Electrical conductivity, magnetic and fatigue properties of aluminum matrix composites reinforced with nano-titanium dioxide (TiO₂)

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ABSTRACT
Aluminum matrix composites are widely used in many structural application due to properties like high specific strength, wear resistance, lightweight and fatigue properties. The aim of the current work is to investigate the effect of the addition of nanosize TiO₂ particiles to AA6063-T4 aluminum alloy on the electrical conductivity, magnetic and fatigue properties of composites produced by stir casting method. It was revealed that the AA6063-T4/TiO₂ composites were successfully prepared with 3 wt%, 5 wt% and 7 wt% TiO₂ using a stir casting technique with electrical conductivity increasing with increasing amount of TiO₂. Also it was found that the electrical conductivity of all composites was higher than the base metal matrix and the conductivity was proportion to the frequency for both matrix and composites. The magnetic studies revealed an improvement of the nanocomposites in comparison with the base metal. The fatigue life and fatigue strength of 7 wt% TiO₂ composite was found to be higher than that of other composites and base matrix.

GRAPHICAL ABSTRACT

Introduction
Aluminum and aluminum alloys are commonly used as a matrix for metal matrix nanocomposites due to their high corrosion resistance, easy production, low weight and low cost [1]. In some cases aluminum matrix composite parts are produced by stir casting methods and used in electrical application. Mechanical properties of aluminum matrix nanocomposites (AMNC) are generally improved by the addition the nanoreinforcements for strength- hardness, wear resistance and fatigue. Zhao et al. [2] characterized the properties of Al-matrix composites reinforced with the Al₂O₃ nanoparticles. It was concluded that ultimate tensile strength, elongation and yield stress of the nanocomposites are improved with an increase in the nanoparticle loading. A356.1 aluminum alloy reinforced with nano ZrO₂ was successfully fabricated via a stir casting technique. Mechanical properties revealed that the presence of ZrO₂ in aluminum matrix significantly improved the wear and hardness of A356.1 aluminum alloy reinforced with 0.5, 1.0, 1.5 and 2.0 wt % ZrO₂. The results reported that the hardness and, wear properties of the nanocomposite are enhanced compared to the neat metal matrix [3]. Divagar et al. [4] studied the production of metal nanocomposites by using a stir casting process and examined the impacts of SiC and Al₂O₃ nanoparticulates on the fatigue strength of metal matrix composites with varying weight percentage of SiC (5, 10 and 15 wt%) and constant weight percentage of Al₂O₃ (5 wt%) as reinforcements and AA7075-T651 as base metal. It was concluded that the effects of reinforcement on

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fatigue life increased with the amount of nanofiller. Also the fatigue strength of metal matrix composites have shown a proportional relationship with the percentage of nanoparticle reinforcement. Khairy and Gouda [5] tested the electrical conductivity ($\delta_{AC}$) of polyaniline NiFe$_2$O$_4$ nanocomposites (PANI/NiFe$_2$O$_4$) with different amounts of filler (2.5, 5 and 50 wt%). They reported that the electrical conductivity of nano composites was found to increase with NiFe$_2$O$_4$ nanoparticle content. AC conductivity of nano grain size TiO$_2$ filled epoxy composites was also measured. A nanosize TiO$_2$ with a grain size 50 nm was used as filler at 3 and 5 wt % and it was found that AC conductivity for all samples increased with increasing frequency and $\delta_{(w)}$ referred to electronic polarization [6]. AC electrical conductivity ($\delta_{AC}$) depends on frequency ($w$) according to the following relationship [2].

$$\delta_{AC}(w) = Aw^S$$  \hspace{1cm} (1)

where, $A$ is the proportional factor and $S$ is the exponent factor.

Islak et al. [7] fabricated CU-TiC composites by hot pressing technique and examined the electrical conductivity at 600, 700 and 800 °C sintering temperature for 4 min and 50 MPa applied pressure. It was concluded that the CU composite with 10 wt% TiC exhibited the highest electrical conductivity for 800 °C sintering temperature and that the value for the highest AC conductivity was 68% of the International Annealed Copper Standard (IACS). Hussain [8] fabricated and electrically tested hybrid nanocomposites based on pure aluminum as a matrix with nanoreinforced Fe$_2$O$_3$ (1.5, 2.5 and 5 wt%) and 2 wt% Al$_2$O$_3$ filler content using a powder metallurgy route. It was found that the highest electrical conductivity was observed for composites containing 1.5 wt% Fe$_2$O$_3$ + 2 wt% Al$_2$O$_3$. The synthesized nanocomposites containing 1.5 wt% Fe$_2$O$_3$ + 2 wt% Al$_2$O$_3$ showed good electrical conductivity with improvement of 42% compared to neat metal matrix. Therefore, the nanocomposites seem to be promising for micro-electromechanical systems. Farah et al. [6] study the AC conductivity of a composite filled with TiO$_2$ with grain sizes of 1.5 μm and 50 nm. The concentration of TiO$_2$ was 3 and 5 wt%. They observed that the AC conductivity increased with frequency for both micro and nano composites according to the relation $\delta (w) = A w^S$ with the value of exponent ($S$) less than unity. Subbash et al. [9] prepared cadmium oxide (CdO) nanocomposites using a chemical oxidation polymerization technique. It was observed that the conductivity of these nanocomposites increased by 5% with increasing content of CdO. Magnetic materials have become an attractive research subject for industrial applications such as computers, video, electrical motors and transformers. The main properties for magnetic materials are the absolute magnetic ($\mu_{abs}$) and magnetic susceptibility ($X_m$) and these parameters can be described by the following formula [10].

$$\mu_{abs} = (1 + X_m) \mu_e$$  \hspace{1cm} (2)

where

$$X_m = \frac{M}{H}$$  \hspace{1cm} (3)

$\mu_e$ is the vacuum permeability ($4\pi \times 10^{-7}$) Henry/m, $H$ is the magnetic field A/m and $M$ is the magnetization A/m.

It was found that an increase in the amount of nanoparticles increased the magnetic properties of the composite material. The magnetic properties are generally improved with smaller grain size [11]. The reason for this decrease in grain size could be the presence of nanoparticles and introduction of low conductive nature of the reinforcements. Ferreira et al. [12] prepared and tested aluminum matrix composite reinforced by naniron oxide (Fe$_3$O$_4$) and they concluded that these composites can be produced easily by controlling the parameters of the process of fabrication. The composites incorporating 10, 20 and 30 wt% Fe$_3$O$_4$ were examined to obtained their electrical conductivity and magnetic properties and a significant improvement in magnetic properties was observed while electrical conductivity decreased with increasing nanoparticles content compared to the pure matrix.

Fatigue behavior has progressively be more understood and is key in the development of a large amount of equipment such as aircraft components, oil and gas, compressors, tanks, pumps, turbines, pistons and many more components subjected to repeated loading and vibrations. Published data on nanoparticle reinforced metal matrix composites have traditionally focused on quasi-static tensile properties and tribological behavior but published data on fatigue properties are more scare. Divagar et al. [4] studied the effect of dual nano sized particle reinforcement on the fatigue life of AA7075-T651 based metal matrix nanocomposites produced by a stir casting method. They reported that the nanocomposites containing 10 wt% SiC + 5 wt% Al$_2$O$_3$ exhibited 12% higher fatigue strength than the matrix and other composites, with the grain size distribution responsible for the high and improved fatigue behavior of the nanocomposites. As the amount of particulate increased, it changed the fatigue properties due to matrix strengthening as a result of increased dislocation density. Those fine nano size particles were responsible for improved fatigue life and strength [13]. The present study
focuses on the fabrication and evaluation of electrical conductivity, magnetic properties and constant fatigue behavior of AA6063-T4 matrix reinforced with TiO2 nanocomposites.

**Experimental**

**Electrical testing**
The nanocomposites and metal matrix samples were electrically tested using the (1V1UMSTAT-XR) device shown in Figure 1. The test conditions were (1 A) average current, (1 V) voltage and room temperature (RT). The frequency tests ranged from 10 KHz to 10MHz. The electrical test outputs are capacitance ($C_s$), real and imaginary permittivity ($\varepsilon_r$), resistivity ($R_s$) and dielectric loss ($\delta$). All the above tests were conducted at Tarbiat Modares University in Tehran.

**Magnetic testing**
A vibrating sample magneto meter (VSM) device was used to measure the magnetic field against magnetization at 25° C (RT). At a constant frequency.
The tests were done at Tehran University and Figure 2 shows the VSM device. This test measures the magnetic properties of specimen using a vibrating sample magnetometer device (Magnates Doghight Kavir) at instants constant frequency. The test was carried out to measure the magnetization (M) against the magnetic field (H) at room temperature. As the magnetic field varied over the ultimate range, the magnetization was measured by sense coils with a lock in amplifier. The VSM device output is the variation of magnetic field (H) versus the magnetization (M) range. It also measures $X_m$ to obtain $\mu_{abs}$ from Equation (2).

**Composite fabrication**

The AA6063-T4/TiO2 composites were fabricated using the stir casting method. The average size of the TiO2 particles was 40 nm [14]. The procedure of the mixing process can be summarized by the following steps:

- Melting the AA6063-T4 using an electrical furnace at 850 °C for 25 min.

| AA6063-T4 (g) | TiO2 (wt%) | TiO2 (g) | Total weight (g) |
|---------------|------------|----------|------------------|
| 970           | 3          | 30       | 1000             |
| 950           | 5          | 50       | 1000             |
| 930           | 7          | 70       | 1000             |

**Fatigue testing**

Rotating bending fatigue tests were used to create an S-N curve based on, constant amplitude tests, for both the composites and the matrix. The applied stress is calculated from the following equation:

$$\sigma_b = \frac{125.7 \times 32 + P}{\pi d^3} \quad (4)$$

where, $\sigma_b$ is the bending stress (MPa), $P$ the applied load (N), the force arm is equal to 125 mm and $d$ is the minimum diameter of the fatigue sample which is shown in Figure 3.

The fatigue testing machine used is a Schenck type PUNN rotating bending machine. A rotating sample is clamped on both sides and loaded and rotating about its own axis by a motor. After a specific number of load cycles, the sample undergoes rupture due to material fatigue. The cycles until fracture are monitored using a counter while keeping the applied stress constant. A value of load ($P$)

| Element | S | Ti | Zn | Cu | Fe | Mg | Mn | Cr | Sr |
|---------|---|----|----|----|----|----|----|----|----|
| Standard | 0.503 | 0.010 | 0.048 | 0.043 | 0.43 | 0.451 | 0.048 | 0.011 | 0.0006 |
| Experimental | 0.490 | 0.009 | 0.040 | 0.033 | 0.41 | 0.392 | 0.029 | 0.009 | – |

**Figure 3.** Fatigue specimen, shape and dimension in mm according to standard specification of DIN 50113.

**Figure 4.** Fatigue testing (SCHENCK PUNU).
in Newton (N) is applied on the specimen leading to a known value of stress \( \sigma_0 \) in MPa and is extracted from Equation (4). All fatigue tests are performed at 1400 rpm (23.34 Hz). The fatigue test rig is shown in Figure 4.

**Results and discussion**

**Chemical composition**

Chemical analysis of AA6063-T4 was performed by the Iraq Geological Survey and results are compared with standard. The chemical analysis has been implemented in an Atomic Fluorescence Spectrometer (AFS) device. Table 2 gives the chemical composition of the AA6063-T4 base metal.

**Mechanical testing**

To obtain the mechanical properties of the metal matrix and their composites, tensile tests were carried out using a computerized uni-axial testing machine as per ASTM standard [15]. Figure 5 shows the specimen dimensions according to ASTM E8/E8M-09.

Three samples were used for each test and average values are reported. Tensile properties such as, ultimate tensile strength (UTS), yield stress (YS), modulus of elasticity (E) and strain at break were extracted from the stress-strain curves. Testing was carried out at a speed of 1 mm/min. The stress-strain curves for pure AA6063-T4 and AA6063-T4 reinforced with 3, 5 and 7 wt% TiO2 nanoparticles are shown in Figure 6.

The mechanical properties of pure AA6063-T4 and its composites reinforced with different amount of TiO2 were determined from Figure 6 and are illustrated in Table 3.

The mechanical tensile properties listed in Table 3 of the composites were significantly enhanced. The UTS, YS and modulus increased with increasing amount of TiO2. The UTS and YS with 7 wt% TiO2 were improved by around 9 and 18%, respectively. The increase in UTS and YS is attributed to the presence of hard particles of TiO2 in the matrix and to the low degree of porosity and uniform distribution of nano particles. It is believed that the introduction of TiO2 nanoparticles into the aluminum matrix provides some heterogeneous nucleation sites during solicitation resulting in refined grains [16,17]. Figure 7 shows SEM image of base metal and 7 wt% TiO2 composites which reveals a uniform distribution of TiO2 nanomaterials and less porosity along the grain boundaries. The enhancement in mechanical properties of the composites, in comparison to the base metal, can also be ascribed to the strong multiaxial thermal stresses at the

![Figure 5. Tensile test sample with dimensions.](image)

![Figure 6. Stress-strain curves for AA6063-T4 and its composites reinforced with different amounts of TiO2.](image)

| Material                  | UTS (MPa) | YS (MPa) | E (GPa) | Strain at 
|---------------------------|-----------|----------|---------|------------|
| AA6063-T4                 | 177       | 99       | 70      | 21         |
| AA6063-T4 + 3 wt% TiO2   | 181       | 105      | 70.3    | 20.5       |
| AA6063-T4 + 5 wt% TiO2   | 186       | 111      | 70.8    | 19.2       |
| AA6063-T4 + 7 wt% TiO2   | 194       | 121      | 71      | 18.4       |

Table 3. Mechanical properties of AA6063-T4 and its composites.
AA6063-T4/TiO2 interface [18]. Moreover, it has been reported by other investigators that a low degree of porosity leads to an effective load transfer between uniformly distributed, strong TiO2 particulates.

**Electrical properties**

The materials used were AA6063-T4 as a matrix and three different composites containing 3, 5 and 7 wt% TiO2. The electrical properties of the aluminum alloy and its composites were investigated as a function of frequency at room temperature (RT). It should be noted that DC conductivity is different from AC conductivity as DC displays a constant frequency of the electrical field while the frequency of the electrical field varies when using AC conductivity. The main parameter affecting AC conductivity, the frequency of the electric field \( w \), has been studied. The empirical formula for the frequency dependence AC conductivity is given below [19].

\[
\sigma_{AC}(w) = A w^S \tag{5}
\]

where, \( A \) and \( S \) are material constants or power law equation constants which can be obtained by curve fitting the experimental data of the conductivity test. Application of Equation (1) to the experimental results for AC conductivity \( \sigma_{AC} \) with frequency \( w \) gives four relationships for the AA6063-T4 matrix and its nanocomposites with the material constants as given in Table 4.

The values of conductivity at different frequency are listed in Table 5 for the four different materials.

The above results are compared with the experimental conductivity of the four materials. It is clear from Table 5 that the AC conductivity varied between 0.0624 Sm\(^{-1}\) to 0.013 Sm\(^{-1}\) for aluminum matrix at \( 10^5 \) and \( 5 \times 10^7 \) respectively while it changed between 0.2884 Sm\(^{-1}\) to 0.3887 Sm\(^{-1}\) at \( 10^5 \) and \( 5 \times 10^7 \), respectively for 7 wt% TiO2. The high AC conductivity was improved in an almost linear fashion with increasing frequency. The high value of AC conductivity of the nanocomposites may come from the induced polarization, i.e. the interface induced polarization and TiO2 filler attributing to improved AC conductivity and dielectric properties [12]. Electrical properties of pure aluminum and the three composites have been measured in laboratory circumstances \((27^\circ \text{C and relative humidity 43\%})\). It was found that the maximum conductivity was observed for \((1.5\%\text{Fe}_2\text{O}_3 + 2\%\text{Al}_2\text{O}_3)\) from 1170 to 69521 \( \Omega \text{m}^{-1} \) [3]. Saravanan et al. [20] investigated the electrical properties of AA6063-T4 composites with micronsize titanium carbide (TiC) particles at different loadings of 0, 3, 6, 9 and 12 wt% TiC produced by stir casting. With the addition of TiC particles to the AA6063-T4, the electrical properties are initially increased and then decreased, due to the homogeneous dispersion of TiC particles in metal matrix. Alalkawi et al. [21] fabricated hybrid nanocomposites by adding various amount of iron oxide (Fe2O3) with constant weight percentage of aluminum oxide (Al2O3). They concluded that composite containing 1.5% Fe2O3 + 2% Al2O3 showed improved electrical and magnetic properties. Jianlei et al. [22] electrically tested Al/SiC nanocomposites with various amounts of SiC and they found that increasing the weight percentage of SiC leads to improved AC electrical conductivity and a significant increase dielectric loss. Athraa

| Material               | Frequency (Hz) | 10\(^5\) | 10\(^6\) | 10\(^7\) | 5 \times 10\(^7\) |
|------------------------|----------------|----------|----------|----------|-------------------|
| AA6063-T4              | 0.0624         | 0.0351   | 0.0197   | 0.013    |
| AA6063-T4 + 3 wt%TiO2  | 0.1028         | 0.1104   | 0.1186   | 0.1247   |
| AA6063-T4 + 5 wt%TiO2  | 0.2852         | 0.3128   | 0.3429   | 0.3657   |
| AA6063-T4 + 7 wt%TiO2  | 0.2884         | 0.3221   | 0.3598   | 0.3887   |

![Figure 7. SEM image for (a) base metal and (b) 7 wt% TiO2 nanocomposite.](image)

![Table 4. Frequency dependence AC conductivity equations with correlation coefficient R\(^2\) for different materials.](image)

| Material               | \( A \)  | \( S \)  | \( \sigma_{AC}(w) = A w^S \) | \( R^2 \) |
|------------------------|---------|---------|-------------------------------|---------|
| AA6063-T4              | 1.110   | 0.25    | \( 1.11 w^{-0.25} \)         | 0.962   |
| AA6063-T4 + 3 wt%TiO2  | 0.072   | 0.031   | \( 0.072 w^{0.031} \)        | 0.975   |
| AA6063-T4 + 5 wt%TiO2  | 0.180   | 0.040   | \( 0.18 w^{0.04} \)          | 0.990   |
| AA6063-T4 + 7 wt%TiO2  | 0.166   | 0.048   | \( 0.166 w^{0.048} \)        | 0.982   |
et al. [23] studied the influence of 6 wt% Al₂O₃ on electrical properties. They reported that the 6 wt% Al₂O₃ shifted the conductivity and permittivity from reducing in neat matrix (AA7100) to increasing in the composites when the frequency increased.

Electrical and mechanical properties were examined for the matrix and the hybrid composites (Al + SiC + Al₂O₃). It was found that the addition of hybrid nano reinforced materials leads to an increase in electrical conductivity of the composite [24]. Many researchers have found that the AC conductivity increases with increasing frequency, and the frequency exponent (S) in Equation (1) decreases as frequency increases for the nanocomposites [3, 4, 10]. Saravana et al. [20] have reported that the electrical resistivity of AA6063/TiC composites increased with increasing TiC content while the electrical conductivity (σₐₐ) decreases with increasing TiC.

### Magnetic Properties

Table 6 lists the magnetic property (magnetization range) of the base metal and the nanocomposites for different amounts of TiO₂.

| Material                  | Magnetization range (M) |
|---------------------------|-------------------------|
| AA6063-T₄                 | 0.166 ± 0.000          |
| AA6063-T₄ + 3 wt%TiO₂    | 0.162 ± 0.000          |
| AA6063-T₄ + 5 wt%TiO₂    | 0.166 ± 0.000          |
| AA6063-T₄ + 7 wt%TiO₂    | 0.174 ± 0.000          |

Figure 8. Magnetization against magnetic field for AA6063-T₄ and nanocomposites.

Table 7. Fatigue test results for base metal and 7 wt%TiO₂ nanocomposite.

| Material                  | Applied stress (MPa) | Nₖ cycles |
|---------------------------|----------------------|------------|
| AA6063-T₄                 | 138                  | 7000, 7800, 6000 |
| AA6063-T₄ + 3 wt%TiO₂    | 103                  | 41800, 40600, 42600 |
| AA6063-T₄ + 5 wt%TiO₂    | 69                   | 45000, 448000, 460000 |
| 7 wt% TiO₂ nanocomposite | 138                  | 9000, 6900, 10200 |
| AA6063-T₄ + 7 wt%TiO₂    | 103                  | 50900, 48200, 51000, 486000, 505000, 472000 |
| AA6063-T₄ + 7 wt%TiO₂    | 69                   | 460000, 505000, 472000 |

Figure 9. Comparison of S-N curves for base metal and 7 wt%TiO₂ nanocomposite.
Fatigue properties

The obtained fatigue results are presented in Table 7. Fatigue data were directly taken from the fatigue test rig for pure AA6063-T4 (0 wt%) and a nanocomposite containing 7 wt% TiO2 (i.e. the most improved composite). The fatigue strength of the different composites are subjected to similar loading. It is convenient to express the fatigue strength in terms of the stresses corresponding to a particular life time on the mean S-N curve using Basquin’s formula.

$$\sigma_f = AN_f^{\alpha}$$  \hspace{1cm} (6)

where $A$ and $\alpha$ are material constant.

The experimental data points of Table 7 are curve fitted and this curve is described by the following S-N relationship:

$$\sigma_f = 600N_f^{-0.166}$$ for AA6063-T4 with correlation coefficient ($R^2$) of 0.98,

$$\sigma_f = 700N_f^{-0.166}$$ for 7 wt% TiO2 nanocomposite with correlation coefficient ($R^2$) of 0.975.

The above equation can be plotted in Figure 9 for both matrix and nanocomposite. Traditionally, $10^7$ cycles have been used and indeed some design code refer to S-N curve in terms of corresponding stresses [25]. The addition of TiO2 nanoparticulates produced an initial increase in fatigue strength of about 13% above that of the metal matrix. Divagar et al. [4] found an increase in fatigue strength of 4.6% for hybrid nanocomposites containing 5% SiC + Al2O3 and 9.8% increase in fatigue strength from its base metal but further improvement for hybrid nanocomposites 15% SiC + 5% Al2O3 while Figure 9 shows the effect of the addition of 7 wt% TiO2 to the AA6063-T4 on the S-N curve.

It clearly shows that, the fatigue behavior (life time and strength) are improved with the addition of 7 wt% TiO2 compared to the fatigue behavior of the base aluminum matrix. The enhancement in fatigue behavior is presumably due to presence of the hard TiO2 particles, which impart strength to the soft aluminum matrix leading to greater strength of the composite. This may be occur as the dispersion of hard nanoparticles in the base metal leads to restricted plastic flow, thereby providing improved strength to the composite. A notable rise in fatigue life and fatigue strength of the base alloy can be seen with the addition of the TiO2 nanomaterial. This is again mainly due to the presence of the hard nano fillers restricting plastic flow of the AA6063-T4 matrix as well as due the strong interfacial bounding between the metal matrix and the nano reinforcements [26].

Conclusion

An aluminum based metal matrix composite was prepared by incorporating 3, 5 and 7 wt% of nanosize TiO2 particles into an aluminum AA6063-T4 matrix using a stir casting technique. The nanocomposite thus prepared exhibited good mechanical, electrical, magnetic and fatigue properties. It was revealed that ultimate tensile strength and yield strength of prepared composites improved for the composites. Strength of the samples increased with increasing the weight percentage of TiO2 particles, while ductility of composites reduced when the weight percentage of TiO2 increased. SEM images of 7 wt% TiO2 composites showed a fairly uniform distribution of TiO2 in the metal matrix. Electrical conductivity of AA6063-T4/TiO2 composites increased with increasing TiO2 content as expected. The highest conductivity was obtained for composites containing 7 wt% TiO2 and conductivity was proportional to the frequency for all composites and the pure matrix. Results indicated that the composite AA6063-T4/7 wt% TiO2 exhibited better magnetic properties while all composites have improved magnetic properties compared to the bulk matrix. Fatigue properties of the 7 wt% TiO2 were found to be increased by 13% compared to the base metal. It is believed that nanoparticles reinforcement, distribution of TiO2 and restricted grain sizes are responsible for the mechanical and electrical properties of the produced nanocomposites.

Disclosure statement

No potential conflict of interest was reported by the authors.

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