

LASER SCANNING STRATEGIES TO INVESTIGATE THE EFFECTS OF OXIDATION IN PURE TANTALUM

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Abstract

Additive manufacturing of tantalum has exhibited issues with the final mechanical properties of the material. This paper suggests that this is due to the oxidation proclivity of tantalum powder before printing, and that the oxygen content of the material can be managed through refinement of laser scanning strategies. It was found that the tantalum powder showed evidence of oxidation before printing, and that different scanning strategies resulted in clear porosity changes and significant hardness variations.

Keywords: tantalum, powder bed fusion, oxidation, scanning

1. Introduction

Tantalum is well-known for its ability to maintain structural integrity at high temperatures, up to 3000°C [1]. This is a desirable property in aerospace applications, where rocket components and hypersonic airfoils can reach these temperatures easily [2].

Table 1: Standard pure Tantalum properties [1], [3]:

Tensile Strength, UTS (MPa)	276
Hardness (HV)	60-100
%elongation	50
Thermal conductivity (W/m)	83.7 (at 2500K)
Melting point (K)	3269
Boiling point (K)	5731

The aerospace community also makes use of additive manufacturing for research and development of new technologies. PBF-LB/M (laser powder bed fusion) is the preferred method for this type of manufacturing due to its efficiency and excellent finish [4]. This is incredibly useful for a high-value material like Tantalum, where wastage is minimal and the leftover powder can simply be reused for another part.

A study on the properties of PBF-LB/Manufactured Tantalum has determined that the microstructure tailoring of Tantalum is critical for this process [5]. It is highlighted that Tantalum produced through PBF-LB/M is not as strong or ductile as it should be (according to the properties given in Table 1). A

method is suggested for combating this through control of the microstructures.

Moreover, another study on the properties of printed Tantalum suggests that the difference between the material's properties and those of wrought Tantalum is due to the amount of oxygen present in the build chamber during print [6].

Tantalum, however, oxidises very easily at low temperatures and forms a layer of Tantalum Pentoxide (Ta_2O_5) [7]. This paper suggests that a standard PBF-LB/M process does not address the effects of oxygen in pure tantalum powder before printing, and explores whether the oxygen content can be mitigated through scanning strategies. This effect is further investigated through hardness and porosity analysis of the final samples.

Ta_2O_5 forms as a passivating layer on the surface of tantalum below 500K when the metal is at equilibrium. This can occur in environments even when oxygen content is minimal [7].

Tantalum Pentoxide (Ta_2O_5) has many good mechanical properties, including hardness and electrical insulation properties [3], [8].

Ta_2O_5 is used extensively for its ability to increase the hardness and corrosion resistance of other materials [9]. By adding a layer of Tantalum Pentoxide to another material, its hardness increases significantly, its elastic modulus decreases (making it more brittle), and its strength can increase [8], [9].

The melting temperature of Ta_2O_5 is approximately 2060K, with vaporization temperature exceeding 2100K in vacuum conditions [8] and the density is much lower than that of pure tantalum, at 8.2 g/cm^3 [8], [10].

Table 2: Research showing the effects of adding Ta_2O_5 to other materials

	Before addition of Ta_2O_5		After addition of Ta_2O_5	
Material	Hardness (HV)	Young's Modulus (GPa)	Hardness (HV)	Young's Modulus (GPa)
SS316 (thin film) [11], [12]	204	190	846	108
$La_2Ce_2O_7$ (doped) [13]	245	27	316	34

From the previous researchers' data, it is clear that Ta_2O_5 causes an increase in hardness that is useful in some cases. It can be a concern, however, that an increase in hardness results in a trade-off with ductility, making the material more brittle [14]. This could be an issue for tantalum where the expected high percent-elongation (~50%) is compromised due to the formation of Ta_2O_5.

A preliminary simulation of the PBF-LB/M process was run on ThermoCalc, with assumed parameters as follows:

Table 3: Parameters used in ThermoCalc simulation of PBF-LB/Manufacturing of tantalum

Laser speed	600 mm/s
Power	195 W
Layer height	0.03 mm
Assumed baseplate temperature	300 K
Laser diameter	0.08 mm
Powder density	80%
Gas pressure	1000 kPa
Beam quality factor	1

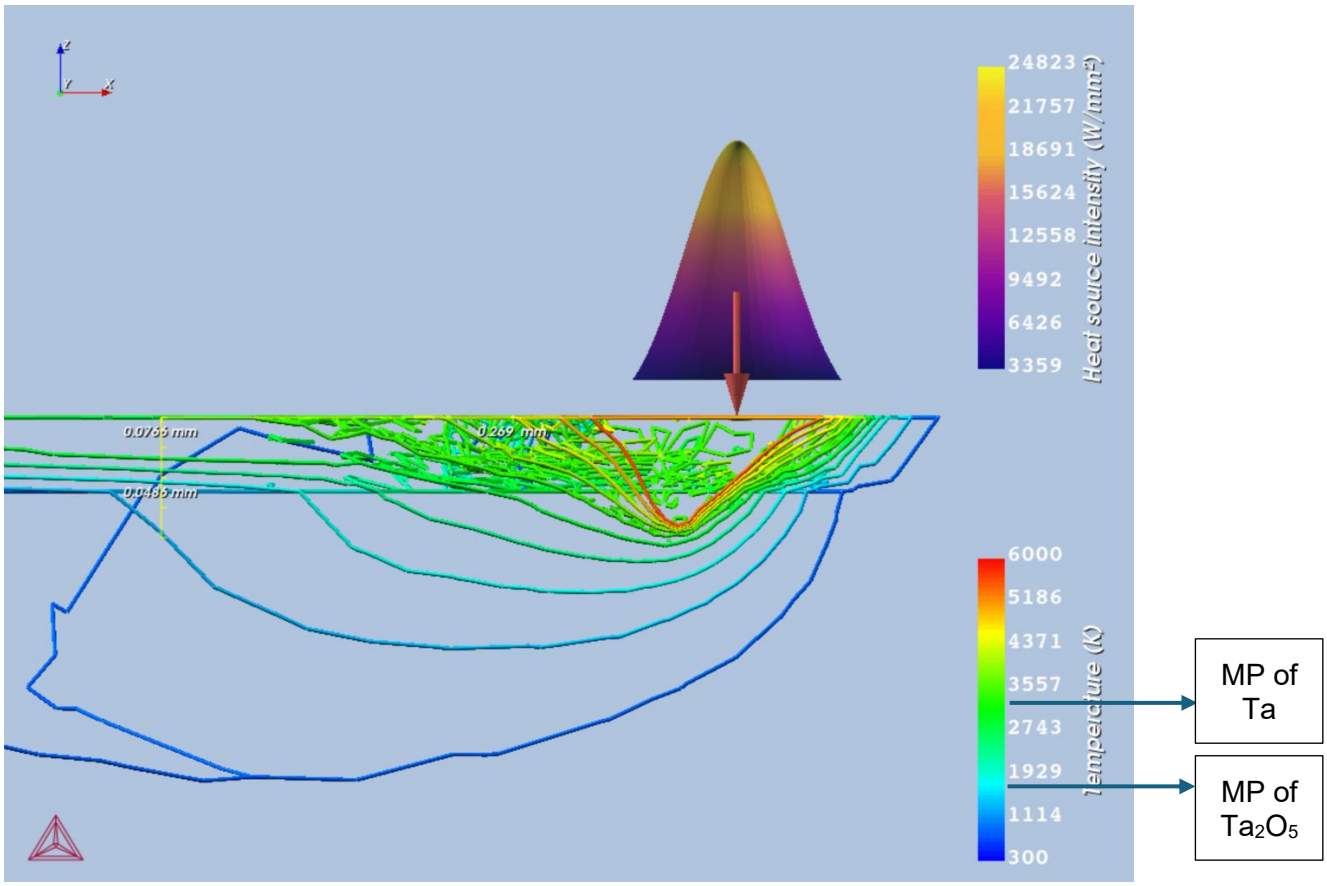


Figure 1: AM simulation of Ta temperature during print showing the anticipated temperature experienced by the material at steady-state conditions

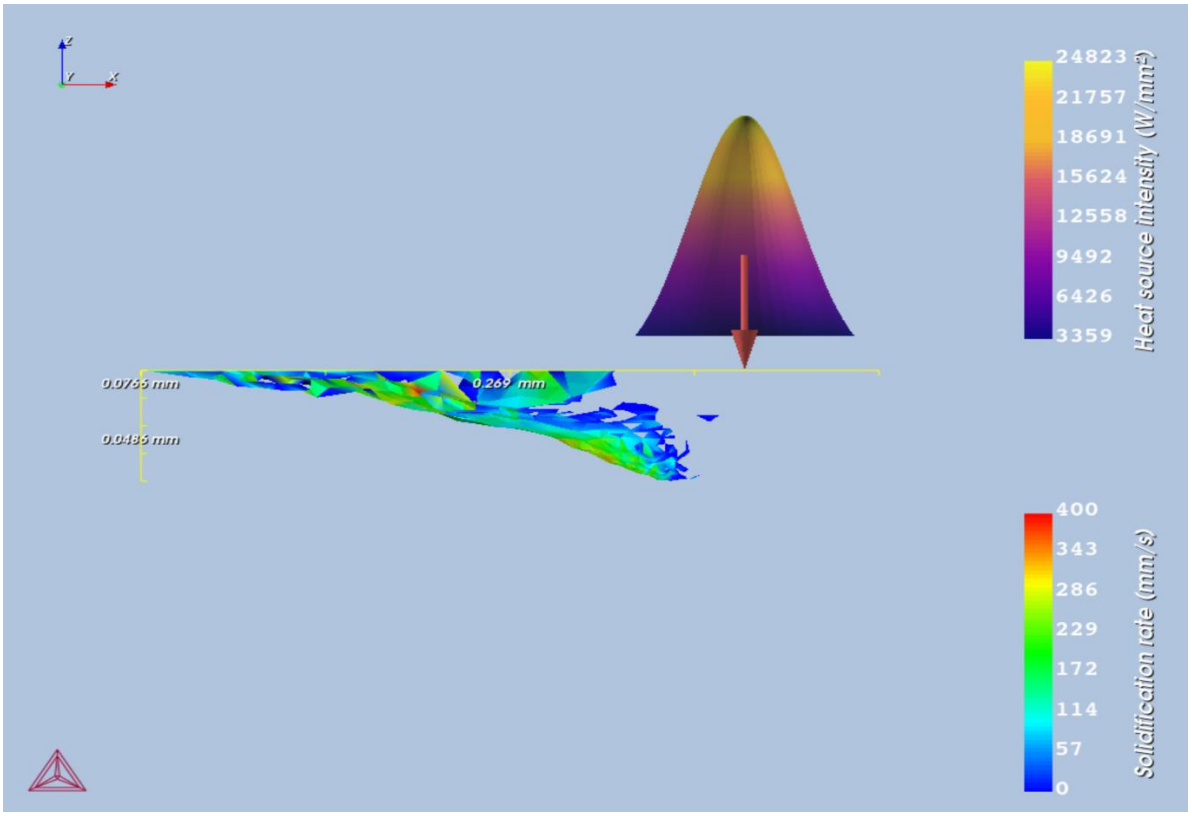


Figure 2: AM simulation of Ta solidification rate during print at steady-state conditions

From a preliminary AM calculation on ThermoCalc, it is clear that the material undergoes extreme temperatures (6000K) in order to melt and fuse. This is good for pure Tantalum but not for Ta_2O_5, for which the vaporization temperature is 2400K. Tantalum has a very high thermal conductivity and so it will solidify quite quickly after reaching its melting and boiling points (Figure 2). While the simulation indicates that the tantalum may vaporize during printing at 6000K, the rapid solidification rate will likely mitigate these effects. These temperatures do, however, make it highly likely that any Tantalum Pentoxide present in the material will be vaporised during printing. Vaporisation is a key factor that can cause keyholing defects or porosity in the final build of a material [15]. It may also be possible that the Tantalum Pentoxide dissolves into the final build, and this must be investigated.

2. Purpose

Current PBF-LB/M techniques for tantalum do not address the issue of oxygen (and by consequence, Ta_2O_5) being present during printing. The purpose of this research is to determine whether oxidation plays a role during a tantalum PBF-LB/M build procedure, what the potential effects will be for such a component, and whether an optimised scanning strategy will mitigate the effects.

3. Objectives

a. Determine whether an oxide layer affects the printing process of pure Tantalum.

b. Establish potential effects of oxidation using microscopy and microhardness tests.

c. Determine whether scan strategies, with the intent of mitigating oxidation effects, will affect the properties of PBF-LB/Manufactured Tantalum.

4. Materials and Methods

4.1 Print samples

Four cylindrical (10 mm) samples were printed using differing print strategies, A and B, and analysed. They were produced using PBF-LB/M at similar energy inputs, but varying scan patterns, shown in Table 3. Samples will be referred to as 1A, 1B, 2A and 2B dependent on the scanning strategy used and the Volumetric Energy Density applied. The oxygen content in the build chamber was kept to below 100ppm.

Table 3: Scanning strategies and VED applied to each sample

	Strategy A	Strategy B
Method	Single strip exposure, with defined rotation	Double strip exposure with defined maximum vector length, with defined rotation
VED 1 (J/mm^3)	181	181
VED 2 (J/mm^3)	211	211

Each sample was printed at a speed of 600mm/s and 190W of power, to match the inputs used for the ThermoCalc simulation.

It should be noted that strategy B applied a higher power wattage in the initial scan, and then a wattage at 80% of the first scan for the second. The VED in the second scan was reduced to a range of 70-85 J/mm^3.

4.2 Analyse for oxygen content and inspect surfaces under microscope

Both the cross-sections and top surfaces of the samples were analysed for porosity after printing.

4.3 Test microhardness on surface of sample vs internal cross-section

Vickers microhardness was tested to determine whether the print strategies had any effect on the internal structures of the samples. This is also to determine if the samples were harder than pure Tantalum is meant to be, potentially signifying the effects of oxidation.

5. Results and discussion

5.1 Measured oxygen content

The oxygen content of each stage of the process was measured through combustion analysis.

Table 4: Oxygen content in parts per million of tantalum

Specification of powder	90 ppm
Virgin powder	49.1 ppm
Sieved powder (reused)	45.4 ppm
Printed material	8 ppm

The data shows that the powder initially used in printing has a good oxygen content according to the specification.

Furthermore, the oxygen content is clearly reduced when the powder is exposed to heat, shown in the results of the used powder (which was present in the build chamber during a different print).

Finally, the printed material shows a significant reduction in oxygen content, supporting the assumption that the oxygen (i.e. a factor inducing Ta_2O_5 and therefore, higher hardness and lower density) can be mitigated through the PBF-LB/M process. Many prints, however, have shown evidence of high porosity and reduced mechanical properties.

5.2 Microhardness

Table 5: Vickers hardness tests on cross-section and top surfaces for each scanning strategy

	Strategy A		Strategy B	
	Top	CS	Top	CS
Mean (HV)	158	175	149	143
Standard deviation (HV)	16.2	17.6	31.6	26.7
Minimum hardness (HV)	112	138	95.6	97.4
Maximum Hardness (HV)	176	202	190	186

An evaluation on the data was also done to determine whether there was a significant difference between the two scanning strategies along the top and cross-sectional surfaces.

Table 6: t-Test for scanning strategies A and B on top surface

Alpha	0.05
T-Statistical	0.801
T-Critical (one-tailed)	1.71
T-Critical (two-tailed)	2.07

Table 6: t-Test for scanning strategies A and B on cross-sectional surface

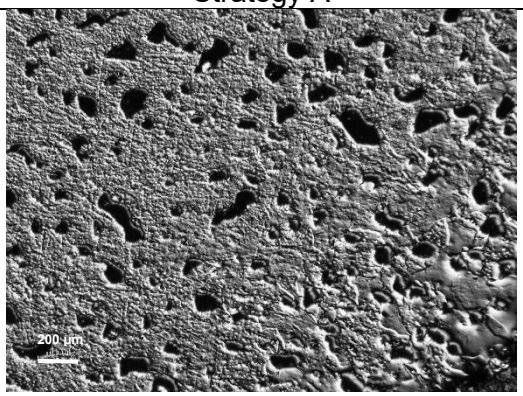
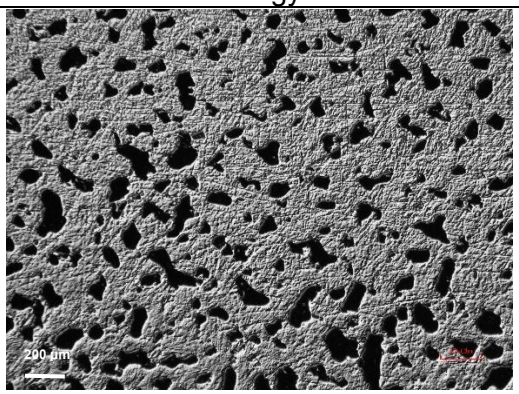
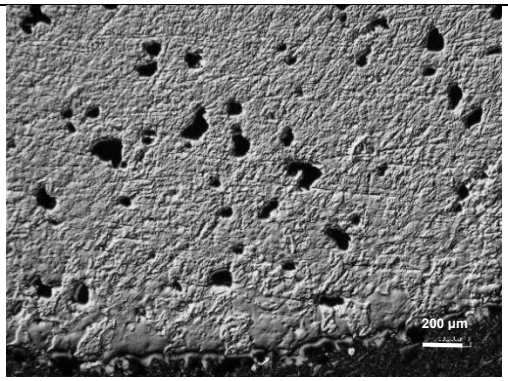
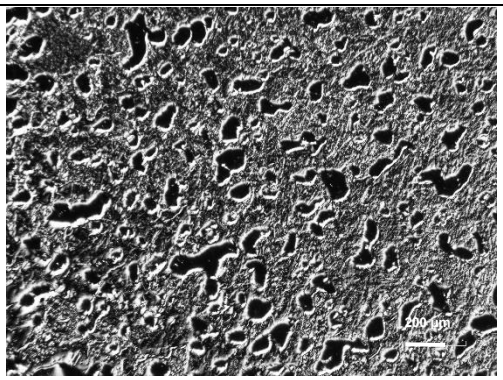
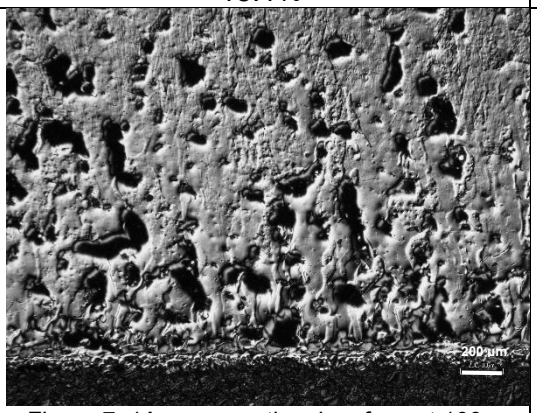
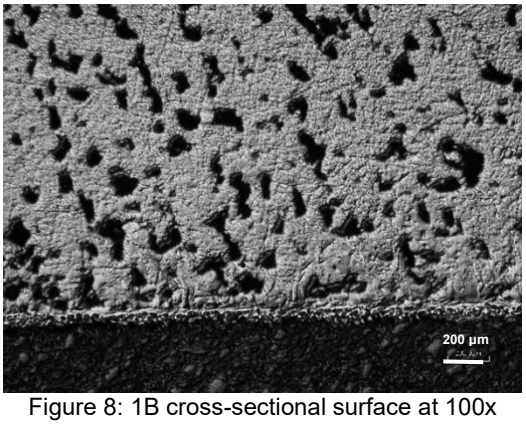
Alpha	0.05
T-Statistical	3.53
T-Critical (one-tailed)	1.71
T-Critical (two-tailed)	2.06

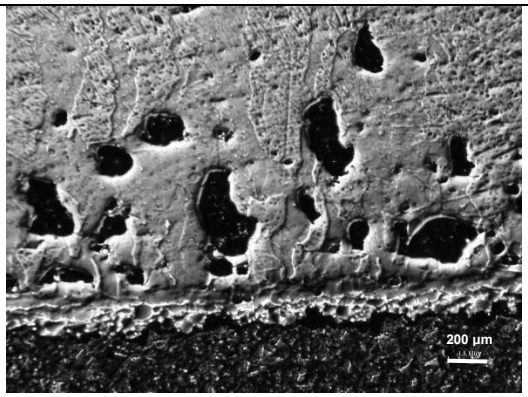
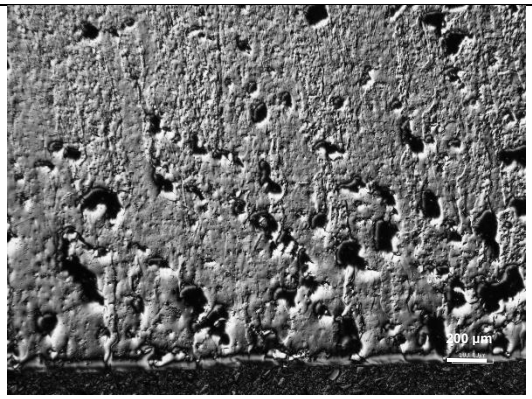
After analysis of the data, it was found that the top surface values do not have a significant difference in the populations (T-stat<T-crit). This means that the top surface hardness values do not differ much across the scanning strategies, which could be attributed to the formation of the Tantalum Pentoxide layer that is known to form on pure tantalum at its equilibrium state, and below 500K.

The cross-sectional surfaces, however, show a large statistical difference (T-stat>T-crit). This means that there is a significant difference in the two tests in terms of hardness values, giving the scanning strategies major influence on the internal properties of the material. This could be due to the vaporization of Ta_2O_5 in the material, as hypothesized, but could also be due to a difference in the melt pool geometry.

5.3 Microscopy images

Table 7: Comparison of microscopy images and porosity markers for scanning strategies A and B

	Strategy A	Strategy B
Top	 Figure 3: 1A top surface at 100x magnification	 Figure 4: 1B top surface at 100x magnification
	%area covered by porosity markers = 26.7%	%area covered by porosity markers = 25.04%
	 Figure 5: 2A top surface at 100x magnification	 Figure 6: 2B top surface at 100x magnification
	%area covered by porosity markers = 18.1%	%area covered by porosity markers = 23.1%
Side	 Figure 7: 1A cross-sectional surface at 100x magnification	 Figure 8: 1B cross-sectional surface at 100x magnification
	%area covered by porosity markers = 44.5%	%area covered by porosity markers = 21.0%

	 Figure 9: 2A cross-sectional surface at 100x magnification	 Figure 10: 2B cross-sectional surface at 100x magnification
	%area covered by porosity markers = 19.5%	%area covered by porosity markers = 19.5%

The images show that scanning strategy A is much less consistent in results than strategy B. B has a more reliable outcome, with no significant change in the porosity markers due to the variance in energy input (VED and double exposure strategy).

A shows some improvement in the porosity indicators when exposed to a higher energy density (2A at 211J/mm3).

6. Conclusion and recommendations

The effect of oxygen content and oxidation on the properties of additively manufactured Tantalum was investigated through scanning strategies. It was found that there was a reasonable oxygen content already present in the pure tantalum powder, but it must be considered that tantalum oxidises easily. It was found that exposing the powder to heat did reduce the oxygen content, and so the scanning strategies were developed to test whether a double laser exposure would make a difference in the part properties.

Two scan strategies were investigated, A (single strip) and B (double exposure), and were assessed for differences in hardness and visible structural differences. It was evident that strategy 2A resulted in fewer pores than B, as it was likely not vaporising as much powder. This, however, made it much harder than B, as the presence of Tantalum Pentoxide is known to do in metals. Further elemental analysis must be done to confirm whether Ta_2O_5 was the cause of this.

The printed material also still showed some oxygen content (8 ppm). A further strategy may be developed to mitigate this, that incorporates the better density seen in strategy A. However, a better strategy may be to expose the powder to a low energy density first before the high fusion exposure step. This may eliminate most of the oxygen content before the pure tantalum is melted. The softer nature of strategy B, however, may indicate a higher purity of tantalum within the structure, which must be further evaluated through elemental analysis.

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