# Additive manufacturing titanium powder oxygen variation within a single powder bed due to differences in powder size and oxygen content

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# Abstract

Thermogravimetric analysis of Ti-6Al-4V powders (virgin Grade 5 and a once used Grade 23) was conducted up to 800 °C in an industrial grade Ar environment. A standard starting particle size distribution (PSD) for electron beam powder-bed fusion (PBF-EB) additive manufacturing (AM) was divided into two smaller ranges based on the D50 to observe the effects of oxidation (e.g. mass gain) resulting from the differences in available, reactive surface area during testing. Significant mass gain was still recorded throughout the testing owing to titanium's extreme reactivity at elevated temperatures even with the Ar cover gas. The mass gain can be attributed to impurities in the purge gas and absorbed water in the powder. The smaller particle size distribution powders accumulated more % mass gain than the full range and larger PSD, likely due to the increased surface area per unit volume of the smaller PSD powder. The virgin Grade 5 powder generally accumulated more oxygen/mass gain than the once used Grade 23 powder, likely resulting from a previously existing thicker TiO<sub>2</sub> shell on the once used Grade 23 powder. The results suggest that within a single AM PBF powder-bed, there may be varying levels of oxidation (both interstitial and oxide) depending on the particle size distribution and resulting surface area exposed to high temperatures for a given volume, and level of reuse (thickness of the oxide layer) on a per-particle basis if the batch had been blended. <sup>1</sup> *Keywords:* Additive Manufacturing, Ti-6Al-4V, Oxidation, Thermogravimetric analysis, Powder reuse

## 1. Introduction

Metal powder-based additive manufacturing (AM) [1], specifically laser or electron beam powder-bed fusion (PBF-L or PBF-EB), often incorporate the blending and reuse of unmelted feedstock powder to minimize process costs [2]. Aside from sustainability [3] and environmentally-driven [4] efforts to mitigate powder

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waste, there is a need to understand the complex processing-structure-properties-performance (PSPP) relationships from feedstock to finished part to ensure viability and consistency of the AM part performance [5]. Titanium alloy (Ti-6Al-4V) is widely used in PBF applications for its high strength-to-weight ratio and good corrosion resistance, which makes it a desirable material for biomedical implants [6] as well as aerospace applications [7]. A recent review [8] catalogs the microstructure and mechanical properties of Ti-6Al-4V typically observed when produced via PBF-L and PBF-EB. These properties can vary greatly depending on the number of times the unconsumed powder has been reused, owing to changes in chemistry (e.g. oxidation) [9–13], powder size distribution (PSD) [13], shape/morphology [11, 13] and flowability [14– 16]. In particular, oxidation in powder batches can occur in varying localized oxide shell thicknesses on a per-particle basis when reused [17]. These variations in surface chemistry in conjunction with changes in PSD from repeated reuse [13] presents a challenge when trying to fully understand the effects of powder reuse as it pertains to available powder surface area and powder bed packing density.

Traditionally, Ti is often used as a gettering media [18, 19] for inert gas purification [20]. One of the major contributing factors to Ti-6Al-4V powder reuse in the PBF process is its propensity to pickup oxygen at elevated temperatures [21]. Coincidentally, PBF-EB is carried out in a partial vacuum at elevated temperatures of 650 °C to 800 °C, whereas PBF-L is conducted at room temperature typically with Ar as a cover gas which can enable the titanium powder bed to purify both environments as well as scrub the cover gasses for any impurities. Traditional powder metallurgical techniques for titanium often require purification of the inert cover gas prior to introduction into a titanium processing environment, as described in a recent review of the powder metallurgical processing of titanium alloys [22]. However, no such purification/gettering mechanism is currently in place for the PBF technologies and production environments.

Recent work by the authors has shown that oxidation can preferentially occur at the beginning stages of the build in a PBF-EB process due to the high background processing temperatures [23], with the implications being that the beginning layers of the Ti-6Al-4V powder-bed purified the processing atmosphere of any residual moisture and oxygen. This build height oxygen variation effect in the powder was also found to exist in melted material, leading to significant variation in tensile properties [24]. The authors have also shown that current standard industrial powder mixing and reuse practices, which include frequently mixing powder with different oxygen content, leads to additional oxygen and tensile property variation [25]. Current approved quantitative chemical analysis of oxygen in AM PBF titanium alloys presents a challenging problem, such that accurate analysis of the oxygen content in titanium requires destructive testing of the entire sample, which is then used to infer the chemistry of the batch the material was sampled from. This leads to an averaging of the oxygen on a per-particle basis if measuring a batch of powder, such that it does not discriminate between individual particles. If testing bulk titanium, this same averaging occurs and it is impossible to determine potential chemical heterogeneity in a microstructure. Higher resolution methods of oxygen analysis, such as atom probe tomography for example, while incredibly accurate, requires such small sample sizes that it is difficult to make confident general statements about a macroscopic sample. It is important to investigate all possible sources of oxygen variation for PBF Ti-6Al-4V, which is why the present investigation seeks to understand the effect of particle size distribution (PSD) and extent of reuse on the rate of oxidation by means of thermogravimetric analysis (TGA).

# 2. Methods and Materials

Two Ti-6Al-4V powders were chosen for this work: Plasma atomized, virgin, Grade 5 powder conforming to ASTM F2924 [26] and a plasma atomized, 1× used Grade 23 powder conforming to ASTM F3001 [27]. The PSD for both powders was standard for PBF-EB and ranged from 45  $\mu$ m to 106  $\mu$ m as reported from the manufacturer via ASTM B214 [28]. The D10, D50, and D90 (as per ASTM B822 [29]) for the Grade 5 powder was determined to be 51 µm, 70µm, and 98 µm respectively. The D10, D50, and D90 for the Grade 23 powder was 52  $\mu$ m, 72  $\mu$ m, and 100  $\mu$ m respectively. A 75  $\mu$ m sieve was chosen to split the PSDs of both batches approximately in half, such that the following three test samples were obtained:  $45 \ \mu m - 75 \ \mu m$ , 75  $\mu m - 106 \mu m$ , and the full range of 45  $\mu m - 106 \mu m$ . The powders used in this experiment were collected in atmosphere and housed in plastic containers. The sieving also took place at atmosphere by pouring the powder into the sieve, shaking, and collecting the fines beneath, whereas the coarse particles were collected from top of the sieve. Chemistry of the virgin powders was determined as such: aluminum, vanadium, and iron were measured by optical emission spectroscopy (OES) per ASTM E2371-13 [30]. Oxygen and nitrogen were measured per ASTM E1409-13 [31], and hydrogen per ASTM E1447-09[32], all by inert gas fusion. Carbon was measured by the combustion method per ASTM E1941-10 [33]. The chemistry values can be found in Table 1. Note that the Grade 23 powder in this work was used once prior to the experiments in this work and may have slight variations in chemistry from the data presented in Table 1.

The powder morphology was characterized via secondary electron (SE) imaging using a field emission scanning electron microscope (SEM) with an accelerating voltage of 2 kV before the TGA testing. Microstructure of the powders prior to TGA testing was also analyzed via metallographic preparation, such that the powder was hot mounted in a conductive phenolic resin, abraded using SiC paper, and finally vibratory polished for 24 hours in 0.05 µm colloidal silica. The microstructure was imaged using a backscatter

Table 1: Chemical composition in weight % of the virgin Grade 5 and virgin Grade 23 (prior to its  $1 \times$  use) Ti powders as measured via inert-gas fusion. Tr. = trace elements. ASTM standard specification maximum limits (unless otherwise noted) for PBF Ti-6Al-4V Grade 23 (ASTM F3001 [27]) and Grade 5 (ASTM F2924 [26]) are listed with the measured values.

	Al	V	Fe	0	C	N	Η	Υ	Tr.	Tr. Max.	Ti
Grade 23	5.5 < x < 6.50	3.5 <x<4.5< td=""><td>0.25</td><td>0.13</td><td>0.08</td><td>0.05</td><td>0.012</td><td>0.005</td><td>0.10</td><td>0.40</td><td>bal.</td></x<4.5<>	0.25	0.13	0.08	0.05	0.012	0.005	0.10	0.40	bal.
This work	6.40	3.83	0.19	0.09	0.01	0.02	0.002	< 0.001	< 0.10	< 0.40	bal.
Grade 5	5.5 < x < 6.75	3.5 <x<4.5< td=""><td>0.30</td><td>0.20</td><td>0.08</td><td>0.05</td><td>0.015</td><td>0.005</td><td>0.10</td><td>0.40</td><td>bal.</td></x<4.5<>	0.30	0.20	0.08	0.05	0.015	0.005	0.10	0.40	bal.
This work	6.52	3.91	0.18	0.18	0.02	0.01	0.002	< 0.001	< 0.10	< 0.40	bal.

electron (BSE) detector with accelerating voltages up to 30 kV. Oxygen testing of the powders was carried out via inert gas fusion as per ASTM E1409 [31] before and after TGA.

The TGA measurements were performed up to 800 °C at a rate of 20 °C/min in an industrial grade Ar (purity = 99.995 %) environment with flow rate of 20 mL/min for the testing, and 20 mL/min for protection of the mass balance. The TGA furnace was purged to  $\approx 10^{-3}$  mbar and backfilled with Ar three times prior to each test. In between TGA runs, the powder samples sat in air at 22 °C and 11 % relative humidity. The TGA pans were 85 µL Al<sub>2</sub>O<sub>3</sub> pans filled approximately to the top, which resulted in approximately 100 mg of powder per test. The masses of the Al<sub>2</sub>O<sub>3</sub> TGA pans were measured prior to the addition of the powder. A single automated TGA sequence consisted of the three PSDs and both titanium grades were tested in triplicate, resulting in 18 TGA measurements for a given sequence. The TGA sequence data sets, and triplicate testing within the sequence itself (such that nine tests were used in the average values that are presented).

#### 3. Results and Discussion

The powder morphology and microstructure is presented in Fig. 1, where the virgin Grade 5 powder is shown in the full PSD range of 45  $\mu$ m to 106  $\mu$ m (Fig. 1a). Once the 75  $\mu$ m sieve was used, the powders were approximately split into the 45  $\mu$ m – 75  $\mu$ m range (Fig. 1b) and 75  $\mu$ m – 106  $\mu$ m range (Fig. 1c). The images of the singular particles in Fig. 1a–b are shown at the same magnification, and were acquired to pictorially demonstrate the approximate size difference between the two PSDs. The powder is generally spherical and contained artifacts from the atomization process, such that small satellite particles are adhering to the surface of the larger powder particles. The BSE images of the Grade 5 and Grade 23 powder microstructures pre-TGA is presented in Fig. 1d–e. The virgin Grade 5 powder microstructure , likely consists of mostly  $\alpha'$  martensite as previously observed in virgin powders [34]. This microstructure likely transformed to  $\alpha/\beta$  Widmänstatten during TGA up to 800 °C, based on the  $\alpha'$  decomposition ranges identified in Ref. [35].



Figure 1: SEM images of the Grade 5 powders used in this study: a) full PSD (45  $\mu$ m - 106  $\mu$ m), b) Particles from the sieved PSD range of 45  $\mu$ m - 75  $\mu$ m, c) Particles from the sieved PSD range of 75  $\mu$ m - 106  $\mu$ m, d) microstructure of the virgin Grade 5 powder prior to TGA, e) microstructure of the 1× used Grade 23 powder prior to TGA.

The grain morphology of the 1 × used Grade 23 consists of mostly  $\alpha/\beta$  Widmänstatten. Note, the white area around the particle perimeter is a brightness difference between the conductive resin and the mounted particle. Given that the Grade 23 powder had been used one time in PBF-EB in an industrial workflow, the  $\alpha/\beta$  Widmänstatten likely already existed due to the high background processing temperatures inherent to PBF-EB (up to 800 °C), which was previously observed in the reused powder study by the authors [36].

Representative TGA curves from the testing are shown in Fig. 2, with the axes representing temperature and mass percentage increase from the baseline measurements at room temperature. The initial bump in the mass percent up to 200 °C is likely an artifact due to buoyancy effects of the heated gas at the beginning stages of the TGA measurements. At 400 °C, the buoyancy effects appear to subside, as indicated by a drop in mass. Following this drop, a continuous mass gain is recorded up to 800 °C, similar to the non-isothermal TGA curves for commercially pure Ti (performed in air) in Ref. [37].

Commercially pure Ar is often insufficient to provide a fully inert atmosphere for titanium processing at elevated temperatures, and typically requires purification prior to use as a cover gas during powder metallurgy of titanium alloys [22]. The 99.995 % bottle purity used in the TGA testing for this experiment



Figure 2: Example/representative TGA curve of a triplicate data set starting from a baseline of mass at room temperature being 100%. Representative curves of Grade 23 Ti-6Al-4V tests powder shown.

can be assumed to contain 0.005 % air by volume (common assumption among gas vendors), and therefore approximately 0.001 %  $O_2$  by volume. Using the Ideal Gas Law, we can get an approximation of the moles of  $O_2$  exposed to the heated titanium from the Ar cover gas by first calculating the amount of molar volume  $O_2$  as a function of temperature:

$$V = \frac{1 \text{ mol of } \mathcal{O}_2 \times 0.082057 \frac{\text{L} \cdot \text{atm}}{\text{mol} \cdot \text{K}} \times T}{1 \text{ atm}}$$

Where T is the temperature in K. We can approximate the flow rate of  $O_2$  relative to the flow rate of Ar (20 mL/min) to be 0.42  $\mu$ L/min of  $O_2$  based on the volume percentages described earlier. At a heating rate of 20 °C/min, we can estimate that heating from room temperature to 800 °C approximately took place over 40 min. Therefore the moles of  $O_2$  as a function of temperature per given volume of 0.42  $\mu$ L of  $O_2$  supplied for 40 min is approximately  $3.4 \times 10^{-7}$  moles of  $O_2$ , and therefore  $\approx 5.4 \ \mu$ g of  $O_2$  exposed to the reactive Ti over a single test, coming directly from the Ar bottle.

The summarized results for the mass increase over the TGA tests are presented as bar charts in Fig. 3. The mass percent increases are higher than that strictly of oxidation coming directly from the Ar bottle. That is, the Ar cover gas was unable to completely rid the testing environment of all residual sources of oxygen/air. Given that these tests were repeated multiple times, the additional mass gain along with the Ar bottle contributions of  $O_2$  are not believed to be transient, but rather inherent to the setup for this particular experiment. From the bar charts, the PSD of 45  $\mu$ m – 75  $\mu$ m for the Grade 5 powder appeared to



Figure 3: Average measurable percent increases from each of the conditions (starting from 100%), averaged over 9 tests. Error bars represent  $\pm 1\sigma$ .

accumulate the most additional mass gain from the experiments compared to the other two PSDs (p value < 0.05). It is the opinion of the authors that the smaller PSD for the Grade 5 powder led to more surface area for the Ti-6Al-4V to react with any contaminants. The Grade 23 powder showed no statistical difference based on PSD differences alone. One explanation for why the Grade 23 powder does not appear to follow the same trend could be that the Grade 23 powder had been used once before in a PBF-EB process. When compared to the virgin Grade 5 powder, the Grade 23 powder could have previously built up a thicker oxide layer, and was therefore slower to accumulate additional TiO<sub>2</sub> relative to the virgin Grade 5 powder due to the parabolic nature of oxidation in Ti-6Al-4V [38].

Powder samples were also collected from one of the test sequences and sent for inert gas fusion oxygen analysis for verification of the presumed oxidation-based mass gain from the TGA testing. The chemistry analysis is also presented as bars in Fig. 4, where the diagonal textured bars represent the pre-TGA testing oxygen analysis (conducted over 9 tests on extra powder of the full PSD) whereas the solid bars represent the post-TGA triplicate tests for each PSD. The 45  $\mu$ m – 75  $\mu$ m Grade 5 powder, shaded blue, displays the highest oxygen content accumulation relative to the other PSDs likely due to the increased surface area of the smaller PSD powder. The tests resulted in significant oxygen concentrations that push the limits of the powder over standard specifications. When comparing the % increases of just oxygen relative to the total mass gain in the TGA tests, there is a small discrepancy such that the oxygen concentrations are not directly equal to the mass increases seen in TGA. This could be due to other reactants such as H<sub>2</sub>, N<sub>2</sub>, or H<sub>2</sub>O as water vapor in the powder leading to additional mass gain.



Figure 4: Average oxygen content for both grades of titanium and the three powder size distributions. Tests for the oxygen uptake are taken from one TGA sequence of testing, whereas the oxygen content of the powders pre-TGA were averaged over 9 tests. Error bars represent  $\pm 1\sigma$ .

#### 4. Conclusion

Thermogravimetric analysis was carried on industrially relevant PBF-EB Ti-6Al-4V powders up to 800 °C. It was observed that:

- Thermogravimetric analysis using industrial purity Ar gas is not sufficient to fully create an inert atmosphere that inhibits titanium's propensity to getter its environment at elevated temperatures.
- Smaller particle size distributions in virgin powder reacted with the processing atmosphere due to the larger surface area of the powder in the chamber. The larger particles also reacted, but to a lesser extent.
- The lower oxygen content containing powder that had been used once in PBF-EB (Grade 23) was slower to oxidize than the higher oxygen content, virgin (Grade 5) powder, likely due to the parabolic oxygen behavior previously observed in Ti-6Al-4V.

These results suggest that within a single powder bed there may be varying oxidation rates on powder particles based on particle size, packing density, and lifetime of a particular particle batch that has been subjected to many reuses. This suggests that both particle size and extent of powder reuse are contributing to oxygen variations in AM PBF Ti-6Al-4V.

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