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Synthesis and Evaluation of Mechanical Properties of Nano TiO_2 Reinforced Aluminium Metal Matrix Nano Composites (MMNCs)

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In the present investigation, aluminium Metal Matrix Composites (MMCs) were synthesized by reinforcing nano TiO_2 particles at varying weight percentages (0.5 ,1.5,2.5 and 3.5 wt%) into an AA6061 molten aluminium matrix using the stir casting method. The synthesized composites were characterized using optical microscopic analysis, Scanning Electron Microscopy (SEM) and X- ray diffraction (XRD). The hardness and tensile properties of the composites were evaluated. The analysis of mechanical properties revealed that the T6 composite containing 0.5 wt% nano TiO_2 particles had a maximum hardness of 79.6 BHN compared to other T6 samples and as well as ac-cast samples. Ultimate tensile strength and yield strength of T6 composite containing 0.5 wt% TiO_2 was found to be (180 MPa and 135 MPa) higher than other T6 composites and as cast samples. Fractography of Al MMC containing 0.5 wt% nano TiO_2 particles revealed ductile -brittle fracture mode in the T6 condition.

Keywords: *Al MMC, Nano TiO_2 particles, Weight percentage (wt%), Heterogenous nucleation sites, Grain refinement, Wettability, Fractography.*

1. Introduction

Aluminium Metal Matrix Composites (MMCs) have been widely used in automotive, construction and aerospace applications due to its properties such as; high strength to weight ratio,wear resistance and corrosion resistance[1-6]. Ceramic nano particulate reinforcements are considered as a favorable option for aluminium to impart high strength to weight ratio compared to microsized ceramic particulate reinforcements[7]. Nagaral et al.[8] reported that Al MMCs reinforced with nano B_4C particulates, exhibited notable improvements in mechanical properties. Similar results were reported by Nagaral et al.[9] in the study of mechanical properties of Al MMCs reinforced with nano ZrO_2 particulates. There are different ways where Metal Matrix Nano Composites (MMNCs) can be synthesized such as; stir casting, powder metallurgy, squeeze casting, etc. Among them, stir casting is well recognized for its cost-effectiveness and simplicity. Stir casting process mostly worked in the manufacture of MMCs[10] and as well as MMNCs as it produces composites with uniform reinforcement distribution[11].

Nano composites are materials that integrate nano particles into a matrix of standard materials and alloys. The addition of nano particles result in a significant enhancement of performance, which may include strength, durability in addition to electrical and thermal conductivity. The efficiency of nano particles is such that the extent of added material generally represents merely 0.5 to 15% by weight. Higher surface area of the nano particles increases interface surface area of the matrix and particles. The material properties of the MMNCs are significantly superior to those of the traditional Metal Matrix Composites.

According to several studies[11], MMNCs are a combination of a metal matrix and dispersed nano ceramic particles or a metal phase. SiC, Boron, Al_2O_3, TiO_2, SiN, B_4C, B_4N are widely used ceramic reinforcements. MMNCs are used in space shuttles, airplanes, electronic media, bicycles, automobiles, mechanical and wear applications. Compared with aluminium MMCs reinforced with microscopic ceramic particulates, Al composites reinforced with nano particulates exhibited significant improvements in physical and mechanical properties such as strength, modulus of elasticity, fatigue strength and resistance to wear loss[12-15].

In the present investigation AA6061 Alloy reinforced with varying weight percentages (wt%) of TiO_2 nano ceramic particles were synthesised by using stir casting route in contrast to several investigations carried out with micro ceramic reinforcements as observed in the studies on the wear behaviour of Al MMCs reinforced with ZrO_2 and graphite reinforcements, carried out by Sampath Kumar et al.[16]. The synthesized composites were characterized and tested to study the effect of wt% of TiO_2 particles in influencing the mechanical properties of Al MMCs. Several investigations indicated that particle weight percentage and particles size influenced the microhardness of Al MMCs reinforced with ceramic reinforcements.

2. Materials and Methods

AA6061 alloy was used as the base metal in the present work. TiO_2 nano particles having average particle size of 30nm was used as the reinforcement at varying wt% in this study.

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The chemical composition of the as-cast base alloy and composites obtained using Optical Emission Spectroscope (OES) is showed in Table 1.

OES analysis revealed that Ti wt% increased with increasing wt% of TiO_2 ceramic particulates , confirming that TiO_2 particulates have got mixed in the Al alloy.

Stir casting setup used for Al MMNCs synthesis is showed in Figure 1. The aluminium base alloy was placed in a crucible and heated in the furnace to melt the aluminium. The molten aluminium was maintained at a constant temperature of 850°C for pouring. The nano TiO_2 particles (Figure 2) preheated at 400^0 C for 1 hour,were gradually added to the molten aluminium and the mixture was stirred using a stirring rod at a constant speed of 500 rpm for 15 minutes to achieve initial dispersion. A small amount of magnesium (Mg) was introduced into the molten composite material to improve wettability.

To further enhance the dispersion and prevent agglomeration of nano TiO_2 particles, ultrasonification was done at 20 kHz for 3 minutes. Ultrasonication was followed by coveral flux addition in the molten composite to prevent oxidation of Al MMNCs and for removal of impurities. The molten composite material was then carefully poured in to mould preheated at 200^0C. The samples were then allowed to cool and solidify at room temperature.

The synthesized composites were subjected to T6 treatment. In the T6 treatment, composites were solutionized at 530°C for 1 hour in order to homogenise the solid solution, followed by quenching in water. Solutionized composites were artificially aged at 185°C for 6 hours followed by air cooling. This process enables the precipitation of the alloying elements and improves the wettability of TiO_2 particles with the matrix leading to an increase in the strength and hardness of the material.

3. Results and Discussions

3.1. Microstructure

Microstructure of base alloy exhibited α - Al phase and eutectic network as showed in Figure 3. Microstructure of the as-cast composites and T6 composites (Figures 4, 5, 6 and 7) showed α-Al phase and eutectics network with grains found to be finer than as-cast composites.

Grain size of composites found to decrease with increasing wt% of TiO_2 particles due to increase in number of heterogenous nucleation sites in the as-cast and T6 composites. In heat treated condition grains were found to be comparitively finer and equiaxed than as-cast composites.

Figure 1. Stir casting set-up used in the investigation.

Figure 2. Preheated TiO_2 nano particles.

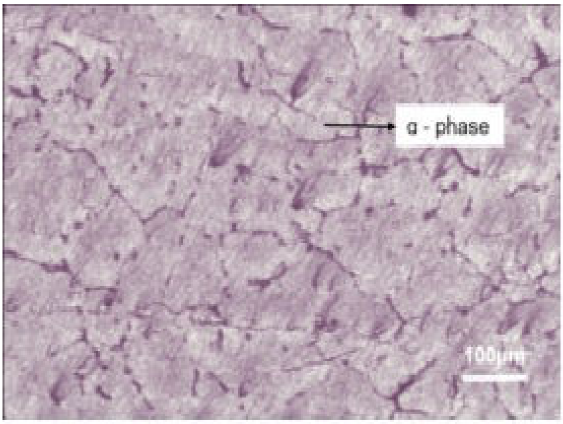

Figure 3. Microstructure of base alloy AA 6061.

Table 1. Chemical composition of base alloy and composites in as-cast condition.

Samples	Wt% of elements						
	Ti	Fe	Si	Mg	Cr	Cu	Al
Base alloy (AA6061)	0.026	0.280	0.585	0.861	0.081	0.228	Remaining
AA6061 + 0.5 wt% TiO_2	0.360	0.2786	0.5821	0.8567	0.0806	0.2269	Remaining
AA6061 + 1.5 wt% TiO_2	0.9246	0.2758	0.5762	0.8481	0.0798	0.2252	Remaining
AA6061 + 2.5 wt% TiO_2	1.5237	0.2730	0.5702	0.8385	0.0790	0.2236	Remaining
AA6061 + 3.5 wt% TiO_2	2.1228	0.2702	0.5645	0.8309	0.0782	0.2223	Remaining

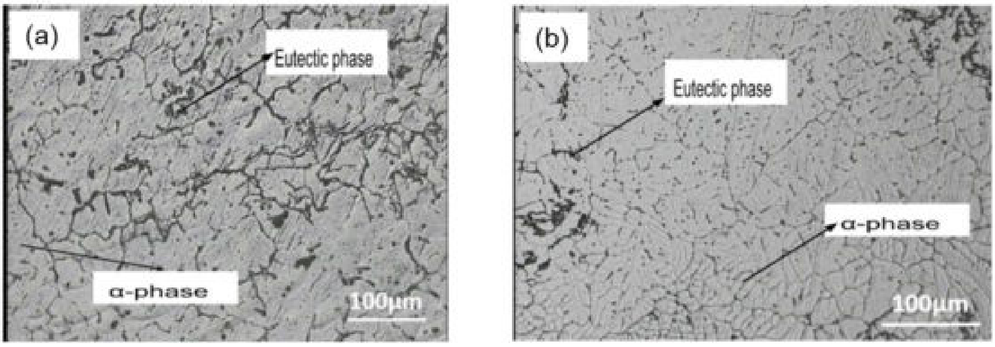

Figure 4. Microstructure of Al MMNCs reinforced with 0.5wt%TiO_2 (a) as cast condition (b) heat treated condition.

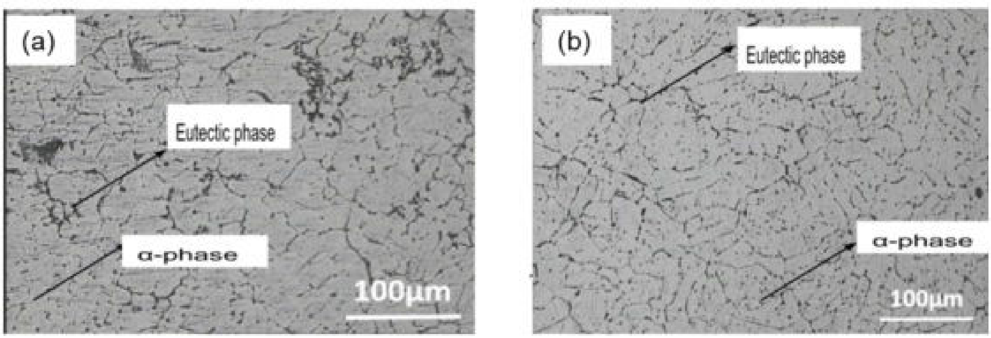

Figure 5. Microstructure of Al MMNCs reinforced with 1.5wt%TiO_2 (a) as cast condition (b) heat treated condition.

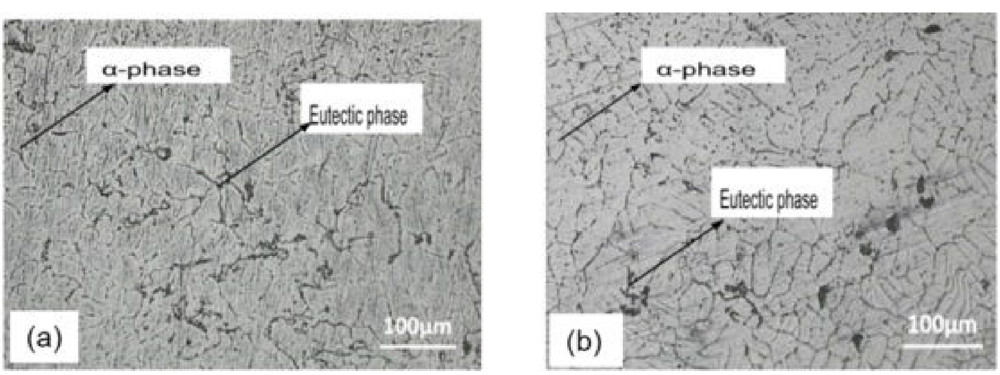

Figure 6. Microstructure of Al MMNCs reinforced with 2.5wt%TiO_2 (a) as cast condition (b) heat treated condition.

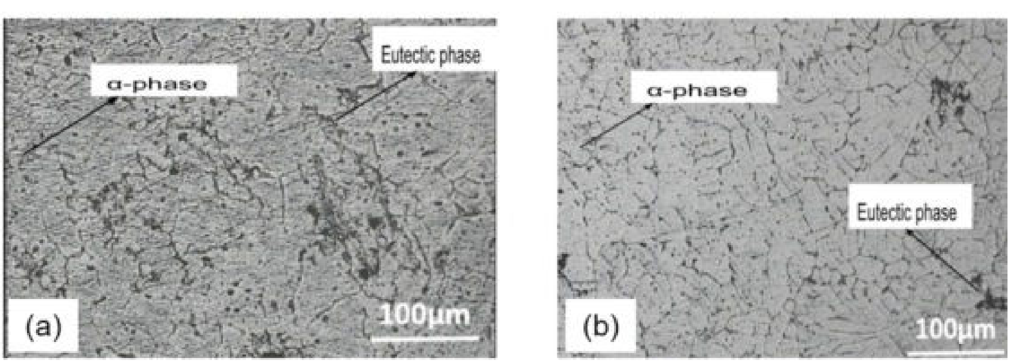

Figure 7. Microstructure of Al MMNCs reinforced with 3.5wt%TiO_2 (a) as cast condition (b) heat treated condition.

This is possibly due to overcoming segregations imparted by the addition of TiO_2 particles in the as-cast composites by T6 treament which also enhanced wettability of TiO_2 particles with the matrix.

3.2. XRD analysis

Results of the XRD analysis of the base alloy and composites containing 0.5wt% TiO_2 particles are shown in Figures 8 and 9 respectively. Presence of TiO_2 peak at 2θ value of 41.2° for MMC reinforced with 0.5wt% TiO_2 in the obatined XRD pattern (Figure 9) confirmed the successful incorporation of TiO_2 in the composites.

Base alloy and 0.5 wt% TiO_2 nano particles containing composite exhibited Al peaks at 2θ values of 38.61°, 44.5°, 65.3°, 78.2° and 82.3° as showed in Figures 8 and 9. Further, Al_2TiO_5 peak was also observed in 0.5 wt% TiO_2 nano particles containing composite at 2θ value of 34.5°.

3.3. Hardness

The hardness test was conducted in accordance with ASTM E 10 standard using a brinell hardness tester with a ball indenter of 10mm diameter and a load of 500 kgf. Table 2 shows the hardness values of base alloy and composites in the as-cast condition. In the as cast condition, Al MMNC reinforced with 0.5wt% TiO_2 had showed highest hardness than other composites (Figure 10). Hardness of 0.5 wt% TiO_2 nanoparticles reinforced composite was found to be 54.77 BHN. Table 3 shows the hardness value of base alloy and composites in T6 condition. The hardness values found to be higher in the T6 samples compared to respective as-cast samples.

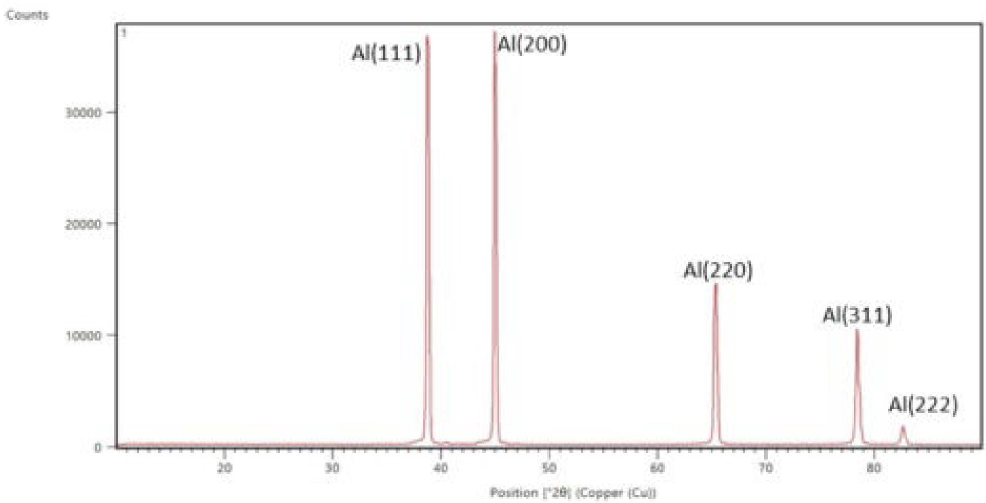

Figure 8. XRD of base alloy AA 6061.

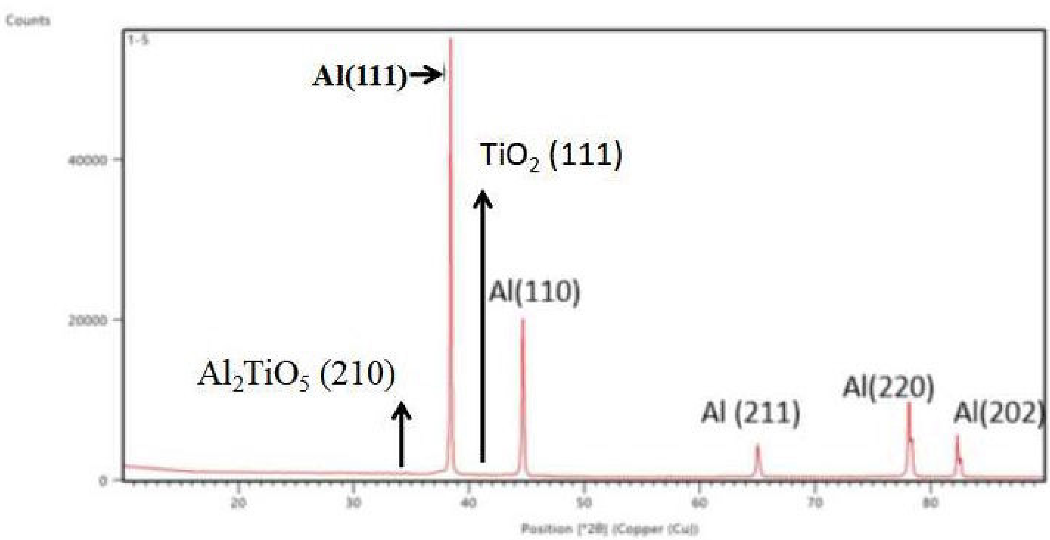

Figure 9. XRD of Al MMNC reinforced with 0.5wt% TiO_2 nano particles.

Table 2. Hardness values of base alloy and composites in as-cast condition.

Wt% of TiO_2 in composites	Brinell Hardness Number (BHN)
Base alloy	48
0.5	54.77
1.5	52.49
2.5	48.61
3.5	38.95

Table 3. Hardness values of base alloy and composites in heat treated (T6) condition.

Wt% of TiO_2 in composites	Brinell Hardness Number (BHN)
Base alloy	63
0.5	79.61
1.5	69.14
2.5	64.64
3.5	54.21

This can be attributed to grain refinement in the T6 samples which impedes dislocation, leading to increase in hardness since impedement to dislocation increases force to cause deformation (indentation). 0.5 wt% TiO_2 reinforced Al MMNC showed higher hardness (79.6 BHN) than other composites in the T6 condition (Figure 11). Hardness of composites found to decrease with TiO_2 wt% due to non-uniform wettability of reinforcement particles with the matrix.

Studies in nanoparticles reinforced Al MMNCs, reported that hardness and tensile strength increased significantly compared to base alloy[17], as well as to micro ceramic particles reinforced Al composites[18] due to uniform dispersion of nano-particles , orowan strengthening and grain refinement[19-21].

3.4. Tensile studies

The Al MMNC reinforced with 0.5wt%TiO_2 showed higher tensile properties compared to 1.5wt%, 2.5wt% & 3.5wt% TiO_2 reinforced composites (Table 4 and Figure 12).

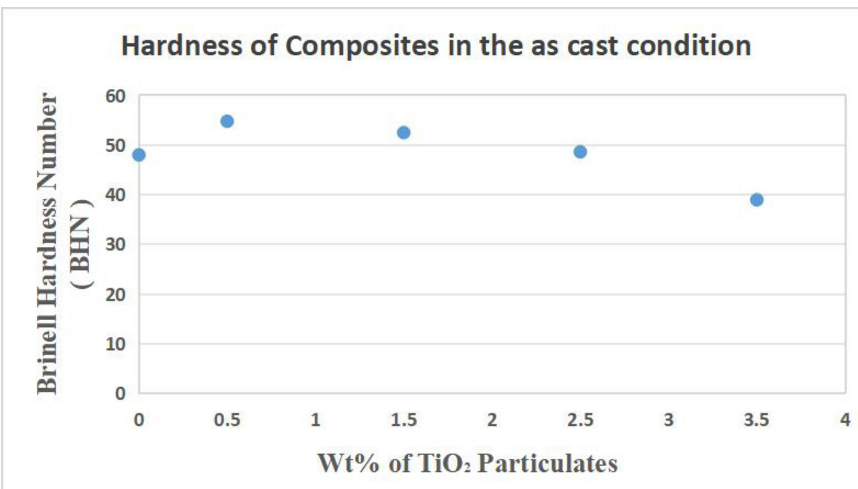

Figure 10. Hardness of the composites in the as cast condition.

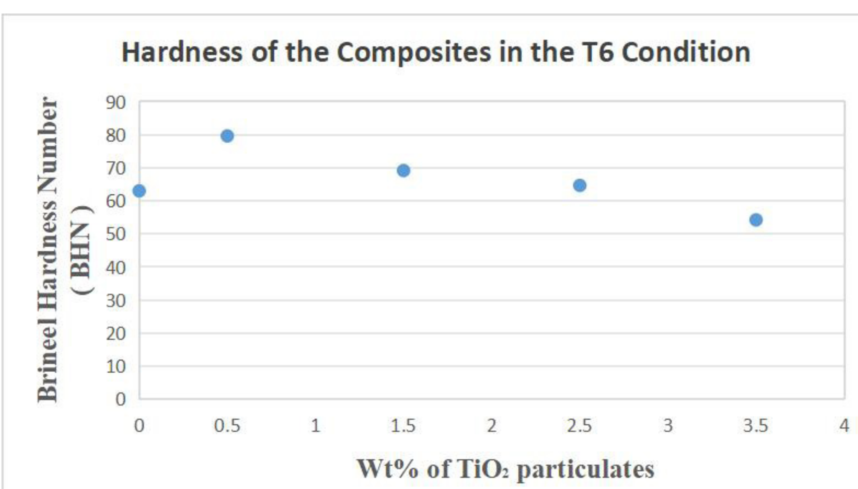

Figure 11. Hardness of the composites in the T6 condition.

Fractured surface analysis of the T6-treated Al MMNC reinforced with 0.5 wt.% nano TiO_2 revealed a combination of fine dimples and localized cleavage facets, indicating a mixed mode of ductile–brittle fracture. The presence of uniformly distributed fine dimples can be associated with micro-void nucleation, growth, and coalescence around the well-bonded TiO_2 nanoparticles, suggesting effective load transfer from the aluminium matrix to the reinforcement. The strong interfacial bonding and uniform dispersion of nano TiO_2 particles restricted localized plastic deformation and delayed crack initiation, thereby contributing to enhanced tensile strength. Strong interfacial bonding and uniform dispersion of nano particles were also reported in some studies as main factors for the enhancement of tensile strength[1,5,14,17,20] due to imepedement of dislocations.

In contrast, composites containing higher weight percentages of TiO_2 exhibited comparatively flatter fracture surfaces with increased cleavage features and reduced dimple density. This behavior can be attributed to particle agglomeration and non-uniform wettability at higher reinforcement levels, which act as stress concentration sites and promote premature crack initiation. The reduced plastic deformation capability and easier crack propagation in these regions lead to a predominantly brittle fracture mechanism, resulting in a decline in tensile properties.

T6 composite containing 0.5 wt% TiO_2 particles exhibited an ultimate tensile strength of 180 MPa which is 10% greater than T6 base alloy. Yield strength of 0.5 wt% TiO_2 containing composite (135 MPa) was found to be 12% higher than yield strength of T6 base alloy. These results are comparable to Al MMCs reinforced with hybrid reinforcements[4,6,12,18].

Fractography analysis of 0.5wt% TiO_2 reinforced Al MMNC in the T6 condition showed the presence of cleavage and dimple features confirming mixed mode of fracture (both ductile and brittle fracture) in the composites as showed in the Figure 13. Mixed mode fracture occurred due to simultaneous ductile deformation of the aluminium matrix and brittle fracture associated with stress concentration around stiff nano-TiO_2 particles and their interfaces. This also indiacted that with increasing wt% of TiO2 particles composites would have undergone brittle fracture.

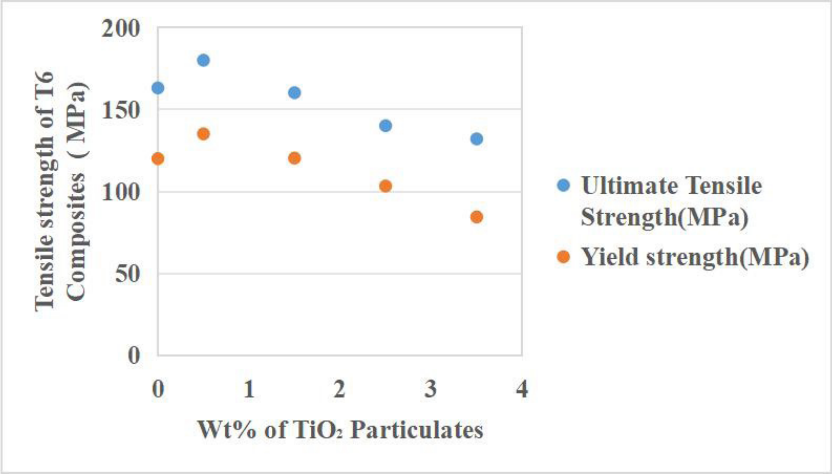

Figure 12. Tensile properties of TiO_2 reinforced Al MMNCs in the T6 condition.

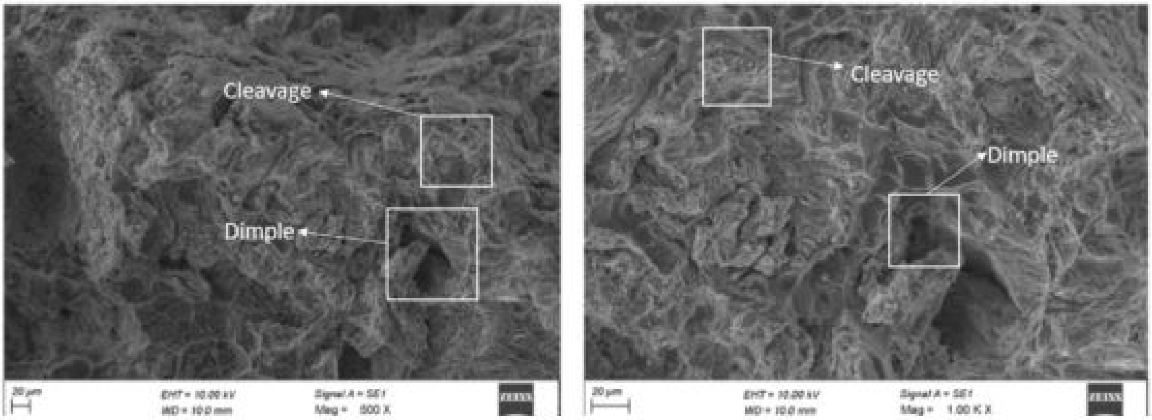

Figure 13. Fractography images of 0.5wt%TiO_2 reinforced Al MMNC in the T6 condition.

Table 4. Tensile properties of base alloy and composites in the T6 condition.

Wt% of TiO_2	Ultimate Tensile Strength (MPa)	Yield strength (MPa)
Base alloy	163.13	120.05
0.5	180.01	135.12
1.5	160.22	120.36
2.5	140.10	103.32
3.5	132.05	84.45

4. Conclusion

- OES analysis showed that samples reinforced with varying wt% of TiO_2 nano particles exhibited higher amounts of Ti than the base alloy.
- XRD analysis confirmed the presence of TiO_2 at 2θ value of 42.2° in the 0.5 wt% TiO_2 reinforced composite.
- Microstructure of the as-cast and T6 samples showed α-Al phase and eutectic network. Microstructure of the T6 composites showed relatively fine and equiaxed grain structure. Al MMC containing 0.5wt% TiO_2 particled had undergone higher grain refinement due to strong wettability of TiO_2 particles at the heterogenous nucleation sites in the matrix.
- In the as-cast condition and T6 condition, the hardness value of composite containing 0.5wt% TiO_2 particles (54.77 BHN and 79.61 BHN) was found to be higher than the other composites and as well as base alloy.
- Composite containing 0.5wt% TiO_2 particles showed higher ultimate tensile strength (180 MPa) and yield strength (135 MPa) compared to base alloy and other compoistes in the T6 condition.
- Higher hardness and tensile properties of 0.5 wt% TiO_2 particles containing composite can be attributed to effective impedement of dislocation in the matrix by reinforcements which had strong wettability with the matrix.
- Fractography of 0.5wt% TiO_2 reinforced composite showed mixed mode of fracture and indiacted brittle fracture in other composites.

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Data Availability

The entire dataset supporting the results of this study was published in the article itself