Microstructure and mechanical characterisation of ZrO$_2$ reinforced Ti6Al4V metal matrix composites by powder metallurgy method

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Keywords: titanium metal matrix composites, powder metallurgy method, microstructure and mechanical characterisation, ZrO$_2$, mechanical properties

Abstract

This study investigated the mechanical and microstructural properties of ZrO$_2$ reinforced titanium metal matrix composites (TMMCs) fabricated using powder metallurgy. The base matrix of the Ti6Al4V alloy was reinforced with ZrO$_2$ at mixing proportions of different wt percentage points at 0, 3, 4, 5 and 6. Microstructure evaluation was carried out to study the bonding characteristics of the matrix and reinforcement, and it was confirmed that the reinforcement was homogenously mixed with the base matrix. The objective is to study the effect of zirconia on mechanical properties such as hardness, compression strength and thermal expansion coefficient of Ti6Al4V alloy. The hardness, compression strength, and shrinkage rate are increased with the increase of ZrO$_2$. Finally, it was observed that, 6 wt percentage of ZrO$_2$ reinforced composite showed better characteristics in that the hardness and compression strength were the highest among all the proportions used and the coefficient of thermal expansion was low. Due to these promising results, the fabricated ZrO$_2$ reinforced Ti6Al4V composite can be a potential material for structural, aerospace and automotive applications.

1. Introduction

Titanium matrix composites are popularly known for their excellent mechanical characteristics such as low weight to high strength ratio, high Young’s modulus, chemical resistance, promising wear resistance and manufacturability. Their application is widespread in the military, aeronautical, biomedical and automotive industries, especially in biomedical bone implants due to the low average life growth and high population. Bone implant materials are mostly metallic alloys, of which Ti alloys and stainless steel are widely used. However, Ti6Al4V is preferred because of its low elastic modulus compared with stainless steel [1–7]. On the other hand, Zirconia plays a significant role in reinforcements compared to other materials such as SiC [8], TiC [9], WC [10] and TiB [11], owing to its high strength, enhanced biocompatibility, high fracture toughness and high thermal shock resistance. It is also used as medical implants and prosthesis [8–13].

Most recently, Titanium was reported as the base matrix in composite research. Therefore, it is worthwhile to achieve a promising tailor-made property of novel composite material by using Zirconia as reinforcement. However, no such combination was reported as a composite to the author’s knowledge. The selection method for fabricating a new novel composite is also challenging. A conventional technology in fabricating TMMCs like casting involves high embodied energy, time and high cost. Moreover, it experiences problems of porosity, chemical reactions and wetting. Furthermore, it increases the difficulty in machining due to the presence of brittle and hard materials as reinforcement. The powder metallurgy method was chosen to overcome these
limitations due to the flexibility in direct fabrication of parts with the advantage of high precision in components. In recent years, this method has developed swiftly and used to manufacture complex structured mechanical components due to less material utilisation with the least expensive production. Furthermore, it enables the distribution of powder particles uniformly, eliminating clustering problems and porosity to a certain extent and saving energy [14–20].

This literature study shows that the typical applications of Ti6Al4V alloys and Zirconia match in most areas; the combined tailor-made properties of these composites would be required in those specific applications. Therefore, in this study, to investigate the characterisation of microstructure and mechanical properties such as hardness, compression strength and thermal expansion coefficient of this novel composite, the new mixture of Ti6Al4V-ZrO2 fabricated by powder metallurgy was examined with different mixing proportions of wt percentages of 0, 3, 4, 5 and 6 for enhancing the tailor-made properties of base matrix and reinforcement.

Table 1. Chemical composition of Ti6Al4V alloy (in %).

|   | Ti  | Al  | V   | Fe  | O   | C   | N   | H   |
|---|-----|-----|-----|-----|-----|-----|-----|-----|
|   | 87.6–91 | 5.5–6.75 | 3.5–4.5 | 0.040 | 0.20 | 0.080 | 0.050 | 0.015 |

Table 2. Mechanical properties of Ti6Al4V alloy and ZrO2.

| Material | Density (mg/m^3) | Hardness (VHN) | Tensile strength (MPa) | Compression strength (MPa) |
|----------|------------------|----------------|------------------------|----------------------------|
| Ti6Al4V  | 4.512            | 3730           | 1200                   | 1080                       |
| ZrO2     | 6.15             | 15750          | 711                    | 5200                       |

Table 3. Weight percentage of Ti6Al4V alloy and ZrO2.

| Specimen ID | Ti6Al4V | ZrO2 |
|-------------|---------|------|
| Pure Ti6Al4V | 100     | 0    |
| TMMC1       | 97      | 3    |
| TMMC2       | 96      | 4    |
| TMMC3       | 95      | 5    |
| TMMC4       | 94      | 6    |

Figure 1. Ball milling set-up.
2. Materials and fabrication method

Ti6Al4V alloy was used as a base matrix and ZrO2 as reinforcement to form a Titanium matrix composite. The chemical composition of Ti6Al4V alloy and the mechanical properties of both matrix and reinforcement are shown in tables 1 and 2. The TMCs were prepared with 0, 3, 4, 5 and 6 wt% of ZrO2 through powder metallurgy method and the weight proportions of Ti6Al4V alloy and ZrO2 powders are presented in table 3. Two steps carried out this method: compacting and sintering. Firstly, the Ti6Al4V alloy and ZrO2 powders were processed to blend and size powders in the ball mill. As per the weight ratio of base matrix and reinforcement, five different wt proportions of the powders were fed separately into the ball mill and set for a spindle speed of 600 rpm and a
duration of one hour to obtain the homogenous mixture of powders and the desired uniform particle size of powders. Secondly, compacting is done by compressing the metallic powders in a hydraulic operated die of press capacity 10 tons to obtain the desired shape. Thirdly, the sintering process in which the compacted specimens were heated in a box furnace at the temperature of 1400 °C was maintained as constant for 2 h. The powder particles were fused together during the sintering process due to the diffusion of atoms and boundary-crossing of atoms between the base matrix and reinforcement. The setup of ball milling, compacting, and sintering in the powder metallurgy method is depicted in figures 1–3. The sintered specimens are shown in figure 4.

3. Results and discussion

3.1. Microstructure evaluation

Figures 5(a) to (o) show the SEM image of pure Ti6Al4V alloy and TMMCs. It is clearly evident that the homogenous distribution of reinforcement particles ZrO2 in TMMCs is seen. It is observed in figures 5(a) to (c) that the bonding strength of Ti6Al4V alloy particles is increased due to elevated temperature maintained at
1000 °C in the sintering process. The large void present in the surface is due to the poor flow characteristics of metal powder during compaction. In figures 5(d) to (o) of TMMCs, it is observed that the reinforcement particles for TMMC4 and TMMC5 compared to TMMC2 and TMMC3 are strongly bonded with matrix alloy, and the voids are lesser due to the structural shape variation between the matrix and reinforcement powder particles and the increased percentage of reinforcement particles.

Table 4. Observed values during hardness test.

| Specimen ID | Trial 1 (HV) | Trial 2 (HV) | Trial 3 (HV) | Average (HV) |
|-------------|--------------|--------------|--------------|--------------|
| Pure Ti6Al4V | 100          | 102          | 105          | 102.33       |
| TMMC1       | 107          | 103          | 104          | 104.67       |
| TMMC2       | 120          | 118          | 115          | 117.67       |
| TMMC3       | 116          | 120          | 118          | 118          |
| TMMC4       | 126          | 130          | 128          | 128          |
3.2. Hardness test
The hardness of the TMMCs was carried out using Vickers’s hardness machine based on ASTM standards. 5 kg load was applied in the test for determining the hardness of the TMMCs. Three trials were conducted for each specimen during the test. The observed values for three trials and average values are presented in table 4. The graphical representation of the hardness test is depicted in figure 6. The figure clearly shows that the hardness increases with an increase in ZrO2 reinforcement from 0% to 6% due to the higher density and hardness of ZrO2. The percentage improvement for hardness from 0% to 6% is 23%.

3.3. Compression test
The reinforcement effect from 0% to 6% is depicted as a graphical representation in figure 7. The figure clearly shows that the increase in reinforcement from 0% to 6% leads to an increase in compression strength of TMMCs. The increased compressive strength is achieved due to the uniform dispersion of the reinforcement phase in the matrix. In addition, due to the higher sintering temperature of 1000 °C, the interfacial reaction between the matrix and reinforcement leads to greater diffusion of atomic particles.
3.4. Thermal expansion

The thermal expansion curves were obtained by testing the sintered cylindrical specimens at 5 °C per min heating rate. The temperature range for the measurement is between 30 °C–1000 °C by using a dilatometer in a...
helium atmosphere. Figures 8(a) to (e) shows the shrinkage rate variation (total percentage) and expansion coefficient of Ti6Al4V alloy and TMMCs as a function of heating temperature. The maximum temperature for transformation is maintained at about 1000 °C, increasing with ZrO2 content. Due to the beginning of bulk sintering, the minimum temperature corresponding to the front of a sudden drop is the sample length. From the figure 9, it is clear that the Zirconia percentage also influenced the shrinkage rate of the specimens. The lowest value is seen in pure alloy specimens. The addition of Zirconia increases the shrinkage rate. The linear dimensions of all samples were increased during the sintering. The characteristics of changes were different that shrinkage for pure alloy was linear up to 1000 °C while the TMMCs were curved.

4. Conclusion

In this study, the Ti6Al4V alloy reinforced with ZrO2 from 0 to 6 wt percentage was fabricated using the powder metallurgy method. Hardness, compression and thermal expansion coefficient were investigated for the TMMC specimens. The significant findings of the present study are summarised as follows.

- The addition of Zirconia into titanium alloy strongly transitioned the microstructural characteristics and mechanical properties. The microstructural examination confirmed the homogenous distribution of ZrO2 particles with Ti6Al4V alloy for all weight percentages and the strong interfacial bonding.
- The hardness and compression strength of the TMMC4 reinforced with 6% of Zirconia is higher than other TMMCs and pure Ti6Al4V alloy.
- The thermal expansion curves also were different for the pure titanium alloy and other TMMCs. The changes resulted from the existing transition temperature conversion of titanium alloy transformation. The expansion coefficient is increased with the increase of Zirconia wt percentage.
- The analysis of the sintering process and the characteristics temperature proved the effect of Zirconia addition on the onset and final temperature of shrinkage, which increased with increased Zirconia content.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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