Research Article

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Retracted: Investigating the Fatigue Behaviour of TiO_2 Nanoparticle Reinforced AA7071 Aluminium Alloy

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ABSTRACT

Aluminium metal matrix nanocomposites have become a major focus in the industry as they possess exceptional strength and ductility. The experiment included incorporating TiO_2 nanoparticles into the AA7071 alloy at a volume ratio of 5 wt. % using the stir casting technique. The matrix was infused with TiO_2 nanoparticles, which had an average particle size of 30 ± 5 nm, using a powder injection method. A study was carried out to assess the influence of different levels of TiO_2 on the durability of the material, both before and after undergoing heat treatment. The results indicated that the fatigue properties of AA7071 were enhanced after heat treatment when TiO_2 nanoparticles were incorporated. After undergoing heat treatment, a 5 wt. % TiO_2 addition resulted in a significant improvement in fatigue properties, leading to a noteworthy 14.71% increase in fatigue life when compared to the base sample. The observed improvement may be ascribed to the larger quantity of finely dispersed precipitates that are evenly distributed after the heat treatment procedure. The fatigue strength of the matrix in composite materials showed a discrepancy of just 4[illegible] between the experimental and numerical values. Furthermore, the research found that the absence or decrease in the size of the [illegible] without precipitates inside the space between dendrites happened after the aging process. Nevertheless, despite the inclusion of 5 weight percent of titanium and the use of heat treatment, the creation of Al_2CuMg precipitates remained unachievable. However, the formation of the Al_3TiCu and Al_7TiCu phases occurred.

Keywords: AA7071; TiO_2 Nanoparticles; Fatigue behaviour

1. INTRODUCTION

Producing engineering materials with a high strength-to-weight ratio suitable for vehicles is a challenging task. It requires achieving strength, lightness, and cost-effectiveness simultaneously (Yang *et al.* 2017). Researchers in the area of composite materials have shown significant enthusiasm about the market demand for these goods in the [illegible] and aerospace sectors (Park *et al.* 2010; W[illegible] *et al.* 2020). Aluminum-matrix composites (AMCs) are regarded as advanced materials d[illegible]tstanding mechanical capabilities. These materials combine the characteristics of a lightweight and durable matrix material with a robust ceramic reinforcement (Miyamoto *et al.* 2010). AMCs fulfil the market demand for lightweight, durable, and high-performance components (Awasthi *et al.* 2021). Carbides, oxides, and nitrides have been the primary ceramic reinforcements used in AMCs (Aluminum Matrix Composites) in the last several decades. Examples of carbides include SiC (silicon carbide) and TiC (titanium carbide), while oxides include Al_2O_3 (aluminum oxide), ZrO_2 (zirconium oxide), and TiO_2 (titanium oxide). Nitrides often employed include TiN (titanium nitride) and AlN (aluminum nitride) (Das *et al.* 2012; Mahan *et al.* 2018). Experimental findings revealed that the fatigue strength of the 7075-T651 aluminum alloy experienced a significant 13% increase upon the addition of 10w/% SiC and 5w/% Al_2O_3 nanoparticles. Moreover, the inclusion of ZrO_2 nanoparticles in the aluminum matrix of the A356 alloy demonstrated a substantial enhancement in both wear resistance and hardness. According to the findings of Anbukarasi *et al.* 2024 the inclusion of 7w/% TiO_2 nanoparticles in the 6063-T6 aluminium alloy resulted in a remarkable 13% increase in fatigue strength when compared to the base metal. The addition of 2w/% TiN nanoparticles to Al2024 and subsequent aging results in a notable enhancement in the tensile strength by 38%. Nevertheless, this improvement is accompanied by a decrease in both the yield strength, which declines from 376.5 MPa to 359.1 MPa, and the elongation, which decreases from 12.7% to 9.4% (Divagar *et al.* 2016). The composition of aluminium alloy 2024 (AA2024) includes copper, magnesium, manganese, and other minor alloying elements. This alloy demonstrates a remarkable ratio of tensile-to-yield strength at high temperatures, along with high ductility, fatigue, and fracture resistance (Al-Furjan *et al.* 2021). The properties and ability to form second-phase precipitates for increased strength (age-hardening) contribute to the high demand of AA2024 in the aerospace and automobile industries. The research revealed that the addition of 10w/% TiO_2 nanoparticles, having an average diameter of 50 nm, into the AA2024 alloy via mechanical milling and hot-pressing led to a 54% enhancement in Vickers hardness and a 13% improvement in strength (Gajalakshmi *et al.* 2020).The properties of the composite are enhanced in comparison to the monolithic alloy. However, it is worth noting that this technology may have a potential drawback in the form of a low elongation value. This could potentially impact the reliability of the alloy when subjected to external stresses. The addition of 3 % TiO_2 nanoparticles (15 nm in size) to an Al matrix improves creep resistance. This is because the presence of the nanoparticles restricts the diffusional flows of the matrix, which are the main factor influencing creep behavior. After conducting a thorough analysis of the accessible materials, it has come to light that there is a scarcity of research regarding the consequences of integrating titanium into Al-Cu-Mg alloys through the process of stir casting (Jaber *et al.* 2020). The AA2024 aluminium alloy is a member of the Al-Cu-Mg alloy series and derives its strength primarily from the presence of precipitates such as (Al_2CuMg) and (Al_2Cu). To enhance the strength characteristics of the alloy, the addition of titanium can be employed, resulting in the formation of titanium aluminides that possess exceptional strength. One issue that arises with Al-Cu-Mg alloys is their vulnerability to thermal instability when subjected to high temperatures. However, the thermal stability of these alloys may be enhanced by producing titanium aluminides that possess robust thermal stability and assuring their uniform dispersion within the aluminum matrix (Sabari *et al.* 2024b).

This study aims to investigate the mechanical properties of AA7071 alloy fabricated through conventional stir casting with the addition of different weight fractions of TiO_2 nanoparticles. AA7071 has shown promising properties in various fields, and Al-Cu-Mg alloys can be hardened by incorporating hard particles during material production.

2. MATERIALS AND METHODS

This research focused on the AA7071 aluminium alloy because of its exceptional mechanical properties, including remarkable toughness, strength, and wear resistance. The versatility of this material makes it well-suited for a diverse array of applications, ranging from automobiles and aircraft to aerospace components and missile parts. Prior to conducting the experiments, the alloy underwent chemical analysis at Saveetha University, Chennai, India to ensure that its composition met the necessary specifications. An analysis was conducted on the fracture surface and microstructure of the AA7071 aluminium alloy-based nanocomposite using a TESCAN VEGA microscope. Additional examination was conducted using scanning electron microscopy (SEM). The microstructure of the alloy was examined using scanning electron microscopy (SEM) to generate micrographs, which offered valuable information on its shape and distribution of phases. Table 1 displays the precise mechanical characteristics of the alloy, especially its tensile strength, yield strength, and elongation. The decision to use TiO_2 nanoparticles as reinforcing material was based on their average size of 30±5 nm. Fig. 1 shows a scanning electron microscope (SEM) image of the nanoparticles. In order to achieve a consistent distribution of nanoparticles throughout the matrix, the composite was fabricated with a reinforcement component of 5 weight percent. The chosen amount of reinforcement was found to be ideal for achieving the specified mechanical qualities, while also maintaining favorable processability (Azhagiri *et al.* 2023).

Table 1. Mechanical properties of AA7071

Ultimate strength (MPa)	Yield Strength (MPa)	Hardness (HN)	Reduction in cross sectional area (%)
334	302	105	12.24

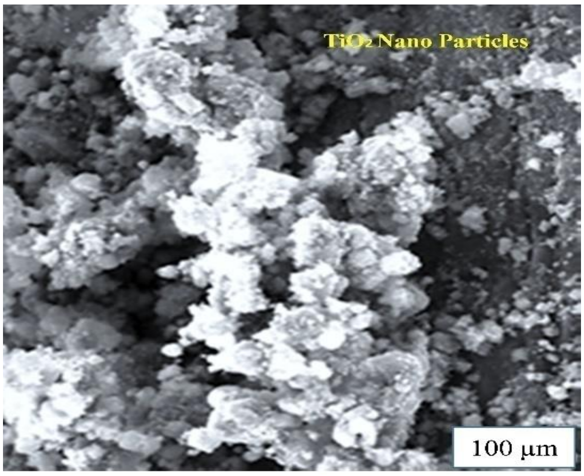


Fig. 1: SEM image of TiO_2 Nano particles

A meticulous technique was adhered to in order to prepare the matrix material samples. The specimens were subjected to a temperature of 700 °C by means of an electric furnace, using a graphite crucible. This controlled heating process ensured the thorough melting of all components, resulting in a uniform material. Afterwards, TiO_2 reinforcement nanoparticles were introduced into the molten matrix and blended for approximately 4 minutes at a speed of 200 rpm to ensure a homogeneous distribution within the matrix. Following the mixing process, the samples were subjected to solution treatment in a furnace equipped with air circulation. The test samples underwent a treatment process where they were exposed to a temperature of 500 °C for a duration of 3 hours. The solution treatment successfully dissolved any additional solute atoms, resulting in the establishment of a homogeneous microstructure. After the solution treatment, the samples were quickly cooled in water at room temperature to stop their progress. Afterward, they were subjected to a 3-hour aging treatment at a temperature of 175 °C. Precipitation hardening during the aging process greatly enhances the mechanical characteristics of materials by promoting the production of tiny precipitates. Subsequently, the samples were left to cool in the surrounding atmosphere until they reached the temperature of the room. Ultimately, the heat treatment procedure was crucial in augmenting the mechanical characteristics of the composite material via the promotion of a homogeneous distribution of reinforcing nanoparticles, resulting in an overall enhancement in performance (Gao *et al.* 2004).

2.1. Fatigue Testing

A fatigue test was performed in order to fully assess the material's fatigue strength, taking into account the statistical character of fatigue and the need for a sizable number of tests to get accurate findings. The research used the rotating-beam technique, which applies a steady bending force and records the number of stress cycles that result in failure. Initially, the stress was maintained slightly lower than the material's ultimate strength to guarantee the specimen could withstand the load. Additional tests were conducted using progressively lower stress levels to investigate the material's response across a spectrum of stress levels. Through this iterative process, a thorough comprehension of the fatigue characteristics of the material was achieved. The test results were plotted on an S-N diagram to illustrate the correlation between stress amplitude and fatigue life. The specimens used in the fatigue test were manufactured with great precision using a CNC milling machine and a computer program called [illegible]t. Accurate machining was crucial in order to meet the dimensional requirements for fatigue testing, guaranteeing that every sample possessed exact measurements and a smooth surface. In addition, the machining process was conducted with the goal of reducing residual stresses in the samples, in accordance with established standards to ensure precise fatigue test outcomes. In order to ensure consistent loading conditions for all fatigue specimens, a modern rotating-bending fatigue testing machine (Fig. 2) was utilized. The machine effectively maintained consistent loading conditions, regardless of the amplitude's constancy or variability, throughout the entire test duration. The machine underwent modifications to provide accurate and constant loading conditions, hence assuring the correctness and dependability of the test findings. The fatigue test samples were manufactured from the base alloy (AA7071) in full compliance with the necessary specifications, as outlined in the DIN 50113 standard. Thorough machining was conducted on all samples to attain a superior surface finish and minimize residual stresses, guaranteeing compliance with standard specifications for fatigue testing in terms of the specified dimensions. The meticulous preparation conducted guaranteed the production of accurate and dependable fatigue test outcomes, providing valuable understanding into the fatigue properties of the material under different loading circumstances (Sabari and Muni[illegible]an, 2024).

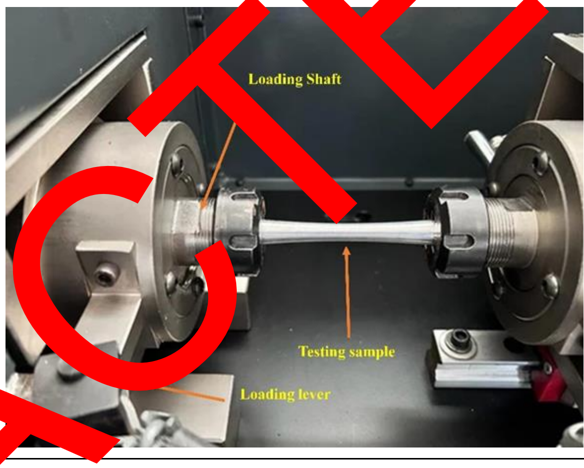


Fig. 2: Machine for Fatigue test

3. RESULTS AND DISCUSSION

The early efforts to understand the fatigue response of materials and buildings were based on the practical knowledge gained from real construction projects. Engineers and researchers have conducted extensive analyses on the performance of materials under cyclic loading conditions to ascertain their fatigue life. Fatigue life refers to the maximum number of stress cycles a material can withstand before experiencing failure. There are various factors that can impact the fatigue life of a material. Factors that influence the behaviour of materials under cyclic loading include the level of applied stress, the type of stress (tensile, compressive, or shear), the waveform of the loading, environmental conditions, and the metallurgical properties of the material. Materials under high stress or harsh conditions may experience shorter fatigue lives compared to those in lower stress or more benign environments. The examination on fatigue behavior included using data derived from Table 2 to create S-N curves for both the basic material AA7071 and the composites. The shown curves illustrate the correlation between stress amplitude (S) and the number of cycles to failure (N), offering a visual representation of the fatigue properties of the material. Through the analysis of these curves, researchers can acquire valuable insights into the response of various materials to cyclic loading and the impact of factors like the incorporation of nanoparticles on their fatigue performance. The S-N curves enable researchers to assess the fatigue properties of materials under different loading conditions and draw comparisons regarding their fatigue behaviour. This information is vital for the design of structures and components that need to withstand repeated loading over their lifetimes without any failures. In addition, a thorough comprehension of material fatigue is crucial for accurately estimating the durability of engineering components and establishing effective maintenance and inspection protocols to guarantee their continued safe functioning. Understanding the degradation of materials over time due to cyclic loading allows engineers to devise strategies for reducing fatigue-related failures and prolonging the lifespan of essential infrastructur[illegible]. One approach to address this issue is to optimi[illegible] materia[illegible] selection, adjust design parameters, a[illegible] implement periodic inspections and maintenance [illegible]rotocols to effectively monitor and manage fatig[illegible]e-rela[illegible] risks.

Table 2. The fatigue testing outcomes for each sample under constant amplitud[illegible]ading

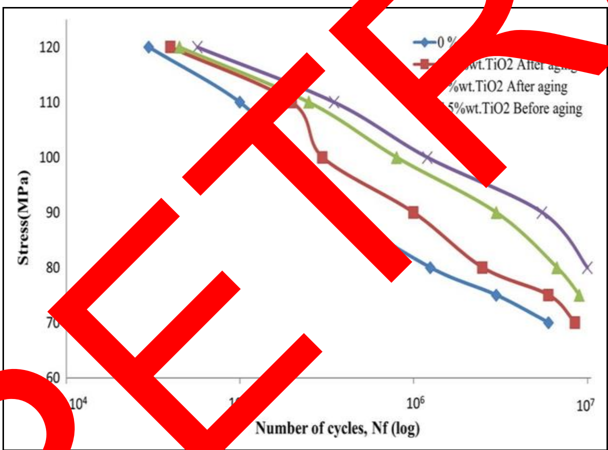
Condition	Spec. no	Heat treatment	N_f cyclic (avg.)	Ap[illegible]d stress, (MPa)
Base material	1, 2, 3	Before	55,[illegible]	100
	4, 5, 6		[illegible]30,[illegible]	[illegible]5
5 wt.% TiO_2	13, 14, 15	Before	57,000	120
	16, 17, 18		180,000	100
Base material	1, 2, 3	After	100,[illegible]0	78
	4, 5, 6		2,000[illegible]0	70
5 wt.% TiO_2	13, 14, 15	After	[illegible],000	130
	16, 17, 18	[illegible]	4,700,000	82

Aluminium alloys are wi[illegible]y re[illegible]nized a[illegible] excellent choices for structura[illegible] applicati[illegible]s. They possess the ability to develop [illegible]rong and [illegible]ng-lasting passive layer, which grant[illegible] the[illegible] adeq[illegible] pro[illegible] against corrosion in ty[illegible]al atmo[illegible]ric conditions. Basquin's equation can [illegible] used to chara[illegible]ize the fatigue behaviour.

$$\sigma_f = \text{[illegible]} N_f \quad (1)$$

[illegible] the provided equatio[illegible] N_f indicates the numb[illegible]f strain c[illegible]es that result in failure, σ_f represents the co[illegible]cient f[illegible]atigue strength, and A and σ_f represent the cons[illegible]or the mat[illegible]ls as specified in Table 2. [illegible]3 illu[illegible]tes the [illegible]igue characteristics obtained duri[illegible] consta[illegible]m[illegible]ude testing (RT) for a material con[illegible]ing 5 wt.%[illegible]O_2 nanoparticles, in comparison to a[illegible] without nanoparticles. The behaviour is [illegible]bserved bo[illegible]efore and after undergoing heat treatment and aging. The findings indicate that the nanocomposite [illegible]monstrates improved fatigue strength in comparison to th[illegible] base material without nanoparticles. The nanocomposite exhibits a noteworthy enhancement in its endurance fatigue limit, surpassing that of the base metal by 7.3%. The maximum fatigue strength achieved is 82 MPa. The improvement in fatigue strength can be credited to the reinforcement effect resulting from the inclusion of TiO_2 nanoparticles into the aluminium matrix. The arrangement of solid nanoparticles inside the base metal limits the movement of ductile metals, leading to increased strength of the composite material. The interaction between nanoparticles and dislocations is essential in defining the fatigue properties of aluminum alloys under cyclic stress. The incorporation of TiO_2 nanoparticles into the aluminum matrix enhances its fatigue resistance. The presence of nanoparticles inside the material impedes the migration of dislocations, leading to a significant enhancement in fatigue resistance. Eventually, the incorporation of TiO_2 nanoparticles into the aluminum matrix results in a reinforcing effect, which eventually enhances the fatigue strength of the composite material. The enhanced strength may be attributed to the interplay between nanoparticles and dislocations, as well as the widespread distribution of nanoparticles inside the base metal. The dispersion of malleable metals effectively limits their mobility, resulting in an overall increase in the strength of the composite.

Nanoparticles act as barriers inside the material, impeding the movement of dislocations and so increasing the material's strength and resistance to fatigue. When 5 wt.% TiO_2 nanoparticles were introduced, the AA7071-5 wt.% TiO_2 nanocomposite exhibited the maximum fatigue strength compared to the other evaluated samples. This led to a substantial enhancement of 7.18% when compared to the original content. The rise in fatigue strength is substantiated by the correlation coefficient R2, which offers statistical proof of the concurrence between the experimental data and the line equation derived from the fatigue tests. Heat treatment is essential for modifying the microstructure of aluminum alloys. It impacts several microstructural characteristics, such as the arrangement and dimensions of precipitates, the size of grains, and the existence of dislocations. The presence of nanoparticles also influences these alterations in the microstructure. By integrating nanoparticles into the matrix, the formation and propagation of fractures may be decreased, resulting in improved resistance to fatigue. Furthermore, the incorporation of Al-Ti-based intermetallic compounds (IMCs) is essential for enhancing the fatigue threshold of the material. The existence of these intermetallic compounds (IMCs) leads to the creation of areas of stress within the material, thereby improving its resistance to fatigue. The presence of minute precipitates and particles in the microstructure significantly contributes to the improvement of the material's strength and the prevention of fracture propagation. Overall, the addition of nanoparticle reinforcement and the creation of intermetallic compounds (IMCs) greatly improve the fatigue strength and overall performance of aluminum alloys. This improvement is crucial for a wide range of engineering applications, particularly when components are exposed to repeat loading conditions. It is of utmost importance to comprehend the microstructural alterations caused by heat treatment and the incorporation of nanoparticles in order to develop materials that possess improved fatigue resistance and durability (Sundaraselvan *et al.* 2023).



[illegible]ves for AA7071 aluminium alloy with different wt.% of TiO_2 nanoparticles, test temperature 25 °C

Fracture region microstructure

During the cooling process, both the matrix and the nanocomposite undergo different stress conditions due to their distinct thermal characteristics. The matrix is subjected to compressive stress in a tangential direction, whereas the nanocomposite receives tensile stress in a radial direction with equal pressure in all directions. The differences in stress occur due to discrepancies in thermal expansion and contraction between the two materials. In order to prevent small fractures in the composite material from spreading, compressive stress must be applied to the matrix. This improves the material's resistance to fracture. This phenomenon arises from the presence of compressive stress, which serves as an obstacle, impeding the spread of little fractures and enhancing the overall resilience of the material (Sabari *et al.* 2024c).

Fig.4 (a-d) demonstrates that fracture propagation often occurs at a right angle to the tensile tension and in line with the compressive stress. However, in high-stress situations, such as when a second phase is present, the crack tip's trajectory within the matrix may deviate. The observed deviation may be attributed to the radial tension and tangential compression imparted by the reinforcing particles. The presence of residual stress in the matrix is evident from the formation of spherical nanoparticles, which exhibit higher strength and elastic modulus in comparison to the matrix material. In addition, heat treatment processes may cause changes on the surface, such as the creation of oxide layers or the scattering of alloying elements. Surface alterations, especially on the outermost layer of a material, may significantly influence its resistance to fatigue.

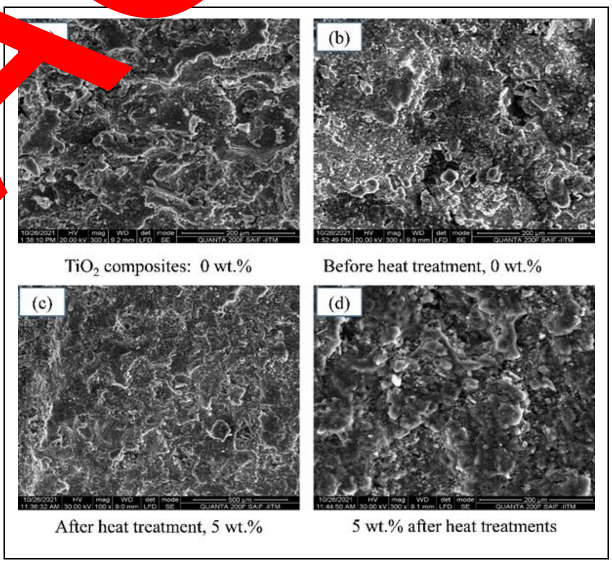


Fig. 4: SEM image of fracture surface of AA7071- TiO_2 Nano composites

Crack propagation can be affected by variations in the locations where cracks start or the existence of residual stresses. Furthermore, the correlation between nanoparticles and surface modifications is crucial in ascertaining the fatigue characteristics of the material. This behavior is often seen in composites that include a variety of nanoparticles. Nanoparticles can either reduce or worsen the impact of surface changes on fatigue performance. A thorough comprehension of stress conditions and surface alterations is crucial in order to improve the fatigue performance of composite materials. Comprehending this data enables the development of materials with improved resistance to fatigue and increased durability by optimizing the interaction between nanoparticles and surface treatments.

3.2. Microstructure of materials

Fig. 5 displays optical microscopy images that provide a comprehensive examination of the microstructures in different samples, both prior to and following heat treatment. The microstructures exhibit various inter-dendritic intermetallic compounds (IMCs) and numerous dispersed small precipitates. Following the heat treatment process, a significant improvement in the quality of precipitates was observed in the artificially aged samples. The microstructures displayed a reduction in particle size and a more even distribution in comparison to their state before treatment. An absence of precipitates was observed in the inter-dendritic area of the aged samples. It is important to note that this zone shrank in size after heat treatment, indicating a change in the microstructural makeup. Precipitate formation was significantly impacted by the presence of titanium, with titanium-containing samples showing lower precipitate sizes than titanium-free ones. It may be deduced that titanium contributes to the enhancement of precipitation, which results in the formation of finer microstructures. In summary, the findings suggest that the addition of titanium has a notable impact on the material's microstructural features, leading to potential enhancements in its mechanical properties and overall performance. A gray block phase was formed with the inclusion of titanium, mainly found next to the dark phase in the interdendritic zone. Following the aging treatment, the particles in the microstructure experienced growth. The microstructure, precipitate type, and presence of intermetallic compounds (IMCs) were analysed using scanning electron microscopy (SEM), as shown in Fig. 5. The examination of samples having different concentrations of TiO_2 (0% and 5% by weight) revealed that the solid particles underwent a purification process and had a more uniform arrangement within the microscopic structure. The inter-dendritic zone consisted of Al_7Cu_2Fe and Al (Cu, Mn, Fe, Si) due to the higher temperature at which these intermetallic complexes dissolved. Upon the addition of titanium, the microstructure underwent changes, leading to the formation of Al_3TiCu and Al_9TiFe intermetallic compounds (IMCs). Copper dissolution in the alloy is decreased when titanium is introduced to the Al-Cu-Mg system. As a consequence, intermetallic compounds such as Al_3TiCu and Al_7TiCu are formed. The ratio of copper to magnesium in the aluminum matrix is altered by this change. The presence of titanium compounds in the aluminum matrix is altered by the addition of 5 weight percent of TiO_2.

The presence of a significant quantity of copper hinders the creation of Al2CuMg precipitates, leading to the occurrence of Al3TiCu and Al9TiFe IMCs in the microstructure. Previous research has also noted the presence of intermetallic compounds (IMCs) located near particles comprising Al7Cu2Fe and silicon. Copper, when present, substitutes a portion of the crystal lattice inside the titanium aluminide structure. The results emphasize the significant influence of titanium on modifying the microstructure and composition of the aluminum alloy, resulting in enhanced mechanical qualities and performance.

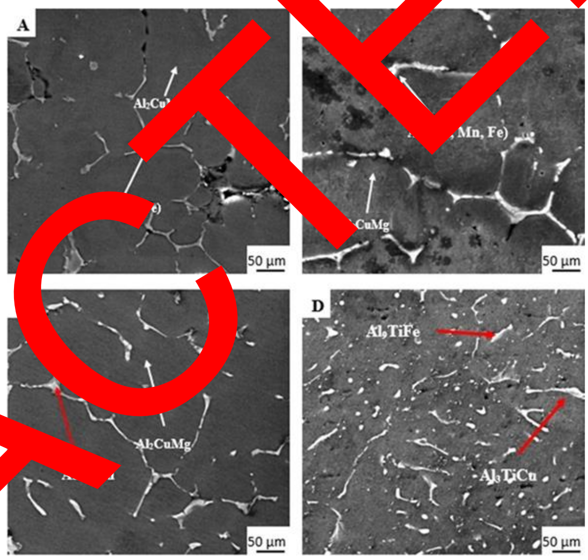


Fig. 5: SEM images of AA7071 alloy are shown in Fig. 5: (a) AA7071 before heat treatment, (b) AA7071 after heat treatment, (c) AA7071 with 5 wt.% TiO_2 before heat treatment, and (d) AA7071 with 5 wt.% TiO_2 after heat treatment

4. CONCLUSION

By utilizing a blend of empirical investigations and computational models, a number of significant findings can be deduced:

- The fatigue strength of the AA7071-5 wt.% TiO_2 nanocomposite surpassed that of the original metal matrix when subjected to constant amplitude loading conditions, reaching an impressive 82 MPa at 107 cycles. This demonstrates a significant 14.7% enhancement in comparison to the original material.
- All composites studied at different stress amplitudes (80, 75, 70, and 60 MPa) showed significant improvements in their fatigue life factor (FLIF%) as compared to the metal matrix. The AA7071-5 weight percentage TiO_2 composite significantly improved after heat treatment.

- An analysis using scanning electron microscopy (SEM) was conducted on the heat-treated AA7071-5 weight percent TiO_2 nanocomposite. The results showed that the TiO_2 nanoparticles were evenly distributed throughout the AA7071 substrate. When heat treatment and aging were applied, a microstructure with smaller and more refined grains than the metal matrix was formed.
- Following heat treatment, the Al_2CuMg precipitates in the titanium-free sample underwent a refinement process, resulting in a more uniform distribution across the microstructure.
- Additionally, it was discovered that the inter-dendritic zone included second-phase particles, namely Al Cu Fe and Al (Cu, Mn, Fe, Si). After undergoing heat treatment, the titanium samples showed a decrease in the quantity of Al_3NiCu intermetallic compounds, along with an increase in the occurrence of Ti-Fe rich compounds (Al_9TiFe).

The study's findings indicate notable improvements in fatigue properties and microstructural features by integrating TiO_2 nanoparticles. The use of nanoparticles, in combination with the heat treatment process, greatly enhanced the mechanical properties of the AA7071 alloy. The integration of empirical investigations and computational modeling offers valuable understanding of the fatigue characteristics and changes in the microstructure of the composite material. This has the potential to enhance its mechanical properties and overall efficiency.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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