

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

Abstract

Hybrid metal matrix composites incorporating multiple reinforcements exhibit immense potential for meeting the evolving requirements of engineering applications. These composites offer exceptional mechanical properties, including enhanced hardness and ultimate tensile strength, owing to the significant influence of reinforcements on their material characteristics. This study focuses on experimental investigations conducted on a hybrid metal composite of Aluminum alloy (A356/LM25) reinforced with micro-sized ceramic particles (Al_2O_3 , MgO , SiC , ZrO_2 , and graphite) using the Stir casting technique. The research aims to analyze and present the mechanical properties of the alloy.

To achieve this objective, various sets of composites were fabricated, maintaining a constant 1% weight fraction of graphite particles (sized between 100-200 μm), while incorporating ceramic particles at weight fractions of 1%, 3%, and 5% (also sized between 100-200 μm). Additionally, unreinforced A356/LM25 samples were included as a reference. Following solidification, the samples underwent preparation and testing for X-ray Radiography to assess their structural integrity. Moreover, mechanical properties, such as tensile strength and hardness, were evaluated, while XRD and SEM techniques were employed to analyze the microstructural properties.

The XRD analysis confirmed the crystalline nature of the composites, while the SEM images provided valuable insights into microstructural defects such as agglomeration, micro-cracks, and void formation. Tensile and hardness tests revealed notable improvements in the performance of the hybrid MMC composites when compared to the base material of A356/LM25. These findings underscore

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EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

the potential of the developed composites to meet the demanding requirements of engineering applications.

SEM images reveal that agglomeration is a prevalent manufacturing defect in LM25 alloy composites, worsening with increased reinforcement material. Void and crack formation, identified in SEM images, significantly enhance material properties, particularly tensile strength, as confirmed by XRD maintaining the base alloy's crystalline nature. The highest tensile strength (305.7 MPa) occurs in LM25+1%MgO+3% SiC, and the highest hardness (96.95 BHN) in LM25+5% Al₂O₃. Weak adhesive bonding causes agglomerations, affecting structural integrity due to filler-filler interaction. XRD shows maintained crystalline structure. Alloy additives like Al₂O₃ and graphite-ZrO₂ minimally impact LM25 alloy microstructure, while MgO and SiC noticeably decrease counts for specific peaks, influencing microstructural properties. Tensile strength in secondary composites varies with SiC, MgO, and Al₂O₃ percentages, influenced by work hardening, load carrying, mismatch strengthening, grain refinement, and particle strengthening. Brinell hardness varies with reinforcement, impacted by the Hall-Petch mechanism, particle strengthening, and grain refinement. The study guides the selection of micro particles for enhancing composite material properties.

Keywords: Stir casting process, Aluminum Alloy, Graphite, Tensile Strength, Hybrid MMC

I. INTRODUCTION

The realm of engineering materials is delineated into four principal cohorts: metals, polymers, ceramics, and composite materials, with the latter encapsulating a pivotal subcategory. Among these, the advent of composite materials represents a seminal accomplishment in the interdisciplinary domains of science and engineering. Noteworthy within this classification are metal matrix composites (MMCs), specifically those constituted by metals and their alloys.

Distinguishing themselves from polymer matrix composites, MMCs exhibit commendable attributes such as heightened resilience to elevated temperatures, superior transverse mechanical characteristics, augmented electrical and thermal conductivities, and heightened resistance to moisture. Aluminium metal matrix composites (MMC), in particular, warrant scrutiny due to their diminutive density coupled with elevated specific mechanical attributes. The AL A356 matrix material, chosen for its distinct properties, typifies the superior characteristics sought after in the creation of lightweight components, especially in commercial and automotive sectors. The alloy's density, reduced by 67% vis-à-vis conventional materials like cast iron, is complemented by a threefold increase in heat conductivity, coupled with wear resistance and strength on par with cast iron, rendering it an optimal choice for lightweight applications.

1. Introduction to MMCs: Metal matrix composites (MMCs) commonly employ aluminum (Al) and magnesium (Mg) as matrix materials, with alternatives including copper (Cu), iron (Fe), and titanium (Ti). Magnesium-based composites have garnered attention for their favorable mechanical properties compared to monolithic magnesium alloy. However, widespread use in automobiles is impeded by limitations such as low ductility and fracture resistance, attributed to magnesium's reactivity at high temperatures. Protective coatings or naturally occurring oxides are employed to mitigate reactivity. In the case of iron matrix materials, brittleness and lower impact strength pose challenges, limiting applications to those prioritizing wear resistance. Copper-based MMCs find utility in applications emphasizing electrical and thermal conductivity, compensating for pure copper's poor strength. Aluminum and its alloys stand out as preferred matrix materials due to their light weight, economic feasibility, processability, high strength-to-weight ratio, and corrosion resistance.

Reinforcements in MMCs can take various forms, such as particulates, fibers, layers, or interpenetrating structures. Particle-reinforced composites, particularly those using Al matrix reinforced with SiC, Al₂O₃, or B₄C, are spotlighted for their accessibility, affordability, and uniform dispersion. Engineers widely adopt aluminum metal matrix composites (AMMC) for applications like braking rotors, drive shafts, pistons, and cylinder liners due to their exceptional mechanical properties and cost-effectiveness. In composite material production, ensuring appropriate interfacial bonding between matrix and reinforcing components is crucial for achieving desired properties. Figure 1.1 illustrates a range of matrices and reinforcing materials applicable in MMC production

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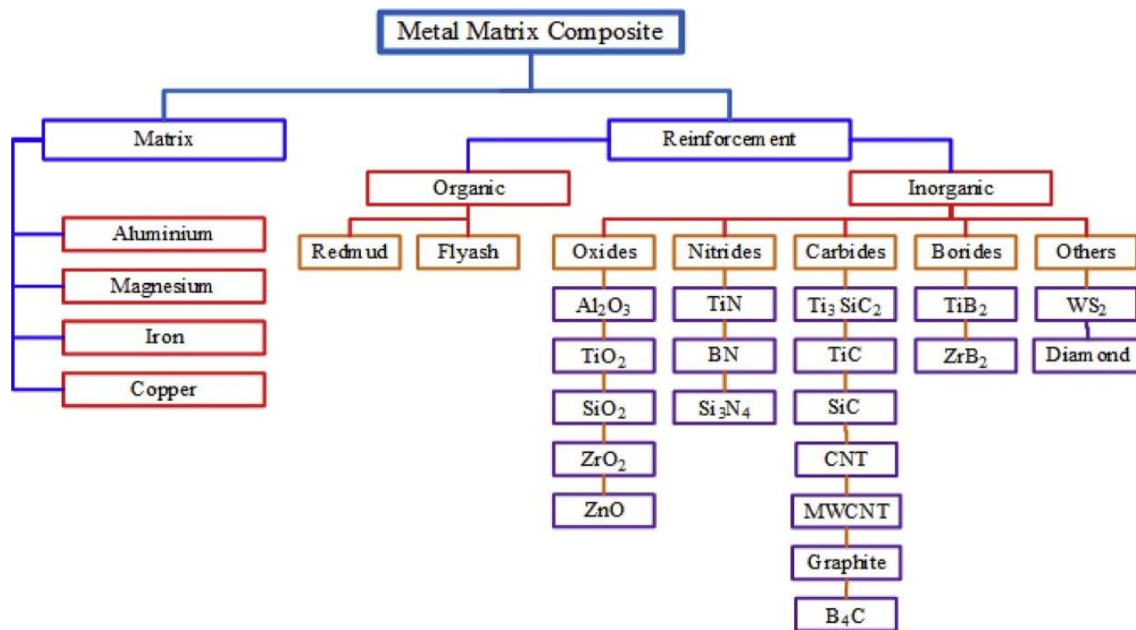


Figure 1.1: Various matrix and reinforcement materials used for the production of MMCs.

2. Production Processes for MMCs: The intricate production processes for Metal Matrix Composites (MMCs), classified in Figure 1.2 based on primary processes such as liquid or solid metal matrix treatments, including semi-solid and in situ methodologies. The chosen production procedures significantly influence manufacturing costs and mechanical properties. This section focuses on MMCs produced through bulk processing, exploring various techniques outlined in the classification.

- **Solid State Processing/Powder Metallurgy:** Solid-state MMC manufacturing involves elevated temperature and pressure for mutual diffusion between matrix and reinforcements, critical for successful completion. Conventional approaches for MMC production within this category are discussed.
- **Spark/Plasma Sintering:** Spark plasma sintering (SPS) is employed in the effective sintering of Al₂O₃ (Matrix)-SiC (Reinforcement) composites, rapidly elevating temperature and pressure, enhancing the sintering process. Achieving maximum hardness at 20% Al₂O₃-SiC composition, SPS presents a viable method for homogeneous alumina dispersion in aluminum matrix composites.

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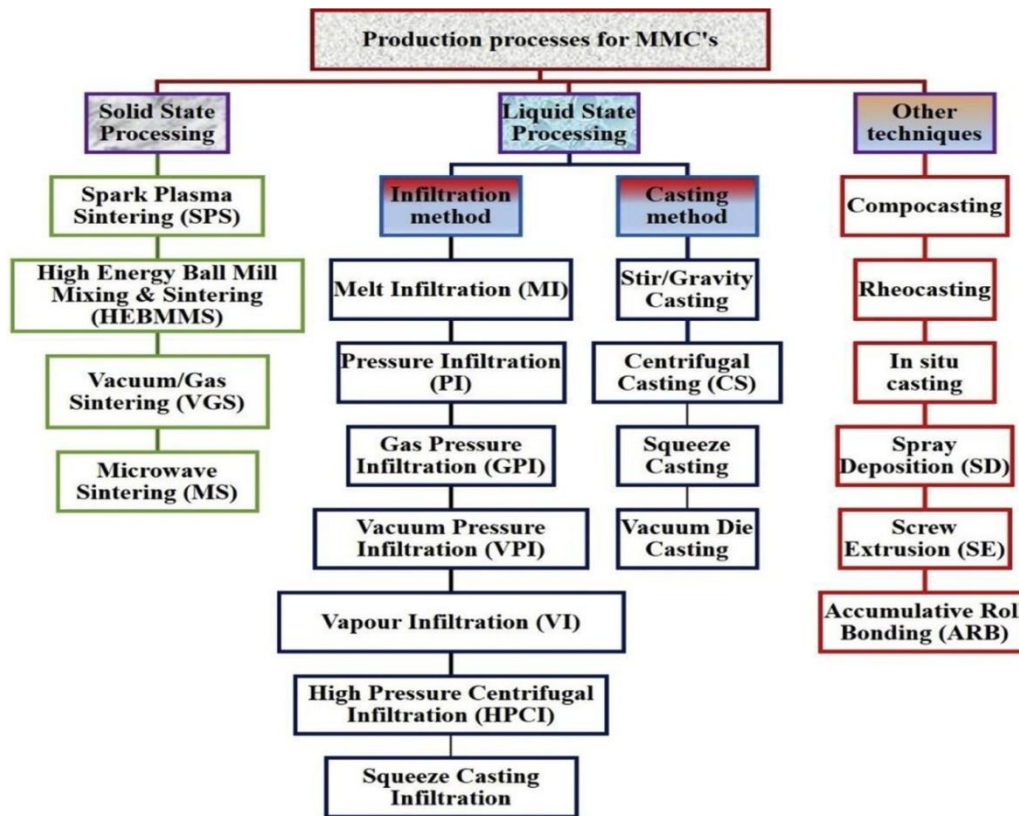


Figure 1.2: Classification of the various production process for MMCs

- **High Energy Ball Mill Mixing and Sintering (HEBMS)** :Utilizing high-energy ball milling and sintering (HEBMS) at 300 revolutions per minute, Bhatt et al. successfully manufactured nanostructured Al-Mg composites reinforced with amorphous silica particulate. Nano-reinforced composites exhibited significantly increased hardness compared to micro-reinforced counterparts, demonstrating uniform reinforcement particle distribution.
 - **Vacuum/Gas Sintering:** Widely employed vacuum and gas pressure sintering method is analogous to HEBMS but differs in subsequent sintering, offering manageability and scalability. Controlling porosity and grain size during sintering influences micro hardness and fracture toughness.
 - **Microwave Sintering:** Microwave sintering utilizes electromagnetic field energy directly applied to the substance, ensuring uniform volumetric heating. Desirable for larger specimens, it outperforms plasma arc sintering in certain applications. Microwave-sintered nanocomposites demonstrated superior mechanical and thermal properties, reaching compression and tensile strengths of 392 MPa and 178 MPa.
3. **Liquid State Processing:** It delves into the intricacies of liquid state processing for Metal Matrix Composites (MMCs), exploring methods deemed straightforward and cost-effective by various industries. Liquid state processes involve casting techniques, encompassing mixing molten metal with reinforcing particles, infiltrating molten metal

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

into preforms or fiber packs, and other related processes such as melt, pressure, gas, vacuum, vapor, high-pressure centrifugal, and squeeze casting.

- **Infiltration Methods:** Infiltration involves the permeation of a preform by molten metal, achievable through pressure infiltration or melt infiltration (pressureless infiltration). Pressureless infiltration involves introducing reinforcements into a die, followed by molten alloy infiltration without external pressure. Pressure infiltration, on the other hand, employs external pressure, either directly or indirectly through inert gas, vacuum pressure, vapor, centrifugal force, or squeezing infiltration.
 - **Melt Infiltration:** Achieved low-temperature manufacturing of Al 6061-Ti3SiC2 composites using pressureless melt infiltration, resulting in noteworthy compressive strength and hardness.
 - **Pressure Infiltration:** Applied the pressure infiltration method for graphene nanoplates reinforced pure aluminum composites, demonstrating enhanced mechanical properties. This method is lauded for its quality and improved thermal conductivity, critical for applications like piston engines.
 - **Gas Pressure Infiltration:** Gas pressure infiltration utilizes pressurized gas to force molten metal into a prepared dispersion phase, exemplified successful generation of Al/diamond composites with enhanced thermal conductivity.
 - **Vacuum Pressure Infiltration:** Vacuum pressure infiltration involves applying increased gas pressure for successful operation, exemplified by Ma et al.'s construction of two-dimensional carbon fiber-reinforced aluminum matrix (Cf/Al) composites, demonstrating reduced porosity and increased tensile strength.
 - **Vapor Infiltration:** Reinforced composites are formed through vapor infiltration, where matrix material infused into fiber preforms with reactive gases yields composites like porous silicon carbide nanowire/silicon carbide (SiCnw/SiC) with excellent mechanical properties.
 - **High-Pressure Centrifugal Infiltration:** High-pressure centrifugal infiltration employs rotating molds and higher pressures for the infiltration process, showcasing potential future use for MMC fabrication.
 - **Squeeze Casting Infiltration:** Squeeze casting infiltration involves applying ram force to liquid metal, permeating preforms. demonstrated improved hardness in AlSi12/Al2O3 using this technique, successfully produced carbon fiber-reinforced aluminum matrix composites, reducing aluminum carbide production and enhancing wettability and particle distribution.
4. **Casting Methods:** It delves into casting methods, a widely employed manufacturing process known for its versatility in shaping various materials at a reasonable cost. Liquid metal is poured into a mold during casting, allowing it to cool and solidify into the intended form, commonly used in the construction of components like lathe beds, milling

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

machine frames, and internal combustion engine parts, known for their high compressive strengths and overall cost-effectiveness.

- **Stir/Gravity Casting:** Stir casting employs mechanical stirring to blend ceramic particles or short fibers with a molten metal matrix, yielding a composite material with enhanced properties. It demonstrated increased mechanical properties, such as hardness and tensile strength, in an A6063/TiC composite using the stir gravity casting method.
 - **Centrifugal Casting:** Centrifugal casting involves pouring molten metal into a rotating mold, subjecting the metal to significant pressure by centrifugal force. It demonstrated exceptional control of microstructure, remarkable mechanical qualities, and effective mold filling in Al-B-Mg composites produced through centrifugal casting. Wang et al. studied SiC/Al composites, revealing non-homogeneous particle distribution influenced by centrifugal force, with wear resistance behavior tied to specific process parameters.
 - **Squeeze Casting:** Squeeze casting combines casting with hydraulic forging, pouring liquid metal into a die and immediately applying high-pressure hydraulic pressing. It improved AA7050 aluminum alloy with graphene nanoparticles through stir and squeeze casting, achieving consistent particle distribution and a tensile strength increase.
 - **Vacuum Die Casting:** Vacuum die casting involves keeping the die in a vacuum to remove gases from the melt, resulting in lower porosity and increased cast density and strength. It introduced advanced stir vacuum casting for large-scale production of AA6061-31%B₄C, achieving even particle distribution and a 112.5% tensile strength increase compared to AA1100-31%B₄C
5. **Stir Casting Furnace Design used for the Production of AMMCs:** explores the design and types of stir casting furnaces used in the production of Metal Matrix Composites (MMCs). Stir casting, a versatile technique conducted in the liquid state, involves blending ceramic particles or short fibers with molten metal, creating composites with enhanced properties. The simplicity, versatility, and cost-effectiveness make it widely adopted. Factors considered in choosing stir casting procedures include achieving uniform particle dispersion, establishing a perfect matrix-reinforcement link, minimizing porosity, preventing chemical reactions, and averting interactions with air components. The section categorizes stir casting furnaces based on the metal-melting process. Various classifications include coal-fired stir casting, electrical stir casting, stir casting using resistance heating, quick quench stir casting, two-step stir casting, stir casting under inert atmosphere, modified stir casting, ultrasonic processing, stir casting under a modified inert atmosphere, bottom pouring stir casting with squeeze casting attachment, Disintegrated Melt Deposition (DMD), and electromagnetic stir casting.

Detailed discussions and case studies accompany each classification, examining methodologies, experimental outcomes, and the impact on composite properties. Examples range from coal-fired furnaces enhancing wear characteristics in Al matrix

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

composites to electrical resistance stir casting improving tensile strength in Al (A356) with fly ash composites. Diverse techniques, such as quick quench stir casting, two-step stir casting, and modified inert atmosphere stir casting, showcase the versatility of the stir casting process in creating tailored MMCs with improved mechanical properties.

6. Factors Influencing Stir Casting: Research findings highlight key factors impacting the stir casting process: stirring speed, stirring duration, and stirring temperature.

- **Stirring Speed:** Achieving uniform reinforcement particle distribution enhances particulate MMC properties. Low stirrer RPM limits shearing force, hindering particle dispersion. This results in agglomeration and clustering due to insufficient resistance. Higher stirrer speeds create a vortex, allowing dispersed particles to uniformly distribute in the matrix. However, increased speeds may introduce gas particles, elevating porosity.
- **Stirring Duration:** Critical for uniform dispersion, insufficient stirring time leads to particle clustering. Some matrix areas may lack reinforcement inclusions.
- **Stirring Temperature:** Matrix metal temperature impacts viscosity and particle distribution. Higher temperatures accelerate chemical reactions between reinforcement particles and the metal matrix.

7. Challenges in Stir Casting Processes: Several considerations arise when fabricating metal matrix composites (MMCs) through stir casting, impacting their mechanical properties significantly. Achieving uniform distribution of micron-sized reinforcement particles poses a substantial challenge, affecting MMC characteristics. Adjusting variables such as melt viscosity, stirrer speed, stir time, and particle size is crucial for even particle distribution. The density difference between matrix and reinforcement can lead to non-uniform distribution, particularly when particles float or settle. The use of pricey nano-sized reinforcements introduces risks of agglomeration and demands careful handling. Wettability between solid reinforcement particles and liquid Al matrix influences bonding, impacting MMC mechanical properties. Porosity is a substantial concern affecting strength, with various methods available to minimize it. Erosion of stirrer blades during AMMC manufacture necessitates high-temperature lubricants, posing challenges for large-scale production. Future furnace designs must address reinforcement mixing rates, as current designs often lack consistent rates for optimal results in MMC production. Consistency in matrix alloy and reinforcement distribution, enhanced wettability, and mitigation of porosity and chemical reactions are imperative for achieving high-quality metal matrix composites.

8. Aluminum alloy LM25/A356: The 300 series aluminum alloys, exemplified by A356, are highly regarded in various industries for their exceptional castability, mechanical properties, and impressive strength-to-weight ratios. A356's composition includes silicon (6.5-7.5%), magnesium (0.3-0.45%), iron (0.2% max), copper (0.1% max), manganese (0.1% max), zinc (0.1% max), and titanium (0.2% max), along with trace metals. Commonly used in aerospace, automotive, and industrial machinery, A356's low shrinkage and high fluidity make it ideal for intricate structures with thin walls. This alloy

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

boasts superior corrosion resistance, weldability, and machinability, making it suitable for various casting methods, including die casting and sand casting. Its impressive mechanical properties, like high tensile and yield strength, further enhance its versatility.

Table 1.1: Chemical composition of Aluminium LM25 alloy

| Elements | Si | Fe | Cu | Mn | Mg | Ni | Zn | Ti | Pb | Sn | Al |
|-----------|------|-------|--------|------|-------|---------|---------|--------|--------|---------|-------|
| Weights % | 6.91 | 0.124 | 0.0091 | 0.01 | 0.395 | <0.0010 | <0.0010 | 0.0077 | 0.0021 | <0.0010 | 92.47 |

A356 exhibits a low melting point, good wear resistance, fluidity, and machinability, making it suitable for diverse applications, including automotive components, aircraft fittings, electronic cases, and more.

Table 1.2: Properties of A356 alloy material (ASM Hand book)

| Properties | Value |
|--|------------|
| Tensile Strength (MPa) | 221 to 232 |
| Yield Strength (MPa) | 165 to 185 |
| Elongation (%) | 5 to 6 |
| Elastic Modulus (GPa) | 72.4 |
| Hardness (HB) | 70 to 80 |
| Hardness (HV) | 58 to 60 |
| Density (kg/m ³) | 2.685 |
| Liquidus Temperature (°C) | 615 |
| Solidus Temperature (°C) | 577 |
| Linear Coefficient of Thermal Expansion at 20 to 300°C (µm/mK) | 23.5 |
| Thermal Conductivity at 25°C (W/mK) | 151 to 155 |

II. LITERATURE REVIEW

- 1. Introduction:** This chapter has attempted to provide a summary of the literature review on modern engineering facilitates the production of hybrid metal matrix composites with exceptional ductility, minimal weight, and impressive strength-to-weight ratios. These composites, increasingly applied in aerospace and automotive industries, offer advantages over standard metals. Ceramic particles, lighter than zinc, stainless steel, or copper, have replaced traditional materials in experiments on reinforced composites. Researchers commonly use matrix alloys for their unique properties comparable to aluminum. The choice of reinforcement material is crucial, meeting requirements for discontinuity, ductility, and corrosion resistance. Stir casting processes reduce costs and enhance output, leading to new hybrid composites with improved physicomachanical properties, controlled weight, and enhanced characteristics compared to conventional composites.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

- 2. Literature Summary:** After through quantitative literature review, the research extensively focuses on evaluating the mechanical, wear, machining, and corrosion properties of aluminum metal composites, with particular emphasis on the alloy AA 2024. This alloy, utilizing AA6061 as the matrix material, incorporates magnesium (Mg), titanium dioxide (TiO_2), and silicon carbide (SiC) particles at varying percentages. While the potential applications of these composites are vast, there is a gap in comprehensive literature addressing microstructure, mechanical properties, wear, machining, and corrosion behavior in aluminum alloy composites. To address this gap, the study employs the versatile stir casting method for manufacturing, aiming to contribute to the understanding of its mechanism in fabricating aluminum metal matrix composites. The fabrication process involves uniformly distributing reinforcement materials within the aluminum alloy matrix through stirring, chosen for its effectiveness, simplicity, and cost-effectiveness. The resulting composites undergo a thorough analysis covering mechanical properties (tensile strength, hardness, and ductility), machining properties (machinability, surface finish, and tool wear), wear resistance through tribological studies, and corrosion characteristics to assess their performance in diverse real-world scenarios.

III. RESEARCH METHODOLOGY

The research employs the stir casting process for fabricating a hybrid composite, utilizing LM25 alloy as the primary material and 1wt% Al_2O_3 and 1wt% Gr particles as reinforcements. The induction electric resistance furnace melts LM25 alloy bricks at 850 °C, and reinforcements are preheated to 400 °C (Al_2O_3) and 300 °C (Gr particles). A stir casting machine operates at 400 rpm for continuous stirring, and a preheated die shapes the well-mixed melt. The fabrication process involves careful melting of LM25 alloy, preheating reinforcements, timed addition of Al_2O_3 and Gr particles, and continuous stirring for 60 to 120 seconds. The final step is pouring the melt into a preheated die to shape the hybrid composite.

Key processing parameters include LM25 alloy melt temperature, preheating temperatures of reinforcements, stirring speed (400 rpm), and stirring duration (60 to 120 seconds). The study varies the weight fraction of Al_2O_3 and Gr particles to understand their impact on mechanical and structural properties.

Fabricated hybrid composites undergo comprehensive analysis, starting with microstructural examinations through metallographic studies. Mechanical testing assesses tensile strength, hardness, and wear resistance using standardized methods. Thermal analysis, using techniques like differential scanning calorimetry or thermogravimetric analysis, evaluates material stability under different temperatures.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

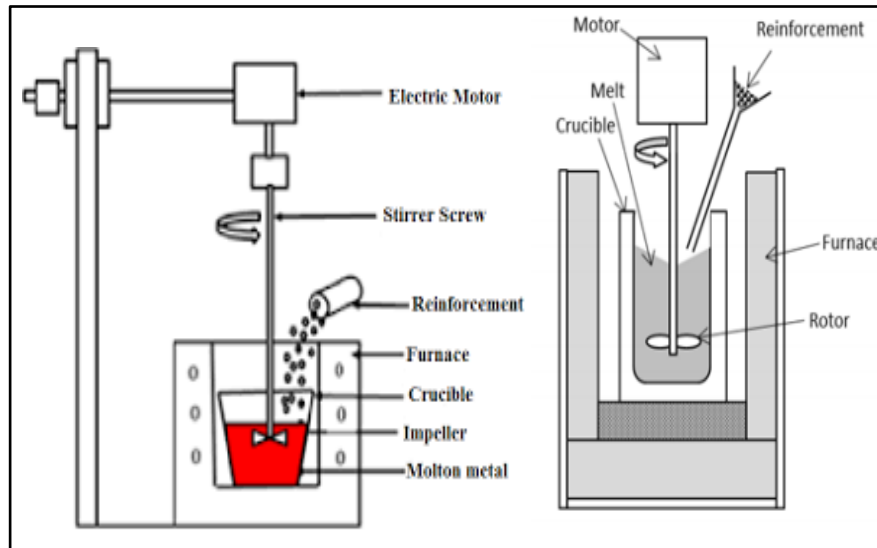


Figure 3.1: Stir casting process

Table 3.1: Parameters used for stir casting for sample preparation

| Sr. No. | Parameters | Value |
|---------|---|-----------|
| 1 | Pre-heated temperature of alumina particles (°C) | 350 |
| 2 | Pre-heated temperature of graphite particles (°C) | 350 |
| 3 | Temperature of melting charge (°C) | 850 |
| 4 | Stirrer speed (rpm) | 1500-1800 |
| 5 | Stirring time (sec.) | 30-40 |

Systematic data collection throughout the fabrication process includes material properties, processing parameters, and test results. Statistical methods analyze data, exploring variability, significance, and correlations between processing conditions and composite properties, ensuring a comprehensive understanding of the hybrid composite's characteristics. A similar method is employed for aluminum matrix composites with 3 and 5 wt% Al_2O_3 /1wt% Gr, with important manufacturing parameters outlined in Table 3.1:1. Prepared samples are machined and grained for further studies.

Table 3.2: Composition percentages taken in this experiment

| Sr. No. | Composite material | Coding |
|---------|--|------------|
| 1 | LM25+1% Gr+1,3,5% ZrO_2 | A1, A3, A5 |
| 2 | LM25+1% SiC+1,3,5% Al_2O_3 | B1, B3, B5 |
| 3 | LM25+1% MgO+2,3% SiC | C2, C3 |

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY
(A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

| | | |
|---|--|------------|
| 4 | LM25+1,3,5% SiC | D1,D3,D5 |
| 5 | LM25+1,3,5% MgO | E1, E3, E5 |
| 6 | LM25+1% Gr+1,3,5% Al ₂ O ₃ | F1, F3, F5 |
| 7 | LM25+1,3,5% Al ₂ O ₃ | G1, G3, G5 |

1. Work Flow Chart

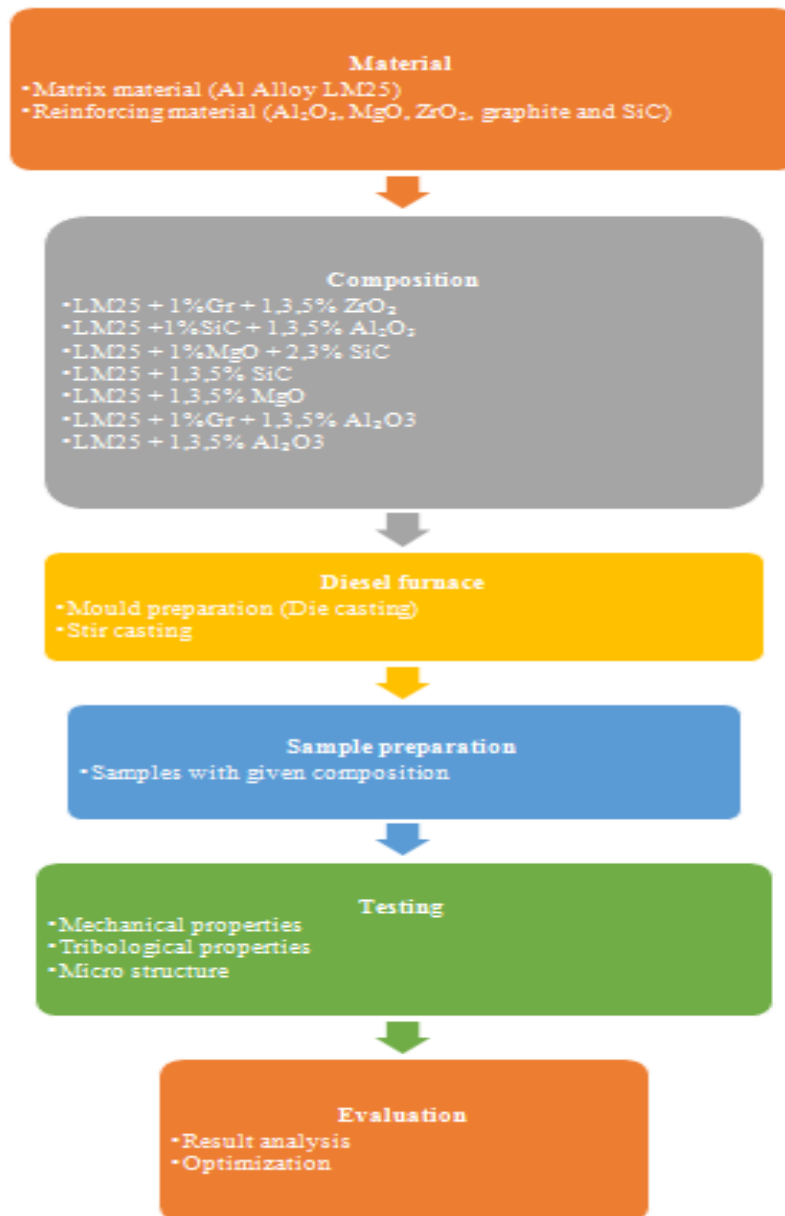


Figure 3.2: Flow chart of the current research.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

- 2. Mechanical Testing:** Unveiling Material Strength through Methodical Analysis. Tensile testing is a fundamental mechanical assessment employed to unravel the intrinsic properties of materials under axial loading. This examination provides valuable insights into a material's response to applied forces, enabling the determination of essential characteristics such as tensile strength, yield strength, and elongation. In the realm of materials science and engineering, tensile testing serves as a cornerstone for evaluating material behavior and performance employing a computerized 50KN Servo hydraulic universal testing machine. This advanced technology ensures precise control over testing parameters, including a strain rate of 1.0 mm/min, to capture the material's response at a controlled deformation rate. Tests were conducted at 30 °C to simulate real-world conditions, adhering to ASTM E8 standards for determining tensile properties. This standardized framework enhances the credibility and comparability of results, fostering cross-study analysis.

The meticulous approach includes three independent tests for each combination of material and testing parameters, ensuring reliability and accuracy. Real-time data from the testing machine, monitoring applied forces, deformation, and other metrics, forms the basis for a comprehensive analysis of the material's response to tensile loading.

Tensile testing provides crucial insights into material behavior, with parameters such as tensile strength, yield strength, and elongation explored. Tensile strength indicates the material's highest stress before fracturing, while yield strength signifies the transition from elastic to plastic deformation. Elongation quantifies a material's ability to deform before rupture, with higher values indicating greater ductility.

Conducting tests at room temperature aligns with practical scenarios, ensuring relevance to real-world applications and facilitating standardized testing conditions. The study, conducted with a state-of-the-art testing machine and adherence to standards, contributes valuable data to the field of materials science and engineering, enhancing our understanding of material behavior under axial loading.



Figure 3.3: Universal Testing Machine

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

- **Hardness Test:** Brinell hardness testing using a specialized apparatus to assess the hardness of materials by applying a known load on a hardened steel ball, measuring the resulting indentation diameter. To ensure accuracy, specimens are polished, and tests are conducted at three locations on each to account for potential variations in hardness. The average Brinell hardness values are calculated, providing a representative measure of overall hardness characteristics. This standardized and reliable testing method is significant in material science and engineering, offering insights into the macroscopic response of materials to applied forces.
- **Scanning Electron Microscopy:** The morphological testing section introduces Scanning Electron Microscopy (SEM) as a crucial method for visualizing the surface morphology of alloy composites. SEM utilizes a focused electron beam to scan cross-sections, generating high-resolution images that reveal structural and compositional attributes. The electron beam interacts with atoms, producing signals like secondary electrons, backscattered electrons, and characteristic X-rays. The controlled raster scan pattern allows for precise exploration of the surface, capturing even the minutest details. SEM's advantages include high resolution, revealing features at the nanoscale, and the ability to analyze composition through techniques like Energy-Dispersive X-ray Spectroscopy (EDS).

SEM plays a pivotal role in characterizing materials, aiding in optimizing processing techniques and understanding material behavior. Challenges such as sample preparation and electron beam-induced damage are acknowledged, emphasizing the careful optimization of imaging conditions. Overall, SEM stands as a powerful and indispensable tool for researchers striving to unravel the mysteries of material behavior and enhance material performance across diverse applications.

- **X-Ray Diffraction:** The synergies of material science and engineering through the incorporation of reinforcement materials into alloy composites, ushering in enhanced material properties. Reinforcement materials, including ZrO₂, SiC, Al₂O₃, and MgO, with diverse chemical compositions and microstructures, play a pivotal role in shaping the fundamental characteristics of the resulting composites. The precision crafting of alloy composites allows for tailored material solutions, influencing not only mechanical properties but also thermal, electrical, and wear characteristics.

Optimizing the performance of alloy composites is a meticulous process that hinges on surface chemistry, dispersion of reinforcement particles, and the adhesion of the metal matrix onto these particles. A profound understanding of surface chemistry is crucial for achieving optimal dispersion, ensuring a uniform distribution of reinforcement materials and preventing concerns like clustering or agglomeration. Equally important is the robust adhesion between the metal matrix and reinforcement particles, directly impacting the strength and durability of the composite.

X-ray diffraction (XRD) spectroscopy emerges as a linchpin in the characterization and optimization of alloy composites. Traditionally used for studying crystalline and amorphous structures, XRD employs X-rays to probe the crystallographic features of materials. This non-destructive technique provides

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

valuable insights into the structural properties of materials, offering a versatile platform to study parameters such as phase composition, crystallite size, and orientation. Despite challenges in sample preparation and data interpretation, the simplicity, reliability, and non-destructive nature of XRD make it a widely accepted and accessible method for material characterization.

3. Design of Experiments: Statistical analysis is a efficient approach for designing experiments to optimize process parameters. The goal is to determine the optimal values for factors that predict the maximum strength of composite materials. The design of experiments involves one or more factors to achieve optimized values and predict the best outcome for composite strength. The experimental results are succinctly explained through the design of predicted information. In the Design of Experiment (DOE), process models, typically linear or quadratic, are commonly used. These models employ a black box technique to analyze multiple controlled parameters and assess one or more output responses. The relationships between input and output parameters are captured through first and second-order terms in the experimental model.

- **Taguchi method of Design of Experiments:** The Taguchi Method, also known as Taguchi Quality Engineering or Taguchi Robust Design, is a statistical approach developed by Dr. Genichi Taguchi in the 1950s. It aims to optimize the design and manufacturing process of a product or system. The method focuses on building quality into the product or process rather than inspecting it at the end. Key steps in the Taguchi Method include problem definition, parameter design, tolerance design, and robustness design. It identifies key factors affecting quality, determines optimal factor settings, establishes tolerances, and ensures robustness against variation.

Benefits of the Taguchi Method include cost-effective optimization of production processes, leading to improved product quality and performance. It allows targeted improvements, enhancing customer satisfaction, loyalty, and manufacturer profitability. The method is known for its simplicity and ease of implementation, making it accessible to a wide range of industries. Additionally, the Taguchi Method can be employed for pre-production improvements by identifying and addressing key factors influencing quality before manufacturing begins, resulting in significant cost savings.

IV. EXPERIMENTAL WORK

1. Experimental Procedure: This chapter delineates the meticulous experimental procedures employed to fabricate hybrid metal matrix composites (HMCs) using the stir casting technique. The investigation is centered on elucidating the influence of distinct weight fractions of reinforcing agents, specifically graphite and ceramic particles, on the mechanical and thermal attributes of the resulting composite material. LM25, a well-established aluminum alloy, serves as the base metal for this study due to its renowned engineering applications. The manipulation of stirring parameters, including rotational speed and duration, plays a pivotal role in achieving homogeneous mixing of reinforcing particles within the matrix.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY
(A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

2. Materials: The materials utilized in this study include:

Base Metal: LM25 Aluminum Alloy

Reinforcing Particles:

Graphite: Integrated at a consistent weight fraction of 1%. Particle size ranges



Figure 4.2: shows the pouring of molten metal in preheated mould for casting I section from 100 to 200 μm .

Ceramic Particles: Al_2O_3 , MgO , SiC , ZrO_2 , with weight fractions of 1%, 3%, and 5%. Particle size ranges from 100 to 200 μm .

3. Experimental Procedure: The experimental procedure unfolds through the following stepwise approach, as illustrated in Figure 4.1:

Stir Casting Process

- **Preheating of Reinforcing Particles:** Both graphite and ceramic particles undergo preheating at 350 degrees Celsius. This thermal treatment eliminates residual moisture and volatile entities, fostering superior interfacial adhesion between particles and the molten LM25 matrix.
- **Preheating of Base Metal:** LM25 base metal undergoes meticulous preheating to an optimal temperature of 850 degrees Celsius within a controlled atmosphere. This preheating procedure ensures complete alloy melting and enables the efficacious incorporation of reinforcing particles during subsequent stirring.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

- **Furnace Loading and Crucible Placement:** An appropriate refractory crucible is chosen to house the preheated LM25 alloy. The alloy is carefully placed into the crucible, which is then positioned within the furnace for further processing. Pouring of Molten Metal
- **Reinforcement Integration and Stirring Parameters:** Upon reaching the designated preheating temperature, both preheated reinforcing particles (graphite and ceramic) are introduced into the crucible containing the base metal. Stirring commences using the stir casting apparatus, maintaining a consistent speed (1200 to 1500 rpm) to ensure uniform dispersion of particles within the molten matrix. Stirring duration is regulated (30 to 40 seconds) to avoid overagitation and unwanted particle agglomeration.
- **Composite Specimen Formation:** Post-stirring, the molten composite amalgam is poured into pre-fabricated and preheated molds designed per the desired specimen dimensions. Stringent precautions are observed to prevent external contamination during this critical phase.
- **Solidification and Cooling:** The cast composite mixture undergoes gradual cooling, culminating in solidification within the molds. This gradual solidification promotes even dispersion of reinforcing particles throughout the composite structure.



Figure 4.5: Specimen before tensile test.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS



Figure 4.6: Actual samples of LM 25 + 1%, 3% and 5% MgO after tensile test



Figure 4.7: Actual samples of LM 25 + 1%, 3% and 5% Al_2O_3 + 1% Gr after tensile test

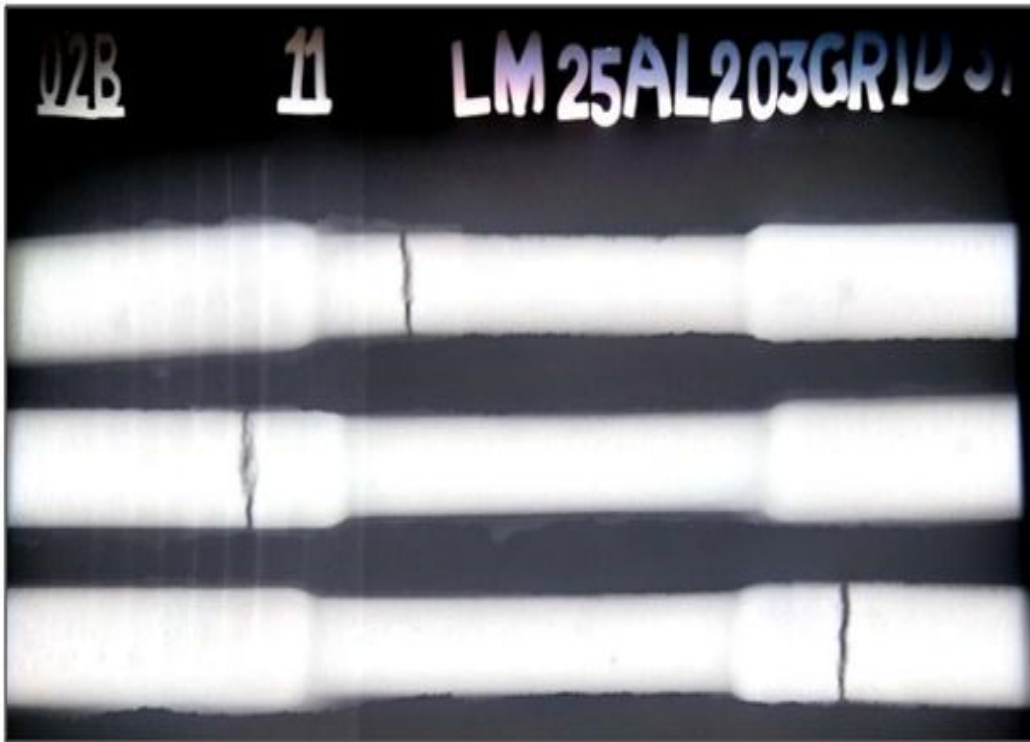
- Specimen Extraction:** After complete solidification, specimens are meticulously removed from the molds. Subsequent procedures involve machining, grinding, and polishing to achieve prescribed dimensional precision and surface finish.
4. **Radiographic Testing:** Non-Destructive Testing (NDT) is pivotal for ensuring the integrity and quality of materials in engineering applications. Radiographic testing, a specific NDT method, proves valuable for assessing aluminum cylindrical I-section samples adhering to the E8 standard. This method involves X-rays to scrutinize the internal structure, primarily for detecting and evaluating porosity.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

- **Importance of Radiographic Testing:** Radiographic testing provides a non-destructive and comprehensive assessment of the material's internal structure. It is particularly advantageous in industries where structural reliability is paramount, such as aerospace, automotive, and construction. The automotive industry, utilizing aluminum alloys to reduce weight, requires stringent quality control to ensure safety and reliability. Radiographic testing allows for the assessment of porosity without destructive testing, maintaining rigorous quality control.
- **Radiographic Examination Procedure:**
Radiographic testing involves several key steps:
 - **Preparation:** The aluminum cylindrical I-section sample is cleaned and positioned for examination, free from debris.
 - **X-ray Source and Detector:** X-rays are emitted from a machine, penetrating the material. On the other side, a detector records the X-rays after passing through the specimen.
 - **Exposure:** X-rays pass through the specimen, and the detector records their intensity. The resulting image provides information about the internal structure.
 - **Imaging:** The X-ray images, or radiographs, show variations in intensity, indicating differences in material density and the presence of defects like porosity.
 - **Interpretation:** Trained technicians analyze radiographs. Porosity appears as dark spots or areas with reduced intensity due to X-rays passing more easily through voids.
 - **Evaluation:** Porosity size, shape, and distribution provide information about the aluminum alloy casting's quality. Larger or clustered porosity may indicate significant defects.
 - **Acceptance Criteria:** Radiographic testing adheres to industry standards and specifications, providing guidelines for evaluating radiographs and determining if porosity levels are acceptable.



(a)



(b)

Figure 4.8: LM 25 + 1% Gr + 1%, 3% and 5% Al_2O_3 after tensile test (a) Actual samples (b) Images from Radiographic testing machine.

V. RESULTS AND DISCUSSIONS

- 1. Morphological Properties:** It is widely accepted fact that the mechanical properties of hybrid composites are basically influenced by the reinforcement defects, size of the reinforcements, distribution of reinforcements, surface irregularity and reinforcement matrix bonding. This demands for the study of microscopic analysis to understand the microstructural characteristics.
- Figure 5.1 indicates the SEM image with magnification of 25000x, indicating the morphology of LM 25 alloy composite formed using ceramic micro-particles of graphite (1%) and ZrO_2 (3%).

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

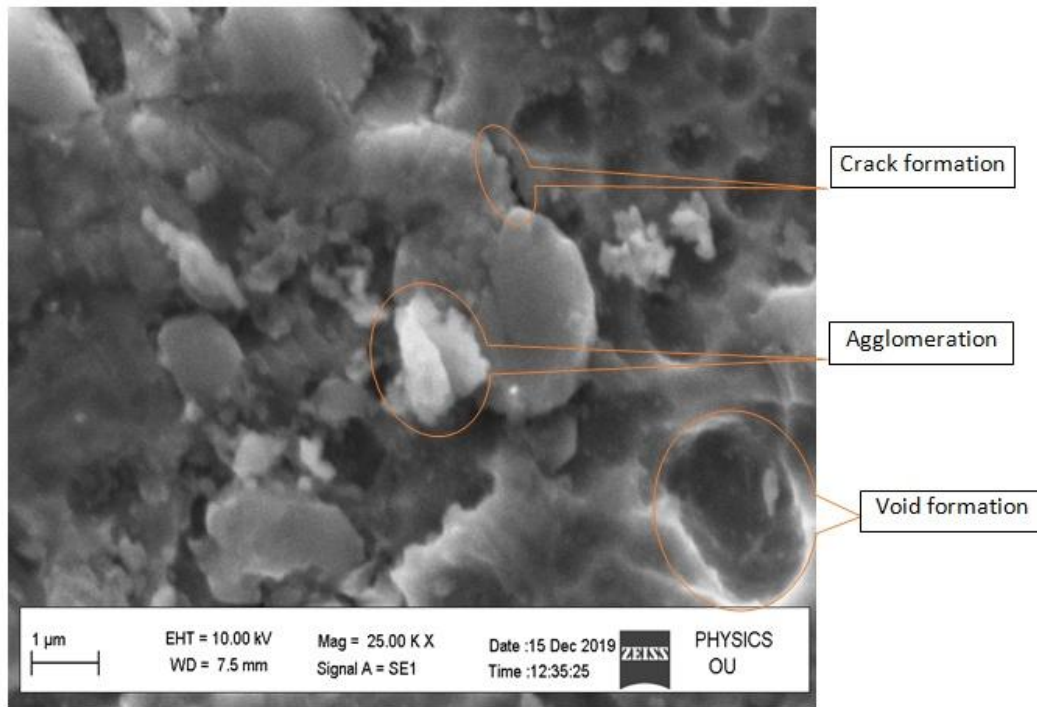


Figure 5.1: A 25000x SEM image of composite of LM 25 alloy with 1% Gr and 3% ZrO₂. The various defects like crack formation, void formation and agglomeration of reinforcement material are visible in

Figure 5.1, in black, dark grey and white colour, respectively. The void formation and crack propagation makes way for the de-bonding between the matrix material and the reinforcement material that result in the reduction of the ductility.

The agglomeration of reinforcement material with its dimensions, The dimensions of agglomeration ranges from 100 nm to 300 nm.

Figure 5. indicates the SEM image with magnification of 2500x, indicating the morphology of LM 25 alloy composite formed using ceramic micro-particles of graphite (1%) and ZrO₂ (1%, 3%, and 5%).

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

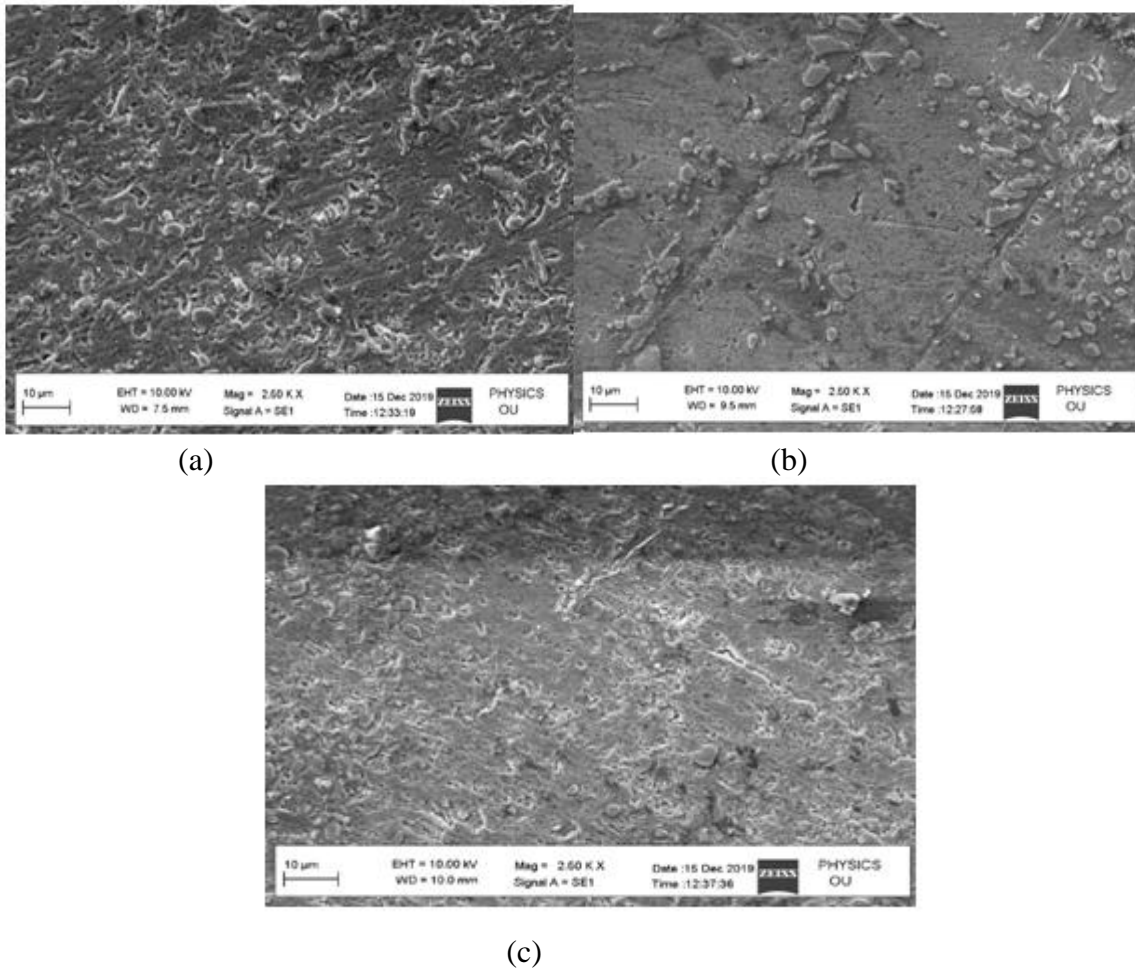


Figure 5.2: 2500x SEM image of composites of LM 25 alloy with 1% Gr and ZrO_2 of (a) 1% (b) 3% (c) 5%

Upon careful observation can be seen from the comparison of (a) (b) and (c) of

Figure 5. that with the increasing amount of reinforcement material, the whitish spots, dark grey polygons and blackish lines are increasing. This is the indication that with the increasing amount of reinforcement material, the formation of morphological defects like agglomeration, crack formation and void formation is increasing.

Consider the

Figure 5. indicating the SEM images at 10,000x zoom of the composites of LM 25 alloy with 1% Gr and 5% ZrO_2 , 1% SiC and 5% Al_2O_3 , and 5% Al_2O_3 .

It can be seen that the dominant microstructural defect for LM 25 alloy with 1% Gr and 5% ZrO_2 is micro-void formation, while that for LM 25 alloy with 1% SiC and 5% Al_2O_3 and 5% Al_2O_3 is agglomeration of reinforcement particles.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

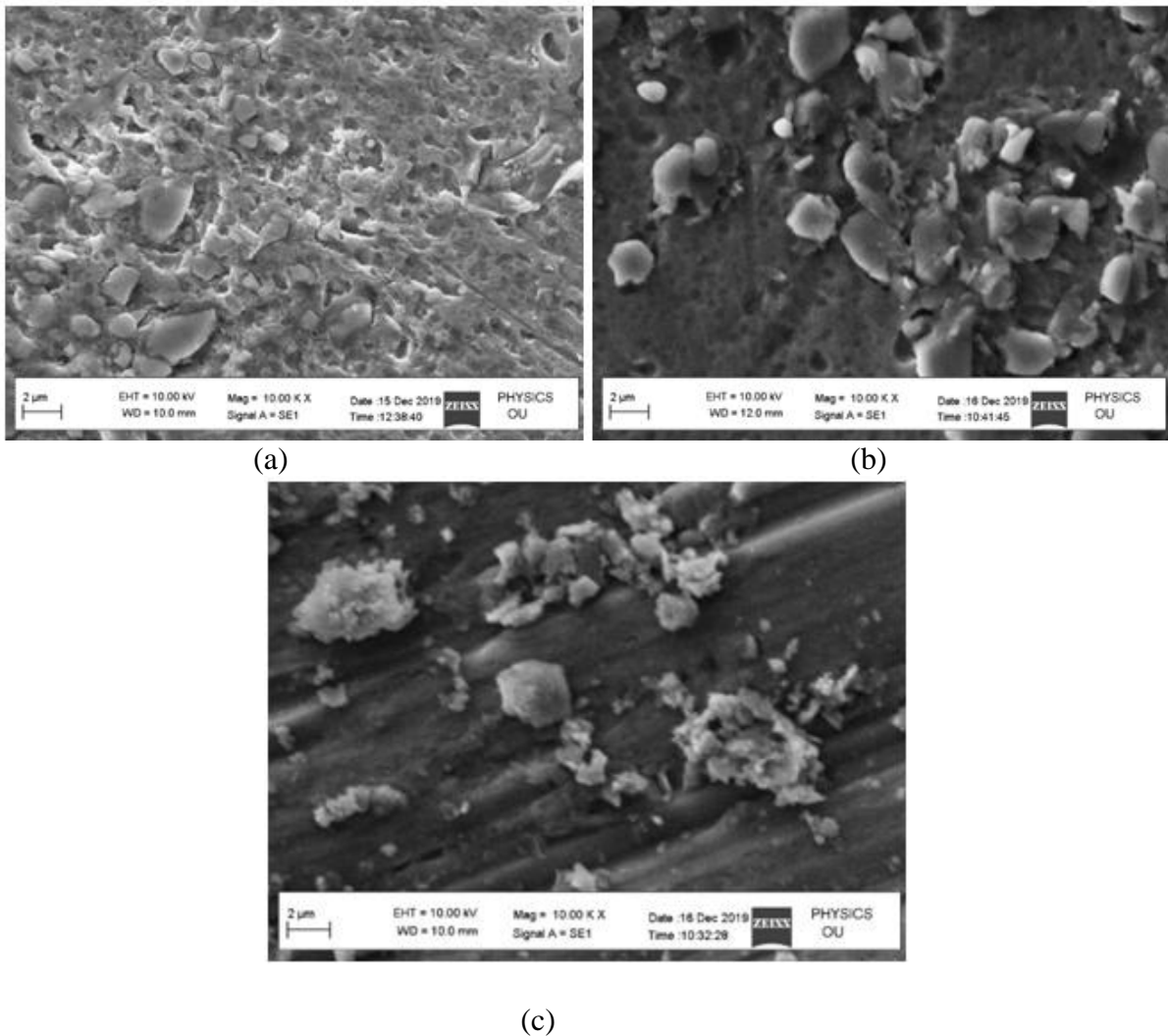


Figure 5.3: A SEM image of composites of LM 25 alloy with (a) 1% Gr and 5% ZrO_2 (b) 1% SiC and 5% Al_2O_3 (c) 5% Al_2O_3

- 3. Microstructural Properties:** The microstructural analysis is considered by using X-ray Diffraction (XRD) technique and is plotted for the variation of counts with 2θ angle, as shown in
- 4. Figure 5..** The primary observation indicates the existence of the multiple peaks which further points to the fact that the powders of the alloy composites have maintained its crystalline structure even after addition of material with different chemical composition.

The XRD plot of the alloy LM 25 is used for reference from earlier literature. The composite of LM 25 alloy with Al_2O_3 (

Figure 5. (a)) and with graphite (Gr) and ZrO_2 (

Figure 5. (d)) has indicated that the peak located between angle 77° to 78° is having higher intensity which is same as that for the LM 25 alloy itself. This means that

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

the addition of Al_2O_3 and combination of graphite (Gr) and ZrO_2 , has little effect on microstructural properties of LM25 alloy

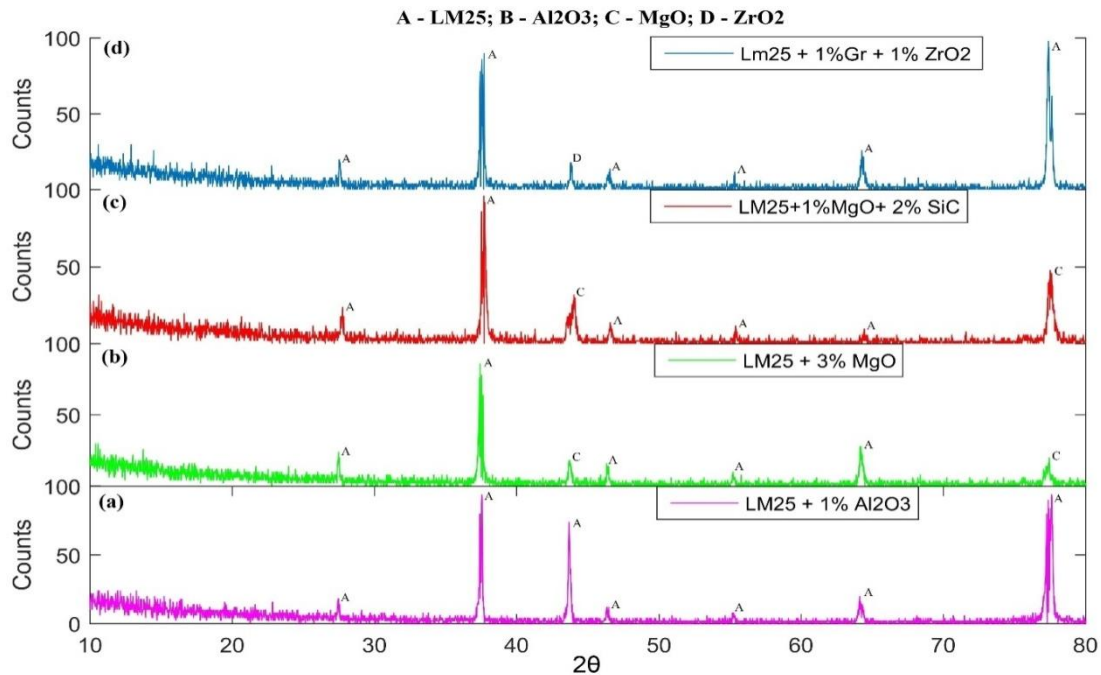


Figure 5.4: XRD data for composite of LM 25 alloy with (a) 1% Al_2O_3 (b) 3% MgO (c) 1% MgO and 2% SiC (d) 1% Gr and 1% ZrO_2

The

Figure 5. (b) and (c) indicates that the addition of MgO and SiC, in LM 25 alloy has resulted in decrement in the counts for the peak located between angle 77° to 78° which indicates that microstructural properties of LM 25 alloy are affected by the addition of MgO and SiC. The addition of graphite is usually found to indicate its presence in the form of a peak between angle of 25° to 30° . that is absent in fig 5.4 (d) which reveals that graphite has very little or negligible effect on LM25 alloy. It is worthwhile to note that the peak located between angle 43° to 44° is also considerably affected by the presence of all the reinforcement materials (used in this study) in LM 25 alloy. Consider the variation of XRD results for LM25 alloy composite formed using Al_2O_3 as a reinforcement material.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

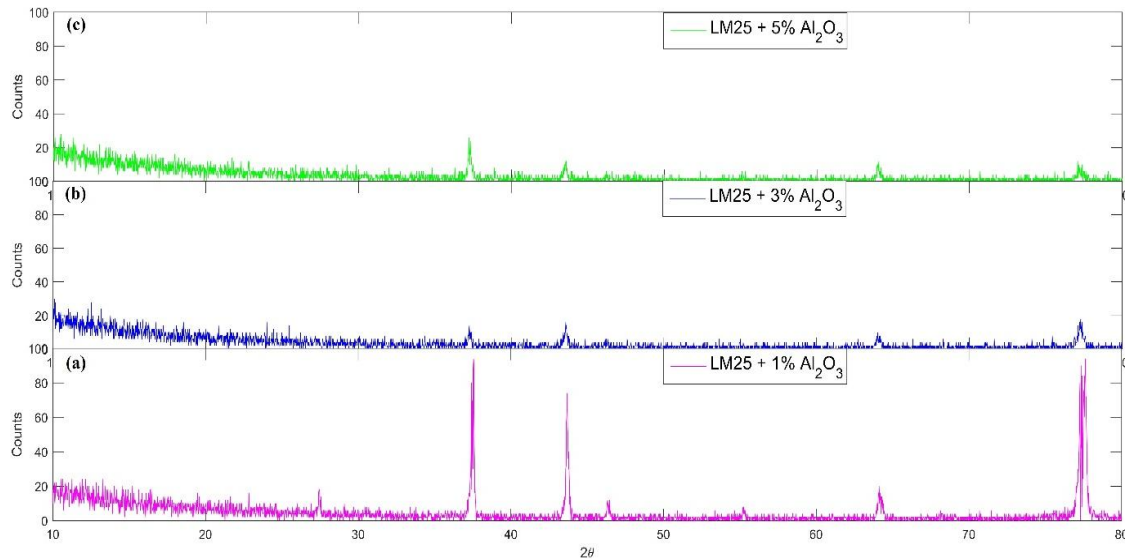


Figure 5.5: XRD data for composite of LM 25 alloy using Al_2O_3 with (a) 1% (b) 3% (c) 5% Al_2O_3 . The

Figure 5. indicates that with increase in the amount of reinforcement of the Al_2O_3 , the intensity of the peaks has decreased. Consider the variation of XRD results for LM25 alloy composite formed using 1% Gr and Al_2O_3 as a reinforcement material.

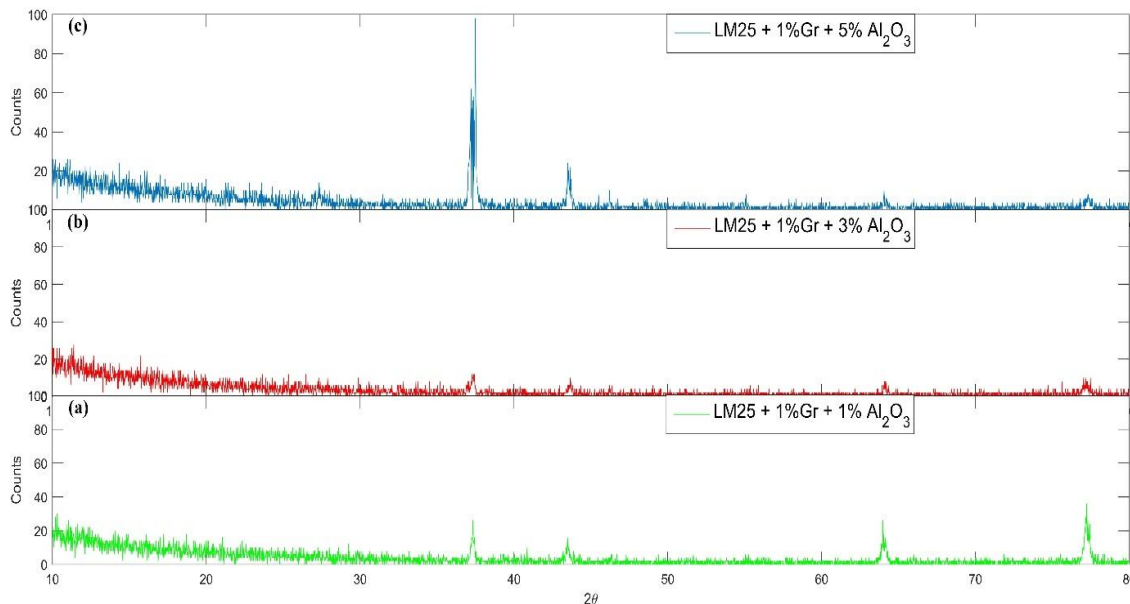


Figure 5.6: XRD data for composite of LM 25 alloy using 1% Gr and Al_2O_3 with (a) 1% (b) 3% (c) 5% Al_2O_3 .

5. Tensile Strength: To start with the discussion of results of tensile strength, a set of secondary composites of LM25 aluminium alloy formed by addition ceramic micro-particles in the form of SiC, MgO and Al_2O_3 are considered.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

6. Figure 5 indicates the variation of tensile strength with the amount of reinforcement of ceramic micro-particles made of SiC, MgO and Al_2O_3 .

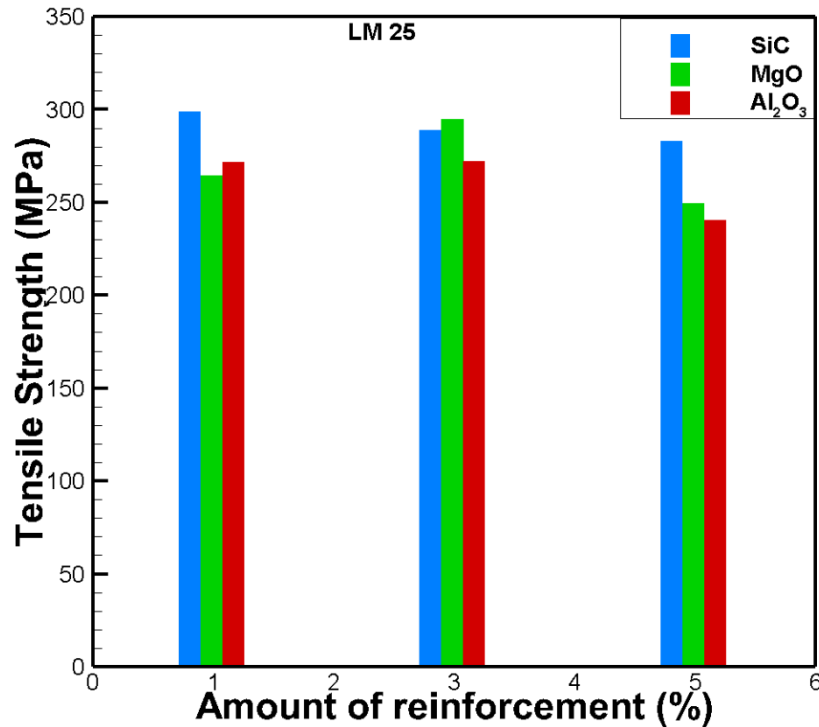


Figure 5.7: Variation of tensile strength with amount of reinforcement of SiC, MgO and Al_2O_3 in LM25 alloy

Highest tensile strength is observed for the LM25 composite sample with 1% reinforcement of SiC while the lowest tensile strength is observed for the LM25 composite sample with 5% reinforcement of Al_2O_3 . The decreasing trend of variation of tensile strength with increase in the amount of reinforcement is observed for the LM25 composite samples with reinforcement of SiC and Al_2O_3 . The tensile strength of the LM25 composite samples with reinforcement of MgO first increases and then decreases with increase in the amount of reinforcement.

The enhanced tensile strength in certain materials can be attributed in part to the accelerated work hardening rate due to particle-containing components. Moreover, factors such as increased load-bearing capabilities and mismatch strengthening brought about by these micro-particles contribute to this strength enhancement. Alongside these factors, grain refinement and particle reinforcement further play pivotal roles. Upon cooling from the solidification temperature, it is expected that the thermal mismatch stress will lead to a higher dislocation density within the material matrix. Such dislocations can induce localized stresses, fortify the matrix's strength, and consequently amplify the overall strength of the composite.

However, a contrasting trend is observed when examining the influence of MgO content on tensile strength. As the MgO content escalates, the composite tends to exhibit greater particle agglomeration, an increased prevalence of defects, and elevated micro-

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

porosity levels. Notably, the MgO particles serve as catalysts for the nucleation of porosities. With a surge in MgO content, there's a concurrent rise in micro-porosities, potentially diminishing the flow stress within the composite. Compounding this effect, the distinct thermal expansion coefficients of LM25 and MgO introduce additional dislocations and defects around MgO particles. This situation might lead to interface debonding, thereby diminishing the tensile strength, especially in composites with a higher MgO volume ratio.

Similar findings emerge when evaluating the tensile strength variations in LM25 aluminum alloy, particularly when integrated with ceramic micro-particles like SiC and Al_2O_3 .

Further exploration focuses on ternary composites of the LM25 aluminum alloy, combining ceramic micro-particles like ZrO_2 and Al_2O_3 with the secondary alloy of LM25 and 1% Graphite (Gr). Figure 5.9 sheds light on the tensile strength fluctuations based on the quantity of ZrO_2 and Al_2O_3 ceramic micro-particle reinforcements. Interestingly, the LM25 composite featuring a 3% Al_2O_3 reinforcement showcased the highest tensile strength, while the counterpart with a 1% ZrO_2 reinforcement exhibited the least. Delving deeper, the tensile strength trend in LM25 composites with ZrO_2 reinforcement demonstrated a consistent increase with rising reinforcement levels. In contrast, the tensile strength trajectory for LM25 composites with Al_2O_3 reinforcement depicted an initial decline followed by an upswing as the reinforcement increased.

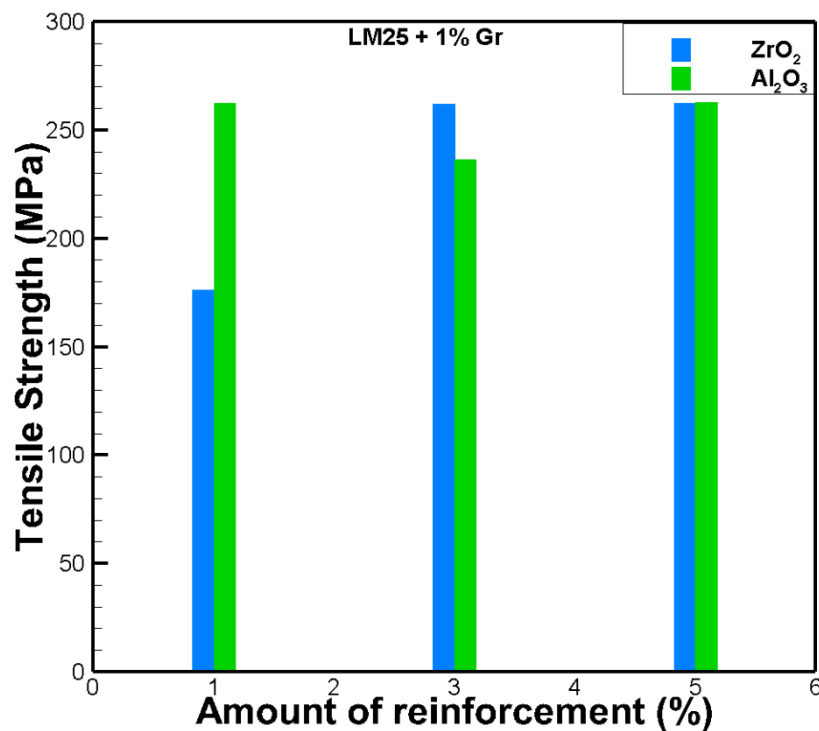


Figure 5.8: Variation of tensile strength with amount of reinforcement of ZrO_2 and Al_2O_3 in LM25 + 1% Gr alloy

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

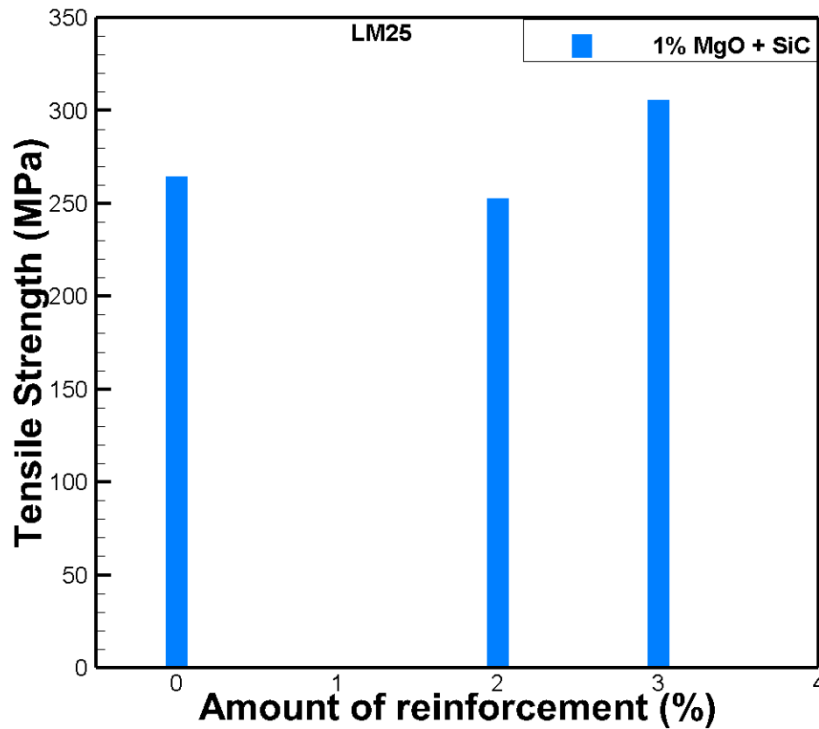


Figure 5.9: Variation of tensile strength with amount of reinforcement of SiC in LM25 + 1% MgO alloy

Highest tensile strength is noticed for the LM25 composite sample with 3% reinforcement of SiC while the lowest tensile strength is detected for the LM25 composite sample with 2% reinforcement of SiC. The tensile strength of the LM25 composite trials thru reinforcement of SiC in 'LM25 + 1% MgO' composite, first decreases and then increases with upsurge in the quantity of reinforcement.

Consider a set of ternary composites of LM25 aluminium alloy formed by addition ceramic micro-particles in the form of Al_2O_3 to the secondary alloy of LM25 containing micro-particles with 1% Gr (Graphite) and 1% SiC.

Figure 5. directs the dissimilarity of tensile strength through the quantity of reinforcement of ceramic micro-particles made of Al_2O_3 .

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

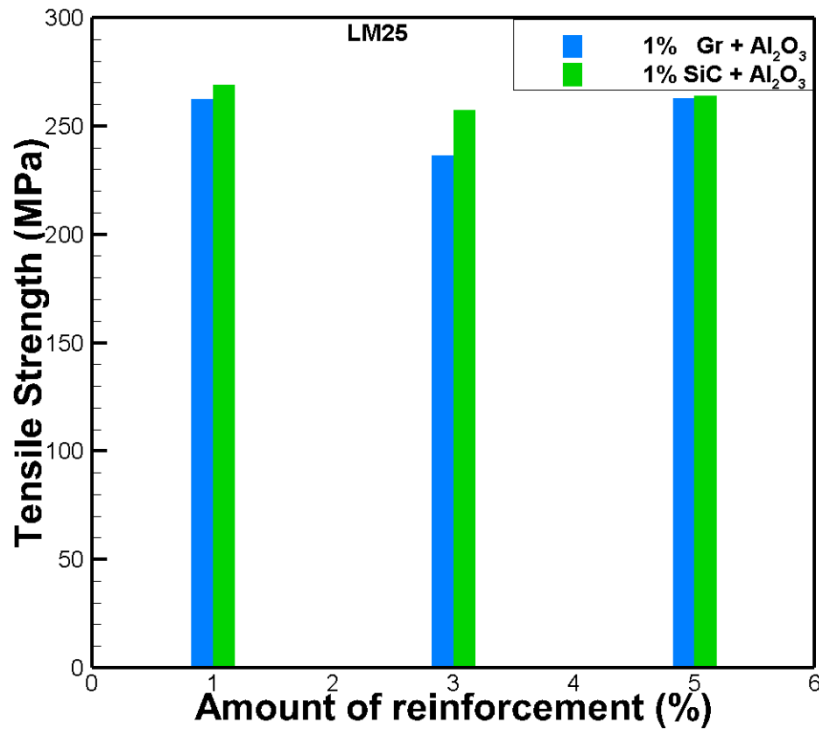


Figure 5.10: Variation of tensile strength with amount of reinforcement of Al₂O₃ in LM25 + 1% Gr and 1% SiC alloy

Highest tensile strength is observed for the LM25 composite sample with 1% reinforcement of Al₂O₃ in LM25 + 1% SiC while the lowest tensile strength is noticed for the LM25 composite sample with 3% reinforcement of Al₂O₃ in LM25 + 1% Gr. The tensile strength of the both the types of ternary composites of LM25 alloy with reinforcement of Al₂O₃ first decreases and then increases with upsurge in the quantity of reinforcement.

Lastly, Figure 5.10 provides insights into ternary composites of the LM25 aluminum alloy infused with ceramic micro-particles such as MgO and SiC, alongside the secondary alloy of LM25 and 1% MgO.

7. **Brinell hardness number:** It is good to start the discussion of results of Brinell hardness number with a set of secondary composites of LM25 aluminium alloy formed by addition ceramic micro-particles in the form of SiC, MgO and Al₂O₃.
8. Figure 5. Indicates the variation of Brinell hardness number with the amount of reinforcement of ceramic micro-particles made of SiC, MgO and Al₂O₃.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

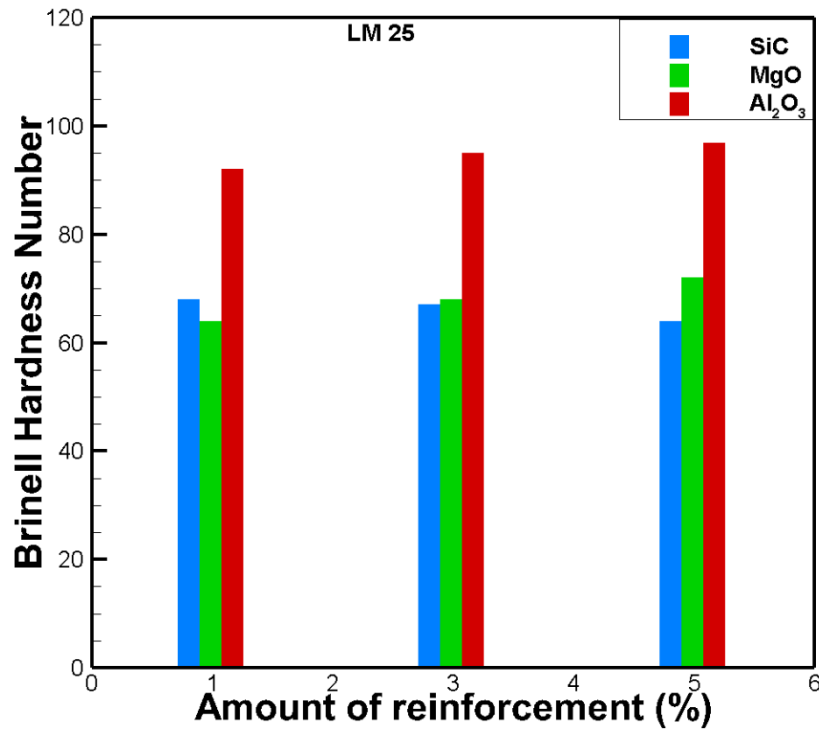


Figure 5.11: Variation of Brinell hardness number with amount of reinforcement of SiC, MgO and Al₂O₃ in LM25 alloy

Highest Brinell hardness number is observed for the LM25 composite sample with 5% reinforcement of Al₂O₃ while the lowest Brinell hardness number is noticed for the LM25 composite sample with 1% reinforcement of MgO and that with 5% reinforcement of SiC. The increasing trend of variation of Brinell hardness number with increase in the amount of reinforcement is noticed for the LM25 composite samples with reinforcement of MgO and Al₂O₃. The Hall-Petch mechanism, particle firming effects, and grain refinement, which act as barriers to the motion of dislocations, are primarily responsible for the observed increase in the hardness levels with increasing amount of micro-particles. A significant number of disorders and thermally induced residual stresses are produced at the particle-matrix edge during the solidification process because the coefficient of thermal expansion (CTE) of the reinforcing particles is lower than that of the aluminium alloy. The level of hardness is increased by the thermal stresses at the particle-matrix contact, which make plastic deformation more challenging.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

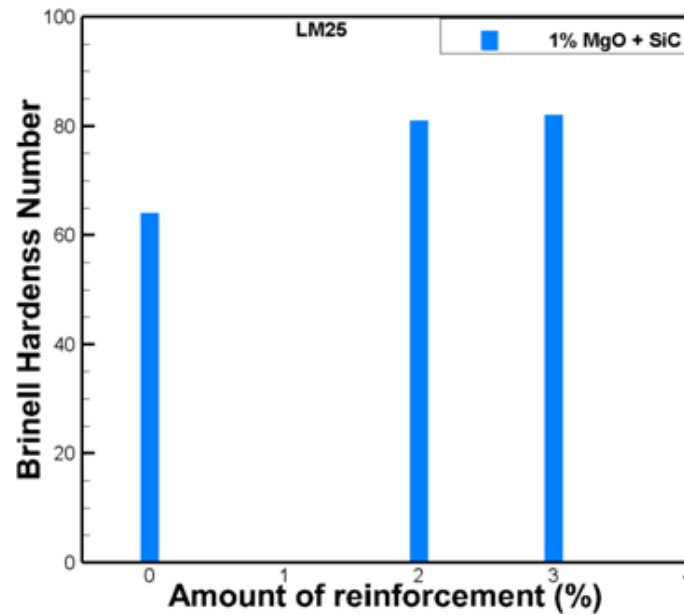


Figure 5.12: Variation of Brinell hardness number with amount of reinforcement of SiC in LM25 + 1% MgO alloy.

Moreover, the data reveals an interesting trend in the variation of Brinell hardness number with the increase in the amount of reinforcement. For LM25 composite samples with Al_2O_3 in LM25 + 1% Gr, there is a noticeable increasing trend, indicating that a higher percentage of graphite reinforcement corresponds to a rise in hardness. This trend suggests that graphite, in this particular composite, has a positive influence on hardness, possibly due to its unique mechanical properties or its interaction with the LM25 alloy matrix.

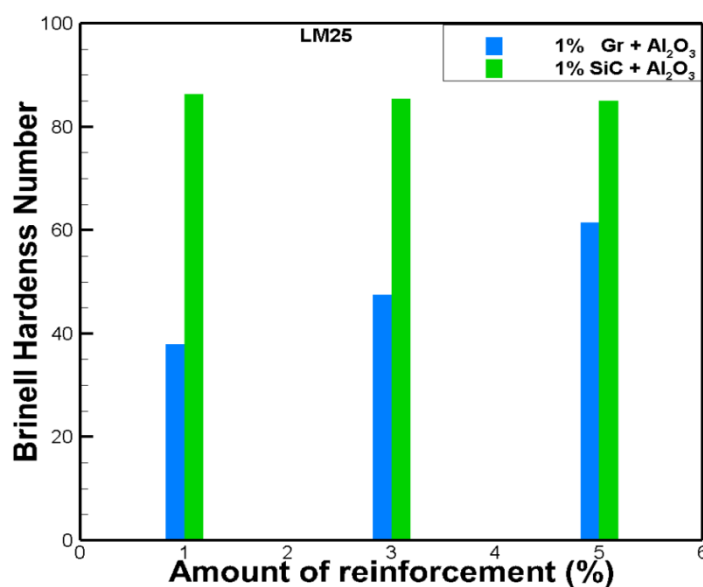


Figure 5.13: Variation of Brinell hardness number with amount of reinforcement of Al_2O_3 in LM25 + 1% Gr and 1% SiC alloy

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY
(A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

Conversely, the opposite trend is observed for LM25 composite samples with Al_2O_3 in LM25 + 1% SiC, where the increase in SiC reinforcement does not lead to a proportional increase in Brinell hardness number. This counterintuitive observation could be attributed to saturation effects or the intricate interplay between SiC and the LM25 alloy, emphasizing the importance of carefully balancing reinforcement content for optimal material performance.

The detailed analysis of Brinell hardness numbers highlights the nuanced effects of different reinforcement types and amounts on the mechanical properties of LM25 composites, providing valuable insights for the optimization of these materials in various industrial applications.

VI. CONCLUSIONS

Aluminum alloy hybrid metal matrix composites are considered as future generation potential materials for many engineering applications. A356/LM25 alloy reinforced with ZrO_2 , Al_2O_3 , SiC, MgO and Gr particulates with varied weight percentage (1, 3 and 5) and particle size of 100–200 μm , are used to fabricate the hybrid composites by using stir casting method. For morphological study, advanced techniques like SEM and XRD are employed while tensile and Brinell hardness test are carried out to determine tensile strength and the hardness of the alloy composite, respectively.

1. SEM images revealed that agglomeration is a prevalent manufacturing defect in LM25 alloy composites, intensifying with increased reinforcement material. This defect significantly impacts the structural integrity of the composite.
2. Impact: Manufacturing defects like void and crack formation, highlighted in SEM images, play a significant role in enhancing material properties, particularly tensile strength. XRD results confirmed the maintenance of the base alloy's crystalline nature despite the addition of reinforcement material.
3. The study determined that the highest tensile strength (305.7 MPa) was obtained for the sample with the composition of LM25+1%MgO+3% SiC, and the highest hardness (96.95 BHN) was achieved for the sample with the composition of LM25+5% Al_2O_3 . These findings provide valuable insights into the specific combinations of micro particles that significantly enhance the mechanical properties of the composite material.
4. Alloy additives such as Al_2O_3 and a combination of graphite (Gr) and ZrO_2 had minimal impact on LM 25 alloy microstructure, while the addition of MgO and SiC resulted in noticeable changes. The study identified factors such as work hardening, load carrying, mismatch strengthening, grain refinement, and particle strengthening as contributors to the improvement in strength.
5. Variations in tensile strength and Brinell hardness in secondary composites were attributed to the Hall-Petch mechanism, particle strengthening effects, and grain refinement. The study provides comprehensive insights into the factors influencing dislocation motion and thermal stresses at the particle-matrix interface.

EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY
(A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

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EXPERIMENTAL INVESTIGATION OF HYBRID METAL COMPOSITES OF ALUMINIUM ALLOY (A356/LM25) REINFORCED BY MICRO-SIZED CERAMIC PARTICLES BY STIR CASTING PROCESS

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List of Publications from This Work

➤ International Journal Paper Publications

- 1) **Rahamathulla Khan Sarvani**, Dr. Mohd Mohinoddin and Dr. L. Siva Ramakrishna, Experimental Analysis of Al LM25 Composite with Various Reinforcement Compositions Using Stir Casting, *International Journal of Mechanical Engineering and Technology (IJMET)*, 14(03), 2023, pp. 23-30
- 2) **Rahamathulla Khan Sarvani**, Dr. Mohd Mohinoddin and Dr. L. Siva Ramakrishna, "Characterization and Mechanical testing of Hybrid Metal Composites of Aluminium Alloy (A356/LM25) Reinforced by Micro-sized Ceramic Particles", *Journal of The Institution of Engineers (India): Series c(Scopus Indexed)* Accepted.

➤ Conference Paper Publications

- 1) **Rahamthulla Khan Sarvani**, Mohd Mohinoddin, L. Siva Ramakrishna, "Experimental analysis of Al composites toughened by aluminium oxide and graphite using stir casting progression", *Materials Today: Proceedings*, 2023, ISSN 2214- 7853, <https://doi.org/10.1016/j.matpr.2023.05.221>.