

Influence of Al_2O_3 whisker concentration on flexural strength of $\text{Al}_2\text{O}_3(\text{w})\text{--ZrO}_2$ (TZ-3Y) composite

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Abstract

On investigating the possibility of using alumina whisker reinforced 3 mol% Yttria stabilized Tetragonal Zirconia (TZ-3Y) composite for bioceramic applications, presented here is the influence of varying whisker concentration (2, 5, 10, 15 and 20 wt%) on flexural strength of the composite. Whiskers of hydrothermally synthesized Ammonium Aluminum Carbonate Hydroxide (AACH) were used for composite synthesis. These whiskers transformed in situ into alumina during sintering. It was found that with addition of alumina whiskers, strength was increased and reached a maximum value of 1212 ± 60 MPa and 1325 ± 65 MPa, in pure and 1 wt% CTAB added samples respectively, at a concentration of 10 wt% Al_2O_3 whiskers. The strength values of the synthesized composite can compete well with commercially available materials for dental applications.

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1. Introduction

Ceramic materials are attracting a lot of attention recently for dentistry and other orthopedic applications due to better biocompatibility and esthetics as compared to their metal counterparts. Importance of ceramics for dental applications is evidenced by the availability in market of all types of ceramic restorations like bridges and crowns etc. [1]. For most of these dental implants, ceramic materials like glass, glass-reinforced ceramics and feldspathic are being commonly used [2,3]. However, zirconia and alumina or combination of both is a novel idea [4,5] for biomedical applications. Owing to their better mechanical properties, biocompatibility and esthetics, a lot of research on development of $\text{Al}_2\text{O}_3\text{--ZrO}_2$ composites [6] is in progress.

On account of increasing demand of ceramic materials for different load bearing structural applications, research activities on the fiber reinforced ceramic composite materials have recently enhanced. Earlier Xu et al. have reported that by

reinforcing the dental resin composite with whiskers, its flexural strength can be enhanced twofold [7]. But like Xu's work, majority of the research concerned with the whisker reinforced composite has focused on using silicon carbide or silicon nitride as whiskers [8,9]. However, these non-oxide whiskers may lose their properties at high temperature (1400 °C) due to oxidation or reaction with the matrix. Hence alumina whiskers could be suitable choice for composite reinforcement due to their good thermal and chemical stability, high temperature creep resistance and adequate fracture strength [10–12]. In spite of these advantages, very limited work has been reported on alumina whisker reinforced zirconia composites [13–15]. The main hindrance seems to be the high production cost of whiskers for composite formation [16]. However, authors have already reported a novel and economical way of producing alumina whiskers with different morphologies through hydrothermal synthesis [17] by using cheap and readily available raw materials. The composites obtained by this method exhibiting improved hardness have also been reported [14].

Determination of strength of a material is very important for its application, design data, quality assurance and predicting its performance over service life. Ceramic

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materials are brittle in nature and determination of their tensile strength is almost impossible due to the difficulties associated with the preparation of sample as well as its gripping and alignment in tensile testing machine. That is why, flexural strength testing is widely recommended for ceramics [18]. Furthermore, another very important property of ceramics i.e. fracture toughness is also directly related with flexural strength [19]. Therefore, determination of flexural strength gives quite important information about a ceramic material. To the best of the author's knowledge, no study on the flexural strength of alumina whisker reinforced TZ-3Y composite has been reported and this is the first study of its kind.

In this article, effect of whisker concentration on flexural strength of the alumina whisker reinforced TZ-3Y composites is being reported. The stabilization of tetragonal phase of zirconia is confirmed with the help of XRD. Electron microscopy of fractured samples has been employed to verify the retention of whisker morphology in the sintered composite samples. Moreover, the effect of the addition of Cetyl Trimethyl Ammonium Bromide (CTAB) on the flexural strength (by enhancing dispersion of whiskers in ceramic matrix) is also investigated. It is expected that current findings may help to enhance the operational life of bioceramics for dental and orthopedic applications.

2. Experimental procedures

AACH whiskers were prepared by hydrothermal synthesis technique using aluminum nitrate (25 g) and urea (12 g) as precursors at 120 °C for 24 h [17]. For this purpose, a high-pressure reactor (Buchi Glas Uster, Model "Limbo 350") was used.

For composite formation, starting materials were AACH whiskers, high purity 3 mol% yttria stabilized tetragonal ZrO₂ (referred as TZ-3Y) and cetyl trimethylammonium bromide. For determination of the effect of CTAB addition on strength of the composite, two types of samples with and without 1 wt% CTAB were prepared. In each type, five samples with varying amounts of AACH whiskers corresponding to 2, 5, 10, 15 and 20 wt% alumina were synthesized. For preparation of the samples, measured amount of AACH whiskers and TZ-3Y were

separately dispersed through ultrasonication in analytical grade ethanol with and without 1 wt% CTAB. Both suspensions were mixed and the mixture was kept at 60 °C while stirring until nearly all the ethanol was evaporated.

To remove moisture and other volatile ingredients, the samples without and with CTAB were calcined at temperatures of 400 °C and 650 °C, respectively. The calcined mixture was cold pressed at 500 MPa in a steel die (of 10 mm diameter size) using a hydraulic press. The pressed samples were sintered in a muffle furnace (Carbolite HTF-18/8) at 1500 °C for 2 hr at a heating and cooling rate of 5 °C/min. Density of the sintered pellets was determined by Archimedes principle in ethanol using a high accuracy weighing balance. The sintered and fractured surfaces as well as the alumina whiskers were characterized using scanning electron microscope (JEOL, JSM-6490LA, Japan).

Following the results of Giordano et al. for flexural strength of the composite samples their surface flaws were removed through polishing [20]. For this purpose they were ground on successively finer silicon carbide abrasive paper down to 1200 grit and polished with 6 and 1 µm diamond paste. These samples were then dried at 250 °C for 4 h for removal of any residual water vapors.

Flexural strength of the polished composite samples was measured by using universal testing machine. Due to simplicity of the process and ease of sample preparation, the ring on ring flexural strength test method [21] was adopted as per ASTM F394–78 [22] standard with a cross head speed of 0.1 mm/min. For each composition, the measurement was performed on at least six samples and data was reported as an average along with standard deviation showing how much variation or "dispersion" existed from the average.

3. Results and discussion

3.1. Microstructure

Fig. 1(a–b) shows the hydrothermally synthesized AACH whiskers. From Fig. 1(a) it is observed that the whiskers are agglomerated in the form of thick clusters (with approximate diameter of 1 µm and length of 10 µm).

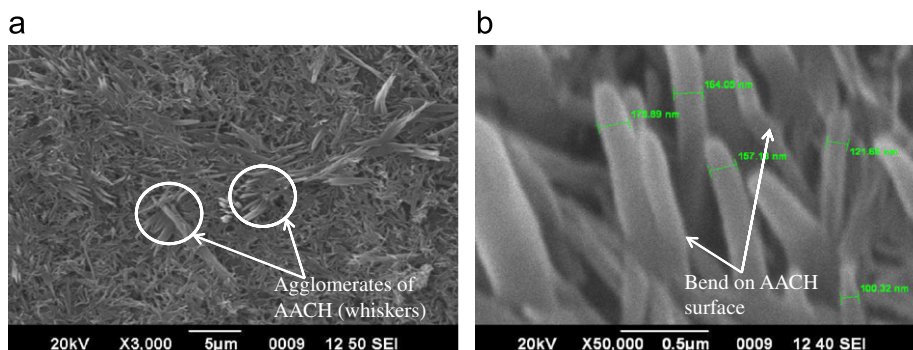


Fig. 1. (a–b) SEM images of hydrothermally synthesized AACH whiskers at (a) lower and (b) higher magnifications.

This agglomeration may be due to Van der Waal's forces and/or hydrogen bonding [23] between individual whiskers, which have diameters of 100–200 nm as shown in a higher magnification image in Fig. 1(b). Some whiskers are observed to have slight bends. Such bending is useful for better mechanical locking with the matrix phase [24].

Fig. 2 shows a SEM micrograph of non-fractured surface of a sintered composite sample. No porosity is visible on the surface. This pore free structure may be attributed

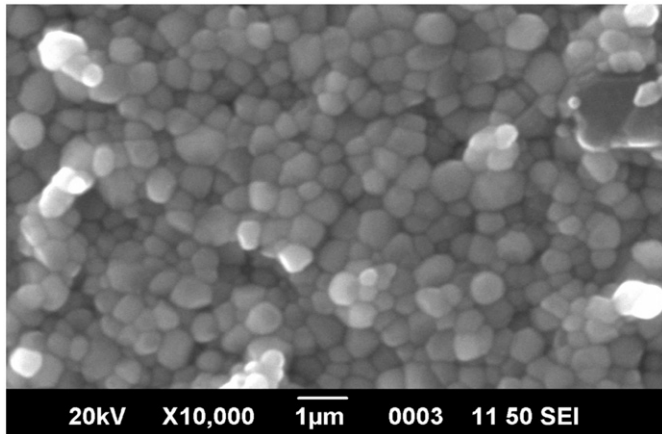


Fig. 2. SEM micrograph of the un-fractured surface of the sintered composite sample containing 10 wt% $\text{Al}_2\text{O}_{3(w)}$.

to the use of a suitable sintering temperature (1500 °C) based on our previous study [13]. Porosity is a highly deleterious element for the flexural strength of a ceramic. Pores not only reduce the effective cross-sectional area, but also act as stress concentrators [25] amplifying the applied stress. For a spherical pore, the applied stress is amplified by a factor of two. The well known empirical relationship between flexural strength and porosity suggests that flexural strength decreases exponentially with increasing the volume fraction of porosity [26]. Therefore, to obtain a material with good flexural strength a pore free micro-structure is indispensable.

To observe the morphology of the whiskers inside the composite, fractography of the samples was performed as shown in Fig. 3(a–c). The SEM micrograph (Fig. 3(a)) shows a bright whisker like particle in a relatively dark background. An EDS spectrum from a $0.75 \times 0.75 \mu\text{m}^2$ area (including the bright whisker) shows peaks from aluminum and zirconium (Fig. 3(b)). The zirconium peak is highly pronounced in comparison with that of aluminum indicating that the overall composition is zirconia rich. The EDS spectrum from whisker like particle shows alumina rich phase (Fig. 3(c)), which bespeaks of this particle to be an alumina whisker. The zirconium counts may come from the zirconia surrounding the whisker or lying in its neighborhood. This confirms that the added alumina whiskers keep their morphology even after sintering.

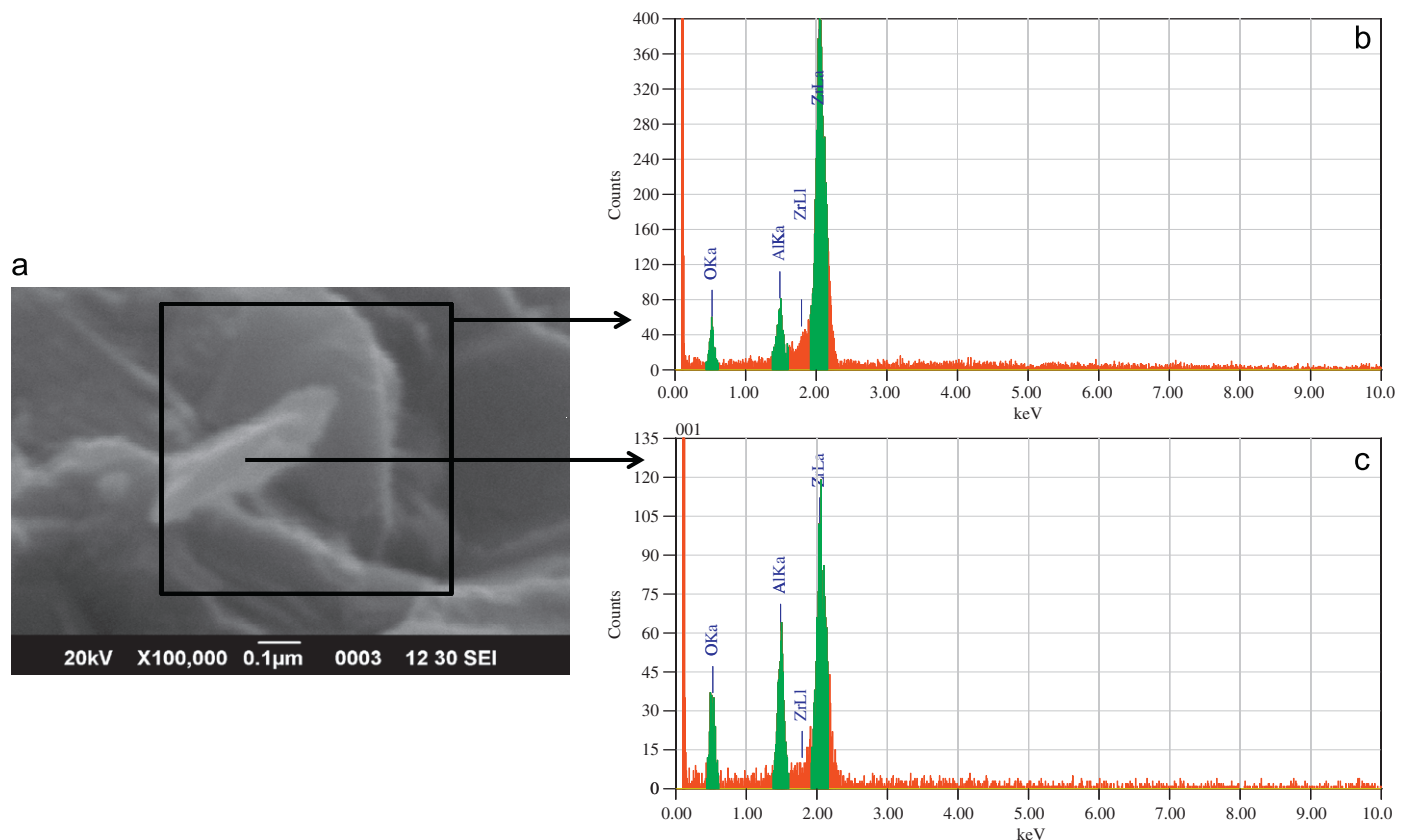


Fig. 3. (a–c) SEM micrograph and EDS of the fractured surface of the sintered/polished composite samples containing 10 wt% $\text{Al}_2\text{O}_{3(w)}$: (a) SEM micrograph; (b) EDS of large area of fractured surface; and (c) spot EDS at a whisker.

The XRD pattern of the composite containing 10 wt% alumina whisker in TZ-3Y matrix is shown in Fig. 4. In this XRD pattern, peaks from tetragonal zirconia and alpha alumina are clearly visible while no peaks from monoclinic zirconia are observed. The key to transformation toughening is stabilization of metastable tetragonal phase at low temperatures, and this results in improving mechanical properties of the composites [27].

Since whiskers were added in the form of AACH and the XRD pattern shows only the peaks of alpha alumina, it is confirmed that AACH has completely transformed to alpha alumina after sintering. It is observed that alumina peaks are highly diminished in comparison with zirconia. It may be due to smaller concentration of alumina and lower X-ray scattering cross-section of aluminum as compared to zirconium [14].

3.2. Flexural strength

Fig. 5(a–b) shows the effect of addition of alumina whiskers on flexural strength of the composites. For pure

TZ-3Y, the value of flexural strength is 945 ± 35 MPa (measured through ring on ring test method). This flexural strength value for TZ-3Y is a little bit lower than its previously reported value of 1000 MPa [28] measured through three or four point flexural test technique. The flexural strength is generally lowered due to presence of cracks or flaws. The measurement of flexural strength through ring on ring technique is more authentic because, in three/four point test methods where the specimens used are in form of bars, cracks that are parallel to longitudinal direction of the bar do not participate in lowering the flexural strength. While in the ring on ring technique, the crack that is present in any direction in sample lowers the flexural strength [29] and depicts the real strength value. This is the reason that strength value varies from one technique to the other [30].

The strength data in Fig. 5(a and b) shows some scattering. In case of ceramics, the failure starts from a flaw that may be introduced during synthesis. These flaws may be in form of cracks, pores, inclusions or surface scratches. Due to statistical distribution in the size, location and geometry of the flaws, strength also shows some scattering [31].

The value of flexural strength (Fig. 5(a and b)) increases with addition of whiskers and reaches maximum values of 1325 ± 65 MPa and 1212 ± 60 MPa at 10 wt% whisker concentration, for the samples with and without 1 wt% CTAB, respectively. These strength values are much higher than TZ-3Y and real tooth [32]. However, further addition of whiskers results in decrease in the flexural strength gradually for both types of samples. The reason for this response of change in strength with whisker concentration is discussed in the following paragraphs.

3.2.1. Flexural strength increase with whisker addition from 0–10 wt%

To investigate the reason for increase in composite strength with increasing whisker concentration (from 0–10 wt% whiskers), refer back to Fig. 3(a). It shows that alumina whiskers have retained their morphology; therefore these whiskers have produced the crack deflection and bridging effects which result in improving the mechanical properties of the composite. The phenomenon of crack deflection can also be verified from Fig. 6; a scanning electron micrograph showing the zigzag path of Vickers indentation-induced crack. Crack deflection is the operative process reported for these whisker reinforced composites. The morphology encircled on the micrograph in Fig. 6 appears like a ligamentary bridge (formed by the whisker) between opposite surfaces of the crack. We know that whiskers are randomly oriented in the TZ-3Y matrix. Under applied indenter load a crack is produced which encounters whiskers as it propagates. This encounter can happen near whisker edge or its center. If the encounter is near the whisker edge, the crack deflects around it due to crack bowing and follows a zigzag path [33]. But if this encounter is near the whisker center then the crack tip appears to arrest as it encounters the interface and then reappears in the matrix

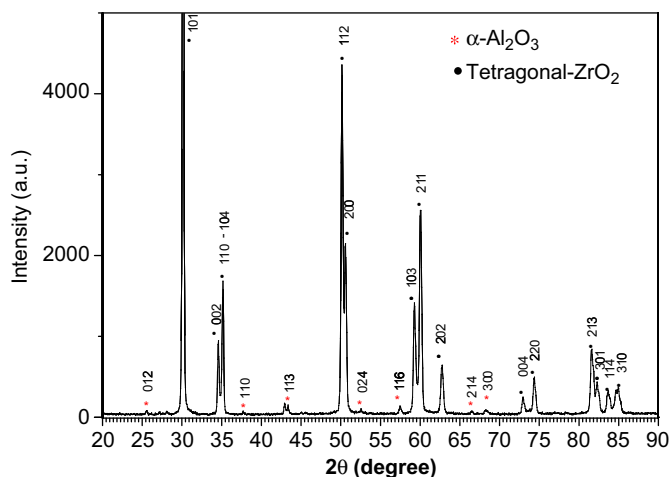


Fig. 4. X-ray diffraction of 10 wt% $\text{Al}_2\text{O}_{3(w)}$ /90 wt% TZ-3Y composite.

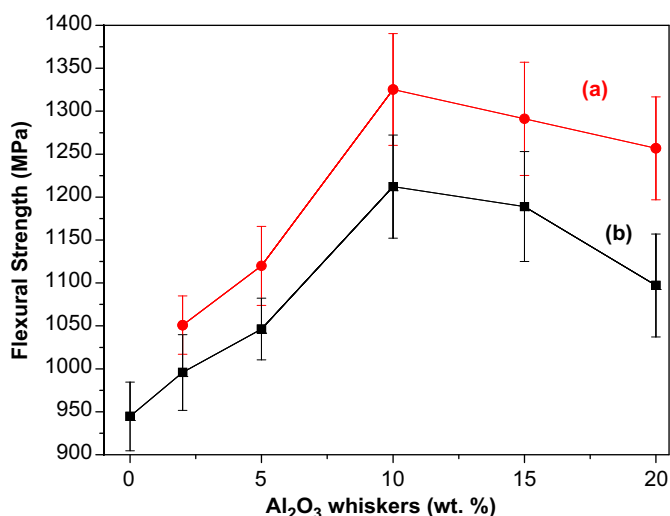


Fig. 5. (a–b) Flexural strength of the TZ-3Y + Al_2O_3 composite with different weight fraction of Al_2O_3 whiskers: (a) with CTAB and (b) without CTAB.

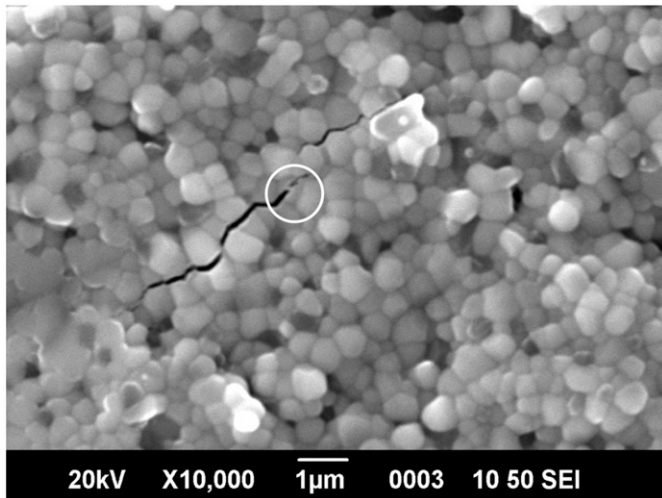


Fig. 6. SEM micrograph of crack formed at the corner of the Vickers hardness indent on the sintered surface of the TZ-3Y+10 wt% Al_2O_3 composite sample, showing crack deflection and crack bridging effects. The morphology encircled on the micrograph is a ligamentary bridge between two surfaces of the crack formed by alumina whisker.

ahead of the whisker, leaving whisker intact and functioning as a ligamentary bridge [34]. This intact type bridging by the whisker is termed as “frictional bridging” [35–37] and results in improved strength of the composite.

3.2.2. Flexural strength decrease with whisker addition above 10 wt%

From Fig. 5(a–b) we can conclude that whisker toughening effect is prominent up to the concentration of 10 wt% $\text{Al}_2\text{O}_3(\text{w})$, beyond which the toughness decreases. It must be emphasized that two opposing factors are operative in the whisker reinforced composites. One is the toughening effect and the other is agglomeration and a consequent decrease in density. The toughening effect plays role through various processes to hinder crack propagation such as crack deflection, crack bowing and crack bridging etc. by the whiskers. Agglomeration of whiskers is not so prominent at lower concentrations and therefore with increasing whisker content, the flexural strength and toughness increase. However, as the concentration of whiskers goes beyond a critical value (10 wt% in the present case) [38], the agglomeration effect starts dominating. The agglomerates create low density regions [39] and result in lowering of strength. As the whisker content is increased further, more low density regions are created and more decrease in flexural strength is observed. This conclusion is also supported by the density versus whisker content data shown in Fig. 7(a and b). Up to 10 wt% whisker content, the density remains almost constant and it starts decreasing as the whisker content is increased further. This is true for both kinds of composites, i.e., with and without CTAB.

The low density regions are formed due to formation of pores/ flaws around or inside agglomerates, thus

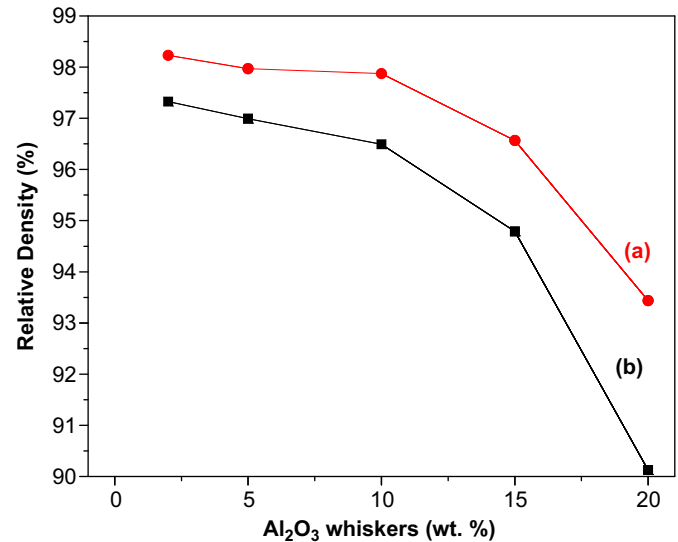


Fig. 7. (a–b) Relative density of the TZ-3Y+ Al_2O_3 composite with different weight fraction of Al_2O_3 whiskers: (a) with CTAB and (b) without CTAB.

deteriorating the strength of composites [40]. According to some reports, incompatibility stresses between whiskers and matrix are responsible for agglomeration of the whiskers [41]. These stresses arise with increase in whisker concentration [42]. Therefore, when the whisker concentration is lower than 10 wt% then these stresses are small, and the strengthening mechanism is whisker toughening. On the other hand, when the whisker content increases above 10 wt%, harmful effect of incompatibility stresses becomes dominant and this results in whisker agglomeration and decrease in flexural strength.

From Fig. 5(a), it is observed that the flexural strength of the samples containing 1 wt% CTAB [43] is higher than samples without CTAB. The CTAB increases the dispersion of the whiskers [15], which results in improving strength of the composite.

From above discussion we can conclude that by optimizing the alumina whisker loading (10 wt%) in TZ-3Y matrix, the composite with excellent flexural strength (i.e. 1325 ± 65 MPa) superior to that of real tooth (225 MPa) [32] can be synthesized. The reason for high strength may be the retained morphology of the whiskers resulting in crack bowing, crack deflection and frictional bridging type of effects. The energy absorbed in these crack whisker interactions results in improving the composite strength. With improved strength, it is expected that these composites can be good candidates for dental implants in future.

4. Conclusions

1. The effect of the addition of various concentrations of alumina whiskers in TZ-3Y on the flexural strength of the composites was studied and it was found that the

- composites were quite compact for concentrations of up to 10 wt%. It was also observed that whiskers kept their whisker like morphology after sintering at 1500 °C for 2 h.
- Flexural strength increased with increasing whisker concentration up to 10 wt% above which the strength decreased. The increase in flexural strength was attributed to whisker toughening mechanisms such as crack deflection, crack bridging and ligamentary bridging between crack surfaces, which resulted in hindrance in crack propagation. Up to 10 wt% alumina whiskers the density was not affected much with increase in alumina whisker content. However, at higher whisker contents density decreased with increasing whisker concentration. It was attributed to whisker agglomeration resulting in formation of low density regions and consequent decrease in flexural strength.
 - Flexural strength was also enhanced with addition of 1 wt% deflocculating agent (i.e., CTAB) due to better dispersion of alumina whiskers.

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