Evaluation the magnetic and microstructure properties of Al/TiO$_2$ nanocomposites using various stir casting temperature

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Abstract. The effect of three stir casting temperatures (ST) (900, 1000, and 1100°C) on magnetic and microstructure properties of AA5052 reinforced with 5wt% TiO$_2$ nanoparticles has been investigated using the stir casting technique. It was found that the magnetic properties decrease with increasing stir casting temperature and the least magnetic saturation (Ms) and magnetic susceptibility (Xm) occurred for nanocomposite fabricated under stir casting temperature (1000°C) also the maximum coercivity (Hc) and residual magnetization (Mr) occurred in this nanocomposite. The values of magnetic saturation (Ms), coercivity (Hc) and residual magnetization (Mr) obtained for nanocomposite under (1000°C) are $2.714 \times 10^{-6}$ (A/m), 4676.55530 (A/m) and $3.25032 \times 10^{-8}$ (A/m), respectively. Scanning electron microscopy (SEM) is used to analyze the microstructure, and it is discovered that well-distributed nanoparticles lead to decrease grain size in the parent phase.

Key words: AA5052, TiO$_2$ nanoparticles, (ST), magnetic, microstructure, stir casting route.

1. Introduction

To reinforce the matrix of aluminium matrix composites (AMCs), hard particles like carbides (Al$_4$C$_3$, SiC, TiC), oxides (Al$_2$O$_3$), or nitrides (TiN, AlN) are widely used [1]. Because of their high corrosion resistance, ease of processing, low weight, and low cost, aluminum and aluminum alloys are widely used as a matrix for metal matrix nanocomposites [2]. Fathy et al., [3], demonstrated the influence of (Fe) addition with (5, 10 and 15wt%) on the microstructure, mechanical and magnetic characteristics of Al-matrix using powder metallurgy (P/M) method. They discovered that the magnetization value for 5% Fe is $0.3816 \times 10^{-3}$ A/m, increases to $0.6597 \times 10^{-3}$ A/m for 10% Fe and decreases to $0.0702 \times 10^{-3}$ A/m for 15% Fe samples. The highest $\mu_{abs}$ was observed in the 1.5 % Fe$_2$O$_3$ + 2 % Al$_2$O$_3$ composite, which was $1.257 \times 10^{-6}$ H/m and reduced to $1.256 \times 10^{-6}$ H/m for (5 % Fe$_2$O$_3$ + 2 % Al$_2$O$_3$) nanocomposite, while this value was $1.254 \times 10^{-6}$ H/m for zero nano. In a vacuum furnace at 600 °C for 1 hour, Al–Fe mixtures were compacted and sintered. The matrix grain refinement and uniform distribution of Fe particles, as well as the formation of Al$_3$Fe$_4$ intermetallic, are all correlated.
with the strengthening process. Ferreira et al. [4], tested magnetic and electrical characteristics of Al matrix reinforced with Fe$_3$O$_4$ nanomaterial with particle size 45-70 nm using (P/M) method. The outcomes showed that the magnetic properties improved as the wt% of Fe$_3$O$_4$ increased and the highest improvement at 30%. Ferreira et al. [5], examined the effect of different weight percentage of Fe$_3$O$_4$ nanomaterial on magnetic, microstructural, electromagnetic and mechanical properties. It was discovered that the photograph obtained by SEM had a homogeneous distribution, while the agglomeration of particles increased as the wt% of reinforcement increased. Increasing the weight percent of Fe$_3$O$_4$ particles also improved the magnetic properties significantly. Hussain AMA, [6], studied electrical and magnetic properties of pure aluminum reinforced with (Fe$_3$O$_4$+ Al$_2$O$_3$) nanoparticles. For composites containing 1.5wt% Fe$_3$O$_4$+2 wt% Al$_2$O$_3$, As compared to a base metal matrix, the highest electrical conductivity was discovered, with a 42% improvement, the synthesized nanocomposites containing 1.5 wt% Fe$_3$O$_4$+2 wt% Al$_2$O$_3$ showed good electrical conductivity. Maximum magnetization (M) was observed for a composite containing 1.5 % Fe$_3$O$_4$ + 2 % Al$_2$O$_3$), which is equivalent to 0.11666 A/m and a 26 % increase in (M) compared to pure aluminum magnetization. Manal Hadi Jaber et al, [7], fabricated AA6063-T6 supported with TiO$_2$ by using stir casting technique with different weight percentage of TiO$_2$ nanoparticles 3, 5 and 7 wt%. The outcomes revealed the distribution of TiO$_2$ in the metal matrix was fairly uniform in SEM images of 7 wt% TiO$_2$ composites. In contrast to the base metal, the magnetic tests showed that the nanocomposites performed better. The composite AA6063-T4/7 wt% TiO$_2$ showed better magnetic properties than the bulk matrix, whereas all composites had better magnetic properties than the bulk matrix. Negin Ashrafi et al, [8], investigated the effect of dual nano size particles (SiC–Fe$_3$O$_4$) on microstructural, thermal, electrical, and magnetic properties of aluminum matrix using the powder metallurgy process. The magnetic analysis revealed that adding SiC to Al-15Fe$_3$O$_4$ in the range of 10 to 30 wt.% increased Ms from 2 to 6 (emu/g) while decreasing Hc from 238 to 171 G. Ms increased from 5 to 11.058 (emu/g) and Hc reduced to 131 after the addition of 30 wt.% ferrimagnetic Fe$_3$O$_4$ and (10 to 20) wt.% SiC nanoparticles to aluminum. Al-15Fe$_3$O$_4$ and Al-30Fe$_3$O$_4$-30SiC have magnetic saturation (Ms) of 6.01 and 11.058 (emu/g), respectively, which is an improvement of 83.99 %. IsmayadiIsmail et al, [9], studied the effect of stir casting temperature and soaking time on mechanically alloyed ferrites' crystallinity degree and magnetic properties of ferrite with the composition Ni$_{0.5}$Zn$_{0.5}$Fe$_3$O$_4$. it used three parameters two parameters for stir casting temperature and the third parameter the soaking time, which varied from 1 to 96 hours with a constant stir casting temperature of 800°C. The findings revealed that as the stir casting temperature rises, the XRD intensity rises, indicating higher crystallinity and possibly higher magnetization values. The aim of this work is to investigate the influence of various stir casting temperature (900, 1000 and 1100 °C) on magnetic and microstructural properties of AA5052 reinforced with constant weight percentage of TiO$_2$ (30nm).

2. Experimental Work
This section discusses the materials used, their chemical composition with simple used devices, as well as the processing of nanocomposite materials. The aluminum alloys are the major materials in many industries such as ships, pipes, structures, airplanes and other applications [10].

2.1 Utilized materials
In this study, the aluminum metal matrix AA5052 is used. AA 5052 is an Al-Mg alloy that is mainly used in the automobile, aerospace, and marine industries due to its high strength [11-13]. AA5052 has been enhanced with TiO$_2$ and particle size (30nm) [14]. Table (1) shows the chemical composition of AA5052 alloy.
Table 1. Chemical composition of AA5052 alloy.

| Alloy      | Mg   | Cr   | Cu  | Fe  | Mn  | Si  | Zn  | Other elements |
|------------|------|------|-----|-----|-----|-----|-----|----------------|
| Standard   | 2.2  | 0.15 | 0.1 | 0.4 | 0.1 | 0.25| 0.1 | 0.05           |
|            | 2.8  | 0.35 |     |     |     |     |     | 0.15           |
| Balanced   |      |      |     |     |     |     |     | Balance        |
| Experimental| 2.6 | 0.29 | 0.09| 0.33| 0.08| 0.24| 0.1 |                |

2.2 Fabrication of Samples

Stir casting was used to render the AA5052/TiO2. The method can be carried out as follows:

1- The required sample weight is initially calculated using the volume x density law.

\[ m = V \times \rho \rightarrow m = 0.6 \times 12 \times 5 \times 3 \rightarrow m = 108 \text{gm} \]

2- Determine the proportions of the additional elements (magnesium and chromium).

Mg weight = 108 \times 0.0317 \rightarrow \text{Mg weight} = 3.4236g

Cr weight = 108 \times 0.0043 \rightarrow \text{Cr weight} = 0.4644g

Al weight = 3.4236 - 0.4644 \rightarrow \text{Al weight} = 2.9592g

3- Then using a gas furnace, fuse the aluminum wires for 5 minutes at 750°C. A sand mold is created based on the dimensions of the sample during the aluminum smelting process.

4- Chromium is added to the melt, which is then wrapped in aluminum foil and immersed in the molten metal. Allow three minutes for cooling before mixing via an electric mixer at 600 rpm for one minute.

5- After the mixing is over, add the magnesium and cover it with aluminum foils, then remove the air, add it to the inside of the molten, holding it under the slag in the melt as well, and mix until the magnesium and chromium are perfectly distributed. After that, 1 gram of aqueous aluminum chloride (AlCl3) is added to extract slag and release gases, and the first casting is poured without adding nano titanium oxide after it has frozen and it is removed from the mold.

6- The same measures are used for the second, third, and fourth castings, but nano titanium oxide is added after coating it with aluminum foil to expel gases before adding the magnesium. It's mixed for five minutes with an electric mixer, then magnesium is added in the same way as the previous one, mixed well, and aluminum chloride is added as a slag-removal aid for ten minutes before the sample freezes, then it's removed from the sample. The mixture is then poured into the mold and temperature regulated, with the second casting implemented at 900°C, the third casting at 1000°C, and the fourth casting at 1100°C.

7- The variation of stirring temperature from 900°C to 1100°C leads to change the viscosity of liquid which is decreased with increasing temperature.

8- The distribution of reinforcement particles in the metal matrix creates interface bond between metal matrix and nanoparticles.

9- Air is withdrawn from the oven by vacuum and Alarcon gas is pumped into the oven and heat the oven to the required temperature. The addition of nano materials to the molten aluminum alloy with gas pump.

2.3 Rule of mixture

Table (2) demonstrates the rule of mixture using AA5052 - alloy as a metal matrix.
2.4 Magnetic test
To calculate the magnetic properties of powders and composites at a constant frequency, this test uses a vibrating sample magnetometer (VSM) system model (Magnates Doglight Kavir Co., Iran). This test is carried out at room temperature to determine the magnetization of a material in the presence of a magnetic field. Since the magnetic field differed over a limited range, the magnetization of the powders is determined by sense coils with a lock in amplifier. This examination takes place at Tehran University. The VSM device's output is the variation of magnetic field (H) versus magnetization (M) range. It can also be used to acquire $\mu_{abs}$ by measuring $X_m$. Figure (1) shows the (VSM).

### Table 2. Rule of mixture of AA5052/TiO$_2$ nanocomposite.

| TiO$_2$ (wt%) | TiO$_2$ (gm) | AA5052 alloy (gm) | Total nanocomposite (gm) | (ST) °C |
|---------------|--------------|-------------------|--------------------------|---------|
| 0             | 0            | 500               | 500                      | 900     |
| 5             | 25           | 475               | 500                      | 900     |
| 5             | 25           | 475               | 500                      | 1000    |
| 5             | 25           | 475               | 500                      | 1100    |

![Figure 1. Vibrating sample magnetometer device.](image)
2.5 Microstructure examination
The preparation of specimens for scanning electron microscopy test requires four stages. These steps are as follows:

1. Selection and cutting of specimens.
2. The grinding and polishing method.
3. Cleaning and drying.
4. Etching.

Grinding through (120, 400, 600, 800, and 1200) grit papers was used to prepare samples, which were then polished with 1µm diamond paste and etched with (HF, HCL). Scanning electron microscopy is used to examine the matrix and composites on a microscopic level (SEM TESCAN MIRA3). The grain size of the material was determined by image analysis. The microstructure of the specimens was examined using a scanning electron microscope (SEM TESCAN MIRA3) at Tehran University in Iran. The device used for this test is shown in Figure (2).

Figure 2. The scanning electron microscopy (TESCAN MIRA3) devices.

3. Results and Discussion

3.1 Magnetic results
Table (3) shows the magnetic parameters of the base metal and nanocomposites (saturation magnetisation (Ms), remanence magnetisation (Mr) and coercive field (Hc) for various stir casting temperatures. The findings in Table (3) show that increasing stir casting temperature causes a reduction trend in magnetization, magnetic field and other parameters. The maximum reduction in saturation magnetization and magnetic susceptibility (Xm) occurring for the composite containing 5wt% TiO$_2$ obtained at 1000°C stir casting temperatures. The maximum coercivity (Hc) and residual magnetization (Mr) were obtained for nanocomposite under stir casting temperature (1000°C). The Ms, Mr and Hc are reduced together may be due to decrease in the particle size which will effected strongly on the magneto crystalline. The magneto crystalline of the material can be effected by size, shape, temperature, method and porosity. Also, the nanoparticles have high reactivity with thermal treatment per unit value which arise from magnostatic or exchange interaction between particles. Coercivity (Hc) can be influenced by dislocations, grain borders, precipitates, pores, impurities, and the distribution of non-magnetic particles, which is an important parameter in soft magnetic behavior. Nonmagnetic inclusions and defects, such as airgaps and grain boundaries, have a negative impact on magnetic properties. The magnetic properties such as magnetic saturation (Ms), coercivity (Hc) and
residual magnetization (Mr) can be determined by using hysteresis loop while magnetic susceptibility (Xm) can be determined using the following equation:

\[ X_m = \frac{M}{H} \]  

(1)

The magnetic behavior of AA5052 and its nanocomposites is shown in Figure (3). Fabio Luis Zabotto et al, [16], studied the effect of various stir casting temperature (1000, 1100, 1150 and 1200 °C) on the magnetic properties of nickel ferrite samples made by solid state reaction. As a result of the increased domain size caused by grain growth, the coercive field is reduced at higher stir casting temperatures. Samples sintered at 1200 °C showed a coercive field approaching superparamagnetic values while retaining high saturation magnetization values, indicating the possibility of a mixed spinel structure. M A Ali et al, [17], investigated the influence of stir casting temperature on the magnetic properties of Ni0.6Zn0.4Fe2O4 (NZFO) ferrites synthesized using the traditional double sintering method. Temperatures of 1200, 1250, and 1300 °C are used to sinter the samples. Magnetic parameters such as saturation magnetization (Ms), coercive field (Hc), remanent magnetization (Mr), and the Bohr magneton (B) are measured and compared to previous studies’ values. Ms and Hc were found to be 71.94 emu/gm and 1.2 Oe, respectively, at Ts = 1300°C.

**Figure 3.** Magnetization (magnetic moment) against magnetic field for AA5052 and nanocomposites.
Table 3. Magnetic properties of nanocomposites.

| Condition                        | Ms (A/m)  | Mr (A/m)       | Hc (A/m)  | Xm          |
|---------------------------------|-----------|----------------|-----------|-------------|
| AA5052 metal matrix             | 2.89×10^{-6} | 2.28116×10^{-8} | 4478.49342 | 6.45306×10^{-10} |
| Nanocomposite 5wt% TiO₂ (900ST) | 4.962×10^{-6} | 1.26592×10^{-8} | 1502.28657 | 3.30296×10^{-9} |
| Nanocomposite 5wt% TiO₂ (1000ST)| 2.714×10^{-6} | 3.25032×10^{-8} | 4676.55530 | 5.80341×10^{-10} |
| Nanocomposite 5wt% TiO₂ (1100ST)| 4.208×10^{-6} | 7.88236×10^{-9} | 1142.16160 | 3.68424×10^{-9} |

3.2 Scanning Electron Microscopy (SEM) results

SEM photographs of (a) zero-Nano (at 900°C), (b) 5wt% TiO₂ (at 900°C), (c) 5wt% TiO₂ (at 1000°C), and (d) 5wt% TiO₂ (at 1100°C) are shown in Figure (4). It can be noted from Figure (4) that the shape of particles is almost spherical and the particle size decreases with increasing stir casting temperature. Also, it has been noted that all the nanocomposite reveal uniform distribution, high agglomeration, and few clusters at inter-dendrite regions. This agglomeration may be due to the nano scale show high surface area which leads to high interfacial tension. It was also noted that with increasing temperature the amount of porosity decreases substantially and the particles move closer. From Figure (4) it has been noted that there is no cracks. However, the 5wt% TiO₂ (at 1000°C) has the best uniform distribution. Furthermore, the addition of TiO₂ to the base metal melt increases the viscosity of the melt, which slows TiO₂ movement. Controlling the solidification rate is critical for achieving a high distribution of TiO₂ during the stir casting process. Strong interfacial bonding has evolved at the interfaces between ceramic TiO₂ nanoparticles and aluminum matrix due to the thermal reaction and diffusion mechanism used in the sintering process. This means that the increase in stir casting temperature decreases grain size and this leads to improve mechanical and fatigue properties. These findings coincide with Zahraa et al., [18], studied the effect of different stir casting temperature on fatigue and mechanical properties of AA5052 reinforced with 5wt% of TiO₂ nanoparticles. The results observed that grain refinement, uniform distribution of TiO₂ particles into metal matrix and the minimization of grain size defects contribute to an improvement in the mechanical properties and strength of fatigue.
Figure 4. SEM photographs of AA5052/TiO$_2$ nanocomposite at (a) as received at 900°C (ST), (b) 5wt% TiO$_2$ at 900°C (ST), (c) 5wt% TiO$_2$ at 1000°C (ST) and (d) 5wt% TiO$_2$ at 1100°C (ST).

The addition of TiO$_2$ nanoparticles could be attributed to the fact that TiO$_2$ particles act as obstacles to the movement of dislocation. Kok, [19], reported that the introducing TiO$_2$ into the aluminum matrix resulted in raising the distribution in uniform manner. This happen when applying 1000°C (ST) while the other temperature showed that the distribution is not completely uniform.

4. Conclusion
The current study used a stir casting technique to produce and evaluate four AA5052 nanocomposites reinforced with a constant weight percentage (5 wt%) of TiO$_2$ with particle size (30nm) and different stir casting temperatures (900, 1000 and 1100°C). Experimental study has attempted into the impact of stir casting temperature on the magnetic and microstructural properties of AA5052 matrix and nanocomposites. The maximum coercivity (Hc) and residual magnetization (Mr) for nanocomposite are obtained at 1000°C stir casting temperature. Also, the maximum reduction in saturation magnetization (Ms) and magnetic susceptibility ($X_m$) is observed for this nanocomposite as well. The residual magnetization (Mr) and coercivity (Hc) improved by 29.8% and 4.23% when increasing stir casting temperature to (1000°C) compared to as-received metal. SEM analysis revealed that the TiO$_2$ nanoparticles were incorporated into the metal matrix. SEM images of composites with 5 wt% TiO$_2$ and a stir casting temperature of 1000°C revealed a relatively uniform distribution of TiO$_2$ in the metal matrix.

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