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The Effect of Holding Time on the Mechanical Properties of Y-TZP-TITANIA-TCP Composites

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Abstract: *The impact of sintering temperature, additive content and holding time on Y-TZP-TiO2-TCP composites was investigated in this research, with respect to the densification behaviour and Vickers hardness. A high-purity titanium oxide (TiO2), tricalcium phosphate (TCP) and Y-TZP composites were prepared in various wt% whereby TCP content was fixed at 15wt%, TiO2 content was varied from 0wt% to 10wt% while Y-TZP content was varied from 75wt% to 85wt%. The samples were prepared into circular discs and rectangular bars i.e uniaxially pressed at 200MPa. Then, the samples were pressureless-sintered at temperatures ranging between 1200˚C to 1400˚C with a ramp rate of 10˚C/min for 60, 90 and 120 minutes holding time. The mechanical properties: bulk density and Vickers hardness were measured and analysed. The results revealed that the higher sintering temperature and higher additive content of TiO2 played a vital role, as good contributing factors towards the enhancement of mechanical properties of this composite especially when sintered at 90 minutes holding time. The results obtained for the composition of 10wt% TiO2/15wt% TCP and 75wt% ZrO3 ceramics were 4.89Mgm-3 for bulk density and 10.67GPa for Vickers hardness, sintered at 1400˚C with 90 minutes holding time. The results show a significant contribution towards mechanical properties accelerated from the holding time and sintering temperature.*

Keywords: Titanium Oxide, Tricalcium phosphate, Zirconia, Mechanical Properties, Holding Time

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1. Introduction

This research work mainly focusses on the optimization of holding time on Y-TZP-TiO_2-TCP composites with various additive content sintered at temperatures ranging from 1200˚C to 1400˚C. The holding time experimented were 60 minutes, 90 minutes and 120 minutes to its significant contribution towards mechanical properties namely Bulk density and Vickers hardness. Whereby a convincing result was obtained for composition of 10wt% TiO_2/15wt% TCP and 75wt% ZrO_3 sintered at 1400˚C for 90 minutes holding time. Zirconia naturally gives a very good impact towards mechanical properties; however the addition of Titanium Oxide and Tricalcium Phosphate were done to enable bone regeneration and begin reaction with the tissues surrounding it.

2. Literature Review

It is a remarkable fact that ceramic materials have been an excellent contributing factor for implants in biomedical applications. Among the different ceramics, the Zirconia ceramics have shown significant outcomes due to their high mechanical characteristics and biodegradability.

Zirconia ceramics have been extensively used in biomedical applications like in orthopedic applications for bone defects, prothesis, dental implants and fragments of skeleton. These applications constantly work for the enhancement in mechanical properties and tribological performance especially in advanced ceramics technology. (M.H. Ghaemi et al., 2017). It is an undeniable fact that ceramic materials have high hardness, low wear rate and biocompatibility, thus it is widely used in biomedical components (Rahaman et al., 2007). In this recent time, the famous material of choice that is commonly used in biomedical implants are tetragonal polycrystal zirconia, specifically the 3mol% yttrium oxide (yttria) stabilized zirconia. The reason Y-TZP is chosen because of its good mechanical properties, which possess relatively good fracture toughness, Youngs Modulus, chemical and dimensional stability, low bacterial affinity and resistance towards wear (X. Guo, 2003; L. Blaise et al., 2001; Reveron et al., 2017; Piconi and Maccauro, 1999; Cho et al., 2014; Buciumeanu et al., 2017; Cionca et al., 2017).

Therefore, in the recent years, Y-TZP has been utilized in biomedical field for the use of bone implants due to its excellent combination of high mechanical properties and good chemical inertness (C. Piconi and Maccauro, 1999; G. Heimke et al., 2002; E. Marcella et al., 2014).

Although Zirconia is known to have excellent fracture toughness (K_{IC} =5 –8MPam$^{1/2}$), it is still considered as a brittle material, as it has the tendency to crack easily due to high impact and thermal shocks (Turon-vinas and Anglada, 2018). Zirconia is also known as bioinert; hence it is normally coated with bioactive materials like beta-tricalcium phosphate (β-TCP), hydroxyapatite (HAp) to improve osseointegration. Osseointegration simply means the immediate structural and functional connection between living bone and the surface of a load-bearing artificial implant. Therefore, these bioactive materials are equivalent to the calcium phosphates found in human bones which can help to improve osseointegration. In the current practice, metallic materials are used to perform knee, dental, femur, bone and other implants. Meanwhile, there is an obvious trend in this recent time, to use more biocompatible and inert materials to perform these implants that avoid corrosion, allergic reactions, ions release, by Bollen, 2017 and Soni et al., 2016.

Despite, on a different school of thoughts, titanium in the form of metallic material is made as a choice to produce dental implants, knee joints and femur implants, on the other hand, different researchers mention a clear inclination to produce these implants from more biocompatible and inert materials. It is reported that these implants inhibit corrosion, ions release, any kind of allergic reactions by Bollen, 2017 and Soni et al., 2016. With reference to this, researchers have reported that implant materials developed immensely in these recent few decades, which is also expected to further grow in this era in relation to the use of better biomaterials, surface modifications, implant's design, and functionalization (Gaviria et al., 2014).

Moreover, implant premature failure could happen when the bioactive coatings detached from the zirconia surface (D. Faria, 2019). Zirconia is unable to begin a positive reaction with the tissues surrounding it after implantation, as it is biocompatible and bioinert. Thus, this characteristic can negotiate osseointegration since bone cannot naturally develop on Zirconia surface as the cells cannot be regenerated (Caravaca et al., 2016; Dehestani et al., 2012).

Hence, this research was conducted to focus on the bone generation of this Y-TZP-TiO_2-TCP composites.

It is known that; sintering is a vital process in an atomic diffusion powered by capillary pores. At this stage, the inter-particle pores in a granular material are eliminated. There are two variables that affect this sinterability, namely the material variables and process variables. The material variables are inclusive of particle size of powder, chemical composition of the powder compact, the grade of powder agglomeration, size distribution and shape. Whereby the process variables include time, temperature, pressure, atmosphere, heating and cooling rates (U. Sutharsini et al., 2014) Thus, the process variable, time was selected to be investigated in detail in this research work.

3. Materials and Methods

In this research work, the TiO_2-TCP doped Y-TZP, with different TiO_2 and TCP content were prepared through co-precipitation method. 0wt% to 10wt% of TiO_2 and 15wt% TCP were mixed with Y-TZP in ethanol solution. An ultrasonic machine was used to mill the mixture for 1 hour through wet milling process. Then, the slurry was dried at 60°C in an oven for 12 hours continuously. Then after, the powder was sieved using a 212µm mesh stainless steel sieve which would be ready to be pressed. The TiO_2-TCP-Y-TZP powder was then uniaxially pressed in a hardened steel into circular discs of 20 mm diameter and rectangular bars of 80mm × 50mm × 8mm mold and die set using a hydraulic pressure of 500 MPa. Followed by that, the pressed samples were sintered in a heating furnace (ModuTemp) between 1200ºC to 1400ºC. The ramp-rate was set at 10ºC/min for both heating and cooling, and holding time of 60, 90 and 120 minutes before the temperature cools down to room temperature as shown in Figure 1. These sintered samples then, were polished using SiC papers (120, 240, 600, 800); coarse to rough grades. Next, the samples were polished with a diamond paste to 6µm to obtain an optical reflective surface. In order to obtain the mechanical property results, the samples were immersed in distilled water to measure density using the Archimedes' Principle where a Mettler Toledo Balance AG204 densi-meter was used. While, Vickers indentation method was incorporated to perform Vickers hardness tests on the polished samples. The load applied to the samples was at 98.1 N, which was kept constant with a 10s loading time.

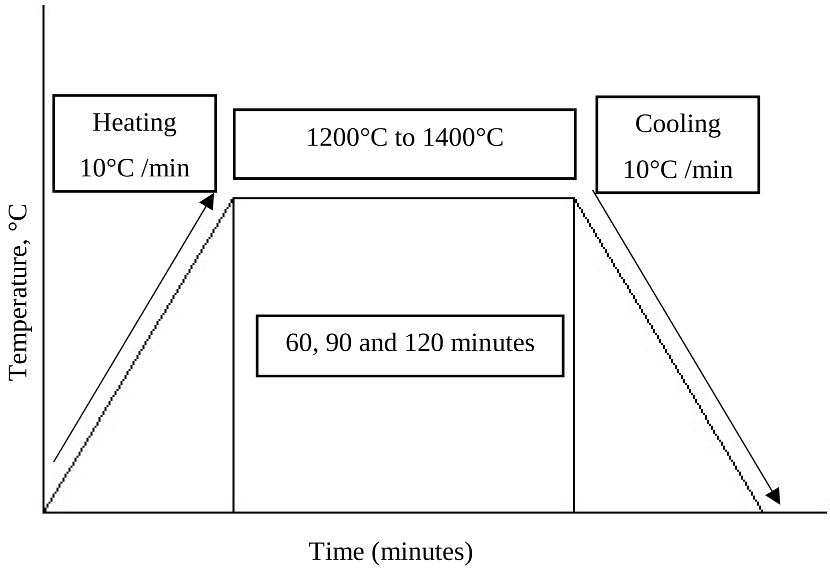

Figure 1: Graph of Sintering profile

4. Results and Discussions

4.1 Bulk Density

Figures 2, 3 and 4 show the bulk density results of the Y-TZP-TiO_2-TCP composites sintered at different holding times (i.e. 60, 90, 120 minutes), whereby these samples were investigated at the temperatures ranging from 1200℃ to 1400℃. Mostly all compositions of all holding times have shown increasing trend in the Bulk density, especially at higher sintering temperatures ranging between 1300ºC to 1400ºC. The highest Bulk density result, 4.97 g/cm^3 was obtained for the composition of 0wt% TiO_2/15wt% TCP and 85wt% ZrO_3 sintered at 1300ºC for 120 minutes holding time. This result recorded about 83% of the theoretical value of density for Y-TZP (6.1 g/cm^3). This density is remarkably high due to its higher zirconia content in the composition. In addition to that, the composition of 10wt% TiO_2/15wt% TCP and 75wt% ZrO_3 sintered at 1400ºC for 90 minutes holding time attained a density value of 4.89 g/cm^3. This result recorded about 82% of the theoretical value of density for Y-TZP, which is quite close to the density value obtained for the 0wt% TiO_2/15wt% TCP and 85wt% ZrO_3 composition. However, this composition required more holding time as compared 10wt% TiO_2/15wt% TCP and 75wt% ZrO_3. Besides that, it can be observed, relatively higher density values were obtained for all compositions, when sintered at the highest sintering temperature i.e 1400ºC. Hence, it can be concluded that composition sintered at 1400ºC for 90 minutes holding time for 10wt% TiO_2/15wt% TCP and 75wt% ZrO_3 showed one of the best density values and would be chosen as the best optimised variables and composition. Both sintering temperature and holding time play a crucial role in increasing the bulk density. This is because during the process of sintering, the samples are fired at high temperatures causing the molecule particles to move close to each other. Hence, the samples go through shrinkage resulting in reduced porosity. Therefore, this directly contributes to the increase in Bulk density values.

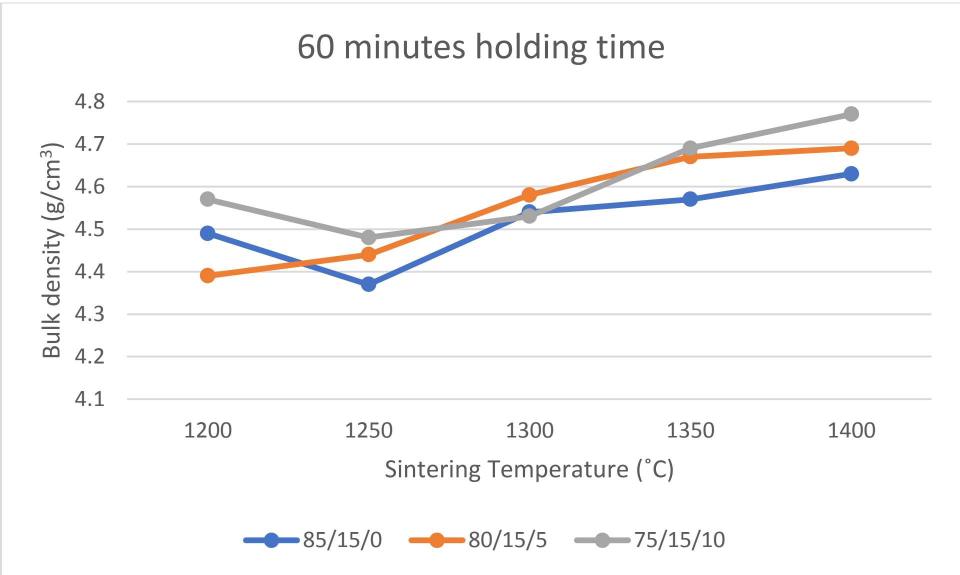

Figure 2: The effect of Bulk density on sintering temperature for 60 minutes holding time

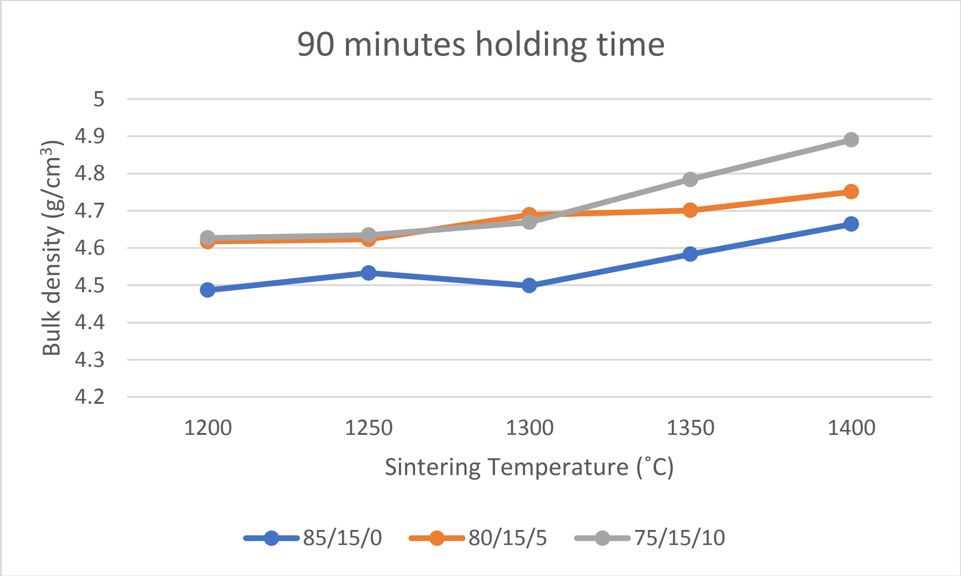

Figure 3: The effect of Bulk density on sintering temperature for 90 minutes holding time

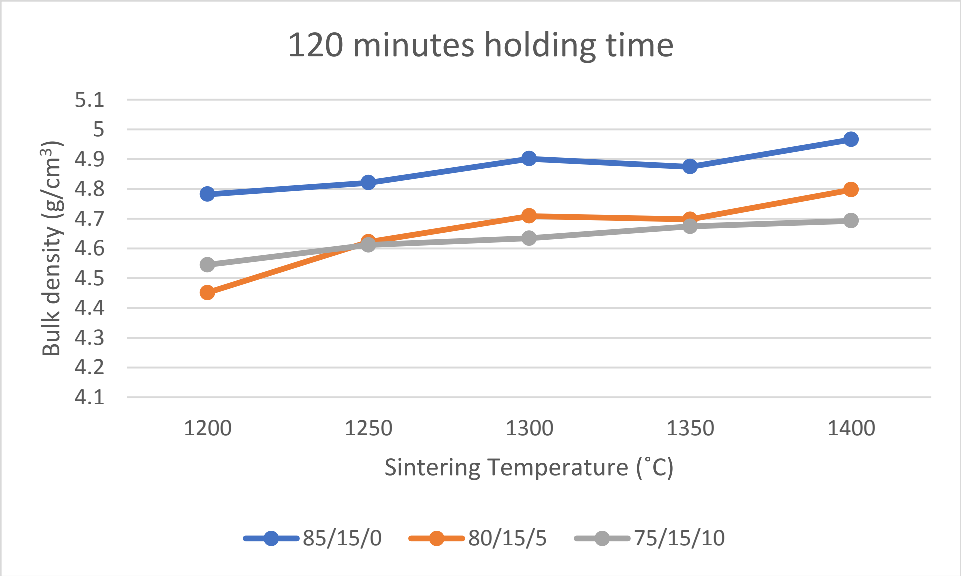

Figure 4: The effect of Bulk density on sintering temperature for 120 minutes holding time

4.2 Vickers Hardness

Generally, it is known that the Vickers hardness and Bulk density results are co-related and directly proportional to each other. Whereby, the Vickers hardness of the sintered samples were investigated, in relation to sintering temperatures, holding times and additive contents, in this research work. The effect of Y-TZP-TiO_2-TCP composites sintered at 60, 90, 120 minutes holding times for temperatures ranging from 1200℃ to 1400℃ are shown in Figures 5, 6 and 7. The Vickers hardness results varied in a similar trend as the Bulk density, as all compositions displayed higher Vickers hardness values, at higher sintering temperatures especially sintered at 1400℃. The highest Vickers hardness result, 11.35 GPa was obtained for the composition of 0wt% TiO_2/15wt% TCP and 85wt% ZrO_3 sintered at 1300ºC for 120 minutes holding time. This result recorded about 87% of the theoretical value of Vickers hardness for Y-TZP (13 GPa). On the other hand, the composition of 10wt% TiO_2/15wt% TCP and 75wt% ZrO_3 sintered at 1400ºC for 90 minutes holding time attained a Vickers hardness value of 10.69 GPa. This result recorded about 82% of the theoretical value of Vickers hardness for Y-TZP, which is also quite close to the Vickers hardness value obtained for the 0wt% TiO_2/15wt% TCP and 85wt% ZrO_3 composition. The results obtained showed that the 1400ºC sintering temperature, 90 minutes holding time and 10 wt% of TiO_2 content enhanced the hardness of Y-TZP. The obtained results for high density reveal the crystalline structure and bond between the grains of the sintered composites are strong. Therefore, this finding lead to higher Vickers hardness and strength.

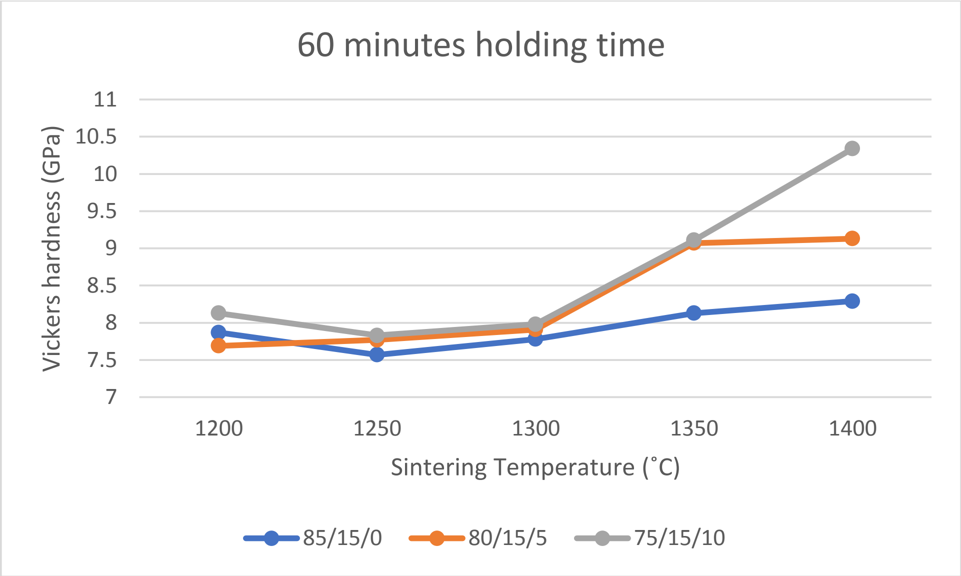

Figure 5: The effect of Vickers hardness on sintering temperature for 60 minutes holding time

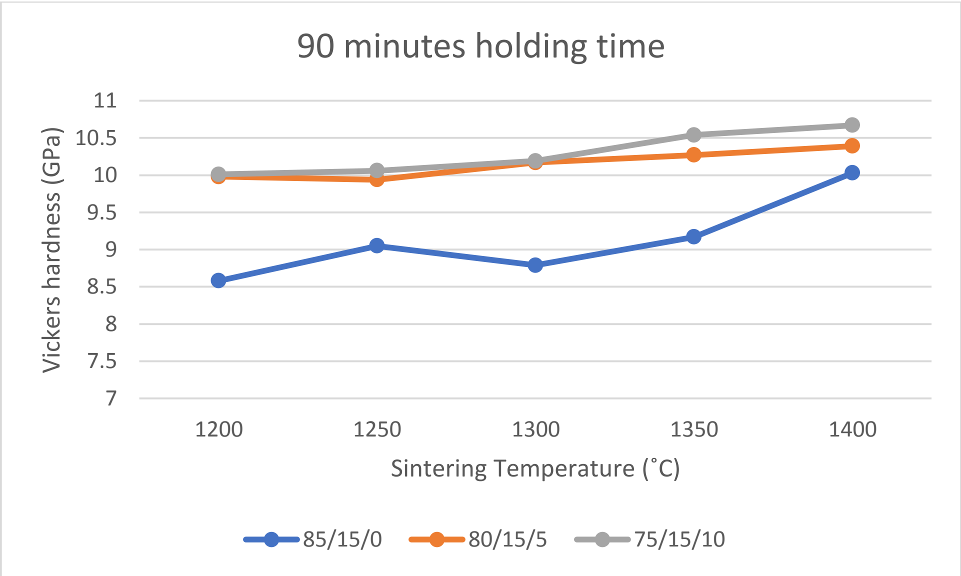

Figure 6: The effect of Vickers hardness on sintering temperature for 90 minutes holding time

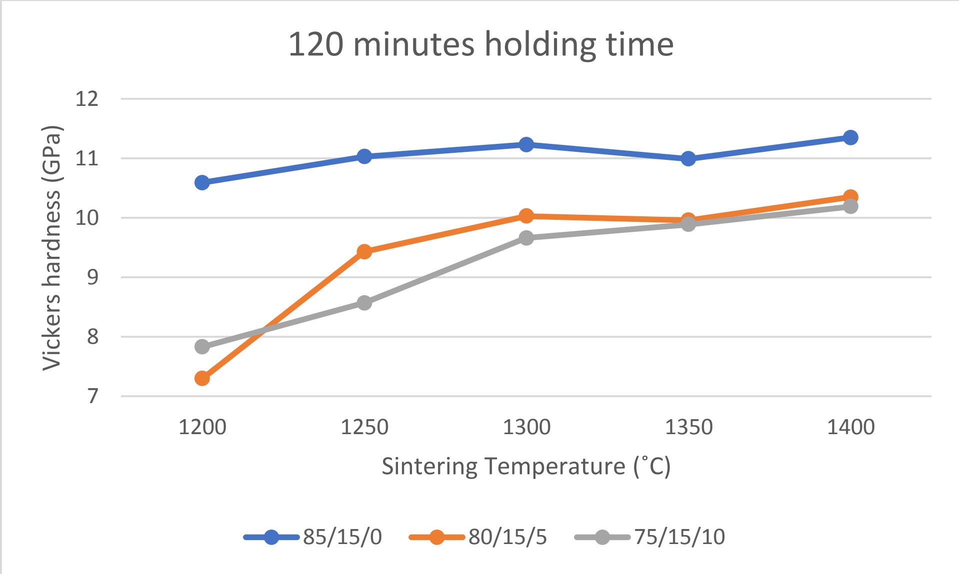

Figure 7: The effect of Vickers hardness on sintering temperature for 120 minutes holding time

5. Conclusion

In this research work, the effect of holding times on the TiO_2 and TCP additions was investigated to improve the Bulk density and Vickers hardness of Zirconia. The obtained results show that 90 minutes holding time significantly enhanced the mechanical properties at 1400ºC for the composition of 10wt% TiO_2/15wt% TCP and 75wt% ZrO_3. The density obtained was about 4.89 g/cm^3 approximately 82% of the theoretical density value (6.1 g/cm^3) of zirconia and the Vickers hardness obtained was 10.69 GPa, about 82% of the theoretical value of Vickers hardness (13 GPa) for zirconia. In addition to that, the composition of 0wt% TiO_2/15wt% TCP and 85wt% ZrO_3 sintered at 1300ºC for 120 minutes holding time marked the highest density and hardness values, whereby this results also can be considered as preferred results due its good mechanical properties. In a nutshell, sintering at high temperatures (1350-1400ºC) with a holding time of 90 minutes was seen to be the optimum temperature and holding time to achieve the best mechanical properties. Sintering at 60 and 120 minutes holding time was found not significantly enhancing the mechanical properties.

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