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Dependence of Densification, Hardness and Wear Behaviors of Ti6Al4V Powders on Sintering Temperature

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***Abstract*—**The sintering step in powder metallurgy (P/M) processes is very sensitive as it determines to a large extent the properties of the final component produced. Spark plasma sintering over the past decade has been extensively used in consolidating a wide range of materials including metallic alloy powders. This novel, non-conventional sintering method has proven to be advantageous offering full densification of materials, high heating rates, low sintering temperatures, and short sintering cycles over conventional sintering methods. Ti6Al4V has been adjudged the most widely used α+β alloy due to its impressive mechanical performance in service environments, especially in the aerospace and automobile industries being a light metal alloy with the capacity for fuel efficiency needed in these industries. The P/M route has been a promising method for the fabrication of parts made from Ti6Al4V alloy due to its cost and material loss reductions and the ability to produce near net and intricate shapes. However, the use of this alloy has been largely limited owing to its relatively poor hardness and wear properties. The effect of sintering temperature on the densification, hardness, and wear behaviors of spark plasma sintered Ti6Al4V powders was investigated in this present study. Sintering of the alloy powders was performed in the 650–850°C temperature range at a constant heating rate, applied pressure and holding time of 100°C/min, 50 MPa and 5 min, respectively. Density measurements were carried out according to Archimedes' principle and microhardness tests were performed on sectioned as-polished surfaces at a load of 100gf and dwell time of 15 s. Dry sliding wear tests were performed at varied sliding loads of 5, 15, 25 and 35 N using the ball-on-disc tribometer configuration with WC as the counterface material. Microstructural characterization of the sintered samples and wear tracks were carried out using SEM and EDX techniques. The density and hardness characteristics of sintered samples increased with increasing sintering temperature. Near full densification (99.6% of the theoretical density) and Vickers' micro-indentation hardness of 360 HV were attained at 850°C. The coefficient of friction (COF) and wear depth improved significantly with increased sintering temperature under all the loading conditions examined, except at 25 N indicating better mechanical properties at high sintering temperatures. Worn surface analyses showed the wear mechanism was a synergy of adhesive and abrasive wears, although the former was prevalent.

***Keywords*—**Hardness, powder metallurgy, Spark plasma sintering, wear.

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I. INTRODUCTION

THE growing needs for the use of high strength and lightweight materials for guaranteed fuel efficiency in the automobile and aerospace industries have attracted enormous research activities on Ti-based alloys. These alloys are indispensable due to their high specific strength, exceptional corrosion resistance and excellent mechanical properties [1], [2]. Among these group of alloys, Ti6Al4V over the years has been the most widely used α + β titanium alloy due to its competitive and comprehensive mechanical performance in engineering service environments [3]. However, applications of the alloy have been limited largely by its relatively low hardness and wear properties [1]. Consequently, materials scientists and engineers have made different attempts at improving the hardness and wear performance of Ti6Al4V alloys in service. One such attempt is the use of the powder metallurgy (P/M) route in the fabricating of parts made from Ti6Al4V alloys. This method of fabrication offers competitive advantages of low cost, material loss reductions, ability to produce near net/intricate shapes, as well as producing components with impressive mechanical properties [3].

The sintering step in P/M processes is very sensitive as it determines to a large extent the properties of the final component produced. Spark plasma sintering (SPS) is a promising and relatively novel technique associated with P/M processes extensively used in consolidating varieties of metallic alloy powders. This non-conventional sintering method offers full densification of materials, high heating rates, low sintering temperatures, and short sintering cycles over the conventional sintering methods. Processing parameters however need to be carefully chosen and controlled during consolidation processes to fabricate fully dense alloys possessing the required enhanced mechanical properties [3], thus this present work investigated the dependence of densification, hardness and wear behaviors of spark plasma sintered Ti6Al4V powders at varied sintering temperatures.

II. MATERIALS AND METHODOLOGY

A. Ti6Al4V Powders

The starting material used in this study was Ti6Al4V powders having a particle size of 100 mesh received in pre-alloyed condition from Flowmaster^{TM}.

B. Sintering Experiments

Measured amount of alloy powders were poured into a graphite die with a diameter of 30 mm and sintered in consecutive runs using the SPS system, model HHPD – 25 FCT Germany at temperatures ranging from 650–850°C at a constant heating rate, applied pressure and holding time of 100°C/min, 50 MPa and 5 min, respectively. Discs of sintered samples (Ø 30 mm x 5 mm thick) were obtained covered with graphite; these were subsequently ground off the sintered alloy.

C. Density Measurements

The densities of sintered samples were measured following Archimedes' principle at a room temperature of 23°C, where water density was considered as 0.997538 g/mL. The recorded density was an average of at least five measurements carried out on each sample. The relative density was thereafter calculated as a function of both the theoretical and measured densities of the Ti6Al4V alloy.

D. Hardness Testing

Vickers microhardness tests at room temperature were performed on sectioned as-polished surfaces at a load of 100 gf (1.0 N) and dwell time of 15 s using the FUTURE – TECH FM 800 microhardness tester. The test results for each sample was the arithmetic mean of five successive indentations.

E. Wear Tests

Dry sliding wear tests were performed at varied sliding loads of 5, 15, 25 and 35 N under room temperature and humidity with frequency set at 5 Hz (300 rpm) using a UMT – 2 – CETR tribometer. Wear test samples were cut into 2 cm x 2 cm x 0.3 cm and polished to a mirror finish using a SAPHIR 520 auto-polisher. The ball-on-disc configuration with tungsten carbide (WC) as the counterface material was used. The coefficient of friction (COF) and wear depths were monitored continuously under sliding condition for a time span of 1000 s.

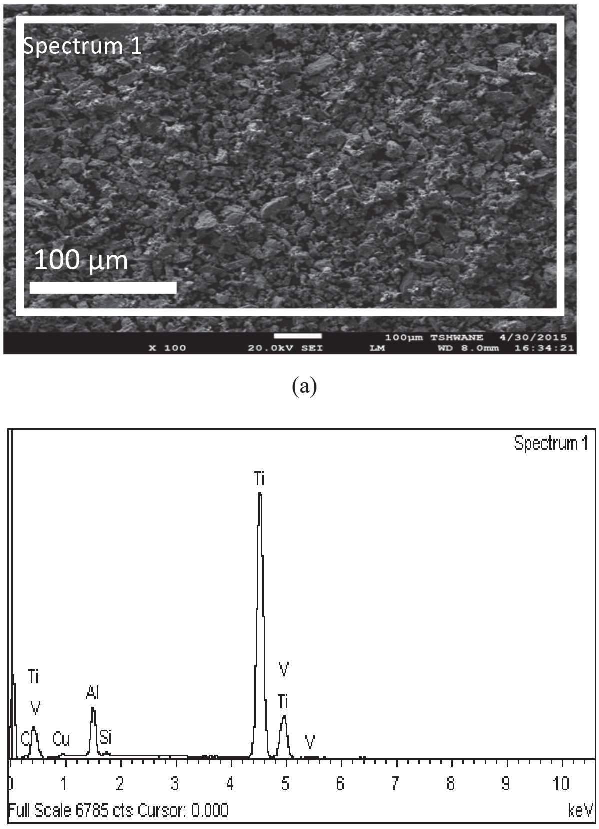
F. Characterization of Samples

The morphology of as-received pre-alloyed Ti6Al4V powder, microstructural characterization of as-polished sintered samples and wear track studies were achieved using field emission scanning electron microscope (FESEM, JSM-7600F, Jeol, Japan) equipped with energy dispersive X-ray spectrometer (EDS). The polished samples were etched with Kroll reagent (6 ml HF, 12 ml HNO3 in 150 ml H2O) before SEM analyses were carried out on them.

III. RESULTS AND DISCUSSION

A. As-received Powder Characterization

The as-received Ti6Al4V powder as characterized by SEM and EDS, respectively, (Fig. 1), showed the powder particles were irregular and angularly shaped, while the corresponding EDS analysis showed the powder contained traces of C, Si and Cu aside the main elements (Ti, Al and V) from which the alloy was formed.



(a)

(b)

Fig. 1 Characterization of as-received Ti6Al4V powder (a) SEM and (b) EDS

B. Densification Mechanism, Microstructural Evolution and Hardness of Sintered Samples

Fig. 2 shows the densification mechanisms of the samples during sintering as retrieved from the SPS system. Near full densification and maximum shrinkage was attained at 850°C.

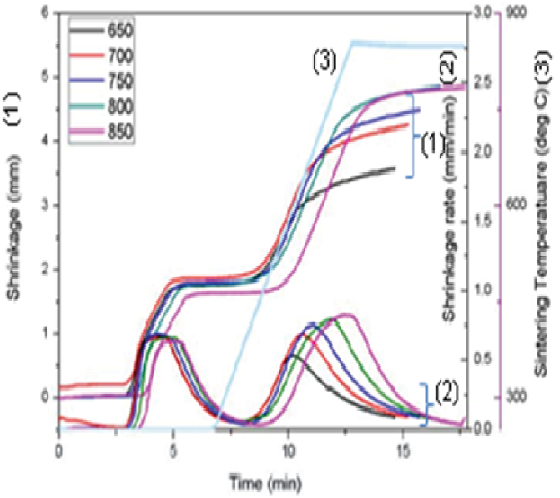


Fig. 2 Sintering profile of sintered samples

As proposed by [4], [5], four distinct regimes of activities occur during a sintering process, these were identified as: 1)

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activation and refining of powder, 2) formation of sintering neck, 3) growth of sintering neck and 4) plastic deformation densification. All these regimes are obvious on the sintering profile obtained (Fig. 2). Extensive sintering and densification took place at the fourth stage where maximum shrinkage was also achieved. The recorded relative density, calculated as a function of both the theoretical and measured densities of the sintered samples (Fig. 3) was enhanced by 14% as the sintering temperature increased from 650°C to 850°C suggesting densification of sintered samples improved with increased sintering temperature. The Vickers microhardness values also increased from 168 HV at 650°C to 360. 5 HV at 850°C, translating to about 53% enhancement in the measured hardness of samples as the sintering temperature increased. This, according to [6] was attributed to the decrease in porosities with increased sintering temperature (Fig. 4) and the associated increase in density.

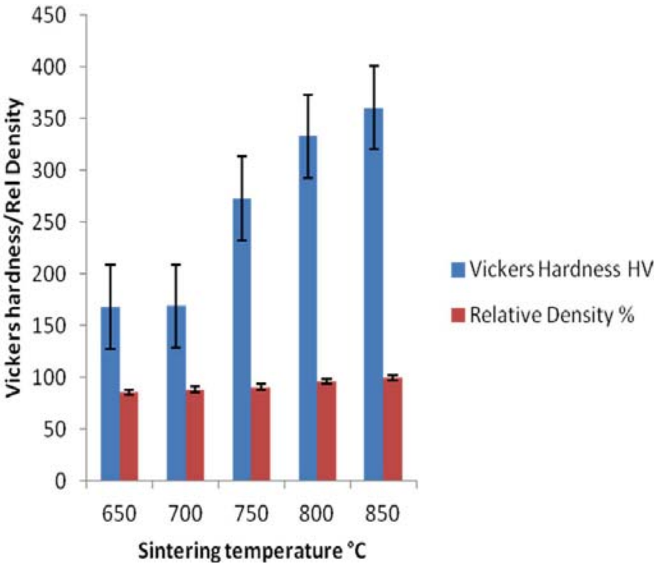

Fig. 3 Vickers microhardness and relative density at varying sintering temperatures

Fig. 4 shows the microstructural evolution during the sintering of the pre-alloyed Ti6Al4V powders. At 650°C (Fig. 4 (a)), a porous structure characterized with open pores (seen as dark spots) located at the grain boundaries was observed. Basket weave, (otherwise described as acicular α+β structure) characteristic of Ti6Al4V alloys were observed at 650 and 750°C (Figs. 4 (a) and (c)) respectively. The proportion of each phase was dependent on the sintering temperature. The α phase was more prevalent at lower temperatures (650 – 750 °C) and being less dense than the β phase, coupled with the high level of porosity evident in the microstructure after sintering at these temperatures, explains the lower density of the samples sintered at 650 and 700°C respectively. A reduction in pore size and its density was observed with increased temperature (Fig. 4). Samples sintered at lower temperatures (Figs. 4 (a)–(c)) still showed particulate features, the open pores were dominant at these temperatures but tend to close up from sintering temperature of 800°C with the pores disappearing almost totally at 850°C (Fig. 4 (e)) leading to the observed enhancement in densification. As pointed out by [6], small and intergranular pores do coalesce to form closed ones at higher sintering temperatures. The β phase (dark grey), as revealed by EDS, was more prominent at high temperature (850°C) and plates of the α phase lamella were situated within the β matrix. Diffusion bonding at particle boundaries in addition to other diffusivity paths is usually enhanced at high temperatures resulting in a re-orientation of the Ti6Al4V particles, thereby closing up the pores due to boundary sliding and grain rotation, and thus leading to an enhanced material density [6], [7].

C. Wear Behaviour

Fig. 5 shows the observed COF and wear depths of SPS sintered samples at different temperatures over a time span of 1000 s in dry sliding condition. COF was lowest at 850°C (almost zero) for all the applied loads except at 25 N where the lowest COF was found to be about 0.3 at 650°C and this was fairly constant from about 500–700 s. The COF increased thereafter to about 0.35 and finally dropped back to 0.3 at 1000 s. Generally, the trend of the COF is a decrease as the sintering temperature increased; this is expected, as the density and hardness of samples improved with increase in temperature. This wear behavior, as previously observed by [8], suggests that wear rate in the samples will be lowest at 850°C. Also, it could be inferred that wear resistance of the sintered alloy powders increased as sintering temperature increased with the highest resistance at 850°C. The reason for the reverse behavior observed with the sample sintered at 650°C under applied load of 25 N, as explained by [2], could be that the contact area between the sample and the WC counterface material had the highest interface temperature at this applied load level, thereby leading to a lower shear stress, which consequently led to reduced COF as against the values obtained at other temperatures under the same applied load. On the other hand, possibility exists that an oxide layer was formed during sliding of the counterface material on this sample at 25 N that becomes more resistant to wear as the dry sliding action progressed on the sample. This is expected as the COF was initially highest (above 0.4) but dropped to 0.3, and was fairly constant thereafter till about 700 s and then increased to 0.35 in a relatively stable manner before it finally dropped back to 0.3. This fluctuation in COF values implies a formation and breakage of the protective oxide layer as the sliding wear test progressed. According to [9], the surface of titanium gets oxidized during sliding conditions due to its poor thermal conductivity when the flash temperature reaches a critical value. This oxidized surface, in turn, either gets fragmented or becomes stable to a certain extent as the sliding progressed. Although, the COF value was higher at this load and sintering temperature compared to the values obtained at the other applied load levels with samples sintered at 850 °C, which means that comparatively wear rate is lowest and wear resistance highest at the highest sintering temperature of 850°C investigated.

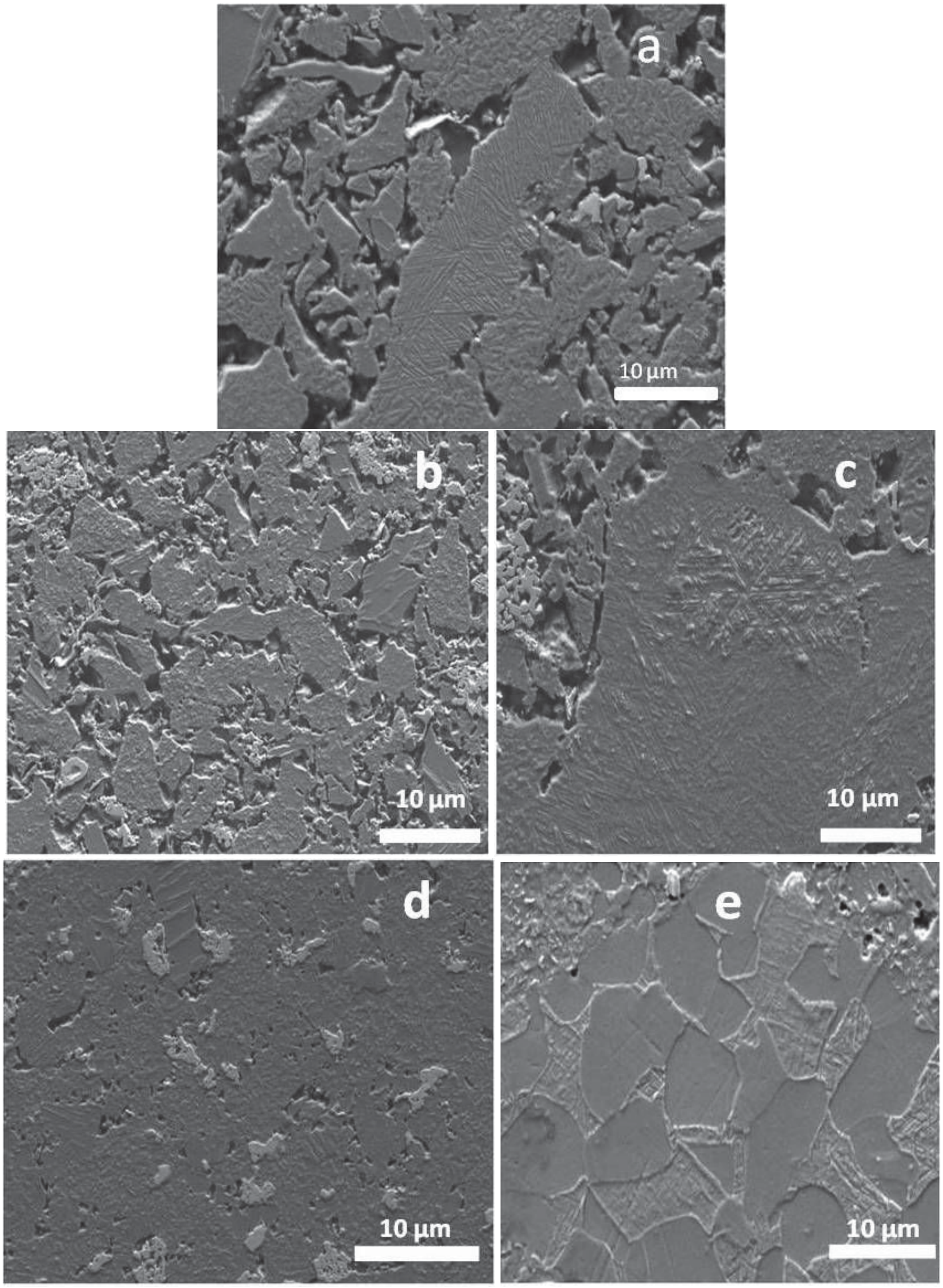

Fig. 4 SEM micrographs of samples sintered at varied temperatures (a) 650 °C (b) 700 °C (c) 750 °C (d) 800 °C (e) 850 °C

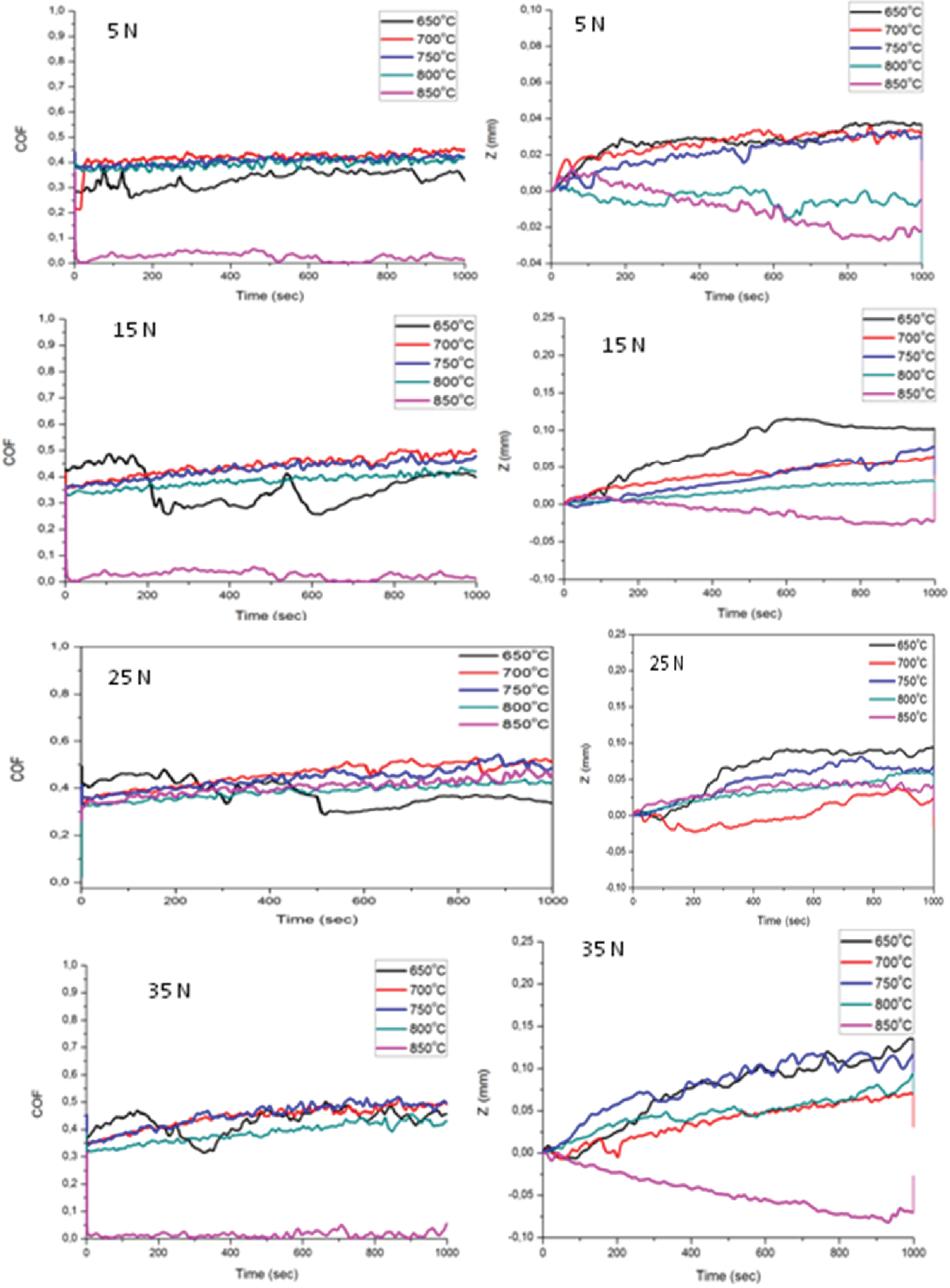

Fig. 5 Graphs of coefficient of friction (COF) and wear depths against time under varied applied normal loads and sintering temperatures

Generally, the observed wear depths in the sintered samples increased as the dry sliding wear test progressed from 0–1000 s for samples sintered at temperatures 650–800°C under applied normal loads 5, 15 and 35 N, respectively, with the highest gradient obtained at 35 N (Fig. 5). However, the wear depths drastically followed a decreasing trend for the sample sintered at 850°C under all the applied loads except at 25 N, where all the wear depths increased progressively throughout the period of sliding. Comparatively, the sample sintered at 650 °C had the highest wear depth under all the applied load levels studied. Therefore, it would be safe to conclude from these wear behaviour results that the wear rate in the sintered Ti6Al4V pre – alloyed powders decreased as the sintering temperature increased with the highest rate attained at 35 N applied normal load. Inherently, the sample sintered at 850°C had the highest wear resistance, while the lowest was obtained at 650°C. This wear behaviour trend is reasonable as the density and hardness values of samples, as previously discussed, increased as the sintering temperature increased.

Fig. 6 shows the microstructural analysis results on the wear tracks of samples sintered at 650 and 850°C, respectively, under 25 N applied normal load as obtained from SEM. The wear scar morphologies showed features of both abrasive and adhesive wears, though the former was prevalent at 850°C. The scars had features typical of grooves, abrasive furrows, brittle detachments, and delaminations. The sample sintered at 650°C had evidently deeper grooves in comparison with the sample sintered at 850°C, which also showed a smoother worn surface than the former. These features confirm the earlier position that wear resistance of sintered samples improved significantly as the sintering temperature was increased.

Fig. 7 shows that examined worn surfaces at 650°C had obvious features of microfragmentation at an applied load of 5 N but more of brittle detachment at 35 N. This shows that wear depth increased with increased applied normal load. These were in agreement with the results from similar work by [9]. At 850°C, abrasive wear was prevalent and the worn surfaces were smoother compared to the worn surfaces obtained at lower sintering temperature. Inferentially therefore, the tribological properties of sintered samples improved with increased sintering temperature. This follows from the fact that wear resistance was improved at high sintering temperature due to the higher hardness and lower coefficient of friction achieved at that temperature. These findings are also supported from an earlier investigation by [10]. Analysis of wear debris of sintered Ti6Al4V alloy samples by EDS showed mostly Ti – rich flakes with traces of Al. This indicates that the debris were purely from Ti6Al4V and not from the WC ball counterface.

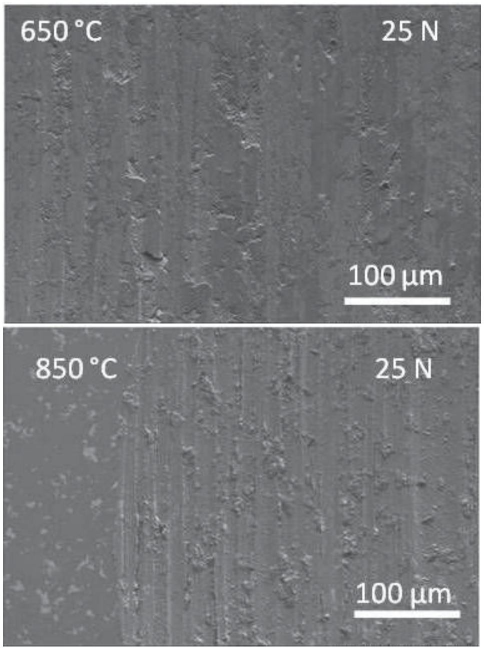

Fig. 6 SEM micrographs of worn sintered samples under 25 N applied normal load and varied sintering temperatures

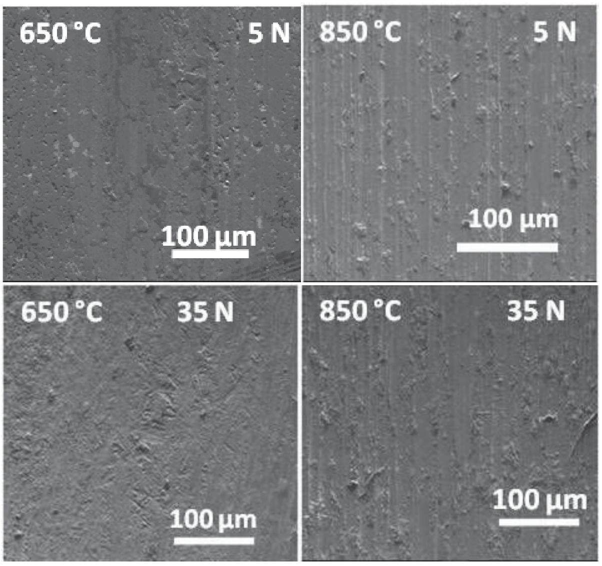

Fig. 7 SEM micrographs of worn sintered samples under different sintering temperatures and applied loads

IV. CONCLUSION

The dependence of the densification, hardness and wear behaviors of spark plasma sintered pre-alloyed Ti6Al4V powders on sintering temperature was investigated. The results obtained showed that the density and Vickers microhardness values were enhanced significantly by 14 and 53 %, respectively, as sintering temperature increased from 650 to 850°C. Generally, COF and wear depths also drastically improved with increased sintering temperature. Wear depths were directly proportional to the applied normal loads, while wear scar morphologies showed inherent wear mechanism was synergistically that of adhesive and abrasive wears, though the abrasive wear features were prevalent at 850°C.

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