

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

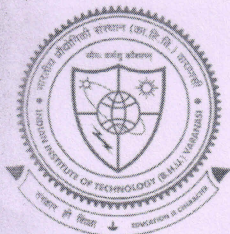
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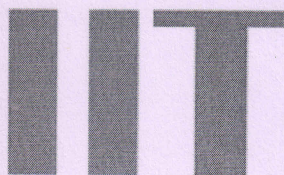
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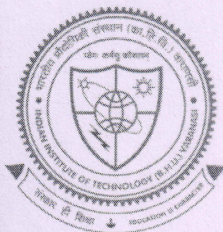
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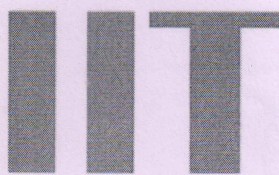
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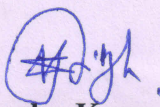
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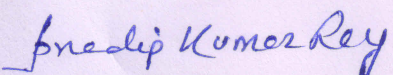
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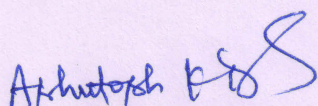

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Dedicated
To
My Beloved Parents, Wife
and daughter (Ishanvi)

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List of Figures

Figure No	Content	Page No
Figure 1.1	<i>Biomaterials market share by application in different biomedical fields</i>	3
Figure 1.2	<i>Biomaterials materials market share (%) in 2022.</i>	4
Figure 1.3	<i>Globally forecasted growing market size trends for biomaterials development</i>	5
Figure 1.4	<i>The reasoning for an effective biomaterial to be employed in the medical field</i>	7
Figure 1.5	<i>The crystal forms and transformations of ZrO_2</i>	11
Figure 1.6	<i>Different forms of ZrO_2 at different temperatures</i>	11
Figure 1.7	<i>Schematic representations of the insertion of Yttria (Y_2O_3) in the zirconia (ZrO_2) lattice and the creation of oxygen vacancies</i>	13
Figure 1.8	<i>Types of biocomposites based on the reinforcement system</i>	17
Figure 1.9	<i>Properties and applications of biocomposite materials used as biomedical products</i>	18
Figure 2.1	<i>A schematic flow chart of different techniques for the synthesis of bioactive glasses</i>	31
Figure 2.2	<i>Mechanisms strategy for osteoconduction in bioactive glass</i>	32
Figure 2.3	<i>Schematic representation of overall research objectives of the present work</i>	56
Figure 3.1	<i>Flow chart represents the 3Y-TZP synthesis method.</i>	59
Figure 3.2	<i>Flow chart represents the 8Mg-PSZ synthesis method</i>	60
Figure 3.3	<i>Flow chart represents the ZTA synthesis method</i>	61
Figure 3.4	<i>Flow chart of synthesis 13-93 bio-glass by sol-gel method</i>	63
Figure 3.5	<i>Schematic representation of dynamic light scattering-</i>	67

	<i>based particle size analyzer</i>	
Figure 3.6	<i>Schematic Basic scheme of an X-ray diffraction experiment</i>	68
Figure 3.7	<i>Schematic representation of regularly spaced atomic planes and their interaction with incident X-ray radiations</i>	69
Figure 3.8	<i>Rigaku Miniflex 600 benchtop diffractometer</i>	70
Figure 3.9	<i>A schematic diagram of Fourier transforms infrared (FTIR) spectroscopy</i>	72
Figure 3.10	<i>FTIR (BRUKER, TENSOR 27) operating in attenuated total reflectance</i>	72
Figure 3.11	<i>The FE-SEM used in this thesis work has been recorded by EVO18, Zeiss</i>	73
Figure 3.12	<i>Universal testing machine (UTM)</i>	76
Figure 3.13	<i>(a) Air jet erosion tester machine and (b) Schematic diagram of material removal mechanics</i>	82
Figure 3.14	<i>The schematic component-wise diagram of the erosion tester setup</i>	82
Figure 3.15	<i>The schematic component-wise diagram of the erosion tester setup</i>	84
Figure 3.16	<i>Schematic diagram of wear experiment</i>	86
Figure 4.1 (a-d)	<i>Characterization of prepared 3Y-TZP (a) Particle size distribution (b) XRD, (c) SEM images, and (d) FTIR</i>	90
Figure 4.1 (e-h)	<i>(e-h) Characterization of prepared 13-93BG (e) Particle size distribution (f) XRD, (g) SEM images, and (h) FTIR</i>	91
Figure 4.2	<i>XRD patterns of (a) sintered composite materials and (b) ZBG25 sample separately</i>	93
Figure 4.3 (a)-(h)	<i>SEM images of 3Y-TZP-based biocomposite materials sintered at 1250°C</i>	95
Figure 4.3	<i>EDS mapping images of 3Y-TZP-based biocomposite materials sintered at 1250°C.</i>	97

(i)-(m)		
Figure 4.4	<i>(a) pH measurement for different time intervals, (b) In-vitro biodegradability analysis, (c) XRD result after 35 days of immersion in SBF, (d) FTIR spectra after 35 days of immersion in SBF</i>	100
Figure 4.5	<i>FE-SEM images with EDS spectra of different samples after 35 days of immersion in SBF.</i>	104
Figure 4.6	<i>MTT assay result for 3Y-TZP/13-93 BG bioceramic composite, cultured for 3, 5 and 7 days. The symbol ‘*’ represents the statistically significant difference in the mean optical density for all samples in comparison to the control sample, and ‘#’ represents the statistically significant difference in the mean optical density for all bio-composite samples in comparison to the base samples ZBG0. ‘**’ represents the statistically significant difference in the mean optical density for ZBG10, ZBG15, and ZBG25 samples in comparison to ZBG5 sample</i>	106
Figure 4.7	<i>Florescence microscopy images of MG-63 cells, adhered on 3Y-TZP/13-93 BG bioceramic composites, while cultured for 48 hours, (a) Control, (b) ZBG0, (c) ZBG5, (d) ZBG10, (e) ZBG15, (f) ZBG25. The scale bar represents 100 μm</i>	108
Figure 4.8	<i>Antibacterial response of 3Y-TZP and 3Y-TZP –xBG ($x = 0, 5, 10, 15$, and 25 wt%) composite for gram-negative (<i>E. coli</i>) and gram-positive (<i>S. aureus</i>) bacteria. Asterisks (*) and (#) represent the statistically significant difference among all the samples with respect to control for gram-negative (<i>E. coli</i>) and gram-positive (<i>S. aureus</i>) bacteria, respectively, at $p \leq 0.05$. Asterisks (@) and (&) represent the statistically significant difference among the composite samples (ZBG5, ZBG10, ZBG15, and ZBG25) with respect to the ZBG sample (ZBG0) for gram-negative (<i>E. coli</i>) and gram-positive (<i>S. aureus</i>) bacteria, respectively at $p \leq 0.05$</i>	110
Figure 5.1	<i>Characterization of prepared 8Mg-PSZ powder (a) Particle size measurement (b) XRD analysis (c) SEM, and (d) FT–IR spectra</i>	115
Figure 5.2	<i>XRD patterns of the 8Mg-PSZ powder at different calcination temperatures</i>	116
Figure 5.3	<i>(a-c) XRD patterns of the composite materials sintered at different temperatures, (d) XRD pattern of MBG25</i>	117

composite at 1350°C

Figure 5.4	<i>(a-f) SEM images of 8Mg-PSZ-based bioceramic composite materials sintered at 1250°C temperature</i>	118
Figure 5.5	<i>(a-d) SEM images of 8Mg-PSZ-based bioceramic composite materials sintered at 1350°C temperature</i>	119
Figure 5.6	<i>(i-k) Elemental mapping of bioceramic composite with different BG content (0%, 10%, and 25%) at 1350°C</i>	120
Figure 5.7	<i>(a) pH measurement with different time intervals, (b) In-vitro biodegradability analysis, (c) XRD patterns after 35 days of immersion in SBF, (d) FTIR spectra after 35 days of immersion in SBF</i>	126
Figure 5.8 (a)	<i>HR-SEM with elemental mapping of MBG0 sample after 35 days of immersion</i>	129
Figure 5.8 (b)	<i>HR-SEM with elemental mapping of MBG10 sample after 35 days of immersion</i>	129
Figure 5.8 (c)	<i>HR SEM with elemental mapping of MBG25 sample after 35 days of immersion</i>	130
Figure 5.9	<i>MTT assay result for 8Mg-PSZ/13-93 BG bioceramic composite, cultured for 3, 5 and 7 days. The symbol ‘*’ represents the statistically significant difference in the mean optical density for all samples in comparison to the control sample, ‘#’ represents the statistically significant difference in the mean optical density for all bio-composite samples in comparison to the base samples MBG0. ‘**’ represents the statistically significant difference in the mean optical density for MBG10, MBG15, and MBG25 samples in comparison to the MBG5 sample</i>	131
Figure 5.10	<i>Florescence microscopy images of MG-63 cells, adhered on the surface of 8Mg-PSZ/13-93 BG (0 to 25 wt% of BG) bioceramic composites cultured for 48 h, (A) Control, (B) MBG0, (C) MBG5, (D) MBG10, (E) MBG15, (F) MBG25. The scale bar represents 100 µm</i>	132
Figure 5.11	<i>Antibacterial response of 8Mg-PSZ and 8Mg-PSZ –xBG (x = 0, 5, 10, 15, and 25 wt%) composite for gram-negative (E. coli) and gram-positive (S. aureus) bacteria. Asterisks (@) and (#) represent the statistically significant difference among all the samples with respect to control for gram-negative (E. coli) and gram-positive</i>	135

(*S. aureus*) bacteria, respectively, at $p \leq 0.05$. Asterisks (*) and (&) represent the statistically significant difference among the composite samples (MBG5, MBG10, MBG15, and MBG25) with respect to the MBG sample (MBG0) for gram-negative (*E. coli*) and gram-positive (*S. aureus*) bacteria, respectively at $p \leq 0.05$.

Figure 6.1	Characterization of the prepared ZTA powders, (a) Particle size distribution (b) XRD, (c) SEM images, and (d) FTIR	140
Figure 6.2	XRD patterns of the prepared powder of ZTA calcined at different temperatures	141
Figure 6.3	XRD patterns of the composite materials sintered at 1250°C, (b) XRD pattern of ABG25 composite sintered at 1250°C	142
Figure 6.4	SEM images of ZTA-based bioceramic composite materials sintered at 1250°C.	143
Figure 6.5	Elemental mapping of bioceramic composite with different BG concentrations (0%, 5%, 15%, and 25%) at 1250°C	144
Figure 6.6	(a) pH measurement with different time intervals, (b) In-vitro biodegradability analysis, (c) XRD patterns after 42 days of immersion in SBF, (d) FTIR spectra after 42 days of immersion in SBF	147
Figure 6.7	HR-SEM of the composite samples after 42 days of immersion in SBF	149
Figure 6.8	Elemental mapping of composite samples, (a) ABG0, (b) ABG5, (c) ABG10, (d) ABG15, (e) ABG25, after 42 days of immersion in SBF	150
Figure 6.9	MTT assay result for ZTA/13-93 BG bioceramic composite cultured with MG-63 cells for 3, 5, and 7 days. The symbol ‘*’ represents the statistically significant difference in the mean optical density for all samples compared to the control sample, and ‘#’ represents the statistically significant difference in the mean optical density for all bio-composite samples compared to the base samples ABG0. ‘***’ represents the statistically significant difference in the mean optical density for ABG10, ABG15, and ABG25 samples compared to the ABG5 sample	152

Figure 6.10	<i>Florescence microscopy images of MG-63 cells, adhered on the surface of ZTA/13-93 BG (0 to 25 wt% of BG) bioceramic composites, cultured for 48 h, (A) Control, (B) ABG0, (C) ABG5, (D) ABG10, (E) ABG15, and (F) ABG25. The scale bar represents 100 μm</i>	154
Figure 6.11	<i>Antibacterial response of ZTA and ZTA-xBG ($x = 0, 5, 10, 15$ and 25 wt%) composite for gram-negative (<i>E. coli</i>) and gram-positive (<i>S. aureus</i>) bacteria. Asterisks (*) and (#) represent the statistically significant difference among all the samples with respect to control for gram-negative (<i>E. coli</i>) and gram-positive (<i>S. aureus</i>) bacteria, respectively, at $p \leq 0.05$. Asterisks (@) and (&) represent the statistically significant difference among the composite samples (ABG5, ABG10, ABG15, and ABG25) with respect to the ZTA sample (ABG0) for gram-negative (<i>E. coli</i>) and gram-positive (<i>S. aureus</i>) bacteria, respectively at $p \leq 0.05$</i>	155
Figure 7.1	<i>(a) HR-SEM image and (b) elemental analysis of erodent (SiC) material</i>	162
Figure 7.2	<i>HR-SEM and EDS images of (a) $x = 0$, (b) $x = 25$ wt% sintered $[(100-x)(3Y-TZP) - x(13-93 BG)]$ composites at room temperature</i>	163
Figure 7.3	<i>MRR of composite materials as a function of BG content and machining temperatures.</i>	164
Figure 7.4	<i>HR-SEM of the machined surface at room temperature</i>	167
Figure 7.5	<i>HR-SEM of the machined surface at 400°C temperature</i>	168
Figure 7.6	<i>HR-SEM of the machined surface at 600°C temperature</i>	169
Figure 7.7	<i>Topography of the machined surface at room temperature</i>	171
Figure 7.8	<i>Topography of the machined surface at elevated temperatures</i>	172
Figure 7.9	<i>Surface roughness (nm) of the machined surface at room and elevated temperatures</i>	173
Figure 7.10	<i>Topography of the machined surface at elevated temperatures</i>	175
Figure 8.1	<i>Image of prepared sintered composite samples</i>	183

Figure 8.2	<i>(a) Coefficient of friction vs running cycles, (b) Coefficient of friction vs bioactive glass concentration. The average friction coefficient of sintered composite samples running after 1800 cycles at 20 N load</i>	184
Figure 8.3	<i>Wear scar profile of all sintered composite samples obtained from profilometer</i>	187
Figure 8.4	<i>HR-SEM images of the wear track (105X) magnification</i>	189
Figure 8.5	<i>HR-SEM images of the wear track (1000X magnification)</i>	190
Figure 8.6	<i>EDS spectra of worn surfaces of all composite samples</i>	191
Figure 8.7	<i>Worn surface topography (Left side 2D and Right side corresponding 3D image) of the composite samples (a) ZBG0, (b) ZBG5, (c) ZBG10, (d) ZBG15, (e) ZBG25 at 20 N</i>	193
Figure 8.8	<i>Worn surface roughness pattern of 3Y-TZP/BG (0 to 25 wt%) composites.</i>	194

List of Tables

Table No	Content	Page No
Table 1.1	<i>Application of biomaterials as synthetic organs</i>	6
Table 1.2	<i>Types of bioceramics</i>	9
Table 3.1	<i>Sample identification and different compositions of 3Y-TZP/13-93 BG biocomposite materials</i>	64
Table 3.2	<i>Sample code and different compositions of 8Mg-PSZ/13-93 BG biocomposite materials</i>	64
Table 3.3	<i>Sample identification and different compositions of ZTA/13-93 BG biocomposite materials</i>	65
Table 3.4	<i>Ionic concentration of SBF solution</i>	78
Table 3.5	<i>Proportionate weight of compounds to prepare SBF solution</i>	78
Table 3.6	<i>Process parameters during machining.</i>	83
Table 4.1	<i>Volume percentage of different phases (m-ZrO₂, t-ZrO₂), bulk densities, relative densities, and porosities of 3Y-TZP/13-93BG composites</i>	93
Table 4.2	<i>Mechanical properties of 3Y-TZP/13-93BG composites</i>	98
Table 5.1	<i>Theoretical density along with experimental density, relative density, and percentage porosity of sintered composite at different temperatures</i>	122
Table 5.2	<i>Mechanical properties of Mg-PSZ-based composite materials sintered at different temperatures</i>	122
Table 5.3	<i>Elastic modulus of Mg-PSZ-based composite material sintered at 1350°C</i>	124
Table 6.1	<i>Theoretical density, experimental density, relative density, porosity, flexural strength, and hardness of ZTA-based composite materials sintered at 1250°C</i>	146
Table 6.2	<i>Comparison of 3 composites (3Y-TZP/BG, 8Mg-PSZ/BG, and ZTA/BG)</i>	157
Table 7.1	<i>Comparison of previously reported research works on machining of bioceramic/ceramic-based composite materials</i>	178

with present machined materials

Table 8.1	<i>The cross-sectional area of wear scar, width of wear scar, average wear depth, wear volume, and wear rate of composite samples</i>	185
Table 8.2	<i>The details of surface roughness of worn surface after wear experiment</i>	192
Table 8.3	<i>Correlates the findings of the current study with those of earlier reported research on the tribological response of the current material</i>	196

List of Abbreviations and Symbols

<i>3Y-TZP</i>	3 mol% yttria-stabilized tetragonal zirconia polycrystal
<i>8Mg-PSZ</i>	8 mol% magnesia partially stabilized zirconia
<i>ZTA</i>	Zirconia toughened alumina
<i>HA</i>	Hydroxyapatite
<i>TCP</i>	Tricalcium phosphate
<i>13-93BG</i>	13-93 Bioactive glass
<i>SBF</i>	Simulated body fluid
<i>HCA</i>	Hydroxy carbonate apatite
<i>Ca-P</i>	Calcium phosphate
<i>AJM</i>	Abrasive jet machining
<i>MRR</i>	Material removal rate
<i>SR</i>	Surface roughness
<i>AAJM</i>	Air abrasive jet machining
<i>RT</i>	Room temperature
<i>MRM</i>	Material removal mechanics
<i>NTD</i>	Nozzle tip distance
<i>PVA</i>	polyvinyl alcohol
<i>DLS</i>	Dynamic light scattering
<i>XRD</i>	X-ray diffraction
<i>FTIR</i>	Fourier transforms infrared spectroscopy
<i>FE-SEM</i>	Field emission scanning electron microscope
<i>AFM</i>	Atomic force microscopy
<i>UTM</i>	Universal testing machine
<i>O.D.</i>	Optical density
ρ_t	Theoretical density
ρ_e	Bulk density
<i>RD</i>	Relative density
<i>E</i>	Young's modulus
<i>G</i>	Shear modulus
<i>K</i>	Bulk modulus
σ_f	Flexural strength

σ_c	compressive strength
L	Span length
W	Flexural load
H_v	Vicker hardness value
V_L	Longitudinal wave velocity
V_T	Transverse wave velocity
L_m	Longitudinal modulus
σ	Poisson's ratio
W_L	Weight loss
W_1	Weight of the sample before the machining
W_2	Weight of the sample after the machining
t	Machining time
D_{avg}	Average depth
ΔV	Volume loss
W_v	Wear rate
A_w	Average wear loss
θ	Bragg's angle
n	Diffraction order
λ	Wavelength
ρ	Density of water
W_c	Sample's weights in the air
W_d	Sample's weights in the water
A	Area
R	Radius of the sample
W_{C1}	Pellet weights before immersion in SBF
W_{d1}	Pellet weights after immersion in SBF
OD	Outer diameter
Nm	Nano meter
μm	Micrometer

PREFACE

The advancement of modern medicine has brought forth remarkable improvements in the quality of healthcare, yet the need for more effective and reliable implant materials remains a critical challenge. With the aging global population and the increasing incidence of bone-related diseases and injuries, the demand for durable, biocompatible, and bioactive implant materials has never been greater. Traditional materials such as metals and polymers, while effective, often fall short in terms of biocompatibility and long-term performance, leading to complications and the need for revision surgeries.

In this context, bioceramic composites have emerged as a promising solution, offering a unique combination of properties that address the limitations of conventional implant materials. Bioceramics, known for their excellent biocompatibility and bioactivity, have the potential to integrate seamlessly with biological tissues, promoting bone regeneration and reducing the risk of implant failure. Their mechanical properties can be tailored to closely match those of natural bone, thereby enhancing their performance and longevity as implant materials.

This thesis explores the development, characterization, and application of bioceramic composites in medical implants. It aims to provide a comprehensive understanding of the material properties, fabrication techniques, and biological interactions that make bioceramic composites suitable for use in orthopedic and dental implants. Through rigorous experimental studies and detailed analysis, this research seeks to contribute valuable insights into the optimization of bioceramic composites for clinical applications.

The present thesis explores the development of zirconia-based bioceramic composites with improved biological properties. The structural, mechanical, and biological performances of composites are measured. In addition to this, the machinability and tribological study are also performed on optimized biocomposites. Physical properties such as density are analyzed

by Archimedes' principle. Mechanical properties of the composites along with the parent material are examined using UTM by measuring the flexural and compression strength. The *in-vitro* bioactivity is measured through structural (XRD), functional (FTIR), and morphological (SEM-EDX, EDS mapping) changes due to surface modification and behavioral changes (pH) of SBF (simulated body fluid) due to ion exchange. The *in-vitro* cellular viability of composites is analyzed on the MG 63 cell line (Human osteosarcoma fibroblast cell). The adhesion and morphology of MG-63 cells on the biocomposite samples are observed using a fluorescence microscope. Antibacterial tests on pure matrix and composite samples are performed with Gram-positive (*S. aureus*; MTCC 435) and Gram-negative (*E. coli*; MTCC 443) bacteria. The biological properties of the composites are enhanced as compared to the parent material. The machinability behavior of composite materials is studied in terms of MRR (material removal rate) and SR (surface roughness) at ambient and elevated temperatures is performed on AAJM. In wet conditions, sintered composites are subjected to reciprocating wear and friction testing using an automated Bio-tribometer. This thesis is divided into 9 Chapters.

Chapter 1 is the introduction of the thesis work which includes the background related to biomaterials, composites, and their properties and applications. Also describes the different synthesis and characterization techniques of bioceramic composites. Finally, it is concluded with the scope and objective of the present work.

Chapter 2 includes the literature review of the bioceramic composites associated with this thesis. This chapter includes a brief discussion related to synthesis techniques and different methods for the improvement of biological properties of bio-insert materials. Also describes the effect of bioactive glass addition into zirconia-based ceramics and its properties, and drawbacks.

Chapter 3 provides a detailed overview of the experimental techniques applied in the present thesis work. It includes sample preparation procedures and a detailed introduction to the working principles of different characterizing instruments that are used in the different studies of synthesized samples.

Chapter 4 Bioceramic composite materials, having general weight composition [(100-x) (3Y-TZP) – x (13-93 BG)] where $x = 0$ to 25 wt%, has been prepared. The effect of BG addition on the structural, mechanical, *in-vitro* degradation, cell culture, and antibacterial behavior of the composite materials are evaluated and discussed in detail.

Chapter 5 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 evaluated the effect of BG amounts on the structural, mechanical, *in-vitro* degradation, cell culture, and antibacterial behavior of the composite materials are evaluated and discussed in the details. Elastic moduli are also calculated and discussed. The effect of sintering temperature on the tetragonal phase retention rate and mechanical properties are evaluated.

Chapter 6 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 effect of BG amounts on the structural, mechanical, *in-vitro* degradation, cell culture, and antibacterial behaviour of the composite materials are evaluated and discussed in the details. On the basis of density, mechanical properties, and biological properties results, these three composites (3Y-TZP/BG, ZTA/BG, and 8Mg-PSZ/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

Chapter 7 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).

Chapter 8 Further, the effect of bioactive glass (BG) addition on the tribological behavior of 3Y-TZP/BG composites is evaluated. The tribological test was performed on a biotribometer in simulated body fluid (SBF) at 37°C. The surface morphology of worn surfaces was studied by HR-SEM/EDS.

Chapter 9 provides brief conclusions of the important findings of our study. A decent improvement in the biological properties of biocomposites is deliberated here with the optimal amount of bioactive glass incorporation and optimization. It also highlights the future scope of research in this material.