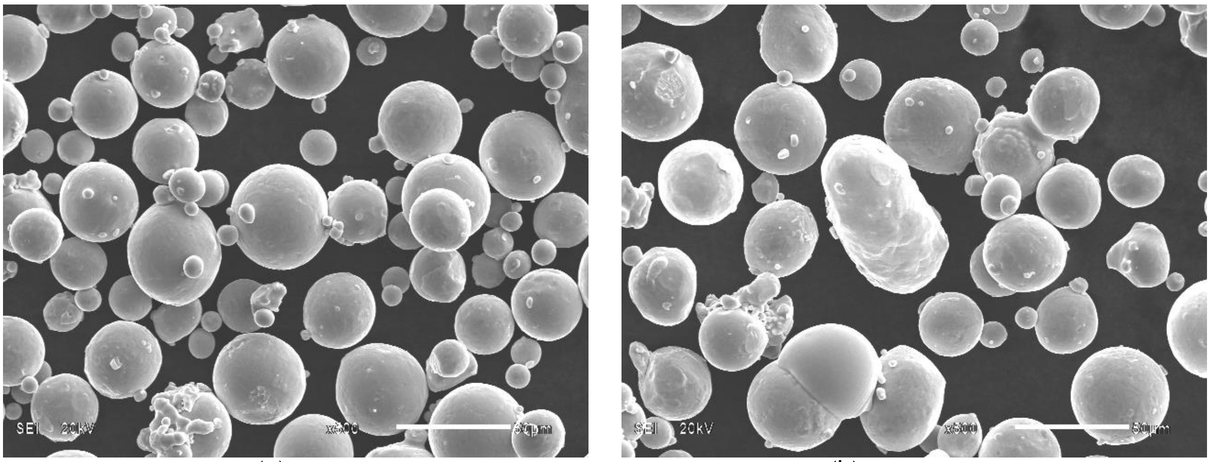
In Figure 2 the comparison of the new powder and reused powder used for 11 build cycles in the CUT EOS M280 machine is shown. The new powder in (Figure 2a) shows spherical particles, with some irregular and elongated shapes in the sample. Some elongated particles were observed after 11 cycles (Figure 2b), but the majority of powder particles were spherical. No clear deterioration of the reused powder was observed when compared to the new powder.



(a) (b)

Figure 2: SEM image of CUT powder: new (a) and after the 11th cycle (b)

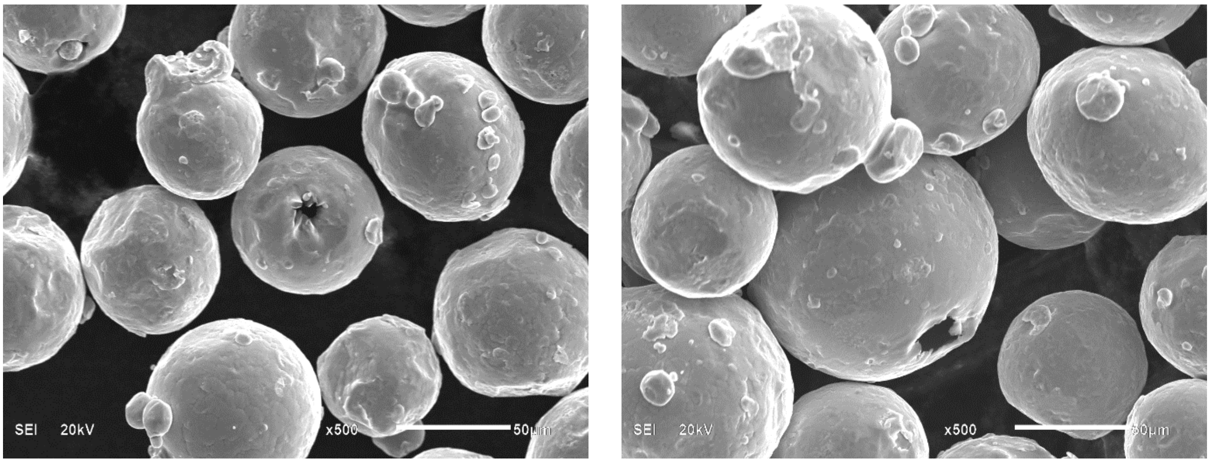
Figure 3 shows the morphology of the SU new powder (Figure 3a) and the powder after 3 build cycles (Figure 3b). The SU new powder showed small, irregular, rough satellites on some particles, but most of the particles were spherical. No convincing general dissimilarities between the new powder and the powder used for 3 build cycles were detected, except for some particles that were elongated.



(a) (b)

Figure 3: SEM image of SU powder: new (a) and after the 3rd cycle (b)

Figure 4 presents large spherical particles from the NLC powder with rougher surface textures than the CUT and SU powders and it also displays pores in some particles from both new powder and after the first cycle. After exposure during one build cycle some changes of the powder were observed. Evidence of fracture of some particles was found. Satellites were attached to larger particles in both the new powder and after one cycle.



(a) (b)

Figure 4: SEM image of NLC powder: new (a) and after 1 cycle (b)

3.3 Particle Size Distribution

Figures 5, 6 and 7 show the PSD curves for the Ti6Al4V (ELI) samples as measured by the Microtrac SI/S3500 laser diffraction analyser and Table 9 gives the corresponding data. The results of the new and un-sieved powder samples from CUT and SU, as well as the sieved powder samples from the NLC, are be presented by integrating various spherical size coordinates with respect to the volume.

The results show the PSD curves of the CUT new powder from five canisters overlap as shown in Figure 5. The overlapping confirms that the new powder samples from the different canisters were from the same batch of powder. No difference is observed between the new powder and the Cycle 11 powder taken from the bottom of the container of the EOS M280 machine. Cycle 11 powder from the middle of the container show small variations from the new powder. The slight shift towards larger sizes may indicate that there are fewer satellites attached to the powder particles. The Cycle 11 powder from the top of the container showed a significant shift towards a smaller particle size range, while there appears to be some particles in the 70 to 90 µm size range. This indicates that the top-up of the powder in the container after the 10th cycle affected the PSD of the top powder, while the PSD of powder lower down in the container was not affected.

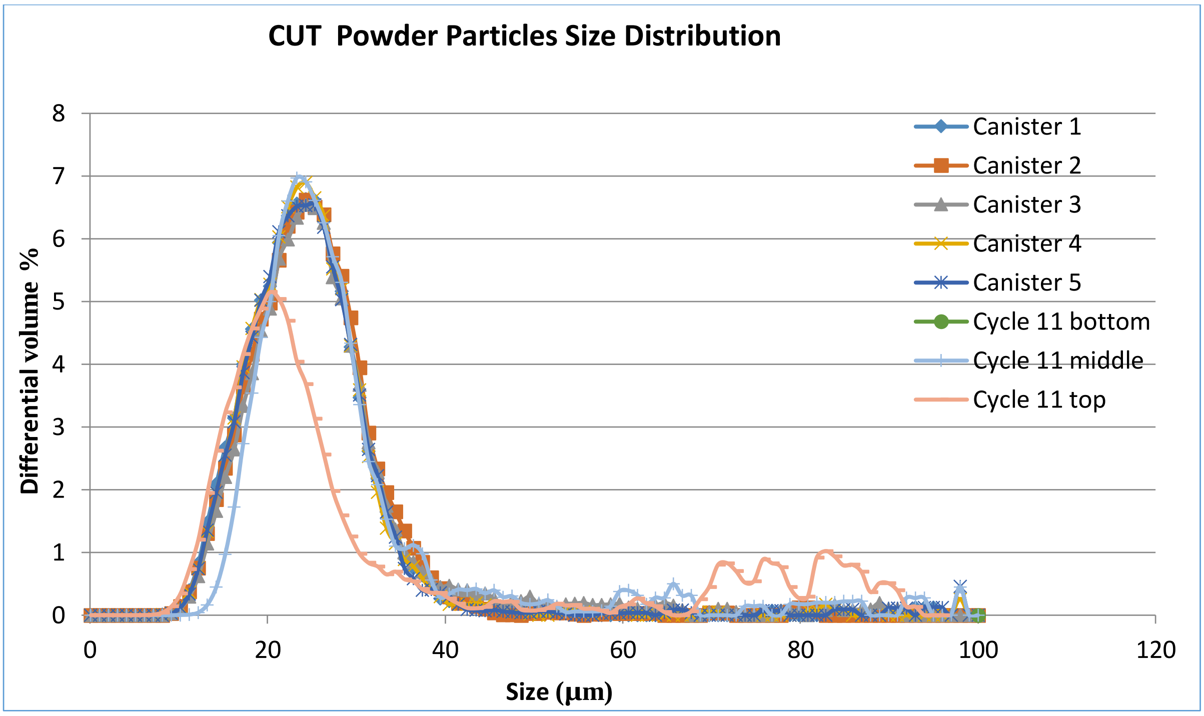


Figure 5: The differential PSD curves for the CUT samples

When comparing the three PSD curves of the SU new powder from the three canisters they appear identical, as shown in Figure 6. After the first reuse of the powder (cycle 1), no significant variations are observed, however after the second and third builds (cycle 2 and cycle 3) the size ranges of the powder particles seem to be larger than the new powder.

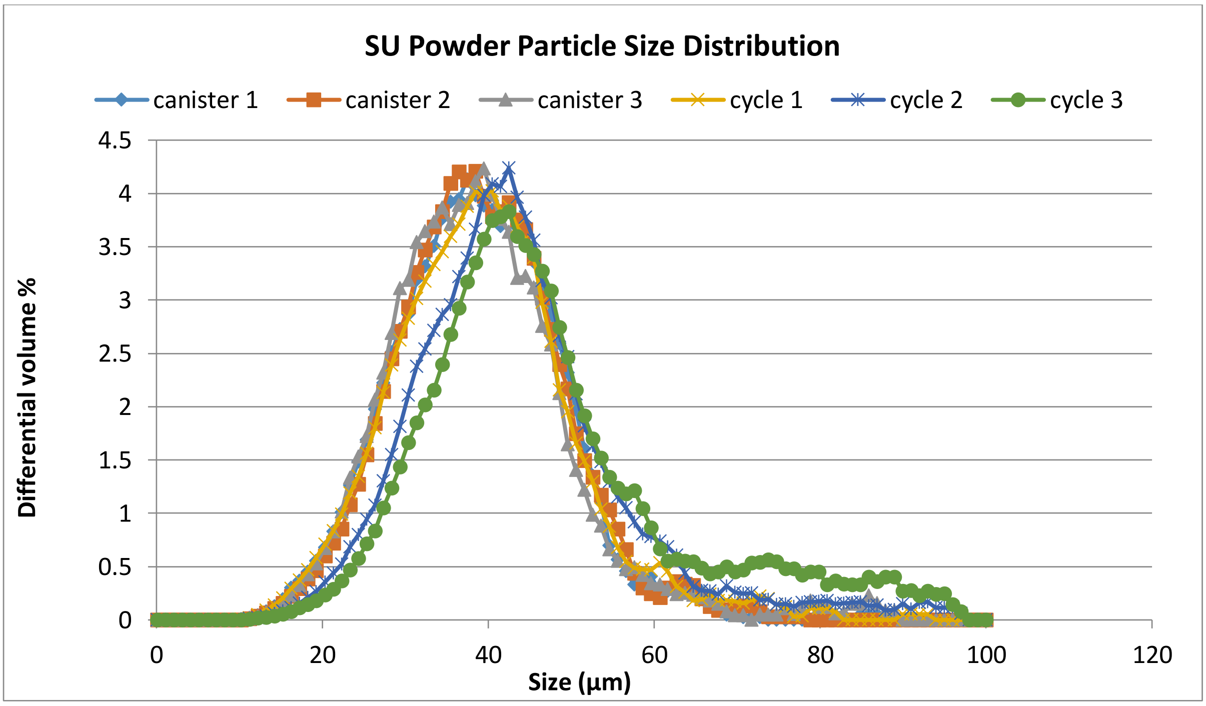


Figure 6: The differential PSD curve for the SU samples

Figure 7 shows the PSD curves for the NLC new powder and reused powder after the first build. When comparing the PSD curves for the new powder from the five canisters, it is clear that the powder samples from the same batch display significant differences. This indicates poorer consistency of particle size for this powder. The rougher, more irregular particles observed in Figure 4 may contribute towards this result. After the first cycle the PSD range seems to have broadened significantly towards larger sizes. It might result from fracture or deformation of particles as seen in Figure 4, but further investigation will be required to clarify this observation.

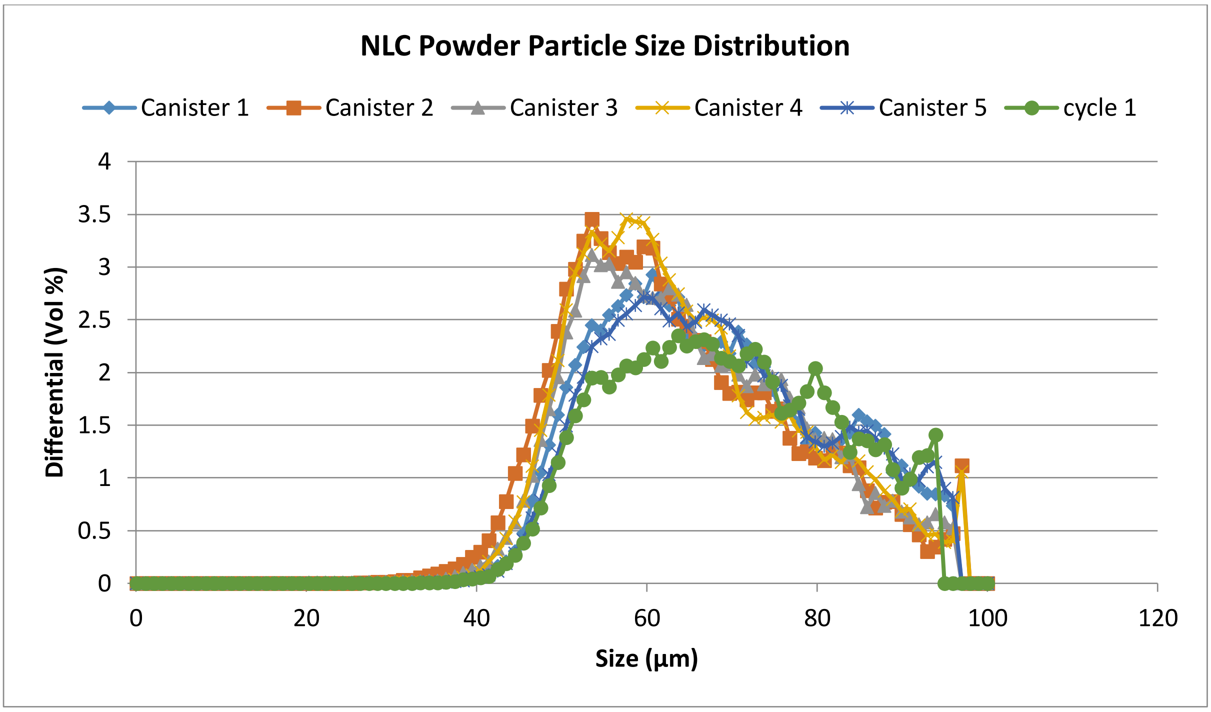


Figure 7: The differential PSD curve for the NLC samples

Table 9 shows the quantitative results derived from the laser diffraction PSD measurements. The D value ("mass division diameter") divides powder samples according to size categories and specifies their diameters in percentages relative to the median diameter D. D_{10} is the diameter at which 10% of the mass sample diameters are less than the D value. D_{50} is the diameter at which 50% are smaller than the D value, while D_{90} is the diameter at which 90% are larger than the D value.

Table 9: Numerical PSD results of new and reused Ti6Al4V (ELI) powders

CUT				SU				NLC			
Sample	D10 µm	D50 µm	D90 µm	Sample	D10 µm	D50 µm	D90 µm	Sample	D10 µm	D50 µm	D90 µm
Canister 1	18	23	34	Canister 1	28	40	54	Canister 1	54	69.8	95
Canister 2	18	26	34	Canister 2	28	41	53	Canister 2	51	64	87
Canister 3	19	26	37	Canister 3	29	41	53	Canister 3	53	67	93
Canister 4	18	26	33					Canister 4	52	65	88
Canister 5	18	26	34					Canister 5	55	71	97
Average	18	25	35	Average	27	41	54	Average	53	67	92
Cycle11 bottom	21	27	42	Cycle 1	29	41	56	Cycle 1	55	74	103
Cycle11 middle	20	27	46	Cycle 2	32	44	64				
Cycle11 top	21	30	107	Cycle 3	33	46	79				

The CUT Cycle 11 reused powder from the bottom and the middle of the container showed only slight increases in the D10, D50 and D90 values above the average values of the new powder and should still be acceptable for use in the EOS M280 machine. However, the Cycle 11 top powder shows a D90 value that is more than double the value for the new powder.

The SU reused powder shows size distributions increasing with the number of reuse cycles. The D10 value increases from 29 µm to 33 µm, D50 increases from 41 µm to 46 µm and D90 increases from 56 µm to 79 µm. This trend might limit the number of times that the powder could be reused in the SU Concept Laser M2 machine.

The NLC reused powder shows significant changes even after the first build cycle. Further investigation on this powder will have to be done to verify the initial observations.

4. CONCLUSIONS

- The results reported in this paper confirm the importance of characterising the Ti6Al4V powders used by the different collaborators in the Collaborative Program in Additive Manufacturing.
- While repeated reuse of the powders in the CUT and SU systems appears to be feasible without negative effects on the AM built parts, the allowable number of reuse cycles have to be determined by extending the duration of this study.
- From this study some concern about the powder used in the NLC system was raised. The composition and quality of these powders that are in the 40 – 100 µm size range do not seem to compare favourably with that of the size ranges used by CUT and SU. Further characterisation of this powder through e.g. pycnometer analyses and nano CT scanning is required to confirm the impression formed from the current work.

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