Nanoindentation studies and analysis of the mechanical properties of Ti-Nb₂O₅ based composites

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Abstract. In this study, nanoindentation tests were used to evaluate the mechanical properties of spark plasma sintered Ti based composites containing 5, 10 and 15 wt.% Nb₂O₅, targeted for potential use as biomedical material. Nanoindentation tests were performed on the samples using indenter loads of 20 and 100 mN, while the microstructures were characterized using scanning electron microscopy. It was noted that with increasing Nb₂O₅ wt.%, there is transition from the lamellar structure of pure Ti to fully bimodal structures for the Ti-10 wt.% Nb₂O₅ and Ti-15 wt.% Nb₂O₅ composites. The hardness (6.0–40.67 GPa (20 mN) and 2.4–12.03 GPa (100 mN)) and reduced elastic modulus (115–266.91 GPa (20 mN) and (28.05–96.873 GPa (100 mN)) of the composites increases with increase in the Nb₂O₅ content, attributed to contributions of load transfer from the Ti matrix to the relatively harder Nb₂O₅ particles, particle and dispersion strengthening mechanisms. The elastic recovery index also improved with increase in Nb₂O₅ content, while the inverse was noted with respect to plasticity index. The elastic strain to failure and yield pressure both improved with increase in Nb₂O₅ content, which suggests that the antiwear properties and resistance to impact loading equally improves with Nb₂O₅ addition.

Keywords: titanium based composites / niobium pentoxide / nanoindentation / microstructure / mechanical properties / biomedical applications

1 Introduction

Presently, Ti has gained glowing reputation as specialist structural material for high tech and sensitive applications such as in aerospace, automobile and biomedical applications [1,2], which is largely due to the spectrum of properties possessed by Ti and its alloys. Load bearing properties such as high specific strength and stiffness, reasonable ductility [3,4]; resistance to environment induced failures such as high corrosion resistance [5]; and biological qualities such as biocompatibility, osteointegration and osteoconductivity [6,7] are some of functional properties it combines, which has given it an edge over several conventional structural materials for specialized applications and environments.

In applications such as orthopedic biomedical applications, Ti and Ti alloys though possessing the closest combination of properties needed in load bearing orthopedic applications (strength, low elastic modulus, biocom-

patibility, and corrosion resistance), are deficient in adequate wear resistance [8,9]. This has been ascribed to the resistance to plastic shearing and the poor protection provided by the surface oxide films [10]. Several problems have been identified to be consequent on this deficiency – namely, increased wear debris/particles from the implants during usage in the physiological environments of the body, which results in complications such as inflammation of the tissues within the vicinity of the implant, acute pains, infections and shortened life span of the implant due to accelerated loosening and failure of the implant, in vivo [11–13].

As a mitigation measure, the development of Ti based composites reinforced with biocompatible and refractory materials have been advanced [14]. The state-of-the-art shows that several metallic oxides (TiO₂, SrO₂, ZrO₂), phosphates, and hydroxyapatite have been investigated for potential use as reinforcement in Ti based composites for load bearing orthopedic applications [15–19]. The consideration of niobium pentoxide (Nb₂O₅) has been the subject of recent interest [20]. Niobium pentoxide has been reported to be biocompatible and still preserves the good

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Fig. 1. Morphologies of starting materials: (a) CpTi powder (b) Nb₂O₅ powder (c) Nb₂O₅ powder dispersed in CpTi matrix.

biocompatibility qualities of the base metal Ti when added as reinforcement. Nonetheless, the mechanical properties and wear behaviour have not been extensively investigated, which could be instructive in appraising its all-round reliability for orthopedic applications; and also forecast the other potential applications of the Ti-Nb₂O₅ based composites.

The present study attempts to address this identified research gap by assessing some of the vital mechanical properties and antiwear properties of the Ti-Nb₂O₅ based composites processed by spark plasma sintering, using nanoindentation method. The SPS process was selected based on its optimal processing and sintering efficiency and improved material properties imparted over conventional powder metallurgy techniques [21–23]. The use of nanoindentation is equally justified as it has been established to be a more amenable and reliable method of assessing mechanical and related properties at small length/size scales than conventional mechanical testing methods [24,25]. The conventional mechanical testing methods often require bulk materials and standard geometries, which are not applicable for nanoindentation testing. Also, the nanoindentation testing is non-destructive as the microstructures are essentially not significantly distorted by testing. Furthermore, it is possible to generate data for the evaluation of a broad range of mechanical and related properties (such as the hardness, elastic modulus, the elastic index, plastic index, fracture toughness, visco-elastic properties, creep rate, and wear resistance), which can hardly be derived from a single conventional mechanical testing data analysis [26,27].

The corpus of literature on the use of nanoindentation indicate that there is hardly any work from authoritative sources which has adopted this technique to study the mechanical and related properties of Ti-Nb₂O₅ based composites, thus the motivation for this study.

2 Materials and methods

2.1 Materials

The starting powders utilized for this investigation were commercial pure titanium powder (APS 25 µm; 99.8% purity) and niobium pentoxide powder (APS 50 µm; 99.9% purity) used as the reinforcement. The CpTi and Nb₂O₅

powders were supplied by TLS Technik GmbH & Co and Sigma-Aldrich, respectively. The Cp Ti based composites were designed to contain 5, 10 and 15 wt.% niobium pentoxide powder as reinforcement, with the unreinforced Cp Ti serving as the control composition.

2.2 Method

2.2.1 Composite production

The rule of mixture was used to determine the amount of Cp Ti and Nb₂O₅ powders required for the designed compositions to be achieved. The powders were mixed using a Retch 100 PM planetary ball milling machine with milling time of 4 hours, and speed of 100 rev/mins, without balls. In order to avoid cold welding and reactions of the powders when milling, 10 mins relaxation time was adopted for each 20 mins milling. The morphologies of the starting and milled powders were assessed using JEOL JSM-7600 F field emission scanning electron microscope and are presented in Figure 1.

The sintering of the mixed powders was performed using an automated spark plasma sintering machine (model HHPD-25, FCT GmbH Germany) following standard spark plasma sintering working principles [26]. The Ti-Nb₂O₅ based composites were sintered in vacuum, using pressure, sintering temperature, heating rate, and sintering holding time of 50 MPa, 1100 °C, 100 °C/min, and 10 min, respectively before cooling to ambient temperature. For ease of removal, the powders were shielded from the die upper and lower punches, using graphite sheets. After sintering, graphite contamination on the products surfaces, were removed by sand blasting. The relative density of the Cp Ti and Ti-Nb₂O₅ composites were measured using Archimede's principle [23]. The relative density of the samples on determination were within the range 99.57–99.94%.

2.2.2 Microstructural evaluation

For microstructural evaluation, Cp Ti and Ti-Nb₂O₅ based composites were metallographically prepared by grinding with silicon carbide papers, after which they were polished with colloidal silica suspension to flat mirror finished surfaces. Thereafter, etching of the polished samples was performed using Carpenters reagent. The microstructures of the Cp Ti and Ti-Nb₂O₅ based composites were then

examined, using JEOL Scanning Electron Microscope (FESEM, JSM-7600F).

2.2.3 Nanoindentation

The nanoindentation tests were performed with Anton Paar ultra-Nanoindenter (UNHT) fitted with a diamond Berkovich indenter. The indentation experiments were performed in accordance with ISO 14577 [23], with the test run under constant load to reach possible maximum depth. The indentation experiments were performed using the user defined profile, using two different loads of 20 and 100 mN to make the indents at a loading rate of 10 mN/min and held for 10 s. A minimum of five indents were made per sample and the average of these indentations served as basis for analyses of the data generated. The user defined profile (with the assistance of a built-in microscope which offers three sets of magnification), permits the selection and proper delineation of areas within the samples where the indents are to be made. The hardness and elastic modulus of the samples were evaluated using Oliver-Pharr analysis [28].

Basically, the hardness (H) is defined as [29]:

$$H = \frac{P_{\max}}{A_c}, \quad (1)$$

where $P_{\max}$ is the maximum load, and A_c is the projected area of the indentation.

The reduced elastic modulus (E_r), is considered as the elastic modulus which factors the elastic contributions of the specimen and the indenter tip, and is determined using the relation [1]:

$$\frac{1}{E_r} = \frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_i^2}{E_i} \quad (2)$$

where, E_i and E_s are the elastic modulus of the indenter and sample, respectively; while ν_i and ν_s are the Poisson's ratio of the indenter and the specimen, respectively.

From the hardness and reduced elastic modulus, the elastic strain to failure and the yield pressure were determined using the expressions [24]:

$$\text{Elastic strain to failure} = \frac{H}{E_r}. \quad (3)$$

$$\text{Yield pressure} = \frac{H^3}{E_r^2}. \quad (4)$$

Also, the elastic and plastic indentation energies for the Cp Ti and Ti-Nb₂O₅ based composites were determined. The total mechanical work done during loading by the indenter, U_t , was determined by from the enclosed area between the loading curve and the displacement axis. This energy is equal to the sum of the elastic, U_e , and plastic, U_p , energies, which is expressed as [25]:

$$U_t = U_e + U_p \quad (5)$$

T values for U_e were determined from the enclosed area between the unloading curve and the displacement axis. Also, the elastic recovery index, $\frac{U_e}{U_t}$, and plastic recovery index, $\frac{U_p}{U_t}$ obtained as the ratio of the elastic energies to total energies and plastic energies to total energies, respectively were evaluated for the Cp and Ti-Nb₂O₅ based composites.

3 Results and discussion

3.1 Microstructural characterization

The SEM images of the Cp Ti and Ti-Nb₂O₅ based composites are presented in Figure 2. Figure 2a shows the lamellar (α) structure of pure titanium (without the addition of the reinforcement) and the corresponding EDS (Fig. 2a1) which shows peaks of Ti and O₂, with the O₂ likely due to oxidation. It is noted that with increasing Nb₂O₅ wt.%, there is transition from the lamellar structure of pure Ti to a bimodal ($\alpha + \beta$) structure for the Ti-Nb₂O₅ based composites (Figs. 2b–2d). The addition of Nb₂O₅ promotes the nucleation and growth of β -Ti which forms at high sintering temperature and are preserved at room temperature due to the fast cooling characteristic of SPS processing [22]. The presence of Niobium (Nb), which is a β stabilizing element [7], could be responsible for the facilitation of the α to β transformation. It is observed from the eds spectra of the Ti-Nb₂O₅ based composites (Figs. 2(b1), 2(c1) and 2(d1)) that Nb and O₂ were identified, alongside elements such as Al, Si, O₂, and Na. The Al, Si, and Na are likely trace impurities from the processing and characterization environment.

3.2 Nanomechanical analysis

3.2.1 Load-displacement and depth-time curves of the Cp Ti and Ti-Nb₂O₅ based composites

The nanoindentation load-displacement curves of the Cp Ti and Ti-Nb₂O₅ based composites subjected to indentation loads of 20 mN and 100 mN, respectively are presented in Figure 3. From Figure 3, it is observed that the Cp Ti and Ti-Nb₂O₅ based composites have similar loading and unloading behaviours that are smooth with no pop-in effect at indentation loads of 20 mN and 100 mN. It is seen that generally, the penetration depth increased with increase in indenter loading from 20 mN to 100 mN. However, the load-displacement curves show that the Cp Ti has a high penetration depth, that is, the indenter penetrates into the material easily, while the penetration depth decreased with increase in Nb₂O₅ content at indenter loads of 20 mN and 100 mN, respectively. The decrease in penetration depth with increase in Nb₂O₅ content is indicative of increased hardness of the Ti-Nb₂O₅ based composites. Similarly, the penetration-depth vs time profiles of the Cp Ti and Ti-Nb₂O₅ based composites when subjected to indentation of 20 mN and 100 mN, are shown in Figure 4. It was observed that after an initial rise, the penetration depth decreased with increasing time, and increase in Nb₂O₅ content. The reduced penetration depth, is an indication of decreased plastic deformability with increasing Nb₂O₅ content.

Fig. 2. SEM micrographs (a) Pure Cp Ti with lamellar structure (a1) EDS showing the chemical composition of Cp Ti. (b) Ti – 5 wt.% Nb₂O₅ (b1) EDS showing the chemical composition of the Ti – 5 wt.% Nb₂O₅ (c) Ti – 10 wt.% Nb₂O₅ (c1) EDS showing the chemical composition of the Ti – 10 wt.% Nb₂O₅ (d) Ti – 15 wt.% Nb₂O₅ (d1) EDS showing the chemical composition of the Ti – 15 wt.% Nb₂O₅.

3.2.2 Hardness and reduced elastic modulus of the Cp Ti and Ti-Nb₂O₅ based composites

The hardness and reduced elastic modulus of the Cp Ti and Ti-Nb₂O₅ based composites are shown in Figures 5a,b and 6a,b. From both Figures, it is observed that there is progressive increase in hardness and reduced elastic modulus with an increase in Nb₂O₅ for both indenter loads of 20 mN to 100 mN. This improvement may be associated to the load transfer from the Ti matrix to the relatively harder Nb₂O₅ particles. It is also linked to increased particle and dispersion strengthening offered by the Nb₂O₅ particles, which serve as barriers to dislocation motion [30].

3.2.3 Mechanical and anti-wear behaviour of the Cp Ti and Ti-Nb₂O₅ based composites

Further insight on the mechanical behaviour of the Cp Ti and TiNb₂O₅— based composites and their anti-wear properties, were assessed from the elastic recovery index $\frac{U_e}{U_t}$, the plastic recovery index, $\frac{U_p}{U_t}$, the Elastic strain to failure $\left(\frac{H}{E_r}\right)$, and the yield pressure $\left(\frac{H^3}{E_r^2}\right)$. The elastic recovery index corresponds to the amount of energy released by the material under the influence of load, and is also a measure of the materials resistance to impact loading; while the plastic recovery index is dependent on the intrinsic plasticity of the material [24,31]. Furthermore, the nano-hardness to elastic modulus ratio $\left(\frac{H}{E_r}\right)$, dictates the ability of the material to resist elastic strain to failure at nanometer length scales whereas the yield pressure $\left(\frac{H^3}{E_r^2}\right)$, serves as a measure of the resistance to plastic deformation iloaded contact and anti-wear resistance of materials [32,33]. Figure 7a,b shows the elastic recovery for the Cp Ti and the Ti-Nb₂O₅ based composites for indentation loads of 20 mN and 100 mN. From Figure 7a, it was observed that the Cp Ti with 20 mN has the least elastic recovery while the value generally improved for the Ti-Nb₂O₅ based composite, albeit the Ti-10 wt.% Nb₂O₅ based composite, had the highest value. The same trend of improved elastic recovery for the Ti-Nb₂O₅ based composites for the 100 mN load was also oerved (Fig. 7b), although the increase was not progressive with wt.% of Nb₂O₅— which could be due to slight inhomogeneous dispersion of the Nb₂O₅ particles in the Ti matrix. Conversely, the plasticity index as shown in Figures 8a,b reduces with the increase in Nb₂O₅ content in the Ti based composites for the indenter loading of 20 mN and 100 mN, respectively. This is an affirmation that with increasing Nb₂O₅ content, the composites undergo less plastic deformation on account of the lower intrinsic plasticity of the composites. Notwithstanding the lower plasticity index of the composites, they are still potentially reliant as hard tissue replacements, as hard tissue implants from biomechanics assessment, are hardly exposed to significant plastic strains based on the typical stress/strain states involved in static and dynamic loads applied on the human body. Figure 9 shows the resistance to elastic strain to failure of the Cp Ti and the Ti-Nb₂O₅ based composites. It is observed that the resistance to elastic strain to failure

Fig. 3. (a) the load at 20mN (b) the load at 100mN.

Fig. 4. (a) Penetration depth with time at 20mN (b) Penetration depth with time at 100mN.

Fig. 5. (a) showing hardness of the material at 20mN (b) hardness at 100mN.

largely improved with the Nb_2O_5 addition. Specifically, the Ti based composite containing 15% Nb_2O_5 had the highest resistance to elastic strain to failure at 20 mN and 100 mN, while the Cp Ti showed the least resistance at both loads. It is also noted that the elastic strain to failure values were highly sensitive to the magnitude of the indenter load, as the values for 20 mN were significantly lower than that for

100 mN. Similarly, from Figure 10, it is noted that the yield pressure of the Ti- Nb_2O_5 based composite improved with increase in the Nb_2O_5 content, with the 15 wt.% Nb_2O_5 having the highest yield pressure. The implication of the improvement of the elastic strain to failure and yield pressure indicates good resistance to impact loading and wear of the composites [25]. Summarily, based on the

Fig. 6. (a) showing the modulus of elasticity at 20mN (b) modulus of elasticity at 100mN.

Fig. 7. (a) Elastic recovery Index at 20mN (b) Elastic recovery Index at 100mN.

Fig. 8. (a) Plasticity Index at 20mN (b) Plasticity Index at 100mN.

analysis of Figures 7–10, the use of Nb₂O₅ as reinforcement in Ti, offers improved mechanical and antiwear properties which are desirable for orthopedic implants and a number of technological applications where Ti based composites

find usefulness. Also, the trends established for these nanomechanically derived mechanical and antiwear properties of the composites, are consistent with findings from related studies [24,33].

Fig. 9. (a) Resistance to elastic strain at 20mN (b) Resistance to elastic strain at 100mN.

Fig. 10. (a) Yield pressure at 20mN (b) Yield pressure at 100mN.

4 Conclusion

In this study, nanoindentation analysis was used to evaluate the mechanical properties and predict the wear behaviour of Ti based composites containing 5, 10 and 15 wt.% Nb₂O₅, developed using spark plasma sintering. The results show that:

- The lamellar structure of the pure Ti transformed to a mixed structure of lamellar and bimodal structure for the Ti-5 wt.% Nb₂O₅ composite, to fully bimodal structures for the Ti-10 wt.% Nb₂O₅ and Ti-15 wt.% Nb₂O₅ composite compositions.
- The hardness (6.0–40.67 GPa (20 mN) and 2.4–12.03 GPa (100 mN)) and reduced elastic modulus (115–266.91 GPa (20 mN) and (28.05–96.873 GPa (100 mN)) of the composites increased with increase in the Nb₂O₅ content.
- The combination of load transfer from the Ti matrix to the relatively harder Nb₂O₅ particles, particle and

dispersion strengthening mechanisms, were linked to the improved hardness and elastic modulus with increase in the Nb₂O₅ content.

- The elastic recovery index also improved with increase in Nb₂O₅ content, while the inverse was noted with respect to plasticity index.
- The elastic strain to failure and yield pressure both improved with increase in Nb₂O₅ content, which suggests that the antiwear properties and resistance to impact loading equally improves with Nb₂O₅ addition.

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