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EFFECT OF SINTERING TEMPERATURES ON MICROSTRUCTURE AND PROPERTIES OF Ti-6Al-4V ALLOY USING POWDER INJECTION MOLDING (PIM)

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ABSTRACT

Binder system consisting of palm stearin (PS) and low linear density polyethylene (LLDPE) were mixed with Ti-6Al-4V alloy powder to prepare feedstock. Ti-6Al-4V compacts were fabricated by powder injection molding (PIM). Influence of vacuum sintering temperature on mechanical properties and microstructure of the sintered compact at 1100 °C, 1150 °C, 1200 °C and 1250 °C with 4 hours soaking were investigated. The experimental results show that the compact sintered, made from 100% gas atomized powder (average particle size is 16 μm), have a relative density of 92.6%-98.5%; the range of hardness value around 336-391 Hv; ultimate tensile strength of 361-830 Mpa; and a lower elongation (<2%).

Keywords: Ti-6Al-4V; Palm Stearine; Low Linear Density Polyethylene;

INTRODUCTION

The excellent properties of Ti-6Al-4V alloy such as high specific strength, high corrosion resistance in many media and excellent biocompatibility have attracted attention for the past decades and make this alloy an excellent choice for many application including medical implants, automotive components and craft parts. However, the utilization of this alloy has been limited due to the high cost related to raw material, the multi-step fabrication process and associated geometry design constrains

[1].Therefore it is considered to be very available that Powder Injection Molding (PIM) process is applied to fabrication of titanium alloy parts [2].

PIM is a technique with and cost advantage for producing titanium alloy component with small, complex shape and precision parts in high volume. Firstly, the products fabricated by PIM are expected to have more homogeneous microstructure, since hydraulic pressure is applied during injection molding. The mold is filled up uniformly, which avoids density gradient in conventional press/sintering process. Secondly, fabrication cost could be eliminated significantly by reducing machining and recycling using of feedstock [3].

Nevertheless, injection molding of titanium alloy presents a real challenge to the processor due to its reactivity. Titanium not only has a strong affinity to oxygen but tends to react readily with carbon, nitrogen or hydrogen from the furnace atmosphere. Therefore contamination by interstitial light element such as oxygen and carbon is a serious problem because they have much influence on the mechanical properties of titanium alloys [4]. So it is necessary that debinding and sintering condition are controlled strictly.

This present work was aimed to investigate the effect of sintering temperature on the sintering behavior and final properties of powder injection molded Ti-6Al-4V. Metallographic techniques were employed to sinter tensile bar to investigate the sintering behavior. Tensile strength, density, elongation and hardness properties of the sintered production were evaluated.

EXPERIMENTAL DETAILS

In this research, gas atomized Ti-6Al-4V powders (Ti-5.9Al-3.9V-0.19Fe-0.12O-0.01C-0.01N-0.004H) provided by TLS Technik Spezialpulzer (Germany) was used. It has particle size distribution of D_{10} = 5.27 µm, D_{50} = 16.11 µm, D_{90} = 28.53 µm. Particle size distributions were determined using CILAS 1190 Dry equipment. Morphology of the Ti-6Al-4V powders observed using scanning electron microscope is given Figure.1. The gas atomized Ti-6Al-4V powder was found in spherical shape.

A multiple component binder system consisting of 40% low linear density polyethylene and 60% palm stearin was used. This binder was mixed with Ti-6Al-4V powder for about 2 hours at 160°C by a Win Worth Z-blade mixer in vacuum atmosphere for prepare feedstock. The powder loading in this mixture was 66 volume percent. After cooling, the feedstock was palletized by hand. The feedstocks were injected by vertical injection molding machine at temperature range of 180°C-200°C. The injection pressure was 80 Mpa and the mold temperature was 50°C. Debinding was performed in two steps which are solvent extraction and thermal operation. The solvent debinding was performed on green molded part for 6 hours in solvent n-heptanes at 60°C. Then, thermal debinding was performed in vacuum atmosphere according to schedule 1:20 ^{o}C

$$\xrightarrow{10\ min} 30\ °C \xrightarrow{30\ min} 100\ °C \xrightarrow{90\ min} 200\ °C \xrightarrow{120\ min} 500\ °C \xrightarrow{120\ min} 500\ °$$

The sintering cycle applied to the samples was a follows: samples were heated to 750 °C at a rate of 0.18 °Cmin^{-1}, then the samples were heated to different temperatures of 1100 °C, 1150 °C, 1200 °C and 1250 °C at rate of 3.75 °Cmin^{-1} and they were held at each for 4 hours respectively in vacuum atmosphere (10^{-3} Mbar).

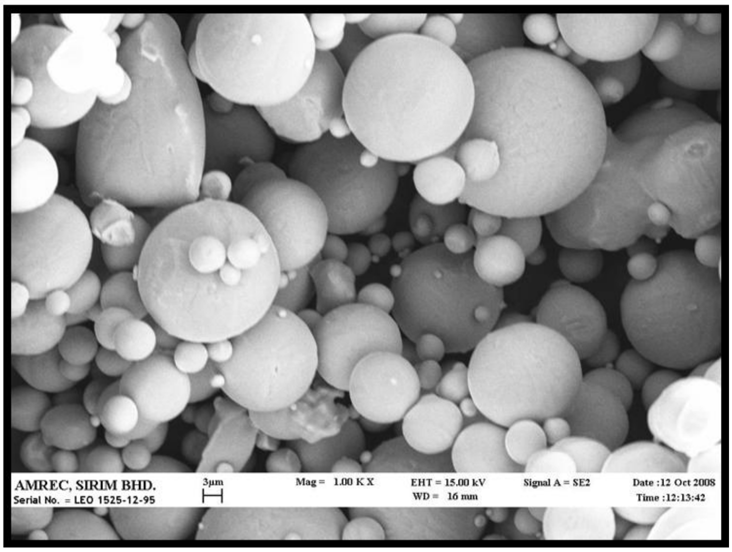

Figure 1: The morphology of Ti-6Al-4V powder

The densities of the sintered samples were measured by means of the Archimedes water immersion method. All tensile tests were performed using Universal tensile machine at a constant crosshead speed of 1 mm min^{-1}. The hardness tests were performed using a Vickers hardness machine. The powder morphologies and optical microstructure of sintering samples were observed using a scanning electron microscope (LEO 1525). For optical metallographic, samples were cut from the sintered tensile test bar. A kroll reagent (3 ml HF, 6 ml HNO_3 in 100 ml H_20) was used to etch the samples.

RESULTS AND DISCUSSION

The physical and mechanical properties of the sintered specimen titanium alloy parts were shown in Table 1. The results show that the Ti-6Al-4V sample sintered at 1150°C have achieved the minimum requirement for sintered PIM compared with Standard Metal Powder Industries Federation (MPIF) 35 for titanium alloy except for the elongation. Among, the Ti-6Al-4V samples sintered at 1100 °C, 1200 °C and 1250 °C, minimum requirement of (MPIF) 35 for titanium alloy have been met except for the tensile strength and elongation.

Figure 2 shows the effect of sintering temperature on the theoretical density of Ti-6Al-4V samples. The figure indicates that the density of Ti-6Al-4V sintered sample

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increase with increasing sintering temperature except at 1250 °C. The Ti-6Al-4V sintered sample has attained a maximum theoretical density of 98.5% with low percent of porosity (1.53%) when sintered at 1150 °C and possesses low percent of porosity (1.45%) when sintered at 1200 °C. However the theoretical density of sample sintered at 1250 °C shown slightly decrease (97.5%) and possess increase percent of porosity (2.41%).The Ti-6Al-4V molded part sintered at 1100 °C possess lower theoretical density (92.6%) but higher percent of porosity (7.37%). So, the sintering temperature at 1150 °C and 1200 °C improved the theoretical density and reduce porosity of Ti-6Al-4V sample.

Table 1: Physical and mechanical properties of sintered Ti-6Al-4V

Properties	Soaking time				MPIF 35
	1100°C	1150°C	1200°C	1250°C	
Density (g/cm^{-3})	4.122	4.382	4.382	4.343	> 97 %
Porosity (%)	7.37	1.53	1.53	2.41	< 5
Hardness (Hv)	366	336	391	351	300-400
Elongation (%)	1.74	0.35	0.47	1.84	10-15
Ultimate Tensile Strength (Mpa)	361.8	830.09	584.24	439.93	> 800

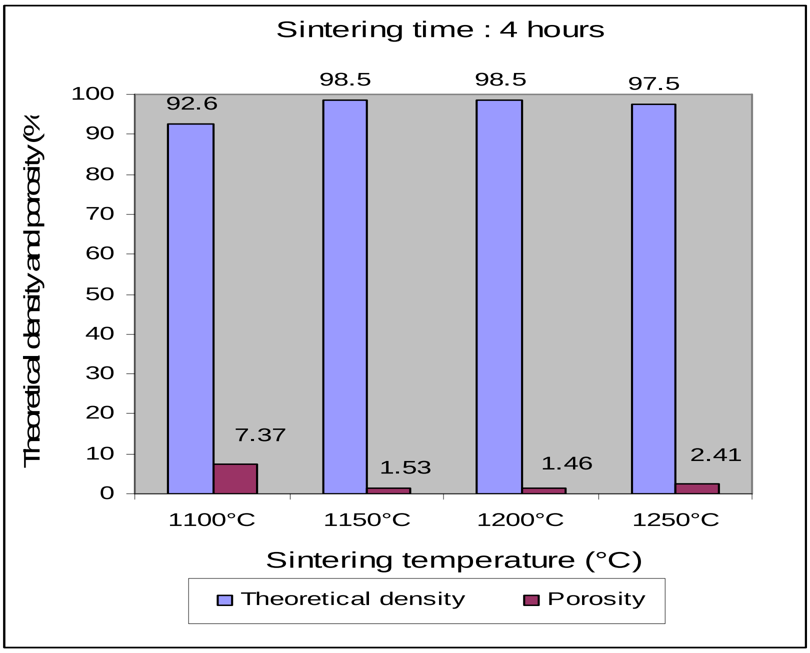

Figure 2: Effect of sintering temperature on theoretical density and porosity of Ti-6Al-4V

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Figure 3 shows the optical micrographs of porosity injection molded Ti-6Al-4V sintered at 1100 °C, 1150 °C, 1200 °C and 1250 °C using scanning electron microscope in secondary electron (SE) mode. This figure indicates that the porosity of Ti-6Al-4V decrease with increasing sintering temperature. However, sample of Ti-6Al-4V molded part which is sintered at 1100 °C shown increase porosity.

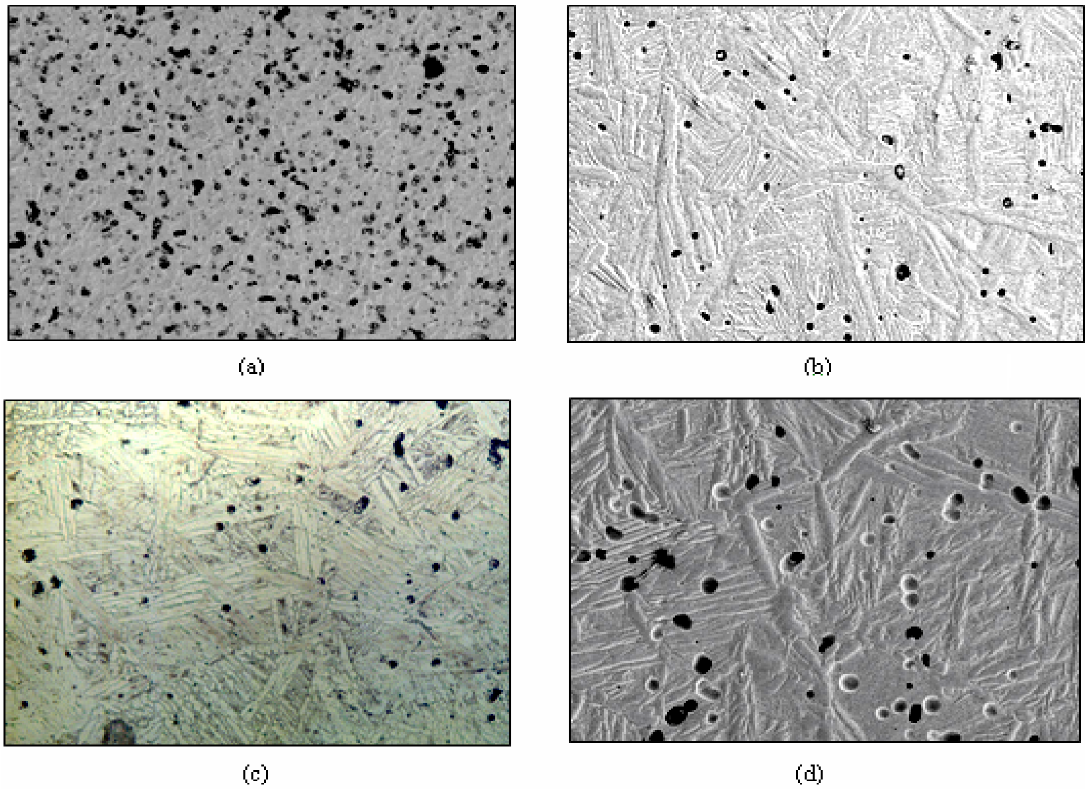

Figure 3: SEM in SE mode optical micrograph of porosity injection molded Ti-6Al-4V sintered (a) at 1100 °C (b) at 1150 °C (c) at 1200 °C (d) at 1250 °C with heating rate dT sinter of 3.75 °Cmin^{-1}

Figure 4 shows optical micrographs of microstructure injection molded Ti-6Al-4V sintered at 1100 °C, 1150 °C, 1200 °C and 1250 °C with 4 hour soaking. The Ti-6Al-4V was found to have coarse acicular microstructure, revealing α grain with β intergranular phase. The percentage of α phase in the alloy depends on sintering condition. Samples sintered at low sintering temperature will affect porosity and the amount of α phase, when it appears precipitated at the grain boundaries and darker than β phase. Samples sintered at high temperature above the β transition temperature (1050 °C) and subsequent slow rate cooling in the furnace, resulted in the development of Widmanstatten microstructure. In this structure, colonies of β lathes (bcc and rich in V) and α platelets (hcp and rich in Al) formed inside the prior β grain [4].

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Figures 4(a) indicates that specimens have dual phase microstructure consisting of equiaxed α particles and β particles. However, the samples possess low strength (361.80 Mpa) and high ductility (1.74%). Figure 4(b) shows a Widmanstatten microstructure, consisting of many smaller β particles and few α particles. So, the samples possess higher strength (830.09 Mpa) with lower ductility (0.35%) because of the occurrence of recrystallization and quickly increase phase in the prior crystal particles and the thickness of crystal boundary between α phases and β phase. So, these factors make the tensile strength to increase and ductility to decrease [5].

Figure 4(c) shows the sample have a few equiaxed β particles but many equiaxed α particles. This factor degraded the mechanical properties of the sample in term of tensile strength (584.24 Mpa) and ductility (0.47%). Figure 4(d) shows typical martensite microstructures which consist of many needles like α particles in the prior β particles. The dimension of equiaxed α particles in this sample increased accordingly. So, these factors make the tensile strength to decrease and ductility to increase.

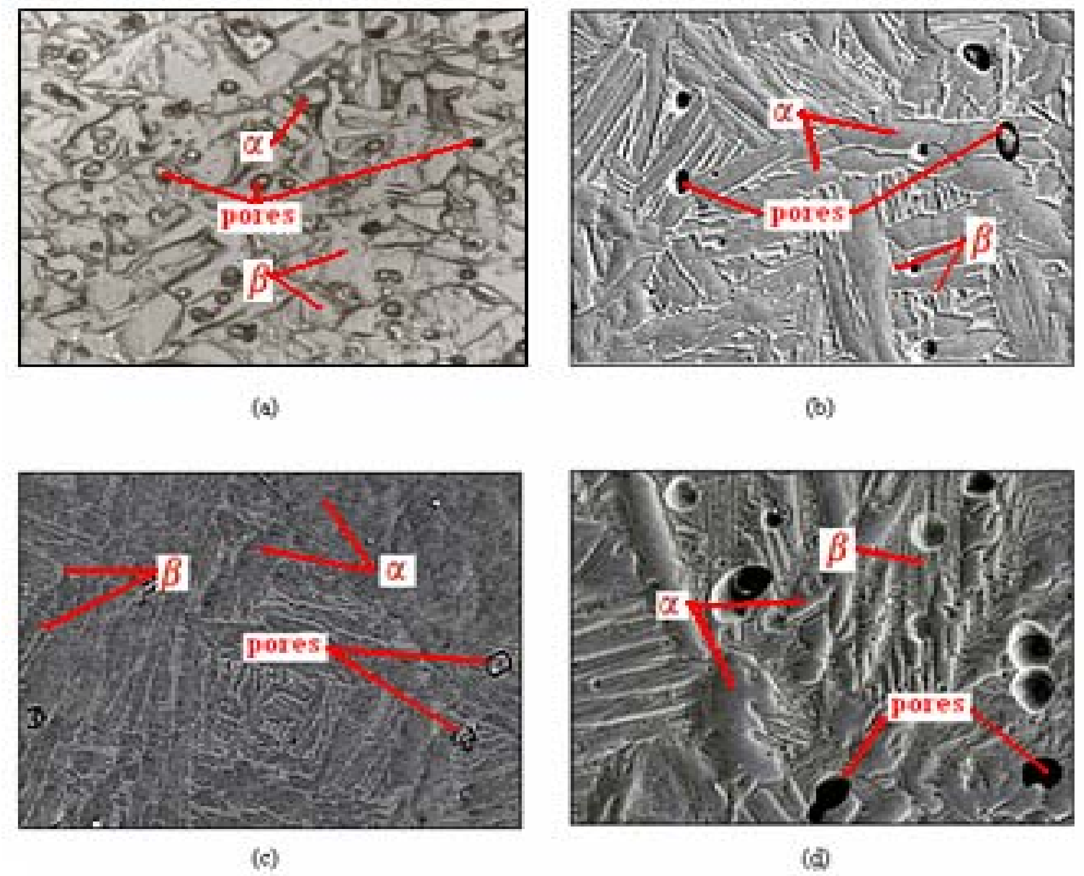

Figure 4: Microstructures of injection molded Ti-6Al-4V parts sintered at different temperatures: (a) at 1100 °C (b) at 1150 °C (c) at 1200 °C (d) at 1250 °C with heating rate dT sinter of 3.75 °Cmin^{-1}. (α = dark region β = white region)

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The effect of sintering temperature on ultimate tensile strength and elongation of Ti-6Al-4V sintered parts is shown in Figure 5. From Figure 5 the tensile strength of sample indicates are increasing when sintered at 1100 °C and 1150 °C. However, the tensile strength of Ti-6Al-4V sintered sample indicates decrease when sintered at 1200 °C and 1250 °C. The Ti-6Al-4V sample which sintered at 1150 °C has achieved higher ultimate tensile strength (830.09 Mpa), while the sample sintered at 1100 °C achieve the lower tensile strength (361.80 Mpa). However, the sample sintered at 1250 °C shower higher ductility 1.84% followed by samples sintered at 1100 °C with ductility 1.74%; at 1200 °C with ductility 0.74% and at 1150 °C with ductility 0.35%.

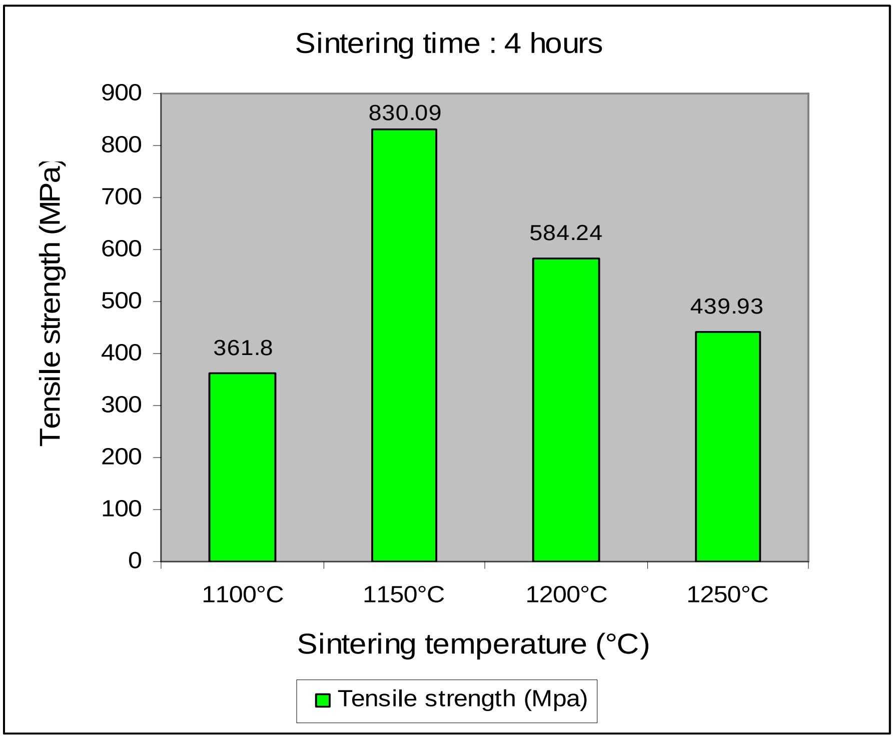

Figure 5: Effect of sintering temperature on ultimate tensile strength of Ti-6Al-4V samples sintered at 1100 °C, 1150 °C, 1200 °C and 1250 °C

Figure 6 shows the effect of sintering temperatures on ductility of Ti-6Al-4V sintered part. The samples sintered at 1250 °C showed higher ductility (1.84%) followed samples sintered at 1100 °C with ductility (1.74%); at 1200 °C with ductility (0.47%) and at 1150 °C with ductility (0.35%).

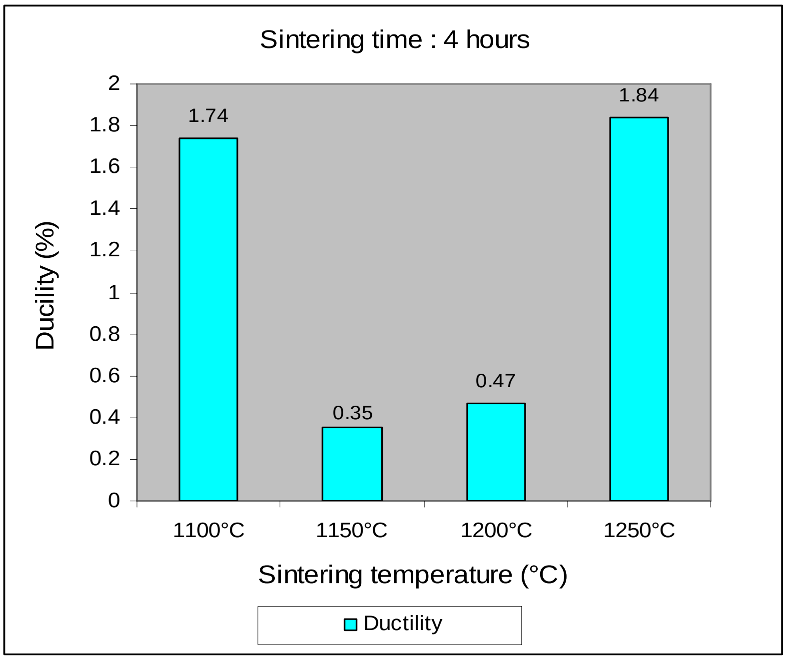

Figure 6: Effect of sintering temperatures on ductility of Ti-6Al-4V samples sintered at 1100 °C, 1150 °C, 1200 °C and 1250 °C

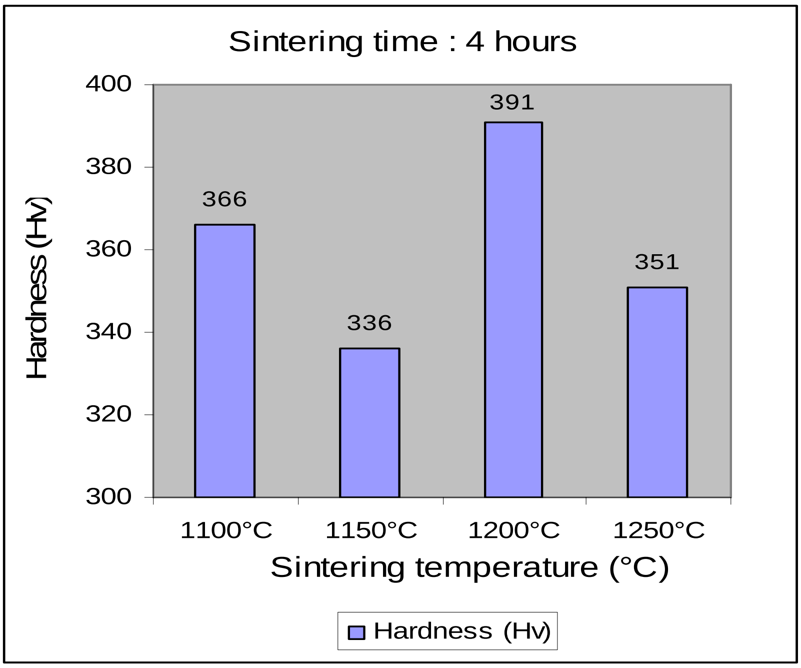

Figure 7: Effect of sintering temperatures on hardness of Ti-6Al-4V sample sintered at 1100 °C, 1150 °C, 1200 °C and 1250 °C

The effect of sintering temperature on the hardness of Ti-6Al-4V samples is shown in Figure 7. Samples attained a maximum hardness of 391 Hv when sintered at 1200 °C for 4 hour. The sample which is sintered at 1150 °C shows the lowest hardness value 336 Hv. With increasing sintering temperature, the hardness value fluctuates results obtained. The hardness for all samples achieved the minimum requirement of MPIF 35 standard.

CONCLUSION

The effect of sintering temperature on microstructure and properties of Ti-6Al-4V sintered part has been investigated. The results revealed Ti-6Al-4V sample sintered at 1150 °C possess higher strength and met all minimum requirement of MPIF 35 in term of density, porosity, hardness and strength except ductility because of the occurrences of recrystallization and quickly increase phase in the prior crystal particles and the thickness of crystal boundary between α phases and β phase. These factors make tensile strength increase and ductility decrease. Overall, it is concluded that sintering temperature does plays an important role on the microstructure and properties of Ti-6Al-4V sintered part.

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