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Spark plasma sintered Ti6Al4V-ZrO₂ bio-composites: optimization of ball milling and turbula mixing of powders and their subsequent sintering parameters

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Abstract. The use of Ti6Al4V as a biomaterial has been limited due to the dissociation of V and Al ions into the surrounding environment which can cause inflammations, cell damage and toxicity. To prohibit these undesirable challenges, the addition of ZrO₂ into Ti6Al4V was proposed because ZrO₂ is biocompatible, it has good wear and corrosion resistant properties, it has low affinity to bacterial colonization and it has high fracture toughness. The aim of this work is to determine the optimum powder synthesising parameters by Tubular mixer and ball mill for fabrication of sintered composites. Powder mixtures were prepared by 49 rpm, 72 rpm and 74rpm for 2, 4 and 8 hours followed by spark plasma sintering at 1100 °C at heating rate of 100 °C/min for 10 min and pressure of 50 MPa. From the results obtained it was discovered that Tubular mixing of 90 Vol% Ti6Al4V and 10 Vol% ZrO₂ at 72 rpm and ball milling of 90 Vol% Ti6Al4V and 10 Vol% ZrO₂ at 74rpm speed have proved to produce homogeneous admixed powders, which can further produce fully dense SPS compacts with higher microhardness values.

1. Introduction

Ti6Al4V is a grade of titanium alloy which is considered suitable for the fabrication of implants. This alloy was developed to improve the properties of commercially pure titanium such as low strength and wear resistance for hard tissue replacements [1]. Ti6Al4V properties range from good biocompatibility, good mechanical properties to good corrosion and wear resistance as such, it was considered a superior metallic biomaterial compared to cobalt-alloys and stainless [2]. However, it was discovered by Manivasagam [3] that under a biological environment such as the human body it releases toxic metal ions of vanadium and aluminium thus leading to diseases such as Alzheimer. This release of toxic ions occurs even though the metal is protected by the surface oxide layers from the environmental attack; as such this was a clear indication that the corrosion resistance of Ti6Al4V is not sufficient and may result in the implant failure. To hinder the release of these ions and the formation of debris which can go into the blood, the addition of ZrO₂, which possesses high fracture toughness, high wear resistance, high strength and excellent biocompatibility to Ti6Al4V was



proposed [3]. The bio-composite was fabricated by Spark Plasma Sintering, a powder metallurgy technology based on simultaneous application of high temperature, high axial pressure, and low-mode (low voltage, high current) field (plasma) associated with an electric current on powders through a graphite die and plungers. In comparison with the conventional sintering methods, it offers products with fine microstructures and high density at relatively low sintering temperatures, very short sintering times and easy control of the sintering conditions [4]. Hence the work was aimed at studying the effect of the ad-mixed and milled powder metallurgy preparation methods on the densification and microhardness of Ti6Al4V-ZrO₂ bio-composites produced via SPS.

2. Materials and Methods

Ti6Al4V (average particle size of 18.94 μ m) and ZrO₂ (65, 26 μ m) powders were utilized as the starting materials for this research. The two powders were mixed and milled in a dry environment using a Turbular shaker mixer (T2F) and planetary ball mill (RETSCH PM 400 MA-type) at a speed of 49 and 72/74rpm for 2, 4 and 8 hours with the powder ratio of 90Vol%Ti6Al4V and 10Vol%ZrO₂. The morphology and powder particle size distribution (PSD) of the mixed and milled powders were examined using scanning electron microscope (SEM) and particle size analyser (Microtrac s3500). The amount of mixed powders required to produce a 40mm diameter and 5mm thickness of Ti6Al4V-ZrO₂ Nano structured bio-composites were poured into graphite die and then sintered using a spark plasma-sintering machine (HHPD-25 FCT GmbH Germany).

The optimized powders were sintered at 1100°C at a heating rate of 100°C/min and 50Mpa pressure in vacuum condition. The relative densities of the samples were measured using an OHAUS density kit and by Archimedes principle of the starting powders constituents using the rule of mixtures. The samples were prepared for metallography following the metallographic standard procedure prescribed for grinding and polishing for Ti6Al4V-ZrO₂. The samples were etched using kroll etchant for 30 seconds and the microstructure was analysed using a (Tescan) scanning electron microscope. The Vickers micro hardness (Hv20) was measured by a micro-hardness tester machine (model falcon 500 series) whereby each sample was indented 10 times. The samples were labelled based on the ZrO₂ ratio added to Ti6Al4V, the mixing/milling speed, the mixing/milling time and the type of mixing process. For example, 10-49-2T means that this sample has 10Vol% ZrO₂ added, it was mixed at 49rpm for 2 hours in a Turbula mixer and 10-49-2B means the sample has 10Vol% ZrO₂ at a speed of 49rpm for 2 hours in a ball mill.

3. Results and Discussion

3.1 SEM results

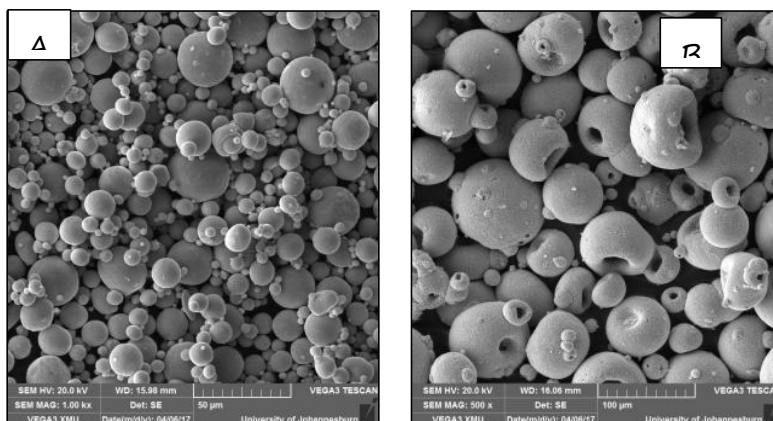


Figure 1: SEM Micrographs of as-received (a) Ti6Al4V and (b) ZrO₂ powders.

The SEM results of as-received powders are shown in figure 1, revealed that the Ti6Al4V powder is spherical and non-porous while the ZrO₂ powders are doughnut shaped, hollow and porous.

3.1.1 Turbular mixing.

Figure 2 illustrates SEM Micrographs of (a)10-49-2T ,(b) 10-49-4T, (c) 10-49-8T, (d)10-72-2T,(e)10-72-4T and (f)10-72-8T. In the obtained results, it is evident that the doughnut-like shape of ZrO₂ particles was strongly broken down in all Turbula admix powders due to the combination of motion of rotation, translation and inversion as well as the impact of the balls that led to the effective reduction of the particle size of the product during mixing. The ZrO₂ particles are homogeneously distributed in the Ti6Al4V particles. Furthermore, an interesting trend discovered in these SEM results was the powders became more homogenous with an increase in speed and time from 49-72rpm and 2-8 hours.

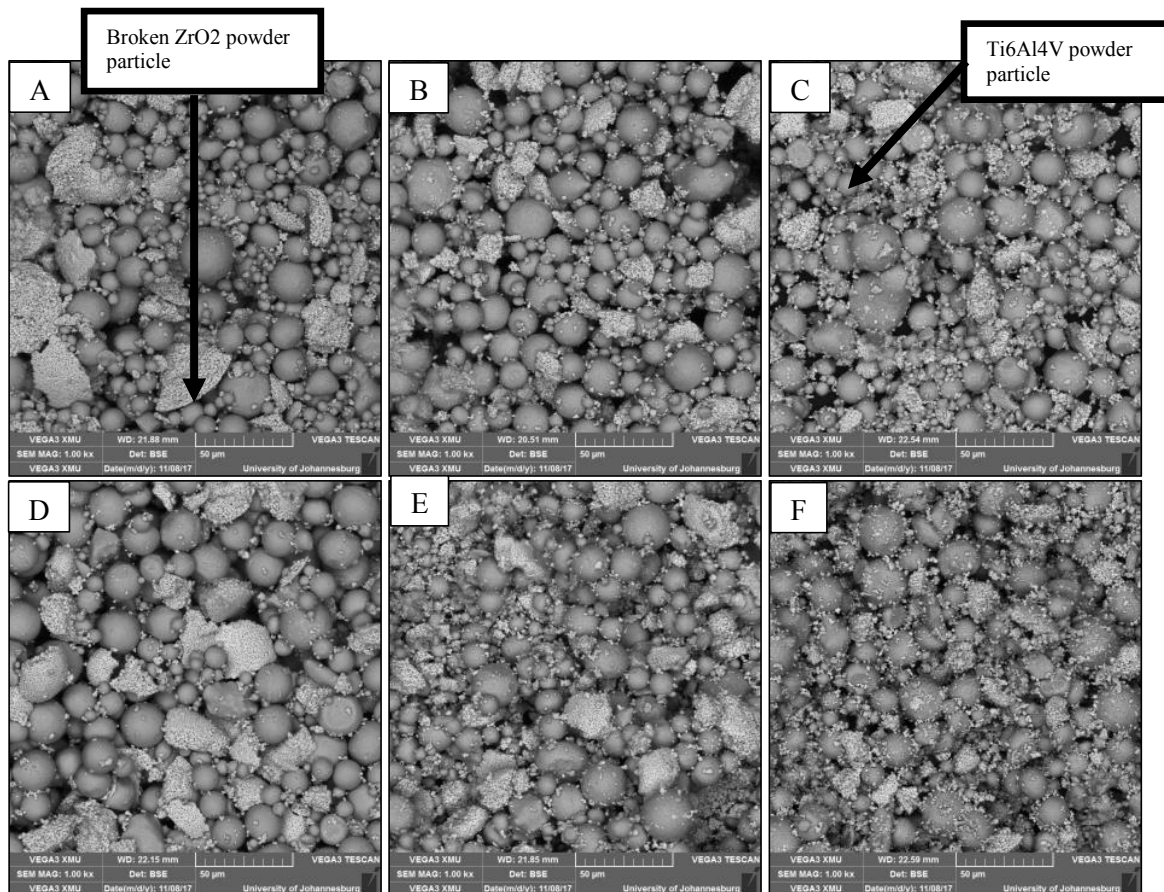


Figure 2: SEM Micrographs of (a)10-49-2T,(b)10-49-4T,(c)10-49-8T,(d)10-72-2T,(e)10-72-4Tand (f) 10-72-8T.

3.1.2 Ball milling

Figure 3 illustrate the SEM Micrographs of (a)10-49-2B,(b)10-49-4B,(c)10-49-8B,(d)10-74-2B,(e)10-74-2B and (f) 10-74-8B. An interesting trend discovered in these results was that satellites of Ti6Al4V have agglomerated in the unbroken hollow ZrO₂ powder particles, this trend is more evidential in

(c)10-49-8B, additionally the results indicate that the ZrO_2 powder particles are gradually homogeneously distributed in the Ti6Al4V particles as the speed and time of milling increases. According to these results, it is evident that ZrO_2 powder particles are also broken down during ball milling but only at high speeds such as 74rpm.

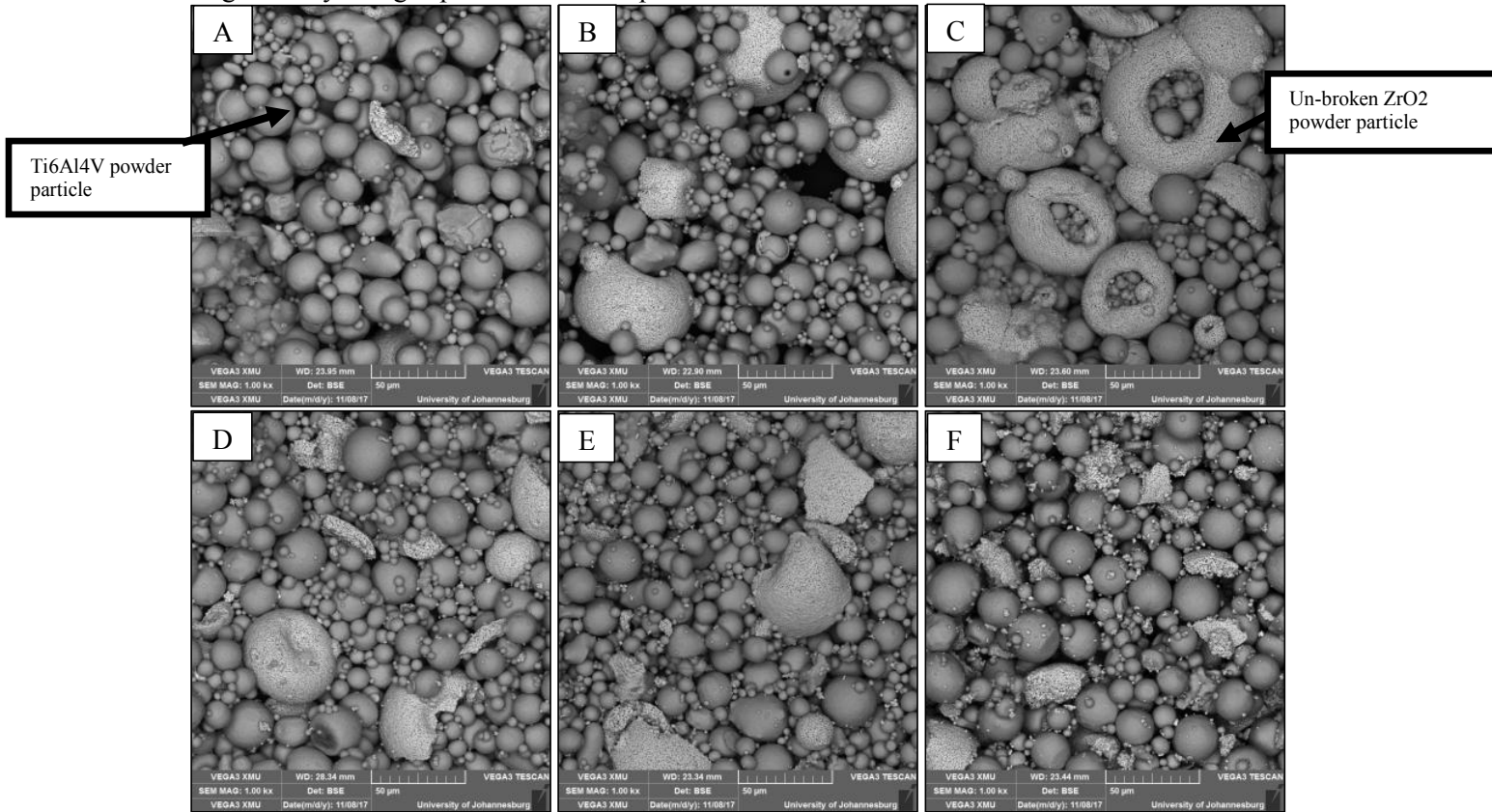


Figure 3: SEM Micrographs (a)10-49-2B,(b)10-49-4B,(c)10-49-8B,(d)10-74-2B,(e)10-74-2B and (f) 10-74-8B.

3.2 PSD results

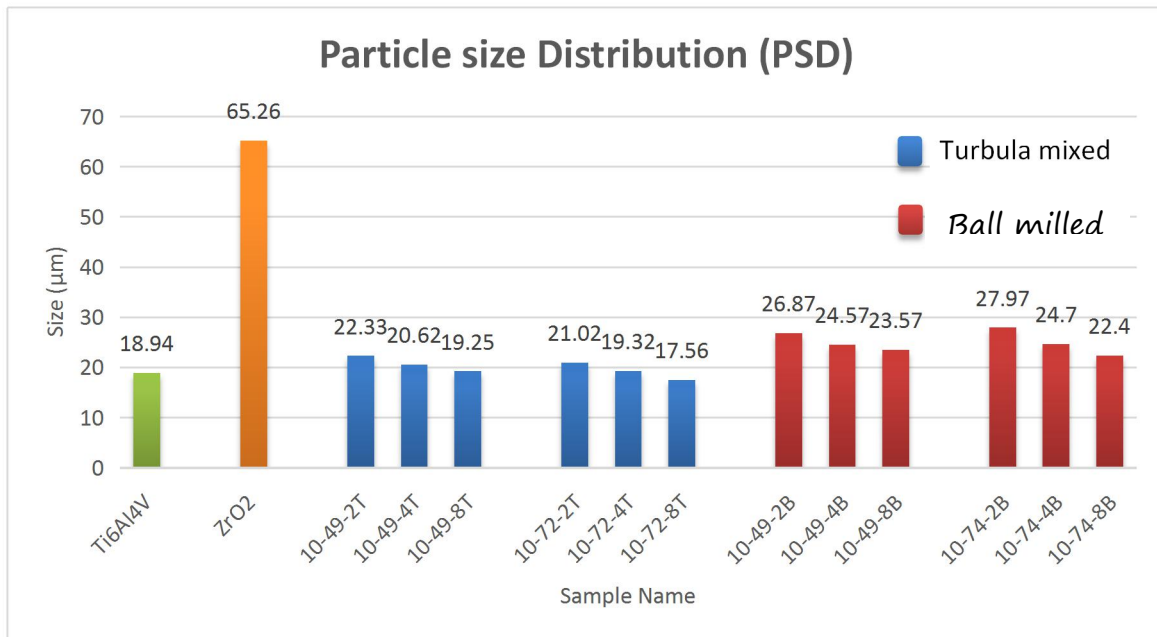
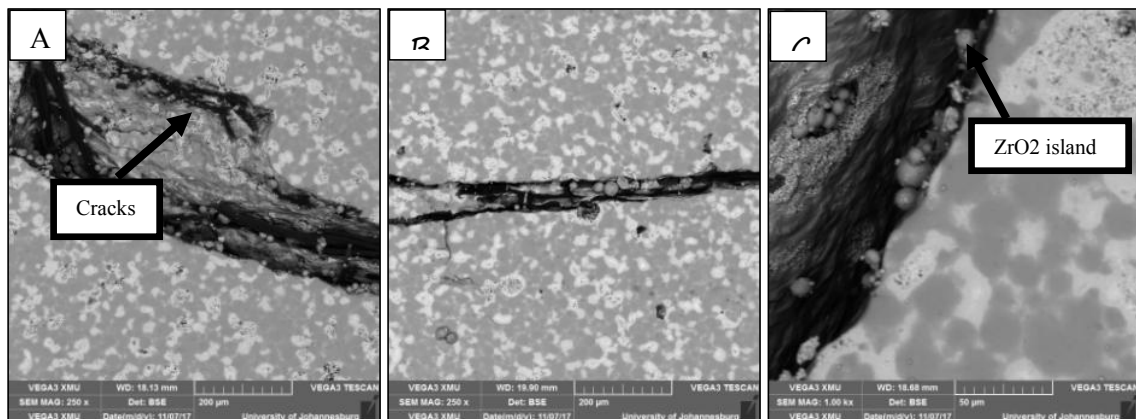


Figure 4: shows the particle size distribution of received powders and mixed powders

The obtained PSD results showed that the ZrO₂ powder has coarse particle size of 65 μm while Ti6Al4V has fine particle size of 19 μm as shown in figure 4. It was discovered that the PSD results compliments the SEM results whereby the Turbula mixed powders have the lowest values of average particle size than that of ball milled powders (illustrated in figure 4). A pattern was also discovered in these results where there is a continuous reduction in powder particle size reduction as we increase mixing/milling speed and time. Moreover, the SEM results alongside with the PSD results indicate that 72 rpm and 74rpm are the optimum mixing/milling speed since they produce powders with low particle size distribution than powders mixed/milled at 49rpm. The samples from the optimised speeds of 72rpm and 74rpm were considered to be the best choice to sinter Ti6Al4V-ZrO₂ bio-composites.

3.3 Sintered Composites Results

SEM Micrographs of sintered Ti6Al4V-ZrO₂ composites, which were mixed and milled at the speed of 72rpm and 74rpm, are illustrated by figure 5. The microstructure of all sintered samples contains a pool of Ti with island of ZrO₂ and they also contain micro pours that lead to crack propagation during metallographic preparation.



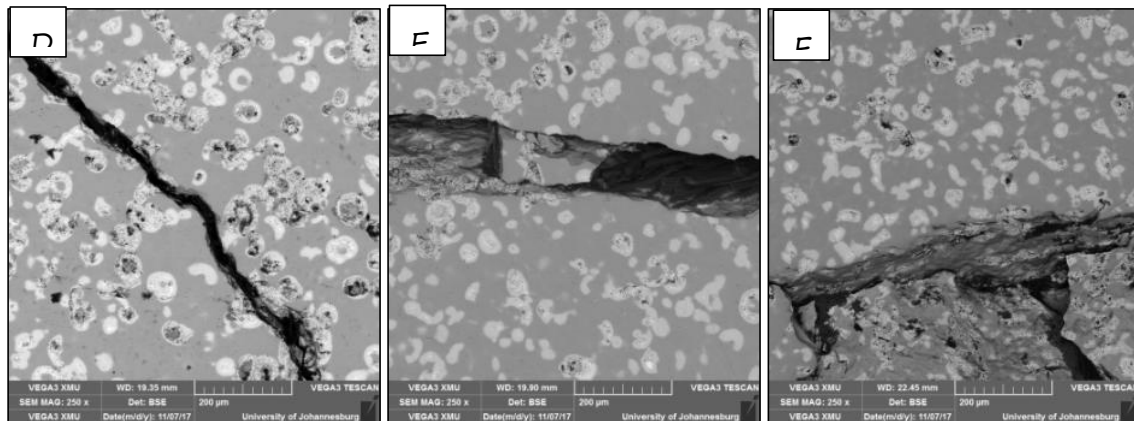


Figure 5: SEM Micrographs of sintered composites (a) 10-72-2T, (b) 10-72-4T, (c) 10-72-8T, (d) 10-74-2B, (e) 10-74-4B and (f) 10-74-8B.

3.4 Densification

Table 1 illustrates that density increases with increasing sintering temperature, as porosity decreases when atoms are bonded together during sintering. Considering the above statement, this could be one of the reasons that the standard sample (Ti6Al4V) sintered at 1000 has a low densification value of 99.6% and the Ti6Al4V-ZrO₂ bio-composite samples sintered at 1100 have high densification values of 100%.

Table 1: Densities of Sintered samples.

Sample name	Holding Time, min	Pressure, MPa	Heating Rate, °C/min	Sintering temperature (°C)	Densification %
Standard sample (Ti6Al4V)	10	50	100	1000	99.6
10-72-2T	10	50	100	1100	100
10-72-4T	10	50	100	1100	100
10-72-8T	10	50	100	1100	100
10-74-2B	10	50	100	1100	100
10-74-4B	10	50	100	1100	100
10-74-8B	10	50	100	1100	100

3.5 Hardness

Figure 6 shows the microhardness of sintered composites (10-72-2T, 10-72-4T and 10-72-8T) and (10-74-2B, 10-74-4B and 10-74-8B). The results show that mixed samples have the highest microhardness values than the ball-milled samples. A trend was observed where microhardness values increase as the mixing and milling time increases from 2 to 8 hours. This could be due to high powder homogeneity as speed and time increased and thus producing a well-dense sintered sample. Theoretically, sintering rates increase with increasing reduction in powder particle size, hence the increase in microhardness values as the mixing time increases.

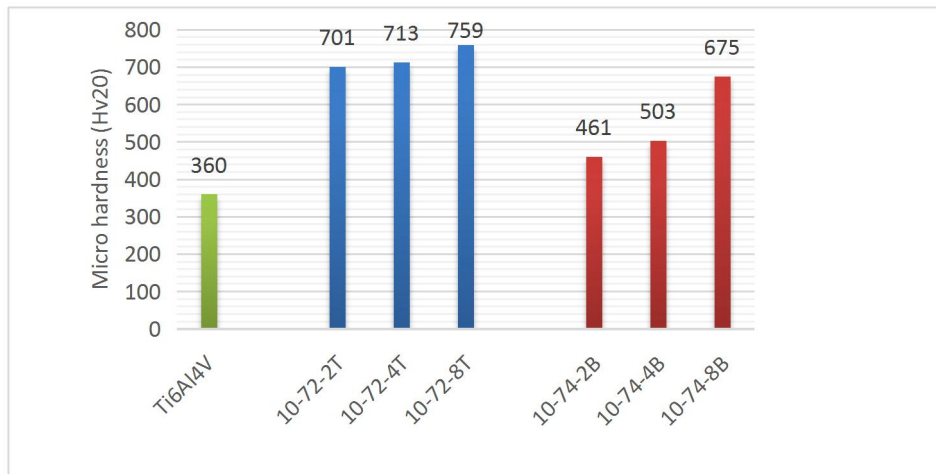


Figure 6: Hardness values of sinterd samples.

4. Conclusion

In conclusion Turbular mixing using balls has proved to be more effective than ball milling in producing homogeneously distributed powder mixtures with finer particles, which can be facilitated in the production of fully dense composites. Furthermore, the optimisation of mixing, milling and sintering parameters done in this paper could be utilised as a reference during further work based on Ti6Al4V- ZrO₂ bio-composites.

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