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Effects of reuse on the properties of tantalum powders and tantalum parts additively manufactured by electron beam powder bed fusion

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Abstract

The cost of additively manufactured tantalum parts can be effectively lowered by reusing or recycling tantalum powder. To evaluate the effects of reuse of the tantalum powder on the properties of tantalum parts and scaffolds, this study investigated the characteristics of tantalum powder during cycling, including oxygen content, particle morphology, apparent density, tap density, and flowability. Besides, the influence of reuse time on the mechanical properties of electron beam powder bed fusion (EB-PBF) fabricated tantalum parts and scaffolds was studied with tantalum powder reused more than 30 cycles. The results indicated that particle size distribution of the tantalum powder was nearly unchanged with the increase of cycle number, accordingly apparent density, tap density, and fluidity. While the powder became less spherical with increasing reuse times and some particles showed noticeable distortion and rough surface after being reused 25 times. Moreover, the oxygen content of the tantalum powder increased progressively with increasing reuse times, leading to the decrease of plasticity of the dense tantalum after 15 reuse cycles, and some potential micro-defects appeared in the tantalum samples fabricated from EB-PBF process. However, the tensile strength of dense tantalum parts was not sensitive to the number of uses within the research range.

1. Introduction

Tantalum is known as a ‘biophilic’ metal and has many applications in high-temperature and biomeicine fields because of its high melting point (2996 °C), appreciable corrosion resistance, and good biocompatibility [1]. Tantalum has been used in orthopedic implants for more than 60 years [2]. However, the high cost of the raw materials and difficulty in processing [3] have limited its broad application.

In recent years, additive manufacturing (AM) has emerged as a technology that allows the fabrication of metal parts of complex geometry. It can be used to customize implants quickly and accurately according to the specific needs of the patients [4, 5]. It affords the advantages of a short operation time, small trauma area, high rate of success, and less pain. The additive manufacturing methods used for implant manufacturing mainly include laser powder bed fusion (L-PBF) and electron beam powder bed fusion (EB-PBF). The L-PBF technology has the key advantages of higher accuracy and larger forming size [6, 7]. Proceeding in a vacuum environment, EB-PBF technology has the advantages of higher energy density, energy utilization, and high production efficiency [8, 9], it is more suitable for manufacturing refractory metals and implant materials, like tantalum implants, which has attracted more and more attention.

At present, tantalum implants manufactured by the EB-PBF technology are still in the stage of clinical trials, and have not been commercialized. On the one hand, the period of Conformite Europeenne (CE), Food and Drug Administration (FDA), and National Medical Products Administration (NMPA) certification for orthopedic implants is invariably long. On the other hand, the cost of the raw materials used in the AM of...
tantalum implants is relatively high; spherical tantalum powder costs 1200–1500 USD per kilogram. Therefore, recycling the powder recovered during additive manufacturing is a common practice for reducing the cost. During the EB-PBF process, the temperature of the powder bed can be up to 1200 °C [10, 11], higher than that during L-PBF process (100 °C–500 °C) [12]. Thus, the residual stress of the manufactured parts decreased during the EB-PBF process [13, 14], the chemical composition and morphology of the reused tantalum powder may also change when exposed to a high temperature environment, thereby affecting the properties of the as-built parts. For example, in the case of the EB-PBF Ti-6Al-4V alloy, the oxygen content of the powder increases as the number of powder reuse cycles increases, leading to a decrease in the elongation of the alloy [15]. In addition, the electrical conductivity of the powder decreases because of the increased oxygen content, causing the ‘powder blowing’ phenomenon, which is detrimental to the formation of the part. Moreover, some scholars studied powder reuse on L-PBF fabricated alloys, such as nickel-based superalloy [16], stainless steel [17, 18], it was found that there was an increment in satellite powder, deformed powder and oxygen content, causing a high-density oxide inclusion in the fabricated parts. However, the effects of reuse on the properties of tantalum powder and tantalum parts fabricated by EB-PBF remain unclear. The correlational research is significant for the quality-control and cost-control of EB-PBF industry-scale production of tantalum implants.

This work studied the effects of reuse on tantalum powder characteristics during EB-PBF recycling, including the oxygen content, particle size, flowability, apparent density, tap density, and morphology. Further, the compressive and tensile properties of the tantalum parts manufactured from the recycled powder via EB-PBF were also investigated.

2. Materials and methods

2.1. Original Ta powder

Spherical Ta powder was produced by plasma rotating electrode process in Xi’an Sailong Metal Materials Co. Ltd. The composition of the purchased tantalum powder is shown in Table 1, which meets the international standard of ISO 13782: 2019. All the dense and porous Ta samples were manufactured using a SAILONG-Y150 EB-PBF system. The initial fabrication was started with 50 kg of the Ta powder.

A substrate with the dimension of 120 mm × 120 mm × 10 mm, which was preheated to 700 °C by electron beam scanning, was used. Preheating was carried out after each layer was printed, and also after the powder was laid. Throughout the AM process, the powder bed was maintained at a temperature not lower than 660 °C. Thereafter, the unmelted powder was blown off the fabricated parts and/or samples in the powder removal system (PRS) using compressed air. The powder residue left in the entire system was blended in the PRS and sieved through a 100 mesh (140 μm) sieve for the next cycle of AM.
2.2. AM parameters

Table 2 summarizes the parameters used in the EB-PBF process. They were kept unchanged in each operation to better understand the effects of the number of powder reuse cycles on the characteristics of the Ta powder and the mechanical properties of the dense tantalum and porous tantalum parts fabricated by EB-PBF. The EB-PBF process with details are shown in figure 1.

The dense samples were prepared with the dimensions of $60 \text{ mm} \times 10 \text{ mm} \times 10 \text{ mm}$, while the porous tantalum samples was prepared with the dimensions of $10 \text{ mm} \times 10 \text{ mm} \times 15 \text{ mm}$ (figure 2(b)). A dodecahedron was used as the unit cell of the microarchitecture of the porous structure (figure 2(a)), similar to that of the human cancellous bone. The size of the dodecahedron unit cell was set as $2.5 \text{ mm}$, and the average strut size and pore size of the porous structure were set as $180$ and $700 \mu \text{m}$, respectively, which results in an overall open porosity of $96.1\%$.

Table 2. Parameters used in the EB-PBF process.

| Variate                        | Value  |
|--------------------------------|--------|
| Filament voltage, kV           | 60     |
| Spot diameter of electron beam, $\mu \text{m}$ | 190    |
| Layer thickness, $\mu \text{m}$ | 50     |
| Preheating temperature, $^\circ \text{C}$ | 700    |
| Beam current for preheating, mA | 20     |
| Scanning speed for preheating, m/s | 10     |
| Beam current for melting, mA    | 11     |
| Scanning speed for melting, m/s | 0.7    |
| Beam current for contour, mA    | 22     |
| Scanning speed for contour, m/s | 0.55   |

2.3. Powder characterization

The size distribution of the powder particles was analyzed using a Malvern laser diffraction analyzer (Malvern MS3000, Worcestershire, UK) following ISO 13320: 2009. The flowability (sample quantity, 50 g), apparent density, and tap density of the reused powder were determined following ISO 4490: 2001, ISO 3923: 2018, and ISO 3953: 2011, respectively. The determination of oxygen content was infrared detection method, Mo, W and Si elements were analyzed by inductively coupled plasma atomic emission. The content of Ni was determined by DC arc atomic emission spectrometer. The content of C, H, N, Fe were determined by high frequency infrared carbon and sulfur analyzer, impulse heating-thermal conductivity tester and inert gas fusion thermal conductivity method, phenanthroline spectrophotometry, respectively. The morphology of the powder was observed by scanning electron microscopy (SEM; JSM-6360LV, Japan). To ensure the analysis of the powder following as many cycles as possible, this study focused on characterizing the powder after 0, 5, 10, 15, 20, 25, and 30 cycles. Each build cycle is continuous, and the time of powder exposed to the air is about two hours for taking out the parts from build chamber and preparing next EB-PBF process. All the collected tantalum powder was mixed and sifted twice after the EB-PBF process, and the screened powder was used in the tests. A sufficient
amount of the powder sample (240 g) was collected after each cycle for testing. Three repeated experiments of flowability, apparent density, and tap density of the reused powder were conducted under the same conditions, to avoid accident errors.

2.4. As-built sample testing

The defects and microstructures of the fabricated dense tantalum sample were analyzed by optical microscopy (OM, Germany Leica). Specimens for OM were etched using a mixed solution of 10 ml HF, 10 ml HNO₃, and 30 ml H₂SO₄. The microstructure of the reused powder and the manufactured tantalum lattice structures was studied by SEM. Before SEM observations, the Ta lattice structures were cleaned ultrasonically for three times. The actual density of the lattice structure was determined by measuring the physical dimensions and mass of each lattice, and the porosity was calculated as 1 - d, where d is the density of the porous tantalum scaffold. The density of the EB-PBF-built dense tantalum was determined by the Archimedes method. X-ray diffraction (XRD) system was an X'Pert PRO machine. The dense specimens used for tensile testing were prepared according to standard ASTM E8M, as showing in figure 3, and compression testing of the porous tantalum followed ISO 13314. Three repeated tests were conducted under the same conditions to minimize errors.
3. Results

3.1. Reused tantalum powder

Figure 4 shows the variation in the oxygen content of the tantalum powder with the number of reuse cycles. Overall, the oxygen content of the powder shows an increasing trend; it increases progressively from 0.004 wt.% for the original powder to 0.018 wt.% after 30 times of reuse.

Figure 5 shows the particle size distribution of the tantalum powder after 0, 5, 10, 15, 20, 25, and 30 reuse cycles. The D10, D50, and D90 of the original tantalum powder were found to be 60, 98.4, and 158 μm, respectively; they increased slightly to 65.9 μm, 105 μm, and 166 μm, respectively, after 30 reuse cycles. Overall, the particle size of the reused tantalum powder did not change significantly as compared to that of the original powder.

Figure 6 presents apparent density, tap density (a) and the fluidity (b) of the tantalum powder after different reuse cycles. Figure 6(a) reveals that there was no obvious change in the apparent density and tap density between 0 and 30 reuse cycles. The apparent density and tap density of the original tantalum powder were measured to be 10.67 and 11.48 g cm⁻³, respectively, and they hardly changed after 30 times of reuse. Further, the fluidity was found to be 7.8 s for the original powder (sample weight: 50 g) and 8.0 s for that reused 30 times. The powder fluidity hardly varied with increasing reuse cycles, and the sample maintained its excellent fluidity even after 30 reuse cycles.

Figure 7 shows the microscopic features of the tantalum powder after different reuse cycles. The powders maintained their spherical morphology and had a smooth surface up to 20 reuse cycles. However, a small distortion in particle shape was noted with further reuse, as observed in the low-magnification views in figures 7(f), (f1) and figures 7(g), (g1). The surfaces of some particles had turned rough after 25 times of reuse, as deduced by comparing the particle morphology in figures 7(a), (a1) (virgin powder) with that in figures 7(f), (f1).
The fusion of some large particles with smaller ones was observed after 30 times of reuse (figures 7, g1).

3.2. Dense tantalum samples fabricated from reused powder by EB-PBF

Figure 8 shows the effect of reuse of the tantalum powder on the relative density of tantalum samples fabricated by EB-PBF. The relative density of the tantalum part was calculated in relation to the theoretical density of 16.68 g cm$^{-3}$ of pure tantalum. As shown in figure 8, the relative density of the EB-PBF-manufactured pure tantalum samples is significantly affected by the number of reuse cycles.
Figure 8. Relative density of tantalum samples fabricated by EB-PBF from reused powder.

Figure 9. OM image of a dense tantalum part fabricated by EB-PBF after 5 reuse times.

Figure 10. XRD patterns of the EB-PBF tantalum parts from reused powder.
tantalum parts decreased gradually from 99.5 to 98.4% after 30 times of reuse. Figure 9 shows the OM image of a dense tantalum part fabricated by EB-PBF from a powder reused 5 times. Although there were no microcracks, some defects (pores) were observed in this sample having a density of 99.4%. Columnar grain structures were formed along the building direction (BD), and the width of the columnar grains was in the range of 50–100 μm.

Figure 11. Tensile properties of tantalum samples fabricated by EB-PBF from reused powder.

Figure 12. Fractured surface morphology with low ((a), (c)) and high ((b), (d)) magnification for EB-PBF tantalum: fabricated from powder reused for: ((a), (b)) 5; ((c), (d)) 30 reuse times.
The XRD patterns of the EB-PBF fabricated dense tantalum from reused powder are shown in figure 10. The manufactured Ta parts all show (110), (200), (211) and (220) peaks of tantalum, and the position and intensity of perks of the manufactured Ta parts are almost unchanged from Ta powder with different reuse times.

The tensile properties of the tantalum samples fabricated by EB-PBF using powder reused for 5, 10, 15, 20, 25, and 30 cycles are shown in figure 11. No significant change in the yield strength and ultimate tensile strength was observed after 5, 10, 15, 20, 25, and 30 times of reusing the tantalum powder. However, the elongation of the sample decreased with increasing number of reuse cycles. When the tantalum powder was reused 5 times, the elongation of the EB-PBF-manufactured tantalum was 49.3%, and it declined to 32.2% when the tantalum powder was reused 30 times. The SEM images of the fractured surfaces of the tantalum parts fabricated from powder reused for 5 and 30 times are displayed in figure 12. The high-magnification SEM image in figure 12(b)
shows many different-sized dimples on the sample surface; the depth of the dimples decreased with increased reuse (figure 12(d)).

3.3. Porous tantalum samples fabricated from reused powder by EB-PBF
The surface morphologies of the porous tantalum samples manufactured using powder from different reuse cycles by EB-PBF are shown in figure 13. As shown in figure 13(a)–(c), the porous tantalum scaffolds exhibit good formability and uniform struts without surface undulations. The average diameter of the porous tantalum scaffolds was measured to be 400 μm, and the struts were coarser than the desired diameter (180 μm). When the tantalum powder was reused more than 20 times, some strut defects were observed. In addition, a balling phenomenon was observed in the tantalum struts after 30 reuse cycles.

Figure 14 presents the measured porosity of the porous tantalum sample manufactured using tantalum powder from different reuse cycles. The porosity was found to be ~83.5%, and it hardly changed with the number of reuse cycles.

Figure 15 shows the effect of reuse of the tantalum powder on the compression performance of the EB-PBF-manufactured porous tantalum sample. As shown, both the compressive yield strength and the platform stress of the porous tantalum decreased by ~20% after 20 reuse cycles. Large deviations were observed in the measured compressive yield strength and platform stress of the porous tantalum samples fabricated from a powder reused 20 times.
4. Discussion

4.1. Effect of reuse on the powder characteristics

The oxygen content of the tantalum powder increased with an increase in the number of reuse cycles. The increase in the oxygen content mainly occurred when the powder was exposed to air during the leveling of the substrate before the EB-PBF process, and the subsequent powder recycling process. According to the data in figure 4, the oxygen content of the tantalum powder increased by 4.7 ppm per EB-PBF process over the 30 reuse cycles. The oxygen content increased slowly because the tantalum particles were heated and melted with an electron beam in a high-vacuum environment during the EB-PBF process.

Figure 5 indicates no significant change in the average particle size after 30 times of reuse. The small change in the particle size with increasing number of reuse cycles can be attributed to several factors: (i) the layer thickness (50 μm) was small, and the coarse powder was scraped with a scraper; (ii) fine-grained powder particles were preferentially melted, which decreased the number of small particles; and (iii) splashing, adhesion, and remelting increased the number of larger particles. Thus, the particle size increased only slightly. As the large particles were screened out, and the adhered particles of the powder were separated, the average particle size decreased after reuse. During the entire EB-PBF process, the changes in the D10, D50, and D90 of the tantalum powder followed the same trend, and hence the particle size distribution hardly changed and the original particle distribution was maintained.

Before the EB-PBF process, an electron beam was used to preheat the substrate to a temperature of 700 °C. Then, the powder bed was maintained at a temperature above 660 °C during the entire EB-PBF process. Therefore, slight surface roughening and distortion of the tantalum particles were expected, particularly during the lengthy EB-PBF process. In addition, some particles were located in the vicinity of the building parts heated to a high temperature during the EB-PBF process; this could lead to noticeable distortion and surface roughening of the particles. The high-pressure air jets induced concave sites on the powder particle surface during the recovery process. Some of these features are observed in figure 7, in particular, after 25 reuse cycles, defects appeared later than in the Ti-6Al-4V powder [15, 19]. The number of surface defects is expected to increase with increasing powder reuse cycles, resulting in an increase in the surface roughness and particle distortion.

4.2. Effect of reuse on the mechanical properties of dense parts and scaffolds

From the XRD pattern of the EB-PBF fabricated tantalum, the position and intensity of perks of the tantalum parts manufactured by tantalum powder with different reuse times are almost unchanged. There is no new phase appeared with increasing reuse times of the tantalum powder, as the oxygen content was too little to induce significant oxygenic phases detected by XRD. As particles with an oxide layer on the surface have poor wettability with the matrix, they cannot be evenly spread on the substrate, and balls and defects tend to form [20]. Some defects were observed in dense tantalum samples fabricated from a powder reused 5 times (figure 9); further, the defects increased as the number of reuse recycles increased. The density (figure 8) and elongation (figure 11) of the EB-PBF-manufactured dense tantalum sample decreased gradually with increasing reuse cycles, mainly due to the increase in the oxygen content. The change in the elongation is consistent with the fracture morphology of the tensile samples; the number of dimples in the tantalum samples fabricated from the powder reused 30 times as well as that used 5 times indicates excellent ductility. The depth of the dimples decreased with increasing reuse cycles, and the elongation decreased significantly (figure 12). Tang et al [21] reported that the elongation of tantalum samples prepared from a powder with a low oxygen content of 90 ppm (0.009 wt.%) was 46 ± 1%, while Zhou et al [22] reported that the elongation of tantalum samples manufactured from a tantalum powder with a high oxygen content (1800 ppm) was only 2%. The variation in the elongation is consistent with the morphology of the fracture surface. When the number of reuse cycles was small, large numbers of large and small dimples were observed on the fracture surface, and the number of dimples decreased with increasing reuse cycles (figure 12).

The measured average diameter of the porous tantalum struts (400 μm) was larger than the desired diameter (180 μm). This is because of the high melting point of the tantalum powder, which requires a high energy input. Thus, the spot diameter of the electron beam is larger than the theoretical value (190 μm), and therefore, the strut diameter is larger. The compression property of the EB-PBF-manufactured porous tantalum sample degraded with increasing reuse cycles, owing to the increase in the number of defects, that is, the balling phenomenon. The oxide layer formed on the particle surface decreases the surface wettability between the tantalum powder and matrix, as reported previously by Good et al [23] and Lei et al [24]. Consequently, the powder does not spread evenly on the substrate, and balls tend to form [20]. As mentioned before, as the oxygen content increased, some defects were observed in the EB-PBF-manufactured tantalum, especially in the case of the porous tantalum strut. Large blocks are less sensitive to defects than smaller ones, and therefore, the tensile...
strength of the former hardly changes with increasing oxygen content. Porous tantalum is composed of periodically arranged porous struts, and the struts are fine and sensitive to defects. When defects form at the strut intersection and in the strut, its performance is greatly affected. Therefore, the compression property decreased as the oxygen content and defects increased in the samples, and a larger deviation in compression performance was observed for porous tantalum fabricated from a powder reused for 20 times.

In summary, the most distinct effect of tantalum powder recycling is the increase of oxygen content, which thereby induced the drop of plasticity and formation of some defects in the EB-PBF manufactured tantalum parts. Therefore, it is necessary to shorten the exposure time of the tantalum powder in the air between the build cycles, and strictly control the humidity and temperature when the powder is exposed in the air. In the next stage, the effects of powder reuse on the biological properties and osteointegration ability of tantalum parts additively manufactured by EB-PBF need to be studied.

5. Conclusion

The following conclusions can be drawn from this study.

1. The oxygen content of the tantalum powder increased from 0.004 to 0.018 wt. % after 30 times of reuse in the EB-PBF process. The fluidity, particle size, apparent density, and tap density of the tantalum powder did not change significantly after multiple cycles of reuse.

2. The tantalum powder particles were spherical and had a smooth surface after 20 times of reuse. A small distortion in particle shape was observed after more than 25 times of reuse, and the fusion of large and small particles occurred during the EB-PBF process.

3. The yield strength and ultimate tensile strength of the tantalum samples did not change significantly, but the elongation decreased from 49.3 to 32% with increasing powder reuse cycles. The compression property of porous tantalum decreased significantly after 20 times of reuse, and a large deviation was observed in the measured values. Further, the balling phenomenon was observed in the struts of the tantalum lattice structure.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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