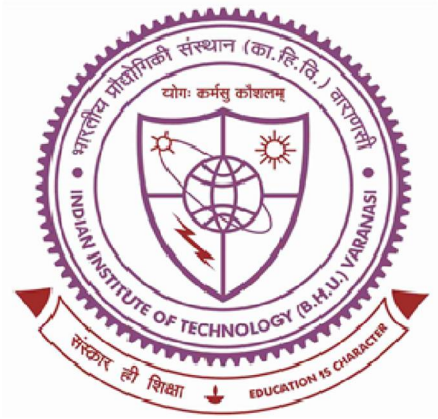
Development and Evaluation of Machinability, Mechanical and Tribological Behavior of Bio-composite Materials



A thesis submitted in partial fulfillment for the Award of Degree

Doctor of Philosophy

Submitted by

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It is certified that the work contained in the thesis entitled "***Development and Evaluation of Machinability, Mechanical and Tribological Behavior of Bio- composite Materials***" by **"Satyendra Kumar Singh"** has been carried out under my supervision, and this work has not been submitted elsewhere for a degree.

It is further certified that the student has fulfilled all the requirements of Comprehensive Examination, Candidacy, and SOTA for the award of Ph.D. Degree.

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DECLARATION BY THE CANDIDATE

I, **Satyendra Kumar Singh**, certify that the work embodied in this thesis is my own bona fide work and was carried out by me under the supervision of **Dr. Pradip Kumar Roy** from July 2018 to July 2024, at the Department of Ceramic Engineering, Indian Institute of Technology (B.H.U.), Varanasi. The matter embodied in this thesis has not been submitted for the award of any other degree/diploma. I declare that I have faithfully acknowledged and given credits to the research workers wherever their works have been cited in my work in this thesis. I further declare that I have not willfully copied any other's work, paragraphs, text, data, results, etc., reported in journals, books, magazines, reports, dissertations, theses, etc., or available at websites and have not included them in this thesis and have not cited as my own work.

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It is certified that the above statement made by the student is correct to the best of my knowledge.

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Dedicated

To

My Beloved Parents, Wife and daughter (Ishanvi)

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List of Abbreviations and Symbols

3Y-TZP	3 mol% yttria-stabilized tetragonal zirconia polycrystal
8Mg-PSZ	8 mol% magnesia partially stabilized zirconia
ZTA	Zirconia toughened alumina
HA	Hydroxyapatite
TCP	Tricalcium phosphate
13-93BG	13-93 Bioactive glass
SBF	Simulated body fluid
HCA	Hydroxy carbonate apatite
Ca-P	Calcium phosphate
AJM	Abrasive jet machining
MRR	Material removal rate
SR	Surface roughness
AAJM	Air abrasive jet machining
RT	Room temperature
MRM	Material removal mechanics
NTD	Nozzle tip distance
PVA	polyvinyl alcohol
DLS	Dynamic light scattering
XRD	X-ray diffraction
FTIR	Fourier transforms infrared spectroscopy
FE-SEM	Field emission scanning electron microscope
AFM	Atomic force microscopy
UTM	Universal testing machine
O.D.	Optical density
ρ_t	Theoretical density
ρ_e	Bulk density
RD	Relative density
E	Young's modulus
G	Shear modulus
K	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}	Pellete weights before immersion in SBF
W_{d1}	Pellete 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 37ºC. 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.