

Structure Modification and Mechanical Characteristics of Hypereutectic Al-16Si Alloy via TiO₂ Nano-Particles

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ABSTRACT

Al-Si alloy is becoming more and more popular for applications such as pistons, liner less engine blocks, pumps, and other parts that require a combination of lightweight, malleability, formability, and excellent corrosion and wear resistance. However, the structure of hypereutectic Al-Si alloys is characterized by dendritic morphology which limits their formability and enhances their cracks susceptibility so, in an attempt to overcome this problem titanium dioxide (TiO₂) with weight rates of 0.5, 1, 1.5, and 3 weight percent nanoparticles were synthesized via Sol-Gel route and added to the 16 percent Al-Si alloy to follow this effect of structural modification as a direct result of TiO₂ NPs addition, mechanical (static and dynamic) tests were performed. Using scanning electron microscopy and x-ray diffraction, the microscopic structure and morphology of produced TiO₂ NPs are investigated. Mechanical properties of monolithic Al-Si 16% alloy and Al-Si 16% alloy Nano composites such as tensile strength, ductility, hardness, and toughness were investigated. Such results reflect that the maximum enhancement percentage in the tensile strength, ductility, hardness, and toughness of aluminum alloy occurred at 3 wt. % of NPs and reached to 29.5%, 42%, 54%, and 80%, respectively as compared to monolithic alloy. The disintegration of NPs in the melt of aluminum alloy, as well as grain dispersion growth and dendritic growth limitation, could explain this improvement in mechanical properties.

Keywords: Al-16%Si alloy, mechanical properties, sol-gel technique, and titanium dioxide (TiO₂) nanoparticle.

1. Introduction

The globe is rapidly evolving these days, particularly in materials production technology. Engineers and scientists anticipate new materials with lightweight and powerful effects. Because of its great strength and lightweight, aluminium and its alloys are widely used in current industrial applications [1]. Hypereutectic Al/Si alloys have the potential to be used in the aerospace, automotive, and electronics fields because of their lightweight, high castability, fluidity, corrosion resistance, ductility, and wear-resistant structural properties [2]. Because of their excellent castability, high stiffness, excellent electrical conductivity, good corrosion resistance, and wear resistance, hypereutectic Al-Si alloys are the most employed in manufacturing processes, mostly for automotive components like head cylinders and valves and electrical applications like electrical gearbox lines [3]. However, all the previously mentioned features of

hypereutectic Al-Si alloys are dependent on the properties of their casting structure: the arrangement of eutectic and principal Si particles, the size shape or form, and the arm spacing or size of the secondary dendritic cells. Primary particles of silicon can take on a variety of morphologies, which vary based on the conditions of solidification, the alloying elements used, and the chemical composition. These morphologies include feathery, polygonal, star-shaped, blocky, and plate-like. Researchers have demonstrated that the incorporation of nanoparticles can further optimize and strengthen the features of Al-Si alloys, making them even more ideal. In recent years, nanoparticles have led to a huge development in many practical applications because of their special properties. Nanoparticles are in size less than 100 nm. TiO₂ is one of the most valuable inorganic semiconductors due to its vast scientific and technological significance in a variety of possible

applications [4]. The use of nanosized TiO₂ as filler in oil transformers resulted in an increase in dielectric strength and dielectric constant [5]. TiO₂ nanoparticles have been employed as an efficient and harmless semiconductor photocatalyst for the destruction of a variety of organic chemicals, including organophosphorus pesticides [6]. TiO₂ has exceptional hardness, a low density, an elevated melting temperature, superior wear resistance, and stability in chemicals, making it a great material for aluminium matrix nanocomposite AMNCs. It has been demonstrated that wear resistance rises with an increase in the percentage of ceramic granules in aluminium. This is owing to the reinforcement level's stiffness and strong strength [7]. Li et al.'s work [8] enhanced the ductility and mechanical strength of Al-20%Si matrix composites by creating nanoparticle-rich zones (NPRs). Using self-propagating high-temperature synthesis (SHS). Song et al. [9] created nano-TiCp-reinforced Al-13Si-5Cu-2Ni composites. The findings show that the addition of nano-TiCp to Al-13Si-5Cu-2Ni alloy increased the alloy's tensile strength, elongation, and yield strength by 5.7%, 29%, and 6.5%, respectively, as compared to monolithic NPs and graphene nano-platelets (GNPs) into hybrid AlSi10Mg composites produced via laser powder bed fusion (LPBF) was studied by Wei et al. [10]. The impact of incorporating both single and hybrid additions on the microstructure and tensile properties was investigated. The (GNPs + ZrO₂)/AlSi10Mg composites had the lowest elongation of 8 ± 1.8% but the highest tensile strength of 520 ± 12 MPa when compared to single-modified composites. The hybrid-modified composites' strength is mostly increased by Orowan strengthening and grain refinement strengthening, whereas the bimodal microstructure, Si precipitation, and some unmelted ZrO₂ NPs within the cells are the main factors that increase ductility. Borodianskiy et al. [11] used TiC nanoparticles to modify the Al-Si alloy A356. The findings show that the elongation of the altered alloys rose around 20 to 50% in various cast sections, while the hardness and tensile strength enhanced by 18 % and remained constant respectively. The altered alloy samples were analysed under an electron microscope, and the results showed a significant amount of dislocations close to grain boundaries. The mechanical characteristics and morphology of Al-Si alloys supplemented with 0.015% NiO NPs have been investigated by Pratama et al. [12] by raising the stir-casting pour temperature. Using the sol-gel method, NiO nanoparticles were created and sintered for an hour at 400 C. The Al-Si alloys were then mixed with nickel oxide nanoparticles using the stir-casting method. These alloys were then sintered at 720, 780, and 840 C. The results indicate that raising the casting temperature

decreases the toughness values of Al-Si alloys to 0.015% NiO NPs while increasing hardness by 5.68% resulting in fracture morphology being more intense and faceted, similar to crystals. To improve the mechanical characteristics of A356 aluminum alloys, El-Mahallawy et al. [13] employed three types of nanoparticles (aluminum oxide (Al₂O₃), titania (TiO₂), and zirconia) as fillers. The mechanical characteristics of the nano-reinforced castings were improved by using 3 wt. % TiO₂ or ZrO₂ and 2 wt. % Al₂O₃ at a stirring temperature of 600 C with speed of 1500 rpm. Furthermore, a group of aluminum specimens (A390) was generated by Al₂O₃/TiO₂ NPs doped Al-Si. The hardness of the cast samples was examined. The insertion of nanosized particles increased the A390 alloy's microhardness from around 100 to 148 HB. The effect of the insertion of TiO₂ NPs on the mechanical features of AA6061 and AA6082 Alloy has been investigated by Al- Jaafari et al. [7]. The best improvements in mechanical properties occurred at 1.5 wt% of TiO₂ NPs which ultimate tensile strength increased by 12.21 % and 16.29 % for AA6082 and AA6061 respectively, increase in hardness by 12.1% for AA6082, and by 32% for AA6061 and ductility decreased by 32% for AA6061, and by 12.1% for AA6082. Mathur et al. [14] used friction-stirring processing (FSP) to adjust the surface of the aluminum alloy AA 5052 by reinforcing TiO₂. At a higher rotational speed, the TiO₂ region was formed. With a welding speed of 65 mm/min and a tool rotation speed of 1000 rpm, the composite's highest hardness number of 78 ± 1 Hv & and tensile strength of 193.1 ± 3 MPa were achieved. El-wazery et al. [15] have also looked at the use of ZnO NPs made using an easy sol-gel process to enhance the mechanical characteristics of hypereutectic Al-16% Si alloy. Kumar [16] investigated Al-16% Si with varying concentrations of ZnO NPs (0.5-1-1.5-3%), which were produced using the permanent mold casting process. The Al-16wt% Si alloy's mechanical features were all enhanced by the addition of ZnO nanoparticles. The greatest value was obtained at 3% NP concentration. Furthermore, the mechanical characteristics of A356 aluminum alloys were enhanced by increasing the weight % of ZnO NPs. Al-Salihi et al. [17] added 5, 10, and 15 wt. % of Al₂O₃ nanoparticles with an average particle size of 40 nanometers to Al6061 by stir casting. Al-12%Si composites were made using Al₂O₃ NPs included via hot pressing and hot extrusion. For the composites containing 5 weight percent Al₂O₃, the highest values were attained. The YS, UTS, and elongation were 286 MPa, ~30%, and 244 MPa with 5 weight percent Al₂O₃. Furthermore, the influence of Al₂O₃ NPs on the tensile behavior of Al-12%Si was examined [18]. The composites' hardness increased at doping levels of 2 and 5 weight percent before declining at doping

levels of 10 weight percent. Chen et al. [19] investigated the effects of Y_2O_3 and Sc-microalloying NPs on the mechanical characteristics and microstructure of Al-5.5% Si alloy. After Sc and Y_2O_3 nanoparticles are added, the mechanical characteristics of the Al-Si alloy are greatly enhanced. The improvement in the characteristics of Al alloy 6061 by the addition of the Aluminum oxide (Al_2O_3) nanoparticles was investigated [20]. When nano- Al_2O_3 was used as reinforcement instead of base alloy Al6061, the mechanical parameters improved in terms of hardness (156%) and tensile strength (130%). A comprehensive analysis of the data regarding structural modification of Al-Si alloy by various techniques seems to be ambiguous and sometimes costly. The present work examined the mechanical characteristics and microstructure of a hypereutectic Al-16%Si alloy supplemented by TiO_2 NPs. TiO_2 nanoparticles were synthesized by simple sol-gel technique and inserted into Al-16%Si alloy with varying concentrations (0.5, 1, 1.5, and 3 wt.%). The microstructural assessment demonstrated that aluminum grain size refinement is the most important component contributing to the enhancement of mechanical characteristics in the aluminum matrix enhanced with nanomaterial.

2. Experimental procedures

2.1 Synthetization of Titanium dioxide Nano crystalline

The following commercial reagents were employed for the titanium (TiO_2) NPs synthetization without any further purification: Titanium (IV) isopropoxide ($TiC_{12}H_{28}O_4$) from Aldrich, purity 97.0%; glacial acetic acid (CH_3COOH), purity 99.9%; distilled water; and ethylene glycol ($C_2H_6O_2$), purity 99%. The using method for synthesizing TiO_2 NPs is a sol-gel technique. The molar ratio of TTIP, ethylene glycol, distilled water, and glacial acetic acid is maintained at 1:1:200:10, respectively. TTIP and acetic acid were mixed for 30 minutes using a magnetic stirrer. Following that, the mixed solution was gradually supplemented with distilled water and stirred for a further half-hour at room temperature. Before adding ethylene glycol, the reaction temperature of the resultant solution was raised to $90^\circ C$. The resulting mixture was held at the same temperature and stirred for 1 hour. The resultant sol-gel was dried at $250^\circ C$ for an hour. Following that, the powder samples were baked for six hours at $400^\circ C$.

2.2 Preparation of Nano composite

This study used Al-16% Si alloy as its foundation material. The reinforcing phase consisted of titanium dioxide nanoparticles at weight concentrations

between 0.5 and 3%. Table (1) displays the fundamental material's chemical composition. Al-16 Si% was produced using the permanent Mold casting (PM) process. TiO_2 NP concentrations in the alloy under investigation ranged from 0.5 to 3 weight percent. TiO_2 NPs and Al-16 percent Si alloy were combined to have a mass of 0.5 kg. At $650^\circ C$, TiO_2 NPs in different weight percentages were introduced to the Al-16 Si alloy, which was either liquid or semisolid. The resulting mixture was then manually agitated for five minutes. Ultimately, the metal Mold was filled with the melt by hand. Al-Si alloy specimens were cut in compliance with ASTM guidelines both before and after TiO_2 nanoparticles were added.

Table 1- Chemical composition of Al-16 wt. % Si Alloys

composition	Al-16%Si
Cu	0.008
Si	16
Mg	0.019
Fe	0.29
Mn	0.002
Al	Bal

2.3 Microstructure

To elucidate the fracture mechanisms, the surface topography and fracture parameters of the produced Al-16% Si alloy were investigated using scanning electron microscopy (JSM-5400 SEM, JEOL Company, Egypt). TiO_2 NPs microstructure was investigated using JEOL 6060 (SEM) equipped with an energy-dispersive spectrum (EDS) chart.

2.4 Mechanical testing

According to ASTM D638, a small universal testing instrument (Model: PC-2000) with a crosshead speed of 0.28 mm/min was used to perform the ductility and tensile of Al-16% Si alloy supplied with different concentrations of TiO_2 NPs. The measured measurements of the sample under investigation were 165 mm in length, 12 mm in width, and 5 mm in depth. Each sample had five measurements done, and the mean value was calculated to guarantee the data's validity. Using a hardness test (Model: MVH-I) in compliance with ASTM standard E384-04a, Brinell-hardness experiments of Al-16% Si supplied with various TiO_2 NPs were conducted. Hardness test was performed using a 125 kg indentation load and dwell duration of 15 seconds. The test specimen used in this experiment was 15 by 15 and had a thickness of 4 mm. To find the dynamic characteristics of the studied

composite at room temperature, a Charpy impact tester was used.

3. Results and discussion

3.1 Structure of the obtained TiO₂ NPs

The synthesized TiO₂ powder's XRD pattern is displayed in Figure (1). The figure shows that all of the observed diffraction lines are well allocated to the TiO₂ anatase phase that is associated with the tetragonal arrangement according to standardized crystallographic information found in JCPDS files no. 71-1166, with no extra peaks from other phases. The Scherrer equation was used to calculate the normal size of the crystallite (D) of the developed powder based on X-ray line broadening. [21, 22]:

$$D = 0.9\lambda / \beta \cos \theta$$

where θ , λ , and β denote the Bragg diffraction angle, the X-ray wavelength, and the whole width at half maximum (FWHM) of the diffraction peak, respectively. The estimated average crystalline size is 14 nm. The morphology of TiO₂ Nanoparticles was carried out using FE-SEM image analysis. The EDX and FE-SEM micrographs of TiO₂ Nano powder are displayed in Figure 2. As is evident from Figure (2), the obtained shape of TiO₂ particles is a spherical shape with slightly non-uniformity. EDX was employed to estimate the weight percentage of elements present in the synthesized TiO₂ nanoparticles. The EDX spectrum confirms the components Ti and O are present.

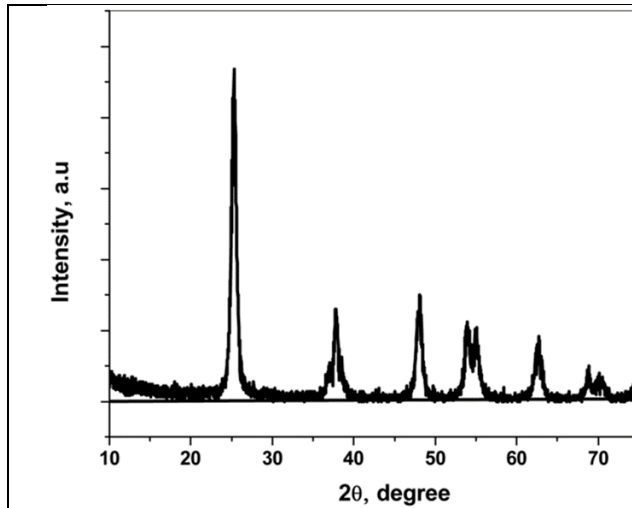


Figure 1- XRD pattern of the obtained TiO₂ NPs

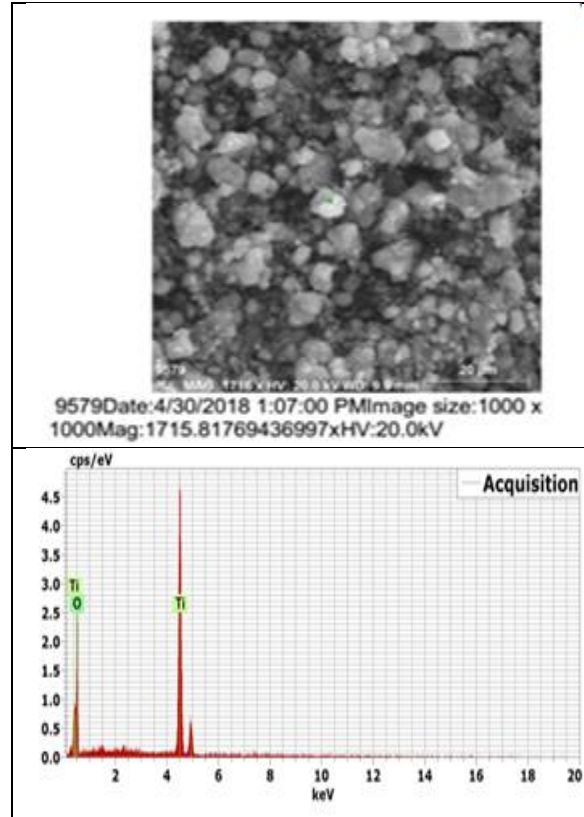


Figure 2-SEM and EDX of TiO₂ nanoparticle, with scale 20µm

3.2 Microstructure

As illustrated in Figure (3), the monolithic hypereutectic alloy is made up of big blocky primary Si and α -Al as needle-shaped eutectic Si. This microstructural feature is attributed to poor mechanical properties. The monolithic Al-Si matrix exhibits extensive pores, cracks, and dendritic structure, as seen in Figure (3.a) and (3.b). The shortage detected in the microstructure of pure Al-Si can be attributed to insufficient mechanical stirring during preparation to confine the plastic strains within the fragmented grains [23], [24]. The fracture surfaces of the Al-16 % Si loaded with various concentrations of TiO₂ nanoparticles from 0.5 to 3 wt. % are shown in Figure (4). It also found that from Figure (4. a) to (4.d), the grain sizes, pores, and cracks in the Al-Si Nano composite are gradually reduced by the insertion of TiO₂ nanoparticles which eliminated the preferred crack preparation channels of Al-Si alloy during the tensile test.

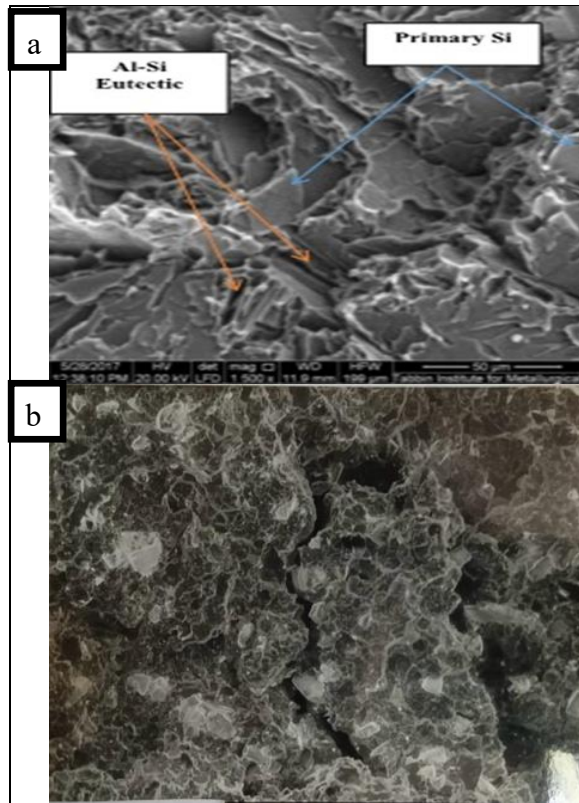


Figure 3- The microstructure of investigation hypereutectic Al–16% Si alloy.

In more specifically, the dispersion of TiO_2 Nanoparticles through the Al-Si matrix may cause restriction of Al grains growth during solidification. This restriction of Al growth leads to refinement in microstructure. Fig (4.d) illustrates the brittle fracture of Al-16%Si with 3 wt. % TiO_2 NPs which show the highest mechanical properties. Due to the possible lack of agglomerated particles, TiO_2 NPs were difficult to identify. As demonstrated by the fracture surface of the Nano-dispersed hypereutectic, which is in agreement with earlier research [25], the primary technique of fracture is the damage of faceted Si particles and debonding at the boundaries between the Al phase and Si elements where the crack crosses the primary Si cleavages location.

3.3 Mechanical behaviour

Tensile strength, ductility, hardness, and toughness tests were performed on the Al-16% Si alloy before and after the addition of varied contents of TiO_2 NPs (0.5, 1, 1.5, and 3 wt.%), as shown in Figures (5–9) and Table 2.

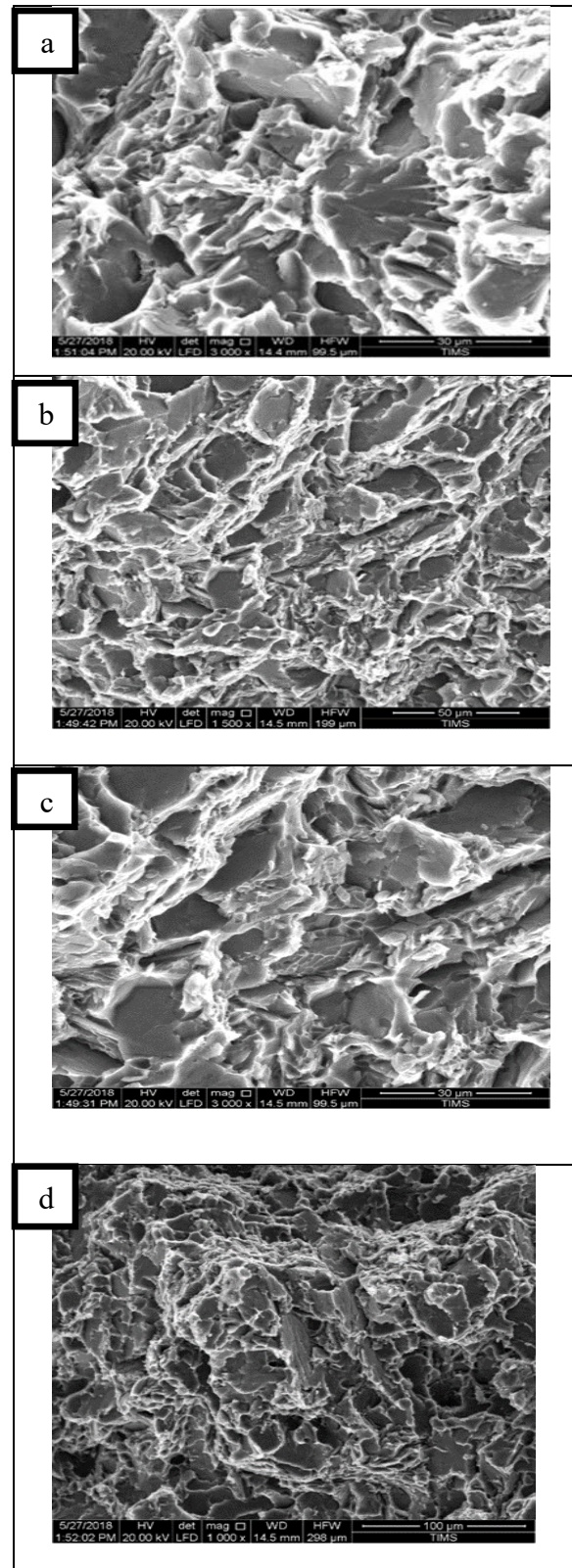


Figure 4-SEM micrographs of hypereutectic Al–16% Si alloys: a) with 0.5wt.% TiO_2 NPs b) with 1wt.% TiO_2 NPs c) with 1.50 wt.% TiO_2 NPs d) with 3wt.% TiO_2 NPs

Such results indicate that the mechanical properties of the alloy were gradually enhanced with adding TiO₂ nanoparticles. In addition, the maximum value of the tensile strength, ductility, hardness, and toughness of this alloy reached 152 MPa, 5.4 %, 54 BHN, and 4.2 J, respectively, at 3wt.% TiO₂ nanoparticle addition. Compared with monolithic alloy, the addition of 3 wt. % NPs resulted in the following increase in percentage in the strength properties for tensile, ductility, hardness, and toughness: 29.5%, 42%, 54%, and 80%. Several strengthening mechanisms are operative in the nanoscale grain size regimen. It is suggested their Orowan hardening contributed to the strengthening in this case. However, as in the present case, the increase in strength while maintaining a high level of ductility suggests the summation of strengthening mechanisms, namely coefficient of thermal expansion, hall-petch orowan, load transfer effect, and elastic modules misfits. The coefficients of thermal expansion for Al, Si, and TiO₂ NPs are respectively 22.2x10⁻⁶ °C⁻¹, 3 x 10⁻⁶ °C⁻¹, and 9 x 10⁻⁶ °C⁻¹. Furthermore, the existence of NPs can lower grain size and boost dislocation production, both of which can increase strength. In addition, TiO₂ NP additions result in an intense strain hardening. This is a result of NPs restricting the matrix's plastic flow. So, the matrix could only flow with the amount of NPs or over the particles during plastic deformation. Figure (5), Figure (6), and Figure (7) shows the gradual increase in tensile strength and ductility percentage with increasing content of TiO₂ NPs. The highest ductility reached 5.4 and occurred at 3wt. % of TiO₂ NPs. The enhancement in tensile strength and elongation ratio is assigned to the refinement of grain size, discussed in the earlier section, and the creation of a robust interface zone between the base matrix and the NPs.

Table 2- Mechanical properties of hypereutectic Al-Si 16% with TiO₂ nanoparticle addition

S. No	1	2	3	4	5
TiO ₂ nanoparticle weight (%)	base	0.5	1	1.5	3
Tensile strength (MPa)	117.5	124	131	140	152
Impact energy (Joule)	2.34	3.45	3.52	3.7	4.2
Hardness (BHN)	35	42	45.5	48	54
Ductility (%)	3.8	4.1	4.3	4.8	5.4

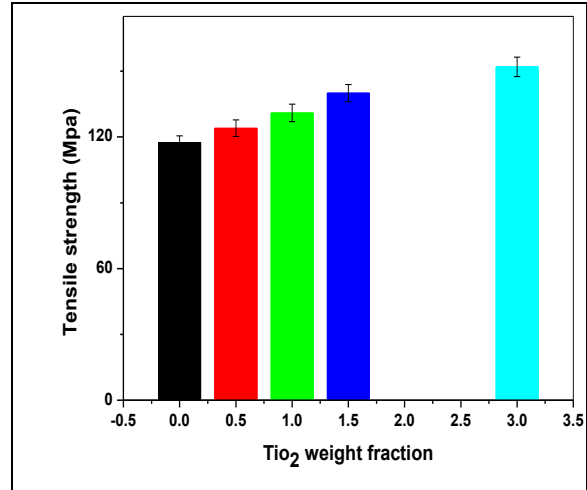
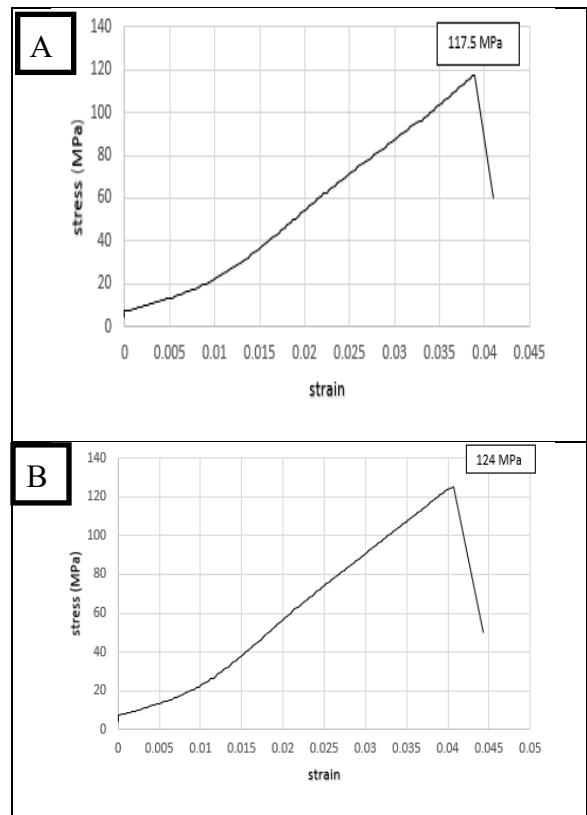


Figure 5 -Variation in tensile strength of hypereutectic Al-16 wt. % Si Alloys with TiO₂ nanoparticle content



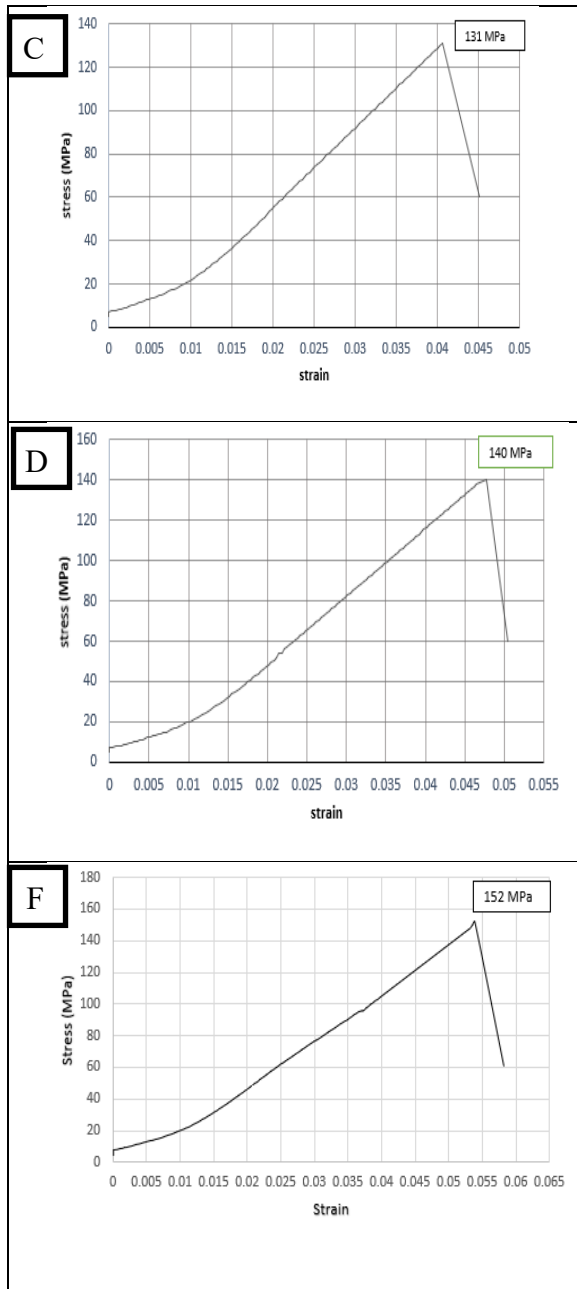


Figure 6-contin.: stress-strain curve for Al-Si 16% at A) without TiO₂ NPs B) with 0.5wt. % TiO₂ NPs C) with 1wt. % TiO₂ NPs D) with 1.5 wt. % TiO₂ NPs E) with 3wt. % TiO₂ NPs

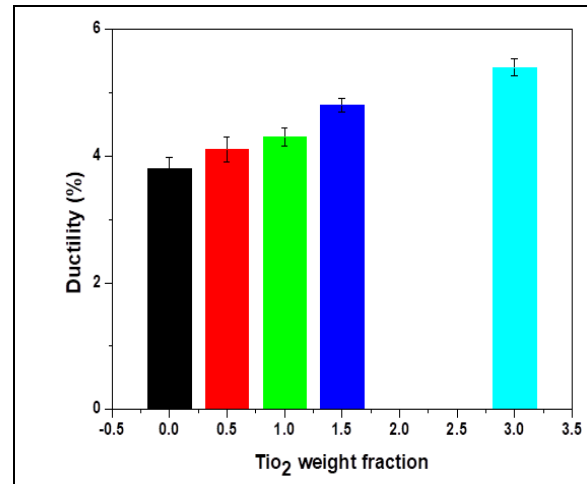


Figure 7 -Variation in Ductility of hypereutectic Al-16 wt. % Si Alloys with TiO₂ nanoparticles content

In addition, NPs enhanced work hardening rate which, in addition to refined effect attributes the high level of strength whether under static or dynamic (impact) conditions while keeping ductility at a high level. So, it seems of high interest to examine the combination of various grain refining techniques as an effective strategy for enhancing the comprehensive mechanical properties of hypereutectic Al-Si alloy. The impact of adding titanium oxide (TiO₂) NPs on the monolithic hardness value is depicted in Figure (8). Hardness is trending upward as the NP addition amount increases. The hardness value enhanced from 35 BHN for base Al-16 Si % alloy to 54 BHN for nanocomposite with 3 wt. % of TiO₂ NPs. The aluminium alloy's refinement and the presence of tougher TiO₂ NPs, which can resist deformation from indentation loads, are responsible for this improvement. More precisely, the TiO₂ nanoparticles scattered throughout the matrix may be useful in preventing the dislocations created by plastic deformation from moving, enhancing the composite's resistance to deformation and raising its hardness as a result [26]. Moreover, the NP layer that is created when TiO₂ nanoparticles are assembled onto the border of the grains can successfully limit the local deformation of the matrix during indentation, increasing the hardness of the material. Finally, finer grain size leads to more grain boundaries and better strengthening of those boundaries, which helps to raise hardness. Figure (9) displays the impact strength value for all Al-Si composite specimens with various concentrations of (TiO₂) nanoparticles. The highest improvement percentage in impact value was obtained at 3 wt. % of TiO₂ nanoparticles and increased by 80% as compared to the Al-Si matrix. An increase in impact strength results from the distribution of nanoparticles

in the Al-Si matrix and the interaction between the added (TiO₂) nanoparticles and the Al-Si metal matrix. Energy absorption at inter-phase interaction between the two phases is considered the main reason for the delayed fracture rate during the impact.

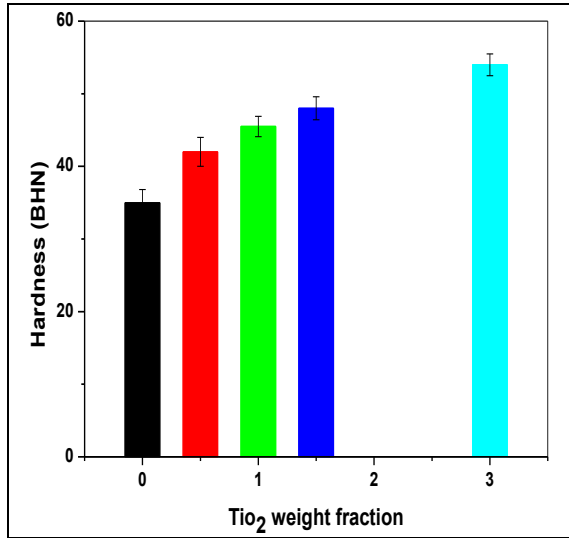


Figure 8 -Variation in hardness of hypereutectic Al-16 wt. % Si Alloys with TiO₂ nanoparticles content

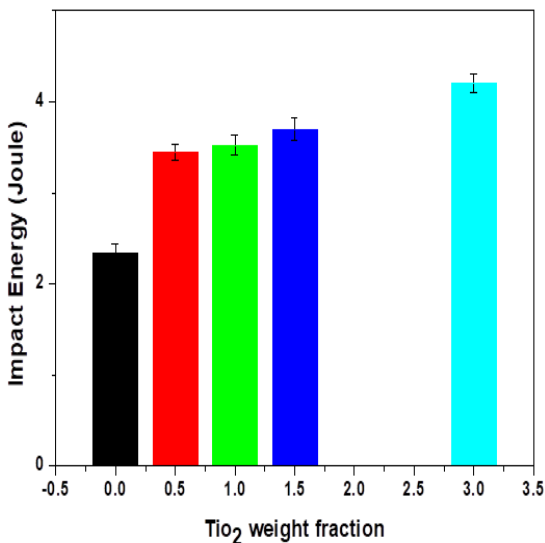


Figure 9-Variation in impact energy of hypereutectic Al-16 wt. % Si Alloys with TiO₂ nanoparticles content

4. Conclusions

The sol-gel method was used to synthesize titanium dioxide (TiO₂), which was then added to the 16 percent Al-Si alloy. By using XRD measurement, the TiO₂ anatase phase with a crystalline size of 14 nm is confirmed. The addition of TiO₂ nanoparticles to hypereutectic Al-16wt % Si alloy enhanced the mechanical properties and microstructure. The tensile strength, ductility, hardness, and toughness of this alloy were enhanced by percentages 29.5%, 42%, 54%, and 80%, which occurred at 3 wt. % of TiO₂ NPs. Microstructural evaluation of the composite material is done by structural analysis that shows that TiO₂ NPs are uniformly distributed in the metal matrix with slight agglomeration, which depends upon the process that is followed for the composite fabrication. At high magnification, there is evidence that nanoparticles may be able to be incorporated and trapped within the interdimeric interface that forms as the scattered alloys solidify.

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