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Effect of Aging Treatment and TiO_2 Nano Particles Addition on the Microstructure and Mechanical Properties of 2024 Aluminum Alloy

Hamid M. Mahan[1,2]*, Konovalov Sergey[2,3], Nada M. Al-Nedawi[4], Ahmed H. Ali[5]

[1] *Department of Mechanical/ Baqubah Technical Institute*
Middle Technical University, Baghdad 10074, IRAQ
[2] *Department of Metals Technology and Aviation Materials*
Samara National Research University, Samara 443086, RUSSIA
[3] *Department of Natural Sciences*
Siberian State Industrial University, Novokuznetsk, 654007, RUSSIA
[4] *Department of Civil, College of Engineering*
University of Diyala, Diyala, 32002- IRAQ
[5] *Department Materials Engineering*
University of Technology, 10074 – IRAQ

*Corresponding Author: hamid.m.mahan@mtu.edu.iq
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Abstract

In this research, aluminum alloy 2024 was enhanced with varying mass percentages (0 wt. %, 2.5 wt. %, 5 wt. %, and 7.5 wt. %) of titanium dioxide nanoparticles through the stir casting technique. Subsequently, the material underwent a solution annealing process at 500 °C for a duration of 3 hours, followed by rapid cooling in water and aging at 175 °C for another 3 hours. The primary aim of this investigation was to examine the impact of incorporating TiO_2 nanoparticles on both the microstructure and the mechanical properties of the 2024 aluminum alloy composite produced through the stir casting method. Scanning electron microscopy, energy-dispersive analysis, as well as X-ray diffraction analysis were implemented to characterize the microstructure, elemental and phase composition of the samples. The tensile and Vickers hardness tests were carried out to evaluate the mechanical properties. The results showed that the addition of 7.5 wt.% TiO_2 nanoparticles increases the ultimate tensile strength by 37 % and elongation by 71 % while decreases the hardness by 14 % comparing with the initial alloy. The highest hardness was demonstrated in the alloy with 5 wt. % TiO_2.

1. Introduction

Developing engineering materials that are simultaneously strong, lightweight, and cost-effective presents a significant challenge, particularly in achieving a favorable strength-to-weight ratio suitable for vehicle applications [1]. The global demand for such materials, especially in the automotive and aerospace sectors, has sparked the interest of researchers specializing in composite materials [2], [3]. Aluminum matrix composites (AMCs) have emerged as advanced materials that offer outstanding mechanical properties, blending the attributes of a lightweight and resilient matrix material with the addition of robust ceramic reinforcement [4]. AMCs consistently address the market's requirements for components that are both lightweight, durable, and capable of delivering high performance [5], [6]. Traditionally, the most common ceramic reinforcements used in

AMCs in the last few decades are carbides (SiC, TiC), oxides (Al_2O_3, ZrO_2, TiO_2), and nitrides (TiN, AlN) "e.g., [7], [8]. Divagar et al. [9] showed that the addition of 10 wt. % SiC and 5 wt. % Al_2O_3 nanoparticles in 7075-T651 aluminum alloy increases the fatigue strength by 13 % compared with the base metal. The presence of ZrO2 nanoparticles in aluminum matrix significantly improves the wear resistance and hardness of A356 aluminum alloy [10]. Jaber et al. demonstrated that the 6063-T6 aluminum alloy reinforced by 7 wt.% TiO_2 nanoparticles has higher by 13% fatigue strength than the base metal [11]. An addition of 2 wt. % TiN nanoparticles in Al2024 followed by aging increases the tensile strength by 38 %, while the yield strength and elongation decrease from 376.5 MPa and 12.7 % to 359.1 MPa and 9.4 %, respectively [12].

Aluminum alloy 2024 (AA2024) contains Cu, Mg, Mn, and some other minor alloying elements and has excellent tensile to yield strength ratio at elevated temperatures, high ductility, fatigue, and fracture resistance [13]. These properties as well as the capability to form second-phase precipitates for improved strength (age-hardening) determine high demand of AA2024 in aerospace and automobile industries. Shing J.H. et al. demonstrated that the addition of 10 wt. % TiO_2 nanoparticles with an average size of 50 nm in AA2024 matrix by mechanical milling and hot-pressing increases the Vickers hardness by 54 % and the strength by 13 % [14]. Although the properties of the composite are improved compared with the monolithic alloy, the drawback of this technology could be a low elongation value which might negatively influence on the reliability of the alloy when external stresses are applied. Creep resistance also enhances by incorporating of 3 vol % TiO_2 nanoparticles (15 nm in size) in Al matrix since the creep behavior depends dominantly on the diffusional flows of the matrix that are strictly limited by the nanoparticles [15]. After reviewing the available literature, it has become evident that there is a lack of extensive research concerning the effects of introducing titanium into Al-Cu-Mg alloys through the stir casting process. The AA2024 aluminum alloy is a member of the Al-Cu-Mg alloy family that relies on S (Al_2CuMg) and θ (Al_2Cu) precipitates as the primary sources of strengthening. The incorporation of titanium into this alloy category can expedite the formation of high-strength titanium aluminides. One limitation associated with Al-Cu-Mg alloys pertains to their susceptibility to thermal instability when exposed to elevated temperatures. However, by creating thermally stable titanium aluminides and ensuring their even distribution throughout the aluminum matrix, the thermal stability of these alloys can be improved [16], [17].

Since AA2024 has promising properties in different fields and as Al-Cu-Mg alloys can be hardened by aging by introducing the precipitations of second-phase particles in the matrix, as well as by an addition of hard particles during material production, in this study we aim to investigate the mechanical properties of AA2024 alloy fabricated by conventional stir casting followed by solution aging with the addition of different mass fractions of TiO2 nanoparticles.

Nomenclature is included if necessary	
SEM	Scanning electron microscopy
HV	Vickers hardness
XRD	X-ray

2. Material and Methods of Research

In this study, 2024 aluminum alloy with the composition of 92.8 wt.% Al, 1.04 wt. % Mg, 0.78 wt. % Mn, 5.33 wt. % Cu, 0.1 wt. % Zn and 0.2 wt. % Fe was selected as a matrix material. As a reinforcement material we used nanoparticles of TiO_2 with the purity of 99.8 % and the size of 30 ± 5 nm as shown in Fig. 1.

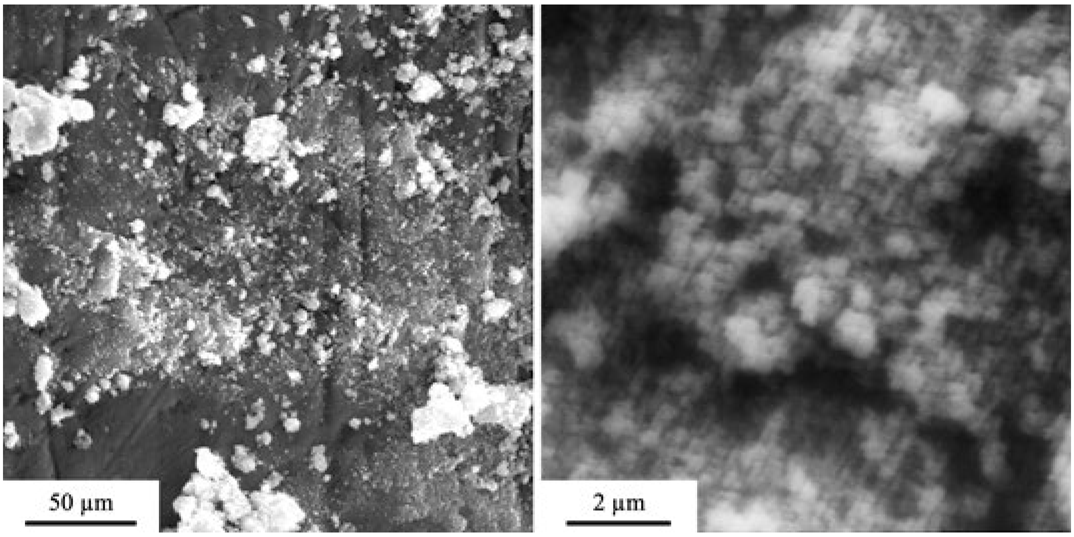

Fig. 1 *SEM micrographs of TiO2 nanoparticles*

Before adding TiO_2 nano powder with the different loading fracture of 2.5 wt. %, 5 wt. %, and 7.5 wt. %, 2024 aluminum alloy was preheated to 700 °C (more than the matrix melting temperature) in a graphite crucible using an electrical furnace to ensure the complete melting of all its components. Using argon as the carrier gas, it was introduced into the molten material as illustrated in Fig. 2. Subsequently, the resulting molten material was poured into a cylindrical mold measuring 22 mm in diameter and 200 mm in length. The stir casting technique was utilized for 4 minutes at 200 min^{-1} to ensure proper mixing and dispersion of the reinforcement material. The stirring action helps achieve a homogeneous distribution of the reinforcement, reducing the possibility of agglomeration or clustering. The specified time and rotation speed were determined based on previous studies or experimental optimization to achieve the desired level of dispersion and ensure the quality of the resulting composite material. Then the molten material was poured into molds and removed after solidification. The samples were then solution annealed by heating until 500 °C for 4 h in an air circulated furnace, water quenched at room temperature and precipitation annealed (aged) at 175 °C for 3 h [18].

Tensile tests were conducted at room temperature using an INSTRON 1125 universal testing machine in accordance with ASTM standard E8/E8M. The tensile specimen used was a flat shape, with a load of 5 KN applied at a deformation rate of 2 mm/min. The yield stress of the composites was determined using the offset method at 0.2 % strain from the stress vs. strain curve.

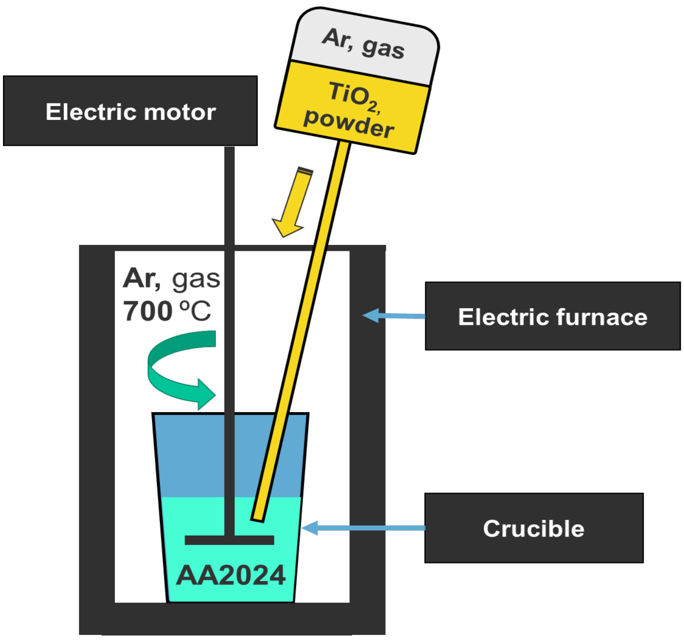

Fig. 2 *The Stir casting furnace for melting*

Fig. 3 illustrates the sequence of heat treatment procedures employed, which include quenching and aging. Initially, the solution of AA2024 aluminum alloy was subjected to heating in an electric furnace for a duration of 3 hours, reaching temperatures within the range of 500 °C to 510 °C. Subsequently, the sample was rapidly cooled by immersion in water to bring it to room temperature. Subsequent to this, the sample was positioned in an electric furnace, specifically the Gallen Hamp hot stop BR-17M / XD-17M furnace, for a period of 3 hours to attain the aging stage (precipitation heat treatment) within the temperature range of 180 °C to 190 °C, followed by cooling in ambient air. The schematic representation in Figure 3 serves to visually depict the sequential heat treatment processes [19]. For microstructural characterization the samples were prepared by the standard metallographic procedure and etched for 15 s using Kroll's reagent (H_2O: HNO_3: HF = 92:6:2). Scanning electron microscopy (SEM) (by TESCAN VEGA) and energy disperse spectroscopy (EDS) (by INCA Energy) analyses were carried out to reveal the microstructure and elemental composition distribution of materials. XRD analysis was performed to examine the structure of the phases and precipitates revealed by SEM. These results were obtained using DRON-7 instrument (Russia) with Cu Kα radiation operated at 40 kV, 30 mA and Bragg angles from 20° to 100° with a step size of 0.02° and a scan step time of 5 s. The Vickers hardness was determined on the polished surface using HV-1000 tester at a load of 0.025 N for a dwell time of 10 s. At least 10 indentations were made on each sample to obtain the average values.

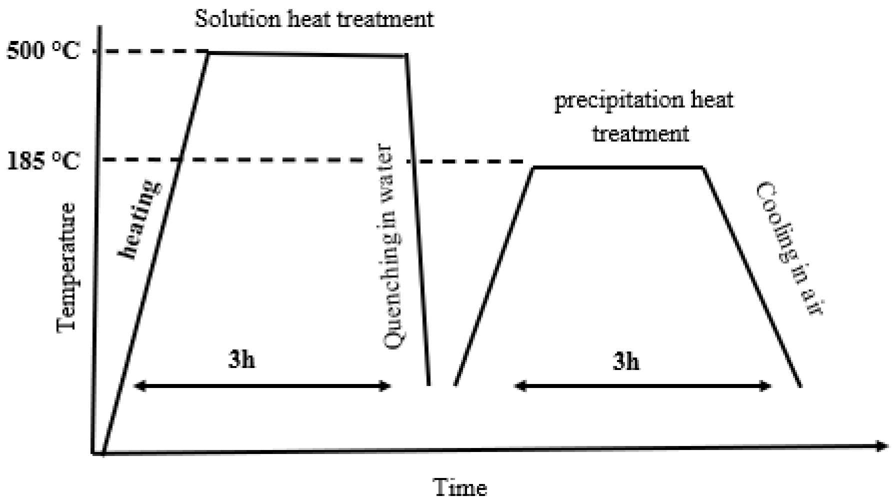

Fig. 3 *Heat treatment processes*

3. Results and Discussion

Fig. 4 demonstrates the results of tensile tests of the obtained samples with the various mass fractions of the reinforcing TiO_2 particles. In general, the samples behaved in a brittle manner, as can be deduced from the low values of the elongation at break (not more than 6 %). The initial sample shows the ultimate tensile strength (UTS) of 208 MPa and elongation before fracture of 3.5 %. An addition of 2.5 wt. % of TiO_2 particles increased the UTS by 43 % to 299 MPa and elongation by 18 % to 4 %. The further increasing of the content of TiO_2 up to 5 wt.% slightly decreased the UTS from 299 MPa to 273 MPa and almost not changed the elongation. In terms of balance between the strength and plasticity the alloy obtained by addition of 7.5 wt. % of TiO_2 showed the optimal properties comparing with the others due to the relatively high UTS of 286 MPa and elongation of 6 %. The yield stress of the fabricated composites reaches its maximum value of 240 MPa at the 2.5 wt. % TiO_2 which is higher than in the initial material by 20 % [20].

Crucially, following the heat treatment, there is a noteworthy enhancement in the tensile strength of TiO_2-AA2024 nanocomposites, as illustrated in Fig 4-b. In a statistical sense, the tensile strength attribute shows a substantial increase of 60 % following the heat treatment of the 5 wt. % TiO_2-AA2024 nanocomposite. Moreover, this represents an 50% enhancement in tensile strength when compared to the hardness of untreated AA2024. Fig. 5 illustrates the plotted values of Vickers hardness as a function of TiO_2 mass content. The addition of 2.5 wt. % and 5 wt. % of TiO_2 increases the hardness up to 13 % and 40 %, consequently, while at 7.5 wt. % TiO_2 the value decreases by 14 %. Such an increase can be attributed to the presence of TiO_2 particles in the aluminum matrix that hampers the movement of dislocations (solution hardening).

Comparing with the results of previous research the composites fabricated in this study have less UTS, YS and Vickers hardness but better elongation before fracture [21]. This might be attributed to the different routes of fabrication of the materials. The route in [22] consisted of mechanical milling followed by hot-pressing. The external stress during the fabrication led to the higher density of composite (maximum 2.87 g/cm^3 at 10 wt. % of TiO_2 nanoparticles) and, consequently, the higher hardness values. Stir casting followed by solution aging used in this study probably provided less dense composites but also more mobile dislocations that contribute to plastic deformation [18]. Therefore, the maximum elongation was quite higher (by 3 %). Crucially, the heat treatment has led to a substantial enhancement in the hardness characteristics of TiO_2-AA2024 nanocomposites, as illustrated in Fig. 4. Notably, the hardness property exhibits a remarkable 68 % increase following the heat treatment of the 5 wt. % TiO_2-AA2024 nanocomposite. Moreover, this represents an 80 % improvement in hardness when comparing the results to the hardness of AA2024 prior to the heat treatment.

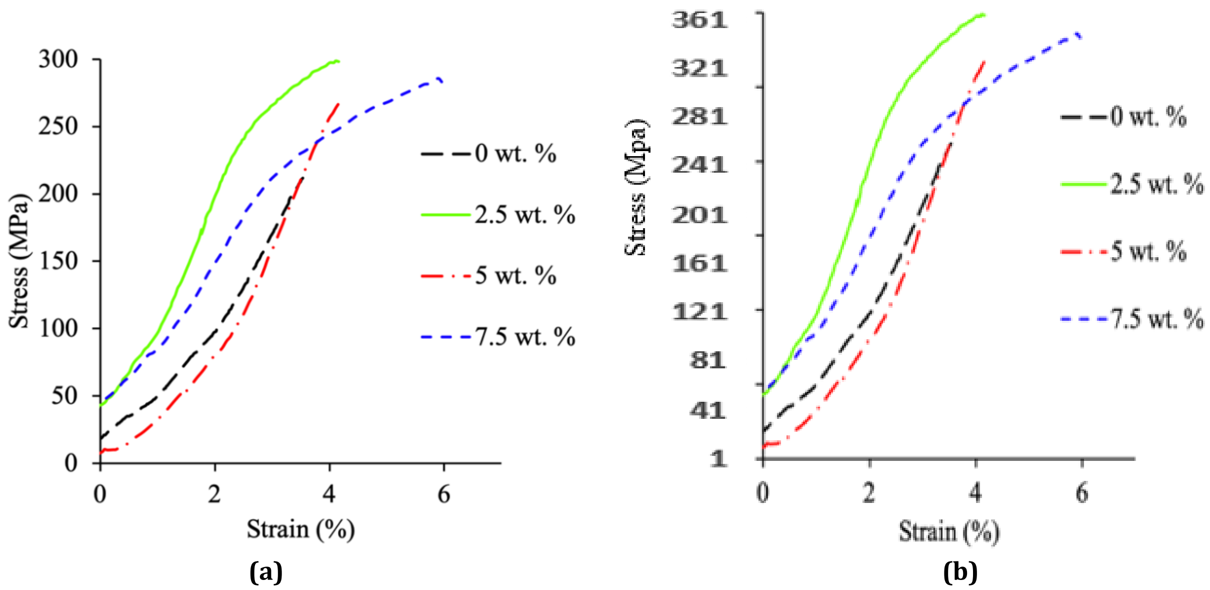

Fig. 4 *(a) Stress-strain curves before heat treatment and; (b) Stress-strain curves after heat treatment of 2024 aluminum alloy with the different mass fractions of TiO2*

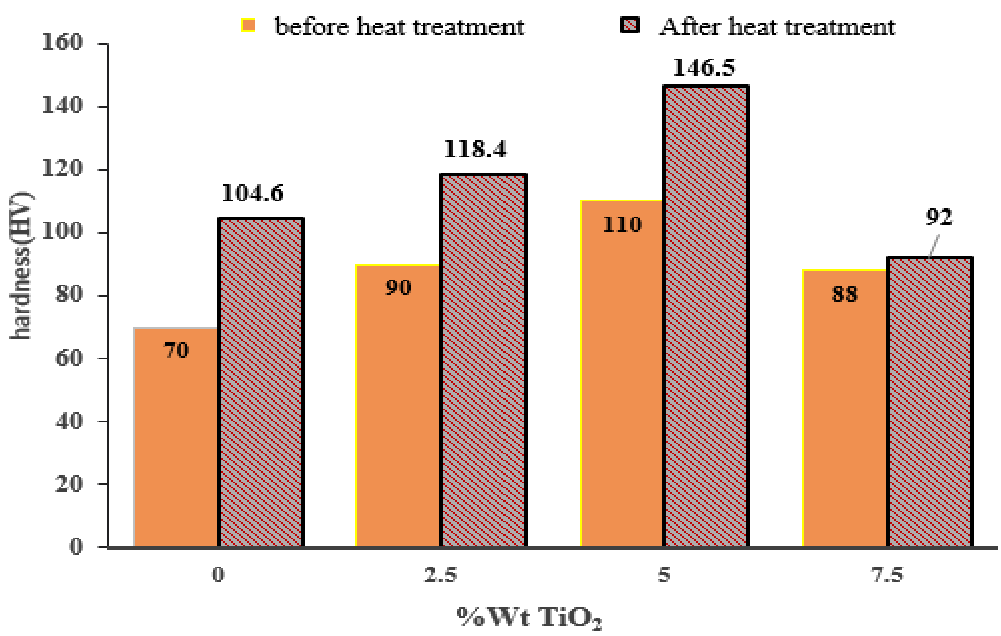

Fig. 5 *Effects of nanoparticles on the hardness of AA2024 composite*

Fig. 6 shows the microstructure of the fabricated samples after casting and heat treatment. The secondary phases composed of the alloying elements are distributed alongside the grain boundaries and as separate particles inside the boundaries. The area fractions of the intermetal ides (white regions) obtained using image analysis software ImageJ revealed the following results: initial sample has 4.4 % of intermetal ides, at 2.5 wt. % TiO_2 – 7.3 %, at 5 wt. % TiO_2 – 3.5%, and at 7.5 wt.% TiO_2 – 5 %.

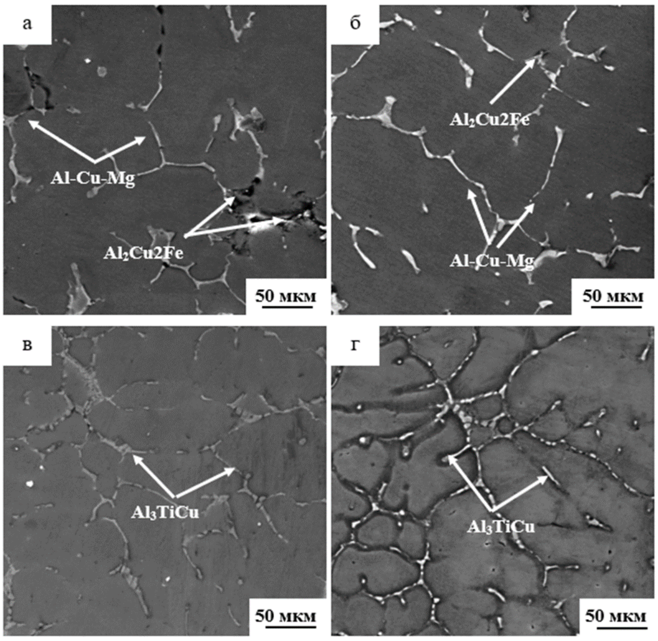

Fig. 6 *SEM micrographs of 2024 aluminum alloy with the different mass fractions of TiO2 before heat treatment*

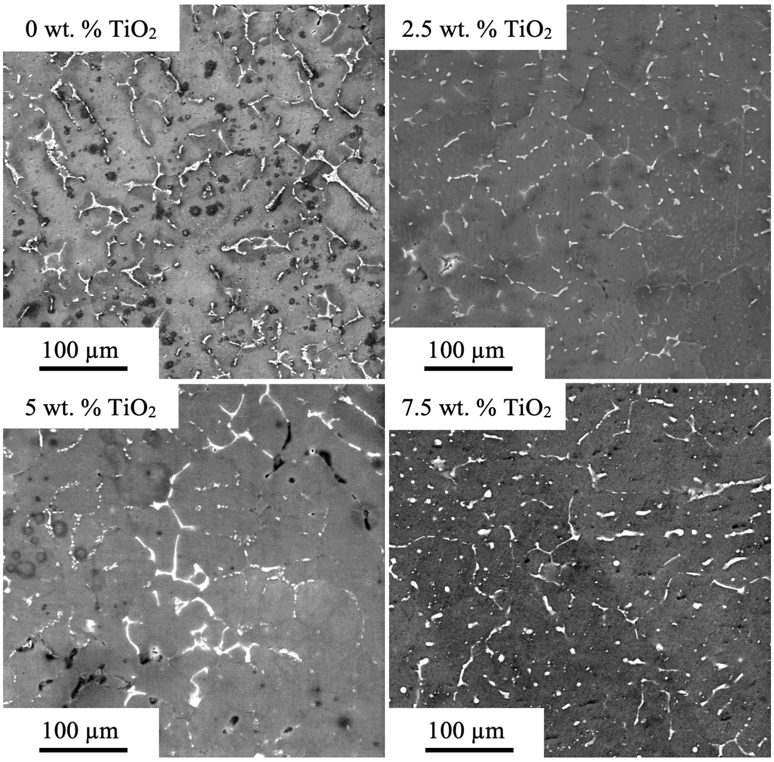

Fig. 7 *SEM micrographs of 2024 aluminum alloy with the different mass fractions of TiO2 after heat treatment*

Solution annealing and aging of 2024 aluminum alloy results in the formation of $CuAl_2Mg$ and $CuAl_2$ precipitates (Fig. 7, 8). The addition of TiO_2 particles may increase the number of precipitates after homogenization and aging treatment [23]. However, Fig.4 shows that Ti particles are distributed uniformly in the microstructure of the 2024 alloy without formation of precipitates even alongside the grain boundaries where the accumulation of particles and precipitates usually occurs. This might be related to the high solubility of TiO_2 nanoparticles in the structure of the matrix. According to the X-ray diffraction patterns for 2024 aluminum composite presented in Fig. 8, the intensities of the peaks gradually rise as the amount of TiO_2 increases due to the broadening effect [24]. Al peaks are observed at 38.50°, 44.64°, 65.14°, 78.18° and 82.5°. The major peaks of precipitations of second-phases are related to Al_2Cu, $MgCuAl_2$. The peaks related to TiO_2 nanoparticles have low intensities even with the increasing of the mass fraction. In the literature, there are reports on reactions between Al and TiO_2 which yields Al_3Ti and Al_2O_3 as final resultants [19], [25]. In this study, however, no obvious reacted product is apparent, which is in good agreement with the results SEM-EDS analysis. The diffraction patterns before heat treatment did not reveal significant changes with increasing TiO_2 content. From the standard data for TiO_2 it is clear that the peak with a diffraction angle of 25.2806° has the highest intensity, and the second most intense peak is 48.0487°. However, in the diffraction patterns these peaks are practically indistinguishable from the background. This may indicate that TiO_2 nanoparticles in the studied samples have nanoscale sizes and a low concentration in the matrix, which makes them weakly visible in the diffraction patterns.

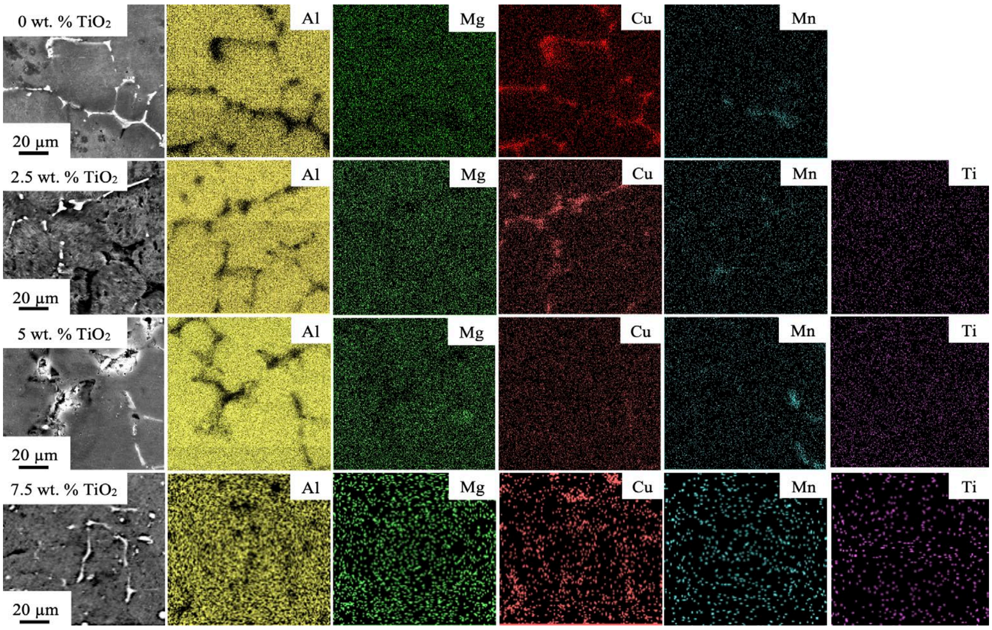

Fig. 8 *SEM image of AA 2024 alloy with the different loaded TiO2 fraction. Elemental mapping was utilized to prove the presence of Ti atoms in the microstructure*

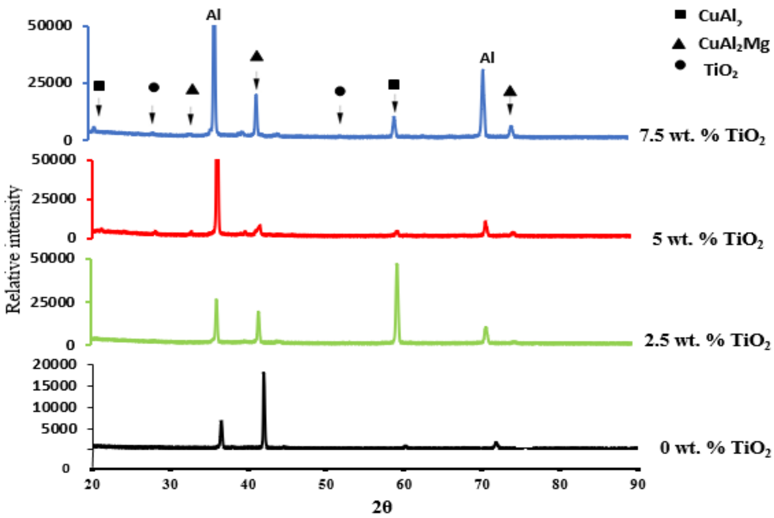

Fig. 9 *Diffraction pattern of the 2024 with the different loading fractions of TiO2 nano powder before heat treatment*

4. Conclusions

In this research, AA2024 was enhanced by the inclusion of TiO_2 nanoparticles at various mass percentages through the stir casting method. To enhance the mechanical properties of the alloys, a combination of solution annealing and aging processes was applied. From these experiments, the following conclusions can be derived:

1. The strength and plasticity of the composite increases with the increasing of mass contents of TiO_2 nanoparticles. The highest UTC of 299 MPa was obtained in the alloy with 2.5 wt. % TiO_2. The optimal combination between strength and elongation is obtained at 7.5 wt. % TiO_2 which showed UTS of 286 MPa and elongation of 6 %.
2. The Vickers hardness was increased by 40 % when 5 % TiO_2 was added compared with the initial A2024 alloy. This improvement might be related to the solution hardening mechanism in which TiO_2 particles behave as obstacles and hamper the movement of dislocations.
3. Scanning electron microscopy showed that Ti is uniformly distributed in the microstructure in each sample not depending on the mass fraction of TiO_2. There were no distinguishable precipitations found alongside the grain boundaries.
4. XRD analysis showed no evident resultants of reactions between TiO_2 and Al confirming SEM results.

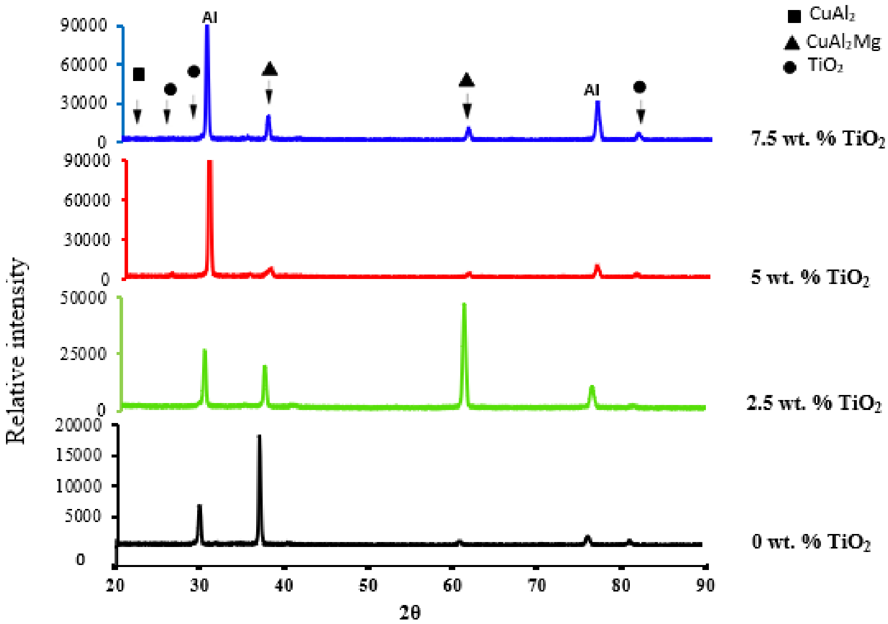

Fig. 10 *Diffraction pattern of the 2024 with the different loading fractions of TiO2 nano powder after heat treatment*

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Hamid M. Mahan;* ***data collection:*** *Nada M. Al-Nedawi;* ***analysis and interpretation of results:*** *Konovalov Sergey, Ahmed H. Ali, Hamid M. Mahan;* ***draft manuscript preparation:*** *Konovalov Sergey, Hamid M. Mahan. All authors reviewed the results and approved the final version of the manuscript.*

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