

Effect of cast iron chill thickness on the microstructure and mechanical properties of cryogenically solidified LM13 Aluminum Alloy/10 wt.% Nano-ZrO₂ composites

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Abstract

Aluminum matrix composites (AMCs) are used in aerospace, automotive, and marine industries for their strength and wear resistance. In this study, LM13 aluminum alloy was reinforced with 10 wt.% nano-ZrO₂ by stir casting combined with cryogenic solidification. Cast iron chills of five thicknesses (5, 10, 15, 20, and 25 mm) were used to study their effect on microstructure and mechanical properties. Tensile strength rose from 145 MPa at the 5 mm chill to 200.63 MPa at the 25 mm chill, a gain of about 38.4%. Micro-Vickers hardness rose from 71.20 VHN to 99.60 VHN, a gain of about 39.9%. These values are averages of three tensile tests and five hardness indentations per condition. The gains track the finer microstructure seen with thicker chills, most likely due to a faster cooling rate; cooling rate itself was not measured, so this remains an interpretation rather than a proven mechanism. The percentage gains describe the trend across the 5–25 mm series, not a comparison against an unreinforced LM13 control, since such a control was not separately cast in this work. Among all conditions tested, the 25 mm chill gave the best overall hardness and tensile strength. Combining nano-ZrO₂ reinforcement with chill-assisted, cryogenically accelerated solidification appears promising for improving LM13 composites. Since the study followed a single-factor design, the result should be read as a demonstrated trend rather than a statistically confirmed synergistic effect; dedicated control experiments are needed to confirm this.

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1. Introduction

Metal matrix composites (MMCs) combine the ductility of a metal matrix with the strength, hardness, and wear resistance of a ceramic reinforcement. Among MMCs, aluminum matrix composites (AMCs) are attractive for their low density, high specific strength, and good corrosion resistance, which suit them to aerospace, automotive, marine, and structural use [1-3]. AMCs also offer better stiffness, as well as tribological and high-temperature performance than monolithic aluminum alloys, meeting the demand for lighter, stronger engineering materials [4, 5].

LM13 aluminum alloy, a hypereutectic Al-Si-Cu-Ni alloy, is widely used for pistons, cylinder heads, pump impellers, and valve components, owing to its good castability, wear resistance and dimensional stability at high temperatures [5]. Silicon improves abrasion resistance, while copper and nickel add thermal stability and strength retention. Even so, unreinforced LM13 falls short for applications needing higher load capacity, wear resistance, and long service life. Reinforcing LM13 with ceramic particulates is therefore an effective way to improve its performance.

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Ceramic reinforcements such as silicon carbide (SiC), alumina (Al_2O_3), boron carbide (B_4C), silica (SiO_2), and zirconium-based compounds have been widely studied for improving the mechanical and tribological behavior of AMCs [2,6-9]. These reinforcements aid load transfer between the ductile matrix and hard ceramic phase, which restricts plastic deformation and improves wear resistance. Zirconium oxide (ZrO_2) is a particularly attractive reinforcement due to its high melting point, chemical stability, fracture toughness, and thermal shock resistance. Unlike most brittle ceramics, ZrO_2 shows transformation toughening, which improves crack and damage resistance. Earlier studies report clear gains in tensile strength, hardness, fracture toughness, and wear resistance for ZrO_2 -reinforced aluminum composites over unreinforced alloys [10-13]. Nano-sized reinforcements have extended these gains further. Nano- ZrO_2 particles have a much higher surface area-to-volume ratio than micron-sized particles, giving stronger matrix-particle bonding and more efficient load transfer [7, 9]. Dispersed nanoparticles also act as nucleation sites during solidification and hinder dislocation motion through Orowan strengthening, which refines grains and improves mechanical properties. By the Hall-Petch relationship, finer grains raise strength and hardness and improve resistance to crack initiation. Several studies report better tensile strength, hardness, and fracture behavior in nano- ZrO_2 -reinforced aluminum and magnesium alloys compared with micro-particle reinforced systems [10-12]. However, dispersing nanoparticles in molten aluminum remains difficult because of poor wettability, agglomeration, and non-uniform distribution from their high surface energy [14].

The fabrication route strongly affects the resulting microstructure and properties of AMCs. Powder metallurgy, squeeze casting, spray deposition, in-situ synthesis, and stir casting have all been used to produce particle-reinforced AMCs. Stir casting is among the most economical and scalable of these, owing to its simplicity and low cost [9,14]. Even so, achieving uniform nanoparticle dispersion and low porosity remains a challenge, so optimizing the solidification conditions is essential for a refined microstructure and good mechanical properties.

Metallic chills are one of the most effective tools for controlling solidification in cast composites. Chills are high-conductivity inserts placed in the mould to raise local cooling rates [15,16]. Cast iron chills are common for their good thermal properties, mechanical strength, and low cost. Prior work shows that chill-assisted solidification refines grains, limits porosity, improves reinforcement distribution, and improves the mechanical properties of aluminum alloys and composites. Chill thickness in particular affects heat extraction, and through it, grain size, dendritic arm spacing, reinforcement distribution, and overall microstructural uniformity.

Cryogenic solidification is another route to a finer microstructure in MMCs. Using liquid nitrogen during solidification raises the cooling rate considerably, giving ultrafine grains, less segregation, better reinforcement dispersion, and stronger matrix-particle bonding [17]. Cryogenically solidified composites therefore tend to show higher hardness, tensile strength, fracture toughness, and wear resistance than conventionally cast ones [15]. Combining chill-assisted casting with cryogenic solidification thus offers good potential for tailoring the microstructure and mechanical performance of aluminum nanocomposites.

Recent studies on zirconium-containing aluminum composites report further gains in mechanical and tribological properties through optimized reinforcement and processing [18,19]. Most of this work, however, looks at conventional solidification, micron-scale reinforcement, or a limited set of chill configurations. The individual effects of nano- ZrO_2 reinforcement and cryogenic solidification are known [10-13], but their combined effect together with varying cast iron chill thickness on LM13 alloy has not been studied in a systematic way. In particular, how chill thickness governs heat extraction, microstructural evolution, and mechanical properties in cryogenically solidified LM13/nano- ZrO_2 composites remains unexplored. Related work shows that stir-cast Al2024/nano- ZrO_2 composites gain hardness, compressive strength, and wear resistance with increasing ZrO_2 content [20], and that friction-stir processed Al- ZrO_2 systems show improved tensile strength, hardness, fatigue strength, and wear resistance through more even nanoparticle dispersion [21]. Nano-oxide-reinforced 2024 aluminum alloys have shown tensile-strength gains alongside a drop in hardness at higher reinforcement levels, showing that such trade-offs depend on the reinforcement and process used, not a universal rule [22]. Stir-cast A356.1 and Al2219 alloys

reinforced with ZrO_2 nanoparticles have also been studied, further supporting the general benefit of ZrO_2 nano-reinforcement in cast aluminum alloys [23, 24].

Recent studies have demonstrated the potential of aluminum-based MMCs for improving mechanical, tribological, machining, and functional performance through the use of diverse reinforcements and processing techniques. Investigations involving rice husk ash, graphene, powder-metallurgy processing, and sustainable reinforcements have highlighted the importance of reinforcement type, particle distribution, processing conditions, and microstructural evolution in determining composite properties [25–29]. These studies confirm that appropriate reinforcement and processing strategies can significantly enhance the performance of aluminum alloys; however, their focus has been mainly on different alloy systems, reinforcement materials, or processing conditions. The combined influence of nano- ZrO_2 , cryogenic solidification, and varying cast-iron chill thickness in LM13 aluminum alloy therefore remains insufficiently explored. The novelty of this work lies in combining nano- ZrO_2 reinforcement, cryogenic solidification, and a systematic variation of cast iron chill thickness to tailor the microstructure and mechanical properties of LM13 composites. These three factors have been studied separately before, but not together. This study looks at how chill thickness affects heat extraction, grain refinement, reinforcement distribution, and mechanical performance under cryogenic solidification. It should be noted that this is a single-factor study on one material system (LM13 + 10 wt.% nano- ZrO_2); the individual and interactive contributions of nano- ZrO_2 reinforcement, chill thickness, and cryogenic cooling cannot be statistically separated here, and the trends reported should be read with this in mind. Based on this gap, the present work fabricates LM13 aluminum alloy reinforced with 10 wt.% nano- ZrO_2 by stir casting followed by cryogenic solidification, and studies how varying cast iron chill thickness affects microstructure, tensile strength, and hardness. Correlations among cooling conditions, microstructural refinement, and mechanical behavior are also drawn. The results are expected to help in developing high-performance aluminum nanocomposites for automotive, aerospace, marine, and wear-intensive applications.

2. Materials and Methodology

2.1 Materials

2.1.1 Matrix Material: LM13 Aluminum Alloy

LM13 aluminum alloy was chosen as the matrix material for its good castability, wear resistance, and wide use in automotive and industrial parts. Its mechanical strength and tribological performance, however, need improvement to meet the growing demand for lighter, higher-performing components. This was addressed here through nano-ceramic reinforcement and controlled solidification. Table 1 lists the physical and mechanical properties of the LM13 alloy used.

The LM13 alloy was procured from Intelligent Materials Pvt. Ltd. (Nanoshel, India), a certified supplier. Liquid nitrogen (LN_2) for the cryogenic step was supplied by Mysore Gases, Mysuru, India.

Table 1. Physical and mechanical properties of LM13 Aluminum alloy

Density	2.7 g/cm ³
Solidus Temperature	695 °C
Elastic Modulus	70 GPa
Thermal Conductivity	130–190 W/m·K
Ultimate Tensile Strength	200 MPa
Hardness	90 VHN

The values in Table 1 are the nominal properties reported by the supplier, not values obtained by independently casting and testing an unreinforced LM13 control under the same chill and cryogenic conditions used for the composites. The percentage gains reported in Section 3 therefore describe trends within the composite system across the 5–25 mm chill-thickness range, not a direct comparison against identically processed unreinforced LM13. A dedicated unreinforced-LM13 control, cast and cryogenically treated under matching conditions, is recommended in future work to confirm this comparison quantitatively.

2.1.2 Reinforcement Material: Nano-Zirconium Oxide (ZrO_2)

Nano-sized ZrO_2 particles were chosen as reinforcement for their hardness, thermal stability, fracture toughness, and ability to promote grain refinement. The nanoparticles act as nucleation sites during solidification, improving matrix–reinforcement bonding and mechanical properties. The nano- ZrO_2 was procured from Intelligent Materials Pvt. Ltd. (Nanoshel, India), with an average particle size of 80 nm. A constant reinforcement content of 10 wt.% was used for all specimens. Table 2 lists its properties.

Before fabrication, the nano- ZrO_2 particles were cryogenically pre-treated in liquid nitrogen, with the aim of improving particle stability and dispersion during casting. It should be noted that the particles were later preheated to 400 °C before addition to the melt (Section 2.2.1), and how the earlier cryogenic step continues to affect dispersion after this later heating is not fully established in the literature; this is flagged here as a point needing further study.

Table 2. Properties of Nano-zirconium oxide (ZrO_2)

Particle Size	80 nm
Molecular Formula	ZrO_2
Melting Point	2715 °C
Density	5.68 g/cm ³

2.2 Composite Fabrication Methodology

2.2.1 Stir Casting Process

The composites were made by stir casting, chosen for its simplicity, low cost, and scalability. LM13 alloy was weighed and placed in a graphite crucible. Permanent moulds were preheated and dried to avoid gas entrapment and casting defects. The alloy was melted in an electric resistance furnace at 710 °C and held there for 10 min for complete melting and thermal homogenization. Hexachloroethane tablets (Sri Venkateshwara Metallurgical Chemicals, Bengaluru, India) were then added to degas the melt and reduce casting defects.

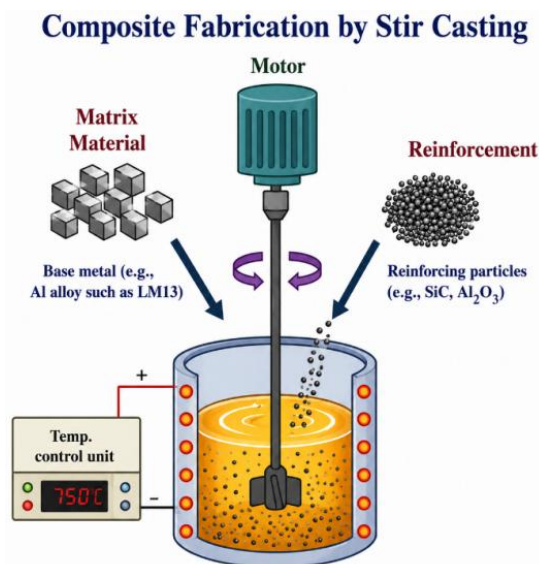


Fig. 1. Illustration of stir casting process

The nano- ZrO_2 particles were separately preheated to 400 °C to remove moisture and improve wettability with the molten aluminum. A graphite-coated impeller stirred the melt at 300–400 rpm to form a stable vortex; stirring speed was not deliberately varied between the chill-thickness conditions. The preheated nano- ZrO_2 was added gradually into the vortex, and stirring continued for 10 min for uniform dispersion. Slag and impurities were skimmed off, and the melt was poured into permanent moulds fitted with cast iron chills of varying thickness. Figure 1 shows the stir-casting setup, and Figure 2 outlines the full fabrication procedure.

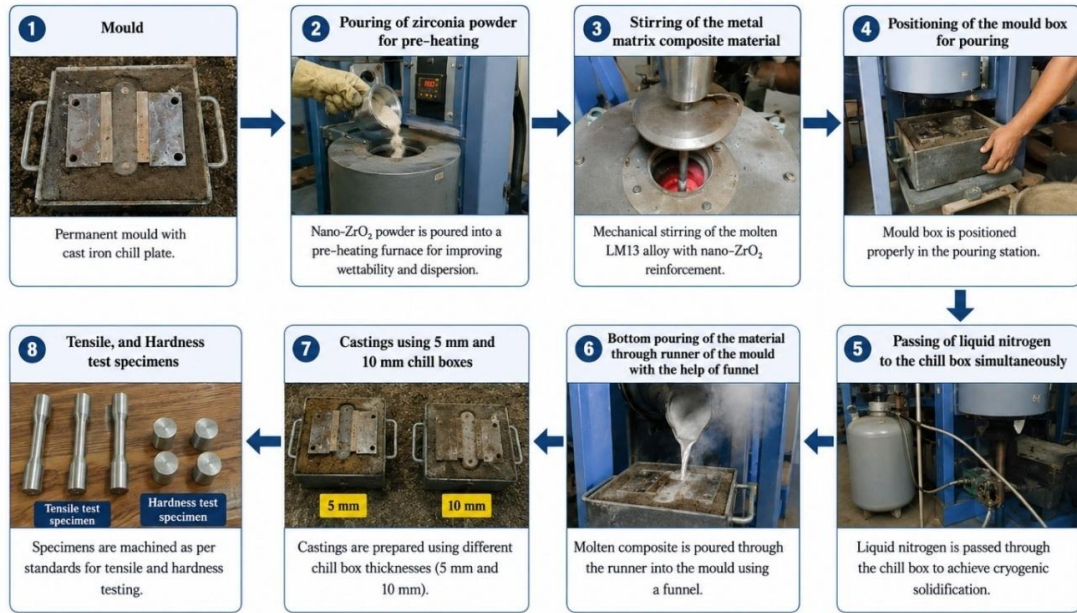
FABRICATION PROCESS OF Al–LM13 / NANO-ZrO₂ COMPOSITES BY STIR CASTING

Fig. 2. Flow diagram illustrating the stir-casting and cryogenic solidification process for AL–LM13/nano-ZrO₂ composites

2.2.2 Chill Casting and Cryogenic Solidification

Cast iron chills were placed at the base of the mould to control solidification rate and refine the microstructure. Five chill thicknesses – 5, 10, 15, 20, and 25 mm – were used to study their effect on heat extraction, cooling rate, and solidification behavior. Varying the chill thickness allowed control over cooling rate, which in turn affects grain size, porosity, dendritic structure, and reinforcement distribution. Liquid nitrogen (LN₂) was also used for cryogenic solidification and rapid heat extraction.

Cryogenic solidification aids grain refinement, reduces micro-segregation and porosity, and improves microstructural homogeneity and matrix–reinforcement bonding. After solidification, castings were removed from the moulds and cooled to room temperature under ambient conditions. Figure 3 shows the cryogenic treatment chamber schematic along with photographs of treated specimens. The chamber had LN₂ inlet and outlet ports, circulating nitrogen at about –196 °C for rapid, uniform cooling. Based on the microstructural and mechanical results together with the wider literature on rapid solidification, this step is expected to have promoted grain refinement and microstructural homogeneity; residual stress was not measured directly, so any reduction in residual stress is inferred, not confirmed, consistent with reported cryogenic-treatment residual-stress reduction mechanisms in aluminum alloys more broadly [30,31].

The accompanying photograph confirms that the cryogenic setup worked as intended, with specimens uniformly exposed to liquid nitrogen. Surface oxidation seen on the specimens is due to moisture condensation during warming to room temperature, and does not indicate material degradation.

An important factor in chilling effectiveness is the sensible heat the chill can absorb as its temperature rises during solidification, which depends on the chill's density, specific heat capacity, volume, and temperature rise.

Heat Absorption Capacity

$$Q = \rho V C_p \Delta T \quad (1)$$

where: ρ = Density of the chill material (kg/m³); V = Volume of the chill (m³); C_p = Specific heat capacity of the chill material (J/kg·K); ΔT = Temperature rise of the chill material during solidification (K).

Equation (1) gives the sensible heat absorbed by the chill for a given temperature rise; it grows with chill density, volume, specific heat capacity, and temperature rise. It is only a thermal-capacitance estimate, though, and does not by itself describe the rate of heat extraction from the casting. Actual chilling effectiveness also depends on the chill's thermal conductivity, the casting–chill contact resistance, the temperature difference between casting and chill, the interface heat-transfer coefficient, the chill geometry and contact area, and the available solidification time. Direct cooling-rate measurements (such as embedded thermocouple records) were not taken in this study; Equation (1) is used only as a first-order indicator of relative heat-absorption capacity among the chill thicknesses tested, not as a validated predictor of cooling rate.

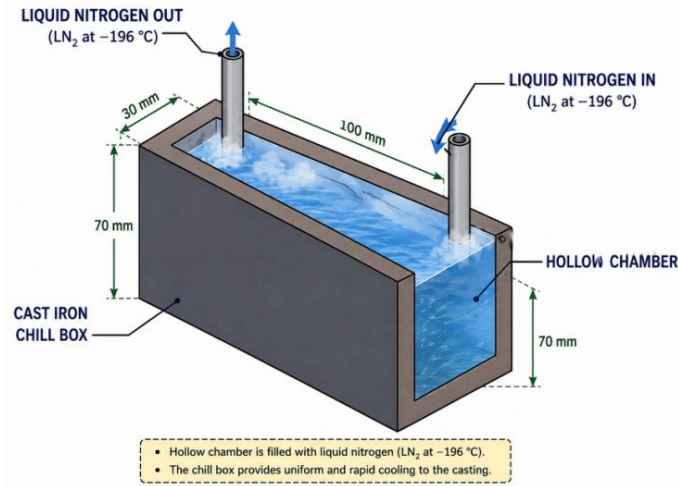


Fig. 3. Schematic diagram of cast iron (CI) chill box for cryogenic treatment

2.3 Specimen Preparation

The solidified castings were machined into standard test specimens as per ASTM standards. Tensile specimens followed ASTM E8 (Figure 4), and hardness specimens followed ASTM E92-17 (Figure 5). All specimens were machined and finished for dimensional accuracy and surface quality.

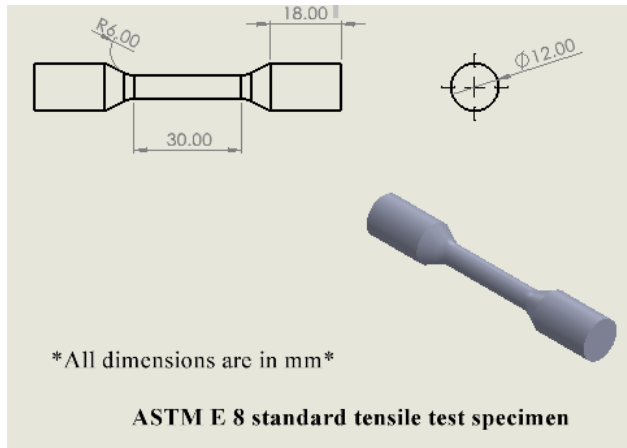


Fig. 4. ASTM E8 standard tensile specimen

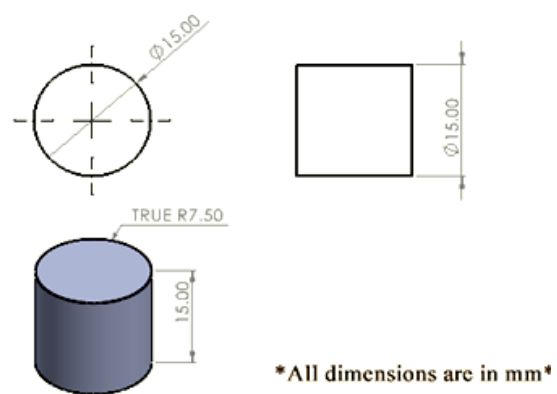


Fig. 5. ASTM E92 standard hardness testing specimen

2.4 Mechanical Testing Methodology

2.4.1 Micro-Vickers Hardness Testing

Hardness was measured using a Micro-Vickers tester, to assess resistance to localized plastic deformation and the effect of chill thickness on the microstructure of the Al–LM13/nano-ZrO₂ composites. Specimens were sectioned perpendicular to the heat-flow direction, mounted, and polished using progressively finer emery paper (100, 220, 400, 600, and 1000 grit), followed by alumina slurry polishing to a mirror finish suitable for indentation testing.



Fig. 6. Hardness test specimens

A diamond pyramid indenter was used, with a 100 g load applied for a dwell time of 25 s. At least five indentations were made per specimen at different locations to account for microstructural variation, and the average was taken as the hardness. Figure 6 shows the hardness specimens used.

2.4.2 Tensile Testing

Tensile testing used a computerized Universal Testing Machine (UTM) as per ASTM E8, at room temperature. Tensile behavior of the nano-ZrO₂-reinforced composites was assessed from the stress-strain curves for each chill-thickness condition; a separately cast, identically processed unreinforced LM13 specimen was not included (see the note under Table 1). Ultimate tensile strength (UTS) was determined from this data. Three specimens were tested per chill-thickness condition, and the average taken, to check repeatability. For each condition, the three tensile specimens came from a single casting batch; independent replicate castings per condition were not made. The reported repeatability therefore reflects specimen-level variation within a batch, not batch-to-batch reproducibility – a limitation of the current design.

2.5 Microstructural Characterization

Microstructure was examined by optical and scanning electron microscopy (SEM). Specimens were ground with successive grades of silicon carbide paper, polished with alumina slurry, and etched with Keller's reagent. This analysis assessed grain refinement, reinforcement distribution, porosity, dendritic morphology, and matrix-particle interfacial characteristics across chill thicknesses and cryogenic conditions. All mechanical tests were run in triplicate; reported values are the arithmetic mean, and the observed variation was within acceptable limits.

3. Results and Discussion

3.1 Hardness Testing

Figure 7 shows that the hardness of the Al-LM13 alloy with 10 wt.% nano-ZrO₂ rises as chill thickness increases from 5 mm to 25 mm, from 71.20 VHN to 99.60 VHN, a gain of about 39.9%. This points to a clear link between chill thickness, cooling rate, and the resulting microstructure. At 5 mm, hardness was lowest, owing to the slower cooling rate. Reduced heat extraction let coarse grains and wider dendritic arm spacing form, lowering resistance to localized plastic deformation; the resulting heterogeneous microstructure further reduced hardness.

At 10 mm and 15 mm, hardness rose gradually as heat extraction improved. Faster cooling promoted quicker nucleation and limited grain growth, giving finer dendritic structures and smaller dendrite arm spacing, which offered more resistance to indentation. At 20 mm, hardness rose further, due to greater grain refinement and a more uniform phase distribution; the faster cooling gave a denser microstructure with fewer casting defects. By the Hall-Petch relationship [31,32], finer grains increase grain boundary area, restricting dislocation movement and raising hardness. The highest hardness, 99.60 VHN, was seen at 25 mm. The higher cooling efficiency here gave a very fine, uniform microstructure with evenly distributed acicular phases, improving resistance to localized deformation and wear.

Table 3. Hardness test result of Al-LM13+ZrO₂

Sl. No	Chill Thickness (mm)	Hardness Value (VHN)
1	05	71.20
2	10	79.10
3	15	81.93
4	20	84.06
5	25	99.60

The small error bars in Figure 7 indicate good repeatability and low experimental variation in the hardness measurements. Table 3 lists the hardness values, and Figure 7 shows their variation with chill thickness. Uniform ZrO₂ dispersion and improved matrix-particle wettability restricted dislocation movement and localized plastic deformation under the indenter, raising hardness. This shows the value of optimizing chill thickness for a good balance of hardness and tensile strength in parts such as pump impellers, shafts, and valve components, where both wear resistance and load-bearing capacity matter.

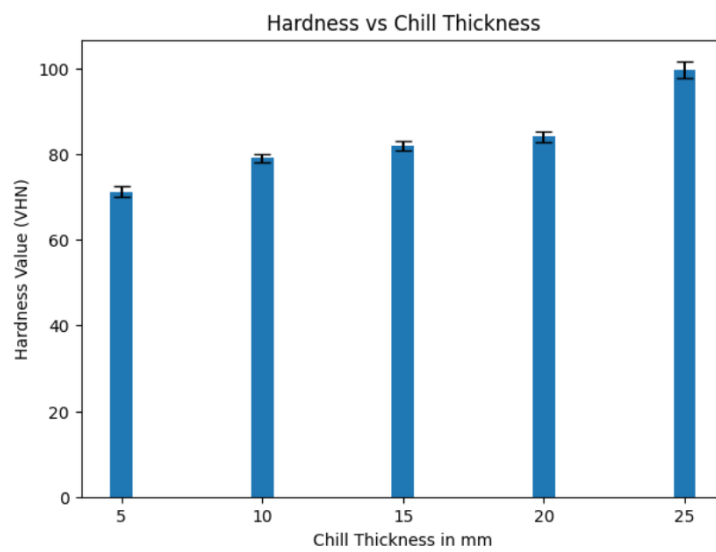
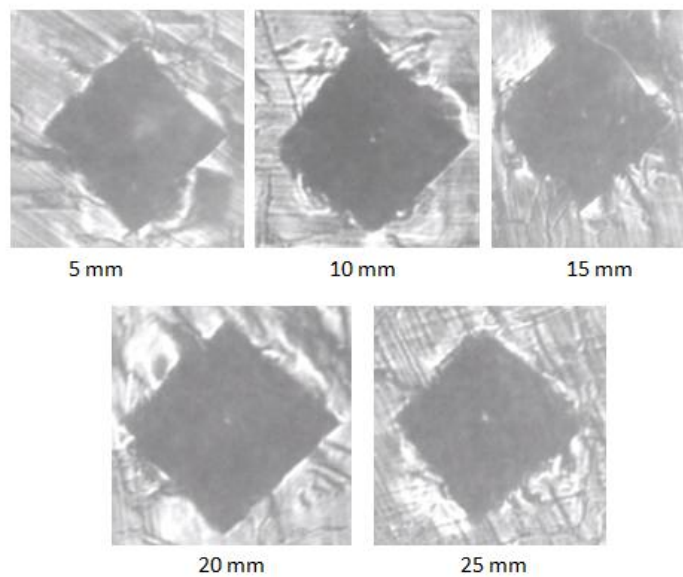
Fig. 7. Hardness of the Al-LM13 alloy reinforced with 10 wt.% nano-ZrO₂

Fig. 8. Optical micrographs of Vickers micro-hardness indentation at varying chill thicknesses of cryogenically treated metallic specimens

Figure 8 shows the optical micrographs of Vickers micro-hardness indentations at each chill thickness for the cryogenically treated specimens; these are discussed together with the hardness trend above rather than repeated here.

3.2 Tensile Testing

Tensile testing was done on Al-LM13 alloy with 10 wt.% nano-ZrO₂ across chill thicknesses of 5, 10, 15, 20, and 25 mm. Table 4 and Figure 9 give the UTS values obtained.

UTS rose from 145 MPa at 5 mm to 200.63 MPa at 25 mm, a gain of about 38.4%. It stayed nearly flat, 145 MPa and 146 MPa, between 5 mm and 10 mm, showing thinner chills have limited effect on solidification behavior. At 15 mm, UTS rose to 150.95 MPa from better heat extraction and microstructural refinement.

Table 4. Tensile strength result of LM-13 + 10 wt.% ZrO₂

Sl. No	Chill thickness (mm)	Tensile Strength (MPa)
1	05	145.00
2	10	146.00
3	15	150.95
4	20	173.03
5	25	200.63

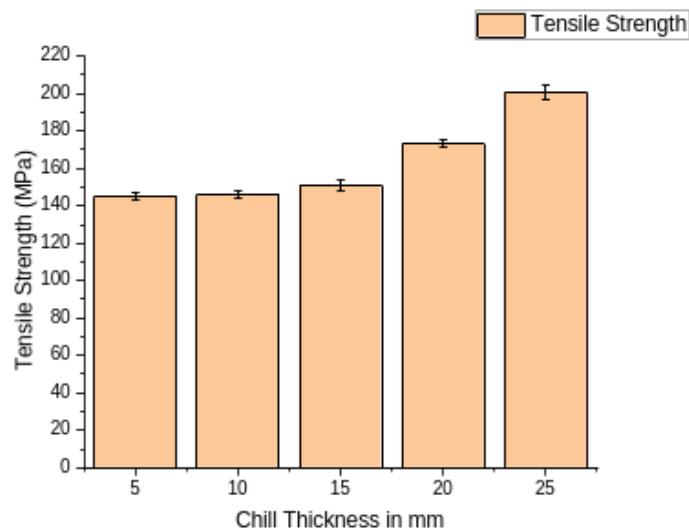


Fig. 9. Ultimate tensile strength (UTS) of the Al-LM13 alloy reinforced with 10 wt.% nano-ZrO₂ as a function of cast iron chill thickness

UTS rose sharply at 20 mm and 25 mm, to 173.03 MPa and 200.63 MPa. This fits with, and is read here as linked to, the higher cooling rates expected at thicker chills, which likely promote grain refinement and a better reinforcement distribution. Porosity, cooling rate, and interfacial bond strength were not measured directly, so these mechanisms should be seen as plausible explanations for the trend, not independently confirmed findings. On this basis, load transfer from the ductile aluminum matrix to the harder ceramic reinforcement is likely more efficient, improving tensile strength.

Figure 10 shows the engineering stress-strain curves. These confirm that chill thickness strongly affects mechanical performance in the cryogenically solidified nanocomposites. At 5 mm and 10 mm, specimens showed lower stress-bearing capacity, from slower cooling, coarser grains, and less uniform reinforcement distribution. At 15 mm and 20 mm, the curves show progressively higher UTS; as yield-strength values were not extracted from the curves here, this gain is reported only in terms of UTS. This fits with the better grain refinement and reinforcement distribution expected from faster cryogenic solidification; as noted, porosity and interfacial bond strength were not measured directly.

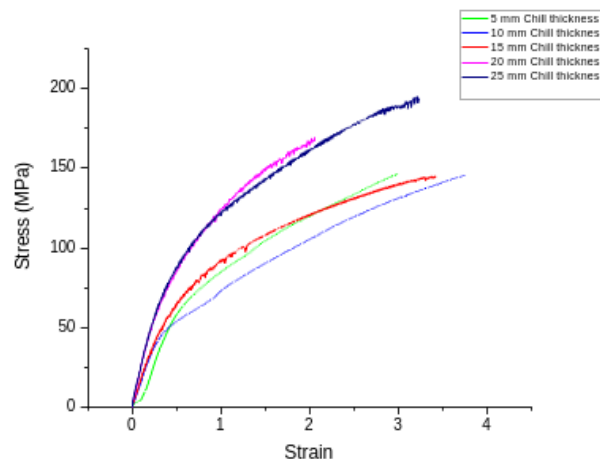


Fig. 10. Engineering stress–strain curves of cryogenically solidified Al–LM13 + 10 wt.% nano-ZrO₂ MMC for varying chill thicknesses (5 mm–25 mm)

The 25 mm specimen showed the highest tensile strength and stress-bearing capacity, along with a visibly, though not separately quantified, lower fracture strain than the lower chill-thickness conditions. This points to a strength gain with the finer microstructure at 25 mm; as yield strength and elastic modulus were not extracted from the curves, this statement is limited to strength and does not imply a separately measured stiffness gain. The refined microstructure is read as having limited dislocation movement and improved load transfer from matrix to reinforcement.

Overall, the stress–strain data confirm that a thicker cast iron chill under cryogenic solidification clearly improves tensile performance in Al–LM13/nano-ZrO₂ composites, through microstructural refinement and better load transfer.

3.3 Microstructural Analysis of the Al–LM13 + 10 wt.% Nano-ZrO₂ Composite at Various Chill Thicknesses

The tensile strength and hardness trends with chill thickness can be linked directly to the microstructural changes in Figure 11, which shows how the acicular phases evolve and distribute, strongly affecting mechanical properties.

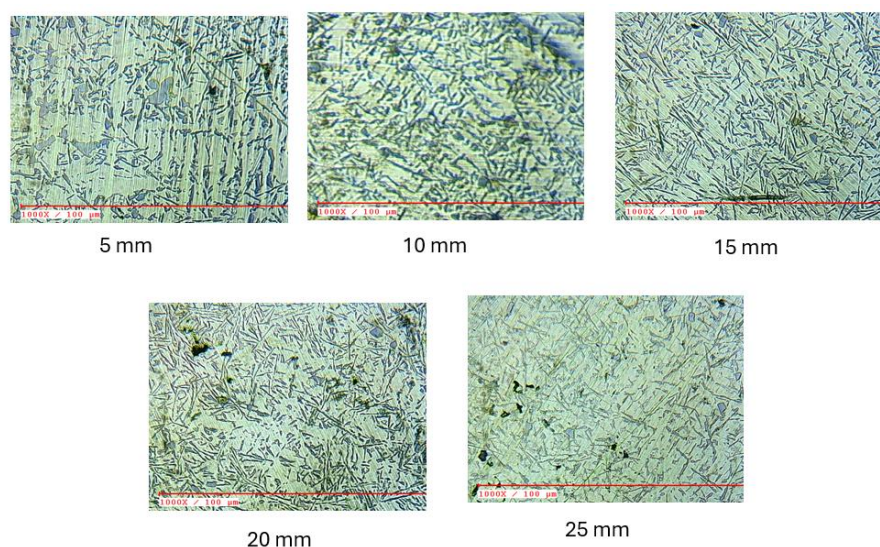


Fig. 11. Optical micrographs showing the microstructural evolution of the Al–LM13 + 10 wt.% Nano-ZrO₂ composite at various chill thicknesses

At 5 mm and 10 mm, the microstructure was relatively coarse, with partially aligned, unevenly distributed acicular constituents. The 5 mm specimen showed elongated features and local clustering, pointing to slower cooling and less grain refinement, matching its lower tensile strength

(145 MPa) and hardness (71.20 VHN). At 15 mm and 20 mm, the microstructure was more refined and even, with acicular phases uniformly distributed and less agglomerated. Better heat extraction and faster solidification here refined grains and improved reinforcement distribution, raising tensile strength from 150.95 MPa to 173.03 MPa and hardness from 81.93 VHN to 84.06 VHN.

The 25 mm specimen gave the highest tensile strength (200.63 MPa) and hardness (99.60 VHN). Its faster cooling drove strong grain refinement and a dense microstructure, which is read as contributing to the improved resistance to plastic deformation and fracture noted above. Microvoid/porosity content and interfacial bond strength were not measured directly (by image analysis, density measurement, etc.), so these should be read as proposed rather than confirmed mechanisms. Overall, a thicker chill is linked to a refined, more uniform microstructure, which likely restricts dislocation motion and crack initiation and contributes to the observed gains in hardness and tensile strength.

3.4 SEM Fractography of Tensile Tested Al-LM13 + 10 wt.% Nano-ZrO₂ Composite

Figure 12 shows SEM fractographs of the tensile-fractured surfaces for the 5 mm and 25 mm chill specimens. These clearly show how chill thickness affects failure behavior. The 5 mm specimen had a coarse, irregular fracture surface, with microvoids, particle agglomeration, and localized crack sites. Larger dimples and broken tear ridges indicate a mainly ductile fracture mode, matching the slower cooling and weaker matrix–particle bonding expected here. Particle pull-out regions also suggest uneven nano-ZrO₂ dispersion.

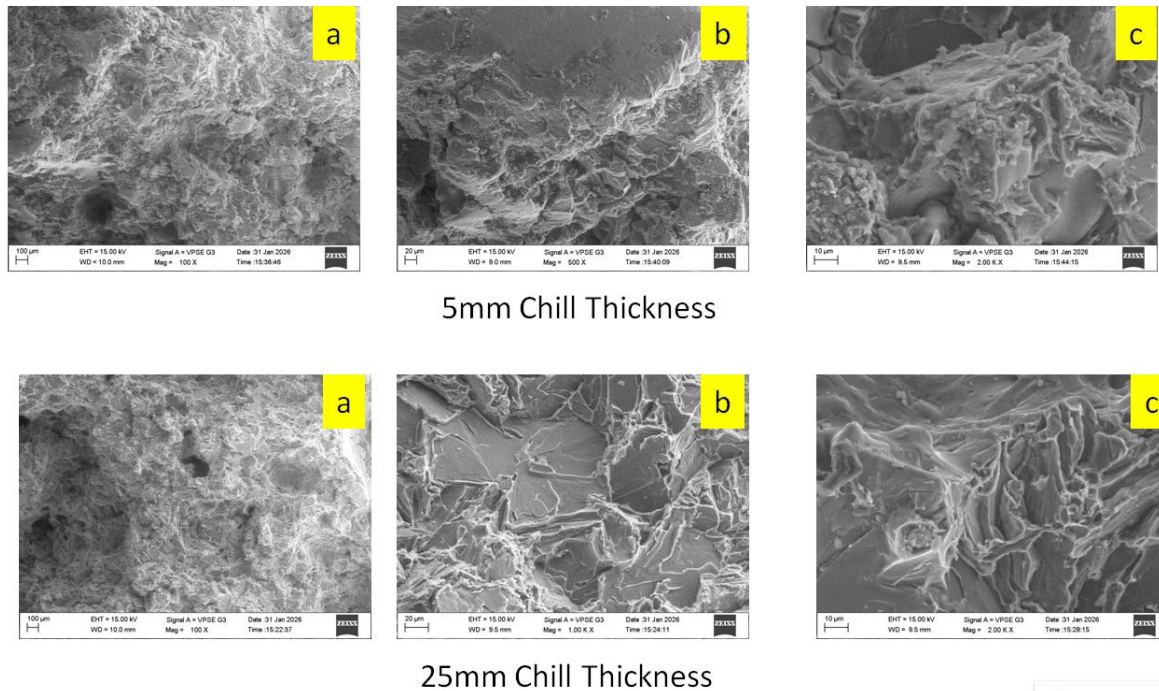


Fig. 12. SEM images of tensile fractured surfaces of cryogenically solidified Al-LM13 alloy reinforced with 10 wt.% nano-ZrO₂ composite at chill thicknesses of 5 mm and 25 mm

The 25 mm specimen showed a more refined fracture surface, with shallow dimples, evenly distributed microvoids, and quasi-cleavage facets. The more compact surface and smaller voids point to better grain refinement and stronger interfacial bonding between the LM13 matrix and nano-ZrO₂, from faster heat extraction under cryogenic solidification. Less plastic deformation and the quasi-cleavage features suggest a shift from mostly ductile fracture at 5 mm to a mixed ductile-quasi-cleavage mode at 25 mm. Note that fractographs were taken only at these two extreme thicknesses, so this shows a change between the lowest and highest thicknesses tested, not a demonstrated progressive shift across all five; fractography at 10, 15, and 20 mm is recommended to confirm the trend. This fits with the sharp rise in tensile strength to 200.63 MPa at 25 mm. Overall, the SEM data fit with a thicker chill improving tensile strength in Al-LM13/nano-ZrO₂

MMCs through microstructural refinement, better reinforcement distribution, and a changed fracture mode.

3.5 X-ray Diffraction (XRD) Analysis

For further phase information, the XRD pattern of the Al-LM13/10 wt.% nano-ZrO₂ composite (25 mm chill) was recorded over $2\theta = 5^\circ$ – 80° and is shown in Figure 13. Strong peaks near $2\theta = 38.5^\circ$, 44.8° , 65.1° , and 78.2° match the (111), (200), (220), and (311) planes of face-centred-cubic aluminum (ICDD 04-0787), with lower peaks near $2\theta = 28.4^\circ$ and 47.3° matching silicon (ICDD 27-1402), reflecting the hypereutectic silicon content of LM13. Given the low ZrO₂ volume fraction (10 wt.%) and small particle size (about 80 nm), distinct ZrO₂ peaks (expected near $2\theta \approx 30.2^\circ$, 35.3° , and 50.3° for the tetragonal phase, or 28.2° and 31.5° for the monoclinic phase) were not clearly seen above the background here. This is a known limitation of lab XRD for dilute nanoscale reinforcements [33] and does not by itself mean ZrO₂ is absent. A longer scan time, a higher-resolution or synchrotron scan focused on these low-angle peaks, and Rietveld refinement are recommended in future work to confirm the retained crystalline phase of the ZrO₂ reinforcement.

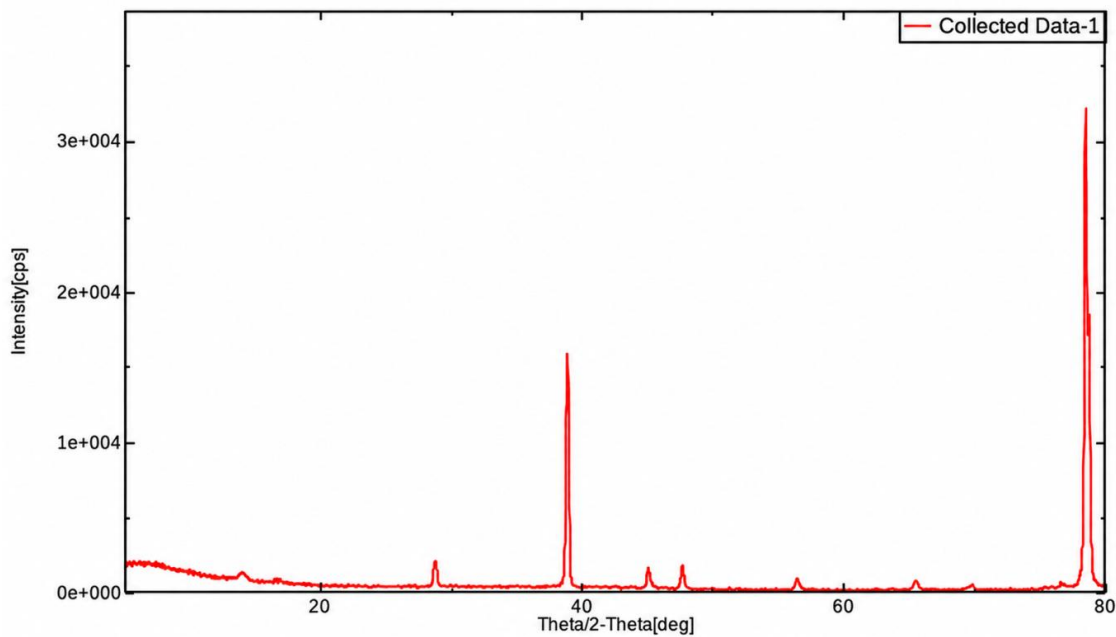


Fig. 13. XRD pattern of the Al-LM13/10 wt.% nano-ZrO₂ composite fabricated with the 25 mm cast iron chill

3.6 EDS Elemental Analysis

Point EDS analysis was also done on the matrix region of the 25 mm chill specimen for semi-quantitative elemental confirmation; results are in Table 5. Aluminum and silicon were the major elements found (82.0 and 18.0 wt.%, or 82.6 and 17.4 at.%), fitting the silicon-rich, hypereutectic LM13 matrix, with an overall analytical uncertainty of 6.94%. Zirconium was not detected in this point scan, which is expected given how localized point EDS is relative to the size and spread of the nano-ZrO₂ particles [34], and does not by itself establish the overall reinforcement distribution. Full-area EDS mapping directly on and around individual ZrO₂ particles, as suggested by reviewers, is identified as necessary future work to quantitatively confirm particle distribution and interfacial characteristics, and was beyond the scope of the point analysis here.

Table 5. Point EDS analysis of the matrix region (25 mm chill specimen)

Element	Weight %	Atomic %	Error %
Al K	82.0	82.6	3.3
Si K	18.0	17.4	8.8

Note: eZAF quantitative analysis; overall analytical uncertainty = 6.94%.

4. Conclusions

This study fabricated an Al-LM13/10 wt.% nano-ZrO₂ composite by stir casting followed by cryogenic solidification, and examined how cast-iron chill thickness (5, 10, 15, 20, and 25 mm) affects its mechanical properties, microstructure, and fracture behavior. UTS rose from 145 MPa at 5 mm to 200.63 MPa at 25 mm (about 38.4%), and micro-Vickers hardness rose from 71.20 VHN to 99.60 VHN (about 39.9%), based on the mean of three tensile tests and five hardness indentations per condition. These gains track the finer, more even microstructure seen with thicker chills, and are read as linked to the higher cooling rates expected there; as cooling rate was not measured directly, this link is an interpretation consistent with the trend, not an independently verified mechanism. Microstructural study showed progressive refinement of the acicular phases and a smaller dendritic scale as chill thickness increased, which likely restricts dislocation motion and so contributes to the hardness and tensile strength gains seen; grain size, dendrite arm spacing, and porosity were not measured quantitatively, and this is recommended for future work. SEM fractography, done at the two extreme thicknesses (5 mm and 25 mm), showed a shift from mostly ductile fracture to a mixed ductile–quasi-cleavage mode; as the intermediate thicknesses were not examined this way, this should be read as a change between the two extremes tested, not a demonstrated progressive shift. XRD of the 25 mm specimen confirmed aluminum and silicon as the main crystalline phases; ZrO₂ peaks were not clearly resolved, given the low reinforcement content and small particle size, and point EDS of the matrix likewise confirmed aluminum and silicon as major elements without detecting zirconium in the specific point scanned, consistent with how localized this technique is.

Among the five thicknesses tested, the 25 mm cast iron chill gave the best overall hardness and tensile strength in this study. As both properties were still rising at the upper end of the range tested, this is reported as the best-performing condition examined here, not a demonstrated optimum, and thicknesses beyond 25 mm were not tested. Several limitations should also be noted: direct thermal measurements (such as embedded thermocouple cooling curves) were not taken; an unreinforced LM13 control, cast and cryogenically processed under identical conditions to the composites, was not separately made, so the reported gains describe trends within the composite series rather than a verified comparison against unreinforced LM13; the design is single-factor and cannot statistically separate the individual or interactive effects of nano-ZrO₂ reinforcement, chill thickness, and cryogenic cooling; and no statistical significance testing beyond reporting means of replicate measurements was done. These limitations do not undo the mechanical trends reported, but they do mean several of the proposed causal mechanisms – reduced porosity, better interfacial bonding, and reduced residual stress among them – remain plausible interpretations rather than confirmed findings, and are presented as such throughout this paper. Overall, combining nano-ZrO₂ reinforcement with chill-assisted, cryogenically accelerated solidification looks promising for improving the strength and hardness of LM13-based aluminum composites. Given the single-factor design used, this should be seen as a demonstrated processing trend, not a proven synergistic interaction among the three variables, pending dedicated control experiments. This evaluation covered tensile strength, hardness, and associated microstructural, fractographic, XRD, and point-EDS characterization; wear, fatigue, corrosion, and elevated-temperature testing were not done. Whether the composite suits the automotive, aerospace, marine, and wear-intensive applications mentioned in the Introduction should therefore be seen as a direction for future validation, not an outcome established here.

Future work should prioritize: (i) an unreinforced LM13 control cast and cryogenically treated under matching conditions; (ii) direct thermal measurements to quantify cooling rate versus chill thickness; (iii) a factorial or other controlled design able to separate the individual and interactive effects of reinforcement, chill thickness, and cryogenic cooling; (iv) quantitative image analysis of grain size, dendrite arm spacing, and porosity; (v) SEM/BSE imaging with EDS mapping directly on ZrO₂ particles, along with higher-resolution XRD and Rietveld refinement, to confirm particle distribution, interfacial characteristics, and the retained ZrO₂ phase; and (vi) tribological, corrosion, fatigue, and elevated-temperature testing, along with checking alternative chill materials such as copper and graphite, to assess the long-term engineering suitability of the composites.

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