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About the Journal

Sustainable Manufacturing and Foundry Practices (SMFP) is an international journal established by *The Institute of Indian Foundrymen (IIF)*. Building on IIF's long institutional 76-year legacy, SMFP provides a respected forum for scholarship and practical insight that advances the metal casting and manufacturing communities worldwide.

The Institute of Indian Foundrymen, founded in 1950 and headquartered in Kolkata, is the national association for India's metal casting industry. IIF operates through four regional offices (Kolkata, Delhi, Mumbai, and Chennai), four Centers of Excellence (Chennai, Delhi, Kolkata, and Pune), and 25 chapters. This nationwide network underpins SMFP's close connection to industry needs and its capacity to translate technical knowledge into operational practice.

SMFP occupies a distinctive position at the interface of metallurgy, mechanical engineering, and industrial sustainability. The journal publishes original research, reviews, industrial case studies, and policy analyses that are relevant to both academic inquiry and industrial application. The journal prioritises work that guides the adoption of technology, enhances process performance, and fosters sustainable industrial transitions.

SMFP serves a broad readership that includes academic researchers, practicing engineers, industry leaders, policymakers, and technology developers. The journal aims to bring about knowledge exchange and technology transfer by presenting work that is both scientifically robust and practically useful, thereby supporting improvements in productivity, resource efficiency, and environmental performance.

Aims and Scope

Sustainable Manufacturing and Foundry Practices (SMFP) is a premier **Open Access** journal, published **bi-annually**, which is an initiative of *The Institute of Indian Foundrymen (IIF)*. The journal aims to provide a comprehensive platform for the publication of a broad range of high-quality research and review papers in those fields of scientific and engineering research appertaining to the development, application, and optimisation of **foundry technologies** and **sustainable manufacturing practices**. Papers describing original work in these areas are submitted to rigorous **double-anonymised peer refereeing** prior to acceptance for publication. This strict process ensures objective evaluation based solely on scientific merit and contribution to the field.

While many journals focus separately on materials science or production engineering, *SMFP* fills a critical gap by bridging the divide between fundamental solidification science and applied sustainable manufacturing. We aim to connect the lab to the ladle, offering a unique venue for research that addresses the dual challenges of digital transformation and environmental stewardship.

We encourage high-quality original research and timely review submissions that offer insights into the processing, characterisation, modelling, prediction, and optimisation of foundry systems. Our goal is to make a significant contribution to the field by publishing work that not only advances theoretical understanding but also demonstrates economic viability and pathways towards net-zero, zero-defect manufacturing.

The scope of *Sustainable Manufacturing and Foundry Practices* encompasses, but is not restricted to, the following core areas:

- Advanced casting processes and solidification science
- Cyber-physical production systems (CPPS) and IIoT
- Digital Twins and smart process control
- Industrial AI, machine learning, and prognostics
- Decarbonisation and energy-efficient melting strategies
- Circular economy, material reclamation, and closed-loop flows
- Life cycle engineering (LCE) and sustainability metrics
- Hybrid and additive manufacturing workflows
- Sustainable binders, coatings, and refractories
- Computational modelling (CFD/FEA) and simulation
- Zero-defect manufacturing and quality engineering

The journal publishes **Original Research Articles**, **Comprehensive Review Articles**, **Industry Case Studies** (emphasizing industrial application and economic viability), **Technical Communications**, and papers focused on **Policy and Standardization**. The target audience includes global academic researchers, R&D professionals, technical managers, and policy stakeholders in the metal casting and advanced manufacturing sectors, with a commitment to advancing the state of knowledge worldwide.

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Contents

Articles

- A Review of Microstructural Evolution and Numerical Studies
in Semi-solid Metal Processing
*Ruchika Dhyani, Debdip Chakraborty, Himadri Roy,
Dipankar Chatterjee and Sudip Samanta* 95
- Micro-fabricated Electrochemical Sensors for Quantitative
Analysis of Tea Polyphenols
Sudip Chattopadhyay and Arnab Sarkar 122
- A Case Study on Failure of Anti-tank Missile Components
S. Madhav and Manish Roy 133
- Predictive Wear Depth Analysis of Al–GnP–hBN Hybrid
Nanocomposites Through Machine Learning Techniques
Arka Ghosh, Hillol Joardar and Ashutosh Das 148
- Advances and Prospects of Biodegradable Magnesium-based
Metal Matrix Composites for Biomedical Implants:
A Comprehensive Review
Kameshwar Mahto, Kuntal Maji and Shashi Bhushan Prasad 165

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A Review of Microstructural Evolution and Numerical Studies in Semi-solid Metal Processing

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**Ruchika Dhyani^{1,2}, Debdip Chakraborty^{1,2}, Himadri Roy^{1,2},
Dipankar Chatterjee^{1,2} and Sudip Samanta^{1,2}**

Abstract

Semi-solid metal processing offers a compelling alternative to conventional casting by enabling the production of alloys with superior, non-dendritic microstructures. Slurry flow dynamics, less porosity formation, higher die and tool life, and final mechanical characteristics are all controlled by primary phase morphology during partial solidification. This review presents a critical assessment of microstructural evolution and mathematical modelling of alloys during semi-solid processing. The observed interaction between solid fraction development, morphological changes, slurry rheology and various semi-solid slurry generation techniques is highlighted. Various techniques for generating non-dendritic structures are provided, covering their working principles, comparative advantages, limitations and effective processing windows. Experimental studies remain constrained in fully capturing microstructure–property relationships and the influence of critical processing parameters. To provide a comprehensive assessment, the latest advancements in volume-averaged solidification process modelling have been examined, with explicit discussion of why interface-tracking methods such as phase-field models and Eulerian–Lagrangian approaches fall outside the present scope. Current two-phase and three-phase solidification models are evaluated with respect to heterogeneous nucleation, solute redistribution, interfacial heat transfer, and inter-phase momentum coupling. Discrepancies between numerical predictions and experimental observations are discussed, particularly under conditions of high shear and non-uniform thermal fields. The review

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demonstrates that computational constraints continue to limit the prediction of nucleation kinetics and microscale features, especially for high-temperature, high-Si and multi-element alloy systems. Although several formulations demonstrate practical potential in laboratory and small-scale industrial castings, further progress requires improved calibration, consistent closure relations, benchmark validation, and expanded material databases capturing micro-macro interactions. Addressing scale-up requirements related to process stability, defect control, tool life and feedstock consistency remains critical for enabling reliable semi-solid processing on an industrial scale.

Keywords

Semi-solid metal processing, microstructural evolution, non-dendritic structure, mathematical modelling, solidification

Introduction

Dendritic microstructures produced by conventional casting are inherently associated with porosity, segregation and mechanical anisotropy. Shear forces are introduced to include the effect of these factors. The resulting enhanced fluid flow disrupts the solid–liquid boundary, fragmenting dendrites and generating a slurry. Instead of the typical dendritic structure submerged in liquid, this slurry consists of fragmented or spheroidised primary phase particles. Non-dendritic primary phase particles are detached and enclosed in a near-eutectic, lower-melting secondary phase once this slurry solidifies. Despite an increased solid fraction, the partially remelted non-dendritic microstructure presents substantially reduced flow resistance. This characteristic offers considerable potential for additional processing in the semi-solid state through forging, pressure die casting and thixo-forming. The aforementioned idea serves as a foundation for the novel manufacturing technique known as semi-solid metal processing (SSMP), in which metal alloys are cast or formed in a semi-solid state (Flemings, 1991; Flemings et al., 1976; Spencer et al., 1972). Semi-solid processing is categorised into rheo-routes and thixo-routes, as explained in Figure 1 by their respective temperature–time trajectories during slurry generation and forming. The rheo-route generates a semi-solid slurry directly from the liquid melt through controlled cooling and shearing, producing near-net-shape products (Spencer et al., 1972). The slurry generation enables continuous, low-turbulence mould filling or forming operations in casting. The thixo-route comprises three steps: preparing a non-dendritic feedstock, reheating it to a semi-solid state and shaping the thixotropic mushy billet (Flemings et al., 1976). Over the last five decades, a wide range of SSMP techniques have been developed, including thixocasting and rheocasting, for manufacturing components critical to the aerospace, automotive and defence sectors. Processing alloys in semi-solid state minimises thermal stresses on moulds, results in reduced cycle times, extends tool life, along with economic and environmental benefits, such as low-cost feedstock, recyclability and reduced waste (Flemings et al., 1976).

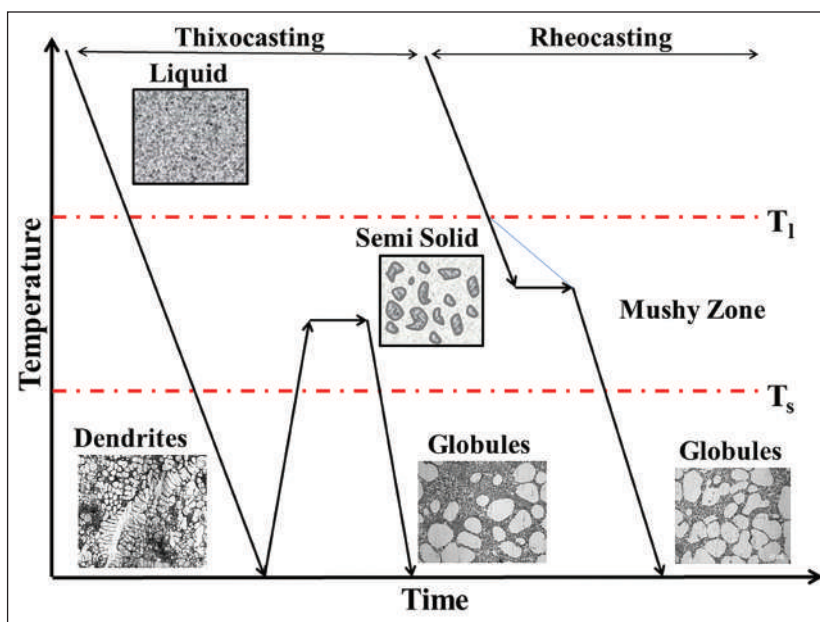


Figure 1. Schematic Representation of Thixo- and Rheo-routes of Slurry Generation.

Source: Nafisi and Ghomashchi (2005).

A crucial step in the semi-solid processing of metals and their respective alloys is the meticulous preparation of slurries that exhibit non-dendritic microstructures, which, in turn, significantly enhance thixotropic flow characteristics during the forming processes (Fan, 2002; Tahamtan et al., 2009; Wang et al., 2011). Conventional casting and solidification inherently produce dendritic morphologies susceptible to porosity and brittleness (Cho & Kang, 2000). Semi-solid processing effectively disrupts dendritic growth through shear-induced fragmentation and particle spheroidisation (Jin, 2022; Qin & Wallach, 2003; Wu et al., 2004).

The formation of non-dendritic microstructures is fundamentally governed by mechanisms including dendrite fragmentation, plastic deformation and spheroidal growth, all of which are significantly influenced by shear intensity, cooling rate and the dynamics of flow within the material (Fadavi Boostani & Tahamtan, 2010; Ji, 2003; Mohammed et al., 2013). Various techniques for the generation of non-dendritic structures include non-shearing methods and shearing methods. Non-shearing techniques produce a semi-solid slurry with a non-dendritic microstructure via thermal cycling or solid-state deformation instead of mechanical stirring. A comprehensive review of the techniques used to generate non-dendritic structures in SSMP is presented in Section 'Methods Used to Generate Non-dendritic Structures in Semi-solid Metal Processing'. Although experimental studies remain essential for understanding material processing and manufacturing technologies, numerical modelling and simulation have increasingly reduced the need for costly and complex experimental trials through advances in computational technology over the past several decades. Simulation-based process

modelling has grown as a tool for predicting micro- and macroscale phenomena of solidification/melting, heat transfer, fluid flow and so on, providing key insights into the study of new process technologies while refining the current ones (Li et al., 2022; Ludwig et al., 2014, 2016; Wu et al., 2019). However, the underlying physical framework significantly impacts a numerical tool's predictive capacity. Thus, it is crucial to comprehend the origin of praxis observation and to develop modelling formulations that accurately represent the relevant flow physics.

In this article, a comprehensive understanding of the processing parameters governing morphological evolution in SSMP is presented, along with relevant mathematical modelling and simulation approaches. Mathematical modelling and simulations provide further insight into microstructural evolution under shear deformation, continuous cooling and isothermal holding conditions. Consequently, microstructural growth kinetics and selected volume-averaged solidification models within this framework are discussed. This review explicitly excludes Eulerian–Lagrangian (E–L) and interface-tracking approaches such as phase-field methods; the rationale for this scope is discussed in the fourth section.

Microstructural Evolution in the Semi-solid State

Most commercially important metal alloys solidify with a dendritic morphology, developing either columnar or equiaxed dendritic structures. In both types, dendrites undergo significant evolution during solidification owing to ripening driven by surface energy minimisation. The structure becomes progressively coarser during solidification due to the remelting of dendrite arms with smaller radii. In castings and ingots undergoing dendritic solidification, multiple processes operate simultaneously in the semi-solid region, such as crystallisation, solute redistribution, ripening, interdendritic fluid flow and solid movement.

The developing dendritic structure is highly sensitive to convection during the initial stages of solidification. When convection is vigorous and cooling is slow, grains evolve into spheroidal forms, and the resulting alloys exhibit semi-solid rheological behaviour that differs substantially from that of fully dendritic alloys (Ji et al., 2022). Semi-solid slurries with non-dendritic microstructures can therefore be produced by solidification under forced convection. In mushy equiaxed solidification, the top surface settles fairly uniformly during the early stages; at this stage, grains remain free to move. Once the solid fraction reaches a critical value, grains interlock and mass feeding stops. Studies show that this network forms at a solid fraction of roughly 0.1–0.2, depending on dendrite size and morphology (Atkinson, 2010; Spencer et al., 1972). Flemings (1991) reported shearing small blocks of aluminium alloys isothermally and observed negligible strength below approximately 0.2 solid fraction. Above this level, shear strength increased with increasing solid fraction and rose slightly with a higher strain rate and larger grain size. In alloys with strong grain refinement, measurable strength did not appear until a solid fraction of approximately 0.4. Spencer et al. (1972) conducted similar tests on Sn–Pb alloy and demonstrated that continuous shearing during solidification dramatically alters rheological behaviour, transforming the alloy into a highly fluid, thixotropic slurry.

Deformation in semi-solid dendrites, up to about 0.9 solid fractions, primarily involves grain-boundary sliding and dendrite distortion. Microstructures show dendrite bending and liquid-filled spaces between grains. Initial stress increases due to more particle contacts, but at high strains, continuous fissures can open, causing stress to drop. Vigorous agitation during solidification initiates the formation of new grains, which then grow dendritically. Continued shear and solidification time transform the dendritic morphology into a rosette shape due to ripening, shear and abrasion (Figure 2). Further cooling perpetuates ripening, leading to spheroidal or ellipsoidal particles, often with entrapped liquid, especially under slow cooling and high-shear conditions. The size of individual grains (dendrites or rosettes) is moderately affected by the shear rate above a minimum value but strongly affected by the cooling rate during the initial stages of solidification (Flemings, 1991).

Mechanisms of Dendrite Fragmentation

Dendritic fragmentation is a widely accepted mechanism for altering the microstructure of semi-solid slurries during shearing. This phenomenon involves the breakdown of primary dendrites into smaller, more spherical particles, which is crucial for achieving a non-dendritic microstructure in SSMP (Ji, 2003). Three principal mechanisms have been proposed for dendrite-arm fragmentation: (a) break-off at the roots caused by shear forces, (b) remelting at the roots due to solute enrichment at high-curvature regions, and (c) bending induced by flow stresses, which generates internal boundaries through the accumulation of geometrically necessary dislocations. Subsequent wetting of high-angle, high-energy grain boundaries by the liquid phase ultimately results in dendrite detachment (Flemings, 1991). Figure 3 presents the schematic sequence for the fragmentation of a typical dendritic morphology and its transformation into a globular grain. A partially solidified dendrite (a) below the liquidus temperature may undergo plastic bending (b) under shear, producing a large misorientation through geometrically necessary dislocations (c). Bending occurs only when the surrounding fluid flows sufficiently faster than the grain. These dislocations store high elastic energy, which is lowered by the formation of a subgrain boundary. When the grain boundary energy exceeds twice the solid-liquid interfacial energy, the boundary is replaced by a thin liquid layer that subdivides the dendrite (Doherty et al., 1984).

The transformation from dendritic to spherical, non-dendritic grains in stirred melts, as described by Molenaar et al. (1986), is a multi-stage process involving initial fragmentation and bending due to shear forces, the formation of rosette structures through liquid entrapment and, finally, ripening of rosettes into spherical grains under high shear and turbulence (Balasubramani et al., 2023). Spheroidal particles, together with equiaxed and dendritic particles, constitute the microstructure of rheocast alloys. Their formation is primarily linked to the fragmentation-by-remelting mechanism, and their number density increases with rheocasting time until it stabilises due to particle sintering and coalescence (Canyook et al., 2010). The microstructural evolution of rheocast billets is strongly influenced by the initial processing parameters. Longer stirring durations and lower cooling rates promote higher particle sphericity at the billet centre, whereas higher

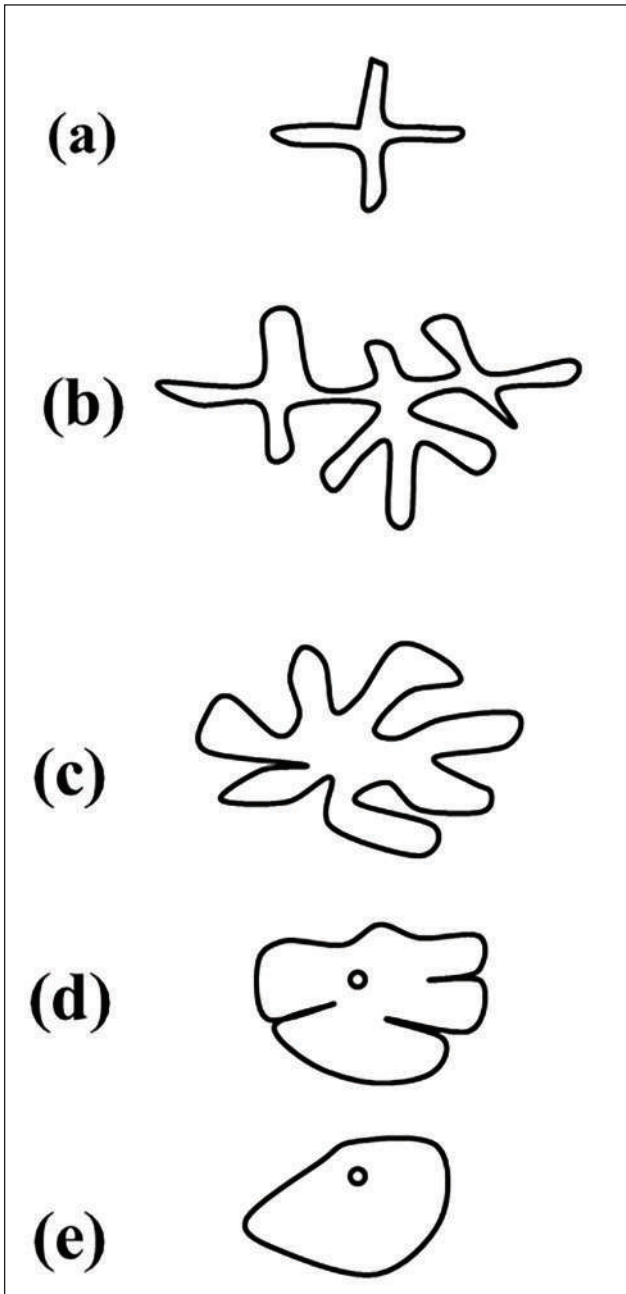


Figure 2. Schematic Illustration of Evolution of Structure During Solidification With Vigorous Agitation: (a) Initial Dendritic Fragment, (b) Dendritic Growth, (c) Rosette, (d) Ripened Rosette and (e) Spheroid.

Source: Shahrooz Nafisi & Ghomashchi, (2019).

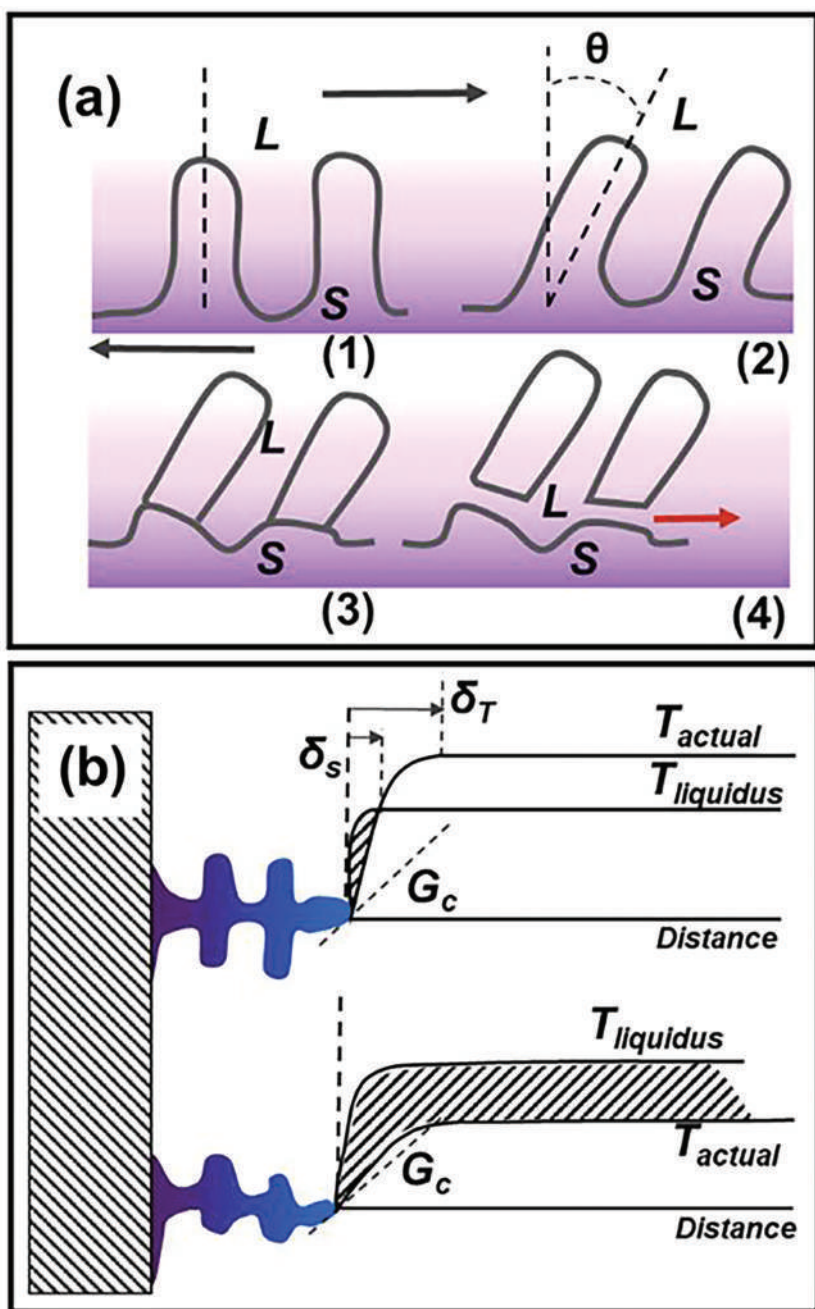


Figure 3. (a) Fragmentation of a Dendrite (Doherty et al., 1984) and (b) the Temperature Field Above the Liquidus and Below the Liquidus Temperature.

Source: Balasubramani et al., (2023).

cooling rates produce finer primary α phases (Zhang et al., 2003). The α phase referred to here is the primary aluminium-rich solid phase that nucleates heterogeneously on inoculant particles or active nucleation sites when the alloy is cooled below the liquidus temperature. Its nucleation is promoted by constitutional undercooling driven by solute rejection at the solid–liquid interface, and its morphology, dendritic, rosette or globular, is determined by the interplay between thermal and solute gradients, shear intensity and cooling rate.

Evolution of Non-dendritic Structures Under Shear

Vigorous agitation during solidification progressively transforms as-nucleated dendritic fragments into increasingly rounded morphologies through a sequence of overlapping mechanisms (Fan, 2002; Spencer et al., 1972). In the initial stage, the imposed shear field induces mechanical bending of primary dendrite arms and promotes fragmentation at the roots (Qin & Wallach, 2003). The fragments, once detached, are transported by convective flow throughout the mushy zone, where they act as additional nucleation substrates, thereby multiplying the grain population. As solidification continues under sustained shear, the fragmented particles evolve into rosette structures through liquid entrapment within concave inter-arm regions (Zhang et al., 2022). Prolonged shear and Ostwald ripening gradually smooth the surfaces of these rosettes, ultimately yielding spheroidal or near-spheroidal grains. The rate of this morphological transition is controlled by the combined influence of shear rate, solidification time and cooling rate (Flemings et al., 1976).

Methods Used to Generate Non-dendritic Structures in Semi-solid Metal Processing

A diverse range of techniques has been developed over the past five decades to generate non-dendritic microstructures in SSMP. These methods can be broadly categorised into mechanical agitation methods, electromagnetic and field-assisted methods, thermal-control-based methods and solid-state deformation methods. Each technique operates through distinct physical mechanisms and offers different advantages, limitations and processing windows. A systematic overview of the principal techniques used for slurry generation and microstructural modification is presented here.

Mechanical Stirring (Rheocasting).

Mechanical stirring is the earliest and simplest technique for generating semi-solid slurries and forms the basis of the original rheocasting process (Spencer et al., 1972). In this method, a rotating impeller or stirrer is immersed in the partially solidified melt to impose shear within the mushy region. Continuous shearing fragments dendrites and promotes their transformation into rosette and near-spherical globular morphologies through ripening and coarsening. The process is applicable to a wide range of Al, Mg, Cu and Sn–Pb alloys. Its main advantages include simple implementation, low capital cost, scalability and compatibility with conventional casting systems (Li et al., 2002). However, the process may

suffer from non-uniform shear distribution, temperature inhomogeneity and gas entrapment under excessive turbulence (Brabazon et al., 2002). Mechanical stirring is generally effective at solid fractions below ~ 0.5 , beyond which the resistance of the solid network hinders stirrer rotation (Kirkwood et al., 2010).

Electromagnetic Stirring.

Electromagnetic stirring (EMS) uses a time-varying magnetic field to generate Lorentz forces within the electrically conducting melt, producing rotational or axial convective flow without mechanical contact. The induced flow shears the mushy zone, fragments dendrites, homogenises temperature and solute fields, and promotes the formation of fine globular microstructures (Bai et al., 2009). The process offers contactless operation, reduced contamination, controllable stirring intensity, and improved grain refinement and macrosegregation control. Despite its advantages over mechanical stirring, EMS remains associated with several challenges, including the formation of partially spheroidised rosette structures and microstructural inhomogeneity across the billet cross-section, both of which may increase reheating times. Production costs are further elevated by the additional stages required for feedstock preparation, reheating before forming and recycling of non-dendritic scrap material (Mohammed et al., 2013). Stirring efficiency also decreases at higher solid fractions (~ 0.3 – 0.4) due to increased slurry viscosity and the development of a solid network. Additionally, non-uniform magnetic field distribution in complex mould geometries may result in heterogeneous microstructures (Bai et al., 2009).

Magnetohydrodynamic Stirring.

Magnetohydrodynamic (MHD) stirring is an advanced form of EMS that employs combined electric and magnetic fields to generate controlled three-dimensional flow within the solidifying melt. Unlike conventional EMS, MHD systems may use superimposed DC and AC fields, pulsed magnetic fields or multi-frequency coil arrangements to produce complex flow patterns (Vives, 1992). The induced Lorentz forces promote dendrite fragmentation, enhance temperature and solute homogenisation, and improve grain refinement. MHD provides a more uniform solid fraction distribution than conventional EMS. The technique offers contactless operation, programmable flow control, and improved thermal and compositional homogenisation. However, it involves significantly greater equipment complexity and a higher capital cost. MHD-cast alloys in the semi-solid state exhibit significant liquid-phase segregation and extensive agglomeration of solid grains compared with strain-induced melt activation (SIMA) and spray-cast alloys. Microstructures remain comparatively less equiaxed and display high rosette growth rates. These characteristics are attributed to differences in grain misorientation distribution introduced during processing (Tzimas & Zavaliangos, 2000).

Cooling Slope Method.

Cooling slope (CS) casting is a simple and low-cost semi-solid slurry preparation technique in which superheated molten metal flows over an inclined, cooled plate before solidifying in a mould (Haga & Kapranos, 2002). Rapid heat extraction at

the slope surface promotes nucleation, while gravity-driven flow generates shear that detaches nuclei and transports them into the bulk melt, producing fine, near-spherical primary particles (Haga & Suzuki, 2001). Key process parameters include channel inclination, slope length, surface temperature and alloy superheat (Birol, 2009). The technique has been successfully applied to various magnesium and aluminium alloys. Major advantages include simple operation, low equipment cost and the ability to generate spheroidal primary phases. However, the process may suffer from porosity, oxide formation and solidified shell formation along the slope surface, which can affect slurry quality and microstructural uniformity (Deepak Kumar et al., 2014). Excessive superheat may remelt nuclei formed on the slope, whereas insufficient superheat can cause premature solidification. The method is therefore most suitable for high-fluidity alloys with relatively wide solidification ranges.

Spray Casting/Spray Deposition.

Spray casting or spray forming is a non-agitation feedstock production process in which liquid metal is atomised into fine droplets using high-velocity gas jets and deposited onto a cooled substrate (Grant et al., 2017). During atomisation, small droplets solidify rapidly, while larger droplets remain liquid, and intermediate droplets are deposited in a semi-solid state. The process has been applied to aluminium, magnesium, copper alloys, nickel superalloys and metal matrix composites. Major advantages include fine non-dendritic microstructures without mechanical stirring, low macrosegregation and the ability to produce near-net-shape preforms. However, the process involves high equipment and operating costs, overspray losses and challenges related to porosity and oxide control (Mohammed et al., 2013).

Swirled Enthalpy Equilibration Device Process.

The Swirled Enthalpy Equilibration Device (SEED) process is a semi-solid feedstock preparation technique based on eccentric swirling of molten metal within a metallic vessel to promote uniform heat extraction and non-dendritic slurry formation (Doutre et al., 2004). Initially, molten metal with low superheat is poured into a rotating vessel, where swirling flow promotes nucleation at the vessel surface while suppressing dendritic growth. The remaining liquid is subsequently drained, leaving a self-supporting semi-solid slug suitable for pressure forming. The enthalpy equilibration approach relies on controlled heat removal, primarily through the cooled crucible wall, and fluid motion through swirling to produce a fine, rosette-to-globular primary phase within a narrow solid fraction window (Gu et al., 2023). The SEED process has been applied commercially to the high-pressure die casting of automotive Al-alloy components. Dendrite fragmentation and controlled cooling during swirling promote the formation of globular microstructures. Stirring intensity and pouring temperature are the key parameters controlling the transition from dendritic to globular structures and the overall microstructural evolution (Doutre et al., 2004). The SEED process enables the on-demand production of semi-solid slurry with precise solid fraction control and

low rotational speeds, thereby minimising gas entrapment. Its crucible-based configuration is also compatible with conventional die-casting systems. However, the process requires careful control of pouring temperature, swirling speed, superheat and cooling conditions to ensure consistent slurry quality. Radial temperature gradients within the crucible may lead to microstructural heterogeneity, while the liquid-draining step increases process complexity (Luo et al., 2019). The method is most effective for near-eutectic aluminium alloys with moderate solidification ranges, whereas alloys with high solidus temperatures or wide freezing ranges are more difficult to process reproducibly.

Strain-induced Melt Activation Process.

The SIMA process is a solid-state deformation route for producing non-dendritic feedstock suitable for thixoforming (Wang et al., 2011). In the SIMA process, a cast alloy billet is first subjected to heavy plastic deformation to introduce a high density of dislocations and stored strain energy. The deformed billet is then reheated into the semi-solid temperature range, where partial melting promotes recrystallisation and liquid penetration along high-energy grain boundaries (Bolouri et al., 2010). This isolates the recrystallised grains and produces a fine, near-spherical primary phase surrounded by liquid, making the microstructure suitable for thixocasting and thixoforging. However, the process requires an additional deformation step before reheating, increasing processing complexity and cost. Careful control of deformation and reheating conditions is necessary to ensure uniform recrystallisation and grain separation. The method is most suitable for wrought aluminium and magnesium alloys and is less effective for alloys with wide solidification ranges or poor workability (Czerwinski, 2020).

Recrystallisation and Partial Melting Process.

The recrystallisation and partial melting (RAP) process is similar to SIMA but employs warm or hot working to introduce stored deformation energy before reheating into the semi-solid range. During partial remelting, recrystallisation and liquid penetration along grain boundaries produce fine, near-spherical grains surrounded by liquid (Jiang et al., 2016). Compared with SIMA, RAP generally provides faster recrystallisation kinetics and more uniform microstructures, particularly in larger billets. The process reduces the risk of deformation-induced cracking and is suitable for alloys with limited cold workability. However, it requires controlled warm working conditions and careful regulation of deformation and temperature to retain sufficient stored energy for recrystallisation. RAP is most effective for wrought aluminium and is less suitable for brittle cast alloys that cannot undergo substantial solid-state deformation. Also, non-uniform strain in large billets leads to a heterogeneous semi-solid microstructure (Wang et al., 2017).

Direct Partial Remelting.

Direct partial remelting (DPRM) is a thixo-route process in which a grain-refined cast billet is reheated directly into the semi-solid temperature range without prior deformation. During isothermal holding, partial melting promotes

spheroidisation through Ostwald ripening and grain boundary migration, producing near-spherical solid grains suitable for thixoforming (Omar et al., 2011). The process typically operates at solid fractions of 0.4–0.6, and the final microstructure strongly depends on the initial grain size and morphology of the feedstock. In this method, the as-received material is reheated directly into the semi-solid range without requiring conventional feedstock preparation routes such as SIMA or RAP (Mohammed et al., 2013). This simplifies processing and broadens the range of materials and processing pathways available for producing thixoformable microstructures. However, precise temperature control and uniform heating are essential to avoid excessive grain coarsening, while the process remains highly dependent on the quality of the initial grain-refined billet. Coarse- or columnar-grained feedstocks are generally unsuitable for this method (Ji et al., 2022).

Ultrasonic Vibration Treatment.

Ultrasonic vibration treatment introduces high-frequency acoustic waves into the solidifying melt through a sonotrode, generating acoustic cavitation and acoustic streaming (Jian et al., 2005). Cavitation causes the formation and collapse of microbubbles, producing intense localised pressure pulses and microjets that fragment dendrite arms, while acoustic streaming enhances convective mixing, homogenises temperature and solute fields, and distributes the fragments throughout the melt. The process provides effective grain refinement, promotes degassing and oxide removal, and can be applied without major modifications to existing equipment. However, sonotrode erosion, limited acoustic penetration depth and sensitivity to probe positioning restrict large-scale application (Jiang et al., 2014). Ultrasonic treatment is most effective during the early stages of solidification at low solid fractions, as the developing solid network rapidly attenuates acoustic propagation and reduces cavitation activity.

Gas-induced Semi-solid Process.

The gas-induced semi-solid (GISS) process produces a semi-solid slurry by injecting inert gas through a porous graphite diffuser immersed in the melt near the liquidus temperature (Wannasin et al., 2010). Rising gas bubbles generate localised shear and convective flow that fragment dendrites and distribute the fragments throughout the melt, while simultaneous heat extraction from the injected gas promotes undercooling and nucleation. The GISS route is suitable for processing different types of alloys, such as zinc alloys, cast aluminium alloys, wrought aluminium alloys and die casting aluminium alloys (Mohammed et al., 2013). The process integrates stirring and cooling in a compact, low-cost setup compatible with conventional casting systems, while the inert atmosphere also helps reduce oxidation. However, gas entrapment, non-uniform shear distribution and graphite tip erosion remain important challenges. The method is most effective at low-to-moderate solid fractions, as increasing solid content reduces bubble mobility and convective efficiency. Techniques such as ultrasonic vibration, SIMA and inert gas purging are not industrially viable at high solid fractions, further highlighting the processing niche of GISS (Gautam et al., 2025).

Effect of Processing Parameters on Microstructure

The viscosity and structural evolution of semi-solid metal slurries are significantly influenced by shear rate and cooling rate, leading to pseudoplastic and thixotropic behaviours. The viscosity of semi-solid metal slurries is highly dependent on both shear rate and cooling rate, with an increasing shear rate and a decreasing cooling rate leading to lower viscosity (Fan, 2002). This is partly due to the formation of denser, more rounded particles that move more easily. Such slurries exhibit pseudoplasticity (viscosity decreases with increasing shear rate) and thixotropy (time-dependent viscosity changes as the material structure builds up or breaks down). This structural evolution, driven by shear and cooling, ultimately governs the rheological properties of the material. Tzimas and Zavaliangos (2000) reported the effects of cooling rate, shear rate and isothermal coarsening on the microstructure of alloys in the semi-solid state. Shearing significantly reduces flow resistance and viscosity by breaking bonds within the solid network. The cooling rate controls solidification kinetics and grain refinement, with higher rates producing finer, more uniform α -phase grains and lower segregation. Isothermal holding determines grain growth and spheroidisation through ripening, where grains become more equiaxed as soaking time and liquid fraction increase. The grain growth rate constant depends on the liquid volume fraction and coalescence kinetics. Ji and Fan (2002) investigated the effect of shearing during solidification under continuous cooling and isothermal conditions using a twin-screw process in Sn–15 wt% Pb alloy. A low shear rate of 500 s^{-1} produced mainly rosette grains, while higher shear rates generated spherical, non-dendritic grains with narrow size distributions. At high shear, the transition from dendritic to rosette or spherical forms occurs through liquid entrapment within the rosette. Increasing the shear rate and turbulence shifts the morphology from equiaxed dendrites to rosettes and then to spheroids. Vigorous convection causes necking and remelting of dendrite arms, accelerating this transformation. Koke and Modigell (2003) discussed the significant influence of both cooling rate and shear rate on the microstructure and rheological properties of semi-solid metal alloys. The average particle size of the globular grains was observed to increase with a decreasing cooling rate and an increasing shear rate. Experiments conducted at three shear rates (100, 200 and 400 s^{-1}) with a constant cooling rate ($-4 \text{ }^{\circ}\text{C/min}$) confirmed that particle diameters increased with higher shear rates. The rheological behaviour was found to be shear-thinning at steady state, with a transient overshoot (shear-thickening) observed immediately after shear-rate increases. Rapid changes in shear rate induce structural breakdown or recovery, leading to transient shear stress and viscosity responses fundamentally linked to the time-dependent agglomeration and deagglomeration of solid particles within the liquid matrix. Liu et al. (2003) further explain the rheological behaviour of a semi-solid Sn–15% Pb alloy under rapid shear-rate changes relevant to thixoforming processes. Shear-rate increases trigger swift structural breakdown and a subsequent gradual viscosity decrease. Recovery upon shear-rate reduction takes longer than breakdown. Both initial and final shear rates influence the transient behaviour, which is closely tied to

microstructural changes, including particle agglomeration and particle size evolution. High turbulence and increased shear rates primarily lead to the beneficial transformation of particle morphology from dendritic to spherical, enhance solute penetration and accelerate solidification, although they do not necessarily improve interface stability (Fan, 2002; Qin & Wallach, 2003). Understanding and controlling these parameters are essential for successful industrial applications and accurate computational modelling (Ji & Fan, 2002). Effective semi-solid processing, especially thixoforming, relies heavily on understanding and precisely controlling process parameters such as shear rate, temperature and die design. The transient rheological behaviour of the material under these conditions is a key factor for both experimental investigation and computational modelling to ensure high-quality products and efficient manufacturing.

Volume-averaged Transport Phenomena in Solidification Processes: Scope and Rationale

Scope of the Review and Exclusion of Phase-field and Eulerian–Lagrangian Methods

Before presenting the volume-averaged framework, it is necessary to clarify why two prominent classes of solidification models, phase-field methods and E–L approaches, fall outside the scope of this review. This is not a reflection of their scientific merit, but rather a recognition that their computational demands and characteristic length scales differ fundamentally from those of the volume-averaged approach discussed here. The E–L method follows individual solid particles (Lagrangian) in a continuous liquid phase (Eulerian). The E–L technique is effective for diluted particle-laden flows, but it proves computationally challenging for SSMP since solid volume percentages typically fall between 0.3 and 0.5, necessitating the tracking of massive amounts of interacting particles. Additionally, the natural consideration of phase transition and solidification physics required for predicting morphological evolution is absent from the method. Phase-field models are very useful for comprehending dendritic morphology and microsegregation because they resolve the solid–liquid interface at the mesoscale. However, these are inappropriate for the macroscale casting models pertinent to SSMP process design, though, because their computing cost limits domain sizes to the microscale. Hence, volume-averaged, continuum-scale formulations that are computationally tractable for engineering-scale predictions are the main topic of this review. Phase-field models resolve the solid–liquid interface explicitly, tracking its position through a diffuse interface parameter that varies smoothly between bulk phases. Coupled with convective flow, as demonstrated by Beckermann et al. (1999), phase-field models can capture dendritic growth morphology, anisotropy and solute trapping with great fidelity. The scale ($\delta \sim 10^{-1}$ – 10^{-3} mm) is orders of magnitude smaller than the macroscopic casting dimension ($\sim 10^2$ – 10^3 mm). Resolving the interface over a

full engineering casting geometry would require computational meshes of prohibitive size. Furthermore, phase-field simulations of solidification under convection are typically restricted to two-dimensional domains or small three-dimensional volumes, limiting their direct applicability to industrial process optimisation. For these reasons, while phase-field models provide invaluable insight into mesoscale solidification physics, they are excluded from the present macroscale-focused review.

E–L methods treat the liquid melt as a continuous Eulerian field and track each solid particle, crystal, or grain individually as a Lagrangian entity. This approach is powerful for capturing particle-level dynamics such as sedimentation, clustering and individual grain growth trajectories. Cellular Automata approaches effectively capture mesoscale phenomena such as grain growth kinetics, crystallographic orientation, and the columnar-to-equiaxed transition (CET) across various solidification and manufacturing processes (Dreelan et al., 2022; Staroselsky et al., 2024; Zhi et al., 2024). However, the computational cost scales with the number of tracked particles, and in industrial castings where grain densities may reach 10^{10} – $10^{14}/\text{m}^{-3}$, full Lagrangian tracking is computationally prohibitive. Moreover, the feedback between individual grain trajectories and the evolving macroscopic flow field (two-way coupling) introduces substantial numerical complexity. Volume-averaged (Eulerian–Eulerian) frameworks overcome this limitation by representing the solid phase as a continuum, making them tractable for engineering-scale simulations while retaining statistical descriptions of grain number density, size and morphology. The present review therefore focuses exclusively on volume-averaged formulations.

General Transport Framework

The volume-averaged approach describes each phase α (liquid, solid, columnar, equiaxed) through conservation equations for mass, momentum, energy and species. For a generic transported quantity Φ_α , the volume-averaged conservation equation takes the form:

$$\partial(\varepsilon_\alpha \rho_\alpha \phi_\alpha) / \partial t + \nabla \cdot (\varepsilon_\alpha \rho_\alpha \phi_\alpha \mathbf{u}_\alpha) = \nabla \cdot (\varepsilon_\alpha \Gamma_\alpha \rho_\alpha \phi_\alpha) + S_\alpha \quad (1)$$

Here, ε_α is the volume fraction of phase α , ρ_α its density, Γ_α the effective diffusivity and S_α a source term that accounts for inter-phase exchange. The pressure gradient force is incorporated as a component of the momentum source term. Physical quantities are most naturally expressed as mass-weighted (Favre-averaged) values, related to volume-weighted quantities through the phase density. For mass conservation ($\phi_\alpha = 1$), the source term S_α represents the net inter-phase mass transfer due to solidification or remelting. For momentum ($\phi_\alpha = \mathbf{u}_\alpha$), S_α encompasses the drag force, buoyancy and solidification-induced momentum exchange. For energy ($\phi_\alpha = h_\alpha$, specific enthalpy), S_α includes the latent heat release and inter-phase heat transfer. For the solute species ($\phi_\alpha = C_\alpha$, solute concentration), S_α accounts for solute redistribution at the solid–liquid

interface. The distinguishing feature of different volume-averaged solidification models lies in how they formulate the closure laws, that is, the expressions for inter-phase exchange terms (mass transfer rate $\dot{M}$, momentum drag M , heat flux Q and solute exchange J). These terms are not computed explicitly at the microscale but are modelled through constitutive relations that encode the assumed grain morphology, growth kinetics and interfacial conditions. The following sections describe how these closure laws differ between the two-phase globular equiaxed model and the three-phase columnar–equiaxed model.

Modelling Examples

Modelling Equiaxed Solidification via a Two-phase Approach (Non-dendritic)

Equiaxed microstructures are prevalent in the solidification of industrial alloys, particularly those refined by inoculants to produce a fine, near-spherical crystal morphology. In the mathematical model, grain morphology is approximated as spherical, and diffusion around the globular structure dictates growth dynamics. Ni and Beckermann (1991, 1993) originally developed the two-phase solidification model for globular equiaxed solidification. Ludwig and Wu (2002) and Wu et al. (2003a) subsequently extended this model to update several closure laws. Further refinements were made by Wu et al. (2013, 2014), Wu and Ludwig (2006, 2007), Bedel et al. (2015) and Heyvaert et al. (2017) by accounting for inoculant transport.

Closure Laws and Inter-phase Exchange Terms

The key inter-phase exchange terms in the two-phase globular equiaxed model are the solidification rate $\dot{M}$ ($\text{kg m}^{-3} \text{s}^{-1}$), the inter-phase momentum drag $M_{\alpha\beta}$ (N m^{-3}), the inter-phase heat transfer $Q_{\alpha\beta}$ (W m^{-3}) and the solute exchange $C_{\alpha\beta}$ ($\text{kg m}^{-3} \text{s}^{-1}$ of solute). These terms couple the solid and liquid phase conservation equations. The solidification rate $\dot{M}$ is the crucial quantity from which all other solidification-induced exchange terms are derived. It is evaluated from the solid–liquid interfacial energy balance:

$$\dot{M} = \rho_s \cdot (df_s / dt) = \left[\rho_s f_s (h'_s - h'_l) + Q_h \right] / \left[L + h_{sl} (T_s - T_l) \right] \quad (2)$$

where f_s is the solid volume fraction, ρ_s the solid density, L the latent heat, h_{sl} the inter-phase heat transfer coefficient per unit interfacial area and Q_h the external thermal source. Once $\dot{M}$ is known, the inter-phase solute exchange C is obtained from the lever rule or Scheil-type conditions applied at the solid–liquid interface, accounting for the impingement factor Ω_{imp} that reduces the effective interfacial area at high solid fractions:

$$C = \dot{M} \cdot C^* \cdot (1 - \Omega_{imp}) \quad (3)$$

where C^* is the solute concentration at the solid–liquid interface. The impingement factor Ω_{imp} accounts for the overlap of diffusion fields between neighbouring grains and can be treated as a function of solid fraction f_s or approximated as $\Omega_{imp} \approx f_s$ (Wu et al., 2013, 2014).

The equiaxed grain radius R_s is related to the grain number density n by the geometric relation: $R_s = \sqrt[3]{3f_s / 4\pi n_s}$. The system of four transport equations (mass, momentum, energy and species for each phase) contains four primary unknowns: the phase volume fractions f_s , the phase velocities $u\alpha$, the temperature T (assuming local thermal equilibrium between phases: $T_s = T_l = T$) and the solute concentrations. The assumption of local thermal equilibrium ($T_l \approx T_s$) is standard in most two-phase models for alloys with moderate cooling rates, as the inter-phase heat transfer is rapid relative to the solidification timescale. Under this assumption, the energy equations for both phases can be combined into a single mixture enthalpy equation, reducing the number of unknowns. Once T is known from the mixture energy equation, the remaining inter-phase exchange terms $\dot{M}$, $\dot{Q}$ and C are computed using the known solidification rate $\dot{M}$ and the grain evolution equation. If the effect of turbulent flow must be accounted for, the diffusion length scale used in computing $\dot{Q}$ and C must be adjusted using turbulent diffusivity correlations (Ni & Beckermann, 1993).

Nucleation Model.

For the majority of engineering alloys, heterogeneous nucleation on inoculant particles governs the equiaxed grain population. An initial number density n_0 of inoculant particles (embryos) is present in the parent melt; undercooling progressively activates these as equiaxed nuclei. The nucleation rate, which has a Gaussian distribution function of undercooling, can be estimated by the source term N_{nu} . The nucleation parameters, $n_{max}\Delta T_N$ and ΔT_σ , can be ascertained through experimentation (Ahmadein et al., 2009; Wang & Beckermann, 1996). Since the solid phase is regarded as a pseudofluid, the associated momentum conservation equation must be solved by defining the solid phase's viscosity, μ_s , which is brought about by interactions between individual equiaxed grains. Ishii and Zuber (1979) demonstrated that the viscosity of a solid/liquid combination is equal to $\mu_{mix} = \mu_l \cdot (1 - f_s / f_s^c)^{-2.5 \cdot f_s^c}$. The viscosity of the solid phase is obtained as follows, considering that the mixing rule (Beckermann et al., 1999) $\mu_{mix} = f_l \mu_l + f_s \mu_s$ is applicable here. A packing limit with a significantly smaller solid volume percentage (e.g., 0.2) can be implemented to simulate the coalescence of dendritic crystals (Combeau et al., 2009; Li et al., 2012). Other calculation techniques need to be taken into account for the turbulent flow (Acharya & Moukalled, 1989; Henkes & Hoogendoorn, 1994; Lipton et al., 1984; Ni & Beckermann, 1993).

Implementations of the Globular Equiaxed Solidification Model

Ni and Beckermann (1993) performed the first simulation using the globular equiaxed solidification model to study crystal sedimentation. Their exploratory two-phase flow simulation demonstrated the coupled interaction between local crystal growth dynamics and grain arrangement, enabling quantitative correlations between sedimentation-induced solidification outcomes (grain size distribution and macrosegregation) and processing parameters (temperature, constitutional undercooling) for the first time. A two-dimensional domain of Al–4.0 wt% Cu alloy was simulated under side and bottom cooling (Ludwig & Wu, 2002; Wu et al., 2003a). Results at 80 s reveal that nucleation and solidification commence along the die wall. A portion of the grains is carried to the central region by melt flow, while others sink and settle at the bottom corners. The coupling of inter-phase momentum exchange drives the formation of two counter-rotating vortices: one clockwise in the right half and one anticlockwise in the left half of the domain. The flow is primarily driven by crystal sedimentation and sedimentation-induced melt convection, though thermo-solutal convection also contributes. Later work extended the globular equiaxed solidification model to study: globular equiaxed crystal formation during channel cooling of A356 aluminium alloy (rheocasting/cooling slope) (Ahmadein et al., 2009; Wang et al., 2005); macrosegregation and breakdown during monotectic solidification (Wu et al., 2003b, 2005); the onset of solidification during mould filling in form casting (Wu et al., 2018); and macrosegregation caused by crystal sedimentation in steel ingots (J. Li et al., 2014; Li et al., 2012; Tu et al., 2014). Validation against laboratory casting experiments for mixed columnar equiaxed solidification has been reported by Wu et al. (2003b) and Wu and Ludwig (2006).

Modelling the Columnar-to-equiaxed Transition

A mixed columnar–equiaxed microstructure is the most common as-cast structure in commercial castings. During solidification, columnar crystals grow preferentially along the thermal gradient from the mould wall, while equiaxed grains nucleate in the undercooled bulk melt and may be advected by convection. Because columnar and equiaxed crystals differ fundamentally in their mobility, that is, columnar crystals are immobile or grow at a prescribed velocity, while equiaxed crystals are free to move, they must be treated as distinct phases in the model, even though both represent the same solid phase from a thermodynamic standpoint. The three-phase model for CET was originally developed by Wu and Ludwig (2006, 2007). It extends the two-phase equiaxed model of Section ‘Modelling Equiaxed Solidification via a Two-phase Approach (Non-dendritic)’ by adding a columnar phase, bringing the total to three phases: liquid, columnar solid and equiaxed solid. The columnar tip advancement is described by the Lipton–Glicksman–Kurz (LGK) model (Lipton et al., 1984), which predicts the columnar tip velocity and tip radius as functions of local undercooling. The equiaxed solidification sub-model is identical to that described in Section ‘Modelling Equiaxed Solidification via a Two-phase Approach (Non-dendritic)’. The columnar-to-equiaxed transition (CET) is predicted using Hunt’s blocking

criterion (Hunt, 1984). The transition from columnar to equiaxed growth occurs when the volume fraction of equiaxed grains ahead of the columnar tip front exceeds a critical threshold (typically 0.49), at which point the equiaxed grains mechanically block further columnar advances.

Boussinesq Approximation and Density Treatment.

Grain sedimentation and thermo-solutal convection are modelled using the Boussinesq approximation (Li et al., 2014; Wu & Ludwig, 2006, 2007) in which the density ρ is treated as constant in all terms of the Navier–Stokes equations except for the gravitational body force term. In the buoyancy term, density variations are linearised as:

$$\rho = \rho_0 [1 - \beta_T (T - T_0) - \beta_C (C - C_0)] \quad (4)$$

where β_T and β_C are the thermal and solutal expansion coefficients, respectively, ρ_0 is the reference density, T_0 and C_0 are the reference temperature and concentration, respectively. This approximation is valid when density variations are small, a condition generally satisfied in Al-alloy systems over typical solidification temperature ranges. The Oberbeck–Boussinesq approximation decouples the flow equations from the energy and species equations except through the buoyancy source term, greatly simplifying the numerical solution. Because the liquid and solid have different densities, the shrinkage-driven feeding flow is handled separately: in open-domain continuous casting, hot melt enters at the inlet; in closed-domain ingot or form castings, a shrinkage cavity must be fed by an additional gas or slag phase (Wu et al., 2017).

Implementations of the Columnar-to-Equiaxed Transition Model.

The three-phase CET model of Wu and Ludwig (2006, 2007) was first applied in an exploratory simulation of a scaled-down model ingot to demonstrate mixed columnar–equiaxed solidification. Heat transfer controls the overall solidification sequence, but melt convection and grain sedimentation exert a significant influence on the final as-cast structure. When the surface temperature falls below the liquidus temperature, solidification commences with the formation of the columnar phase at the mould wall. Equiaxed grains nucleate in the undercooled melt, and melt convection transports them towards the mould wall, where they sink. Sinking crystals drag the melt downwards along the walls, generating a rising melt flow in the casting centre. Thermal-solutal buoyancy further modifies the overall convection pattern. As solidification proceeds, the columnar phase volume fraction increases at the mould wall, while equiaxed grains continue to nucleate, grow and sink along the columnar tip front until the CET occurs. The model was subsequently extended for industrial ingots (Wu et al., 2017), incorporating the formation of shrinkage cavities and dendritic equiaxed crystal morphology as significant additions. It was noted that the prior assumption of an ideal globular equiaxed grain morphology tends to overestimate macrosegregation (Wu & Ludwig, 2006, 2007), highlighting the importance of morphological fidelity in closure laws.

Comparison of Solution Techniques

Volume-averaged solidification models can be solved using several numerical approaches, each carrying distinct advantages and limitations depending on the application:

Table 1. Comparison of Numerical Methods for Modelling Fluid Flow, Heat Transfer, and Microstructure Evolution in Casting and Solidification.

Method	Description	Advantages	Limitations	Best For
Finite volume method (FVM)	Integral conservation on control volumes	Conserves flux; robust for complex geometry	Numerical diffusion on unstructured grids	Melt flow, macro-heat transfer
Finite element method (FEM)	Variational formulation on elements	Handles irregular domains; good for solid mechanics coupling	Computationally intensive for large 3D domains	Thermomechanical coupling in solidification
Lattice Boltzmann method (LBM)	Mesoscale; velocity distribution functions	Natural for complex interface topology; parallelisable	Not yet scalable for full industrial casting domains	Dendritic growth at mesoscale
SIMPLE/SIMPLER algorithms	Pressure–velocity coupling for incompressible flow	Widely validated; efficient for steady-state problems	Convergence issues in high-solid-fraction mushy zones	Inter-dendritic and mould-filling flow
Phase-field method	Diffuse-interface formulation with order parameter ϕ	Resolves solid–liquid interface without explicit tracking	Restricted to microscale domains (mm^3); high computational cost	Dendrite morphology, microsegregation

Future Scope

Research is largely focused on conventional Al, Mg and Sn–Pb alloys, while semi-solid processing of high-temperature alloys, high-Si-content alloys and lightweight multi-element alloys remains less explored, with emphasis on understanding their fragmentation and spheroidisation kinetics. Further work is also needed to improve energy efficiency, cycle time, tool life, defect suppression, process stability, automation, feedstock consistency and integration with existing manufacturing systems to enable reliable large-scale industrial production. Integrating physics-based models with machine learning or statistical inference can improve the prediction of particle morphology, slurry behaviour and defect formation while reducing computation

time, provided that proper validation is ensured. In addition, many closure laws used in volume-averaged models still require refinement through targeted experimental studies on fluid–solid interactions during solidification, especially at intermediate solid fractions. Model validation should increasingly rely on controlled laboratory benchmark experiments rather than only engineering castings, as this allows individual closure laws and physical assumptions to be isolated and tested more effectively. Finally, advancing integrated micro–macro property modelling will require more extensive material input data beyond traditional approaches.

Conclusion

Shear rate, cooling rate and isothermal holding are the dominant parameters governing the transition from dendritic to non-dendritic microstructures in SSMP. Their combined influence controls dendrite fragmentation, spheroidisation, grain growth, slurry viscosity and the overall thixotropic behaviour essential for effective rheocasting and thixocasting. The comparative analysis of stirring methods reveals that each technique has a distinct and limited processing window defined by its effective solid fraction range, shear intensity and scalability. These constraints must be matched to alloy solidification kinetics for optimal microstructure control.

Accurate modelling and simulation of SSMP are necessary because they provide a practical approach to predicting microstructural evolution, such as fragmentation, spheroidisation and rheological transitions, without relying on repetitive experiments. Volume-averaged Eulerian–Eulerian multi-fluid frameworks, reviewed here, are the appropriate engineering-scale tools for this purpose. Several significant advancements have been made over the past two decades: (a) solidification models accounting for mixed columnar–equiaxed structure growth; (b) refinement of equiaxed crystal morphology descriptions; and (c) application of models to shrinkage cavity and macro segregation phenomena.

Further research directions include designing specific laboratory benchmark experiments for model validation, providing more reliable processing parameters and material data, improving closure laws through targeted experimental studies, incorporating turbulence models into multiphase flow frameworks and developing hybrid physics–machine-learning (ML) approaches to accelerate computation. As computing power increases, the industrial applicability of these models will become increasingly feasible.

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Micro-fabricated Electrochemical Sensors for Quantitative Analysis of Tea Polyphenols

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Abstract

Tea polyphenols are key bioactive constituents responsible for the health-promoting properties and quality attributes of tea. Rapid, reliable and cost-effective methods for their quantification are therefore essential for quality control and food analysis. In this work, a micro-fabricated disposable screen-printed electrochemical sensor was developed for the quantitative determination of tea polyphenols, using catechin as a representative standard compound. The electrochemical behaviour of catechin was investigated by cyclic voltammetry, revealing a well-defined anodic oxidation peak at +0.450 V, which was selected as the working potential for amperometry detection. The sensor exhibited a linear response towards catechin in the concentration range of 0–400 mg L⁻¹ with excellent sensitivity. The limit of detection and limit of quantification were found to be 1.151 mg L⁻¹ and 3.838 mg L⁻¹, respectively. The analytical performance of the sensor was validated by comparison with high-performance liquid chromatography, showing strong agreement with a correlation coefficient of 0.994. Repeatability studies performed on 10 different tea samples demonstrated good precision, with coefficients of variation below 3%. The proposed micro-fabricated electrochemical sensor offers a simple, rapid and reliable alternative to conventional chromatographic methods for the determination of tea polyphenols and shows strong potential for on-site tea quality assessment and food analytical applications.

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Keywords

Polyphenols, screen-printed electrodes, cyclic voltammetry, amperometry

Introduction

Tea is one of the most widely consumed beverages worldwide and is highly valued for its characteristic flavour, aroma and significant health-promoting properties. These beneficial effects are primarily attributed to tea polyphenols, a complex group of phenolic compounds including catechins, flavonols and phenolic acids, which are known for their strong antioxidant, anti-inflammatory and chemoprotective activities (Balentine et al., 1997; Cabrera et al., 2006; Dufresne & Farnworth, 2001; Graham, 1992). The concentration and composition of tea polyphenols are therefore considered critical quality parameters for tea and tea-based products, necessitating reliable and efficient analytical methods for their quantification.

Conventional techniques for tea polyphenol analysis, such as spectrophotometric assays based on the Folin–Ciocalteu reagent, high-performance liquid chromatography (HPLC) and other chromatographic methods, are widely used due to their accuracy and selectivity (Prior et al., 2005; Singleton et al., 1999). However, these methods often involve time-consuming sample preparation, expensive instrumentation and limited portability, which restrict their application in rapid or on-site analysis. As a result, electrochemical methods have gained considerable attention as alternative analytical tools because of their high sensitivity, simplicity, rapid response and cost-effectiveness (Bard & Faulkner, 2001; Chevon & Or, 2005; Oliveira & Pimentel, 2009).

Electrochemical sensing of phenolic antioxidants relies on their inherent redox activity, enabling direct quantification using voltammetric techniques. Several studies have demonstrated the suitability of differential pulse voltammetry, linear sweep voltammetry and impedance-based approaches for evaluating the electrochemical behaviour and antioxidant capacity of polyphenolic compounds (Ping et al., 2015; Sánchez-Moreno, 2002). In particular, electrochemical sensors and biosensors have emerged as powerful tools in food and beverage analysis due to their adaptability, miniaturisation potential and compatibility with disposable electrode platforms (Mello & Kubota, 2002; Wang, 2006).

Significant efforts have been devoted to improving sensor performance through electrode surface modification using biological and chemical modifiers. Enzyme-modified electrodes employing tyrosinase extracted from crude sources have been successfully applied for the estimation of tea polyphenols via differential pulse voltammetry, exploiting the enzymatic oxidation of phenolic substrates (Chattopadhyay et al., 2015). Similarly, potato-extracted enzyme-modified electrodes combined with linear sweep voltammetry and electrochemical impedance spectroscopy have provided sensitive and mechanistic insights into tea polyphenol oxidation processes (Chattopadhyay & Sarkar, 2016). Alternative chemical modification strategies, such as the use of chloramines-T and ferric chloride, have also been shown to enhance sensitivity and reproducibility in tea polyphenol

sensing (Chattopadhyay & Sarkar, 2014a; Sen et al., 2016). These studies clearly established the feasibility of electrochemical approaches for reliable quantification of tea polyphenols using modified electrode systems (Chattopadhyay & Sarkar, 2014b).

Among the various electrochemical platforms, screen-printed electrodes (SPEs) have attracted growing interest owing to their low cost, disposability, ease of mass production and suitability for miniaturised and portable sensing devices (Economou, 2010; Honeychurch, 2014; Metters et al., 2011; Renedo et al., 2007). SPE-based sensors have been extensively applied in environmental monitoring, clinical diagnostics and food analysis, including the detection of phenolic compounds and antioxidants (Apetrei & Apetrei, 2013; Pingarrón et al., 2008). Early investigations demonstrated the successful estimation of tea polyphenols using screen-printed electrochemical sensors, highlighting their potential for rapid and practical analysis (Chattopadhyay et al., 2014). The integration of micro-fabrication techniques with SPE technology further enables precise control over electrode geometry, improved reproducibility and enhanced electroanalytical performance.

Recent advances in electrochemical sensor design emphasise the development of compact, label-free and highly sensitive systems capable of on-site analysis with minimal sample preparation (Cheng & Yu, 2024; Li et al., 2022, 2023; Shao et al., 2022; Vestergaard et al., 2007). In this context, micro-fabricated screen-printed electrochemical sensors represent a promising approach for tea polyphenol quantification, combining the advantages of electrochemical detection with scalable manufacturing processes.

Building upon previous work on enzyme- and chemically modified electrodes as well as screen-printed sensor platforms, the present manuscript reports the development and application of micro-fabricated screen-printed electrochemical sensors for the quantification of tea polyphenols. The proposed approach aims to achieve enhanced sensitivity, reproducibility and operational simplicity, offering a robust analytical tool for tea quality assessment and related food analytical applications.

Experimental

Chemicals

Catechin ($\geq 98\%$, TLC grade) was procured from Sigma (UK). A 0.1 M sodium phosphate buffer solution (pH 6.8) was prepared using disodium hydrogen phosphate, sodium dihydrogen phosphate and potassium chloride, all obtained from Merck (India). Milli-Q water (Millipore India Ltd., India) was used throughout for the preparation of buffer solutions. All other reagents employed in the study were of analytical grade and were purchased from Merck (India). Commercially available green and black tea samples were collected from local markets. For HPLC analysis, acetonitrile, acetic acid and water of HPLC grade were obtained from Merck (India).

Instrumentation

Electrochemical measurements, including amperometric responses, were carried out using an Autolab electrochemical analyser (PGSTAT 12, Ecochemie B.V., The Netherlands). The electrodes were connected to the corresponding terminals of the Autolab system through standard connectors. Experimental parameters such as scan rate, applied potential and measurement duration were controlled and recorded using General Purpose Electrochemical Software operated via a computer interfaced with the analyser.

Sensor Fabrication

The sensing system was designed and fabricated for the electrochemical detection of tea polyphenols using a disposable SPE platform. The SPE consisted of a carbon working electrode, a carbon counter electrode and an integrated silver/silver chloride reference electrode. These electrodes were screen-printed onto a 250 μm thick polyester substrate (Cadillac Plastic Ltd., Swindon, UK) using a DEK 248 screen-printing machine (DEK Printing Machines Ltd., Weymouth, UK).

The circular electrocatalytic working electrode was fabricated using a commercially available carbon powder (MCA Services Ltd., Cambridgeshire, UK), which was converted into a screen-printable paste by mixing with 2.5% (w/v) hydroxyethyl cellulose (HEC) in phosphate-buffered saline (PBS) at a weight ratio of 1:3. The reference electrode ink comprised silver paste containing 15% (w/w) silver chloride (MCA, UK) (Singleton et al., 1999). The counter electrode and conductive tracks connecting the electrodes to the measurement system were printed using I45R carbon ink (MCA, UK).

To electrically insulate the basal tracks from the measurement solution, an epoxy-based protective coating ink (242-SB, Agmet ESL Ltd., Reading, UK) was applied. Following fabrication, the electrodes were thermally cured at 125 $^{\circ}\text{C}$ for 2 h to ensure proper curing of the epoxy resin and to enhance the mechanical stability and electrochemical durability of the electrocatalytic surface, enabling prolonged use in aqueous media. The detailed configuration of the sensing system is illustrated in Figure 1.

Preparation of Tea Extract

A variety of commercially available green and black tea samples were procured from the local market. The green tea samples included Tetley, Darjeeling Longview, Twinings, Japanese green tea, Golden Tips and Lipton, while the black tea samples comprised CTC, Tulip Bari, BOPL and Twinings. These samples were assigned identification numbers from 1 to 10, respectively, and stored in airtight containers until further use.

For extract preparation, 0.1 g of each tea sample was accurately weighed and transferred to a beaker. Subsequently, 5 mL of freshly boiled distilled water was added, and the mixture was allowed to infuse for 5 min. The infusion was then filtered to remove insoluble residues, and the resulting supernatant was used directly as the sample for electrochemical measurements.

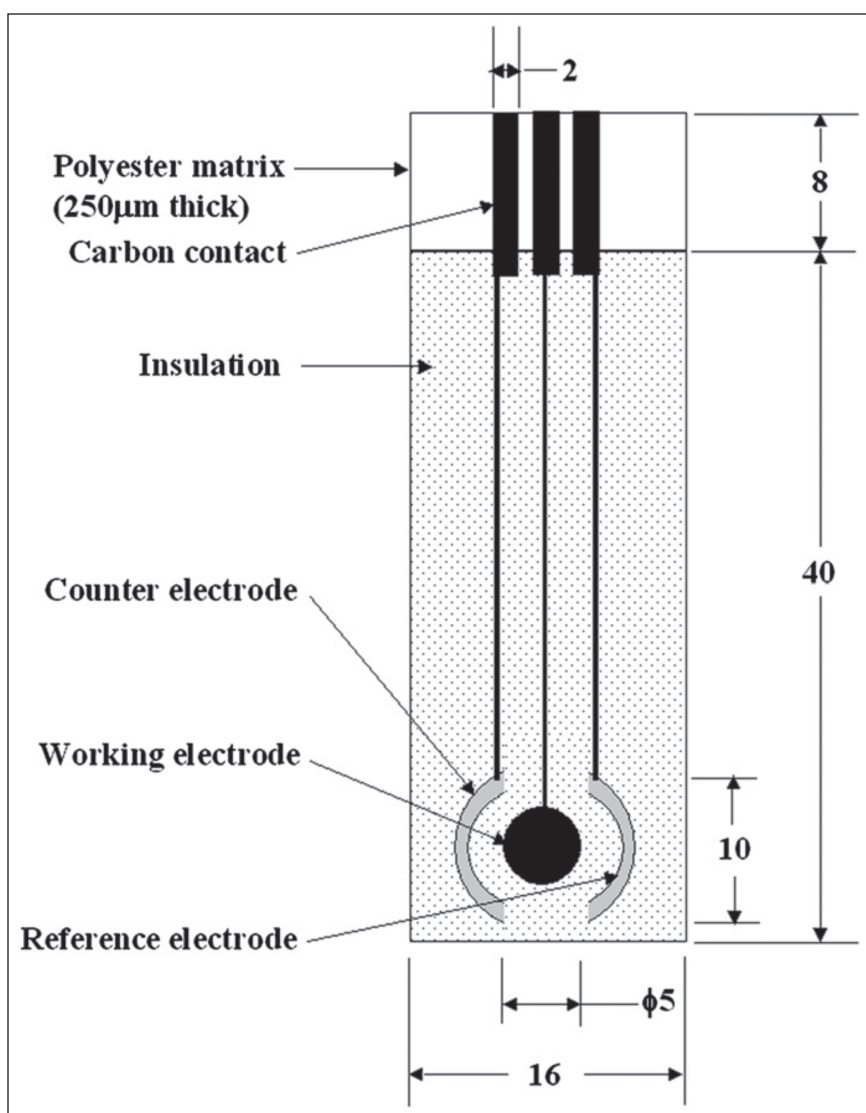


Figure 1. Schematic Illustration of the Transducer Layout.

Note: All dimensions are given in mm.

Electrochemical Measurement

Cyclic voltammetry was employed to investigate the electrochemical behaviour of catechin and to determine the optimal oxidation potential for subsequent amperometric measurements. The experiments were carried out using a 200 mg L⁻¹ catechin solution prepared in 0.1 M phosphate buffer solution (PBS, pH 6.8). The voltammograms were recorded over a potential window ranging from -1.0 V

to +1.0 V at a scan rate of 0.001 V s^{-1} , with a step potential of 0.00305 V. For comparison, cyclic voltammetric measurements were also performed using a blank solution consisting of 5 mL of 0.1 M PBS mixed with 0.5 mL of reverse osmosis (RO) water under identical experimental conditions.

High-performance Liquid Chromatography Analysis

HPLC was employed as an independent and well-established analytical technique to validate the results obtained from the electrochemical detection of tea polyphenols. Catechin was used as the representative polyphenol standard for validation, owing to its predominance in tea and its well-characterised chromatographic behaviour. HPLC analysis was carried out using a Varian LC system (USA) comprising a ProStar 230 solvent delivery module coupled with a ProStar 330 photodiode array (PDA) detector. Chromatographic separation of phenolic compounds was achieved on an OmniSpher C18 reversed-phase column ($25 \text{ cm} \times 4.6 \text{ mm}$, Varian, USA), which was protected by a ChromSep guard column ($1 \text{ cm} \times 3 \text{ mm}$, Varian, USA). The use of a PDA detector enabled reliable detection and quantification of catechin based on its characteristic UV-visible absorbance, thereby providing a robust reference method for comparison with electrochemical sensor data.

For HPLC analysis, an accurately weighed 0.1 g of each tea sample was infused with 5 mL of freshly boiled distilled water in a boiling water bath for 5 min. The infusion was allowed to cool to room temperature and then filtered through filter paper to remove suspended solids.

The filtrate was subjected to a decaffeination step by liquid-liquid extraction with an equal volume of chloroform. After phase separation, the lower chloroform layer was carefully discarded. This extraction procedure was repeated twice to ensure efficient removal of caffeine, following the reported method (Copeland et al., 1997).

The decaffeinated aqueous phase was subsequently extracted with ethyl acetate. The ethyl acetate layer was collected in Eppendorf tubes and evaporated overnight at 45°C to remove the solvent. The resulting dried residue was reconstituted in HPLC-grade water and injected into the HPLC system for analysis. Chromatographic separation and quantification were performed according to a previously reported method (Liang et al., 2001).

Results and Discussion

Electrochemical Study

The cyclic voltammograms of catechin exhibited a well-defined anodic peak, confirming the electroactive nature of catechin at the fabricated sensor surface. The anodic oxidation peak was observed at approximately +0.450 V (vs Ag/AgCl), corresponding to the electrochemical oxidation of phenolic hydroxyl groups present in catechin molecules (Figure 2).

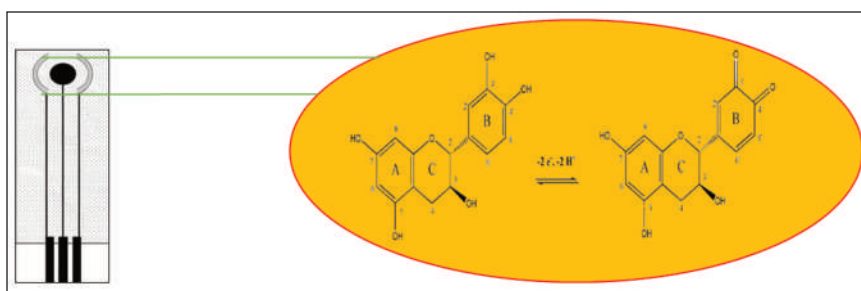


Figure 2. Reaction Mechanism of the Oxidation of Phenolic Hydroxyl Groups Present in Catechin.

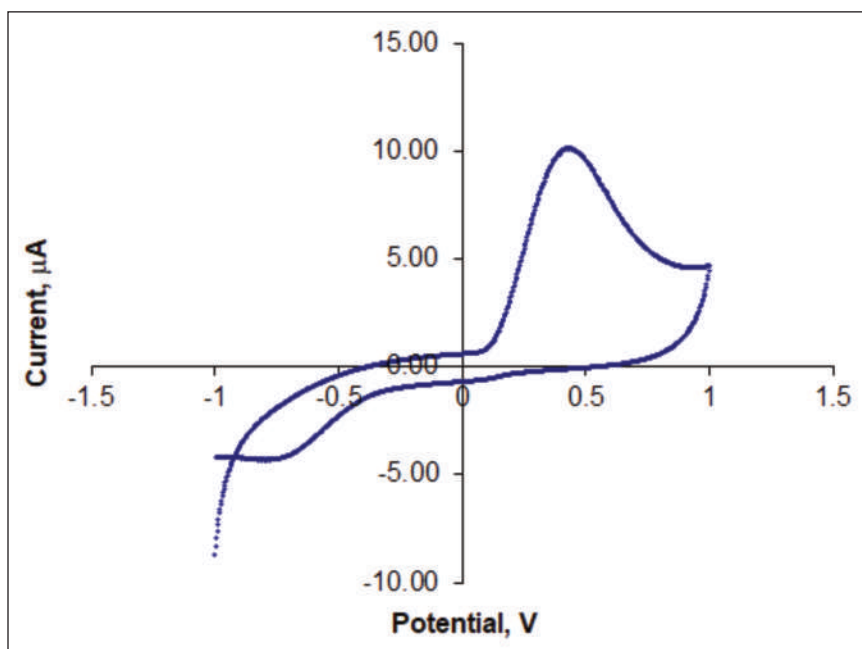


Figure 3. Cyclic Voltammogram of the Developed Sensor for Catechin (200 mg L^{-1}).

This potential was identified as the optimum oxidation potential, as represented in Figure 3, and was subsequently selected as the working potential for amperometric measurements.

To further evaluate the electrochemical response of the sensor towards varying catechin concentrations, amperometric responses were recorded for catechin solutions in the concentration range of $0\text{--}400 \text{ mg L}^{-1}$ under the same experimental conditions. As shown in Figure 4, the anodic peak current increased progressively with increasing catechin concentration, indicating a concentration-dependent electrochemical response. The well-defined and reproducible oxidation peaks

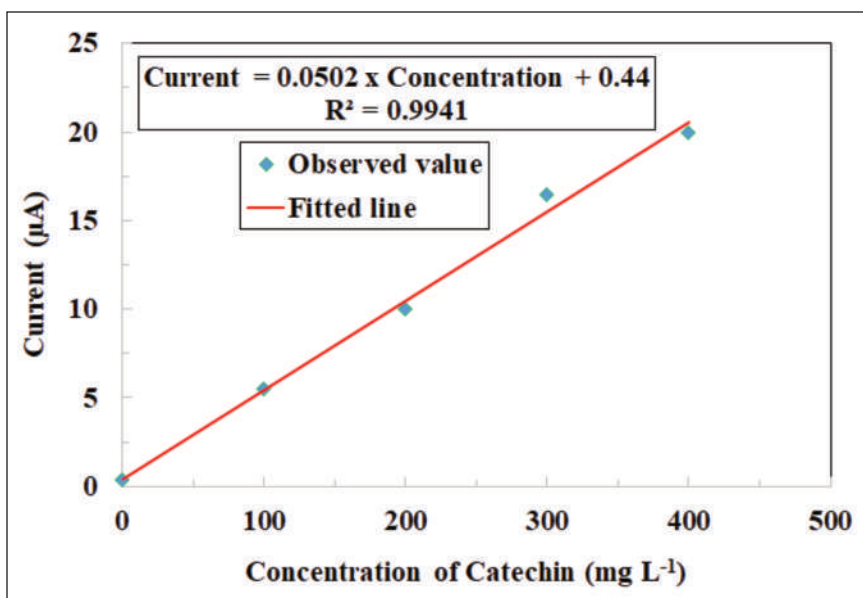


Figure 4. Calibration Curve for the Developed Sensor Using Different Catechin Concentrations.

demonstrate efficient electron transfer at the electrode interface and confirm the suitability of the fabricated sensor for quantitative determination of catechin and related tea polyphenols.

Validation and Statistical Analysis

To validate the quantification of catechin (total polyphenols) in tea samples, the analytical performance of the proposed electrochemical sensor was evaluated by comparing it with the reference HPLC method. The results obtained from both techniques exhibited a strong linear relationship, with a coefficient of determination (R^2) of 0.9877 and a correlation coefficient of 0.994, indicating excellent agreement between the two methods. The sensor demonstrated an LOD of 1.151 mg L⁻¹ and a LOQ of 3.838 mg L⁻¹. The catechin concentrations determined using HPLC closely matched those measured using the electrochemical platform, confirming the reliability and accuracy of the proposed sensor. The high correlation coefficient, approaching unity, further substantiates the strong agreement between the electrochemical and HPLC results, as illustrated in Figure 5A.

The repeatability of the electrochemical measurements was assessed by calculating the coefficient of variation (CV) for catechin determination in ten different tea samples. Each sample was analysed in triplicate using the fabricated sensor. As shown in Figure 5B, the CV values are <3% across all samples, demonstrating excellent precision and good repeatability of the proposed electrochemical sensing platform.

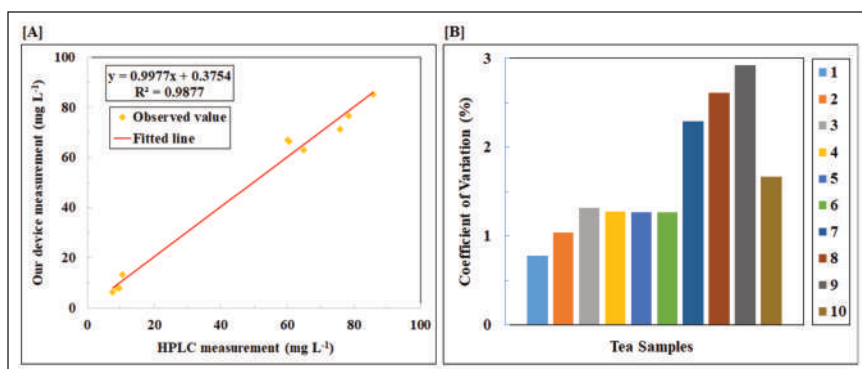


Figure 5. (A) Comparison of Tea Polyphenol Measurements in Our Device with the Standard Results Obtained from High-performance Liquid Chromatography (HPLC) for 10 Different Tea Samples that are Different from the Ones Used to Construct the Calibration Curve. The Experimental Data with Tea Polyphenol Concentrations Demonstrate Good Agreement Between the Present Method and the HPLC Method, with a Correlation Coefficient of 0.994. (B) Coefficient of Variation (CV) of Measured Data of Tea Polyphenols for 10 Samples Using the Present Device. The CV for Every Sample is <3%, Indicating Good Precision Among the Measurements.

Conclusion

A micro-fabricated screen-printed electrochemical sensor was successfully developed for the quantitative determination of tea polyphenols using catechin as a model compound. Cyclic voltammetric studies confirmed the electroactive nature of catechin, with a well-defined oxidation peak at +0.450 V, enabling sensitive amperometric detection. The sensor exhibited a wide linear response range, low detection and quantification limits and excellent repeatability across multiple tea samples. Validation against HPLC analysis demonstrated strong agreement between the two methods, confirming the accuracy and reliability of the proposed electrochemical platform. The low cost, disposability, simplicity of operation and rapid response of the sensor make it well-suited for routine analysis and on-site monitoring of tea polyphenols. Overall, this study highlights the potential of micro-fabricated screen-printed electrochemical sensors as practical analytical tools for tea quality control and broader food analysis applications.

Authors' Contributions

Sudip Chattopadhyay: Conceptualisation, data curation, formal analysis, validation, visualisation and writing—original draft.

Arnab Sarkar: Writing—review and editing.

Declaration of Conflicting Interests

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A Case Study on Failure of Anti-tank Missile Components

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Abstract

The pyro housings of the propulsion systems of three different anti-tank missiles were damaged during flight testing and received in damaged condition. Failure investigations were undertaken with the help of visual observation, optical microscopy, scanning electron microscopy, hardness testing, glow discharge optical spectroscopy, and optical emission spectroscopy to assess the reason for the failure of these components. Results indicated that all pyro housings were made from Al alloy HF15 grade (24345) and used in T6 heat-treated condition. The pyro housing of one missile failed, as the groove was longer and had a higher angle of inclination. This makes the groove thinner with reduced load-bearing capacity. There was improper manufacturing by closed-die forging of the other two pyro housings. This resulted in elongated grains in certain directions and lower load-bearing capacities. There was no metallurgical deviation from the recommended material and composition.

Keywords

Anti-tank missile, propulsion system, failure analysis, booster head

Introduction

An anti-tank missile having an imaging infrared seeker for a guidance system is used extensively in most warfare. These missiles have the striking capability of armoured vehicles located 4–7 km away. These missiles carry tandem warheads to strike main armour and explosive reactive armor. These missiles can be deployed on a land vehicle or rotorcraft.

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Failure analysis of propulsion systems, particularly solid propulsion systems, has been carried out earlier (Jha et al., 2011, 2013). Jha et al. (2011) investigated the failure reason of a stud made of maraging steel used in the flex nozzle control actuator bracket of a solid propulsion system. They found that the synergistic effect of stress and corrosion is responsible for the failure of the component. According to Jha et al. (2013), maraging steel fasteners used in the nozzle assembly of a solid propulsion system failed because of a similar reason. Several articles are available on the failure of domes (Bayraktar et al., 2022; El-Mahdy et al., 2025; Foraboschi, 2014). These domes are primarily civil structures. Failure of domes of pyro housing is not reported. Analysis of failure of various missile components has been carried out previously (Kim et al., 2005; Madhav & Roy, 2025; Schulz & Moran, 2001). It was found that the crack in the rocket motor of the interceptor missile was generated under the simultaneous action of corrosion and stress and propagated through grain boundaries (Kim et al., 2005). Failure of the pin locator was found to be because of the generation of intergranular quench cracking (Madhav & Roy, 2025). The missile fin of another system was found to fail due to cyclic loading by a fatigue crack, as confirmed by striation marks (Schulz & Moran, 2001). Failure investigation of domes of pyro housing of propulsion systems of missiles is not available at all in the literature. These facts prompted us to undertake the proposed work on the metallurgical investigation of failed parts of the propulsion system of the anti-tank missile.

In the present work, it was noted that the booster head of the propulsion system of an anti-tank missile was damaged. In another missile, it was found that the hot gases were directed toward the blast tube, resulting in the melting away of part of the dome of the propulsion system. The gas flow directed toward the centre melted away the aluminium (Al) alloy casing of the blast tube on one side up to half of the length in the propulsion system of another missile. Metallurgical investigations were carried out for all the failed components, and the results of the investigations are analysed in detail in the present article.

Background

The pyro housing, as an integral part of the booster head pertaining to the propulsion system of an anti-tank missile, was received in a damaged condition. This part is termed 'PH-1.' The booster head end is an enclosure for booster motor functioning for 1.4 s at a chamber pressure of 50 Ksc. Metallurgical investigation of the failed component is carried out in detail. The failed rear dome of the pyro housing of another missile designated as PH-2 was also brought for investigation. Another missile exhibited a defective dome of the pyro housing of the propulsion system. This part was designated as PH-3. Both these failed parts were examined extensively to assess the cause of the failures.

Investigations

All the failed components were visually examined with necessary care and details. Samples were cut close to the fracture surface and away from the fracture

surface, hot mounted, and polished progressively on emery paper up to 600-grit polishing, followed by cloth polishing. Samples were then viewed under an optical microscope in etched and unetched conditions. Etching was done with Keller's reagent. Hardness of the samples was measured with a Vickers indenter at a 5-kg load. The loading and unloading time was 10 s, and the holding time was 15 s. The chemical analysis of the pyro housings was carried out by instrumental analysis employing glow-discharge optical spectroscopy and optical emission spectroscopy. A glow-discharge optical spectrometer was used at a discharge voltage of 1,000 V and a discharge current of 50 mA. The pressure of argon gas was around 5 Torr. Optical emission spectroscopy was operated with high-purity argon gas with an inlet pressure of approximately 5 bar, with a high-voltage spark discharge up to 10,000 V and a frequency between 100 and 1,000 Hz. The failed samples were cut carefully so as not to damage the fracture features and ultrasonically cleaned in acetone. Subsequently, they were viewed under a scanning electron microscope. The microscope was operated at a power of 20 kV with a working distance of 15 mm. The aperture size was around 40 μm .

Results

Visual Examination

The failed booster head end of the pyro housing (PH-1) in the as-received condition is shown in Figure 1. Damage was observed to be due to an opening that occurred on the pyro housing where the pyro cartridge is fitted with an M16 threaded joint. Photographs showing the damaged, failed pyro housing, and intact pyro housing are presented in Figure 2. It is seen from Figure 2 that the damaged surface is very rough. The presence of several particles which solidified subsequently on the damaged surface can be



Figure 1. The Failed Booster Head End of the Pyro Housing (PH-1) (a) Front and (b) Back in the As-received Condition.

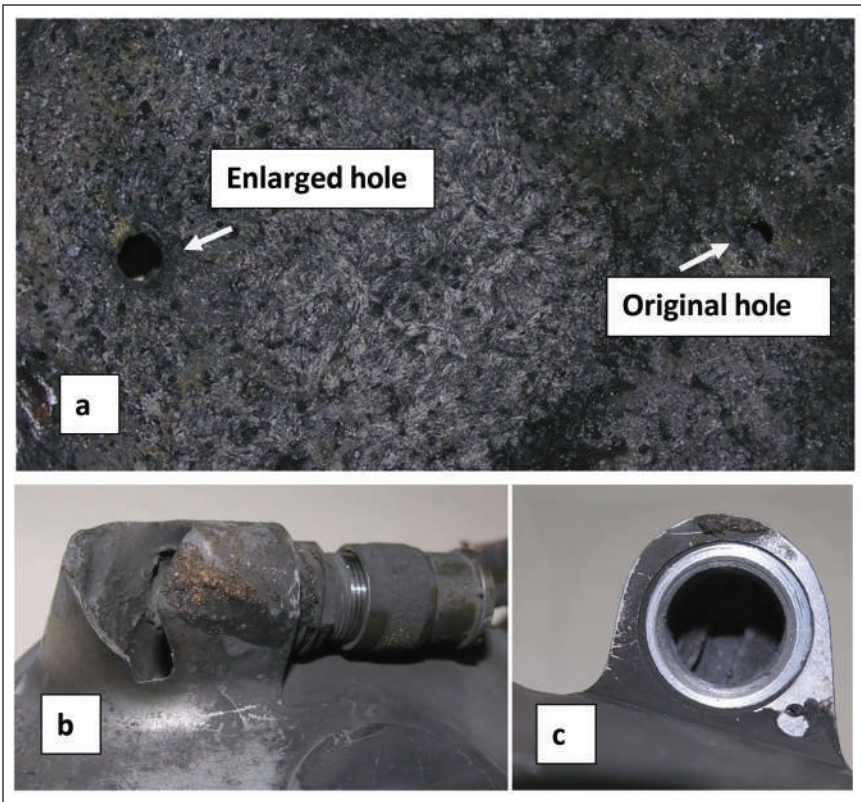


Figure 2. Photographs Showing the (a) Damaged Surface, (b) Failed Pyro Housing, and (c) Intact Pyro Housing.

noted. It is also noted that one hole is larger than the other one. The images of the damaged pyro housing (PH-2) of another anti-tank missile are presented in Figure 3. A part of the dome was found to be melted (Figure 3(a)). The rear wall was broken (Figure 3(b)), and the central hub was facing the pyro; with the loss of dome surface backing, the inner composite liner developed a crack (Figure 3(c)). The liner hole enlarged, and a crack was found to form in the liner hole. The photographs of the failed pyro housing of the third missile are illustrated in Figure 4. The liner is removed from the dome. Some deposition of molten metal can also be seen. Deposition of molten aluminium metal is shown in Figure 4. Part of the aluminium alloy casing of the blast tube melted (Figure 4(c)). The end wall of the flight is damaged (Figure 4(d)). A crack can be seen on the liner as well (Figure 4(e)).

Metallographic Examination

A representative microstructure of the metallic part of an intact pyro housing is provided in Figure 5. The material appears to be in solutionized, quenched, and

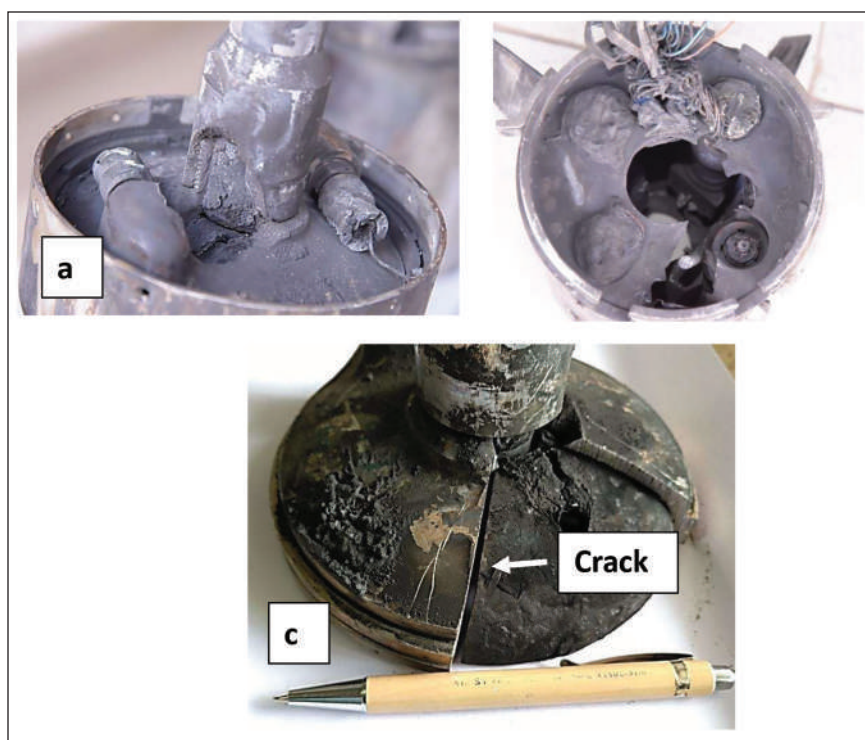


Figure 3. The Images of Damaged Pyro Housing (PH-2). (a) Failed Dome, (b) Broken Rear Wall, and (c) the Liner.

aged condition. Al alloys, based on their composition, are solution treated between 500 °C and 550 °C for a few hours so that strengthening elements are fully dissolved in the matrix. This is followed by rapid quenching in water so as to make a supersaturated, unstable solid solution. Finally, the part is artificially aged between 150 °C and 180 °C to precipitate the hardening particles. This treatment results in an increase in strength and hardness. This treatment is referred to as 'T6 heat treatment' for Al alloys. In the unetched condition, the microstructure of PH-1 indicates the presence of a crack that was propagating from the fracture surface toward the inside, as illustrated in Figure 6. An optical micrograph of damaged parts of PH-1 is given in Figure 7. Figure 7 reveals that, on etching with Keller's reagent, the microstructure reveals elongated grains and undissolved precipitates. Microstructures prepared from heat-affected zones of PH-2 and PH-3 are illustrated in Figure 8 and Figure 9, respectively. Although the heat-affected zone of PH-2 retained the original structure as shown in Figure 8, the microstructure of the heat-affected zone of PH-3 indicates grain boundary segregation and a rosette structure (Figure 9). These features confirm incipient melting of the material of PH-3.

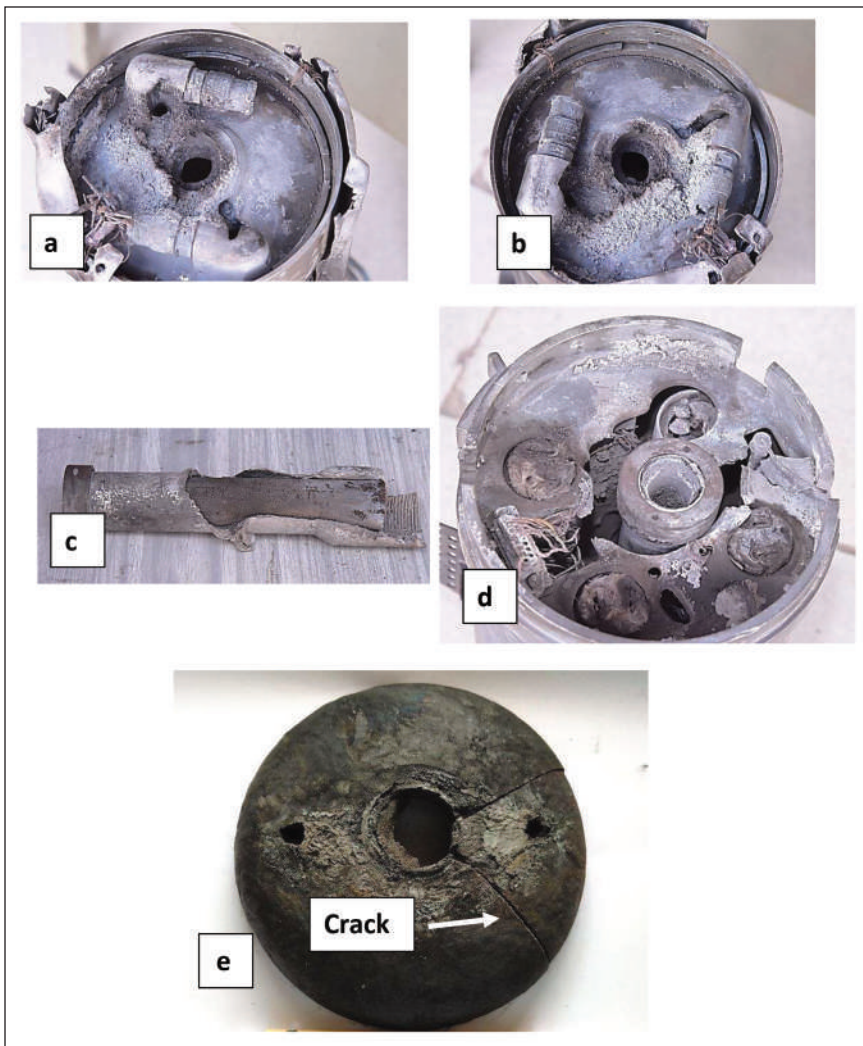


Figure 4. The Photographs of Both Failed Pyro Housings of PH-3, (a) and (b) Damaged Dome, (c) Casing of Blast Tube, (d) Broken End Wall, and (e) the Liner.

Hardness Measurement

Vickers hardness was measured in the regions near the fracture surface and away from the fracture surface. The hardness is given in Table 1. The hardness of PH-1 is significantly higher in the region near the fracture surface than that in the region away from the fracture surface. Hardness of PH-2 and PH-3 compiled in Table 1 reveals that hardness of PH-3 is significantly higher than that of PH-2.

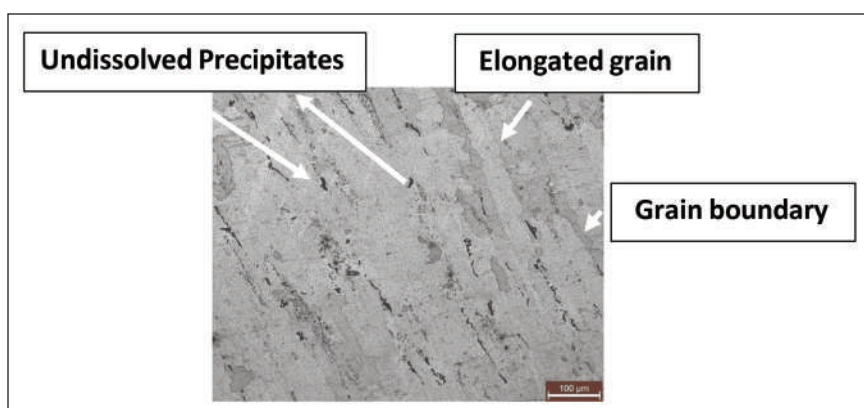


Figure 5. A Representative Microstructure of the Metallic Part of an Intact Pyro Housing.

Chemical Analysis

The chemical compositions of all the pyro housings (PH-1, PH-2, and PH-3) were measured by instrumental analysis. The measured compositions of the damaged component are listed in Table 2. Compositions of all the investigated materials are more or less similar. The material is an Al–Cu alloy containing trace amounts of Si, Mn, and Fe.

Fractographic Examination

The failed sample was cut carefully not to damage the fracture features and ultrasonically cleaned in acetone. The scanning electron microscopy (SEM) fractography of PH-1 given in Figure 10 has shown eroded features with oxide coating. One fine crack is also observed on the fracture surface (Figure 10(a)). SEM micrographs of the failed surfaces of PH-2 and PH-3 are presented in Figure 11. Deposition of molten metal in some places can be noticed. Figure 11 also reveals a clean-cut area possibly obtained by gas cutting.

Discussion

It was informed that in PH-1, there are two pyro housings as an integral part of the booster head end, of which one has failed. The booster head end is an enclosure of the booster motor functioning for 1.4 s at a chamber pressure of 50 Ksc. The booster head end is designed to withstand a proof pressure of 90 Ksc and a burst pressure of 120 Ksc.

The chemical composition of the pyro housing indicates that the material is an Al–Cu–Si–Mg alloy and conforms to aluminium alloy HF15 grade (24345). The microstructure and hardness indicate that the material is used in the T6 heat-treated condition. No abnormalities in the microstructure were observed. Fracture surface examination of PH-1 indicates that the material has eroded from the inner

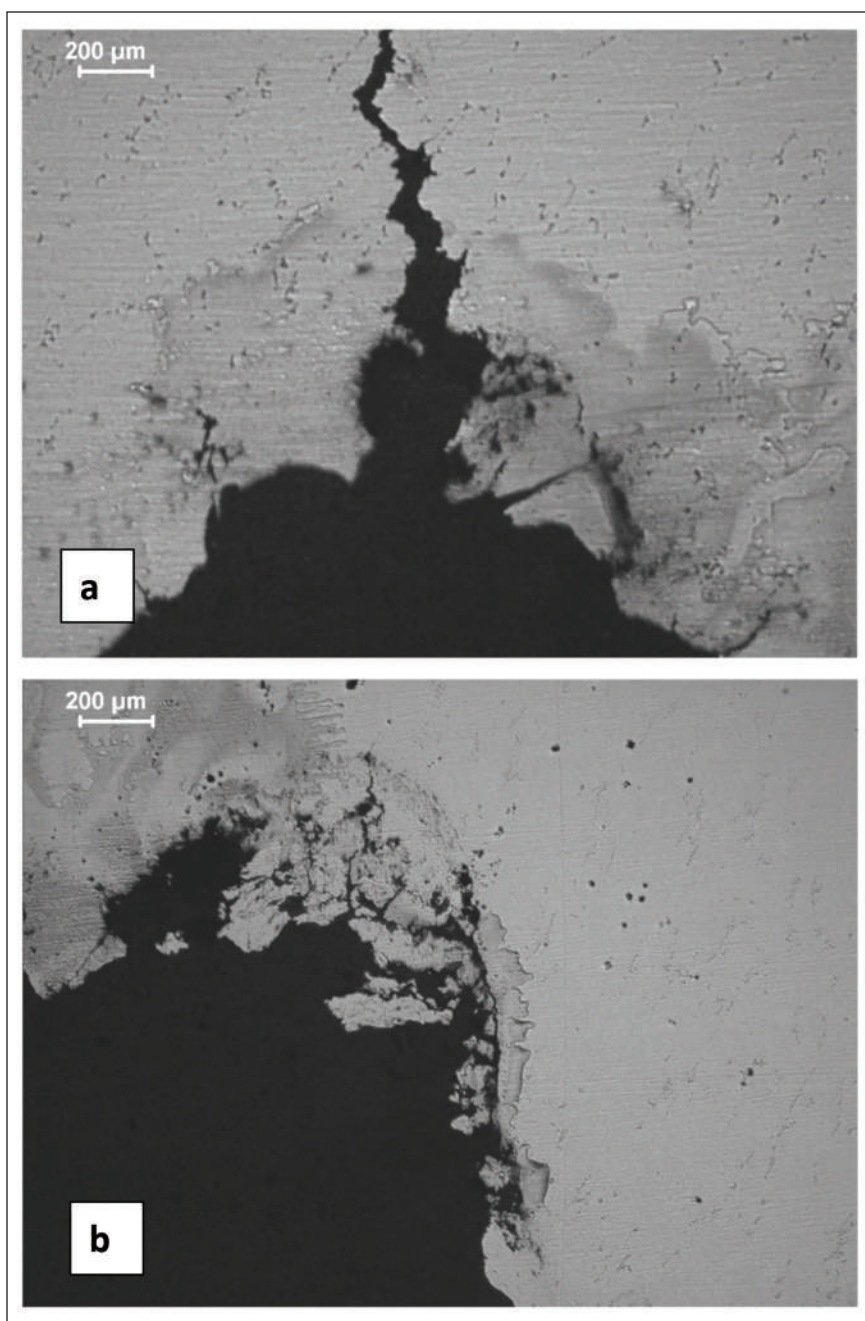


Figure 6. Optical Micrograph of PH-I in Unetched Condition Showing Cracks Emanating from the Fracture Surface.

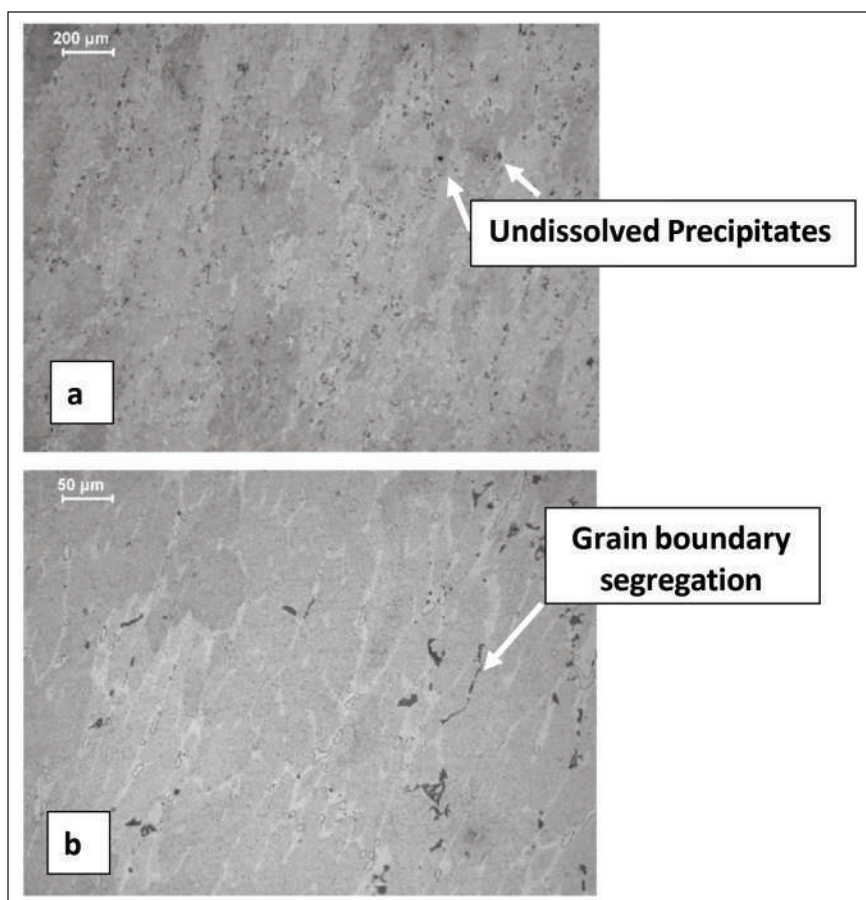


Figure 7. The Optical Microstructures Prepared from Heat-affected Zones of PH-1.

surface to the outer surface of the housing. From the visual observations, it is seen that the pyro hole and the angle at which the groove is made are not the same in both pyro housings. The failed pyro housing has a more inclined angle and a longer groove length when compared to the other pyro housing, which is intact. This longer groove might have resulted in a reduction of the thickness of the housing. The reduction in thickness of the housing (PH-1) and the more inclined angle of the groove could not withstand the pressure developed during ignition. This results in the generation of subsurface and surface cracks, leading to the failure of the housing. The crack initiated at the triple point of the elongated grain boundary, which has significant segregation. A crack can be initiated at the interface of the undissolved precipitate and the matrix. These features are observed in the micrographs of the component (Figure 7). The elongated grains of the failed part indicate tensile stress in the direction of grain elongation. Surface cracks, which are also responsible for failure, are seen in Figure 6. Cracks are generated on the surface as the stresses are maximum on the surface.



Figure 8. The Optical Microstructures Prepared from Heat-affected Zones of PH-2.

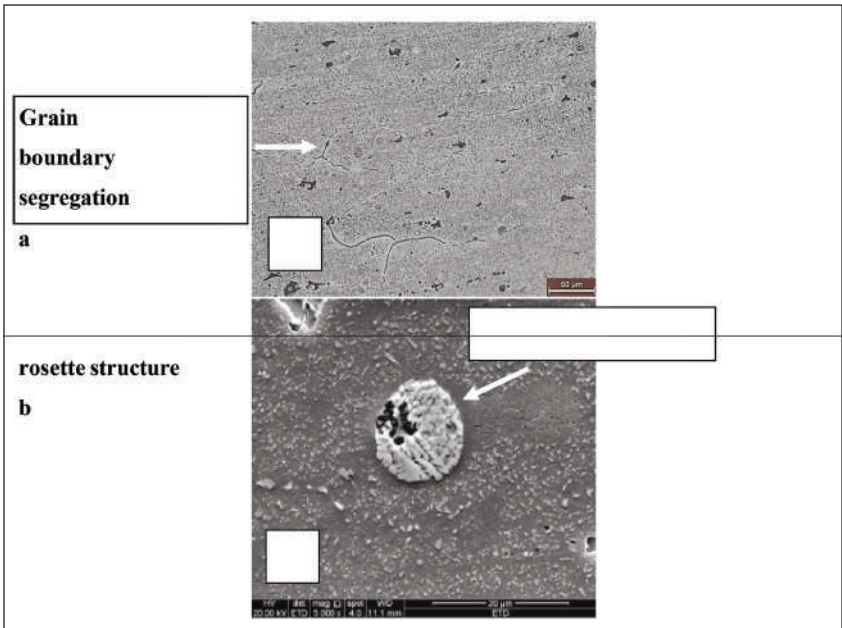


Figure 9. The Optical Microstructures Prepared from Heat-affected Zones of PH-3.

Table 1. Hardness of the Failed Components.

Serial No.	Components	Location	Hardness (HV)
1	PH-1	Near the fracture surface	147 ± 4
		Away from fracture surface	131 ± 4
2	PH-2	Near the failed surface	79 ± 1
		Away from failed surface	155 ± 5
3	PH-3	Near failed surface	89 ± 1
		Away from failed surface	155 ± 5

Note: PH: Pyro housing.

Table 2. Chemical Composition of the Failed Parts.

Components	Elements (wt%)								
	Cu	Si	Mg	Cr	Fe	Mn	Ti	Zn	Al
PH-1	4.65	0.33	0.42	—	—	—	—	—	Bal.
PH-2	4.7	0.68	0.73	0.05	0.15	0.75	0.05	0.13	Bal.
PH-3	4.47	0.6	0.71	0.05	0.2	0.7	0.05	0.12	Bal.

Note: PH: Pyro housing.

The failed parts of PH-2 and PH-3 are made of aluminium alloy similar to that of PH-1, as revealed by composition analysis. The chemical composition, microstructure, and hardness are well within the specified range, as seen above. Therefore, material deficiency is ruled out as the cause of the failure. Signs of hot gas flowing between the liner and the aluminium dome can clearly be understood, as failed parts have features of damage by hot gas. This exposure to the super-hot gas might raise the temperature of the dome, causing loss of strength and subsequent failure of the pyro housing wall. Evidence of pyro housing (PH-2 and PH-3) leaking was noted. The hot gases are directed toward the blast tube of PH-2, resulting in the melting away of part of the dome and central hub facing the pyro, with the loss of dome surface backing; the inner composite liner developed a crack. The hot gases escaped from the last chamber by breaking the rear wall. The gas flow of PH-3 was directed toward the centre, melting away the aluminium alloy casing of the blast tube on one side up to half of the length. A small part of the dome surface was melted. The hot, high-speed gases made some holes in the rear wall of the last chamber and escaped. Some molten aluminium metal is seen deposited. The liner of PH-3 showed a pattern of gas flow from the pyro holes toward the centre. This flow occurred between the aluminium alloy dome and the composite liner. The leak was initially directed horizontally toward the centre and then became vertical due to erosion in the aluminium alloy wall. The quantum of gas flow in PH-3 is significantly higher than that in PH-2, resulting in alteration of the microstructure of PH-3 but not in PH-2. The gas flow left evidence of deposition of molten Al alloy, and higher gas flow in PH-3 gives rise to a clean gas-cut surface.

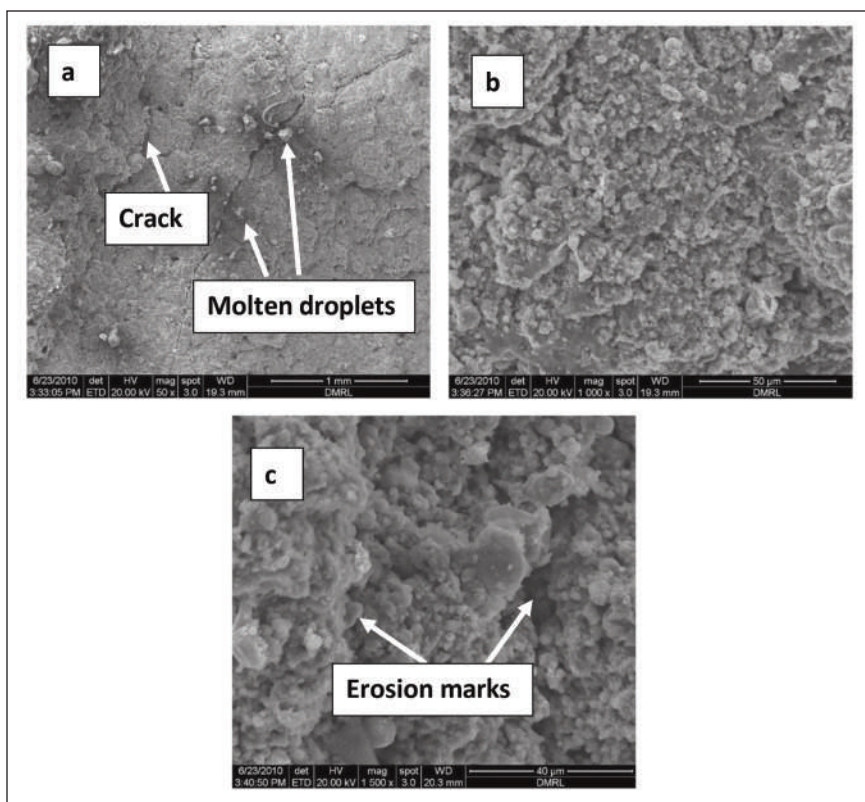


Figure 10. (a, b, c) Scanning Electron Microscopy (SEM) Fractograph Taken from the Failed Surfaces of PH-I.

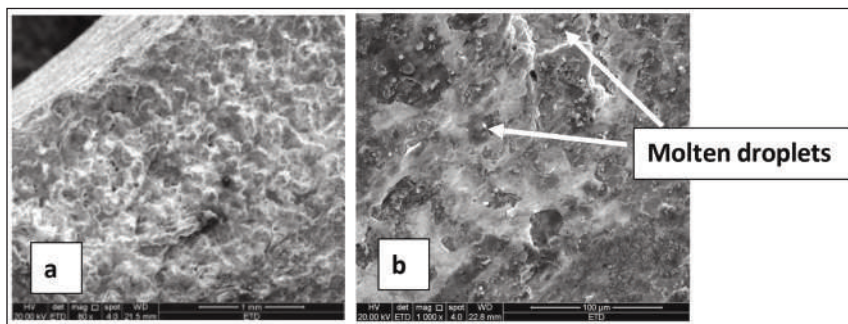


Figure 11. Scanning Electron Microscopy (SEM) Micrographs of the Failed Surfaces of (a) PH-2 and (b) PH-3.

The domes of the pyro housings were made by closed-die forging and machining. They have two integral pyro housings and a central exit. The pyro housings have internal threads made by either machining or tapping. The central

exit has outer threads to fix the blast tube. The rocket motor has an internal heat-resistant composite lining bonded with the aluminium alloy body with adhesive (Araldite). The dome has two oblique holes at 40° connecting the pyro housing internal volume with the rocket engine drilled through the lining and the dome wall. The rocket motor is ignited with both pyros firing simultaneously. The spark from the pyros travels through the 5-mm holes into the rocket motor, where it starts burning the solid fuel. The central exit has a tight-fitting graphite plug with a hole through which the hot gases move out, providing the required thrust. The exhaust gases travel through a cylindrical tube made of carbon–carbon composite up to the end of the missile through the control fin chamber. The C–C composite tube is held in place with an aluminium alloy tube threaded to the dome.

The missile has completed several successful trials without such failure in the past, implying there is no serious design or material problem. The dome in the beginning was made from bar stock that was later modified to closed-die forging. It should be noted that even the domes made from bar stock did not fail in this fashion. It does appear that deviation from the established manufacturing processes is the cause of the failure, specifically the bonding between the inner liner and the dome. The generation of cracks can take place at the triple-point boundaries of elongated grains with grain boundaries having significant segregation, weakening the grain boundary.

This type of failure can be avoided if an Al alloy with elevated temperature capability is used (Huang et al., 2024; Qian et al., 2008; Xue et al., 2023). The other option for improving performance is to coat the Al alloys with thermal-sprayed thermal barrier coating systems (Gokul Lakshmi et al., 2021; Malvi & Roy, 2021) or by coating a heat-resistant layer employing physical vapor deposition (PVD) techniques (Monfared & Dolati, 2026; Roy et al., 2020).

Conclusion

1. Detailed investigations to identify the cause of failure of pyro housings of three anti-tank missiles have been carried out.
2. Detailed analysis revealed that all the components of the propulsion system are made from aluminium alloy HF15 grade (24345) in the T6 heat-treated condition.
3. Metallurgical abnormality related to deviation from recommended material and composition or heat treatment cannot be noted.
4. The angle at which the groove is made is higher, and the grooves are deeper in the case of failed pyro housing than in the case of the pyro housing that was intact. This leads to a reduction in thickness and an increase in stresses, which, in turn, is responsible for the failure of the pyro housing (PH-1).
5. The manufacturing processes of the other two pyro housings (PH-2 and PH-3) were different from the recommended process. These components are manufactured by the closed-die forging process, which is a deviation

from the original manufacturing process. This may be responsible for premature failure leading to a gas leak.

6. The cracks are initiated at the triple point of elongated grain boundaries, which have significant segregation leading to weakening of grain boundaries. A crack can be initiated at the interface of the undissolved precipitate and the matrix. These features are observed in the micrographs of the component. Subsurface cracks are seen in all three failed components. However, a surface crack was seen only in PH-1.

Declaration of Conflicting Interests

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Predictive Wear Depth Analysis of Al–GnP–hBN Hybrid Nanocomposites Through Machine Learning Techniques

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Abstract

Reinforcement content and sliding duration strongly influence the wear behaviour of aluminium (Al)-based hybrid composites, and evaluating these parameters experimentally remains time-consuming and resource-challenging. This study presents an extensive machine-learning framework to forecast the wear depth of Al–graphene nanoplatelet (GnP)–hexagonal boron nitride (hBN) hybrid nanocomposites with a data set of 3,016 experimentally obtained measurements, with wt% reinforcement and sliding time as inputs. Five supervised regression models (Linear Regression, Support Vector Regression, Multi-Layer Perceptron, Random Forest and Extreme Gradient Boosting) were trained and evaluated with the performance metrics of R^2 , root mean square error and mean squared error. The minimal prediction error with the highest accuracy ($R^2 \approx 1.000$) was obtained with the Random Forest model, exceeding both linear and kernel-based approaches capable of describing the non-linear wear responses shown by Al hybrid composites. SHAP analysis indicated that sliding time is the dominant factor governing wear depth, while reinforcement wt% contributes secondary but meaningful effects. The projected machine learning-driven method delivers a fast, dependable and characterisation-free method for expecting wear behaviour and delivers interpretable insights into parameter influence, offering a valuable tool for the design and optimisation of Al hybrid nanocomposites for advanced engineering applications.

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Keywords

Aluminium hybrid composites, GnP–hBN reinforcement, machine learning regression, wear depth modelling, explainable AI (SHAP)

Introduction

Modern engineering systems frequently require materials with multifunctional qualities, although such property combinations are challenging to achieve using monolithic alloys. Using mixed nanostructured reinforcements in MMHnCs offers a feasible approach to enhance metal performance, providing increased strength combined with improved wear and thermal stability.

AMMHnCs have been getting more and more attention lately because they have a strong yet light structure, good stiffness, consistent thermal behaviour, and better corrosion and wear resistance (Abushanab et al., 2023; AlMangour et al., 2019; Chung, 1987; Falin et al., 2017; Firestein et al., 2017). Graphite, carbon nanotubes (CNTs), graphene, graphene oxide, carbides, borides and transition-metal dichalcogenides (TMDs) are among the many nanofillers used to upgrade the mechanical and tribological properties of aluminium matrices (Geim & Novoselov, 2007; Geng et al., 2009; Ghosh et al., 2023; Ghosh, Shukla, et al., 2025; Gostariani et al., 2018). Each nanofiller adds its unique value to the composite by combining different types of reinforcement. When these fillers operate together, they make the material stronger, tougher and more resistant to heat and wear. This method also makes the material less likely to rust and wear out. Due to these benefits, hybrid reinforcement is becoming a popular alternative in fields with high demand, such as aerospace and automotive engineering.

Aluminium is used extensively, but its poor wear resistance at elevated temperatures and low fracture toughness make it less suitable for components that operate under high friction (Li et al., 2023; Sahoo et al., 2026; Surappa, 2003; Udoye et al., 2019). Graphene nanoplatelets (GnP) and hexagonal boron nitride (hBN), as 2D reinforcements added to aluminium, are a good approach to get around these problems (Ghosh et al., 2026; Mohammed et al., 2013). Graphene has exceptional fracture strength, stiffness, thermal conductivity and large surface area (Das et al., 2026). This material enhances load-transfer efficiency and strengthens the surrounding matrix (Chung, 1987; Geim & Novoselov, 2007; Geng et al., 2009; Hatui et al., 2014; Yasmin et al., 2006). Likewise, hBN, with its layered structure, chemical stability, solid-lubricating behaviour and high stiffness, performs as an efficient wear-reducing reinforcement (Falin et al., 2017; Li et al., 2014; Song et al., 2010; Wickramaratne et al., 2018). Even though they have a lot of potential, making Al–GnP–hBN hybrid nanocomposites is hard since the nanoparticles tend to clump together, the bonds between them are not strong enough, and it is hard to spread them out evenly. Research shows that both the mechanical response and wear performance are strongly affected by reinforcement weight percentage and the chosen processing technique, such as powder metallurgy (PM), spark plasma sintering (SPS) or severe plastic deformation (Casati & Vedani, 2014; Ghosh et al., 2023; Ghosh, Alam, et al., 2025; Huang

et al., 2019; Pérez-Bustamante et al., 2014; Sardarabadi et al., 2022; Shrivastava et al., 2021). Combinations of GnP and hBN have yielded notable synergistic improvements, and recent research points to ultrasonic mixing and PM as reliable methods for limiting agglomeration and achieving better dispersion (Ghosh, Alam, et al., 2025; Kumar et al., 2021; Sahoo et al., 2024, 2025; Şenel & Gürbüz, 2021).

In parallel with experimental work, machine learning (ML) is now widely used to study tribological behaviour and to predict the wear performance of aluminium-based composites, helping to cut down on large-scale testing efforts. Models such as Linear Regression (LR), Support Vector Regression (SVR), Random Forest (RF), Extreme Gradient Boosting (XGBoost) and Multi-Layer Perceptron (MLP) neural networks are commonly employed to forecast wear depth, wear rate and friction coefficients from both material composition and testing conditions. Current studies indicate that ML models are capable of learning complex non-linear interactions between composition, reinforcement level, load, speed and sliding duration, allowing them to deliver quick and reliable surrogate predictions for tribology data (Marian & Tremmel, 2021; Moosavi et al., 2020). Tree-based ensemble methods such as RF and XGBoost generally deliver better performance than linear or kernel-based models on aluminium composites and hybrid systems as per the literature. Their advantage comes from their ability to learn non-linear patterns, work well with mixed feature types and maintain robustness against overfitting (Hamrol et al., 2024; Mishra, 2025). While neural networks can produce highly accurate results, they usually depend on sizeable data sets and meticulous tuning. Alongside prediction, methods like SHAP improve interpretability by revealing how material composition and test conditions shape wear behaviour, thereby linking model predictions to the corresponding tribological mechanisms. SHAP is increasingly useful in materials science to recognise key features governing mechanical and wear properties and to support data-driven optimisation of composite formulations (Zhong et al., 2022). Taken together, these results support the choice of RF, XGBoost and SHAP in this study, since they deliver high predictive accuracy while also offering insight into the underlying mechanisms. By combining ML with experimental evidence, a more complete understanding and optimisation of aluminium–GnP–hBN hybrid nanocomposites can be achieved for advanced engineering uses.

To summarise, while many studies have focused on GnP- or hBN-reinforced aluminium composites separately, much less is known about how these reinforcements behave together, the best hybrid proportions, the associated dispersion issues and their combined tribological response. To bridge this gap, the current study develops aluminium hybrid nanocomposites containing an equal-ratio GnP–hBN nanofiller system (G50B50), produced through a carefully controlled ultrasonic-assisted PM process that improves dispersion and reduces agglomeration. Because wear behaviour in aluminium hybrid composites is highly complex and only a few studies have explored data-driven approaches, there is still a noticeable gap in applying ML to reliably predict wear and to analyse the role of different parameters. To address this, the present work develops a comprehensive ML-based framework using an experimentally obtained wear data set of aluminium hybrid composites

with varying wt% reinforcement and sliding time as inputs. Five supervised learning algorithms (LR, SVR, MLP, RF and XGBoost) are systematically evaluated to identify the most reliable predictive model. Furthermore, explainable AI analysis using SHAP is employed to quantify the contribution of each input parameter and reveal underlying non-linear wear mechanisms. This study is among the first to apply a complete ML-interpretability pipeline to aluminium hybrid composite tribology, offering a fast, scalable and characterisation-free approach to understanding and predicting wear behaviour for engineering applications.

Materials and Methods

Experimental Method

In this work, graphite nanoplatelets were obtained by thermally exfoliating a graphite intercalation compound prepared from natural flake graphite. The starting graphite, sourced commercially from Loba Chemie (India), had a mesh size of 60 and a stated purity of 98%. To form the intercalated precursor, the natural flake graphite was treated with a mixture of concentrated sulphuric acid and hydrogen peroxide. Specifically, 6 g of the graphite powder was combined with 16 mL of 98% H_2SO_4 and 1.5 mL of 30% H_2O_2 , both supplied by Merck India, to facilitate the intercalation step before thermal expansion. After the reactants were combined, the suspension was kept under magnetic stirring at room temperature for about four hours, during which it gradually transformed into a light, foam-like mass. This intermediate material was washed five to seven times with distilled water until the filtrate reached neutral pH. After washing, the solid was oven-dried overnight at 80 °C. The dried intercalated graphite was quickly thermally treated in a muffle furnace at 1,000 °C for 30 s. Sudden heating released acidic species and formed thermally exfoliated graphite nanoplatelets. The complete experimental procedure and composition of the composites are presented in Table 1.

After thermal expansion, the graphite was ultrasonicated for 20 h to separate GnP layers. The same length of high-energy milling was used to exfoliate hBN from Oxford Lab Fine Chem LLP (India) with an average particle size of 5 μm . The binary hybrid system was prepared by dispersing GnP and hBN in acetone and ultrasonicated for 2 h. This hybrid dispersion was then combined with aluminium powder in acetone at a 1:50 ratio (1 g of powder in 50 mL of acetone) and ultrasonicated for two more hours to achieve uniform distribution. Ultrasonication was performed with a 1,000-W high-power oscillator (Sonapros PR 1000 MP, Oscar Ultrasonics Pvt. Ltd) at 2–3 kHz and a 12-mm horn. Acetone ($(\text{CH}_3)_2\text{CO}$) was used as a cooling fluid and dispersant to inhibit reactions and ensure nano-filler distribution. After being dispersed, the Al–GnP–hBN powder mix was then pressed together using a uniaxial press at 550 MPa for about 5 min. This formed green pellets that were about 10 mm in diameter and 3.5 mm thick. After that, these compacts were sintered in a tubular furnace (Bysakh & Co., India) for 2 h at 550 °C in an argon environment. The tubular furnace used in this investigation may become as hot as 1,700 °C. A Ducom TR 208 M1 ball-on-plate dry-sliding wear system was used to measure the wear behaviour of the sintered specimens,

such as wear depth, material loss and wear rate. A hardened SAE 52100 steel ball with a diameter of 2 mm was used as the counterbody during testing. The tests were done with a standard load of 15 N, the ball spinning at 20 rpm for 10 min, and the temperature and humidity in the lab were about 298 K and 40%–60%. The dry-sliding experiments were done with only one set of operating settings because the main goal was to see how the generated nanocomposites wear down on their own. The examples were made using PM; thus, a test load of 15 N was chosen to make sure that the wear track did not get too bent or break. The same reason kept the speed of rotation at 20 rpm. The schematics of the experimental process have been shown in Figure 1. Initial observations showed that the penetration depth stayed the same after the first 10 min of sliding. Because of this, each test could only last 10 min.

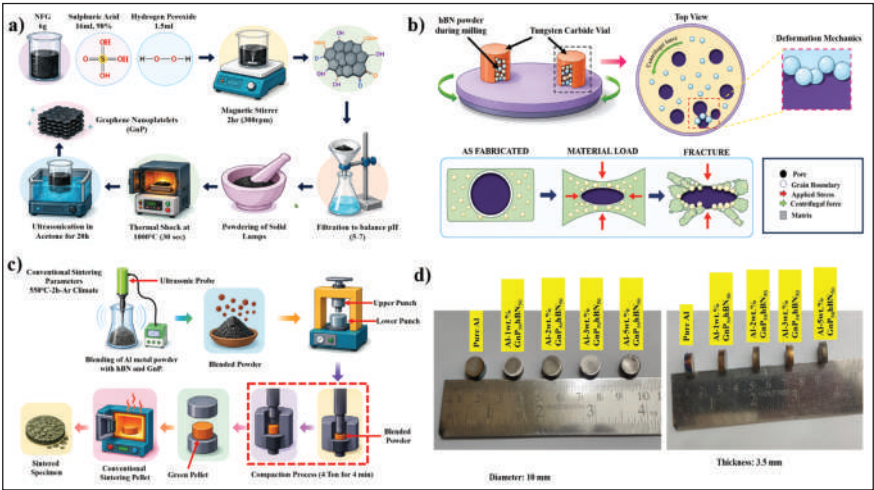


Figure 1. Schematics for (a) Synthesis of Graphene Nanoplatelet (GnP), (b) Milling of Hexagonal Boron Nitride (hBN), (c) Conventional Sintering Process and Preparation of Sintered Pellet, and (d) the As-obtained Green Samples With Their Respective Dimensions.

Table 1. Details of the Composition of Various Aluminium (Al)-based Binary Nanocomposites.

Composition	Pure Al (wt%)	GnP (wt%)	hBN (wt%)	Abbreviation
Pure Al	100	0	0	Al
Al-1wt%GnP ₅₀ hBN ₅₀	99	0.5	0.5	Al-1wt%G ₅₀ B ₅₀
Al-2wt%GnP ₅₀ hBN ₅₀	98	1	1	Al-2wt%G ₅₀ B ₅₀
Al-3wt%GnP ₅₀ hBN ₅₀	97	1.5	1.5	Al-3wt%G ₅₀ B ₅₀
Al-5wt%GnP ₅₀ hBN ₅₀	95	2.5	2.5	Al-5wt%G ₅₀ B ₅₀

Note: GnP: Graphene nanoplatelet; hBN: Milling of hexagonal boron nitride.

Machine Learning Methods

ML approaches help find connections in tribological data that were not known before. This makes it easier to manage and improve the tribological properties of new materials (Hasan et al., 2021). Regression analysis within supervised ML systems proves particularly effective for predicting tribological behaviours. ML algorithms were specifically designed to anticipate the wear depth of aluminium and its composite. Efficient data collection is essential for constructing a strong, data-driven ML algorithm. ML models generally perform better when trained on substantial and well-matched data sets. Yet, generating adequate data from tribological tests demands considerable effort, time and experimental resources. Our data set has 2,412 data points for training and 604 data points for testing out of 3,016 sample data points. This makes it easier to figure out how deep the wear is. The ML algorithms took in two things: the weight percentage of reinforcement and the time in seconds. The variable that predicts is called wear depth.

Machine-learning Models

ML models are computer programs that learn from data and use it to make predictions about new data sets based on the input–output relationships they saw in training cases. We created models using five different ML algorithms: LR, RF, SVR, MLP and extreme gradient boosting (XGBoost). We used Python and its built-in ‘scikit-learn’ package to do the ML study. Below are full explanations of several algorithms. Linear regression is a simple and popular way to learn under supervision that shows how input features and a continuous output are related. This study creates a baseline by positing a linear correlation between wear depth, weight percentage and time. Although it provides easily interpretable coefficients showing how each parameter influences wear, its predictive performance is limited when the underlying wear behaviour is non-linear. Therefore, more advanced ML models are required to capture the complex interactions governing the wear process. People often utilise RF for classification jobs, but it is also quite good at solving regression problems. This approach uses a method called ‘ensemble’, which includes random decision trees. Each decision tree is trained using a sample from the training set of data. The parameter ‘n_estimators’ tells the RF model how many trees were made during training. This amount is what makes the model complex (Breiman, 2001).

The SVR extends the support vector machine framework (Zhong et al., 2022) to handle regression problems (Awad & Khanna, 2015). To ensure that predictions stay within predetermined bounds, SVR creates higher-order hyperplanes to capture strong correlations between the inputs and the intended outputs. By employing kernel functions to produce these hyperplanes, the overall generalisation error is decreased. Both the regularisation parameter and γ during hyperplane formation have a significant impact on SVR accuracy. Tribology research shows that SVR can achieve excellent accuracy even with little data sets and is especially effective with high-dimensional inputs (Hasan et al., 2022). A feedforward neural network with an input layer, one or more hidden layers and an output layer

is called an MLP. Training is typically carried out using backpropagation, which calculates how the loss changes with respect to each weight and adjusts those weights through gradient descent.

XGBoost is a powerful ensemble method that extends gradient-boosted decision trees, offering strong predictive performance and efficient training. XGBoost grows trees one after another, with each tree focusing on the mistakes of the previous ones. This kind of learning works well for modelling the non-linear interactions that can be seen in wear data sets. XGBoost works well with structured data and has regularisation techniques that keep it from overfitting. It often makes better forecasts of wear depth based on wt% and time than many standard methods since it can generalise so well.

In this study, model performance was evaluated using three metrics: the coefficient of determination (R^2), root mean square error (RMSE) and mean squared error (MSE). R^2 measures how effectively the regression model accounts for the variability in the target values.

Data Preprocessing

At first, go through the data set to locate and fix any numbers that are missing or do not make sense. The data set is shuffled to keep hidden biases or unwanted patterns from coming into the ML models before training. Then, Python Colab is used to split the whole data set into training and test sets. The training set is used to build and fine-tune the ML models. Then, their ability to make predictions is tested on the test set, which is made up of data that is not seen before. Here, use a typical 80:20 split to divide the data set. Eighty per cent of the data are used for training and 20% for testing. A total of 2,412 samples are used to train the ML models and 604 samples are used to test them. In total, 3,016 data points are used to forecast wear depth.

Model Validation

To evaluate the robustness and generalisation capability of the developed ML models, k-fold cross-validation was additionally performed. The complete data set was randomly divided into five equal subsets ($k = 5$). During each iteration, four subsets were used for training and the remaining subset was used for validation. This procedure was repeated five times so that each subset served once as the validation set. The average values and standard deviations of R^2 , RMSE and MSE were calculated across all folds. Cross-validation minimises the dependence of model performance on a single train-test split and provides a more reliable assessment of predictive stability and generalisation.

Results and Discussion

Figure 2 shows a radar plot that compares the ML models that were tested for wear-depth prediction. It shows how work on R^2 , inverse RMSE and inverse mean

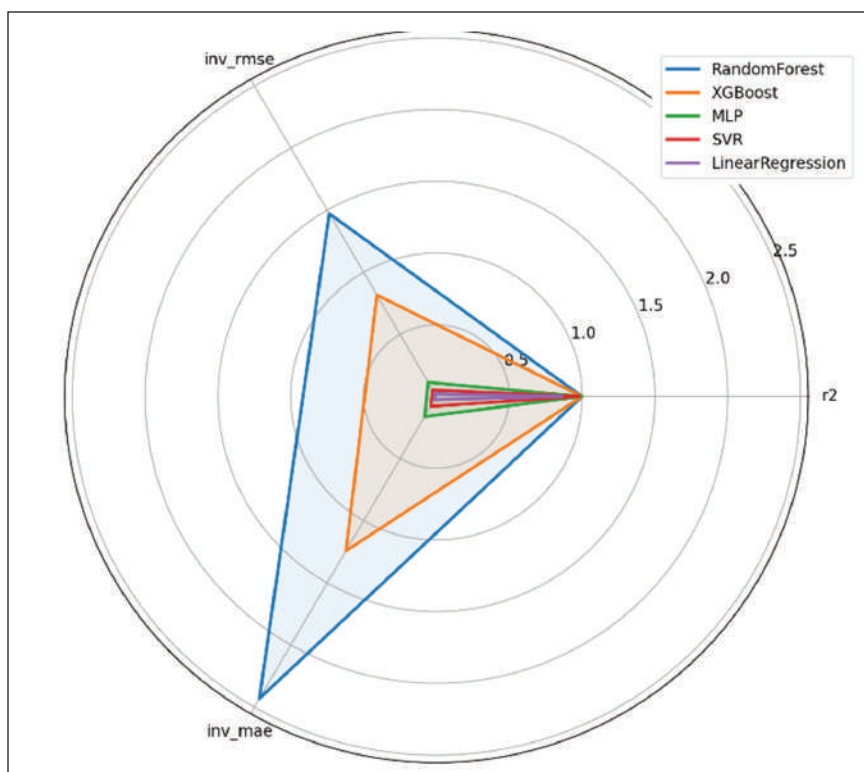


Figure 2. Radar Plot Comparing the Performance of All Machine-learning Models Using R^2 , Inverse Root Mean Square Error (RMSE) and Inverse Mean Absolute Error (MAE).

absolute error (MAE). In the radar diagram, RF covers most of the area, which indicates better predictions and also has less errors than XGBoost, MLP, SVR and LR. It indicates that tree-based ensemble models are superior and how the non-linear interactions between wt% and sliding time affect wear behaviour. The small regions for SVR and LR show that simpler models have trouble capturing the system's non-linear structure. Figure 3 illustrates the correlation matrix that connects the input features (wt% and time) with wear depth. This is our first look at how these things function together. The wear depth has a strong positive association with time ($r = 0.71$), which suggests that, as sliding continues, more material is lost, which is what tribology says should happen. The modest positive correlation between wt% and wear depth ($r = 0.52$) indicates that elevated reinforcement levels affect wear behaviour, possibly due to variations in hardness or microstructure. The slight negative correlation between wt% and time also illustrates that these inputs do not really depend on each other.

Figure 4 shows direct links between wear depth, wt% and time, verifying the matrix correlations. All compositions' wear depth against time graph rises, confirming the significant positive correlation ($r = 0.71$) and that wear grows progressively while sliding continues. The wear depth against wt% graph is more scattered

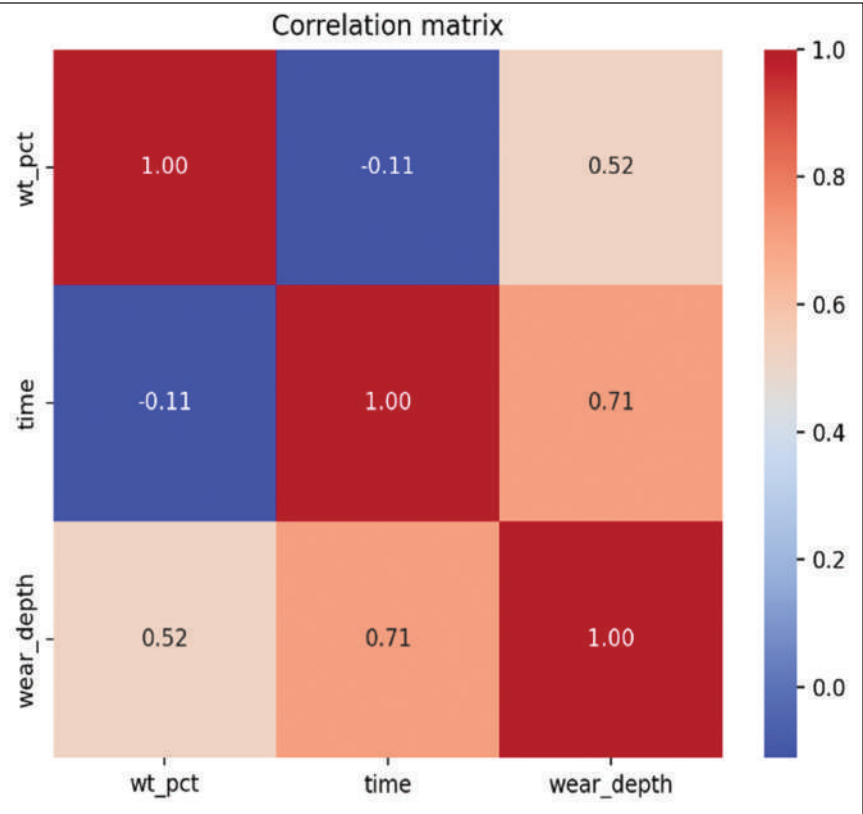


Figure 3. Correlation Matrix Showing the Relationship Between wt%, Time and Wear Depth.

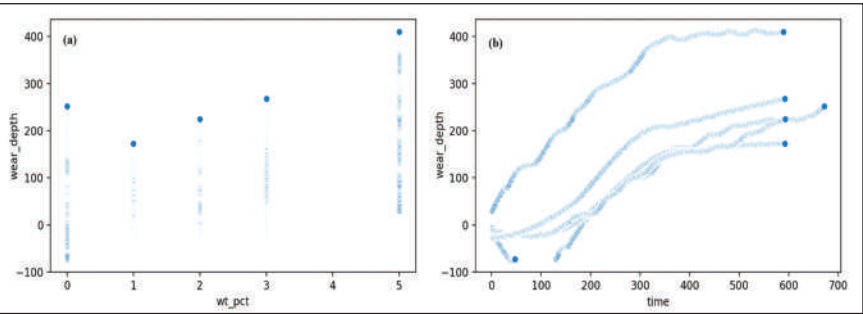


Figure 4. Scatter Plots Showing the Relationship of Wear Depth With wt% (Left) and Time (Right).

but still shows a modest rising trend, matching the moderate correlation ($r = 0.52$) and demonstrating that reinforcement percentage affects wear, though not as substantially as time. The figures support the correlation matrix results, suggesting that time affects wear more than wt%.

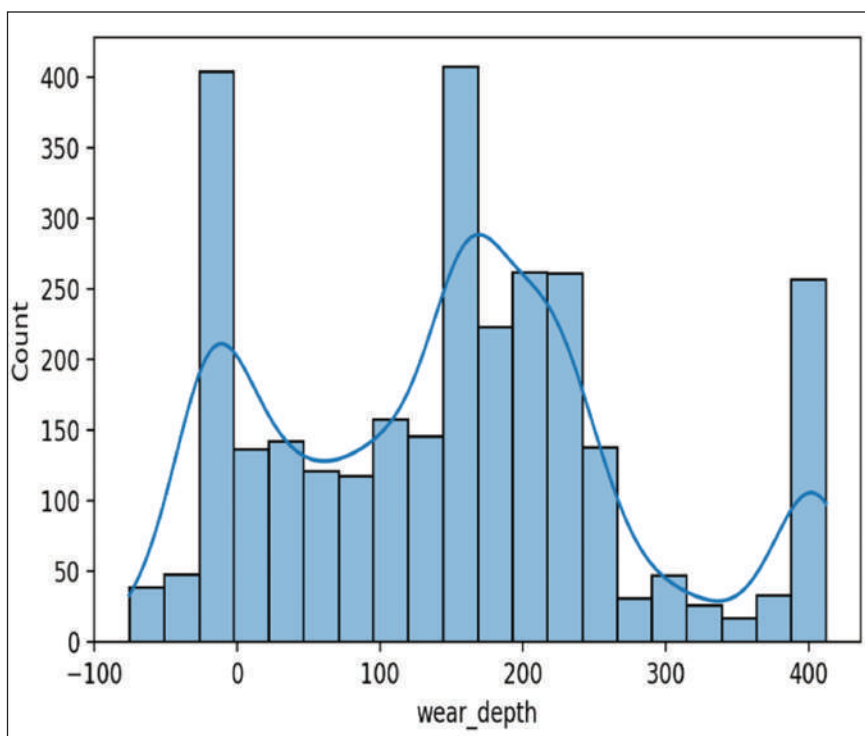


Figure 5. Histogram Showing the Distribution of Wear Depth Across All Experimental Conditions.

Figure 5 shows how wear depth is spread out under all test settings. Instead of a smooth peak, it has a multimodal structure. These distinct clusters match up with different wt% and sliding time combinations, which means that wear behaviour fluctuates a lot depending on the experimental setup. The range of wear depth from negative to big positive values shows that the data set has both mild and severe wear regimes. This kind of diversity shows how the system behaves in a non-linear way and shows how important it is to use advanced ML models that can learn complicated patterns instead of just relying on simple linear relationships. Figure 6 shows the pair plot, which summarises the distributions and pairwise relationships between wt%, time and wear depth, giving a clear overview of how the data set is structured. The diagonal plots demonstrate multimodal behaviour in wt% and wear depth, suggesting separate experimental groups and different wear regimes. The rising trends in both the time–wear and wt%–wear scatter plots confirm that longer sliding times and higher reinforcement levels lead to more material loss, which is consistent with the earlier correlation results. The plot shows that the data set is non-linear and clustered, which shows that we require ML models that can understand these complicated interactions.

Figure 7 compares the projected wear depth results for each ML model to the actual wear depth results. This makes it easy to see how accurate their regression

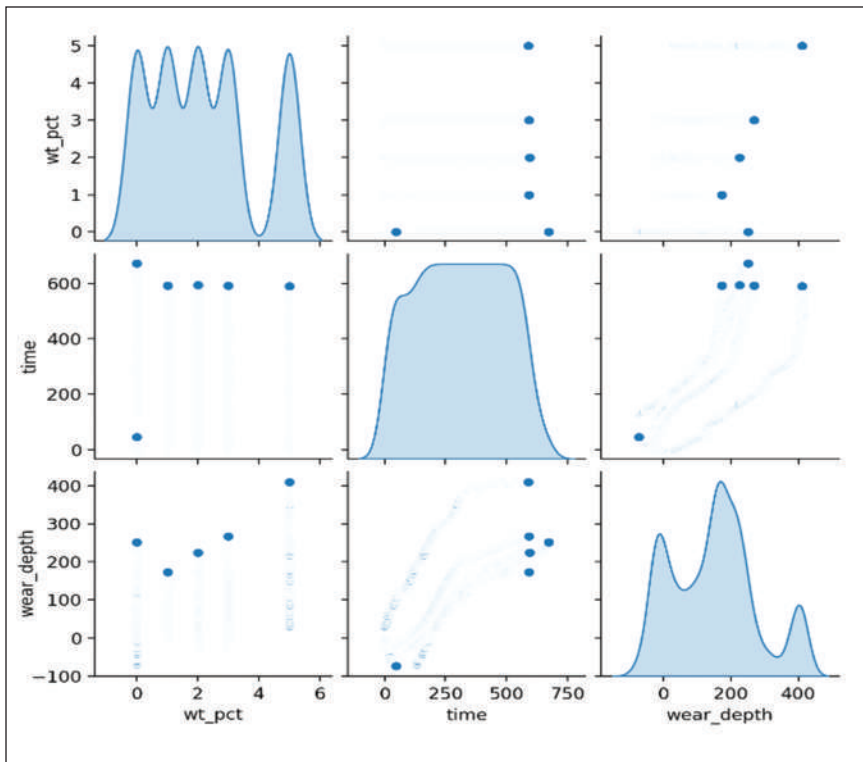


Figure 6. Pair Plot Illustrating the Marginal Distributions and Pairwise Relationships Among wt%, Time and Wear Depth.

is. Both LR and SVR are not very close to the ideal 45° line, which shows how hard it is for them to represent the non-linear relationships between wt%, time, and wear depth. The MLP, on the other hand, shows better alignment, while the RF and XGBoost models are almost perfectly aligned with the reference line ($R^2 = 1.000$), which shows that they are more accurate at making predictions. The almost perfect performance of tree-based ensemble models shows how well they can predict the complicated, non-linear wear behaviour in the data set, which is better than both linear and kernel-based methods. Figure 8 shows the residual distributions of the five ML models. This shows how well they can reduce prediction errors across the wear-depth range. LR, SVR and MLP all exhibit clear patterns and curves in their predictions. The residual dispersion also gets bigger as the wear-depth values increase. This behaviour shows that the model is not fitting well and shows that it cannot accurately predict the non-linear relationships between wt%, time and wear depth. On the other hand, the residuals of RF and XGBoost are tightly grouped around the zero-error line with no clear trend. This shows that they generalise better and have less systematic bias. Of them, RF has the least residual variance and the most even error dispersion, which confirms that it is the most accurate and robust model for forecasting wear depth in this

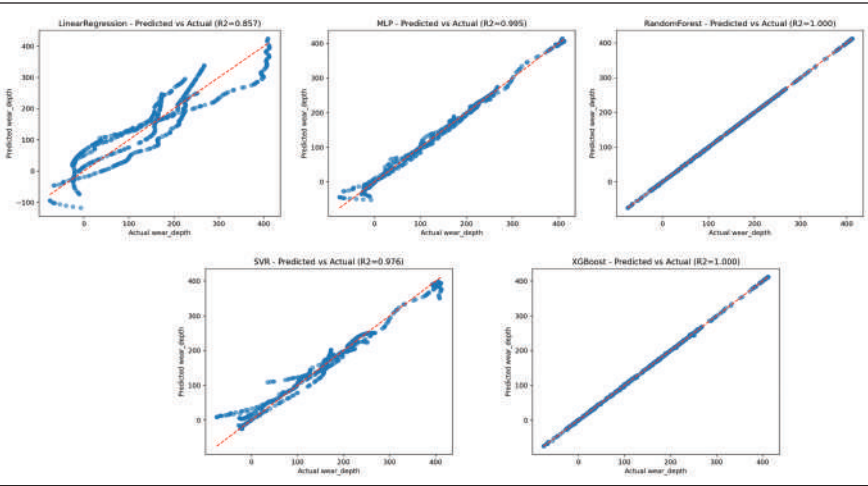


Figure 7. Predicted Versus Actual Wear-depth Plots for All Machine-learning Models.

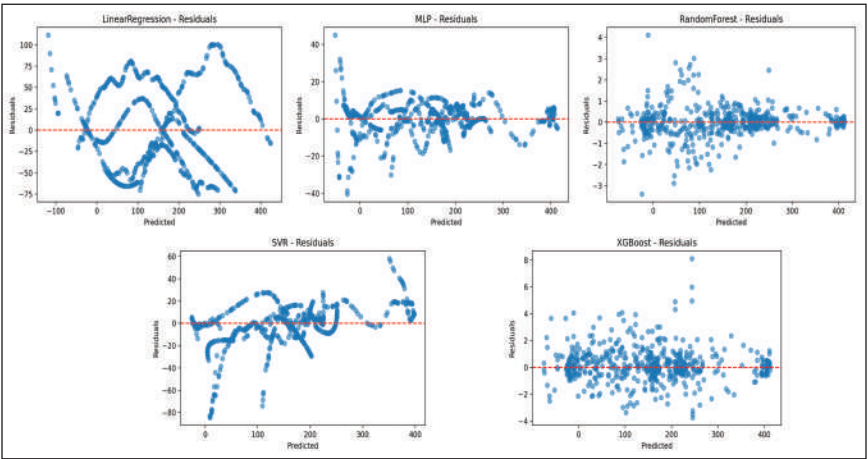


Figure 8. Residual Plots for All Machine-learning Models.

investigation. To further evaluate the robustness and generalisation capability of the developed ML models, a five-fold cross-validation procedure was performed on the complete data set. The results of R^2 , RMSE and MSE demonstrated that the RF model consistently achieved the highest prediction accuracy with the lowest variability among all models. The small standard deviations of the performance metrics indicate that the model performance is stable and not strongly dependent on a particular train–test partition. Similar behaviour was observed for XGBoost, whereas LR and SVR exhibited comparatively larger variations across folds, reflecting their limited ability to capture the non-linear wear behaviour of the

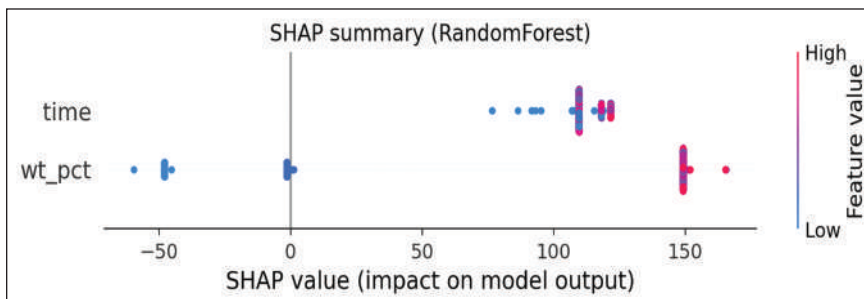


Figure 9. SHAP Summary Plot for the Random Forest Model Showing the Contribution of wt% and Time to Wear-depth Prediction.

Al–GnP–hBN hybrid nanocomposites. The close agreement between the cross-validation results and the hold-out test-set performance further confirms the robustness and generalisability of the proposed ML framework.

The SHAP summary plot for the RF model in Figure 9 shows how each feature helps forecast wear depth. Time has the biggest and most consistently positive SHAP values, which shows that the length of time spent sliding is the main cause of wear. In contrast, wt% has a lesser but still significant effect. For example, larger levels of reinforcement (red points) lead to higher wear predictions in some cases and lower predictions in others. This is because wt% interacts with the wear mechanism in a more complicated and non-linear way. The clear spread and size of the SHAP values show that the RF model does a good job of finding the most important elements that cause wear. This fits with what we know about tribology: longer sliding periods lead to more material loss, and the nature of the reinforcement affects how the material reacts to wear. Furthermore, positive SHAP values associated with higher sliding times indicate an increased contribution to wear-depth prediction, whereas lower sliding times contribute less strongly to material removal. This behaviour is physically consistent with dry sliding wear, where prolonged contact causes progressive surface degradation and deeper wear tracks. The influence of reinforcement wt% is comparatively more complex. The distribution of SHAP values suggests that reinforcement content affects wear through competing mechanisms, including enhanced hardness, improved load-transfer capability and the lubricating action of GnP and hBN. However, higher reinforcement contents may also promote particle agglomeration and local microstructural heterogeneity, which can alter wear behaviour. Consequently, both positive and negative SHAP contributions are observed for wt%, reflecting its non-linear interaction with the wear process. These observations confirm that sliding time is the primary governing parameter, while reinforcement content acts as a secondary factor modifying the overall wear response.

Conclusion

The present work applied an ML framework to analyse and predict the wear behaviour of aluminium hybrid composites reinforced with GnP and hBN, using

wt% and sliding time as input variables. Based on the observations made, the following conclusions were drawn:

- The comparative evaluation of five ML models shows that ensemble-based methods outperform linear and kernel approaches in modelling the non-linear wear behaviour of aluminium hybrid composites.
- RF emerged as the most robust and accurate predictor across all performance metrics, demonstrating strong generalisation and minimal error dispersion throughout the data set.
- LR, SVR and MLP exhibited higher residual variability, indicating limited capability in capturing complex interactions between reinforcement content and wear progression.
- SHAP explainability revealed that sliding time is the dominant factor governing wear depth, whereas reinforcement wt% plays a secondary but still significant role in shaping the overall response.
- The combined use of predictive modelling, residual analysis, radar comparison and SHAP interpretation offers a reliable and transparent workflow for tribology-focused ML studies.
- The findings confirm that ML-driven analysis provides a fast, cost-effective and scalable route for understanding and optimising wear behaviour in aluminium hybrid composites for advanced engineering applications.

Declaration of Conflicting Interests

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Advances and Prospects of Biodegradable Magnesium-based Metal Matrix Composites for Biomedical Implants: A Comprehensive Review

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Abstract

Magnesium-based biodegradable composites offer significant advantages over nondegradable materials for biomedical implants. These materials degrade gradually in physiological environments, thereby reducing stress shielding and eliminating the need for secondary implant removal surgeries. This review explores how alloying elements (e.g., Zn, Ca, Sr) in the magnesium matrix and reinforcing phases (e.g., hydroxyapatite, SiC, TiC) in metal matrix composites influence degradation kinetics, mechanical integrity, and ion release behaviour. Proper regulation of the degradation rate is critical for maintaining mechanical support during the healing phase while ensuring biocompatibility. This study expands the use of magnesium composites in implantable medical devices. Some examples are orthopaedic implants, circulatory stents, bone fixation devices, and soft tissue scaffolds. The above uses demonstrate the ability of magnesium composites to improve patient outcomes and reduce implant-related procedures through gradual breakdown and tissue regeneration. The review also reports the biodegradation control, biocompatibility, manufacturing scale-up, and regulatory requirements for clinical translation. Successful commercialisation depends on balancing the degradation rate with mechanical support, limiting cytotoxicity, and meeting regulatory standards. In conclusion, magnesium-based biodegradable composites have the potential to revolutionise biomedical implants. The unique combination of mechanical properties, biocompatibility, and regulated biodegradation improves

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patient outcomes and therapy efficacy. Medical device design and regenerative medicine will benefit from magnesium composites as researchers improve synthesis methods, material properties, and overcome barriers.

Keywords

Biodegradable, alloying elements, reinforcement, osteogenesis, corrosion, cytotoxicity

Introduction

Biodegradable magnesium-based metal matrix composites (Mg-MMCs) represent a paradigm shift in temporary implant design for bone fractures, bone defects, and cardiovascular applications, addressing key limitations of traditional permanent implants such as stress shielding, chronic inflammation, and the need for revision surgeries (Chandra & Pandey, 2020; Kim et al., 2020; Tsakiris et al., 2021; Zhang et al., 2021). Unlike nondegradable metals (e.g., titanium and stainless steel), Mg-MMCs degrade progressively in physiological environments, ideally matching the rate of tissue healing while providing temporary mechanical support (Choudhary & Singh Raman, 2015; Fu et al., 2011; Seetharaman et al., 2023; Sheikh et al., 2015). This review critically synthesises recent advances in synthesis, reinforcement strategies, degradation control, and clinical translation of Mg-MMCs (Bairagi & Mandal, 2022; Bommala et al., 2019; Haghshenas, 2017; Zhao et al., 2017).

Temporary implants are widely used in bone fixation, tissue scaffolding, and stent implantation. Orthopaedic implants play a critical role in treating bone fractures and defects by providing temporary support and stability during the healing process. The bone plate is the most common internal fixation device, secured to bone fragments with screws (Kim et al., 2020). Magnesium alloys and Mg-MMCs are highly attractive for biomedical applications due to their low density, high specific strength, and excellent biocompatibility, making them suitable for use in aerospace, automotive, and medical fields (Bairagi & Mandal, 2022; Choudhary & Singh Raman, 2015; Zhao et al., 2017).

The capacity of biodegradable materials to undergo slow degradation and subsequent resorption by the human body is in accordance with the inherent tissue repair mechanism, hence facilitating enhanced integration and remodelling of the surrounding tissues. Large bone defect repair and regeneration are still significant clinical issues, and bioactive glass scaffolds are emerging as a promising solution. Due to their lack of mechanical strength and fracture toughness, these scaffolds are often not suitable for use in load-bearing bone defects. Recent advancements in both the manufacturing process and the mechanical qualities of bioactive glass scaffolds have demonstrated that the mechanical strength of these scaffolds is not a significant barrier to their use in the field of bone repair. Using a number of methods, researchers have created a scaffold with compressive strengths on par with trabecular and cortical bone. In spite of these advancements, the literature still highlights limitations of current bioactive glass-based scaffolds. These drawbacks include the low fracture toughness of these scaffolds (Fu et al., 2011; Sheikh et al., 2015). Because the use of current medical implants made with biodegradable biomaterials

removes the need for eventual surgical removal, this aspect of their design and use is of the utmost importance. This not only promotes patient comfort but also mitigates the potential risks of problems (Seetharaman et al., 2023).

Magnesium possesses favourable characteristics that render it a compelling contender for biomedical applications, primarily owing to its biocompatible nature and its likeness to the composition of real bone material. Magnesium implants have these qualities, which aid in their natural integration with the body. The usage of temporary orthopaedic implants made from magnesium-based alloys has gained a lot of attention in recent years due to the biocompatibility, biodegradability, and outstanding mechanical properties of these materials. However, because magnesium alloys corrode at such a high rate in physiological conditions, their clinical application in load-bearing components is limited (Bairagi & Mandal, 2022; Zhao et al., 2017). Magnesium-based composites provide various benefits in biomedical applications. They are biocompatible, which implies that they are well-tolerated by the body and do not cause any negative reactions. Furthermore, these biodegradable materials gradually decompose and assimilate into the body with time. These composites have advantageous mechanical qualities, such as high strength and good ductility, which make them suitable for load-bearing applications. Techniques for altering the surfaces of the composites may increase their resistance to corrosion. In addition, magnesium-based composites may have tailored qualities that allow for application optimisation in response to specific needs. To summarize, these advantages make magnesium-based composites a hopeful prospect for multiple biomedical applications. However, degradation rate of pure magnesium is often considered insufficient for specific applications, and it may not meet the desired level of mechanical strength (Bommala et al., 2019; Haghshenas, 2017). The manipulation of the rate of degradation and enhancement of mechanical characteristics can be achieved by integrating other materials, such as ceramics or polymers, into the magnesium matrix. Biodegradable Mg-MMCs can have their degradation rate regulated and their mechanical qualities improved by including ceramics or polymers in the magnesium matrix. Incorporating hydroxyapatite (HA) particles into a magnesium alloy matrix, for instance, enables one to regulate both the rate of corrosion and the mechanical strength of a composite material. Assimilation of reinforcement materials such as HA, tricalcium phosphate (TCP), SiC, Al₂O₃, TiC, and biodegradable polymers (e.g., polycaprolactone or poly(lactic-co-glycolic acid) [PLGA]) into the magnesium matrix presents a potentially effective technique for regulating the degradation rate and enhancing the mechanical properties of Mg-MMCs (Bommala et al., 2019; Cerqueira et al., 2021; Khorashadizade et al., 2021; Krishnan et al., 2022; Kuśnierczyk & Basista, 2017; Mohanasundaram et al., 2023; Nie et al., 2021; Nirala et al., 2021; Ratna Sunil et al., 2014; Su et al., 2020). For instance, bioactive ceramics such as HA and TCP improve osseointegration and the implant–bone interface, biodegradable polymers (e.g., PCL/PLGA) help regulate corrosion and ion release, and ceramic particles such as SiC and TiC enhance strength, stiffness, and wear resistance. This approach allows tailoring of composites for specific biomedical implant requirements, including orthopaedic fixation devices and cardiovascular stents.

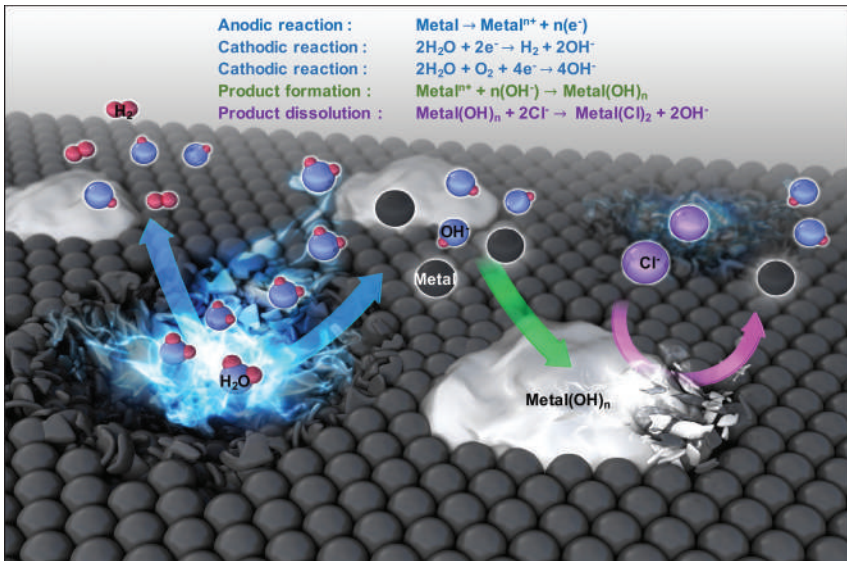


Figure 1. The Mechanism of Biodegradation of Metals.

Source: Han et al. (2019).

A sequence of anodic (oxidation) and cathodic (reduction) events is responsible for the biodegradation of biodegradable metals that occurs within the body. These are primarily a form of corrosion. Usually, this degradation entails the metal being converted into more stable ions. As seen in Figure 1, oxidation occurs in the metal whenever it comes into touch with body fluids. The cathodic reactions employ the resulting electrons. These reactions, which entail the emission of hydrogen gas and hydroxide ions, result in the formation of a thin layer of metal oxide on the surface of the object, which acts as a protective barrier against further chemical reactions. The presence of large concentrations of chloride ions in human fluids causes the protective oxide layer in magnesium alloys to become weakened, which in turn speeds up the process of degradation. As the process continues, the calcium and phosphate ions that are present in biological fluids, along with the alkalinity of the surrounding environment, cause the accumulation of calcium phosphate on the metal oxide layer. This buildup facilitates the attachment of cells to the interface, leading to the eventual formation of tissue (Han et al., 2019).

Biodegradable Magnesium Alloys

Properties and Characteristics of Pure Magnesium

The key properties and characteristics of pure magnesium as a base material for biodegradable alloys and composites are summarized in Figure 2. Magnesium, with a density of approximately 1.74 g/cm^3 , closely matches that of human

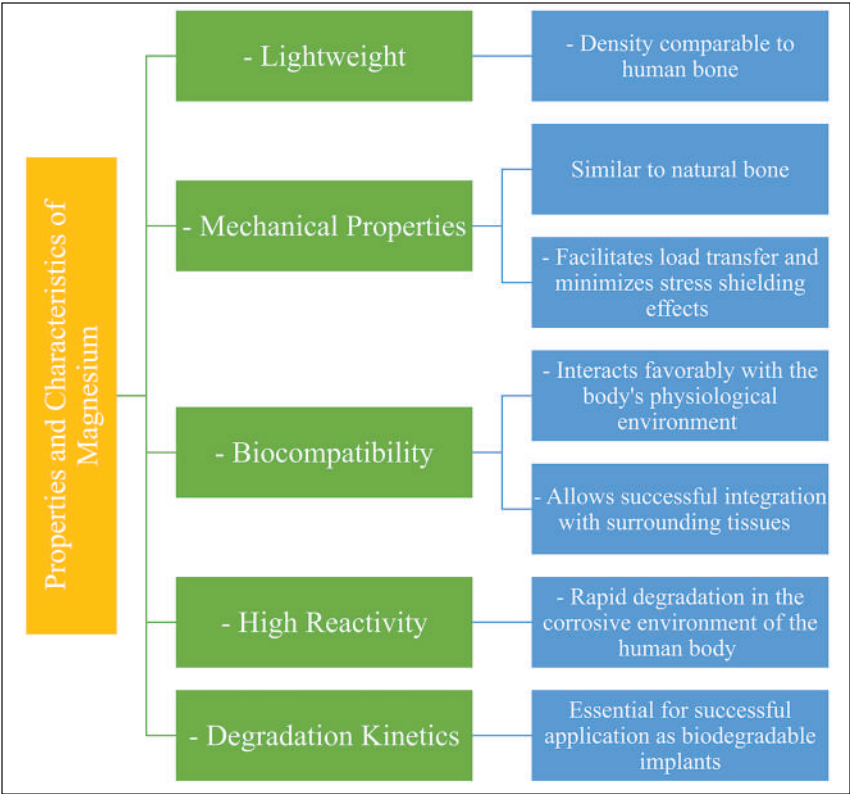


Figure 2. Properties and Characteristics of Pure Magnesium as a Base Material for Biodegradable Alloys and Composites.

Source: Li et al. (2020) and Liu et al. (2018).

cortical bone (1.8–2.1 g/cm³), enabling effective load distribution and minimising stress shielding effects (Li et al., 2020; Liu et al., 2018). Its elastic modulus (40–45 GPa) is also comparable to natural bone, further reducing the risk of bone resorption (Li et al., 2020). Magnesium exhibits excellent biocompatibility and supports tissue integration without eliciting detrimental responses (Ding et al., 2014; Liu et al., 2018). However, pure magnesium undergoes rapid degradation in chloride-rich physiological fluids (pH 7.4–7.6), releasing hydrogen gas and leading to localised pH changes (Agarwal et al., 2016; Persaud-Sharma & McGoron, 2012; Virtanen, 2011; Witte, 2010).

Benefits and Challenges of Pure Magnesium as a Biodegradable Material

Figure 3 outlines the principal benefits and challenges of pure magnesium as a biodegradable implant material and highlights the need for alloying and

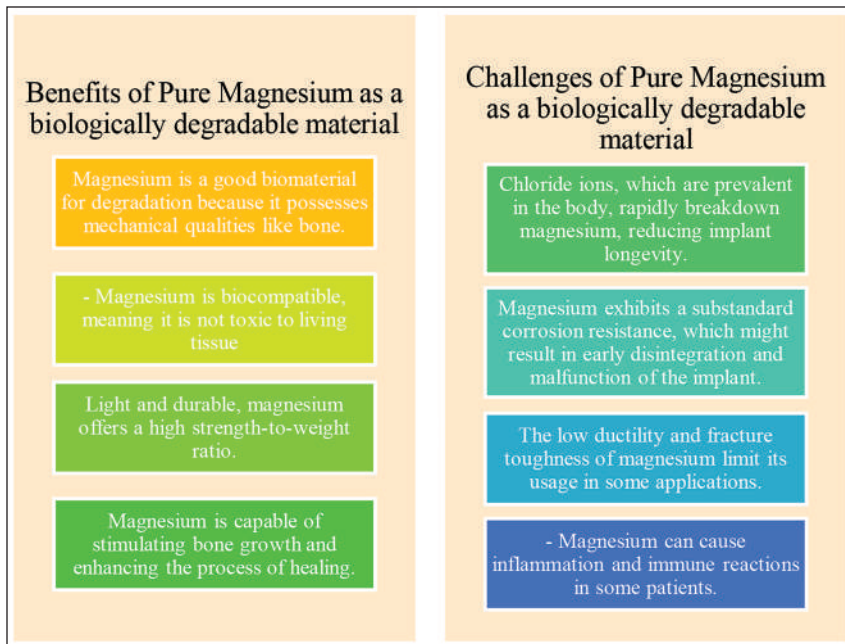


Figure 3. Benefits and Challenges of Pure Magnesium as a Biodegradable Material, Highlighting the Need for Alloying and Reinforcement to Overcome Limitations.

Source: Amukarimi and Mozafari (2021), Chandra and Pandey (2022) and Wang et al. (2022).

reinforcement to overcome its limitations. One notable benefit of pure magnesium lies in its capacity to undergo slow degradation over time, aligning with the healing pace of surrounding tissue (Sezer et al., 2021). This process eliminates the need for secondary surgery to remove the implant, thereby reducing patient discomfort and associated complications (Liu et al., 2018). Moreover, the inherent degradation of magnesium facilitates bone remodelling and tissue regeneration, leading to improved long-term outcomes (Shan et al., 2022).

However, the rapid degradation of pure magnesium poses significant challenges for many applications, necessitating alloying and composite reinforcement strategies to control degradation rates and enhance mechanical properties (Amukarimi & Mozafari, 2021; Chandra & Pandey, 2022; Wang et al., 2022). Localised release of magnesium ions may also influence cellular responses and potentially cause cytotoxicity or inflammation if not properly managed (Abdalla et al., 2020; Ben Amara et al., 2023; Venkateswarlu et al., 2024; Zhang et al., 2009).

The Effect of Alloying on Deterioration

The addition of alloying elements such as zinc (Zn), calcium (Ca), strontium (Sr), and manganese (Mn) to magnesium significantly improves both corrosion resistance (stability) and mechanical strength, making magnesium alloys and Mg-MMCs

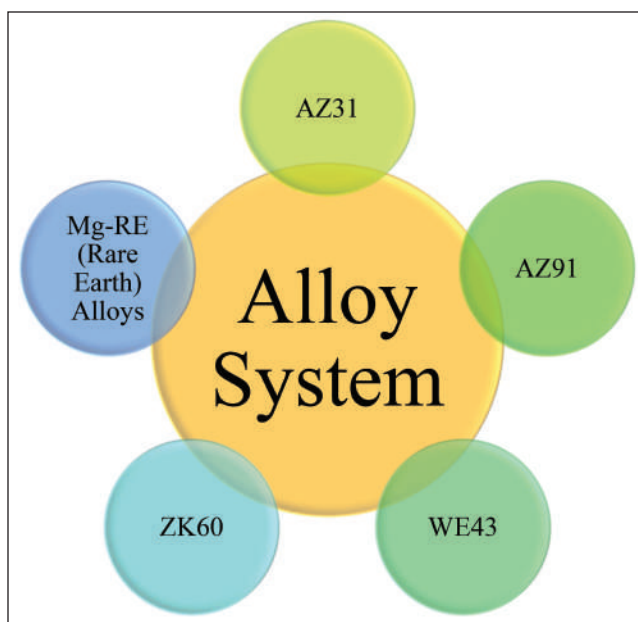


Figure 4. Major Magnesium Alloy Systems Commonly Investigated for Biomedical Applications.

suitable for load-bearing biomedical applications that require structural integrity during the healing period (Aikin et al., 2025; Chen et al., 2019; Kumar & Katyal, 2022; Li et al., 2021; Sharma et al., 2022; Tian & Liu, 2015). For example, Zn and Ca additions refine grain structure, enhance tensile and compressive strength, and slow degradation rates, while Sr at about 3 wt % further refines grains and supports bioactivity, thereby providing adequate mechanical support while minimising premature failure. Careful selection of alloying elements is essential, as some may introduce cytotoxicity concerns or alter degradation behaviour unfavorably. Therefore, a balanced approach is required to optimize these materials for specific implant applications such as bone fixation devices and cardiovascular stents. The major magnesium alloy systems commonly investigated for biomedical applications are illustrated in Figure 4. The influence of common alloying elements on the mechanical properties of biodegradable magnesium alloys and magnesium-based metal matrix composites is summarized in Table 1.

Magnesium-based Metal Matrix Composite Synthesis and Reinforcement Materials

The production of Mg-MMCs can be accomplished through various processing routes. These methods are generally classified into liquid-phase, solid-phase, in situ, deposition, and liquid-solid two-phase techniques (Dash et al., 2018). These metallurgical processing routes are schematically illustrated in Figure 8. Among these, powder metallurgy, friction stir processing, and in situ synthesis are the

Table 1. Effect of Common Alloying Elements on the Mechanical Properties of Biodegradable Magnesium Alloys and Magnesium-based Metal Matrix Composites.

Sl. No.	Elements	Role in Improving Mechanical Properties
1	Calcium (Ca)	Enhances grain refinement; increases tensile strength and creep resistance; promotes formation of protective intermetallic phases
2	Lithium (Li)	Improves ductility while reducing tensile strength; enhances formability in Mg-Li alloys
3	Zinc (Zn)	Strengthens the composite with optimal enhancement up to 5 wt %; improves tensile and compressive strength
4	Strontium (Sr)	Improves strength and refines grain size at ~3 wt % addition; enhances bioactivity
5	Manganese (Mn)	Improves grain fineness and tensile strength
6	Tin (Sn)	Improves both tensile and compressive strength
7	Zirconium (Zr)	Provides high damping capacity, tensile strength, and ductility.
8	Aluminium (Al)	Increases hardness and tensile strength (commonly used in AZ series)
9	Titanium (Ti)	Improves elongation
10	Copper (Cu)	Reduces ductility
11	Silicon (Si)	Enhances fluidity during casting
12	Iron (Fe)	Not suitable for corrosion resistance; generally avoided
13	Neodymium (Nd)	Enhances strength (rare earth element)
14	Molybdenum (Mo)	Increases hardness, Young's modulus, and elongation
15	Cerium (Ce)	Reduces yield strength

Source: Agarwal et al. (2016), Aikin et al. (2025), Chen et al. (2019), Kumar and Katyal (2022), Li et al. (2021), and Sharma et al. (2022).

most commonly used for biomedical Mg-MMCs due to their ability to provide good microstructural control and interfacial bonding.

Powder Metallurgy Techniques

Powder metallurgy is a widely used solid-state method for producing Mg-MMCs. It involves blending magnesium powder with reinforcements (e.g., TiB₂, SiC, HA), followed by compaction and sintering. This technique enables uniform distribution of reinforcements and minimizes contamination (Anbuechziyan et al., 2015; Dinesh Kumar et al., 2020; Jayasathyakawin et al., 2020; Jiang et al., 2005; Liu et al., 1994; Purohit et al., 2018; Saxena et al., 2023; Wang et al., 2004).

Figure 5 shows scanning electron micrographs of TiB₂/Mg composites. Minor microporosity increases with higher reinforcement volume fractions, with the 20 vol % composite showing the lowest porosity under optimized conditions (Dinesh Kumar et al., 2020).

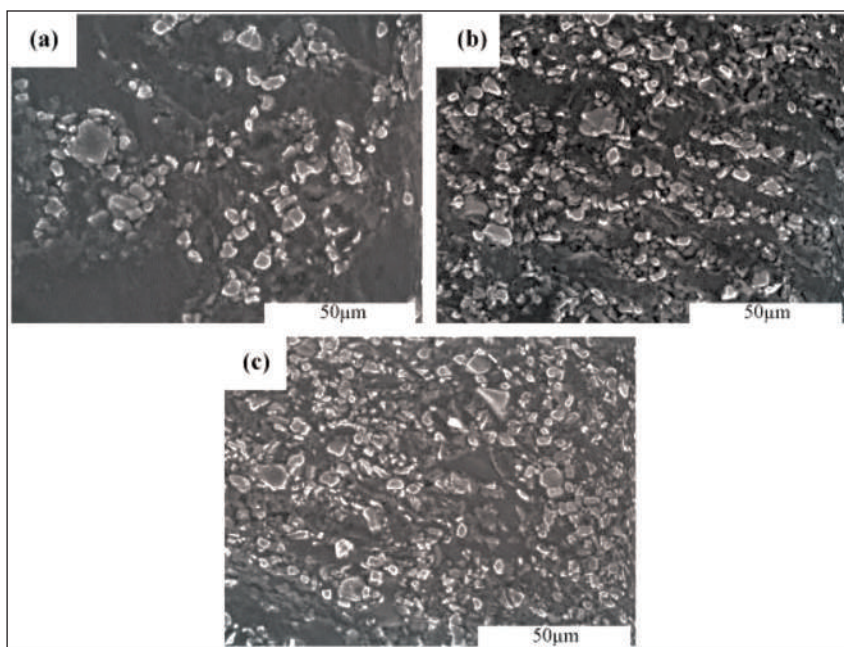


Figure 5. Scanning Electron Micrographs of Powder Metallurgy-fabricated TiB₂/Mg Composites at: (a) 10, (b) 20, and (c) 30 vol % TiB₂.

Source: Wang et al. (2004).

Friction Stir Processing Techniques

Friction stir processing is a solid-state technique that offers superior microstructural uniformity and strong interfacial bonding without melting the matrix. The friction stir processing technique is cost-effective and environmentally sustainable because it eliminates the need for supplementary binders or consumable materials, which reduces waste generation and energy usage. The lack of molten material throughout the processing phase also mitigates the potential for flaws or inclusions in the ultimate composite. These benefits make friction stir processing an attractive choice for producing magnesium composites with microstructural uniformity for biological and engineering applications. Additional research is still needed to optimize processing settings and to understand how different reinforcements affect friction-stir-processed magnesium composites for biomedical Mg-MMCs (Arokiasamy & Anand Ronald, 2018; Bajakke et al., 2019; Balakrishnan et al., 2015; Dinaharan & Akinlabi, 2018; Kumar et al., 2021; Kundurti et al., 2022; Qiao et al., 2021; Ratna Sunil et al., 2014; Sunil et al., 2016; Vedabouriswaran & Aravindan, 2018).

In Situ Fabrication Methods

In situ synthesis involves direct formation of reinforcements during processing, leading to excellent interfacial bonding and improved corrosion resistance. These

methods produce composites with excellent interfacial bonding between the magnesium matrix and the reinforcement phase, including interpenetrating networks formed by reactive infiltration. One common in situ technique is in situ powder metallurgy, where precursor powders of magnesium and reinforcement materials are combined and then processed under controlled conditions to create the composite. Another approach is the in situ solidification method, where the reinforcement materials are introduced to the molten magnesium alloy, and the composite is formed upon cooling. Because in situ fabrication procedures lead to greater bonding between the phases, corrosion resistance and mechanical properties are both improved. However, precise control of the in situ process and potential challenges associated with the inhomogeneous distribution of reinforcement materials require careful consideration during fabrication (Chen et al., 2005; Dong et al., 2004; Meher et al., 2021; Shakil et al., 2020; Shamekh et al., 2012; Wang et al., 2003; Zhou et al., 2022).

Figure 6 shows the scanning electron micrographs of in situ synthesized Mg matrix composites via reactive infiltration at different temperatures (Dong et al., 2004). The figures demonstrate that reaction completeness and microstructure compactness depend strongly on processing temperature and reactant particle size. Lower porosity and better TiC formation are achieved at optimal temperatures (e.g., 1,023 K), highlighting the importance of process control for biomedical applications.

A comparison of these methods reveals that powder metallurgy offers good scalability, friction stir processing provides excellent grain refinement, and in situ routes yield superior bonding. Selection depends on the target reinforcement type and desired implant performance.

Reinforcement Materials

Ceramics, polymers, and others: Reinforcement materials are critical for improving the mechanical performance and biodegradation behaviour of composites with a magnesium metal matrix (Nie et al., 2021; Su et al., 2020). Commonly used reinforcement materials include ceramics (e.g., calcium phosphate, HA) and polymers (e.g., polycaprolactone, PLGA) (Bommala et al., 2019; Khorashadizade et al., 2021; Kuśnierczyk & Basista, 2017; Mohanasundaram et al., 2023; Ratna Sunil et al., 2014). Ceramics provide excellent biocompatibility and help promote bone regeneration, while polymers offer better toughness and flexibility. In order to ensure that the materials are suitable for the intended implant applications, biocompatibility, rate of degradation, and mechanical qualities are taken into consideration (Cerqueira et al., 2021; Kim et al., 2020). The integration of reinforcing materials leads to enhanced mechanical characteristics, including wear resistance, increased strength, and modulus, thereby rendering the metal matrix composites (MMCs) acceptable for applications involving load-bearing implants. In spite of this, it is essential to optimize the volume fraction and distribution of reinforcement materials in order to prevent agglomeration and keep the composite's intended attributes (Khorashadizade et al., 2021; Krishnan et al., 2022; Nirala et al., 2021; Ratna Sunil et al., 2014; Su et al., 2020).

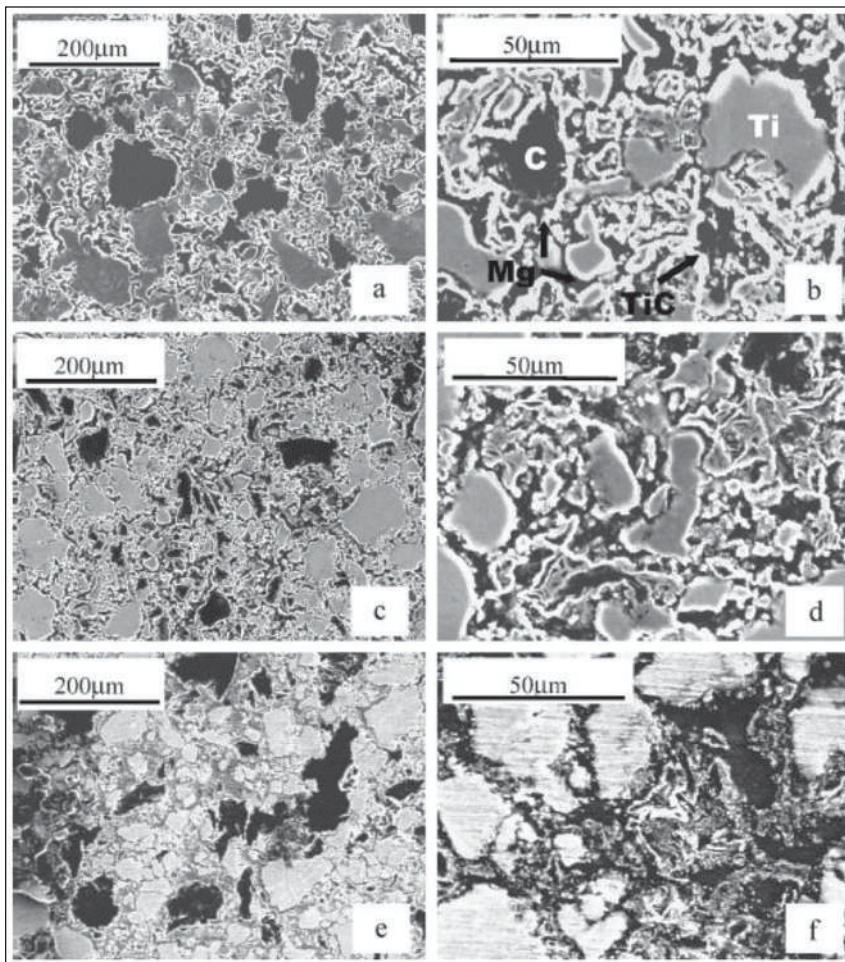


Figure 6. Scanning Electron Micrographs of In Situ Synthesized Mg Matrix Composites Through Reactive Infiltration for System I. Low Magnification: (a) 1,073 K; (c) 1,023 K; (e) 973 K. High Magnification: (b) 1,073 K; (d) 1,023 K; (f) 973 K.

Source: Dong et al. (2004).

Table 2 summarises commonly used reinforcement materials and their effects on mechanical properties, corrosion resistance, and biocompatibility. Dispersed fibres or particles. Reinforcement materials are typically classified into four main categories: continuous fibres, discontinuous fibers, whiskers, and particulates (Figure 7). The choice of reinforcement materials in MMCs depends on the intended composite properties. The way in which the reinforcement and matrix phases interact is essential for optimizing features such as load transfer, wetting behaviour, reactions, and electrical conductivity. MMCs include reinforcement materials such as ceramics (e.g., SiC and Al_2O_3), graphite, CNT, graphene, carbon, diamond, cubic boron

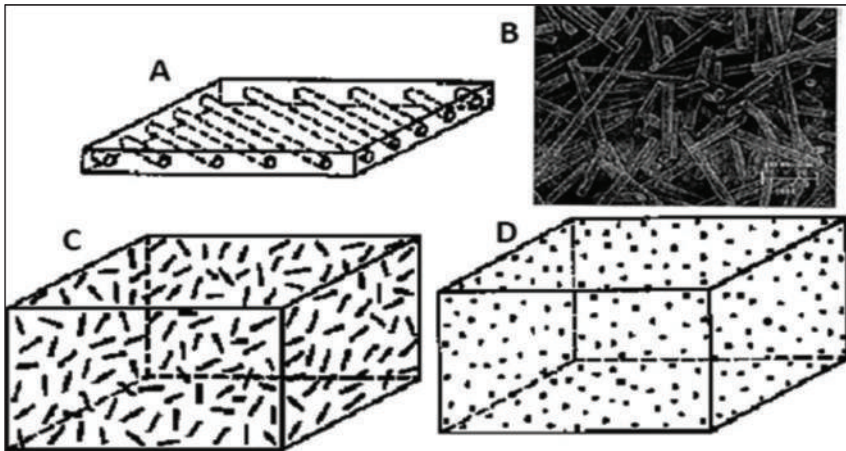


Figure 7. Metal Matrix Composites (MMCs) Categorized Into (a) Continuous Fibres, (b) Discontinuous Fibres, (c) Whiskers, and (d) Particles as Reinforcement.

Source: Kumar and Soren (2020).

nitride, and fly ash. Continuous fibres and whiskers are used as uninterrupted reinforcements, whereas discontinuous fibres and particulates act as fragmented reinforcements. In order to obtain the desired outcomes with MMCs, it is necessary to match and blend the reinforcement material with the matrix material in an effective manner (N. Kumar & Soren, 2020).

Surface Modification Techniques for Enhanced Biocompatibility

Increasing the biocompatibility and bioactivity of Mg-MMCs can be accomplished through the application of surface modification techniques (Delavar et al., 2023). In order to promote cell adhesion and aid osseointegration, the surface of the composite may be modified or coated with bioactive compounds such as HA or biodegradable polymers (Buck et al., 2020). Surface modification has a dual role in improving the initial cellular response and mitigating the release of corrosion by-products, hence minimising potential harmful effects on the surrounding tissues. In addition, the application of surface modification techniques can accomplish effective control of the rate of degradation of composites and make it possible for the regulated release of magnesium ions. This approach serves to mitigate the potential cytotoxic effects associated with the material. Surface modification is frequently performed using techniques such as sol-gel coating, plasma electrolytic oxidation (PEO), plasma spraying, and electrospinning to support controlled ion release and to delay excessive hydrogen gas evolution. Optimizing surface characteristics in magnesium composites is crucial to enhance biocompatibility and ensure the long-term success of implants for biomedical applications (da Conceicao et al., 2010; Daroonparvar et al., 2022; Yang et al., 2011).

Table 2. Reinforcement Materials, Effect on Mechanical Properties, Corrosion and Biocompatibility.

Reinforcement Material	Effect on Mechanical Characteristics	Effect on Corrosion Resistance	Biocompatibility
SiC	Increases the strength as well as the stiffness and resistance to wear	Improves corrosion resistance	Good biocompatibility
Al ₂ O ₃	Increases the strength as well as the stiffness and resistance to wear	Improves corrosion resistance	Good biocompatibility
TiC	Increases the strength as well as the stiffness and resistance to wear	Improves corrosion resistance	Good biocompatibility
BN	Increases the strength as well as the stiffness and resistance to wear	Poor corrosion resistance	Good biocompatibility
ZrO ₂	Increases the strength as well as the stiffness and resistance to wear	Improves corrosion resistance	Good biocompatibility
Hydroxyapatite (HA)	Improves bone-bonding properties	Poor corrosion resistance	Excellent biocompatibility
Tricalcium phosphate (TCP)	Improves bone-bonding properties	Poor corrosion resistance	Excellent biocompatibility

Source: Cerqueira et al. (2021), Khorashadizade et al. (2021), Krishnan et al. (2022), Kuśnierczyk and Basista (2017), Mohanasundaram et al. (2023), Nie et al. (2021), and Su et al. (2020).

Mechanical Performance of Magnesium Composites

Strength in Tension, Strength in Yield, and Ductility

Parameters such as tensile strength, ductility, and yield strength are vital when determining whether magnesium composites are suitable for use as biomedical implants. The mechanical performance of magnesium composites is characterised by parameters such as these. Tensile strength denotes the highest stress that a material can withstand without breaking under tension. The yield strength indicates the stress value at which plastic deformation commences, providing insight into the material's capacity to resist permanent deformation. Ductility describes the degree to which a substance can deform plastically before it fractures. The mechanical properties of magnesium composites are susceptible to being significantly influenced by the reinforcement materials used. It is crucial to keep in mind that the addition of ceramics or polymers, for example, has the potential to raise both the tensile strength and the yield strength of the material; nevertheless, it is important to keep in mind that this may come at the expense of decreased ductility in the final product. The attainment of a harmonious equilibrium among these characteristics is of utmost importance in order to guarantee the composite's

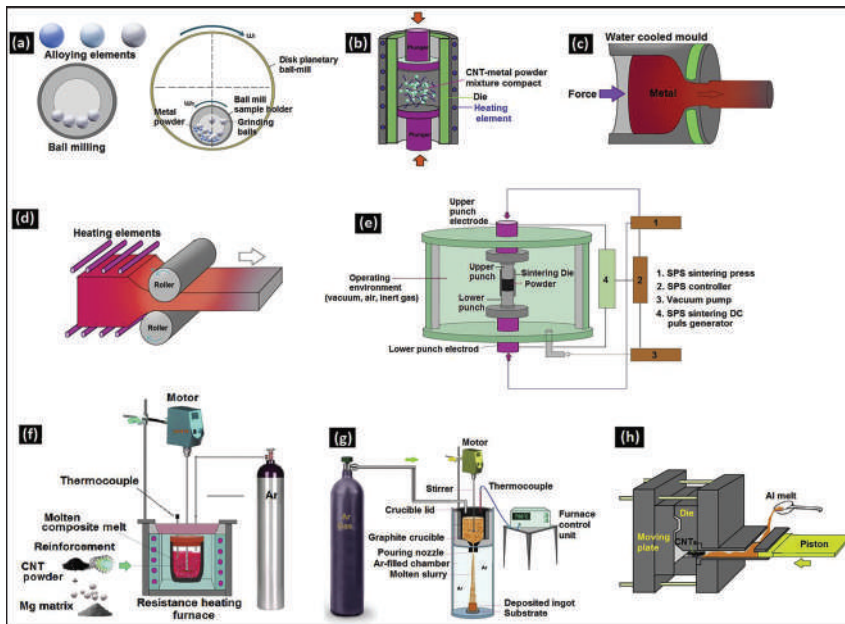


Figure 8. Various Metallurgical Techniques for Producing Composites: (a) Powder Metallurgy, (b) Hot Press Sintering, (c) Hot Extrusion, (d) Hot Rolling, (e) Spark Plasma Sintering (SPS), (f) Stir Casting (SC), (g) Disintegrated Melt Deposition (DMD), and (h) High-pressure Die Casting.

Source: Khorashadizade et al. (2021).

ability to endure mechanical stresses without untimely breakdown, while simultaneously permitting sufficient flexibility to accommodate physiological motions (Haghshenas, 2017; Khorashadizade et al., 2021; Krishnan et al., 2022; Li et al., 2014; Venugopal et al., 2023; Zhang et al., 2009).

Figure 9 illustrates the tensile strength and elongation of Mg–Zn–Mn alloys in their respective as-cast and extruded forms. When looking at the tensile strengths of the material, such as the yield strength and ultimate tensile strength (UTS), Figure 9(a) demonstrates that the tensile strength (both yield strength and ultimate tensile strength) of Mg–Zn–Mn alloys increases with increasing Zn content. The extrusion process results in a substantial enhancement in tensile properties compared to the as-cast condition. For instance, the yield strength of the Mg–1Zn–1Mn alloy increased from 44 MPa (as-cast) to 246 MPa after extrusion (a 459% improvement), while the ultimate tensile strength rose from 174 MPa to 280 MPa (a 60% improvement) (Zhang et al., 2009).

Figure 9(b) shows the elongation behaviour of Mg–Zn–Mn alloys. Although increasing Zn content slightly improves elongation in the as-cast alloys, the elongation of extruded alloys decreases significantly at higher Zn levels. For the Mg–Zn–Mn alloy, the elongation drops to approximately 10% after extrusion, which is lower than that of the corresponding as-cast alloy (Zhang et al., 2009).

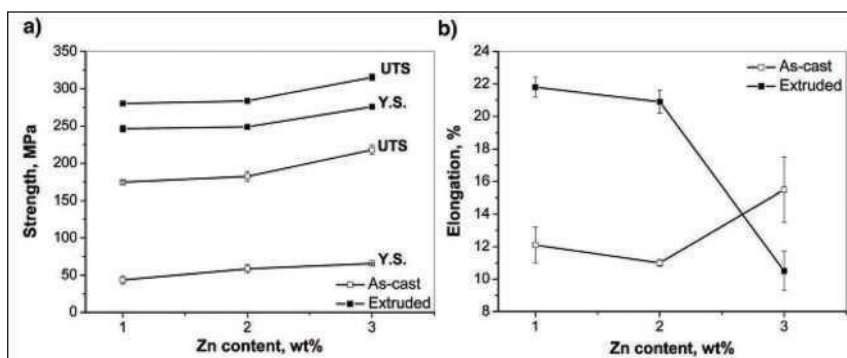


Figure 9. Mechanical Characteristics of Cast and Extruded Materials Mg–Zn–Mn Alloy. (a) Tensile Strengths and (b) Elongation.

Source: Zhang et al. (2009).

This trade-off between strength and ductility must be carefully considered when designing load-bearing biomedical implants.

Hardness and Wear Resistance

Magnesium composites' mechanical properties, particularly their hardness and wear resistance, are critical in determining the longevity and effectiveness of these materials when applied as biomedical implants. The term “hardness” pertains to the capacity of a material to resist deformation and penetration, whereas “wear resistance” denotes its ability to endure abrasion and surface degradation caused by frictional forces. The inclusion of reinforcement materials, such as ceramics, has the prospective to greatly improve hardness and wear resistance, hence rendering the composite more suited for applications that bear loads. Nevertheless, the utilization of an implant that is too rigid might result in heightened abrasion on surfaces that come into contact with it. This necessitates the need for a meticulous equilibrium to be established, in order to attain ideal performance in terms of wear, while simultaneously avoiding excessive wear on natural tissues. To establish which applications are best suited for composites and to assure the continuous success of implants, it is critically necessary to have a full understanding of the hardness and wear parameters of composites (Alahelisten et al., 1993; Madhusudhan Reddy et al., 2013; Soorya Prakash et al., 2016; Wang et al., 2004, 2006).

Figure 10a illustrates how the Vickers hardness varies with depth in the graded coatings. It demonstrates a steady drop in average hardness from the top layer (856.5 HV) to the substrate (67.8 HV) over the course of the coating. Because WC particle doping had good effects on the Ni–WC composite coatings, the top layer's hardness was noticeably approximately 12.6 times larger than that of the substrate. This doping increases hardness by dispersing and strengthening the fine grain. Figure 10b illustrates the potentiodynamic polarisation curves for both the coating and the substrate, respectively. The results show that the coating has a

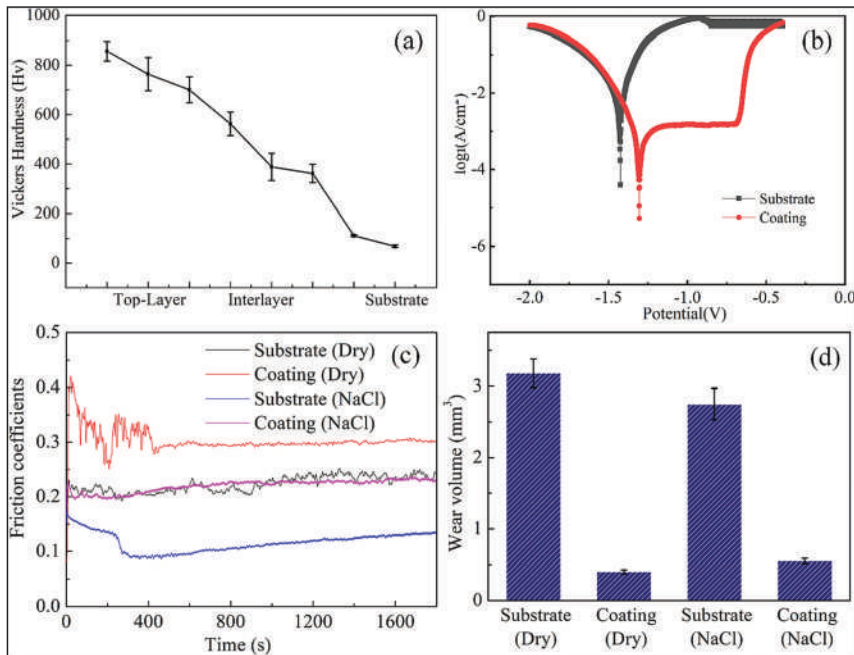


Figure 10. (a) Changes in Vickers Hardness Across the Cross-sectional Depth, (b) a Polarisation Curve, (c) Standard Curves Showing Coefficients of Friction (COF), and (d) Wear Volume Data.

Source: Wang et al. (2023).

higher corrosion potential and lower corrosion current density than the substrate. This indicates improved corrosion resistance. Figure 10c and 10d illustrates the coefficient of friction (COF) and wear behaviour, respectively. The coatings had a higher COF than the Mg alloy because of the rough surface of the coating. The top layer's matrix was worn away first, leaving robust WC particles on the surface during wear testing. This resulted in a rough coating surface and an elevated COF. Despite that, the coating showed a substantial decrease of 87.4% and 80.0% in wear volumes under dry and NaCl circumstances, showing about five to eight times higher wear resistance compared to the Mg alloy (Wang et al., 2023).

Behaviour Caused by Fatigue and Breaking Toughness

The investigation of fatigue characteristics and fracture toughness is of utmost importance when considering the mechanical dependability and safety of magnesium composites in dynamic load-bearing circumstances. The term “fatigue behaviour” refers to the way a composite material behaves when subjected to the influence of cyclic loading and unloading, which has the potential to cause the material to fail due to fatigue over the course of some amount of time. The inclusion of reinforcement materials has the potential to improve the fatigue resistance

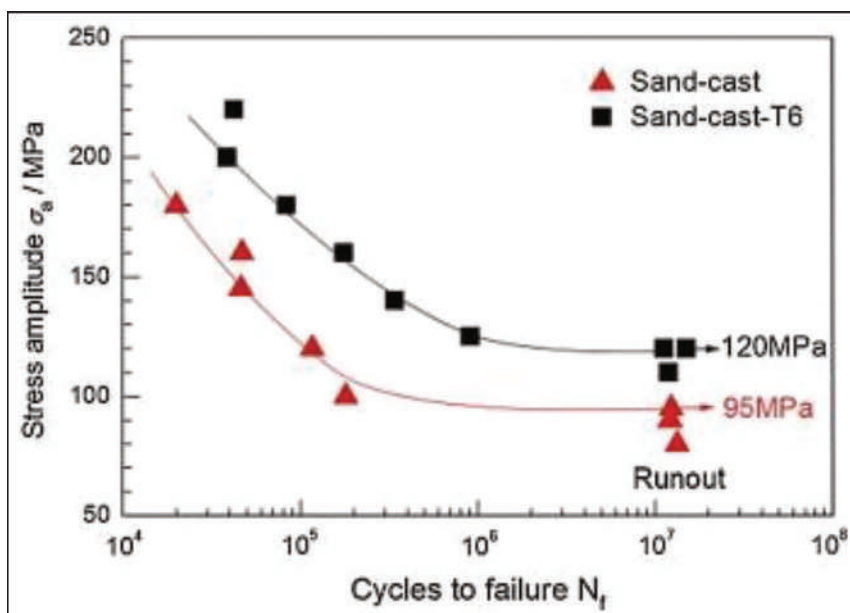


Figure 11. S–N Curves Representing the Fatigue Life Response of Both Sand-cast and Sand-cast-T6 Mg–10Gd–3Y–0.5Zr alloys.

Source: Liu et al. (2014).

of a material by increasing the material's resistance to cracking and reducing the amount of stress concentrated in certain areas of the material. This enhancement is possible as a result of the addition of materials that provide reinforcement to the structure. The fracture toughness parameter quantifies the material's capacity to resist the propagation of cracks, hence reflecting its capability to endure abrupt impacts or stress concentrations without experiencing catastrophic collapse. A composite material possessing a high fracture toughness exhibits enhanced resistance to unanticipated mechanical stresses encountered during the utilization of implants. The comprehension of fatigue characteristics and fracture toughness of magnesium composites enables researchers and engineers to develop implants capable of withstanding physiological loads for prolonged durations while simultaneously reducing the likelihood of abrupt failure and related consequences. The accurate evaluation and optimisation of the mechanical characteristics of magnesium composites are crucial in guaranteeing the sustained performance and dependability of these materials in biomedical implant applications (García-Romero et al., 2010; Hack et al., 1984; Qi et al., 2017; Shin & Bae, 2018; Vaidya & Lewandowski, 1996; Xia et al., 2020; Zhu & Iizuka, 2003).

The S–N curves for sand-cast and sand-cast-T6 Mg–10Gd–3Y–0.5Zr alloys that were subjected to rotating beam loading (with a stress ratio of -1) at about 100 Hz in an air environment are displayed in Figure 11. These alloys were loaded with a stress ratio of -1 , and the frequency of the rotating beam loading was approximately 100 Hz. The resistance to fatigue of these alloys is under

examination. The fatigue strength of the sand-cast Mg–10Gd–3Y–0.5Zr alloy was found to be 95 MPa after 10^7 cycles, but the sand-cast-T6 Mg–10Gd–3Y–0.5Zr alloy demonstrated a fatigue strength of 120 MPa for the same cycle count. This was established after comparing the two alloys' fatigue strengths. The application of the T6 thermal treatment process resulted in a 26% increase in the end product's fatigue strength (Liu et al., 2014).

Corrosion Behaviour of Magnesium-based Composites

Magnesium Degradation Mechanisms in Physiological Environments

The comprehension of the corrosion characteristics exhibited by magnesium-based composites is of utmost importance in order to effectively utilize them as biodegradable materials for biomedical implants. Magnesium degrades through electrochemical mechanisms upon exposure to the physiological environment. This degradation is mostly attributed to the relationship between magnesium and water, as well as the presence of ions in bodily fluids. One of the stages of deterioration is the dissolution of magnesium, which leads to the creation of magnesium ions and ultimately ends in the emission of hydrogen gas. The aforementioned procedure has the capability to induce localised alterations in pH levels, which may result in either alkalisation or acidification in the immediate area of the implant. These changes have the capacity to impact the surrounding tissues and cells. In addition, the liberation of magnesium ions could potentially exert physiological influences on cellular mechanisms and tissue reactions. Several factors, including alloy composition, microstructure, surface area, and exposure circumstances, contribute to the deterioration rate. Understanding these degradation mechanisms comprehensively is extremely important to accurately forecast the performance of the material and customise its degradation rate in line with the healing process for specific biomedical implant applications (Bakhsheshi-Rad et al., 2014, 2016; Eliezer & Witte, 2010; Neves

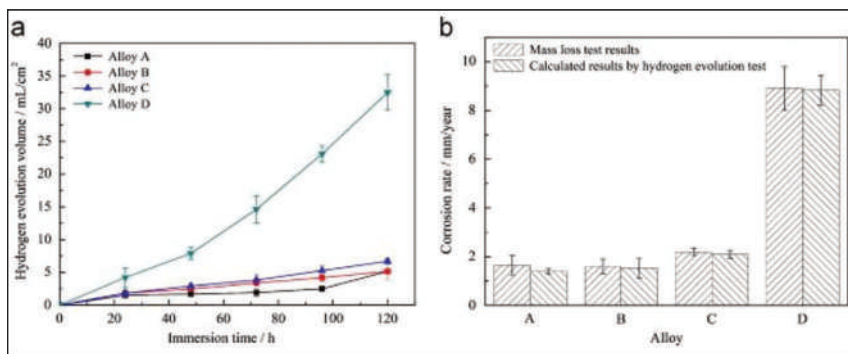


Figure 12. Corrosion Performance of Alloys with Mg–3.0Nd–0.2Zn–xAg–0.4Zr Alloys; (a) Hydrogen Evolution Curves and (b) Corrosion Rate.

Source: Zhang et al. (2013).

et al., 2021; Pulido-González et al., 2020; Razavi et al., 2010; Törne et al., 2017; Xu et al., 2022; Zhang et al., 2013).

The results of studies on hydrogen evolution and mass loss are depicted in Figure 12, which includes hydrogen evolution curves (Figure 12a) and corrosion rates of alloys (Figure 12b). These results reveal distinct corrosion behaviours. After 120 h of immersion, Alloy A's hydrogen evolution volume aligns with Alloy B's, while Alloy C displays elevated hydrogen evolution, and Alloy D's mean volume surpasses those of Alloys A and B by sixfold. The results of mass loss testing indicate that Alloy B has a slightly lower corrosion rate than Alloy A. However, Alloy C and Alloy D have corrosion rates that are 32% and 437% higher, respectively, than Alloy A. Despite the fact that the rates acquired using this approach are slightly lower than those obtained using the mass loss tests, the findings of the hydrogen evolution tests support these findings and reveal a trend that is comparable. This is the case despite the fact that the rates obtained using this method are lower than those obtained using the mass loss tests. This is due to the fact that during the process of simulated body fluids (SBF) renewal, there is a chance that hydrogen may be released into the air and then dissolved into SBF. Remarkably, Figure 12 showcases a direct relationship between Ag content and corrosion rate increase, notably with Alloy B (0.2 wt% Ag) mirroring the rate of Ag-free alloy. These corrosion behaviours are influenced by grain size, second-phase characteristics, distribution, and the presence of the MgZr compound, with finer grains generally improving corrosion resistance while discontinuous second phases enhance microgalvanic-driven corrosion effects (Zhang et al., 2013).

Corrosion Protection Strategies

The topics of interest include alloying, coatings, and surface treatments. In order to mitigate the issues arising from the quick deterioration of pure magnesium, many solutions for corrosion protection are implemented in magnesium-based composites. Alloying is a frequently used technique that involves introducing specific elements into the magnesium matrix to modify its corrosion characteristics and regulate the rate of degradation (Abdalla et al., 2020; Heimann, 2021). The addition of alloying metals such as aluminium, calcium, or zinc has the potential to generate protective corrosion layers or compounds. This, in turn, can effectively decrease the rate at which magnesium ions are released and enhance the overall resistance to corrosion (Agarwal et al., 2016; Aikin et al., 2025; Delavar et al., 2023; Virtanen, 2011). A further approach entails the application of protective coatings or surface treatments to the composite material. Coatings composed of bioceramic materials, such as HA, or biodegradable polymers have the ability to establish a protective barrier between the composite material and the surrounding physiological environment. This barrier serves to decelerate the pace of degradation and improve the biocompatibility of the composite (Ratna Sunil et al., 2014). In addition, the application of surface treatments such as anodization or PEO can result in the manufacture of coatings on the surface of the composite material that are long-lasting and resistant to corrosion. These coatings can be advantageous in a variety of applications. The comprehension and enhancement

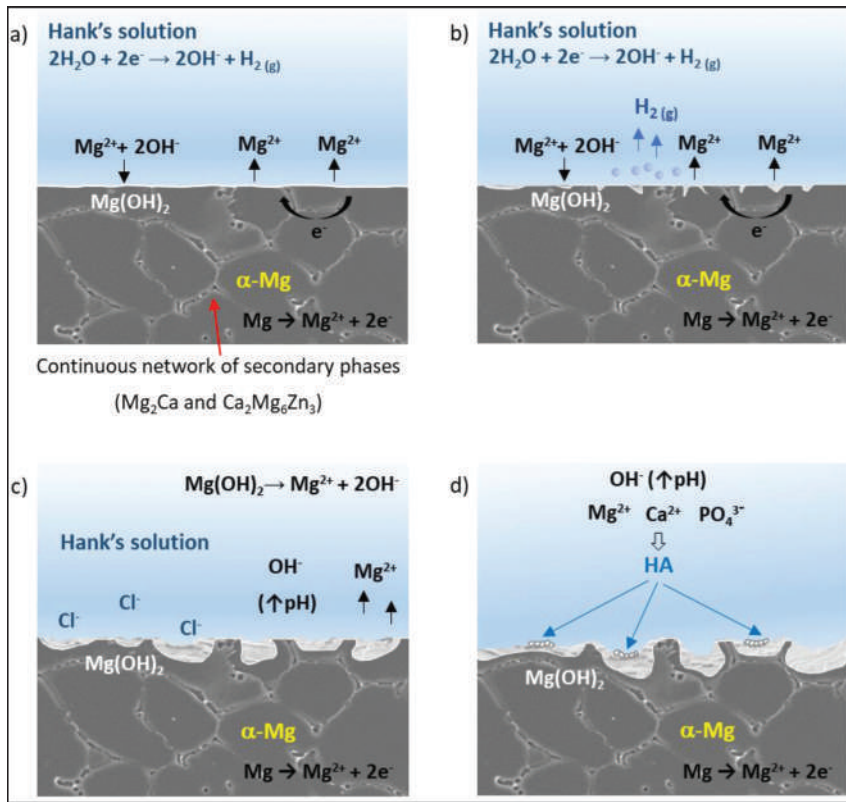


Figure 13. Corrosion Process in As-cast Mg–I Zn–I Ca Alloy Immersed in Hank's Solution: (a) Uniform Corrosion and Protective Layer Formation, (b) Initiation of Initial Pitting, (c) Localised Corrosion Progression, and (d) Hydroxyapatite Precipitation on the Protective Layer.

Source: Pulido-González et al. (2020).

of these corrosion mitigation approaches are of utmost importance in guaranteeing the enduring stability and efficacy of magnesium-based composites employed in biomedical implantation contexts (Allavikutty et al., 2021; Heimann, 2021; Li & Zheng, 2013; Narayanan et al., 2015; Yang et al., 2010).

Figure 13 depicts the mechanism of corrosion that takes place in the Mg–Zn–Ca alloy after it has been cast. This mechanism of corrosion takes place when the resulting alloy is immersed in Hank's solution. The first steps in the process involve the creation of a protective layer and the breakdown of the alloy in a manner that is consistent throughout (Figure 13a), but local disruption of the layer by chlorine ions and hydrogen bubbles leads to the formation of small pits that expose alloy portions to the electrolyte (Figure 13b). The galvanic interaction between the α -Mg matrix and the intermetallic secondary phases also contributes to the acceleration of this process. The presence of the continuous phase at the grain boundary slows corrosion advancement (Figure 13c), even though the

intermetallic phase itself does not disintegrate. As dissolution continues, corrosion progresses through the network of secondary phases (Figure 13d) and finally through the α -Mg matrix via localised corrosion (Figure 13c), exacerbating the overall corrosion. The production of HA is triggered on the residual protective $\text{Mg}(\text{OH})_2$ layer as a result of the interaction between free Mg^{2+} and Ca^{2+} cations and solution phosphates (Figure 13d). This formation is made easier by the rise in pH that results from the presence of OH^- in the solution (Pulido-González et al., 2020).

In Vitro and In Vivo Corrosion Studies

Comprehensive corrosion investigations are carried out both in the laboratory (in the form of *in vitro* testing) and inside living creatures (in the form of *in vivo* testing) in order to evaluate the corrosion properties and biocompatibility of magnesium-based composites. *In vitro* investigations entail submerging the composite specimens in simulated bodily fluid or cell culture conditions to replicate the physiological milieu and evaluate the rate of degradation and kinetics of ion release (Hussein et al., 2022). These findings offer significant insights into the first corrosion behaviour of the material and its possible implications for adjacent

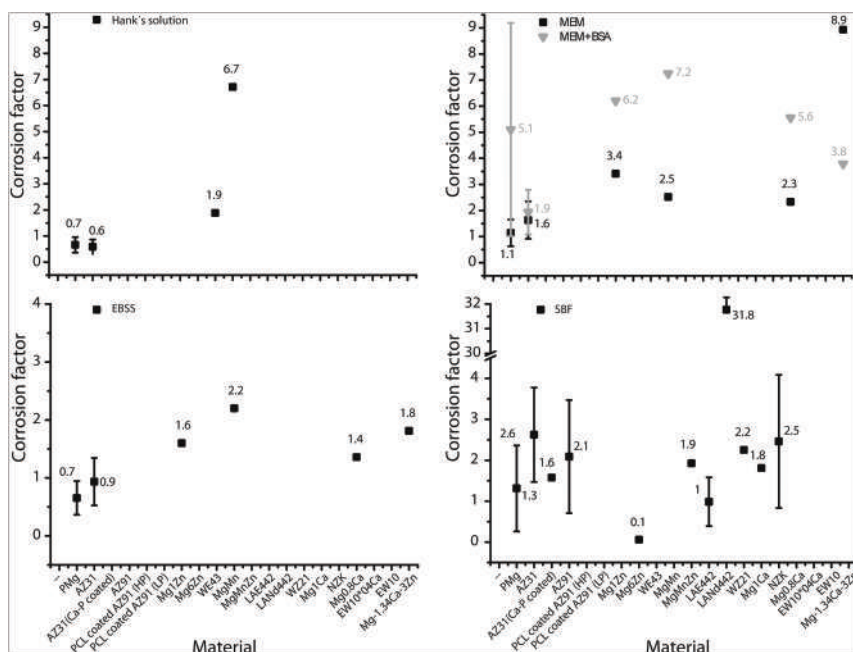


Figure 14. Categorisation of Corrosion Influences Based on the Testing Medium Employed for In Vitro Assessment.

Source: Sanchez et al. (2015).

cells and tissues (Sommer et al., 2022; Tahriri et al., 2018). The process of *in vivo* corrosion investigations entails the insertion of composites into animal models for the purpose of assessing their degradation characteristics, tissue reaction, and suitability for biological applications in a setting that closely mimics real-life biological conditions (Sommer et al., 2022). These studies make it possible to monitor the continuously degrading performance of the implanted composite as well as the host's reaction to the composite. Understanding the biodegradation kinetics, biocompatibility, and safety features of magnesium-based composites requires the combined use of *in vitro* and *in vivo* corrosion research. This is of the utmost importance. These researches provide valuable insights that aid in the refinement of these composites, ensuring their secure and efficient utilization across diverse biomedical implant applications (Cho et al., 2022; Chou et al., 2013; Sanchez et al., 2015; Wen et al., 2021).

Corrosion media used in *in vitro* testing, such as Earle's balanced salt solution (EBSS), SBF, Hanks' balanced salt solution (HBSS), minimum essential media (MEM), and MEM supplemented with bovine serum albumin (BSA), all have distinct effects on the test specimens, as shown in Figure 14. Importantly, the corrosion factors determined for a given corrosion solution show considerable similarity, with only a few exceptions. In general, the corrosion factors for EBSS and SBF are below 3, while MEM exhibits higher values, which range from 1.5 to 3.5. Upon introduction of proteins, the values increase to 4–7. In contrast, the corrosion factors yielded by Hanks' solution differ significantly; yet definitive correlation assessment is hindered due to limited statistical data (Sanchez et al., 2015).

Biocompatibility and Biological Response

Interaction of Magnesium Composites with Cells and Tissues

The assessment of biocompatibility in magnesium composites plays a pivotal role in determining their appropriateness for utilization in biomedical implant applications (Huan et al., 2010; Zhang et al., 2009). Upon being inserted into the human body, these composite materials engage in interactions with cells and tissues, thereby beginning biological reactions that have an impact on their integration and overall functionality. The initial stage of the interaction involves the surface of the composite material, where proteins from the surrounding environment adhere and promote the attachment of cells. Various types of cells, including osteoblasts and fibroblasts, engage in interactions with the composite surface, thereby facilitating the processes of tissue growth and remodelling. The degradation products of the composite material, which encompass magnesium ions, are additionally implicated in biological responses. The concentration of magnesium ions can elicit either stimulatory or inhibitory effects on several cellular processes, including proliferation, differentiation, and apoptosis. Gaining a comprehensive understanding of the interplay between magnesium composites and cellular and tissue components is of paramount importance in facilitating desirable tissue reactions and guaranteeing successful incorporation of the implant within the host

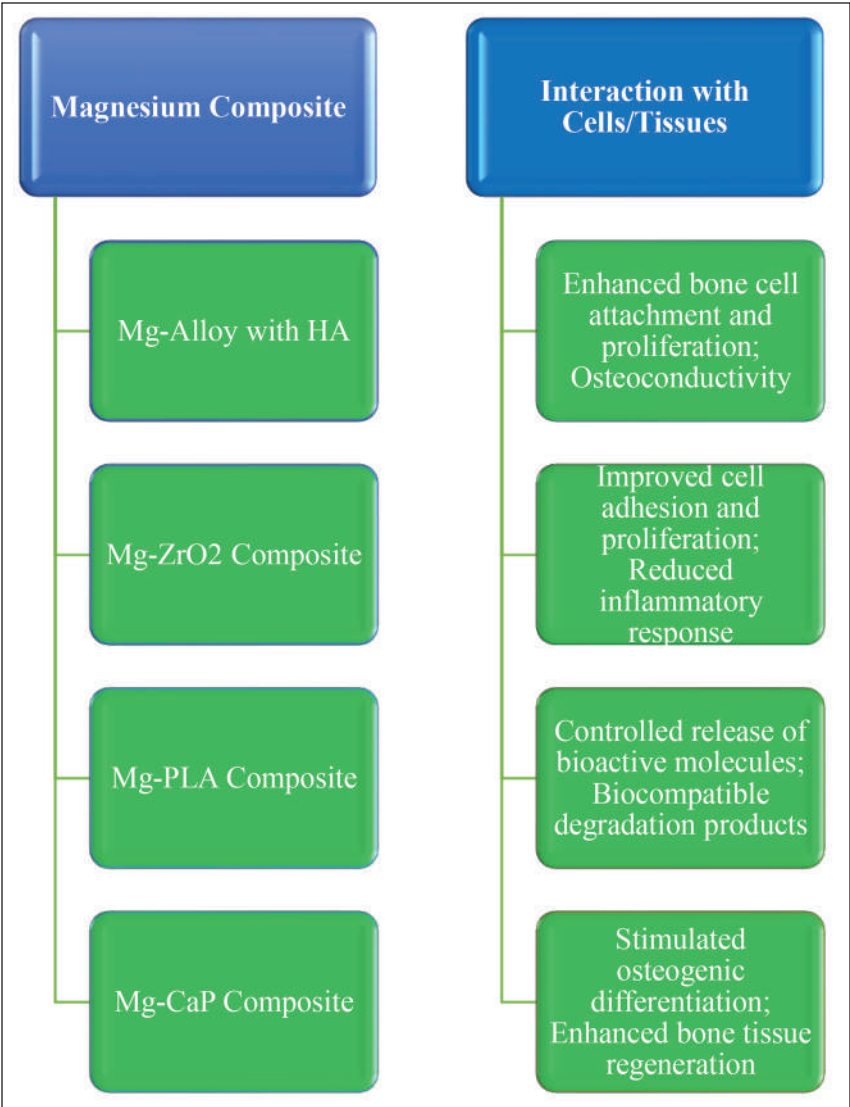


Figure 15. Magnesium Composites' Effects on Cells and Tissues.

milieu (Al-Shalawi, Azmah Hanim, et al., 2023; Castro et al., 2019; Huan et al., 2010; Shunmugasamy et al., 2023). Figure 15 summarises the effects of representative magnesium composites on cells and tissues.

Inflammatory Response and Host Reactions

When introduced into the human body, magnesium composites have the potential to elicit an inflammatory response, which is an inherent component of the

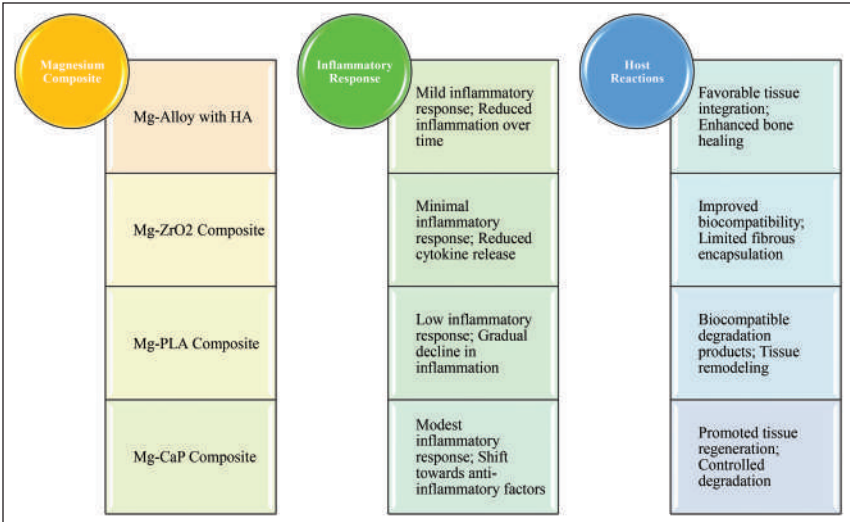


Figure 16. Magnesium-composites-induced Inflammation and Host Reactions.

physiological wound healing process (Al-Shalawi, Azmah Hanim, et al., 2023; Castro et al., 2019; Huan et al., 2010; Li et al., 2022; Shunmugasamy et al., 2023). It is possible to identify the beginning stages of inflammation by the movement of immune cells to the site of implantation. These immune cells may include neutrophils and macrophages. The immune system’s cells perform an essential part in the removal of waste from cells and the regulation of the local microenvironment in the tissue that surrounds them. On the other hand, inflammation that is excessive or persists for an extended period of time can have negative effects on the host, such as the development of chronic inflammation or the production of fibrous encapsulation surrounding the implant. The composite’s rate of degradation can be affected by the inflammatory response, which impacts the local electrolyte balance and pH. The management of degradation rate and the manipulation of surface qualities of composites are crucial factors in mitigating the inflammatory response and facilitating a controlled and positive host reaction towards the implant (Avery et al., 2023; Bakhsheshi-Rad et al., 2017; Hotchkiss et al., 2018; Hua et al., 2023; Huan et al., 2010; Jurczyk et al., 2020; Martinez et al., 2023; Prestat & Thierry, 2021; Saha et al., 2022; Selders et al., 2017; Velnar et al., 2016). Figure 16 summarises magnesium-composite-induced inflammation and the associated host reactions.

Immunomodulatory Effects and Osteogenesis

Magnesium composites possess immunomodulatory properties that can exert an influence on the immune response and subsequent tissue healing processes (He et al., 2023). The composite material has the ability to regulate immune cell activity and cytokine production through the release of magnesium ions. This has an

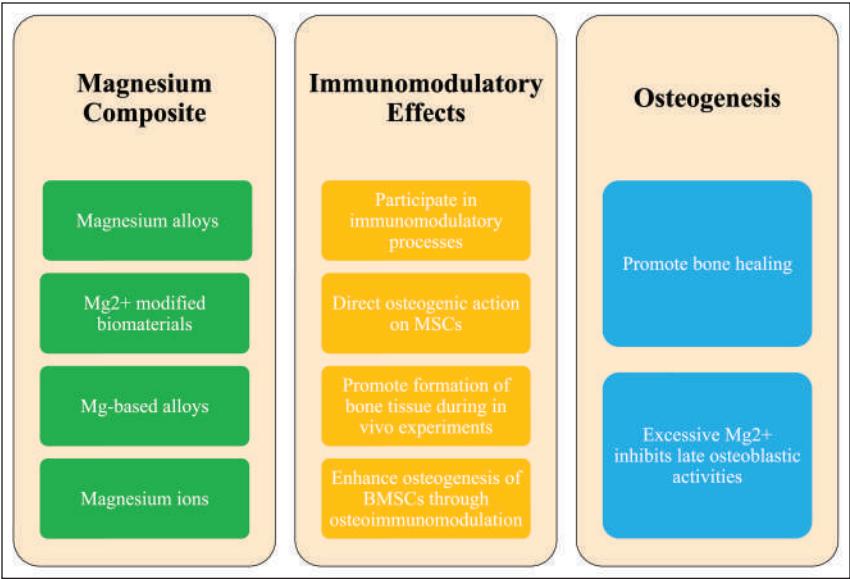


Figure 17. Magnesium Composites Immunomodulatory Effects and Osteogenesis.

impact on the equilibrium between anti-inflammatory and pro-inflammatory responses (Zhou et al., 2021). The appropriate modulation of the immune system can effectively facilitate the process of tissue repair and regeneration, while concurrently mitigating the occurrence of unfavourable reactions. Moreover, magnesium composites have demonstrated potential in facilitating osteogenesis, which is the biological process responsible for bone formation (He et al., 2023). Magnesium ions released during degradation can promote osteogenic differentiation of mesenchymal stem cells, which supports bone regeneration and stronger bonding between the implant and adjacent bone tissue (He et al., 2023; Zhou et al., 2021). Additionally, the similarity between magnesium and natural bone minerals can create a conducive microenvironment for the functioning of osteoblasts and the creation of the extracellular matrix (He et al., 2023). Figure 17 schematically presents the immunomodulatory effects and osteogenic potential of magnesium composites. It is of utmost importance to comprehend the immunomodulatory effects and osteogenic capacity of magnesium composites in order to enhance their biocompatibility and guarantee the successful integration and usefulness of these materials in biomedical implant applications connected to bone (Loi et al., 2016; Medhat et al., 2019; Xue et al., 2022; Zhao et al., 2022).

Preclinical and Clinical Studies

Preclinical Evaluation of Magnesium-based Implants

Prior to conducting human trials, magnesium-based implants undergo thorough preclinical examination in laboratory settings. This stage evaluates implant

biocompatibility, mechanical characteristics, and degradation patterns *in vitro* and *ex vivo* (Chagnon et al., 2019). *In vitro* investigations encompass the exposure of implants to simulated bodily fluids or cell cultures, enabling the examination of their interactions with biological constituents (Hua et al., 2023; Huan et al., 2010; Loi et al., 2016). The degradation rate, release of magnesium ions, and cellular reactions of the implants are meticulously examined in order to comprehend their behaviour within a controlled setting (Chen et al., 2019; Li et al., 2014; Nene et al., 2016; Shan et al., 2022). Furthermore, *ex vivo* tests may encompass the transplantation of the composites into animal models' tissues or organs that exhibit a high degree of similarity to human physiology (Bakhsheshi-Rad et al., 2017; Li et al., 2022; Martinez et al., 2023; Sanchez et al., 2015; Sommer et al., 2022). These experiments offer significant insights into the early biological reaction and degrading behaviour of the implants within a scenario that closely resembles real-world conditions. The preclinical assessment is a critical step in identifying potential problems, improving implant designs, and predicting their performance before proceeding to animal testing and clinical trials (Chagnon et al., 2019; Mathew et al., 2023; Sekar et al., 2021).

Animal Studies: Efficacy and Safety Assessment

The examination of animals is an essential component in the assessment of the effectiveness and safety of magnesium-based implants within living beings. These investigations entail the insertion of the composites into animal models, commonly including rabbits or pigs, in order to evaluate their efficacy inside a functioning biological system (Sun et al., 2021). Table 3 summarises the outcomes of an initial minipig study of magnesium implants, organised by implantation site, together with the associated safety evaluations (Imwinkelried et al., 2020). The present investigation examines implant biocompatibility, deterioration, tissue integration, and host reactions (Rahim et al., 2020; Sun et al., 2021). The implants were monitored over a prolonged duration in order to evaluate the enduring consequences and possible problems. Imaging modalities, histopathological examination, and biomechanical assessment are often employed methodologies for the evaluation of implant efficacy and tissue reactions (Grün et al., 2018). Animal research plays a crucial role in gathering essential information on the biological processes, tissue regeneration, and possible negative effects of implants. The aforementioned findings provide valuable insights for enhancing the design and safety of implants prior to embarking on human clinical trials (Grün et al., 2018; Rahim et al., 2020; Sekar et al., 2021; Sun et al., 2021).

Clinical Trials and Case Studies of Magnesium-based Implants

Clinical studies provide the final assessment of magnesium-based implants' safety and efficacy in humans (Zhao et al., 2017). The trials encompass meticulously planned investigations wherein human subjects are administered implants within controlled environments (Sekar et al., 2021). The primary objectives of clinical

Table 3. Summary of the Outcomes from the Initial Minipig Study, Organised by the Site of Implantation. An Overview of Safety Evaluations Along with Corresponding References from Publications.

Conditions Required for Implantation	Situation of Degradation/Loading	Safety Considerations	Results	References
Nasal bone: Tissue pocket with rectangular plate	Degradation without load Maintenance of strength	Consistency of degradation The initial bending strength is at 80% after 12 weeks	Limited localised corrosion	Imwinkler et al. (2013)
Frontal bone: Contoured plates using non-locking screws	Gas release The plates are against the bone and the screws tightened for tension Gas release Plate—screw connection	Coating prevents initial peak release Screw head rupture during implantation Dependent on screw location No preferential degradation observed	Replaceable over drilled Screws	Schaller, Saulacic, Imwinkler, et al. (2016)
Mandible: Thin-walled rivet-screws for cortical bone	Implant deformed plastically while undergoing load	Effects of plastic deformation and loading	Coating delays gas release despite deformation	Schaller, Saulacic, Beck, et al. (2016)
Rib arches: Contoured plates using non-locking screws	Bone plates are fitted and screws are tightened to move the ribs in a partial osteotomy The rib-specific plates are intact, but some screws are broken	Effect of respiration and animal body weight	One pilot animal experienced a broken Mg mandible plate	Schaller et al. (2017)
Systemic effects: Combined Implants	Total degradation products Release of gas	Buildup of metals in body organs due to alloying No systemic effects observed	No accumulation except for Zr	Imwinkler et al. (2020)

trials include assessing the implants' biocompatibility, degradation kinetics, mechanical performance, and long-term outcomes in real-world scenarios (Prestat & Thierry, 2021). The trials are typically conducted in phases, starting with small groups to evaluate safety, followed by larger groups to assess efficacy and gather statistical data. Case studies may also be conducted to report individual patient experiences and outcomes with magnesium-based implants. The data from clinical trials and case studies help establish the safety profile and effectiveness of the implants in human patients. Positive results from clinical trials pave the way for regulatory approval and widespread clinical use of magnesium-based implants in specific medical applications (Albaraghteh et al., 2022; Espiritu et al., 2021, 2022; Garimella et al., 2026; Jurczyk et al., 2020; Prestat & Thierry, 2021; Sekar et al., 2021; Stürznickel et al., 2021; Suljevic et al., 2022; Venkateswarlu et al., 2023; Zhao et al., 2017).

Applications of Magnesium Metal Matrix Composites in Biomedical Implants

Bone Fixation Devices and Osteosynthesis

Mg-MMCs find application in bone fixation devices and osteosynthesis, where they play a crucial role in stabilizing fractured bones and supporting the healing process (Xue et al., 2022). These implants are used in orthopaedic surgeries to immobilise fractured bones and facilitate bone fusion. The biodegradability of magnesium composites is particularly helpful in this context because, as they gradually disintegrate over time, they lower the risk of implant-associated problems and eliminate the requirement for a further operation for removal (Medhat et al., 2019; Xue et al., 2022; Zhao et al., 2022). Furthermore, the mechanical properties of magnesium composites are remarkably close to those of genuine bone. This promotes load transfer throughout the healing process and lessens the effects of stress shielding. The controlled degradation of magnesium composites in bone fixation devices can stimulate bone formation and remodelling, leading to improved long-term outcomes. In order to ensure an adequate level of implant integrity during the entire healing period, the rate of deterioration and the mechanical properties of the composite are carefully examined. Bone fixation devices and osteosynthesis with magnesium composites represent a promising area for enhancing patient outcomes and reducing the burden of implant-related surgeries (Cerqueira et al., 2021; Espiritu et al., 2022; Fu et al., 2011; Garimella et al., 2026; Kim et al., 2020; Munir et al., 2023; Putra et al., 2020; Tahriri et al., 2018; Zhang et al., 2021; Zhou et al., 2021).

Cardiovascular Stents and Vascular Applications

The investigation of magnesium-based composites for cardiovascular stents and vascular applications is now underway (Walker et al., 2014). Stents are cylindrical instruments employed for the management of coronary artery

disease and various vascular blockages (Ahadi et al., 2023). The utilization of magnesium composites in stents presents numerous benefits, including the provision of temporary scaffolding support throughout the healing process and slow degradation, thereby obviating the necessity for permanent implants (Erbel et al., 2007). Ensuring the biocompatibility of magnesium composites is of utmost importance in mitigating the potential for inflammation and thrombosis commonly associated with conventional stents (Ahadi et al., 2023). Magnesium composites, when intentionally degraded for use in vascular applications, may be able to solve the problem of in-stent restenosis that plagues conventional metal stents (Ma et al., 2016). Nevertheless, it is crucial to optimize the degradation kinetics, mechanical strength, and surface qualities in order to guarantee the stent's mechanical support throughout the healing process and mitigate the risk of premature stent failure. Ongoing research in this domain demonstrates the potential of magnesium composites to significantly transform the field of cardiovascular treatments by providing enhanced biocompatibility and patient-centric solutions (Erbel et al., 2007; Ma et al., 2016; Walker et al., 2014).

Orthopaedic Implants and Soft Tissue Scaffolds

Mg-MMCs exhibit considerable potential for application in diverse orthopaedic implant and soft tissue scaffold designs (He et al., 2023). Within the field of orthopaedics, these composite materials find application in several types of implants, including spinal cages, interbody fusion devices, and craniomaxillofacial plates (Kim et al., 2018; Schaller, Saulacic, Imwinkelried, et al., 2016; Shen et al., 2019). The favourable nature of magnesium composites lies in their biodegradability, which proves beneficial in various applications. Specifically, these implants offer support during the healing process while concurrently undergoing slow degradation as bone tissue undergoes regeneration (Medhat et al., 2019). It is possible to tailor the composite material's mechanical properties to meet the specific requirements of orthopaedic applications; this results in a solution that is both adaptable and individualized for patients (Chandra & Pandey, 2020; Tsakiris et al., 2021; Walker et al., 2014). In addition to this, magnesium composites have the potential to function as scaffolds in the field of tissue engineering and regenerative medicine (Sheikh et al., 2015; Xue et al., 2022). Temporary frameworks are utilized to facilitate tissue growth and remodelling processes by stimulating the development of new tissue (Adetunla et al., 2022; Cerqueira et al., 2021; Huang et al., 2014; Persaud-Sharma & McGoron, 2012). The progressive deterioration of the composite material facilitates the development of a functional tissue architecture, leading to the eventual replacement of the implant by native tissue. However, in order to guarantee the efficiency and safety of these implants and scaffolds in the context of orthopaedic and tissue engineering activities, it is absolutely necessary to conduct a comprehensive analysis of the rate of degradation, the mechanical features, and the biocompatibility of the material (Adetunla et al., 2022; Huang et al., 2014; Radha & Sreekanth, 2017; Rahman et al., 2020).

Challenges and Future Perspectives

Biodegradation Control and Implant Longevity

The management of the biodegradation rate of magnesium-based composites is one of the key challenges that arises in the process of utilizing these materials for the manufacture of biomedical implants (Rahman et al., 2020). The progressive deterioration of implants is advantageous in reducing the necessity for implant extraction procedures. However, it is crucial to strike a balance that guarantees adequate mechanical reinforcement during the entirety of the healing phase (Adetunla et al., 2022; Zhao et al., 2017). Rapid deterioration can cause premature implant failure and excessive hydrogen evolution, while second-phase particles and reinforcements may accelerate attack through microgalvanic coupling; delayed degradation may impede tissue repair (Zhao et al., 2017). The attainment of an optimal degradation rate is contingent upon the meticulous choice of alloying components, composite composition, and surface changes (Aikin et al., 2025). Moreover, the degrading behaviour of implants might be influenced by changes in physiological settings among individuals, necessitating the implementation of individualized approaches in implant design. The maintenance of suitable biodegradation management is of utmost importance in order to attain optimal longevity of implants and favourable outcomes for patients (Adetunla et al., 2022; Zhao et al., 2017).

Cytotoxicity and Biocompatibility Concerns

The release of magnesium ions during the degradation process of magnesium composites has the potential to have an effect on both cytotoxicity and biological responses (Xin et al., 2011). Magnesium ions, when present in low concentrations, have been shown to encourage the growth of new cells and the repair of damaged tissue; however, magnesium ions present in high concentrations have been shown to have the opposite effect, leading to cell death and inflammation (Zhao et al., 2017). Properly tuning the degradation rate and ion release kinetics is essential to minimize cytotoxicity and ensure biocompatibility. Additionally, variations in individual responses to magnesium ions must be considered, as some patients may be more sensitive to their presence. In order to address cytotoxicity and biocompatibility concerns before moving on to clinical applications, it is absolutely necessary to carry out complete biocompatibility assessments. These assessments should include both *in vitro* and *in vivo* research (Chagnon et al., 2019; Sezer et al., 2018; Xin et al., 2011).

Translation to Commercial Products and Regulatory Aspects

Despite significant research advancements, the translation of magnesium-based composites into commercial products for widespread clinical use involves various challenges (Walker et al., 2014). Manufacturing scalability, consistent quality control, and process reproducibility remain significant concerns (Wang

et al., 2022). Additionally, regulatory aspects related to biodegradable materials and medical devices must be thoroughly addressed to meet safety and efficacy standards for clinical applications. Obtaining regulatory approvals can be a lengthy and resource-intensive process. Collaborations between academic researchers, medical device companies, and regulatory bodies are crucial for navigating these challenges and bringing magnesium-based composites to market. Moreover, long-term post-market surveillance is essential to monitor the performance and safety of these implants in real-world scenarios. In order to realize the full potential of magnesium-based composites as novel and patient-friendly solutions in the field of biomedical implants, it is essential to overcome these hurdles and handle regulatory considerations (Al-Shalawi, Azmah Hanim, *et al.*, 2023; Al-Shalawi, Mohamed Ariff, *et al.*, 2023; Chagnon *et al.*, 2019; Chandra & Pandey, 2020; Drelich *et al.*, 2020; Hung *et al.*, 2019; Prakasam *et al.*, 2017; Sezer *et al.*, 2018; Wang *et al.*, 2022; Wu *et al.*, 2015; Zhao *et al.*, 2017; Zhu *et al.*, 2022).

Conclusion

Because of properties such as biocompatibility, mechanical qualities that are similar to natural bone, and controlled biodegradation, magnesium-based composites provide a compelling alternative to standard nondegradable implants. The properties and characteristics of magnesium, including its lightweight nature and biocompatibility, make it an attractive material for medical applications. Alloying with specific elements allows for the optimisation of degradation kinetics and mechanical performance, tailoring the composites to specific implant requirements. The corrosion behaviour of magnesium composites was explored in this article. The article also emphasized the need for biodegradation control to ensure optimal implant longevity and prevent premature failure. Biocompatibility work continues to flag risks linked to magnesium ion release, so careful material design and thorough biocompatibility evaluation remain necessary. The review article contained a complete analysis of the many applications of magnesium composites in the realm of biomedical implants as well as other applications. These applications cover a wide variety of medical devices, including stents for the circulatory system, orthopaedic implants, bone fixation devices, and scaffolds for soft tissue. The exhibited applications have showcased the potential of magnesium composites in improving patient outcomes and minimising the necessity for subsequent surgeries through the facilitation of progressive degradation and tissue regeneration. When considering the future, the outlook for the utilization of biodegradable magnesium-based composites in biomedical implants is highly favourable. Current research endeavors are focused on tackling the obstacles associated with degradation control, biocompatibility, and regulatory considerations in order to facilitate the successful transition of these innovations into commercially viable products. The establishment of collaborations among academia, industry, and regulatory agencies is of utmost importance in effectively navigating the intricate journey from preclinical assessment to clinical trials and eventual market acceptance. In short, biodegradable magnesium composites remain

promising temporary implant materials when mechanical support, biocompatibility, and degradation rate are jointly managed. Continued progress in processing, surface engineering, and clinical evaluation will determine how widely they enter routine medical device practice.

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Data Availability

No data set is available with the article because no new data were created/analysed in this study.

Declaration of Conflicting Interests

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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