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# ELEMENTS ATERIAL SCIENCE AND ENGINEERING **OF INNOVATION: A JOURNEY INTO**



**Elements of Innovation : A Journey into Material Science and Engineering** Swami Vivekananda University Kolkata, 700121, India



## **ELEMENTS OF INNOVATION:**

## A JOURNEY INTO MATERIAL SCIENCE AND ENGINEERING

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## ELEMENTS OF INNOVATION: A JOURNEY INTO MATERIAL SCIENCE AND ENGINEERING



Swami Vivekananda University, Kolkata (Institutional Publisher)

Published by the Swami Vivekananda University (Institutional Publisher), Kolkata-700121, West Bengal, India

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This edition can be exported from India only by the publisher. Swami Vivekananda University (Institutional Publisher), Kolkata-700121, India. ISBN: 978-81-964878-5-0 First Edition: April, 2024 Publisher: Swami Vivekananda University (Institutional Publisher), Kolkata-700121, India Contact us: deptme@svu.ac.in

## PREFACE

"In the expansive realm of material science and engineering, where innovation and discovery intertwine, 'Elements of Innovation: A Journey into Material Science and Engineering' stands as a beacon of intellectual exploration. This volume is a collaborative endeavour, a symphony of insights orchestrated by dedicated researchers, scientists, and engineers who, driven by relentless curiosity, embark on a journey through the frontiers of material science.

The chapters within this compendium are a testament to the breadth and depth of our understanding, tracing the historical trajectory of materials from their early origins to the current landscape of 21st-century technological advancements. From the dawn of metallurgy to the complex dynamics of modern material synthesis, each chapter unfolds a narrative that is both historical and forward-looking.

The convergence of Material properties and biomedical applications is meticulously examined, delving into the intricacies of material selection for biocompatibility. The significance of mechanical properties, ranging from strength and elasticity to future trends such as smart materials and nanotechnology, is explored within the context of their applications in the field of medicine and healthcare.

Advanced materials analysis using techniques such as crystallography and the potential applications of this analytical method. Additionally, practical insights into material characterization techniques and their role in design considerations are provided, offering a comprehensive understanding of the materials landscape.

As part of this compilation, I extend my heartfelt gratitude to the contributors, whose intellectual generosity has brought this volume to life. It is our collective hope that this compendium not only serves as a repository of knowledge but also sparks a cascade of inspiration, fostering a deeper understanding of the multifaceted world of material science and engineering.

May this book be a catalyst for further inquiry, a guide for those navigating the frontiers of material science, and a source of inspiration for future innovations.

(Dr. Ranjan Kumar) Associate Professor, Swami Vivekananda University, Kolkata, West Bengal, India

Dated: 24, April, 2024

## ACKNOWLEDGEMENT

I extend my heartfelt gratitude to Swami Vivekananda University, Kolkata, India, for their steadfast support and encouragement throughout the creation of "Elements of Innovation: A Journey into Material Science and Engineering." The university's dedication to fostering education and research has been instrumental in shaping the content and direction of this publication. We deeply appreciate the collaborative spirit and resources provided by Swami Vivekananda University, Kolkata, which have enabled us to explore and share the latest innovations and technologies across various fields.

We hope that this book serves as a valuable resource for this esteemed institution and the broader academic community, reflecting our shared dedication to knowledge, progress, and the pursuit of excellence.

I extend my deepest appreciation to each of the external reviewers mentioned below for their unwavering commitment to excellence and their indispensable role in ensuring the scholarly merit of this work.

With sincere appreciation,

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## CHAPTER 1 MATERIAL CLASSIFICATION BASED ON STRUCTURE AND COMPOSITION

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#### ABSTRACT

The classification of materials based on their structure and composition is fundamental in understanding their properties and behaviors, which are crucial for various engineering and scientific applications. This abstract explores the diverse categorization schemes employed in identifying materials, encompassing both natural and synthetic substances. The classification methods discussed include crystalline, amorphous, and composite structures, along with distinctions based on elemental composition and molecular arrangement. Furthermore, the significance of these classifications in determining material properties, processing techniques, and performance characteristics is highlighted. Understanding the relationship between structure, composition, and material behavior enhances the ability to select appropriate materials for specific applications, leading to advancements in various fields, including engineering, materials science, and manufacturing.

#### **1.1 INTRODUCTION:**

Materials science and engineering are indispensable in today's era of scientific and technological advancement. A multitude of materials find application across industries, housing, agriculture, transportation, and beyond, tailored to meet diverse needs. The rapid progress in understanding solids through quantum theory has revolutionized our comprehension and utilization of materials. Notably, breakthroughs in space exploration owe much to advancements in high-temperature and high-strength materials.

Selecting the right material for a specific purpose is a complex endeavor. However, simplification is possible when essential details such as operating parameters, manufacturing processes, functional requirements, and cost considerations are known. These factors, summarized in Table 1.1, guide material selection. With countless materials available, it's challenging for engineers to possess comprehensive knowledge of all. Yet, a profound understanding of the fundamental principles governing material properties facilitates optimal selection.

Materials science and engineering draw from various disciplines like metallurgy, ceramics, and polymer science to inform decision-making. The field is expansive, spanning numerous branches, including the science of metals, mechanical behavior of metals, engineering metallurgy, and engineering materials. This breadth underscores the boundless potential and importance of materials science in shaping our technological landscape.

#### **1.2 REQUIREMENTS OF ENGINEERING MATERIALS:**

In the process of selecting materials for engineering applications, properties such as impact strength, tensile strength, and hardness serve as indicators of suitability, but thorough consideration of additional factors like radiography and other material properties is essential to meet specifications. Engineers may need to dictate production methods, service life, and costs, considering the diverse demands placed on metallic materials. Special surface treatments, such as hardening or normalizing, may be required to address specific service requirements. Additionally, chemical properties, such as structure, bonding energy, and resistance to environmental degradation, significantly influence material selection for engineering purposes.

Polymeric materials, or plastics, have gained popularity in engineering applications in recent years, despite being inferior to most metallic materials in strength and temperature resistance. These materials find use not only in corrosive environments but also in applications where minimal wear is desired, such as small gear wheels, now commonly made from nylon or Teflon. These materials offer satisfactory performance, operate quietly, and eliminate the need for lubrication.

Prior to material selection or component design, a thorough understanding of process requirements is essential. Factors such as operating conditions (hazardous or non-hazardous), continuous or non-continuous operation, raw material availability, spares availability, and cost must be carefully considered. Different materials possess varying properties to meet the diverse engineering requirements.

#### **1.2.1 MECHANICAL PROPERTIES:**

- Tensile Strength: Resistance to tensile forces.
- Hardness: Resistance to indentation, scratching, abrasion, and wear.

- Ductility: Ability to be drawn into wires or elongated before rupture.
- Impact Strength: Resistance to shock loading.
- Wear Resistance: Ability to resist friction wear.
- Corrosion Resistance: Resistance to corrosive action.
- Density: Weight/mass criticality, e.g., for aircraft components.

#### **1.2.2 THERMAL PROPERTIES:**

- Specific Heat (c): Heat capacity per unit mass.
- Thermal Conductivity (K): Heat conductance per unit area in Table 1.1.
- Thermal Expansion: Change in size with temperature.
- Thermal Stresses: Stress due to temperature gradients.
- Thermal Fatigue: Fatigue due to thermal cycling.

Type of the material	Material	Thermal conductivity (K) (W/m/k)
Metals	Copper	380
	Aluminium	230
	Cast iron	52
	Mild steel	54
	Stainless steel	16
Ceramics	Alumina	2
	Titanium Carbide	3
	Glass	1
Polymer	Bakelite	0.23
Composites	Concrete	1.4
	Woods	0.14

#### Table 1.1 Thermal Conductivity for specific materials

#### **1.2.3** Electrical properties:

Electrical properties such as conductivity, resistivity, and dielectric strength are vital characteristics of materials in electrical engineering. A material that offers minimal resistance to the flow of electric current is termed a good conductor of electricity.

The electrical resistance of a material is influenced by its dimensions and is expressed by the formula:

Resistance = Resistivity × Length / Cross-sectional area Resistivity, typically provided in literature, is measured in Ohm-meters.

Materials are categorized based on their electrical resistivity into:

- (i) Conductors
- (ii) Semiconductors
- (iii) Insulators

In general, metals exhibit high conductivity and are considered excellent conductors. Insulators, on the other hand, possess extremely high resistivity. Ceramic insulators, commonly found in applications such as automobile spark plugs and Bakelite handles for electric irons, serve as notable examples. Plastic coverings on cables used in domestic wiring also function as insulators.

Upon cooling below a specific transition temperature, Tc, numerous metals and alloys can enter a state of superconductivity, where their DC resistivity drops to zero. The resistivity in the superconducting phase is estimated to be less than  $4 \times 1025$  Ohm -meters, essentially negligible for practical purposes. Mercury cuprate currently holds the record for the highest Tc, reaching up to 133 K.

#### **1.2.4 MAGNETIC PROPERTIES:**

Magnetic Properties encompassing five main categories: diamagnetic, paramagnetic, ferromagnetic, antiferromagnetic, and ferromagnetic. Certain elements such as iron, cobalt, nickel, as well as their alloys and compounds, exhibit spontaneous magnetization.

Magnetic oxides like ferrites and garnets, known for their exceptional magnetic properties and high electrical resistivity, are extensively utilized in various applications. These include magnetic recording tapes, inductors, transformers, memory elements, microwave devices, bubble domain devices, and recording hard cores, among others.

Key magnetic properties such as hysteresis, permeability, and coercive forces play critical roles in the manufacturing of transformers and other electronic components. Understanding

these properties is essential for optimizing the performance and efficiency of magnetic materials in electronic applications.

#### **1.2.5** CHEMICAL PROPERTIES:

Chemical properties encompass various characteristics such as atomic weight, molecular weight, atomic number, valency, chemical composition, acidity, alkalinity, and more. These properties play a significant role in material selection, particularly in chemical plants, where compatibility with specific chemical processes is crucial.

#### **1.2.6** OPTICAL PROPERTIES:

The optical properties of materials, including refractive index, reflectivity, and absorption coefficient, influence light reflection and transmission, impacting applications such as optics, photonics, and telecommunications.

#### **1.2.7** STRUCTURE OF MATERIALS:

The properties of engineering materials are heavily influenced by their internal atomic or molecular arrangement. Understanding the differences and similarities between materials is essential in the selection process.

Pure metals consist of a single type of atom, whereas alloys, which are more commonly used in commercial applications, are composed of mixed types of atoms. Alloys offer a wide range of physical properties, making them versatile for various industrial applications. They can be classified as binary alloys (two components), ternary alloys (three components), or multicomponent alloys, with most commercial alloys being multi-component.

Although the basic atomic arrangement may not be evident in the final component, the properties of individual crystals within the metallic component, controlled by atomic arrangement, determine its suitability for specific industrial uses.

The strength of a metal or alloy is determined by its ability to withstand external loading. When subjected to stress, the internal structure of the metal responds by attempting to maintain the ordered arrangement of atoms. However, if the applied load exceeds the forces holding the atoms in place, the metallic bond becomes ineffective, causing atoms to move into new positions, a phenomenon known as slip. The ease of atom movement or slip within a material directly influences its hardness and mechanical properties. Understanding these factors is essential for designing and selecting materials for optimal performance in various engineering applications.

#### **1.3** CLASSIFICATION OF ENGINEERING MATERIALS:

Engineering materials encompass a wide range of substances used in various industries for construction, manufacturing, and other applications. These materials are classified based on their properties, composition, and intended use. Below is a comprehensive classification of engineering materials. The basis of various systems of material classification in material science and engineering rests upon several factors:

(i) Chemical Composition: The composition of the material influences its properties and behavior, particularly in chemical plants.

(ii) Occurrence in Nature: The mode of occurrence of a material in nature affects its availability and extraction processes.

(iii) Refining and Manufacturing Processes: The processes undergone by a material before acquiring its required properties shape its final characteristics.

(iv) Atomic and Crystalline Structure: The atomic and crystalline structure of a material plays a significant role in determining its properties and applications.

(v) Industrial and Technical Use: Understanding how materials are used in industrial and technical contexts helps in categorizing them effectively.

Common engineering materials within the scope of material science and engineering can be classified into the following groups:

- (i) Metals (ferrous and non-ferrous) and alloys
- (ii) Ceramics
- (iii) Organic Polymers
- (iv) Composites
- (v) Semiconductors

#### (vi) Biomaterials

(vii) Advanced Materials

Among these, metals are broadly categorized into:

(i) Pure Metals: Obtained through refining processes, typically with purity levels around 99.99%.

(ii) Alloyed Metals: Formed by blending two or more metals, resulting in properties distinct from the individual components.

(iii) Ferrous Metals: Principal constituents include iron, with various carbon content classifications determining properties such as strength and weldability.

(iv) Non-Ferrous Metals: Composed of metals other than iron, with aluminum, tin, copper, nickel, zinc, and magnesium being common examples.

(v) Sintered Metals: Produced using powder metallurgy techniques, resulting in materials with unique properties and structures.

(vi) Clad Metals: Created by sandwiching two materials together to combine their advantageous properties, such as corrosion resistance and strength.

Understanding the properties and applications of these material groups is essential for their effective utilization in engineering and industrial settings.

#### 1.3.1 ORGANIC, INORGANIC AND BIOMATERIALS:

1. Organic Materials:

- Definition: Organic materials primarily consist of carbon atoms bonded to hydrogen, oxygen, nitrogen, and other elements. They are often derived from living organisms or synthesized through chemical processes.

- Properties: Organic materials exhibit diverse properties such as flexibility, lightweight, and low density. They can be transparent, translucent, or opaque, depending on their molecular structure. Examples include plastics, rubbers, fibers, and adhesives.

- Applications: Organic materials find extensive applications across various industries. Plastics are used in packaging, automotive components, and consumer goods due to their versatility and cost-effectiveness. Rubbers are utilized in tires, seals, and gaskets for their elasticity and resilience. Organic fibers like cotton and wool are employed in textiles for clothing and upholstery.

#### 2. Inorganic Materials:

- Definition: Inorganic materials are compounds lacking carbon-hydrogen (C-H) bonds and are often derived from non-living sources such as minerals, metals, and ceramics.

- Properties: Inorganic materials typically exhibit high strength, hardness, and thermal stability. They can be transparent, translucent, or opaque, depending on their composition. Examples include metals, ceramics, glasses, and semiconductors.

- Applications: Inorganic materials have diverse applications across industries. Metals like steel and aluminum are widely used in construction, transportation, and manufacturing due to their strength and durability. Ceramics find applications in tiles, bricks, electronic components, and biomedical implants due to their resistance to heat and corrosion. Semiconductors are essential for electronics and computing, enabling the creation of transistors, diodes, and integrated circuits.

#### 3. Biomaterials:

- Definition: Biomaterials interact with biological systems for therapeutic, diagnostic, or research purposes. They may be natural or synthetic and are designed to mimic or replace biological tissues and organs.

- Properties: Biomaterials are biocompatible, meaning they do not elicit adverse reactions when in contact with living tissues. They can be biodegradable or non-biodegradable and possess mechanical properties tailored to specific applications.

- Applications: Biomaterials play a crucial role in medical and healthcare fields. Metallic implants, such as titanium alloys, are used in orthopedic and dental implants due to their biocompatibility and strength. Biodegradable polymers, like polylactic acid, are employed in sutures and drug delivery systems for their controlled degradation in the body. Bioceramics,

such as hydroxyapatite, are used in bone grafts and dental implants for their similarity to natural bone structure.

In conclusion, organic, inorganic, and biomaterials each serve distinct roles across various industries, offering a wide range of properties and applications tailored to specific needs. These materials continue to advance through research and innovation, contributing to technological advancements and improvements in quality of life.

#### **1.3.2** CURRENT TRENDS AND ADVANCES IN MATERIALS:

Current trends and advances in materials encompass a wide array of developments across various sectors. Here's an overview of the advancements in timber, steel, cement, polymers, ceramics, and semiconductors:

#### 1. Timber:

- Sustainable Forestry: There is a growing emphasis on sustainable forestry practices to ensure the long-term availability of timber resources.

- Engineered Wood Products: Advancements in technology have led to the development of engineered wood products such as cross-laminated timber (CLT) and laminated veneer lumber (LVL), offering superior strength and versatility in construction applications.

- Timber Treatments: Innovations in timber treatments, including preservatives and fireretardant coatings, enhance the durability and fire resistance of timber products.

#### 2. Steel:

- Advanced Alloys: Research continues on the development of high-strength steel alloys, including chromium, nickel, molybdenum, and tungsten alloys, for applications in high-temperature environments such as steam and gas turbines.

- Processing Techniques: Emerging processing techniques such as isostatic pressing and isothermal forging improve the fatigue properties of steel components, particularly in aerospace applications.

- Rapid Cooling Technology: Rapid cooling technologies enable the production of metal powders with enhanced properties for powder metallurgy and hot isostatic pressing, leading to temperature-resistant parts with superior mechanical properties.

3. Cement:

- Sustainable Cement Production: Efforts are underway to reduce the environmental impact of cement production through the use of alternative fuels, waste materials, and carbon capture technologies.

- Supplementary Cementitious Materials (SCMs): SCMs such as fly ash, slag, and silica fume are increasingly used as partial replacements for cement, enhancing concrete performance and reducing carbon emissions.

- Self-healing Concrete: Research is ongoing on self-healing concrete formulations that can repair cracks autonomously, improving durability and extending the service life of concrete structures.

#### 4. Polymers:

- Synergistic Plastic Alloys: Advances in plastic technology have led to the development of synergistic plastic alloys with improved properties, surpassing those of individual polymers.

- Plastic Conductors: The discovery of plastic conductors opens up new possibilities for electronic applications and flexible electronics, paving the way for innovations in areas such as wearable technology and smart devices.

#### 5. Ceramics:

- Cermet Composites: Ceramics mixed with metal powders produce cermet composites, offering enhanced cutting properties and wear resistance for tooling applications.

- Ceramic Reinforcements: Efforts are underway to reinforce ceramics with fibers of materials such as molybdenum to improve strength and durability, particularly in high-temperature and high-load environments.

#### 6. Semiconductors:

- Silicon Demand: The increasing demand for silicon chips, driven by advancements in electronics, automation, and robotics, underscores the importance of semiconductor materials in various industries.

- Emerging Applications: Semiconductors continue to play a pivotal role in diverse applications, including communications, computing, biomedicine, defense, and entertainment, fueling innovation and technological progress.

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These trends reflect ongoing efforts to improve the performance, sustainability, and versatility of materials across multiple industries, driving innovation and addressing emerging challenges in the global marketplace.

#### **1.3.3** Smart or intelligent materials:

Smart or intelligent materials represent a cutting-edge category of materials poised to revolutionize various technologies. The term "smart" implies that these materials possess the ability to detect changes in their surroundings and respond to these changes in predetermined ways, akin to the behavior of living organisms. Moreover, the concept of smart materials extends to complex systems comprising both smart and traditional materials.

The field of smart materials aims to integrate sensors (which detect input signals), actuators (which execute responsive and adaptive functions), and control circuits into a single integrated unit. Actuators may be tasked with altering shape, position, natural frequency, or mechanical characteristics in response to changes in temperature, electric fields, or magnetic fields.

Typically, four types of materials are commonly employed for actuators:

1. Shape Memory Alloys: These metals revert to their original shapes after being deformed when the temperature changes.

2. Piezoelectric Ceramics: These ceramics expand and contract in response to an applied electric field or voltage. Conversely, they also generate an electric field when their dimensions change.

3. Magnetostrictive Materials: These materials respond to magnetic fields in a manner similar to piezoelectric ceramics.

4. Electrorheological/Magnetorheological Fluids: These liquids undergo significant changes in viscosity when electric and magnetic fields are applied, respectively.

The integrated system of sensors, actuators, and control circuits emulates a biological system. This setup is known as smart sensors, microsystem technology (MST), or microelectromechanical systems (MEMS). Sensor materials/devices include optical fibers, piezoelectric materials (including certain polymers), and MEMS. For instance, smart systems are utilized in helicopters to mitigate aerodynamic cockpit noise generated by rotating rotor blades. Piezoelectric sensors embedded in the blades monitor blade stresses and deformations. Feedback signals from these sensors are fed into a computer-controlled adaptive device, which generates noise-canceling antidotes.

MEMS devices are characterized by their small size, lightweight, low cost, reliability, and large-scale fabrication capabilities. They typically comprise sensors for gathering environmental data (such as pressure, temperature, and acceleration), integrated electronics for data processing, and actuators for influencing and controlling the environment.

MEMS technology involves a wide range of materials, with silicon being a cornerstone due to its excellent mechanical properties and mature microfabrication technology. Other materials with piezoelectric, piezoresistive, ferroelectric, and other properties are extensively used for sensing and actuating functions in conjunction with silicon.

The field of MEMS is anticipated to impact various aspects of life, including aviation, pollution control, and industrial processes, heralding a new era of technological innovation and advancement.

#### 1.3.4 NANO TECHNOLOGY AND NANOSTRUCTURED MATERIALS

Nanostructured materials are those whose structural elements—cluster, crystallites, or molecules—have dimensions ranging from 1 to 100 nm. These small groups of atoms are referred to by various names such as nanoparticles, nanocrystals, quantum dots, and quantum boxes. Substantial research has been conducted in the field of nanostructured materials and nanotubes over the past decade due to their potential for high-technology engineering applications. As one progresses from an 'infinitely extended' solid to a particle consisting of a countable number of atoms, there are remarkable variations in fundamental electrical, optical, and magnetic properties.

Various types of nanostructured materials have been considered for applications in optoelectronic devices and quantum-optic devices, including nano-sized powders of silicon, silicon nitride (SiN), silicon carbide (SiC), and their thin films. Some of these materials are

also utilized as advanced ceramics with controlled microstructures, as their strength and toughness increase with diminishing grain size. Carbon-based nanomaterials and nanostructures, such as fullerenes and nanotubes, play an increasingly pervasive role in nanoscale science and technology.

Today, nanotechnology is hailed as the next enabling technology that will revolutionize the future of numerous technologies, products, and markets. A brief overview of nanostructured materials, particularly carbon-based nanomaterials and nanostructures, is presented in a separate chapter.

#### 1.3.5 QUANTUM DOTS (QDS):

Quantum dots (QDs) are semiconductor nanocrystals with unique optical and electronic properties. These tiny structures, typically ranging from 1 to 10 nanometers in size, exhibit quantum mechanical behavior due to their small dimensions. Quantum dots are composed of semiconductor materials such as cadmium selenide (CdSe), cadmium sulfide (CdS), or indium arsenide (InAs), among others.

One of the most notable characteristics of quantum dots is their size-dependent bandgap, which allows them to emit light of specific colors depending on their size. This phenomenon, known as quantum confinement, enables quantum dots to emit light with narrow, tunable wavelengths, making them highly useful in various applications such as displays, lighting, solar cells, and biological imaging.

Quantum dots have several advantages over traditional fluorescent dyes, including higher brightness, longer lifetimes, and resistance to photobleaching. These properties make them valuable tools in biological and medical research, where they are used for labeling and tracking biomolecules within cells and tissues with high precision.

Furthermore, quantum dots are being explored for their potential in quantum computing and quantum information processing due to their ability to trap and manipulate individual electrons and photons. Research in this field aims to harness the quantum properties of quantum dots to develop faster and more efficient computing and communication technologies.

Overall, quantum dots represent a fascinating area of research with promising applications across various fields, from electronics and photonics to biomedicine and quantum technologies.

#### **1.3.6 SPINTRONICS:**

Spintronics, short for spin transport electronics, is a field of study that explores the intrinsic spin of electrons as a means to store, manipulate, and transfer information in electronic devices. Unlike conventional electronics, which rely on the charge of electrons to carry information, spintronics exploits both the charge and spin of electrons, offering potential advantages in terms of speed, efficiency, and functionality.

At the heart of spintronics is the concept of electron spin, a fundamental property of electrons that can be thought of as their intrinsic angular momentum. In spintronics, the orientation of electron spins (either "up" or "down") is used to represent binary information, analogous to the "0" and "1" states in conventional electronics.

One of the key components in spintronics is the spin valve, a device that controls the flow of electrons based on their spin orientation. Spin valves typically consist of layers of magnetic and non-magnetic materials, where the relative alignment of the magnetic moments determines the conductivity of the device. By manipulating the spin orientation of electrons, spin valves can be used to create spin currents, detect magnetic fields, and perform logic operations.

Spintronics has the potential to revolutionize various fields, including data storage, memory, and computing. Spin-based devices, such as spin-transfer torque random-access memory (STT-RAM) and magnetic tunnel junctions (MTJs), offer non-volatile memory with fast read/write speeds and low power consumption. In addition, spintronic devices have shown promise for applications in spin-based logic circuits, spin-based transistors, and spin-based sensors.

Furthermore, spintronics intersects with other emerging technologies, such as quantum computing and topological insulators, opening up new possibilities for the development of novel electronic devices with enhanced functionality and performance.

While spintronics is still a relatively young field, ongoing research and technological advancements continue to push the boundaries of what is possible, paving the way for the next generation of electronic devices and technologies.

#### **1.3.7** FERMIONIC CONDENSATE MATTER:

Fermionic condensate matter, also known as fermionic condensates, refers to a state of matter composed of fermions—particles with half-integer values of spin, such as electrons, protons, and neutrons—cooled to extremely low temperatures. In this state, fermions undergo a phenomenon known as Bose-Einstein condensation (BEC), where a large fraction of particles occupy the lowest quantum state, exhibiting quantum mechanical behavior on a macroscopic scale.

Unlike bosons, which can occupy the same quantum state due to their integer spin, fermions obey the Pauli Exclusion Principle, which states that no two fermions can occupy the same quantum state simultaneously. However, at ultra-low temperatures, fermions can pair up and form composite particles known as Cooper pairs. These Cooper pairs behave like bosons and can undergo BEC, leading to the formation of a fermionic condensate.

Fermionic condensates were first predicted by theoretical physicist Leon Cooper in 1956 as part of his explanation of superconductivity, a phenomenon where certain materials lose all electrical resistance at very low temperatures. In 2003, researchers at Duke University and MIT successfully created the first fermionic condensate using ultra-cold lithium atoms trapped in a magnetic field.

Fermionic condensates exhibit several intriguing properties, including coherence over macroscopic distances, similar to superfluids and superconductors. They also offer unique opportunities for studying quantum phenomena and simulating complex quantum systems in a controlled laboratory environment. Moreover, fermionic condensates have potential applications in quantum computing, precision measurement, and fundamental research in quantum physics.

#### **1.4 MATERIAL STRUCTURES:**

In recent years, the diversity and significance of materials relevant to engineering have surged. Each material type possesses specific compositions and properties tailored for particular applications. While it's impractical to cover the properties of all materials comprehensively, understanding their structures aids both students and engineers in analyzing material behavior. Material structure can be categorized as follows: macrostructure, microstructure, substructure, crystal structure, electronic structure, and nuclear structure.

#### Macrostructure:

The macrostructure of a material is observable with low-power magnification or the naked eye. It encompasses the material's shape, size, and atomic arrangement within crystalline structures. In some crystals, such as quartz, the external crystal form mirrors the internal atomic symmetry. Macrostructure analysis, often conducted on fracture surfaces or forging specimens, can reveal flaws, segregations, and cracks, allowing for early detection and rejection of defective materials, thereby avoiding unnecessary expenses.

#### Microstructure:

Microstructure refers to the material's structure observed under an optical microscope. Optical microscopes can magnify structures up to 1500 to 3000 times linearly without sacrificing resolution. They can detect cracks, porosity, and non-metallic inclusions within materials, providing crucial insights into material integrity.

#### Substructure:

Substructure examination involves scrutinizing crystal imperfections, such as dislocations, using specialized microscopes with higher magnification and resolution, like electron microscopes with magnifications up to 10^5. Field ion microscopes are also employed to visualize individual atoms and atomic defects.

#### Crystal Structure:

Crystal structure elucidates the atomic arrangement within a crystal. X-ray and electron diffraction techniques are commonly used for this purpose. Typically, studying the arrangement of atoms within a unit cell provides sufficient insights, as crystals comprise numerous unit cells forming regularly repeating patterns.

#### **Electronic Structure:**

Electronic structure refers to the arrangement of electrons in the outermost shells of individual atoms within the solid. Spectroscopic techniques are employed to determine electronic structure accurately.

#### Nuclear Structure:

Nuclear structure analysis is conducted using nuclear spectroscopic techniques such as nuclear magnetic resonance (NMR) and Mössbauer spectroscopy.

Understanding material structure across these categories is pivotal for predicting and optimizing material performance in engineering applications.

#### **1.5 ENGINEERING METALLURGY:**

Engineering metallurgy encompasses the study of metallurgical principles and practices tailored for engineering applications. For an engineer, having a deep understanding of engineering metallurgy is crucial when making decisions about material selection and treatment processes for specific tasks. This knowledge enables engineers to determine the appropriate treatment processes and their sequence for finished components and structures. Some key processes involved in engineering metallurgy include:

#### (i) Iron-Carbon Alloy System:

This aspect delves into the structure of iron and steel, as well as iron-carbon equilibrium diagrams. Engineers study the transformation of alloys and steels under various conditions to assess their suitability for different applications. A comprehensive understanding of this system aids engineers in selecting the appropriate iron alloy for their projects based on desired properties and performance requirements.

#### (ii) Heat Treatment:

A thorough grasp of heat treatment processes is essential for engineers to make informed decisions about the types of treatments necessary to enhance the performance and durability of components and structures. Heat treatment methods, such as annealing, tempering, quenching, and case hardening, can significantly influence the mechanical properties and microstructure of materials, thereby ensuring their smooth and efficient operation.

#### (iii) Corrosion of Metals:

Corrosion presents a significant challenge in engineering applications, as it can compromise the integrity and longevity of metal components and structures. Engineering metallurgy addresses the mechanisms of corrosion and explores preventive measures to mitigate its effects. Engineers learn about various corrosion protection techniques, including coatings, cathodic protection, alloying, and proper material selection, to improve the lifespan and aesthetics of metal components and structures.

By acquiring expertise in engineering metallurgy, engineers can make informed decisions about material selection, treatment processes, and corrosion prevention strategies, ultimately contributing to the efficiency, reliability, and longevity of engineering projects.

#### **1.6** Selecting the appropriate material:

Selecting the appropriate material for a particular engineering application is a critical task, requiring careful consideration of various factors to ensure optimal performance and cost-effectiveness. Engineers must strive to achieve the ideal combination of material properties while minimizing costs and maintaining quality standards. Several factors influence the selection of materials:

1. Component Shape: The shape and size of a component impact the choice of processing methods and, consequently, the selection of materials. For example, die casting may limit material options to those with lower melting points, such as aluminium, zinc, magnesium, or thermoplastics.

2. Dimensional Tolerance: The required dimensional tolerance for finished components influences material selection. Some materials can be finished to close tolerances, while others cannot.

3. Mechanical Properties: Mechanical properties such as hardness, strength, and ductility guide material selection. Different processing methods can also affect the mechanical properties of components.

4. Fabrication Requirements: Fabrication requirements, including castability, weldability, machinability, and formability, influence material selection. Each material may have different characteristics that affect its ease of fabrication.

5. Service Requirements: Material properties required for specific service conditions, such as dimensional stability, strength, heat resistance, corrosion resistance, fatigue resistance, and electrical/thermal conductivity, impact material selection.

6. Cost Considerations: The cost of the material and processing plays a significant role in material selection. Cheaper materials may result in higher processing costs due to additional operations and increased scrap, making the overall cost higher than using more expensive materials with lower processing costs.

7. Availability of Material: Material availability may dictate material selection, especially when the desired material supply is limited. In such cases, a more readily available but slightly costlier material may be chosen over a cheaper but scarce material.

8. Design Considerations: Material selection affects the detailed design aspects, such as joining methods (e.g., spot welding, screws, rivets), which must be decided at the early design stage.

When multiple materials are suitable for a job or when conflicting factors arise, compromise becomes necessary. Engineers must weigh the relative merits and demerits of each material option, considering factors such as cost, performance, and lifespan of the finished component. Material selection is a dynamic process that may evolve with changes in design requirements and technological advancements.

In summary, efficient material utilization involves making informed choices based on a thorough understanding of the available materials and their properties, along with careful consideration of various influencing factors.

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#### **CHAPTER 2**

#### **BIOMATERIALS: MATERIALS FOR MEDICAL APPLICATIONS**

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#### ABSTRACT

Biomaterials are specifically engineered materials capable of interacting with living tissue and the surrounding environment in a way compatible with biological systems. Biomedical materials are produced from organic sources, such as silk, or artificial sources, such as ceramics, metals, and polymers. These materials can be categorized into many types, including ceramic, metallic, polymeric, and composite. Biomaterials are employed in diverse medical contexts to assist impaired tissue, substitute deteriorated tissue, or augment biological processes. Biocompatibility is a crucial attribute of a biomaterial, which can also be classified as bioinert, biodegradable, or bio-absorbable. They are utilized in various anatomical locations and have many applications, including stick-to-skin medical devices, implants, prostheses, transplants, and tissue and regenerative engineering. The field of biomaterials is multidisciplinary. The design of a simple biomaterial necessitates knowledge and ideas from multiple disciplines. It needs the synergistic integration of materials, biology, medicine, mechanical sciences, and chemistry. This work aims to present a concise overview of contemporary biomaterials, including their key features and clinical applications.

#### 2.1 INTRODUCTION

Biomaterials are pivotal in creating implants, medical devices, and scaffolds for tissue engineering because they exist at the interface of materials science, biology, and medicine. These meticulously developed materials aim to improve patient outcomes and quality of life through safe and effective interactions with biological systems.

The vast array of materials that make up biomaterials—from metals and ceramics to polymers and even natural substances—each has distinct qualities and functions that are fine-

tuned for particular uses in biomedicine. For instance, orthopedic implants and cardiovascular devices are perfect candidates for metallic biomaterials such as stainless steel and titanium due to their resistance to corrosion and high mechanical strength. Dental and orthopedic applications frequently use bioactive ceramic biomaterials like hydroxyapatite to encourage bone development and integration.

Synthetic and natural polymeric biomaterials' design flexibility and adaptability have made them useful in creating scaffolds, wound dressing material for tissue engineering, and drug delivery systems. Biocompatible and long-lasting synthetic polymers include polylactic acid and polyethylene, whereas natural biomaterials like chitosan and collagen resemble the extracellular cell matrix and promote proliferation and tissue regeneration. Biodegradable materials have recently emerged due to advancements in biomaterials; these materials break down naturally in the body, minimizing the likelihood of long-term problems and doing away with the necessity for surgical removal. Promising both short-term use and eventual replacement by natural tissue, biodegradable metals such as magnesium and iron alloys have great potential as orthopedic and cardiovascular surgical temporary implants.

In this view, biomaterials are vital to contemporary healthcare since they pave the way for new and improved medical equipment and treatments that deal with various health issues. Researchers and clinicians are constantly pushing the boundaries of biomedical engineering by utilizing biomaterials' unique qualities and how they interact with biological systems. This has led to improvements in regenerative medicine, personalized healthcare, and better patient outcomes.

#### 2.2 CATEGORIES OF BIOMATERIALS

Biomaterials can be classified based on several criteria, including the material's source, physical and chemical properties, and the intended application (Festas AJ et al., 2020; Ratner BD et al., 2004). The major categories of biomaterials are represented in Figure 1, which is based on the type of materials. A brief overview of different types of biomaterials is discussed in this section.

**Metals:** Metals such as titanium, stainless steel, and cobalt-chromium alloys are commonly used as biomaterials due to their strength, corrosion resistance, and ductility. They are often used in orthopaedic implants such as hip and knee replacements and cardiovascular stents.

**Ceramics:** Hydroxyapatite, alumina, and zirconia are used as biomaterials due to their hardness, biocompatibility, and bone tissue integration. They are often used in dental and orthopaedic implants.

**Polymers:** Polymers like polyethylene, polyurethane, and silicone are commonly used as biomaterials. Their properties include flexibility, biocompatibility, and ease of processing. They are used in various applications, from contact lenses to artificial heart valves.

**Composites:** Composites comprise two or more materials, such as polymers and ceramics. They are often used to combine the features of different materials, such as strength and flexibility. They are used in the fabrication of bone grafts and dental fillings.

**Natural biomaterials:** In tissue engineering and regenerative medicine, natural biomaterials, including collagen, chitosan, and hyaluronic acid, are frequently utilized. They can act as a scaffold for cell proliferation and differentiation and are biocompatible.



Fig 2.1: Major categories of biomaterials

#### 2.2.1 Metals and Alloys:

Metallic and metal alloy biomaterials are monoliths in functional prosthetics, medical implants, and temporary devices. Consolidated metallic monoliths are widely utilized in orthopaedics, dentistry, craniofacial surgery, and cardiovascular devices. Metallic components are commonly employed as surgical instruments due to their reconditioning ability without compromising performance, making them suitable for temporary and permanent applications. Stainless steel, Co-Cr, Ti, and its alloy are used for permanent fixation, but for temporary fixation, Mg, its alloy, and iron are employed; these are also called bioresorbable materials (Pillar RM et al., 2021; Park JB et al., 2007).

#### 2.2.1.1 Stainless steel

The FCC austenitic stainless steel compositions are advised for use in implants. 316L austenitic stainless steel (ASTMF138/139), although it is more prone to crevice corrosion than other commonly used metallic biomaterials, has demonstrated satisfactory performance and reliability over many years of usage in medical devices ((Ratner BD, 2013). The corrosion resistance of stainless steel relies on developing a thin surface oxide layer containing chromium and molybdenum. Molybdenum provides stability in an environment that contains chloride ions (CI<sup>°</sup>). The material transforms into a single phase known as the FCC austenite phase, starting at its forging temperature of approximately 1050°C and continuing until it reaches room temperature. Its strength and resistance to fatigue are achieved through strain hardening, solid solution strengthening, and small grain size. A high nitrogen concentration in certain stainless steels (ASTMF1314, ASTMF1586, ASTMF2229) increases strength thanks to enhanced solid solution strengthening. Additionally, these steels exhibit superior resistance to crevice and pitting corrosion. Austenitic stainless steels, such as alloys 304, 316, and 316L, are used to construct cardiac pacing systems, electrodes, conducting lead wires, and vascular stents (Table 2.1).

Stainless Steel Grade	Composition	Common Uses in Bioimplants
316L	16-18% Cr, 10-14% Ni, 2-3% Mo, <0.03% C	Orthopedic implants, surgical instruments
316LVM	16-18% Cr, 10-14% Ni, 2-3% Mo, <0.03% C, extra low carbon	Orthopedic and cardiovascular implants, surgical instruments

Table 2.1: Stainless steel-based Biomaterials and their biomedical application

Stainless Steel Grade	Composition	Common Uses in Bioimplants
304	18-20% Cr, 8-10.5% Ni, <0.08% C	Surgical instruments, orthodontic appliances
17-4 PH	15-17.5% Cr, 3-5% Ni, 3-5% Cu, <0.07% C	Orthopedic implants, dental implants
455	11.5-13.5% Cr, 7.5-9.5% Ni, <0.05% C, 0.5- 1.5% Ti	Orthopedic implants, dental implants

#### 2.2.1.2 Co-Cr-Mo alloy:

The castable CoCrMo alloy has been utilized extensively in dentistry for a considerable period and, more recently, in fabricating artificial joints. The toxicity of the corrosion products of CoCrMo is higher than those of stainless steel 316L. The benefits of using wrought or forged CoCrMo are that it provides the greatest strength and wear resistance, and the drawback of CoCrMo is that it is difficult to manufacture and may also cause sensitivity or toxicity to cobalt or chromium ions (Niinomi M 2002).

#### 2.2.1.3 Ti and Ti alloys:

Titanium exhibits exceptional strength-to-weight ratio and corrosion resistance compared to other metals. The object has a shiny, metallic-white appearance and demonstrates a notable toughness. In its pure state, Titanium is a ductile material frequently combined with other elements to increase its strength. The extraction of titanium involves the processing of rutile (TiO<sub>2</sub>), a mineral deposit, through a series of stages to obtain the final material. Because of its non-corrosive characteristics, titanium exhibits exceptional biocompatibility. In life, the material undergoes passivation by creating a sticky oxide layer. Titanium exhibits the distinctive characteristic of osseointegration, which involves establishing a structural and functional connection with the underlying bone. It is frequently employed in various medical applications such as dental implants, total joint replacements, artificial heart valves, internal and external fixators, medical equipment, and spinal fusion. Nevertheless, the exorbitant cost of titanium is attributed to its high processing expenses (Oldani C et al. 2012).

Comparison of the properties and characteristics of Titanium alloy (Ti-6Al-4V) and Titanium (Ti) metal. Ti-6Al-4V has greater strength compared to pure titanium metal. Additionally, both titanium alloy and titanium metal possess exceptional corrosion resistance

and demonstrate good bone bonding properties. The materials have a modulus of elasticity of around 110 GPa. The elasticity value is significantly lower than the modulus of stainless steels and Co-base alloys, which are 210 GPa and 240 GPa, respectively. Titanium and its alloys are utilized in various medical applications due to their exceptional strength, lightweight nature, and corrosion resistance. Ti is a primary biomaterial utilized for joint replacements such as hip balls and sockets and as internal fixators, including plates and screws (Geetha M et al. 2009).

Titanium implants possess superior fracture toughness and enhanced fatigue characteristics compared to other metals. The acetabular shell refers to the socket portion, while the femoral stem pertains to the other component. The socket consists of a metal shell with a liner made of medical-grade plastic, which functions as a bearing. The femoral stem is composed of a metallic material, specifically a titanium alloy. Titanium is utilized for bone-fracture fixation in spinal fusion devices, as well as in the production of pins, bone plates, and screws. Due to its nonmagnetic nature, it is safe for implant patients during resonance imaging or electronic device exposure. Titanium is utilized for a diverse array of surgical tools. It remains corrosion-resistant and maintains its surface features even after repeated sterilization. Additionally, its lightweight nature helps reduce surgeon fatigue during multiple surgeries. This biomaterial is used to substitute craniofacial and maxillofacial fractures (Kaur M et al. 2019).

#### 2.2.1.4 Noble Materials

Due to their superior biocompatibility, ease of production, and design flexibility, nickel metal alloys have been used for biomedical applications for many years. However, owing to their high cost and better alternative solutions, such as base metal implants, these alloys are the least preferred candidates for load-bearing orthopedic applications. Nonetheless, they are expensive but essential to load-bearing dental fillings and restorations (Nouri A. et al. 2021).

• Gold: Gold is a chemically inert metal that is very resistant to the growth of bacteria. Throughout history, gold and its components have been used in oriental civilizations to treat various illnesses. It was among the first materials used as an implantable substance for dental tooth implants. Its malleability makes it an important material in restorative dentistry, especially for crowns and permanent bridges. Gold has great electrical conductivity and biocompatibility, making it an ideal material for manufacturing wires used in pacemakers and other medical devices (Basova TV et al., 2021).

- **Platinum**: Platinum has exceptional resistance to corrosion, compatibility with living organisms, and consistent electrical characteristics. It is used to construct electrodes for devices like cardiac pacemakers and cochlear implants, which are employed as a substitute for the inner ear in individuals with hearing impairments. A traditional pacemaker delivers electrical pulses to stabilize the heart rhythm using platinum-iridium electrodes. Endovascular treatment employs small platinum coils to treat aneurysms (Wissel K et al. 2018).
- Silver: Silver is used in surgical implants and functions as a disinfectant. They are employed as studs in earrings to decrease the likelihood of infection in newly pierced ears. Silver compounds are utilized in burn therapy to augment healing and inhibit infection in burn sites. Silver is used to make stethoscope diaphragms and urinary bladder catheters (Elliott C., 2010).

#### 2.2.1.5 Magnesium and its alloys as Biodegradable

Magnesium and its alloys have gained attention as potential biomaterials due to their biocompatibility, biodegradability, and mechanical properties, which are very much related to natural bone. Magnesium is also an essential mineral in the human body, and it plays a crucial role in several biological processes, including bone growth and maintenance. Magnesium alloys can provide several advantages over other metallic biomaterials when used as a biomaterial. One of the main advantages is their ability to biodegrade in the body, eliminating the need for surgical removal of implants after the healing process. The degradation products of magnesium, including magnesium ions, are biocompatible and can be easily eliminated by the body's natural processes. Moreover, magnesium alloys have been found to promote bone growth and regeneration, making them useful in orthopaedic applications. Magnesium has also been shown to have antimicrobial properties, which can reduce the risk of implant-associated infections (Zhang T et al., 2022; Gu XN et al., 2010).

*Biodegradability:* One of the main advantages of magnesium and its alloys as biomaterials is their biodegradability. Unlike permanent metallic implants, which remain in the body for the rest of the patient's life, magnesium and its alloys can degrade over time, eliminating the need for surgical removal. The degradation products of magnesium are biocompatible and can be easily eliminated by the body's natural processes. The biodegradation of magnesium and its alloys is a complex process that involves the formation of a protective oxide layer on the material's surface, followed by releasing magnesium ions into the surrounding tissue. The release of magnesium ions has been shown to benefit bone regeneration, as magnesium ions
promote the differentiation of bone-forming cells and the formation of new bone tissue (Kamrani et al., 2019).

*Mechanical properties*: Another advantage of magnesium and its alloys as biomaterials is their mechanical properties, which are similar to natural bone. Magnesium has a low density, which can reduce the weight of implants and improve patient comfort. In addition, magnesium and its alloys have a high strength-to-weight ratio, which can provide the necessary strength for load-bearing applications while minimizing the implant's weight. Magnesium alloys can also exhibit good ductility and toughness, which can help prevent implant fracture and failure (Tsakiris V et al. 2021).

One of the primary benefits of iron as a biodegradable material is its natural existence and metabolic role in the human body. Iron is necessary for several physiological activities, including oxygen transport in haemoglobin and enzyme catalysis. As a result, the body has well-established systems for controlling iron levels and metabolizing iron-containing substances. Iron, when developed into biodegradable implants, can undergo controlled degradation via corrosion mechanisms that imitate its natural metabolism in the body. Adjusting alloy composition, surface treatments, and implant shape can all influence the degrading behaviour of iron implants. Iron-based alloys, such as iron-manganese or irongallium, have better corrosion resistance and mechanical qualities than pure iron. Surface changes, such as coatings or biodegradable polymers, can help control the disintegration rate and improve biocompatibility. Furthermore, the implant's architecture, shape, and porosity can affect breakdown kinetics and tissue response. Biodegradable iron implants have demonstrated promise in orthopaedic, cardiovascular, and gastrointestinal therapies. In orthopaedics, temporary fixation devices such as screws or plates composed of biodegradable iron alloys can give initial support during bone healing before gradually deteriorating, reducing the need for implant removal operations. Similarly, biodegradable iron stents in cardiovascular procedures can temporarily support blood vessels while facilitating tissue recovery and reducing the long-term consequences of permanent implants (Moravej M et al. 2010). Overall, the development of biodegradable iron materials represents a significant improvement in implantable medical devices, potentially improving patient outcomes, lowering healthcare costs, and increasing biocompatibility compared to standard permanent implants. Ongoing research explores the optimization of iron-based biomaterials for a wide range of clinical applications, generating innovation in regenerative medicine and implantable technologies (Zivic et al., 2018).

#### 2.2.2 Ceramics

The field of ceramics is advancing rapidly due to the diverse properties of ceramics, such as porosity and glass-like characteristics, which make them suitable materials for implant fabrication to replace and repair damaged tissues. Different Ceramics used for bioimplant fabrication are discussed in Table 2.2. Bioceramics can be engineered to replicate the neighbouring tissues' mechanical characteristics, enhancing the implant's durability over an extended period. These materials can be utilized in biomedical applications, including prosthetic components. They can be categorized based on the structure of the glass, including (i) predominantly glass, (ii) glass with other particles filling the mass, and (iii) polycrystalline. The biomaterials can exist in several forms, such as crystalline (sapphire), polycrystalline (alumina, hydroxyapatite), glass-ceramic (Ceravital), and composite. Bioactive and bioresorbable ceramic materials are being used to heal and restore injured areas of the musculoskeletal system. This is done by introducing customized biomimetic scaffolds at the site of fracture. The details of this process are explained in the following sections (Warreth A. et al., 2020; Blum IR et al., 2011; Hench LL et al., 2010). Undoubtedly, the selection of the appropriate bioceramic is contingent upon the specific location of its use.

Ceramic Type	Properties	<b>Common Uses in Bioimplants</b>
Hydroxyapatite (HA)	Biocompatible, resembles natural bone mineral	Coatings for orthopaedic and dental implants, bone fillers
Zirconia (ZrO2)	High strength, biocompatible	Dental implants, hip replacements, femoral heads
Alumina (Al2O3)	High strength, wear resistance	Orthopaedic implants (e.g., hip, knee), dental implants
Bioglass	Bioactive, bonds with bone tissue	Bone grafts, dental implants, coatings for implants

Table 2.2: Types of ceramics, properties, and their applications

## 2.2.2.1 Alumina (Al<sub>2</sub>O<sub>3</sub>) and Zirconia (ZrO<sub>2</sub>)

Alumina  $(Al_2O_3)$  and zirconia  $(ZrO_2)$  are the main ceramic oxides utilized in tissue engineering applications, particularly for repairing and replacing damaged bone tissue and

joints, such as in total-hip and -knee arthroplasty. These materials are chosen for their exceptional wear resistance and biocompatibility. Although they are inactive substances, they can be utilized alongside other substances, such as biodegradable polymers, to administer medications and facilitate the growth of tissues. The material biocompatibility is determined by the chemical stability of its crystal lattice. This arrangement provides corrosion resistance to alumina and zirconia and ensures their reliable behavior inside living organisms. The implant surface of these materials often exposes hydroxyl radicals (-OH). These radicals interact with bodily fluids, providing a lubricating layer on the implant surface. The fatigue strength, mechanical strength, and brittleness of Al<sub>2</sub>O<sub>3</sub> are affected by the size, purity, crystal distribution, and density. Because of its exceptional mechanical strength, it is utilized for the production of endosseous implants in both orthopedics and maxillofacial surgery (Maccauro G. et al., 2020). Al<sub>2</sub>O<sub>3</sub> implants possess a low surface roughness (R $\leq 0.02$  µm) and a tiny average grain size (<4 µm), resulting in outstanding tribological characteristics (Contuzzi, N et al. 2023). In its pure form, Zirconia exhibits a singular crystal structure when kept at ambient temperature but transforms cubic structure and a tetragonal at elevated temperatures. Zirconia's square and cubic structure grid is stabilized by incorporating several oxides, including yttrium oxide, calcium oxide, magnesium oxide, and cerium oxide (Ce<sub>2</sub>O<sub>3</sub>) (Denry I et al. 2021). Zirconia occurs in three primary crystalline-phase structures: monoclinic (m), cubic (c), and tetragonal (t). If the transformation from a tetragonal to a monoclinic crystal structure is regulated, microcracks present in the crystal-mesh structure of zirconia will be restricted to a certain extent.

Zirconium oxide demonstrates a strength that is more than double the strength of polycrystalline aluminium oxide. It also has a low modulus of elasticity and brittle (Hernigou P. et al., 2003). Femoral balls fabricated by many zirconia heads have been successfully implanted, demonstrating favourable biocompatibility and mechanical performance. These implants are also effective in prosthetic and dental applications, with no reported instances of displacement (Gil J et al. 2021). One can effectively regulate the phase transformation by including CaO, MgO, Y2O3, and oxides, which stabilize the zirconia lattice, producing multiphase materials like stabilized zirconia. A partly stabilized zirconia is formed by incorporating 2-3% mole yttrium oxide (Y2O3), and a partly stabilized zirconia is composed of small square zirconia crystals. As the break spreads in the material, the crystals around the crack tip change from a cubic crystal system to a monoclinic crystal system.

#### 2.2.2.2 Hydroxyapatite/ TCP

Another significant category of bioceramics comprises calcium orthophosphates, specifically hydroxyapatite (HA, Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>) and tricalcium phosphate (TCP,  $Ca_3(PO_4)_2$ ). Apatites are typically inorganic compounds characterized by the general formula  $Ca_5(PO_4)_3X_2$ , where X might represent fluorine ions (fluorapatites or FAp), chloride ions (chloroapatites or ClAp), or hydroxyl ions (hydroxyapatites or OHAp). Hydroxyapatite, with the chemical formula  $Ca_{10}(PO_4)_6(OH)_2$ , is the primary mineral in bones and teeth. Its generally low level of crystallinity characterizes it. Its stoichiometric composition includes 39.68% calcium (Ca) and 18.45% phosphorus (P). As the calcium to phosphorus (Ca/P) ratio increases, the resistance also increases, reaching its highest value at a ratio of around 1.67. However, after this point, the resistance starts to decline (Prakasam M et al. 2015; Pajor K et al. 2019). The bone matrix, while resembling hydroxyapatite, comprises sodium, chlorine, and magnesium, along with other supplementary ionic components, and is stable at a pH range of 9-12. The primary characteristic of hydroxyapatite lies in its exceptional biocompatibility, which facilitates osseointegration and renders it very appropriate for bone repair and replacement purposes. Hydroxyapatite is frequently chosen as an ideal material for dental implants due to the same factors. Moreover, this material can integrate various compounds and eventually assign them to their specific surroundings. It can function as a drug-delivery platform for the regulated release of therapeutic substances over a specific duration. Furthermore, it enhances the production of new bone and the restoration of bone tissue, while its primary limitation is its comparatively limited mechanical durability. The strength diminishes rapidly as the porosity increases. The Weibull modulus of hydroxyapatite falls within the range of 5-18, suggesting that it exhibits characteristics similar to ceramic, which are brittle in nature. The Young's modulus of hydroxyapatite ranges between 35-120 MPa. The modest tensile strengths and the vulnerability to gradual crack propagation (particularly in moist environments) validate the limited load dependability of compact hydroxyapatite implants (Aslankoohi N et al. 2021). As the level of porosity increases, there is a significant drop in fracture toughness. It is important to note that porous hydroxyapatite ceramics have lower fatigue resistance compared to dense hydroxyapatite. Modifying the components' percentage concentration or the solid phase's grain size might alter the mechanical characteristics (Hassanajili S et al., 2019). Given that porous hydroxyapatite ceramics form a stable matrix for cell attachment and osteogenic agents, they are widely employed as bone substitutes because they can make contact with the bone.

Osseous tissue forms inside the pores, enhancing the durability of the implant. Porous hydroxyapatite ceramics with hole diameters ranging from 100 to 600  $\mu$ m are often prepared using powder-sintering. This process involves the addition of appropriate additives such as naphthalene, paraffin, and hydrogen peroxide. These additives release gases at high temperatures, which create pores in the ceramics (Gittens R.A. et al., 2014; Yoshikawa H et al. 2009).

Tricalcium phosphate is utilized in various medical fields, including otolaryngology, orthopaedic prostheses, maxillofacial surgery, dental implants, neurosurgery (specifically spinal cord surgery), periodontal therapy, and percutaneous appliances. The metabolic response of calcium phosphates to bodily fluids is influenced by changes in temperature and pH. Under high-temperature conditions, the non-hydrated forms of calcium phosphate react with bodily fluids at 37 °C to produce hydroxyapatite. Nevertheless, calcium-phosphate cement exhibits drawbacks, primarily associated with subpar mechanical properties, restricting its usage compared to pure ceramic materials (Lodoso-Torrecilla et al., 2021; Fiume E et al. 2021).

#### 2.2.2.3 Bioactive glasses

Bioactive glasses are a distinct category of man-made ceramics that can interact with biological fluids, hence boosting the healing capacity of the human body. Bioactive glasses serve as scaffold materials in tissue engineering, facilitating tissue regeneration in many applications, such as nerve regeneration and wound healing. These substances mostly consist of silica, with additional trace amounts of components such as Na<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, and CaO. These constituents have a crucial role in determining their bio absorbability and bioactive activity. One notable benefit is their robust mechanical strength and their potential for usage as concealing materials. The fabrication of Bioactive glasses is done by either the rapid cooling of molten glass at room temperature (melt-derived glasses) or the sol-gel method, facilitating a three-dimensional porous gel network formation from a colloidal solution under varying pH values (Tulyaganov D.U. et al., 2011).

## 2.2.2.4 Silicon Nitride

Among non-oxide ceramic materials, silicon nitride  $(Si_3N_4)$  is superior, especially due to its exceptional dependability in high-temperature conditions. It exhibits greater mechanical strength and hardness than alumina and is commonly manufactured using the hot-isostaticpressing (HIP) technique (Gracis S. et al., 2015; Benzing J et al., 2019). Silicon nitride that has been strengthened, with a tensile strength of around 1 GPa and a stress-intensity factor of 10-12 MPa·m<sup>1/2</sup>, has been utilized in manufacturing femoral heads that exhibit exceptionally minimal wear.

## 2.2.2.5 Silicon carbide

Silicon carbide (SiC) is an example of a non-oxide ceramic that is manufactured using the HIP method. This material exhibits superior hardness and strength compared to alumina while possessing a similar stress-intensity factor. This material has a tensile strength of 650 MPa and a stress-intensity factor ranging from 9 to 10 MPa·m1/2. This substance is highly beneficial in the orthopedic domain. The silicon-carbide bulk is coated with a thin layer of silicon oxide formed through surface oxidation (Du X et al. 2022).

## 2.2.3 Polymers:

Polymer materials play an important role in bioimplants because of their versatility, biocompatibility, and ability to replicate biological tissue features (Banoriya D et al. 2017). The polymers used for biomedical applications are presented in Table 3. Some of the most important polymers are briefly discussed in this section.

## 2.2.3.1 Polyethylene (PE)

Polyethylene (PE) is a frequently used polymer in bioimplants, especially for joint replacement surgery. It is known for its biocompatibility and high wear resistance, making it ideal for bearing surfaces in orthopedic implants like hip and knee replacements (Paxton et al., 2019).PE components in implants undergo significant processing to improve their mechanical properties and limit wear debris production, extending the implant's lifespan and minimizing adverse reactions in the body.

Table 2.3:	Types	of polymers	used as	biomaterial,	their pro	perties and	applications
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Polymer Type	Properties	Common Uses in Bioimplants
	Biocompatible, wear-	
Polyethylene (PE)	resistant	Joint replacements (e.g., hip, knee)
Poly(methyl		
methacrylate) (PMMA)	Biocompatible, rigid	Bone cement for joint arthroplasty
Poly(lactic-co-glycolic	Biodegradable,	Sutures, drug delivery systems

Polymer Type	Properties	Common Uses in Bioimplants
acid) (PLGA)	biocompatible	
Polyethylene glycol		Coatings for implants, drug delivery
(PEG)	Hydrophilic, biocompatible	systems
		Catheters, pacemaker leads, vascular
Polyurethane (PU)	Biocompatible, flexible	grafts
Silicone	Biocompatible, elastomeric	Breast implants, ophthalmic implants

## 2.2.3.2 PMMA

A transparent thermoplastic polymer commonly used in bone cement during joint replacement surgeries. It is biocompatible and offers a firm fixation when used to anchor implants to bone. PMMA bone cement is prepared intraoperatively and injected into the bone cavity to hold prosthetic components in place during joint replacement procedures. Once cured, PMMA produces a robust contact between the implant and the bone, allowing for load transfer and long-term stability (Mousa W F et al. 2000).

#### 2.2.3.3 Poly(lactic-co-glycolic acid) (PLGA)

It is a biodegradable polymer made from lactic and glycolic acids. Its biocompatibility and adjustable degradation qualities make it a popular choice for bioimplants. Through hydrolysis, PLGA degrades in the body, gradually releasing integrated medicines or bioactive compounds. This makes it appropriate for drug delivery systems like biodegradable sutures and implants that release therapeutic compounds over time. Additionally, PLGA scaffolds are employed in tissue engineering applications to promote cell growth and regeneration (Jin S et al. 2021).

#### 2.2.3.4 PEG

A hydrophilic polymer that is both biocompatible and non-toxic. It is widely used as a bioimplant coating to increase biocompatibility and reduce the risk of fouling or thrombosis. PEG coatings can be used on various implantable devices, including stents, catheters, and drug delivery systems, to reduce interactions with proteins and cells in the surrounding biological environment. Furthermore, PEG hydrogels are used in tissue engineering because

they can encapsulate cells and offer a three-dimensional matrix for tissue development (Rhodes A. et al., 2021).

## 2.2.3.5 Polyurethane (PU)

It is a flexible polymer with diverse mechanical properties, making it ideal for various bio-implant applications. Its biocompatibility, flexibility, and endurance make it ideal for implants, including catheters, pacemaker leads, and vascular grafts. Polyurethane catheters are widely employed in medical procedures because of their flexibility and resistance to kinking, whilst polyurethane vascular grafts have high hemocompatibility and resistance to thrombosis, making them excellent for vascular bypass surgery (Shin EJ et al., 2018).

## 2.2.3.6 Silicone

Silicone is a biocompatible elastomeric polymer with a long history of application in bio implants. It is extremely stable, inert, and resistant to deterioration, making it ideal for long-term implantation in the body. Breast implants, ocular implants, and soft tissue fillers are all examples of medical devices that use silicone implants. Silicone breast implants, for example, have a natural appearance and feel and are commonly utilized in reconstructive and cosmetic breast augmentation procedures.

These polymers represent materials designed for specialized bioimplant applications, including load-bearing orthopedic implants, drug delivery systems, and tissue engineering scaffolds. Their biocompatibility, mechanical properties, and degrading characteristics make them indispensable in modern medical practice, contributing to better patient outcomes and quality of life (Woolfson A. D et al., 2003).

## 2.2.4 Composite:

Due to their customized mechanical qualities, biocompatibility, and ability to mimic biological tissues, composite materials of two or more separate elements have been used significantly in bio implants (Park et al., 2007). Different types of composites are utilized in bio implants (Table 4 ); they are briefly described in this section.

## 2.2.4.1 Carbon Fiber Reinforced Polymers (CFRPs)

It comprises carbon fibers incorporated in a polymer matrix, such as epoxy resin. These composites have exceptionally high strength-to-weight ratios, making them ideal for load-bearing implants in orthopedic and dental applications. CFRP implants provide great stiffness and fatigue resistance, replicating bone's mechanical properties while lowering implant weight and minimizing stress shielding. CFRPs can also be adjusted to match bone radiopacity, allowing for more accurate imaging and postoperative assessment (Chua et al., 2021).

#### 2.2.4.2 Hydroxyapatite (HA) Reinforced Polymers

HA-reinforced polymers mix bioceramic hydroxyapatite particles with a polymeric matrix, such as polyethylene or PMMA. HA, a naturally occurring mineral in bone tissue, improves the composite's bioactivity and osseointegration while increasing its mechanical qualities. These composites are commonly employed in bone substitutes, dental implants, and orthopaedic fixation devices, where they induce new bone growth and help the implant integrate with surrounding tissue (Ramesh N. et al., 2018).

## 2.2.4.3 Glass Fiber Reinforced Polymers (GFRPs)

GFRPs use glass fibres embedded in a polymer matrix, often epoxy resin or polyetheretherketone (PEEK). These composites have high strength, stiffness, and fatigue resistance, making them ideal for orthopaedic and spinal surgery structural implants. GFRP implants have more radiolucency than metallic implants, allowing for greater visualization using imaging modalities. Furthermore, GFRPs can be tuned to match bone's mechanical characteristics, reducing stress shielding and improving load transfer to surrounding tissue (Sano K et al. 2022).

## 2.2.4.4 Graphene-reinforced polymers

This composite uses grapheme nanoparticles' superior mechanical, electrical, and biocompatibility capabilities to improve polymer matrix performance. Graphene, a two-dimensional carbon allotrope, is added to polymers like polyethylene or polylactic acid (PLA) to improve mechanical strength, electrical conductivity, and biocompatibility. These composites stimulate cell adhesion, proliferation, and differentiation in various bioimplants, including neural electrodes, biosensors, and tissue engineering scaffolds (Xie, H et al.2017).

#### 2.2.4.5 Titanium-reinforced polymers

It blends titanium particles or fibres with a polymer matrix (e.g., PEEK or PU). These composites improve mechanical strength, stiffness, and fatigue resistance while maintaining polymer biocompatibility and radiolucency. Titanium-reinforced polymers are widely utilized

in orthopaedic and spinal implants because they offer greater load-bearing capacity and implant durability than pure polymers. (Linder, L. K. B et al. 2019).

## 2.2.4.6 Bioglass-reinforced polymers

It includes bioactive glass particles in a polymer matrix, such as PLGA or PCL. These composites combine bioglass's bioactivity with polymers' mechanical qualities to promote bone repair and tissue integration. Bone graft alternatives, dental implants, and tissue engineering scaffolds use bioglass-reinforced polymers to induce new bone production and aid implant osseo integration.

To summarize, composite materials provide varied solutions for bioimplants, offering specific combinations of mechanical characteristics, biocompatibility, and bioactivity to match the wide range of medical applications. Composite bioimplants improve performance, durability, and patient outcomes in various therapeutic contexts by taking advantage of the synergistic effects of numerous elements. Continuous research and innovation in composite materials drive advances in bioimplant technology, paving the way for safer, more effective treatments and therapies in modern medicine (Niemelä, T et al., 2011).

Composite Type	Description	Common Uses in Bioimplants
Carbon Fiber Reinforced Polymers	Carbon fibers embedded in a polymer matrix offer high strength-to-weight ratios, stiffness, and fatigue resistance.	Orthopedic implants, dental implants
Hydroxyapatite Reinforced Polymers	Bioceramic hydroxyapatite particles combined with a polymer matrix enhance bioactivity and osseointegration while improving mechanical properties.	Bone substitutes, dental implants, orthopedic fixation devices
Glass Fiber Reinforced Polymers	Glass fibers embedded in a polymer matrix provide excellent strength, stiffness, and fatigue resistance with superior radiolucency.	Structural implants in orthopedic and spinal surgeries
Graphene- Reinforced Polymers	Graphene nanomaterials are incorporated into polymer matrices, enhancing mechanical strength, electrical conductivity, and biocompatibility.	Neural electrodes, biosensors, tissue engineering scaffolds
Titanium	Titanium particles or fibers combined with a polymer	Orthopedic and spinal

Table 2.4: Types of composites and their usage in bioimplant fabrication

Composite		Common Uses in
Туре	Description	Bioimplants
Reinforced	matrix offer enhanced mechanical properties while	implants
Polymers	retaining biocompatibility and radiolucency.	
Bioglass-	Bioactive glass particles are incorporated into a	Bone graft substitutes,
Reinforced	polymer matrix, stimulating bone regeneration and	dental implants, tissue
Polymers	tissue integration while providing mechanical support.	engineering scaffolds

#### 2.2.5 Natural Biomaterials:

Natural biomaterials are important for the regeneration of tissues due to their inherent bioactivity, including cell adhesion and proliferation, and they are nontoxic to native healthy tissue. Cellulose, chitin, chitosan, alginate, dextran, glycosaminoglycans, collagen, gelatin, elastin, fibrinogen, laminin, silk, and decellularized extracellular matrix (ECM) derived biomaterials are natural biomaterials. The composition and application of polymer-based biomaterials are discussed in Table 2.5.

#### 2.2.5.1 Collagen

It is a structural protein found in the extracellular matrix of tissues like skin, bone, and cartilage. It offers mechanical support to tissues and is essential for cell adhesion, migration, and regeneration. Bioimplants frequently incorporate collagen-based biomaterials due to their biocompatibility and ability to promote tissue integration. These biomaterials can be formed into scaffolds for tissue engineering, wound dressings, and medication delivery systems (Parenteau-Bareil et al., 2010).

#### 2.2.5.2 Chitosan

Chitosan is a carbohydrate produced from chitin, a naturally occurring polymer in crab exoskeletons. It has biocompatible, biodegradable, and antibacterial qualities, making it ideal for many bioimplant applications. Chitosan-based biomaterials are utilized in tissue engineering scaffolds, wound dressings, and drug delivery systems because they promote cell development, improve wound healing, and deliver therapeutic agents to specific areas. Alginate is a naturally occurring polysaccharide derived from brown algae. When crosslinked with divalent cations, it generates highly biocompatible hydrogels with tunable mechanical characteristics. Cell encapsulation, tissue engineering scaffolds, wound dressings, and drug

delivery systems all use alginate-based polymers in bioimplants. These biomaterials promote cell proliferation and differentiation while allowing for controlled release of bioactive chemicals (Croisi F et al. 2013).

## 2.2.5.3 Silk fibroin

Silk fibroin is a protein from silkworm silk. It is biocompatible, biodegradable, and has excellent mechanical strength. Silk fibroin-based biomaterials are used in bioimplants such as tissue engineering scaffolds, sutures, and drug delivery devices. These biomaterials provide an optimal environment for cell adhesion and proliferation, making them ideal for encouraging tissue regeneration and wound healing (Chai S et al., 2024; Altman GH et al., 2003).

#### 2.2.5.4 Hyaluronic Acid (HA)

It is a glycosaminoglycan found in connective tissues, including synovial fluid and extracellular matrix. It supplies tissues with lubrication, hydration, and viscoelasticity while exhibiting high biocompatibility. Dermal fillers, ocular implants, and osteoarthritis treatments use HA-based biomaterials in bioimplants. These biomaterials improve tissue lubrication, hydration, and joint discomfort caused by degenerative diseases (Wolf KJ et al., 2019).

## 2.2.5.5 Decellularized ECM

It refers to tissues treated to remove cellular components while keeping their architecture and metabolic signals. Biomaterials based on decellularized ECM act as scaffolds for tissue engineering and regenerative medicine applications. They create a natural milieu for cell attachment, proliferation, and differentiation, aiding tissue regeneration and repair in bioimplants for tissues such as skin, heart, liver, and nerves (Zhang X et al. 2022).

Natural Biomaterial	Description	Common Uses in Bioimplants
Collagen	The main structural protein found in the extracellular matrix of various tissues provides mechanical support and promotes cell adhesion and migration.	Tissue engineering scaffolds, wound dressings
Chitosan	A polysaccharide derived from chitin, offering biocompatibility, biodegradability, and antimicrobial properties.	Tissue engineering scaffolds, drug delivery systems
Alginate	A naturally occurring polysaccharide extracted from	Cell encapsulation,

Table 2.5: Natural biomaterials and their application in tissue engineering

Natural Biomaterial	Description	Common Uses in Bioimplants
	brown algae, forming hydrogels with excellent biocompatibility and tunable mechanical properties.	wound dressings, drug delivery
Silk Fibroin	A protein derived from silkworm cocoons, characterized by its biocompatibility, biodegradability, and mechanical strength.	Tissue engineering scaffolds, sutures
Hyaluronic Acid (HA)	A glycosaminoglycan found in connective tissues, providing lubrication, hydration, and viscoelasticity, with excellent biocompatibility.	Dermal fillers, ophthalmic implants, osteoarthritis
Decellularized ECM	Extracellular matrix (ECM) is derived from tissues that have been treated to remove cellular components, retaining the tissue's architecture and biochemical cues.	Tissue engineering scaffolds, regenerative medicine

The field of developing biomaterials has had a notable upsurge in the last several years, mostly due to an unparalleled commitment to healthcare technologies and their commercialization process. Because of their remarkable mechanical durability, metallic and ceramic biomaterials have found extensive application as internal fixation devices and load-bearing implants. The commercially available bioimplants are represented in Figure 2. However, in soft tissue engineering, materials based on polymers and biocomposite materials are becoming serious competitors. Although many alloys and soft materials have been proven reliable for biomedical applications, much can be done to improve their ability to replicate the complexities of natural tissues and gain the trust of the larger industrial community. Bioimplants have witnessed an astounding diversity of improvements in recent years. The subject has seen a boom in innovative endeavors, from the creation of biocompatible materials to the fusion of 3D printing and nanotechnology.



Fig 2.2:Commercially available biomaterials-based implants for repair and regeneration (a) Metallic Dental implant (b) Metallic Femoral implant (c) Metallic Acetabular system (d) metallic Bone plate (e) ceramic Dental screws (f) Polymeric Cranial implant

## 2.3 Summary

Modern medicine relies on bioimplant materials for several therapeutic procedures. These materials are chosen for biocompatibility, mechanical strength, and degrading qualities to optimize performance and patient safety. Bioimplant materials range from metals and ceramics to polymers and natural biomaterials, reflecting medical applications and patient and provider needs. Mechanical strength, corrosion resistance, and biocompatibility make titanium and stainless steel biomaterials desirable. Orthopaedic implants, dental prostheses, and cardiovascular devices use them. Due to its biocompatibility and Osseo integration, titanium is a staple in implant dentistry and orthopaedic surgery. 316L and 316LVM stainless steel alloys are also preferred for long-term bodily implantation due to their durability and corrosion resistance. Bioactivity and wear resistance distinguish ceramic biomaterials like hydroxyapatite and alumina. Orthopaedic implants are coated with hydroxyapatite, a bone mineral, to improve Osseo integration and bone development. Hip and knee replacements use biocompatible, strong alumina to create durable articulating surfaces replicating natural joint motion. Synthetic polymers like polyethylene and biodegradable polymers like PLA and PLGA are versatile and adaptable for bioimplant applications. Biodegradable polymers enable wound healing, medication delivery, and tissue engineering, while polyethylene can withstand wear and biocompatibility. Collagen, chitosan, and hyaluronic acid stimulate cell adhesion, proliferation, and tissue regeneration in a biomimetic environment. Tissue engineering scaffolds, wound dressings, and dermal fillers use these materials. As the major structural protein in the extracellular matrix, collagen is biocompatible and supports cell development, making it a versatile tissue repair and regeneration material. Biodegradable biomaterials that dissolve in the body without implant removal operations or long-term consequences have gained popularity in recent years. Biodegradable iron and magnesium alloys are promising for orthopaedic, cardiovascular, and gastrointestinal temporary implants. Controlled breakdown through corrosion mimics the body's metabolism and promotes tissue repair and regeneration.

Bioimplant materials are evolving and diversifying with advances in materials science, engineering, and biomedical research. Medical application, patient characteristics, and intended outcomes determine bioimplant material selection. Clinical researchers and clinicians can use material features to create creative solutions that improve patient care, quality of life, and regenerative medicine.

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# CHAPTER 3 INTRODUCTION TO ENGINEERING COMPOSITE MATERIALS: PROPERTIES AND APPLICATIONS

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#### ABSTRACT

The intersection of materials science and biomedical engineering has led to significant advancements in the development of materials tailored for various biomedical applications. This study investigates the mechanical properties of materials intended for use in biomedical devices and implants. The mechanical behavior of these materials is crucial for ensuring their compatibility with the physiological environment and their ability to withstand biomechanical stresses. The research encompasses a comprehensive analysis of the tensile strength, elasticity, fatigue resistance, and other mechanical characteristics of biomaterials. The study employs state-of-the-art testing techniques, such as tensile testing, fatigue testing, and impact testing, to evaluate the mechanical performance of materials in simulated physiological conditions. Additionally, the influence of fabrication methods, compositional variations, and surface modifications on the mechanical properties is systematically examined. The goal is to establish a correlation between the material's mechanical behavior and its long-term performance within the human body.

Furthermore, the research explores the implications of these mechanical properties on the design and durability of biomedical implants, prosthetics, and other devices. Insights gained from this study contribute to the optimization of materials for enhanced biocompatibility, structural integrity, and overall reliability in biomedical applications.

This interdisciplinary investigation bridges the gap between materials science and biomedical engineering, offering valuable insights that can guide the design and selection of materials for the next generation of biomedical devices, thereby advancing the field towards safer and more efficient healthcare solutions.

## Abstract:

Composite materials have significantly transformed numerous industries, including aerospace and automotive, by providing a distinctive combination of characteristics that conventional materials are unable to replicate. This chapter explores the intricate world of composite materials, examining their composition, production techniques, applications, and future potential. In this present review, an in-depth investigation and assessment of advanced composite materials is conducted. Multifunctional composite materials and structures have attracted considerable interest for their capacity to incorporate diverse capabilities, including mechanical strength, electrical conductivity, thermal management, and others, within a single material or structure. This chapter presents a summary of the latest advancements in multifunctional composites research and their diverse applications in many industries. This paper encompasses the most recent progress in the field of material synthesis, techniques for characterization, and methods for production. Furthermore, it explores the difficulties and potential opportunities in the realm of multifunctional composites.

## **3.1INTRODUCTION**

Composite materials are engineered materials that are formed from two or more constituent materials that have significantly distinct chemical or physical properties. Other constituent materials may also be used. The combination of these elements results in the production of a superior material that possesses enhanced performance properties [1,2]. Recent years have seen a significant rise in the number of countries throughout the world that are making use of miniaturised products. The need for small items that are integrated, compact, and multipurpose has been steadily increasing over the past period of time. Electronics, microtooling, aerospace, medicine and biomedicine. information technology and telecommunications, and microrobots are some of the industries that have a major demand for these products [3,4]. Because of this, items and technology are getting smaller, all the way down to the microscale, because there is a demand for nanoscale in the not-too-distant future. In point of fact, this trend towards miniaturisation has proceeded quite swiftly over the course of the past two decades, with the primary drivers being items based on silicon (Si) and technologies related to electronics. Nevertheless, goods based on silicon have some inherent limits in terms of geometry (they are only available in two dimensions 2D), material (they are only made of silicon), mechanical performance (they have limited motion, strength, and durability), and cost (they cannot be manufactured in mass quantities).

As a result of these problems, researchers have been looking for different ways to manufacture three-dimensional (3D) microparts that have the desired strength, greater durability, complicated geometry, better surface polish, and cost-effectiveness. These alternatives involve the use of metallic and ceramic alloys and their composites. Additionally, there has been a discernible advancement in the micromanufacturing of various components

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based on metal, ceramic, polymer, and composite materials. There has been extensive research conducted on their manufacturing processes, working principles, size effects brought about by miniaturisation, batch production, and the amount of energy and material that may be saved. However, the possibilities of using bulk materials, including as metals, ceramics, polymers, and their alloys, are so abundant that it may be difficult to attain the greatest material attributes, such as durability and dependability of the components, even when using the most modern techniques. This is because the possibilities of using bulk materials are so saturated. Composite materials, on the other hand, provide an infinite number of possibilities for meeting many of the new industrial requirements. These requirements include extreme mechanical, electrical, magnetic, optical, and thermal qualities, which monolithic materials are unable to meet [4-6]. When compared to conventional materials, composite materials have a number of advantages, the most notable of which are their high strength, toughness, stiffness, and resistance to creep. As a result, composite materials experience significantly less corrosion, wear, and fatigue. When compared to individual alloys, a composite of copper-aluminum (Cu-Al) clad is lighter, stronger, more solderable, and more electrically conductive than the individual alloys. This is achievable by selecting an appropriate combination, which also makes it possible to achieve specific features. Therefore, composite materials are essential in a wide range of applications that are being used today, ranging from the microscale to the nanoscale [7-10]. The first step is to present a fundamental definition that defines the notion of micromanufacturing and composite materials. This is then followed by a categorization that is both comprehensive and straightforward. Due to the vastness of the subject of composite materials, the primary emphasis is placed on composite materials that are based on metals and ceramics, while organic and polymeric composites are not taken into consideration. Therefore, cutting-edge micromanufacturing methods of metal matrix composites (MMCs) and ceramic matrix composites (CMCs) are thoroughly discussed, with an emphasis on the most recent breakthroughs as well as future trends and research scope. These elements include size effects and matrix-reinforcement interfacial features, among others [11-14]. By utilising a unique micromanufacturing technique known as hot compaction diffusion bonding (HCDB), a bimetallic composite consisting of ceramic and steel was successfully manufactured. This is done in order to illustrate the potential for the micromanufacturing of composite materials. A presentation and discussion of the results obtained are provided. Finally, a presentation is made regarding the advancements that have been made in analytical modelling and simulation of the micromanufacturing of composite materials. In addition to this, a comparison analysis is offered, which demonstrates the

prospective future applications of composite materials, as well as the potential and versatility of generating composite materials. The purpose of this review is to provide assistance in advancing micromanufacturing technology for the purpose of fabricating miniaturised composite components that possess desirable qualities in order to satisfy the increasing demand in the industrial sector.

# 3.2 FUNDAMENTALS OF MICROMANUFACTURING AND COMPOSITE MATERIALS3.2.1 MICROMANUFACTURING

There have been a number of scholars and industrial professionals that have attempted to define and/or discuss the notion of micromanufacturing, as was described in earlier studies. The most basic description of micromanufacturing describes it as a method of making smalldimensional parts that take up less space, use fewer resources, and need less energy. This is accomplished by reducing the overall production process to a smaller scale. It is possible to significantly lower the mass of the system due to the fact that the size of the equipment has been reduced. This leads to a reduction in the amount of energy consumed, the overhead costs, the number of materials required, the amount of noise, and the amount of pollution, and ultimately makes it possible to have a production process that is more ecologically friendly and viable [15]. Micromanufacturing results in better production rates since it eliminates the need for a longer manufacturing cycle and increases the speed at which tools are used. These components are built of composite materials that combine the hardness of ceramic with the strength of steel. These composite materials are reinforced with aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) and 316 L stainless steel. A fresh approach, which was referred to as the "soft moulding technique," is utilised. According to the research the hardness of 316 L stainless steel is increased by 1.8 times, which made it possible for the components to be more effective in terms of strength, hardness, and resistance to wear.

## **3.2.2 COMPOSITE MATERIALS**

Due to their distinctive properties that overcome the limitations of monolithic materials, composite materials are widely employed as advanced multifunctional materials in various industries including electronics, aeronautics, medicine, automobiles, and machining tools. This increased usage is a result of the rapid growth of the modern manufacturing industry. As an illustration, tungsten carbide (WC) exhibits high hardness and wear resistance, but it lacks strength and toughness. On the other hand, high strength steel has exceptional strength and toughness, but it has low hardness and wear resistance [16]. The utilisation of a stratified composite material consisting of tungsten carbide (WC) and high-strength steel allows for the synergistic integration of their respective benefits, rendering it suitable for a wide range of

technical applications. A composite material is a substance made by combining two or more distinct materials that have significantly diverse physical and chemical properties. The resulting material possesses unique features that are different from those of its separate components. The various components remain separate and distinct throughout the finished structure, distinguishing composites from mixes and solid solutions. Therefore, any composite material inherently consists of two main components: a matrix, which is a continuous phase, and a reinforcement, which is a discontinuous phase. If there are three or more elements, the composite is referred to as a hybrid composite [17-19]. Natural composites, such as wood, bone, and tissue, are abundantly present in our surroundings. Within the industrial sector, the majority of composites are derived from metals, ceramics, and polymers. Nowadays, there is a growing significance placed on the use of Metal Matrix Composites (MMCs) that incorporate reinforcements such as ceramic particles, whiskers, and fibres. The CMCs are regarded as the most recent additions to the field. This study specifically examines the process of micromanufacturing metallic- and ceramic-based composite components.

## 3.2.3 METAL MATRIX COMPOSITES

Manufacturing companies have a perpetual need for novel materials. The development of new materials is driven by the desire to achieve improved mechanical qualities, decreased weight, and reduced cost. Engineers are exploring the use of composites to enhance the strength, stiffness, and longevity of materials, while traditional bulk materials have limitations. Metals and their alloys are mostly produced and formed in huge quantities, but they can also be closely coupled with another substance to enhance their performance. The materials obtained are Metal Matrix Composites (MMCs). Considerable progress has been made in the development of Metal Matrix Composites (MMCs) in recent decades, leading to their integration into crucial industrial applications. These innovative materials have expanded the range of possibilities for current material research and advancement. MMC features can be intentionally incorporated into the material, tailored to specific requirements, and influenced by the intended use. MMC's provide enhanced material characteristics in comparison to polymer matrix composites [20-22]. For instance, as compared to resin, metal matrices offer superior tensile and shear moduli, a higher melting temperature, a lower thermal coefficient of expansion, improved dimensional stability, enhanced joinability, increased ductility and toughness, and the capability to achieve full density. MMCs primarily comprise a matrix material made of metal or alloy, together with a reinforcement of various types and forms.

## 3.3 PROPERTIES OF COMPOSITE MATERIALS

## **3.3.1 MECHANICAL PROPERTIES**

Composite materials have a high strength because the reinforcing fibres or particles, which are responsible for bearing the majority of the load, are present. When compared to the stiffness of individual constituent materials, composites have a higher stiffness, which results in improved structural integrity [23-25]. In terms of toughness, composites are superior to monolithic materials in terms of their ability to endure impact and fatigue loads.

## 3.3.2 PHYSICAL PROPERTIES

Composite materials are often lighter than metals, which makes them appealing for applications that are sensitive to weight. The thermal conductivity of composites might vary depending on the matrix and reinforcement used in the construction of the composite. The electrical characteristics of composites can be adjusted to specific purposes and vary depending on the ingredients that make up the composite.

## 3.4 APPLICATION OF ENGINEERING COMPOSITE MATERIALS

The uncommon combination of qualities exhibited by multifunctional composite materials makes them highly promising for a diverse array of technical applications.

## 3.4.1 AEROSPACE AND AUTOMOTIVE APPLICATIONS

Multifunctional composites provide substantial weight reduction, which is vital for aerospace applications where optimising fuel efficiency and payload capacity is of utmost importance [26-28]. These materials have the potential to enhance the structural robustness of aeroplanes, hence enhancing safety and dependability.

## 3.4.2 RENEWABLE ENERGY

Utilising multifunctional composites in wind turbine blades can enhance their lightweight nature, bolster their strength, and enhance their durability, hence promoting heightened energy efficiency and reliability. These materials can enhance the structural integrity, decrease weight, and incorporate energy storage and conversion capabilities in solar panels.

## 3.4.3 INFRASTRUCTURE AND ELECTRONICS GOODS

Construction can benefit from the usage of multifunctional composites, which offer enhanced strength, reduced weight, and increased durability. This results in lower maintenance expenses and improved sustainability. Wearable devices can utilise multifunctional composites due to their versatility, longevity, and capacity to incorporate sensors and renewable energy components [29,30]. Utilising lightweight and robust composites can enhance the longevity and efficiency of electronic devices such as cell phones, laptops, and

tablets. Conductive flexible composite materials have the potential to facilitate the creation of flexible and rollable displays for a wide range of uses.

## 3.4.4 PROSTHETICS AND MEDICAL DEVICES

Multi-functional composites can be customised to possess biocompatible characteristics, rendering them appropriate for use in implants and medical devices. Advanced manufacturing techniques enable the production of prosthetics and implants that are customised for each patient and have improved mechanical qualities.

## 3.5 DESIGN AND FABRICATION

Due to the complexity of its structures, the design of multifunctional composite materials and structure (MFCMS) is a difficult task to accomplish. However, the selection of materials for fabrication and the process of fabrication plays a very important role in achieving the desired functional capabilities. This is accomplished by maintaining the initial structural functions while also incorporating additional non-structural functions. The incorporation of functional devices that demonstrate additional capabilities inside the structural materials is a method that can be utilised to facilitate the design of multifunctional materials and structures. Xiao et. al [31] have conducted a demonstration of implanted thin film lithium energy cells. These cells are utilised for the purpose of energy storage. These energy cells are constructed with laminated composites, and the difficulties in designing embedded devices, which reflects the structural function of composites, is also explored. The crack propagation of embedded composite laminate was shown to be affected by slippage, as well as frictional forces, according to the results of both experimental and numerical studies. Lubineau and Rahaman [32] have investigated the current developments that have been made in the enhancement of the degrading properties of MFCMs through the utilisation of epoxy that is packed with carbon-based nano-stimulants. Naebeet al.[33] have conducted research on the fracture initiation and propagation in multifunctional polymer composites. In their discussion, they also discussed the development of nature-inspired self-healing and autonomic repair in composite materials. The fatigue characteristics and mode-II inter-laminar fracture toughness of multifunctional graphite epoxy composite material were also demonstrated in a specialised investigation that was carried out by NASA. Another investigation that was carried out by Pang et. al [34] have demonstrated that the segregated structures of conductive polymer composites (CPCs) were responsible for the characteristics of these materials. Additionally, the ultralow percolation behaviour, electromagnetic interference shielding, and thermoelectric shielding of CPCs were examined in this study. Nevertheless, Qian [35] was able to bring about an increase in the strength of MFCMs by employing carbon nanotubes as the filler for

such materials. This was observed and accomplished. Through a review of the multifunctional polymer composite that was filled with carbon particles and the absorption of microwaves in such materials, the study offered a comprehensive assessment of the enhanced electrical, thermal, and mechanical capabilities. Another study that Brosseau [36] investigated revealed that the geometry, morphology, and composition of carbon particles all have an effect on the amount of electromagnetic radiation that is absorbed by the particles. In order to develop high-performance materials that are utilised in the aviation industry, Rafique et al. [37] investigated epoxy resins and hardening methods that make use of carbon nanotubes. Al-Saleh and Sundararaj [38], on the other hand, carried out an in-depth analysis of the mechanical properties of MFCMs by measuring the tensile properties, fracture toughness, dynamic mechanical properties, and rheological properties. This gave them the opportunity to examine the mechanical properties in greater detail. According to the findings, the processing conditions and procedures had a substantial impact on the characteristics of the VGCNF/polymer composites during the manufacturing process. Polylactic acid (PLA)-based materials were the subject of an extensive study that was carried out by Armentano et al. [39] in the field of bio-nano multifunctional composites. The study demonstrated the characteristics, present trend, and potential applications of these materials. The purpose of this work is to investigate these composites and their potential applications in tissue engineering and PLA modification approaches. Dicker et al. [40] have conducted research on green composites, which are nature-based multifunctional materials. Their findings demonstrated that green composites have the potential to be utilised in a variety of applications. Moreover, a review of the mechanical properties, variable fibre properties, renewability, biodegradability, and toxicity of multifunctional green composites was included in another study. As a result of their favourable economic and environmental perspective as well as their capacity to fulfil human requirements, natural composites were found to have a promising potential for usage in infrastructure applications, as demonstrated by the findings.

## 3.6 FUTURE SCOPE OF THE ENGINEERING COMPOSITE MATERIALS

There is a wide range of industries that could benefit from the use of multifunctional composite materials in engineering applications in the future. These industries include aerospace and automotive, as well as renewable energy, construction, healthcare, electronics, manufacturing, and defence. As scientific research and technological advancements continue to grow, these materials will play an increasingly vital role in the process of addressing the complex difficulties that the modern world presents. Furthermore, there is a growing interest in recycling and circularity in big composite components. As a result, there are workshops

and conferences that are dedicated to developing innovation and sustainability within the composites recycling sector. Graphene and its derivatives are also attractive additions for increasing the qualities of composite materials. By cooperating with industry professionals, organisations can increase their chances of effectively sourcing, formulating, and deploying these materials.

#### **3.7 CONCLUSION**

Composite materials, consisting of two or more types of materials, have attracted considerable interest because of their versatile features and possible uses. Composite materials, such as fiber-reinforced polymer composites, provide numerous advantages, including a high ratio of strength to weight, long-lasting durability, resistance to corrosion and wear, and exceptional mechanical qualities. A significant benefit of multifunctional composite materials lies in their capacity to be customised to individual needs through the manipulation of constituent materials in terms of their types, quantities, and configurations. This allows for the enhancement of characteristics like as electrical conductivity, thermal stability, and impact resistance. In addition, the utilisation of sophisticated manufacturing methods, such as additive manufacturing and automated fibre placement, has enhanced the potential of multifunctional composites.

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# CHAPTER 4 SYNTHESIS OF ZNO NANOSTRUCTURED CFRP COMPOSITES BY HYDROTHERMAL METHOD AND ITS MACHINABILITY ANALYSIS

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#### ABSTRACT

This chapter reveals the growth of ZnO nanostructures on woven carbon fibres (WCF) by hydrothermal technique using various process parameters. The impact of molar concentration of precursor solution on structural morphology of the nanostructure has been identified. A variety of nanostructures have been developed on CFRP composites such as nanoflakes, nanorods, and nanowires by varying the molar concentration. In addition, the machinability of ZnO nanostructured CFRP composites has been investigated using high speed micro drilling process. The machinability parameters have been compared for three different hybrid composites; i.e., unstructured CFRP, 35 mM ZnO nanostructured and 45 mM ZnO nanostructured CFRP composites. The drilling operations have been performed applying three different levels of spindle rotational speeds (25000, 37500, and 50000 rpm) and feed rates (1 µm/rev, 3 µm/rev, and 6  $\mu$ m/rev). It has been observed that the delamination factor and the circularity of drilled holes were improved significantly for the nanostructured composites as compared to unstructured one. Additionally, the burr height has been increased for those nanostructured composites having higher molar concentration. Moreover, better results have been observed at a combination of higher rotational speed and higher feed rate for ZnO nanostructured CFRP composites.

## 4.1 INTRODUCTION

ZnO nanostructures attained immense research interest due to their inherent physical properties and applicability in various electronics and photonics industries. ZnO has superior semiconductor properties due to the appearance of the symmetric focal point, wurtzite structure in association with excellent thermal and chemical stability. Wide energy band (3.37eV) and binding energy (60 meV) have improved the semiconductor properties of ZnO<sup>[1]</sup>. Additionally, the applications of ZnO nanostructures have been emerged for several

devices such as LEDs, optical modulator waveguides, solar cells, acoustic filters, gas sensors and varistors <sup>[2][3][4][5]</sup>.

For the last two decades, different approaches have been adopted to develop one-dimensional nanomaterials such as vapour-liquid-solid technique, sol-gel technique, template-assisted solvothermal, electrochemical processes, metal-oxide chemical vapour deposition, physical vapour deposition, vapour-liquid-solid epitaxial, laser pulsed deposition, ultrasonic synthesis and epitaxial electro deposition <sup>[6][7][8][9]</sup>. All the above techniques require large experimental setup with controlled environment such as fixed gas concentration, high temperature and rate of flow. Besides, the modified solvothermal process (Hydrothermal technique) is a simple technique that requires a low-cost setup, and low-temperature phenomenon <sup>[10]</sup>. Meanwhile, this technique can develop a wide variety of eco-friendly metal oxides.

The density and orientation of the implanted ZnO nanostructures are dependent on several factors associated with hydrothermal techniques such as seeding treatment <sup>[11]</sup>. Apart from that, various factors such as orientation of sample, time, temperature and pH affect the growth of nanostructures <sup>[12]</sup>. The size of the nanostructured can be enlarged by increasing the growth temperature and selecting proper chemical agents <sup>[13][14]</sup>. Similarly, the size of nanostructures can be modified by varying the growth duration  $^{[15]}$  and concentration of Zn2+  $^{[16]}$ . The presence of hydroxyl ions governs the chemical reactions for the development of nanostructures. Meanwhile, the pH of the solution governs the amount of hydroxyl ions; and therefore, regulates the growth of nanostructures <sup>[17]</sup>. The growth of ZnO rods using the new hydrothermal process has been initiated by Vayssieres et al. <sup>[18]</sup> in the early 2000s. The technique utilized equivalent molar emulsion of HMTA and (Zn(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O) on different substrates by seed plantation of resynthesized nanoparticles grown into ZnO rods. Further, Hazarika et al.<sup>[19]</sup> have developed ZnO nanorods on Kevlar fabric at a moderate temperature using this technique. Meanwhile, the interfacial strength of the composites was increased by those nanostructures. Additionally, enhancement in energy absorbing capacity of the composite has been accomplished by cross-linking of ZnO nanorods on Woven carbon fibres<sup>[20]</sup>. Moreover, Ehlert et al. <sup>[21]</sup> investigated the surface chemistry and growth mechanisms of ZnO nanostructures at the interfacial carbon fibre content. The concentration of ketone groups played a vital role in improving the interfacial strength.

Researchers investigated that physical and chemical properties of the final materials have been highly influenced by size, shapes, compositions, and orientation of the nanostructures. For example, star like morphology of the nanostructures enhances the photo-degradation rate of methylene blue <sup>[22]</sup>. However, previous researches was mostly focused on metallic substrate rather than hybrid polymer composites. Hybrid composite like Carbon fibre reinforced polymer (CFRP) composites have wide applications in aerospace and automobile industries owing to their high strength-to-weight ratio and prevailing stiffness. Nevertheless, based on a few investigations, the interfacial parameters of hybrid composites can be improved by morphological variations of ZnO nanostructures such as nanostructured fiber-reinforced polymer composites. Additionally, the structural properties are improved as well by the morphological variations of implanted nanostructures. For obtaining the morphological variations of morphological variations are required to be adapted. Variable conditions of seeding and growth reactions are required to ver hybrid composites.

## 4.2 SYNTHESIS OF ZNO NANOSTRUCTURES BY HYDROTHERMAL TECHNIQUE

Unmodified Woven Carbon Fibre (WCF) of grade T-300 consisting of 3000 wires, 200 GSM size, 7  $\mu$ m diameter and 0.25 mm thickness designed as plain woven and bi-directional structure and chemicals precursor of analytical grade were used in the process. Hydrothermal technique has been used to synthesis the ZnO nanostructured CFRP composites from the woven carbon fibre. The first step of the technique involved the preparation of Seed solution and growth solutions. For seed solution, initially 0.30 g of Zinc Acetate dihydrate (Zn(CH<sub>3</sub>COO)<sub>2</sub>.2H<sub>2</sub>O) and 420 ml of ethanol are mixed together using a magnetic stirrer at 75 °C for 45 min. Similarly, another solution has been prepared by mixing 2 mM NaOH with 80 ml ethanol at 70°C for 15 min. Both the solutions were mixed together adding some extra ethanol using the magnetic stirrer for 45 min and cooled at atmospheric temperature maintaining pH of 5-6. The final solution has been used as the seed solution for seeding treatment of the samples. The chemical reactions involved in preparing the seed solution are enlisted as:

$$Zn^{2+} + 4OH^{-} \leftrightarrow [Zn(OH)_{4}]^{2-}$$
(1)

$$\left[\operatorname{Zn}(\operatorname{OH})_{4}\right]^{2^{-}} \leftrightarrow \operatorname{ZnO} + \operatorname{H}_{2}\operatorname{O} + 2\operatorname{OH}^{-}$$

$$\tag{2}$$

$$ZnO_2^{2^-} + H_2O \leftrightarrow ZnO + 2OH^-$$
(3)

$$ZnO + OH^{-} \leftrightarrow ZnOOH^{-}$$
 (4)

Further, the growth solution has been prepared by mixing HMTA and zinc nitrate hexahydrate. It was an equimolar solution of the homogeneous mixture of 15 mM HMTA in 650 ml distilled water prepared by stirring for 20 min. Further, 15 mM of  $(Zn(NO_3)_2.6H_2O)$  has been mixed with the aqueous HMTA solution and stirred for 45 min at room temperature maintaining its pH at 6-8. With the same process, another ZnO growth solution with a different concentration (25 mM) has been prepared. The chemical reactions involved in the preparation of the growth solution are enlisted below:

 $C_6H_{12}N_4 + 6H_2O \leftrightarrow 6HCHO + 4NH_3$  (5)

$$C_6H_{12}N_4 + Zn^{2+} \leftrightarrow [Zn(C_6H_{12}N_4)]^{2+}$$
 (6)

 $NH_3 + H_2O \leftrightarrow NH_4^+ + OH^-$ (7)

$$Zn(NH_3)_4^{2+} + 2OH^- \leftrightarrow ZnO + 4NH_3 + H_2O$$
(8)

Thenceforth, the WCF has been cut into pieces of size 120 mm  $\times$  100 mm and cleaned by ethanol-acetone solution to extract foreign impurities. Further, the oxide layers have been removed from the sample surfaces by desiccating those samples. The treated samples have been dipped in the seed solution for 15 min followed by removal of solvent via thermal annealing at 120°C for 15 minutes. This process is called seeding process which forms a ZnO seed layer on the WCF surface which would further act as growth site of ZnO nanostructure. The seed treatment of WCF with ZnO particles has been performed to restrain the thermodynamic obstruction by giving nucleation locales. It is a significant factor to accomplish uniform development of ZnO nanostructure through aqueous procedure. Further, the seeded samples have been heated for 4 hours at constant temperature of 90°C and rinsed by DI-water for 30 min to stop further development of ZnO nanostructure. Eventually, the nanostructured WCF samples have been dried for 24 hours under ambient condition. The schematic representation of the synthesis technique used for growing the ZnO nanostructures is depicted in Fig. 4.1.


**Fig. 4.1:** Schematic representation of the synthesis technique used for growing the ZnO nanostructures on WCF

Fig. 4.2 depicts the influence of seeding treatment, growth solution treatment and the development of ZnO nanostructures on Woven Carbon Fibre. It has been observed that ZnO nanorods are nucleated only at a few portions on the WCF surface by the seeding treatment as shown in Fig. 4.2 (a). Besides, the growth solution treatment produces low quality nanorods on WCF as shown in Fig. 4.2 (b) which proves the fact that nanorods can be grown by growth solution treatment only. Therefore, high quality nanorods have been produced on WCF samples by successive treatment of seeding followed by growth treatment. The molar concentration of the precursor solution has influenced the size and dimensions of the ZnO nanostructures implanted on WCF. For example, ZnO nanoflakes have been developed over the WCF samples at higher molar concentrations (70 mM) as depicted in Fig. 4.2 (d). On the other hand, nanowires of diameter less than 100 nm and a height of 1.2 µm have been implanted on WCF sample at a lower concentration (10 mM) as depicted in Fig. 4.2 (c).



**Fig. 4.2:** FESEM images of (a) seeding treated, (b) growth solution treated ZnO nanostructures, (c) developed ZnO nanostructures at a molar concentration of 10 mM, (d) developed ZnO nanostructures at a molar concentration of 70 mM on WCF samples<sup>[10]</sup>

#### 4.3 MICROMACHINING OF ZNO NANOSTRUCTURED CFRP COMPOSITES

Miniaturization is attaining global interest in modern manufacturing. Mechanical micro machining is an adaptable emerging technology to produce miniaturized products for several industrial applications. However, the miniaturized components can be easily subjected to rapid deformation due to the action of cutting force as a result of small component stiffness. This phenomenon further affects dimensional accuracy. Additionally, the small stiffness of the micro tools enhances the tendency of breakage during mechanical micromachining. Moreover, the low material removal rate in micromachining further promoted the requirement of high speed micromachining technologies <sup>[23]</sup>. Additionally, lower chip load in

high speed machining resulted in a decrement of cutting force. Subsequently, the deformation of the miniaturized workpieces or micro cutting tools can be reduced upto certain extent. Micro drilling is one of the mechanical micromachining technologies used to develop micro holes on the miniaturized components having diameters ranging from a few microns to one millimeter. However, rapid burr formation and surface degradation are two major challenging issues in mechanical micro drilling that affect the dimensional accuracy and precision of the miniaturized product <sup>[24]</sup>.

Research on micromachining has been emphasized on metal matrix composites. For example, diamond turning operations were carried out on SiC reinforced Al metal matrix composites; and nano-metric finishing with a cutting force of less than 1 N have been achieved. Favorable outcomes have been achieved at a combination of higher cutting speed, lower feed rate, and lower depth of cut <sup>[25][26]</sup>. However, very few research has been focused on polymer composites. Some complicated issues such as smearing of the matrix content, delamination, intra-laminar cracks, uncut fibre content are predominant which further restrained the application of mechanical micromachining for carbon fibre reinforced polymer composites (CFRP) <sup>[27]</sup>. In addition, poor hole quality in terms of circularity and delamination factor further restricts the application of micro drilling operation on CFRP composites; especially, when the feed rate was comparable with tool cutting edge radius <sup>[28]</sup>. Meanwhile, all the machining parameters such as cylindricity, circularity, and delamination factor have been decreased upto certain extent at a combination of lower feed rate and higher spindle speed <sup>[29]</sup>. Although, no previous study has explored the machinability of nanostructured polymer composites.

To determine the machinability of the ZnO nanostructured composites, high speed micro drilling operations have been performed on those samples following the synthesis of the nanostructures. Machining operations have been performed on high speed micromachining center (Model V60, Developed in microfabrication laboratory of Indian Institute of Technology (ISM), Dhanbad) which is shown in Fig. 4.3 as the experimental setup. The spindle can rotate upto 60000 rpm that was mounted on the vertical Z-stage of the micromachining center. The travel range of each linear stage was 150 mm with an accuracy and repeatability of 2.5  $\mu$ m. Machining has been carried out on three different samples. The first one was unstructured CFRP where no nanostructure has been grown, followed by 35 mM ZnO nanostructured CFRP, and 45 mM ZnO nanostructured CFRP. TiAlN coated

cemented carbide micro drill having diameter of 0.5 mm (Axis microtools made) was utilized for machining. The depth upto which the drilling has been performed was 1 mm. The laminated composites contained multiple layers of nanostructured WCF and the thickness of each layer was 0.4 mm. Therefore, each drilling operation could dig upto the third layer of the WCF sample. Three different levels of spindle rotational speeds (25000, 37500, and 50000 rpm) and feed rates (1  $\mu$ m/rev, 3  $\mu$ m/rev, and 6  $\mu$ m/rev) have been incorporated. The selection of the process parameters was based on the recommendation of manufacturer of the cutting tool and the capability of the machine movement. The design of experiments has been prepared based on the Taguchi method (L9 orthogonal array design); and therefore, a total of 27 machining operations have been performed. The machining condition was dry. All the experiments have been repeated twice.



**Fig. 4.3:** Experimental setup for the high-speed micro drilling operations with an extended view of the machining zone

# 4.4 CHARACTERIZATION OF MACHINED SAMPLES OF NANOSTRUCTURED CFRP

# 4.4.1 DELAMINATION AND CIRCULARITY

The delamination factor can be expressed in several ways. However, most of the researchers calculated the delamination factor as the ratio total area of the hole including the delamination or damaged portion  $(A_{max})$  to the actual area of the hole (tool diameter,  $A_0$ )  $\left(F_d = \frac{A_{max}}{A_0}\right)^{[30][31]}$ . Therefore, it can be expressed as the ratio of maximum diameter including the delaminated portion  $(D_{max})$  to the actual diameter of the drilled hole  $(D_0)$   $\left(F_d = \frac{D_{max}}{D_0}\right)^{[32]}$ . In general, the delamination has been examined at the entry section of the drilled hole. Several approaches have been illustrated to reduce the delamination defects. For example, Rodriguez et al. <sup>[33]</sup> utilized minimum quantity lubrication or hybrid MQL

technique in addition with liquid CO<sub>2</sub> to reduce the delamination factor in CFRP composite. For nanostructured CFRP, Fig. 4.4 depicts the variation of delamination factor for different hybrid composite samples (unstructured, 35 mM ZnO nanostructured, and 45 mM ZnO nanostructured CFRP composite) with the variation of spindle rotational speed and feed rate. The delamination factor has been found to be reduced significantly for nanostructured composite as compared to unstructured CFRP. The percentage reduction was ranged from 4.5% to 18% for the nanostructured composite. Higher lamination strength of the nanostructured hybrid composite was responsible for the reduction of delamination. Meanwhile, the delamination factor has been increased imperceptibly for 45 mM ZnO nanostructured CFRP than 35 mM. However, a slightly different trend has been observed for the feed rate of 6  $\mu$ m/rev where the delamination factor has been lower 45 mM ZnO nanostructured composite than 35 mM. Higher concentration of the ZnO nanostructures sometimes induced brittle fracture along the drilled hole which resulted in higher delamination in some cases along the periphery of 45 mM ZnO nanostructured composited. Additionally, elevated rotational speed of the spindle reduced the delamination factor in most of the cases. Stable machining conditions at higher rotational speeds reduced the ploughing effect and the tendency of delamination defects. However, an opposite trend has been found at lower feed rate (1  $\mu$ m/rev), where the delamination factor has been reduced initially by increasing the rotational speed from 25000 rpm to 37500 rpm; and further increased when the rotational speed has been shifted to 50000 rpm. At a federate of 1 µm/rev, the tendency of ploughing was significant which increased the tendency of delamination. The delaminated portions have been scattered at higher rotational speed (50000 rpm) and enhanced the defects. The delamination factors of the nanostructured composites have been reduced at higher feed rate as shown in Fig. 4.4. This was due to higher uncut chip thickness which established stable machining conditions reducing the ploughing phenomenon. Only the unstructured composite has a different tendency for the delamination factor with feed rate. It has been initially increased (1 µm/rev to 3 µm/rev) and then decreased (at 6 µm/rev) with increasing the feed rate.



**Fig. 4.4:** Variation of delamination factor with spindle rotational speed and feed rate for unstructured and nanostructured CFRP composites

Fig. 4.5 illustrates the optical micrographs of the overall drilled holes at 25000 rpm and 1  $\mu$ m/rev for the unstructured hybrid composite and nanostructured composites (35 mM ZnO nanostructured and 45 mM ZnO nanostructured CFRP composites) experimented by optical profilometer (Zygo made, Model: Newview 9000). It has been ascertained from the micrographs that nanostructured composites have possessed better circularity for the hole geometry as compared to the unstructured composite. The implanted nanostructures have improved the fibre strength of hybrid composite material. This phenomenon resulted in a uniform cutting of the laminated structures with less delamination defects. Consequently, the circularity of the drilled holes has been improved as a result of reduction in uncut fibre contents. The circularity has shown a similar trend as the delamination factor with spindle rotational speed. Better circularity has been observed at higher spindle rotational speed for the nanostructured composites.



(c) 45 mM ZnO nanostructured

Fig. 4.5: Optical micrographs of the drilled holes at 25000 rpm and 1 µm/rev

# 4.4.2 BURR FORMATION

Higher burr height has been observed at higher molar concentration of the ZnO nanostructures (45 mM). The delamination defects have been higher for 45 mM nanostructured composite as compared to 35 mM. The delaminated portions might result in material pile-up near the hole periphery. Additionally, the uncut fibre layer near the hole periphery has been plastically deformed and piled up as burr. The variation of burr height with spindle rotational speed and feed rate are depicted in Fig. 4.6 for 35 mM and 45 mM nanostructured CFRP composites. Additionally, the burr height has been reduced at higher spindle rotational speed and feed rate. High rotational speed has resulted in high shear angle which led to lesser friction and friction-induced plastic deformation. Furthermore, high feed rate precipitated increased uncut chip thickness and reduction in ploughing effect. This phenomenon further reduced the plastic deformation of the uncut layer and delaminated fibre

contents. Fig. 4.7 represents the burr formation on different nanostructured composites at varying process parameters.



Fig. 4.6: Variation of burr height with spindle rotational speed and feed rate



(a) 35 mM ZnO nanostructured; 25000 rpm, 1 µm/rev

(b) 45 mM ZnO nanostructured; 25000 rpm, 1  $\mu m/rev$ 



(c) 35 mM ZnO nanostructured; 37500 rpm, 1  $\mu m/rev$ 



(d) 45 mM ZnO nanostructured; 37500 rpm, 1 µm/rev

Fig. 4.7: Burr formation on different nanostructured hybrid composites

#### 4.5 CONCLUSION

In conclusion, this chapter describes the implantation of ZnO nanostructures on woven carbon fibre samples by hydrothermal method and their impact on machinability of hybrid CFRP composites. The size and shapes of the nanostructures can be controlled by adapting various synthesis conditions such as pH of the solution, molar concentration, time and temperature. Various nanostructures can be generated by varying the molar concentration of the precursor solution. The nanostructures can be nanorods, nanowires, nanoflakes, nanoflowers and nanopallets. It is evident that the molar concentration of precursor solution was a predominant factor for structural morphology of the nanostructures and the nucleation density. Additionally, the high speed micro drilling operation of the ZnO nanostructured CFRP composites reveals that the delamination factor has been reduced for nanostructured composite as compared to unstructured one. This phenomenon further improved the circularity of the drilled holes on nanostructured composites. The lamination strength of ZnO nanostructured CFRP composite was higher which restricts the delamination defects. The burr height was profound for hybrid nanostructured composites having higher molar concentrations. Therefore, 45 mM ZnO nanostructured composite possessed larger burr height than 35 mM. Moreover, convenient results have been accomplished at a combination of higher rotational speed with higher feed rate while micro drilling of nanostructured CFRP composites.

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