HOT ISOSTATIC PRESSURE STUDY OF MICROSTRUCTURE ON ELECTRON BEAM MELTIN TUNGSTEN

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Abstract

Tungsten has a wide range of uses thanks to its high melting point and hardness. Often in literature, it is rare to find data about crack-free tungsten specimens and their manufacturing process. This project aims to study the effects on the microstructure of crack-free tungsten manufactured with Electron Beam Melting (EBM) after going through three different High Isostatic Pressure (HIP) cycles, performed at constant pressure and different temperatures. This evaluation was performed in terms of how grain size and defect content are affected when being exposed to these three different HIP cycles. Furthermore, all specimens were manufactured using the same parameters and in the same batch. Upon optical microscopy, some specimens showed a higher amount of defect formation which may be attributed to temperature variation between specimen formation, leading to unsuccessful melting of some tungsten particles. A comparative analysis of the micrographs obtained is performed to determine the effect of the High Isostatic Pressure.

1. Introduction

Tungsten has a high melting point, high density and high thermal conductivity. It is commonly use in extreme temperature environments. Unfortunately, due to its high Ductile-Brittle Transition Temperature (DBTT) that goes from 150º C - 400º C, makes it difficult to machine it, a solution to this problem is powder metallurgy. Processes such as Electron Beam Melting (EBM) allows for refractory metals, such as tungsten to be manufactured.

The advantage of manufacturing tungsten specimens with EBM is each printing layer is preheated, optimizing its cooling rate, allowing for a more uniform microstructure. In addition, EBM prints occur under a vacuum, this prevents oxidation from happening due to the high temperature environment inside the chamber.

Hot Isostatic Pressing (HIP) is commonly used in additive manufactured metal specimens to enhance its properties after print. HIP tends to make the specimen denser by closing internal porosity. In addition, this technique helps to relief stresses as well as improve its fatigue properties and ductility.
This project is currently in progress; however, it is aiming to study the effect of HIP on EBM manufactured tungsten.

2. Materials and Methods

For this project, 6 cylindrical-shaped specimens were printed. The specimens are section into four samples and divided into 4 groups, each group corresponds to a different HIP temperature (1300º C, 1500º C, 1700º) and each group contains a sample of each specimen. Figure 1 shows a) a representation of the printing order of the specimens, b) illustration of the sectioning of each sample c) Sectioning into corresponding planes. Build direction plane (X) and plane containing printing layers (Z) are both analyzed in this project.

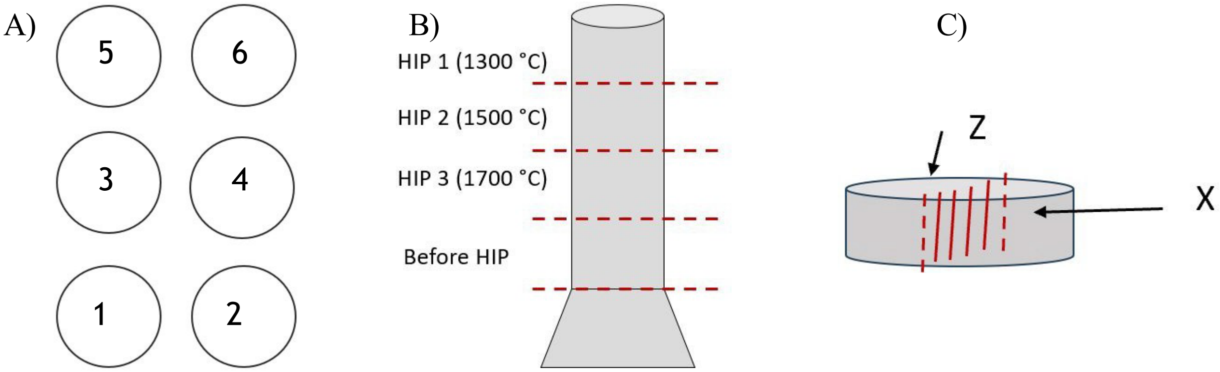

Figure 1 – A) printing order of each specimen manufactured using EBM. B) Sectioning of specimen and separation of three HIP temperature groups. C) Sample sectioning to analyze both planes, X and Z.

2.1. Metallography

Metallographic analysis was performed using standard procedure. All samples were sectioned using ATM BRILLIANT 220 (Germany). Specimens were cold mounted and grinded with grit papers from 240-1200. The samples were later polished using diamond polishing media of 9 μm, 6 μm, 3 μm, 1 μm, 0.1 μm. Samples were etched using Murakami's reagent for 10 seconds. Image J software was used to calculate porosity content.

2.2 Mechanical testing

Micro-hardness measurements were obtain using Qness 30 CHD Master + on a load of 1HV. Seven trials were recorded on each plane. All micro-hardness measurements were taking in the middle of the plane, to obtain accurate values

3. Results and Discussion

3.1 Microstructure

The challenges of processes of additive manufacturing are obtaining a specimen with fully sintered microstructure free of cracks. A study comparing Laser Powder Bed Fusion (LPBF) and EBM shows that specimens achieved high densities with microcracks and lack of fusion in its microstructure. EBM material achieves high density as well and it also presents lack of fusion throughout its microstructure. There is vast evidence that EBM prints under specific conditions can achieve specimens with a crack free microstructure. However, EBM cracking is also possible; it has been attributed that EBM manufactured specimens present a small amount of oxides along the grain boundary causing intergranular cracking.

Figure 2 shows stitching micrographs of the as printed specimens 1- 4 on the X plane. Specimens 1- 4 shows a more sintered microstructure compared to Figure 3 showing specimens 5 and 6. The presence of elongated grains indicates the cooling direction. As printed specimens on the Z plane also show lack of fusion in its microstructure.

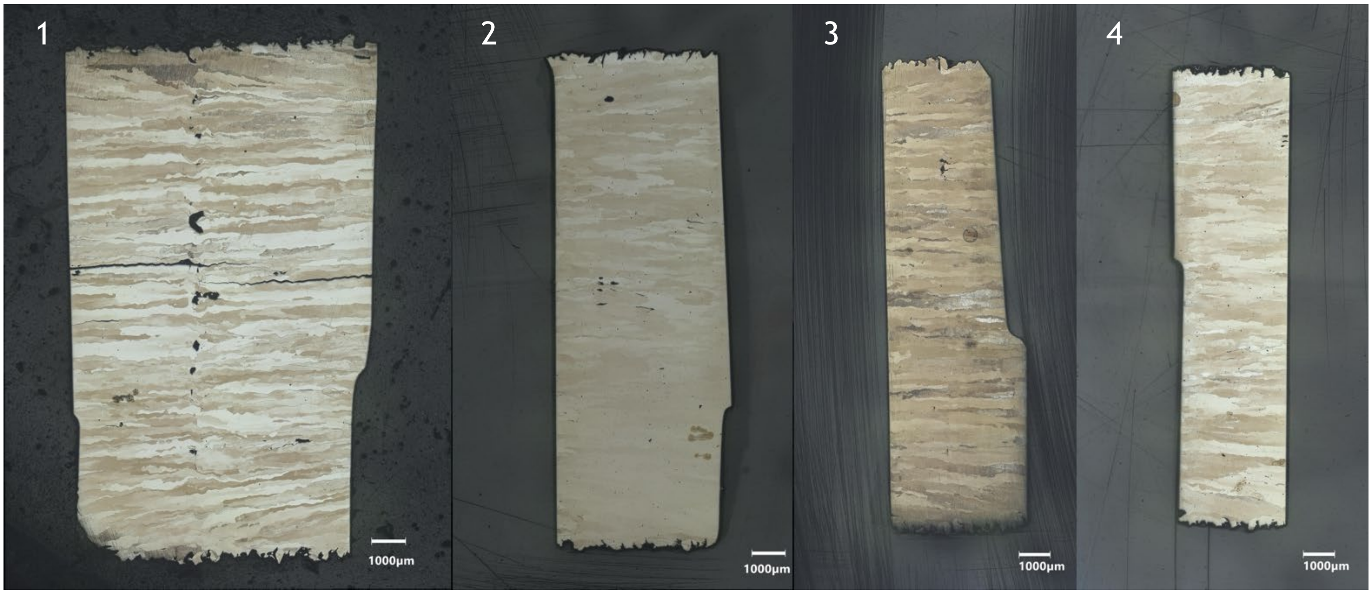

Figure 2 – stitched micrographs of the as printed specimens 1- 4

Figure 3 is showing the stitched micrographs of specimens 5 and 6. There is a greater amount of lack of fusion and porosity in its microstructure.

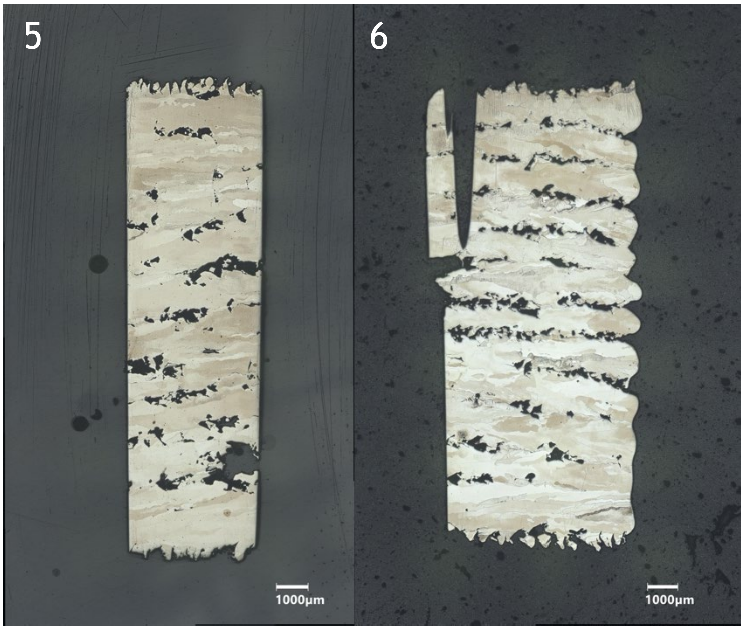

Figure 3 – Stitched micrographs of samples 5 and 6 in the as printed conditions.

The HIP 3 group, which corresponds to a temperature of 1700 º C, showed lack of fusion in its microstructure. Figure 4 is showing the conditions of specimens 1- 4 of HIP 3. Samples 1 through 4 on both, as printed conditions and HIP 3 show similar microstructures, where lack of fusion is still present. Samples 5 and 6 showed continued to show greater amounts of lack of fusion on its microstructure and is represented on Figure 5. However, it is visible that the lack of fusion begins to sinter; all samples from HIP 3 showed this event. Figure 6 shows how the lack of fusion on sample 6 on the as printed conditions began to close at the grain boundary on sample 6 of HIP 3.

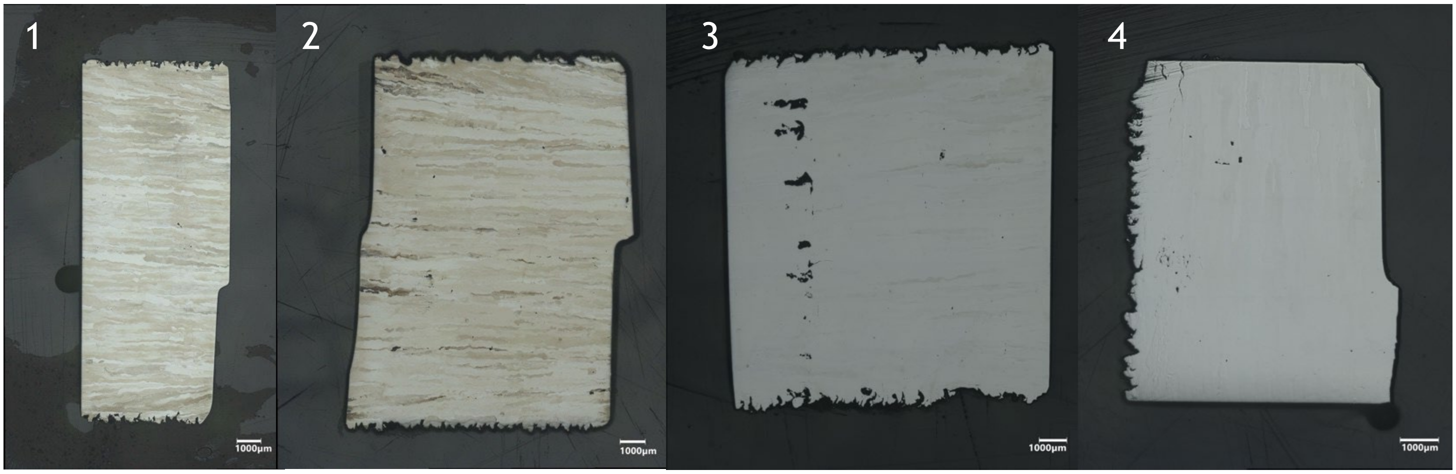

Figure 4 – Stitched micrographs of HIP 3 Samples 1 through 4

Figure 5 – Stitched micrographs of samples 5 and 6 on HIP 3, where lack of fusion is still present.

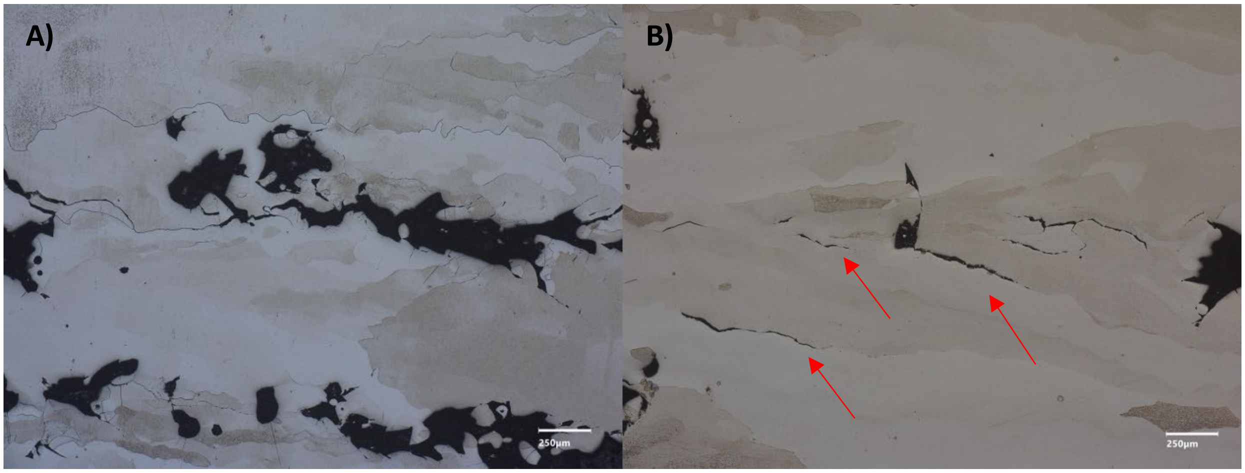

Figure 6 – A) Sample 6 on the as printed conditions presenting lack of fusion on its microstructure. B) Sample 6 on HIP 3 at 1700º C. where it shows the voids at the grain boundary closing.

3.2 Hardness

No HIP	X	Z	HIP 3	X	Z
Sample 1	383	373	Sample 1	381	371
Sample 2	381	372	Sample 2	359	367
Sample 3	381	374	Sample 3	358	362
Sample 4	380	377	Sample 4	383	371
Sample 5	382	373	Sample 5	376	366
Sample 6	384	373	Sample 6	382	380
Average	381.833333	373.666667	Average	373.166667	369.5

Table 1 – Micro hardness results of the as printed conditions and after HIP at 1700º C

Conclusion

This project is currently in process. However, until this moment, EBM manufactured tungsten is showing to decrease its hardness when increasing its HIP temperature. It was noticeable that the layered elongated grains tend to thicker, and this leads to a decrease of hardness of tungsten. The status of the project shows that HIP is having a small impact on closing the voids on the microstructure. Though, it is early to conclude the effects of HIP at this stage. Future work consists of concluding tests for the remaining of the HIP groups. There are plans to manufacture more specimens and subject them to a higher HIP temperature to understand its effect.

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