

**Development and Evaluation of Machinability, Mechanical and  
Tribological Behavior of Bio-composite Materials**



**A thesis submitted in partial fulfillment for the  
Award of Degree**

**Doctor of Philosophy**

**Submitted by**

***Satyendra Kumar Singh***

**DEPARTMENT OF CERAMIC ENGINEERING  
INDIAN INSTITUTE OF TECHNOLOGY  
(BANARAS HINDU UNIVERSITY)  
VARANASI – 221005, (U.P.)  
INDIA**

**18031005**

**2024**

# **Chapter 9**

## **Conclusions and Future Perspectives**

## 9.1 Conclusions

In brief, the present research explains the investigations regarding the structural, mechanical, and biological behavior of stabilized zirconia-based bioactive glass (BG) composites (3Y-TZP/13-93BG, 8Mg-PSZ/13-93BG, and ZTA/13-93BG). The matrix materials (3Y-TZP, 8Mg-PSZ, and ZTA) are synthesized by the combustion route, and reinforcement (13-93BG) is synthesized by the sol-gel route. Composites are prepared by wet mixing of matrix and reinforcement in a certain proportion (0-25wt% BG). Among them, the composite having the best properties is also evaluated for its machinability and tribological behavior. These composites have applications in dental and orthopedics. The incorporation of bioactive glass into zirconia-based materials is in good agreement with earlier reported works. The tuning within the structural, mechanical, machinability, and tribological parameters is correlated with the BG addition into matrix materials. The incorporating materials perform their desired function within the system and enhance the properties according to the expected outcomes. Compared to the earlier utilized zirconia materials, the incorporated reinforcement proves their excellence within a particular or specified application. Overall, the work can be concluded as follows:

- The bioceramic composite having a general formula of  $[(100-x) (3Y-TZP) - x (13-93 BG)]$  where  $x = 0$  to 25 wt%, has been prepared successfully and sintered at 1250°C for 4 hrs. The effect of 13-93 bioactive glass addition on the structural, mechanical, and biological behavior of 3Y-TZP ceramic is studied. Results indicate that the monoclinic phase of zirconia (m-ZrO<sub>2</sub>) is increased with the addition of BG contents, and it is found to be a maximum (76%) for the sample containing 25wt% of BG. Simultaneously, the tetragonal phase of the zirconia (t-ZrO<sub>2</sub>) phase is decreased with increasing the BG contents and found to be a maximum (88%) for the pure 3Y-TZP. The microstructure of the composite materials indicates that the addition of BG improves the sintering process

of 3Y-TZP ceramic at low sintering temperatures. The relative density and mechanical properties, such as flexural and compressive strength, are found to increase up to 10 wt% of BG content; afterward, it decreases. The hardness is increased with BG addition in 3Y-TZP ceramics. The *in-vitro* degradation indicates the dissolution and apatite layer formation after immersion in a simulated body fluid (SBF) solution. The cell proliferation ability of the composite is increased with increasing the BG concentration and found a maximum of up to 25 wt% of BG. The antibacterial result demonstrates that the presence of BG considerably reduces the bacterial cells' viability on the composite samples. Overall, the best findings are obtained with 10 wt% BG contained 3Y-TZP ceramic composite sintered at 1250°C. The highest values of relative density, flexural strength, and compressive strength are about 97.01%, 397.9 MPa, and 488.7 MPa, respectively for the composite sample containing 10 wt% BG concentration.

- The bioceramic composites, having the general formula [(100-x) (8Mg-PSZ) – x (13-93 BG)] where x = 0 to 25 wt%, have been prepared and sintered at 1150°C - 1350°C for 4 hrs. Results indicate that with enhancing the sintering temperature from 1150°C to 1250°C, the retention of the t-ZrO<sub>2</sub> phase is decreased, but the retention is slightly increased with the BG addition (10 to 25wt %). With further enhancement in the sintering temperature from 1250°C to 1350°C, the retention rate of the t-ZrO<sub>2</sub> phase is increased along with enhancing the BG (0 to 25wt %). The microstructure of the composite materials indicates that the addition of BG improves the sintering process of 8Mg-PSZ ceramic at low sintering temperatures. The inclusion of BG frits (0 to 25 wt%) and variation in sintering temperature (1250°C-1350°C) are strongly affected the relative densities and the mechanical characteristics. Adding BG reinforcements increases the mechanical characteristics at lower sintering temperatures (1250°C) while reducing the mechanical characteristics except hardness at higher sintering temperatures (1350°C).

Elastic moduli decreases with the addition of BG content up to 15 wt%; after that, it slightly increases up to 25 wt%. The *in-vitro* bioactivity and bio-degradation are increased by enhancing the BG contents up to 25 wt%. The biocompatibility increases with BG concentration (up to 25 wt%). The antibacterial result demonstrates that the presence of BG considerably reduces the bacterial cells' viability on the composite samples.

- The bioceramic composites, having the general formula [ZTA-xBG (x = 0, 5, 10, 15, and 25 wt%)] composites have been synthesized and sintered at 1250°C for 4 hrs. The XRD of sintered composites shows crystalline phases gehlenite, nepheline, and akermanite which are osteoinductive bioceramic materials. The results indicate that improved sintering characteristics are observed due to BG inclusion. The relative density is decreased with enhancing the BG content except for a sample containing a high amount of BG (25 wt%). Mechanical properties such as flexural strength and hardness are enhanced by BG content up to 25 wt%. *In-vitro* analysis in simulated body fluid (SBF) reveals that both degradation and bioactivity increase as BG concentrations increase. According to the *in-vitro* cell culture data, adding BG to ZTA improves cellular viability with BG content up to 25 wt%. The antibacterial result shows that the viability of the bacterial cells on the composites has significantly decreased with BG content.
- On the basis of density, mechanical properties, and biological properties results, these three composites (3Y-TZP/BG, 8Mg-PSZ/BG, and ZTA/BG) are compared and found that the 3Y-TZP/BG composites have the best result. Further, machinability and tribological studies are performed on 3Y-TZP/BG composite samples only.
- The machinability of 3Y-TZP/BG composites is performed on the abrasive air jet machine (AAJM) and machinability is evaluated in terms of MRR (material removal rate) and SR (surface roughness) and material removal mechanics (MRM). The

machinability is studied as a function of BG composition (0 to 25 wt%) and machining temperature (room temperature - 600°C). It is found that MRR decreases with increasing the concentration of bioactive glass (BG) while it increases with increasing the machining temperature. The SR is found in the opposite trend. The MRR, SR, and MRM are also confirmed by the HR-SEM and AFM (atomic force microscopy) analysis. Analysis of machined surface indicates that mainly ploughing with plastic deformation and large erosion craters are the predominant mechanisms at an elevated temperature. While at room temperature, brittle fracture, grain ejection, micro-crack propagation, and exfoliation are responsible for material removal.

- Further, the effect of bioactive glass (BG) addition on the tribological behavior of 3Y-TZP/BG composites is evaluated. The tribological test is performed on a biotribometer in simulated body fluid (SBF) at 37°C. The surface morphology of worn surfaces is studied by HR-SEM/EDS. Tribological results indicate that the wear rate and coefficient of friction (COF) are first decreased up to 10wt% of BG and afterward increase up to 25 wt% of BG content. 3Y-TZP with 10 wt% of BG displays superior antifriction and wear resistance properties, with an average COF of 0.17 and a wear rate of  $0.63 \times 10^{-6} \text{ mm}^3$  per mm of sliding distance. The optical profilometer is used for surface topography analysis of worn surfaces. Line and surface roughness are almost enhanced by enhancing the BG content into 3Y-TZP ceramic. The surface morphology of worn surfaces of composite samples shows that each sample undergoes pull-out during the rubbing process. Maximum pull-out is observed on the surface of pure 3Y-TZP sample and minimum pull-out is observed on the surface of the sample containing 10 wt% of BG content.

As a conclusive remark, the addition of bioactive glass into three composites (3Y-TZP/BG, 8Mg-PSZ/BG, and ZTA/BG) enhances the biological properties. Also improves the

sintering condition, and mechanical properties at low sintering temperatures. The machinability in terms of MRR and SR of 3Y-TZP is strongly affected by the BG addition. The 3Y-TZP containing 10 wt% of BG indicates high wear resistance. These bioceramic composites can be used in dentistry and orthopedics.

## 9.2 Future perspectives

A good research project should raise additional questions and explore further possibilities. In a similar vein, based on the techniques and results discussed in this study, several ideas for future directions are suggested here.

- (a) The use of 3D printing technologies for the preparation of zirconia-based bioceramics (3Y-TZP/BG, 8Mg-PSZ/BG, and ZTA/BG) composites that allow for the creation of patient-specific implants and prosthetics, improving fit and functionality. Additive manufacturing enables the production of complex shapes and structures that are difficult to achieve with traditional methods.
- (b) Bioceramic-based composites have been widely used in orthopedics for decades due to their exceptional biocompatibility and osteoconductivity. However, their inherent brittleness and high hardness significantly affect their machinability, limiting their application as bone fixation devices. So there is a need to make machinable 3Y-TZP/BG, 8Mg-PSZ/BG, and ZTA/BG bioceramic composites by incorporating the rare earth phosphate.
- (c) Moreover, the machining of bioceramic parts is an expensive, difficult, and time-consuming process, often requiring diamond tools. In this context, electrochemical processes can substitute conventional machining methods for ceramics. These include electro-discharge machining (EDM), electrochemical machining (ECM), and electrochemical grinding (ECG), which are commonly used for machining hard materials to avoid expensive grinding and labor-intensive operations for high-quality

finish machining. However, EDM, ECM, and ECG only work for electrically conductive materials. Since 3Y-TZP/BG, 8Mg-PSZ/BG, and ZTA/BG bioceramic composites are non-conductive, their electrical conductivity can be increased by embedding secondary electrically conductive phases such as carbides, nitrides, and borides.

- (d) These derived matrix materials can be used to make biocomposite with polymeric materials. Because polymeric materials play a crucial role in advancing biomedical technology due to their biocompatibility, versatility, and cost-effectiveness. Their applications range from implants and drug delivery systems to tissue engineering and minimally invasive devices, making them essential in improving patient care and outcomes in the biomedical field.
- (e) Further *in-vivo* investigation of these derived bioceramic composites is necessary to facilitate their clinical translation, achieve Food and Drug Administration (FDA) approval, and make them marketable.