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Pore size studies of high temperature microwave assisted powder sintered Ti6AL4V components using X –ray Micro-Computed Tomography (CT)

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Abstract

Non-destructive testing of the materials continues to emerge, wherein the quality of the produced components can be assessed without destroying or severely damaging the workpiece. In this work, the morphology of the pressureless-high temperature microwave sintered Ti6AL4V components were studied using microscale-computed tomography (micro-CT). Microwave sintering of Ti6AL4V powder (AP & C, Canada) was performed under inert environment of argon gas in a high temperature microwave furnace. Thereafter, morphology of the components such as average pore size, and pore size distribution were assessed using micro-CT (Micro-CT 50, SCANCO). It was observed, that the average pore diameter decreases with isothermal temperature from 950 °C to 1150 °C. However, the average pore diameter saturates and does not vary between 1150 °C to 1350 °C. It was observed that the compressive yield strength increases as the average pore size decreases.

Keywords: Additive manufacturing, High temperature sintering, micro-CT, Ti6AL4V sintering, morphology, quality.

1. INTRODUCTION

Titanium and its alloys are used in different applications owing to its properties of light weight, high strength, corrosion resistance, and biocompatibility. Due to these properties, the Titanium and its alloys are preferentially used in biomedical and aerospace applications. Subtractive machining of Ti6Al4V is relatively time-consuming and involves high tool wear. The subtractive manufacturing process can further increase the cost of Ti6AL4V components. Therefore, additive methods can help in creating complex near-net shapes as well as reduce the time and cost of the manufacturing. Pressureless-high temperature sintering is one of the manufacturing process for low cost part production of Ti6AL4V. Recently, microwave radiations are used for high temperature sintering of metals. Microwave respond well to magnetic metals. However, for non-magnetic metals such as Titanium and its alloys the microwave does not respond well at lower temperatures [1]. Therefore, to obtain controlled and uniform heating, microwave susceptor are used while sintering Ti6AL4V using microwaves [1].

Unlike other processes, sintering also has some limitations. The most prominent limitation is the presence of pores in the components. It is not feasible to obtain components without pores in sintering. The presence of the pores and the size of the pores can affect the quality of the components produced. There are various methods to access the quality of the components produced using sintering. The most common techniques are microstructure evaluation under microscope or scanning electron microscopy. However, these conventional methods require extensive time on sample preparation. Also, 3D visualization of the pores and distribution of the pores is not possible in the conventional methods.

X-ray micro tomography is an effective technique to visualize the pore diameters and the distribution of the pores. Minimal sample preparation is required in this technique to produce high quality images. Pores are identified in the X-ray micro-computed tomography due to their linear attenuation coefficient. This parameter entirely depends upon the electron density of the specimen to be tested, the atomic number of the specimen and the energy of the incoming X-ray beam. A typical setup of the micro-computed tomography consists of an X-ray source, a rotating stage with sample holder, an X-ray detector as shown in Fig.1. The images of the specimen are captured at different viewing angles as the stage is slowly rotated. The captured imaging information is then used by the reconstruction software to recalculate the 3D map of the attenuation from the combination of the obtained images.

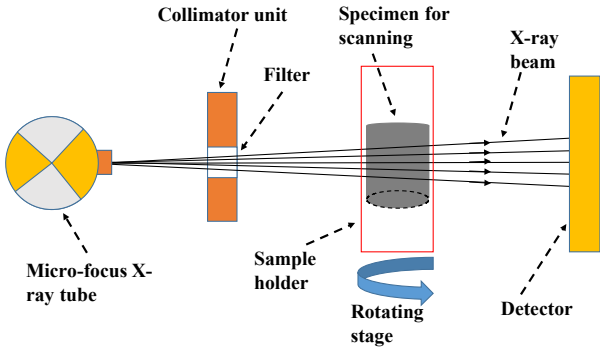

Fig 1. Schematic for Micro-CT arrangement

Researchers have successfully used micro-CT to measure the morphology of the sintered components. Of the few, Giradin et. al., successfully performed the microstructural characterization and pore size analysis for the direct laser sintering (DSL) of metal matrix composite based on *Al-Si-Cu* alloy by X-ray computed micro-tomography technique [2]. Rouholamin and Hopkinson used the micro-CT technique to understand the efficacy of the process for Nylon sintered components. The authors characterized the pore size of the sintered components using micro-CT [3]. Heinl et. al., used the micro-CT to characterize Ti6AL4V cellular structures for its average pore size for the bone implants produced using selective electron beam melting [4]. Ho and Hutmacher scripted a review of the micro-CT techniques, and compared it with other available techniques and concluded that micro-CT can produce high resolution images with almost no damage to the tested sample [5]. Cox, Wilcox et. al., evaluated the porosity of PMMA bone cement using three dimensional pores using micro-CT as measuring technique. The research group compared the results with conventional two dimensional techniques such as X-ray radiography and light microscopy. This study concluded that micro-CT is a reliable source with repeated results to measure pores three dimensionally [6]. In this paper, morphology of the

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microwave sintered Ti6AL4V components were studied. The specimens were sintered on isothermal temperatures of 950 °C, 1150 °C and 1350 °C with 10 °C/minute heating rate and a holding time of 60 minutes. The specimens were scanned using micro-CT and average pore diameter and pore size distribution was identified. Thereafter, the specimens were tested for their yield compression strength. The compressive yield strength was correlated to average pore diameter.

2. MATERIALS AND METHODS

CAD models were prepared with dimensions ϕ 10 mm × 15 mm length for the components to manufacture and thereafter the components were 3D printed (Form labs, USA) using stereolithography technique (SLA). The molds were prepared using an investment material (Bellasum, Bego, Germany). The polymeric material was burn out in a tube furnace. Hollow mold cavity of the component was formed and Ti6AL4V atomized powder particle size 15-45 µm (AP & C, Canada) was filled into the mold cavity. The sintering was experimented in a 1.45 kW microwave multimode sintering furnace (Enerzi Microwave systems, Belgaum, India) with maximum temperature 1400 °C. The sintering was performed inside an Al_2O_3 insulation boards. These boards are transparent to microwaves and does not absorb the microwaves. Titanium alloys does not absorb microwave at lower temperatures. Therefore, to obtain persistence heating rate SiC susceptors were used for the present experimentation. Three isothermal temperatures of 950 °C, 1150 °C and 1350 °C were selected for sintering. A pyrometer (range 350 °C to 1400 °C) was used for the infrared measurement of the temperature of the specimen. The sintering of Ti6AL4V was performed under the constant flow of high purity argon gas. The specimens were furnace cooled after the completion of the heating cycle. Sintering of Titanium alloy involves heating the alloy powder with a heating rate and then holding the temperature till the complete densification of the powder can be achieved. The sintering is performed in between the range of 70-80 % of the melting point temperature [7]. Sintering generally involves three stages, first stage is formation of necks between the powder particles. Second stage involves the densification of the parts and third stage involves further densification by eliminating isolated pores [8].

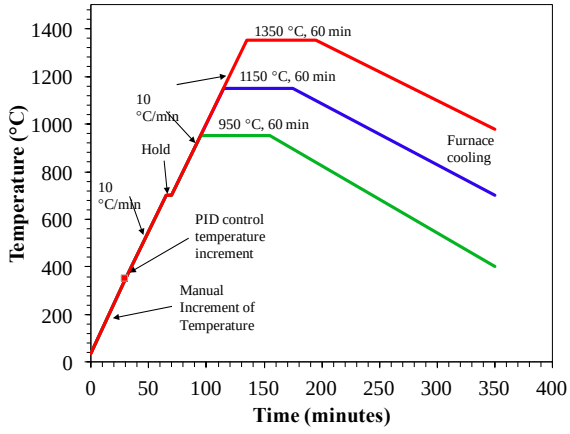


Fig. 2. Heating cycle used for sintering of Ti6AL4V alloy

The average pore size was measured using X-ray micro-computed tomography (µCT) µCT-50 (Scanco Medical, Bassersdorf, Switzerland). The specimens were scanned in a ϕ 15.2 mm sample holder. The specimens were firmly fixed inside the sample holder to avoid any movement during the rotation of sample holder. All the specimens were scanned at higher resolution and the obtained voxel size was 4.4 µm. The specimens were scanned for a depth of 3.55 mm in 800 slices. The specimen was rotated by 180° to obtain projections for the 3D reconstruction. The X-ray voltage and the current intensity used for scanning were 90 kV and 88 µA respectively. The region of interest (ROI) was defined manually approximately covering over ~1000 pixel. The threshold values were maintained same for the reconstruction of 2D and 3D images. An inbuilt evaluation script was executed for the identification of average pore size and pore size distribution. The average pore diameter is calculated by fitting the largest sphere in the void space of the pores fitting through each voxel in contained with the void space. The average is calculated for the all these spheres and average pore diameter is reported. A detailed discussion about the method used for calculating the average pore diameter and pore size distribution is presented by Bouxsein et. al., in their review article [9].

The compression yield strength was tested for the specimen using made using three temperatures on Universal testing machine (Instron 5582, UK). The testing speed was maintained at 1 mm/minute. One sample was tested for each experiment.

3. RESULTS AND DISCUSSION

3.1 Pore size measurement

The average pore diameters were measured using the micro-CT and the results are plotted in Fig.3. It can be observed from the plot that at 950 °C the average pore diameter is ~22.9. whereas the pore size reduces with the increase of temperature till 1150 °C. Thereafter, the change in average pore diameter was not observed to be significant. This could be because at higher temperature as the densification and grain size increases as a result the pore size slightly increases [10].The sintering behavior for Ti6AL4V observed here was solid state sintering. This was also confirmed by Luo *et. al.* in their experiments on Titanium alloys [11].

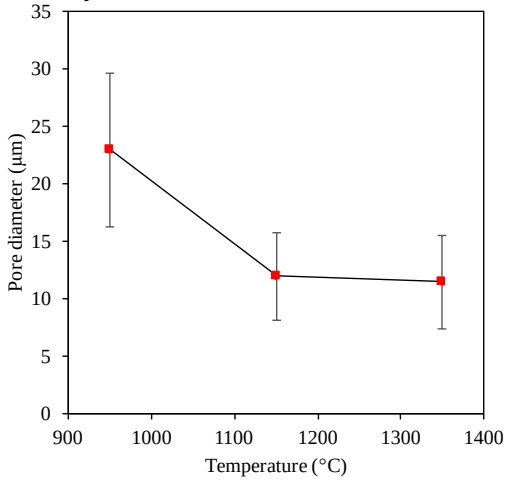


Fig. 3. Plot for pore diameter versus isothermal temperature

In Fig. 4. it can be clearly seen that there are micro size pores. The pores are randomly arranged inside the part. With the help of 3D reconstruction of the micro-CT images internal defects such as voids or local shrinkage defects can easily be observed. However, in the present study the components were carefully produced and hence these defects were not observed.

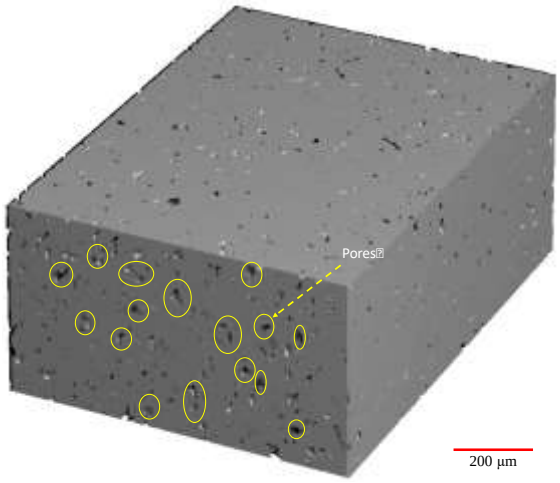
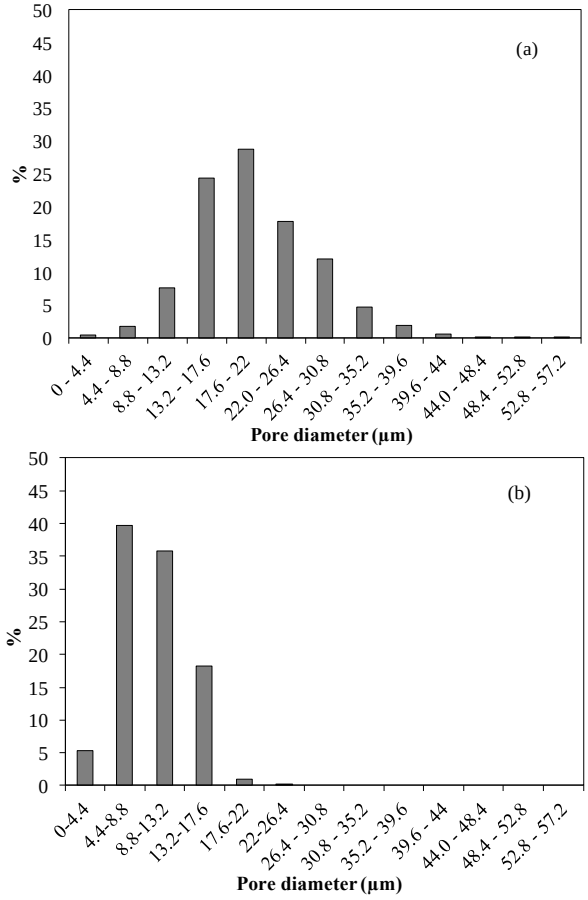


Fig. 4. Micro-CT 3D reconstruction of microwave sintered Ti6AL4V at 1150 °C peak temperature, 10 °C/minute rate of heating and 60 minutes of holding time.

3.2 Pore Size Distribution

Pore size distribution for the Ti6AL4V components produced by microwave sintering were determined using micro-CT technique. The histogram is presented in Fig. 5. (a), (b) and (c). The sizes of pores ranged between 4.4 µm to 57.2 µm at sintering temperature of 950 °C (Fig. 5. (a)). Only ~ 2 % of the total pores were observed between the size of 4.4 to 8.8 µm. This indicates partial densification due to slightly low sintering temperature. As a result, larger size pores were obtained.



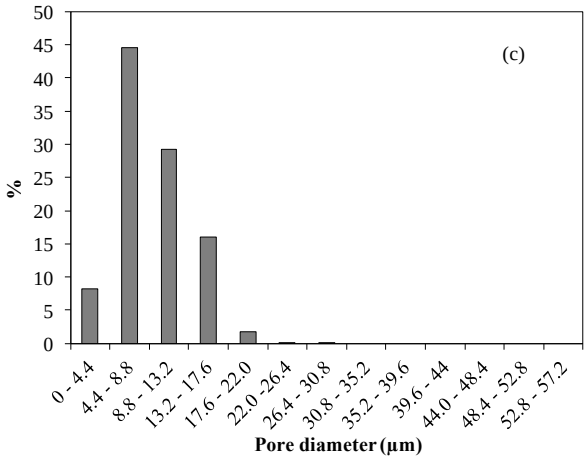


Fig. 5. Pore size distribution for isothermal temperature of (a) 950 °C, (b) 1150 °C, and (c) 1350 °C.

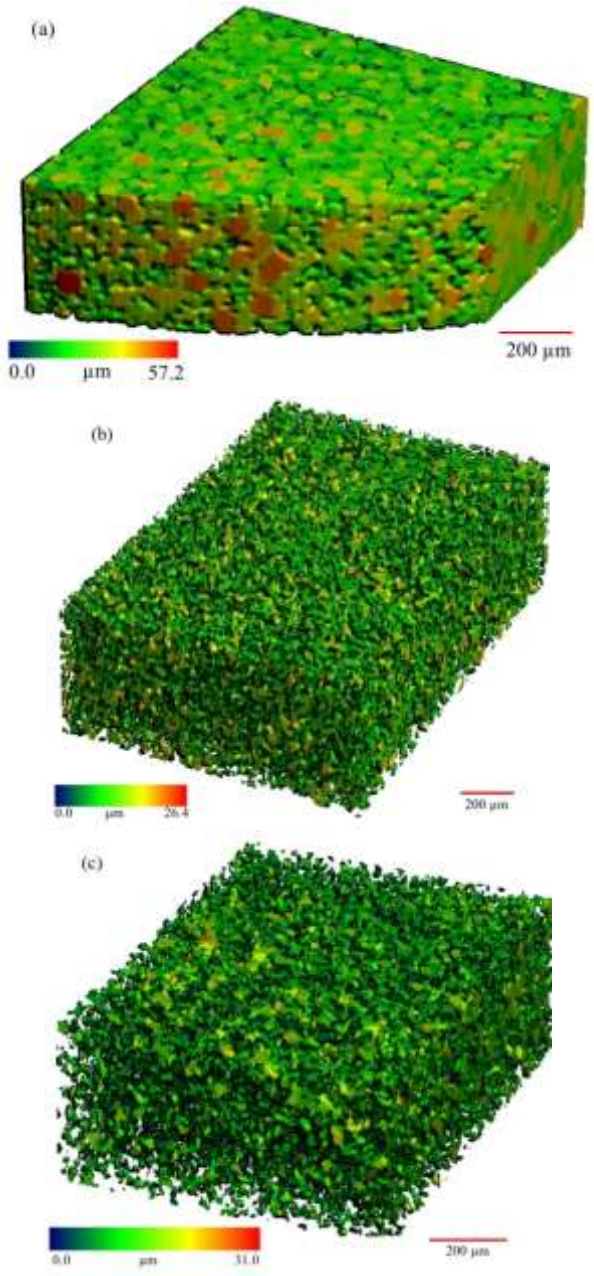


Fig. 6. Micro-CT 3D reconstruction of pores for different isothermal temperatures at (a) 950 °C, (b) 1150 °C, and (c) 1350 °C

The pores size ranges between 4.4 µm to 26.4 µm at sintering temperature of 1150 °C as shown in Fig. 5 (b). It can be observed that at 39 % of the total pores were found in the size range of 4.4 to 8.8 µm. In the case of sintering temperature of 1350 °C, the sizes vary between 4.4 µm to 30.8 µm, (Fig. 5 (c)). In the 4.4 µm to 8.8 µm pore size, accounted for ~45 % of the pores. Fig. 6 (a) show the 3D reconstruction of pores showing larger spheres for the components sintered at 950 °C. It can also be seen from the Fig. 6 (b) and (c) of the components sintered at 1150 °C and 1350 °C that at higher temperatures, only few of the pores are larger in size than the pores in 1150 °C.

3.3 Correlation between pore size and compressive yield strength

The correlation between the average pore size and compression yield strength was identified. Fig. 7 shows the plots for pore size and compression yield strength. It is evident from Fig. 7 that as the average pore size decreases the strength is increasing. At 950 °C the yield strength was observed to be the lowest and the at 1350 the yield strength was observed to be highest.

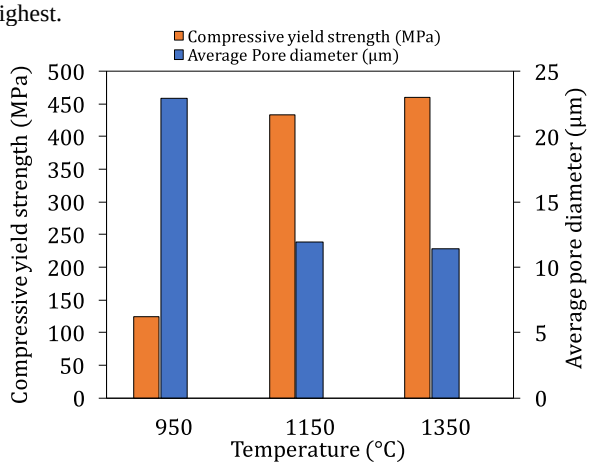


Fig. 7 Average pore size versus compression yield strength graph

4. CONCLUSION

Experiments were conducted to find out the average pore diameter and the pore size distribution in the microwave sintered Ti6AL4V specimen. The sintering was performed on three isothermal temperatures of 950 °C, 1150 °C and 1350 °C. The average pore diameters were determined at the three different temperatures. The average pore diameter was observed to be 22.9 µm at 950 °C, 11.9 µm and at 1150 °C and 11.4 µm at 1350 °C. The pore size distribution was determined and histograms were plotted. The pore size distribution revealed that at lower temperature of 950 °C the pore size varies between 4.4 µm to 57.2 µm, at 1150 °C the pore size varies between 4.4 µm to 26. 4 µm and at 1350 °C pore size vary between 4.4 µm to 31 µm. In addition, the spread of the pores were identified though 3D reconstruction of the X-ray micro-CT images. Lastly, relationship of the pore size with compressive yield strength was identified.

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