# Analysis and Characterization of TIO<sub>2</sub> Nanoparticle Effect on Aluminum Matrix Nanocomposites by Stir Casting

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**ABSTRACT**: The objective of this study was to use stir casting to create aluminum matrix nanocomposites with a uniform dispersion of  $TiO_2$  nanoparticles to evaluate their effect on the performance of aluminum-based products. Microstructural analysis significantly reduced the grain size, with an average drop of 18.2%. This suggests that the TiO2 nanoparticles affect the mineral matrix, which improves the mechanical properties. With the incorporation of TiO2 particles, the results showed improvements in stiffness (6.97%), nominal stress (27.92%), actual failure stress (25.6%), and ultimate stress (26.1%). Outperforming commercially pure aluminum, the mechanical properties of the resulting nanocomposites were significantly improved by adding  $TiO_2$  nanoparticles into the aluminum matrix. Microstructural and X-ray diffraction (XRD) studies confirmed the beneficial effects of the addition of nanoparticles.

Keywords: Aluminum matrix; TiO<sub>2</sub> nanoparticles; Mechanical Properties; XRD.

## تحليل وتوصيف تأثير الجسيمات النانوية TiO2 على مركبات الألومنيوم النانوية عن طريق الصب بالتحريك رياض عزاوي بدر, عبد السلام يوسف عبيد

الملخص: كان الهدف من هذه الدراسة هو استخدام الصب بالتحريك لإنشاء نانو مركبات ذات مصفوفة ألومنيوم بتشتت موحد لجسيمات نانوية من ثاني أكسيد التيتانيوم لتقييم تأثيرها على أداء المنتجات القائمة على الألومنيوم. أدى التحليل المجهري إلى تقليل حجم الحبيبات بشكل كبير، بمتوسط انخفاض بلغ 18.2٪. يشير هذا إلى أن جسيمات نانوية من ثاني أكسيد التيتانيوم تؤثر على مصفوفة المعدن، مما يحسن الخصائص الميكانيكية. مع دمج جسيمات ثاني أكسيد التيتانيوم، أظهرت النتائج تحسنات في الصلابة (6.97٪)، والإجهاد الاسمي (27.92٪)، وإجهاد الفشل الفعلي (26.6٪)، والإجهاد النهائي (26.1٪). تفوقت على الألومنيوم النقى تجاريًا، وتحسنات في الصلابة (6.97٪)، والإجهاد الاسمي (27.92٪)، وإجهاد الفشل الفعلي (26.6٪)، والإجهاد النهائي (26.1٪). تفوقت على الألومنيوم النقى تجاريًا، وتحسنت الخصائص الميكانيكية للمركبات النانوية الناتجة بشكل كبير عن طريق إضافة جسيمات نانوية من ثاني أكسيد التيتانيوم إل التقريرات المفيدة لإضافة الجسيمات النانوية (XRD) الألومنيوم. أكدت دراسات المجهر والحيو، بالميتان بالنوية من ثاني

الكلمات المفتاحية: مصفوفة الالمنيوم ; جسيمات اوكسيد التيتانيوم النانوية; الخواص الميكانيكية ; حيود الاشعة السينية.

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#### 1. INTRODUCTION

Aluminum allovs are frequently employed in the automotive industry due to their good mechanical properties; nevertheless, surface treatment is frequently needed because of their high sensitivity to corrosion. Due to its strong oxidation resistance, TiO<sub>2</sub> is frequently utilized to enhance the corrosion resistance of aluminum surfaces. Due to high-performance demand and affordable materials, the researchers have focused on monolithic to composite materials, such as aluminum matrix nanocomposites (AMNC). The third most common element and one of the lightest industrial metals, aluminum makes up 8% of the earth's crust. Due to its outstanding properties, including low weight, high ductility and good thermal properties. Aluminum and its alloys have found widespread application in many industries. However, their limited use as an automotive material is still due to their low hardness, strength, and wear properties [Samal et al.,2020; Singh et al.,2021; John 1985 ]. Aluminum metal matrix composites (AMMCs) with high specific stiffness and high strength can be used in long-term applications where weight reduction is a key component, such as robots, high-speed rotating shafts, machinery, and automotive engine and brake parts. The ability to produce a homogeneous distribution of reinforcement inside the matrix has a direct impact on the characteristics and quality of nanocomposite materials. Aluminum, magnesium, titanium, and their allovs are the most commonly used matrices in the manufacture of MMCs. Aluminum matrix composites (AMC) are a popular choice among matrix materials due to their low cost and versatile processing capabilities. AMC is superior to other materials in terms of formability, corrosion resistance, and strength-toweight ratio, making it an ideal choice for structural applications in the automotive and aerospace industries [Rajan, R. T. K., et al., (2023)]. This process involves melting the matrix material, mechanically stirring it to create a vortex on the melt's surface, and then adding reinforcing particles to the vortex. Before the casting process, this mixing procedure is then run for a few minutes. During the process, stirring aids in both better particle distribution and maintaining their suspension. Metal Matrix Composites (MMC) can now be produced on a large scale and used in a variety of applications to improve manufacturing techniques and the addition of a range of reinforcing components. The inclusion of ceramic reinforcement reduces the ductility of composites, limiting their use in many structural applications. The overall strength of AMCs is influenced by their low fatigue properties and low fracture toughness, caused by insufficient ductility. Previous studies show that the reinforcement volume fraction and reinforcement particle size have a direct impact on the overall mechanical performance of MMCs [John et al. .2019: Madhu et al..2018: Ghasali et al..2018: Prosviryakov et al., 2018]. By changing the volume fraction, types, and sizes of reinforcing particles, these

characteristics can be improved. Since it is necessary to improve wear resistance, friction resistance, and some promising mechanical properties, AMMC fabricates a wide range of aerospace components [Prosviryakov et al., 2015,2017]. The importance of aluminum in airframe construction has not diminished despite the growing use of composite materials; High-strength aluminum alloys still play a significant role. Aluminum is one of the most easily produced high-performance materials, meaning production and maintenance costs are reduced. It is also a relatively inexpensive and lightweight metal that can be heat treated and loaded with relatively high-stress levels. Recent developments in aluminum aircraft alloys have made them competitive with modern composite materials [Urban et al.,2019; Lakra et al.,2010]. Ceramic oxide particles are the main type of reinforcement used in aluminum MMS. Although reinforcement is more readily distributed in an aluminum matrix [Chen et al.,2013; Welham et al.,2002], carbides [Setoudeh et al.,2016], nitrides [Prosviryakov et al.,2020] and borides [Morris et al.,2010] have been used both in the work site and by local methods. The uniformity of the reinforcing particles over the surface of the composite is another aspect that helps improve the mechanical properties of the composite. However, maintaining the uniformity of reinforcing particles is one of the most challenging tasks for researchers. The main disadvantages of using standard or stir-casting technology are the non-uniform distribution of reinforcing elements, unfavorable interfacial interactions in the melt, and poor wettability between the matrix and the reinforcement. This work aims to determine how titanium dioxide (TiO2) nanoparticles affect the mechanical properties and microstructure. The tensile strength and hardness of the material are assessed, and the results are contrasted with those of aluminum, by obtaining samples of an aluminum matrix nanocomposite doped with TiO2 nanoparticles.

### 2. EXPERIMENTAL PROCEDURE

Technically, the primary component of this work is aluminum. Titanium dioxide (TiO2) nanoparticles with an average diameter, of 40 nm are selected as the reinforcement. The nanocomposite is produced using aluminum as the basis metal and 2.6 wt% TiO<sub>2</sub> nanoparticles. The following is how aluminum sample manufacture is done. A hacksaw is used to first cut the aluminum to the necessary weight. In a shaft furnace, aluminum is deposited in a graphite crucible.

In a shaft kiln, charcoal is burned and stacked around a crucible. An electric fan that is attached to the shaft furnace's one side pulls air from the atmosphere and directs it there to produce heat continuously. With this fan, a decent A certain amount of air is given to the coal. Aluminum starts to melt as a result. Using crucible-raising tongs, the crucible is raised once the aluminum has melted to a temperature of roughly 700 C. After that,



the crucible is inserted into the pouring rod. Using a pouring rod, the melt is then poured into the mold. The molten metal is kept in the molds for a while after pouring to allow it to harden. The specimens are ready for tensile and hardness testing after hardening. A schematic illustration of a shaft furnace installation is shown in Figure 1.



Figure 1. Installation of pit furnace configuration.

depicts the process Figure 1 for creating nanocomposite samples with an aluminum matrix by casting and mechanical stirring the aluminum starts at a speed of 450 rpm, and mechanical stirring casting equipment is employed to stir the melt.  $TiO_2$ nanoparticles of size 40 nm are put into a funnel containing an aluminum matrix and stirred on top of a crucible using a steel tube. The added amount of TiO<sub>2</sub> was 26 g. While the shaft furnace continued to run, the solution was mixed for 5 minutes. Lastly, it is used to cast the specimens required for hardness and tensile Figure 2 shows how aluminum matrix tests. nanocomposite samples are produced using the mechanical stir-casting method.



#### Figure 2. Schematic diagram of powered stir casting setup.

Using an optical microscope equipped with image analysis equipment, the microstructural characterization of the produced nanocomposites is performed. Polishing and etching methods are applied in tests. Using wire electrical discharge machining (EDM) equipment, a sample of approximately 12 mm x 12 mm is cut from various points in the final casting (wire EDM). The pattern is then polished in five steps using emery cloths with grit sizes of 100, 200, 400, 800, and 1600. Following the grinding process, the samples are polished by brushing 1/0 to 4/0 sandpaper across their surfaces. Using a damp spinning wheel that is cloth-covered, the desired smooth surface is produced without any scratches. As an abrasive, diamond paste is employed. The use of a double-disk polishing device for this procedure is applied. The polished sample is etched with Keller's reagent (175 ml of purified water + 6 ml HNO3 + 4 ml HCl + 3 ml HF). The microstructure of samples is examined and their features are evaluated using an optical microscope attached with image analysis tools. All samples of nanocomposite are subjected to X-ray diffraction (XRD) examination utilizing an X-PERT PRO diffractometric equipment. With a Brinell hardness tester with a load, of 550 kg and an indenter with a steel ball having a diameter, of 12 mm, the hardness of every nanocomposite sample is assessed. For the hardness measurement, the holding period is one minute. The nominal and true breaking stress and ultimate strength are the utmost stress that is put through tests on the universal testing machine (UTM), which is used to test the mechanical properties (tension, compression, etc.). Figure 3 shows the casting process as well as the procedure for the samples used in the test setup. Figure 3 shows the casting process as well as the procedure for the samples used in the test setup.







Figure 3. Casting process for three procedures and the chemical analyses equipment.

TiO2 nanomaterials' mechanical and physical qualities were density  $gm/cm^3 = 3.95$ , Shear Modulus=111.2 MPa, Young's Modulus=285 GPa, and Elastic Limit=377.5 MPa. Table 1 lists the mass percent.

**Table 1.** The chemical composition of Al nanocomposite(AMNC) in mass percent

Element	Compostion Wt%	
Chromium	0.011-0.25	
Magnesium	0.7-1.3	
Titanium	0-0.10	
Silicon	0.6-1.2	
Copper	0.0-0.10	
Manganese	0.5-1.0	
Iron	0-0.6	
Zinc	0-0.21	
Others	0.05	
Aluminum	Balance	

Table 2 contains the majority of the mechanical and physical characteristics of Al nanocomposites (AMNC). The compression molding parameters for the creation of nanocomposites (AMNCs) are listed in Table 3. Figure 4 displays SEM images of the powders utilized in this investigation of nanoparticles of titanium dioxide samples of aluminum matrix nanocomposite.

Table 2.	Al nanocomposites' (AMNC) mechanical and	
	physical characteristics.	

	Al nano composites (AMNC )		
	before adding (TiO <sub>2</sub> )	after adding (TiO <sub>2</sub> )	
Modulus of Elasticity (GPa)	75	77	
Yield stress (MPa)	77	79	
Ultimate Stress (MPa)	148.8	152.7	
Hardness (BHN)	29	33	



Figure 4. SEM of the powders utilized in this investigation of nanoparticles of titanium dioxide samples.

**Table 3.**list of the compression molding parameters for the<br/>production of nanocomposites (AMNCs).

S.No.	Process parameters	Values
1	Stirring temperature	700 °C
2	Stirring speed	450 RPM
3	Stirring time	5 mins
4	Preheat temperature of reinforcement particles	320 °C
5	Preheat temperature of permanent die	320 °C
6	Squeeze pressure	210 MPa

#### 3. RESULTS AND DISCUSSION

Using optical microscopy at x 5000 and X 10000 magnification, images of the microstructure of aluminum and aluminum matrix nanocomposite (AMNC) are illustrated in Figures 5 and 6 respectively. The SEM images clearly show that the grain size decreases when TiO<sub>2</sub> nanoparticles of various sizes are introduced to the aluminum matrix from 43.12 to 25.10 nm. Consequently, the aluminum grain size is refined during the production of nanocomposite samples with an aluminum matrix, and as a result, a fine dispersion of grains is observed. Due to divergent boundaries, bigger grain sizes result in a higher dislocation ratio than smaller ones, which have more surface area and resistance. The mechanical characteristics of the resulting nanocomposites depend on the reduction of aluminum grain size. Two alternative mechanisms can be used to explain the decreasing of aluminum grain size upon the addition of TiO<sub>2</sub> nanoparticles: firstly by the fixation of old grain boundary by particles of the second phase and secondly by enhancement of nucleation [Welham et al., 2002]. Figure 5 presents the SEM optical microscopy for pure aluminum particles. Figure 6 reveals primary aluminum dendrites in the alpha phase of the microstructure and aluminum reinforced with 40 nm TiO<sub>2</sub> particles. The proper distribution of the reinforcement particles that dispersed within aluminum matrix is clear as tiny pores seen in Fig. 6 which tend to agglomerate owing to higher surface energy. The optical microscope is used to determine the grain size and we observed that adding TiO<sub>2</sub> nanoparticles to the grains can reduce the average grain size from 43.12 to 25.10 nm (see Fig.7).



Figure 5. Optical microscopy of pure aluminum.

As the grain size becomes smaller, its mechanical properties become stronger. Reinforcing particles (TiO2 nanoparticles) reduce grain size by providing sites for nucleation, which enhances the mechanical properties of composites [Christy, J.V., at el, 2020]. Previous research indicates that another aspect that affects grain refinement in composite materials is the large plastic deformation that occurs during the stirring process and causes dynamic recrystallization of the material [Christy, J. V., et al., (2020)]. The number of objects or grains in the optical micrograph is counted, the corresponding diameter values are estimated and image analysis software is used to calculate the average size.



**Figure 6.** Optical microscopy of reinforced aluminum with 40 nm TiO<sub>2</sub> particles

When reinforcing  $\text{TiO}_2$  nanoparticles are added to the aluminum matrix, the grain size of the matrix decreases, and the surface area of the reinforcing material increases. Therefore,  $\text{TiO}_2$  nanoparticles are added to aluminum, which increases its strength [Setoudeh et al.,2016].



Figure 7. Variation in grain size.

Figure 8 shows how the Brinell hardness value changes for an aluminum nanocomposite with an aluminum matrix. It can be seen from the graphs that the addition of TiO<sub>2</sub> nanoparticles to the aluminum matrix increases the Brinell hardness of the aluminum alloy. Compared to the aluminum alloy, the nanocomposite is harder. In accordance with the fact that aluminum is a soft material, it should be expected that hard TiO<sub>2</sub> will increase the nanoparticles hardness of nanocomposite [Prosviryakov et al.,2020]. It was discovered that hardness enhanced mechanical qualities, notably a 6.97% increase in hardness.



Figure 8. Varaiation of Brinell hardness.

TiO2 nanoparticles possess a significant effect on the aluminum matrix's strength, as evidenced by the noticeably higher hardness of nanocomposite compared to pure aluminum. The study of microstructure has already shown that the fine-grained structure of the aluminum matrix and the role of uniform dispersion of nanoparticles, create barriers to the movement of dislocations which are responsible for this improvement in the hardness value. Figures 9-11 show the variations of actual fracture stress, nominal fracture stress and ultimate stress for aluminum nanocomposite compared to original aluminum stresses.



Figure 9. Variation of nominal breaking stress.

Based on data from the microstructure study, a comparison is made between nanocomposite samples with aluminum matrix and samples of aluminum grains that have changed in size (Figure 7). It is obvious that the nominal fracture stress of nanocomposite, the actual fracture stress and limiting stress increase as a result of an increase in the area of grain boundaries caused by grain refinement and efficient transformation of tensile load necessary for the uniform dispersal of  $TiO_2$ nanoparticles in aluminum microstructure. A higher strength of aluminum reinforced with nanoparticles is observed indicating the presence of different concentrations of dislocations [Morris et al.,2010].



Figure 10. Variation of the true breaking stress.



Figure 11. Variation of Ultimate stress.

The area of the reinforcing surface and grain size of the matrix increase when reinforcing TiO<sub>2</sub> nanoparticles are introduced into the aluminum matrix. As a result, the inclusion of TiO<sub>2</sub> nanoparticles increases the aluminum strength. The strength of created nanocomposites is directly affected by a decrease in the size of the grain. With the inclusion of  $TiO_2$ aluminum nanoparticles, the tensile strength of aluminum increases. This is a consequence of the grain refinement of aluminum microstructure, which has been studied [John et al., 2019; Madhu et al., 2018]. It is significant to note that pre-stressing causes the tensile equipment to start stresses at zero, although this does not influence the alloy's tensile behavior. It was determined that the tensile strength was 114.3 MPa. Additional sample testing could provide a more accurate picture of tensile

strength. After the T6 heat treatment, the alloy still exhibited brittle behavior and had a decreased elongation compared to the mean acquired before the application of heat treatment. [Jaber, A.M. and Krishnan, P.K., 2022.]

XRD is used to determine the phase for all samples. Peak intensities were measured, and any new phases developed during the manufacturing process were identified by XRD analysis. Figure 12 shows the X-ray analysis of pure aluminum. The diffraction patterns obtained show that the aluminum matrix had the highest intensity peaks  $(311^{\circ})$ . While aluminum reinforced with 40 nm TiO<sub>2</sub> particles was subjected to X-ray examination as shown in Figure 13, which showed that had the highest intensity peaks  $(111^{\circ})$ . It was clear that the material was polycrystalline because of the high peak and because of the pressure used in the injection molding process, the atoms were tightly arranged in a well-organized hexagonal configuration.



Figure 12. XRD pattern of pure aluminum.

The eutectic phase is also indicated by elemental mapping. Al and TiO<sub>2</sub> particles were the most abundant components both before and during heat treatment. However, before heat treatment, the eutectic phase is deposited at the grain boundaries; After heat treatment, it is uniformly dispersed and integrated with the aluminum matrix.Moreover, the XRD data shows that aluminum, the base material, has the largest peaks (see Figures 12 and 13). In addition to these elements, peaks of mineral phases were observed. The characteristics of the alloy and the type of fracture are influenced by the appearance of these phases at the grain boundaries. [Jaber, A.M. and Krishnan, P.K., 2022]



Figure 13. XRD pattern of aluminum reinforced with 40 nm TiO2 particles.

By comparing the position and intensity of peaks of XRD in the JCPDS (Joint Committee for Powder Diffraction Standards) database, one can identify the diffraction peaks at two values, which are a distinctive pattern of pure aluminum. As a result, map No. JCPDS agrees with the experimental XRD pattern of pure aluminum. Comparing it with those in the JCPDS database, it indicates the XRD pattern of TiO2 nanoparticles. Concluding the existence of TiO2 nanoparticles in the aluminum matrix, the experimental XRD pattern of TiO2 nanoparticles in the aluminum matrix, the experimental XRD pattern of TiO2 nanoparticles is therefore consistent with the JCPDS map [Chen et al., 2013; Welham et al., 2010; Ramadoss et al., 2022].

#### 4. CONCLUSIONS

The following are significant findings from this research:

- TiO2 nanoparticle introduction led to the most significant benefits through reducing the average grain size by (18.2%), which explains how the nanomaterial interacts with the base metal matrix to improve mechanical properties. Improvements in hardness by (6.97%) were achieved.
- The outcomes demonstrate that the addition of TiO<sub>2</sub> particles to the base aluminum alloy enhanced the nominal stress by (27.92%), the real fracture stress by (25.6%) and the ultimate pressure by (26.1%).
- The results of XRD analysis showed that TiO<sub>2</sub> nanoparticles are present in the aluminum matrix. The improved mechanical performance may be related to increased cohesion between the alloy components of the nanocomposites, such as higher bonding between TiO<sub>2</sub> and aluminum, a relative decrease in the porosity of the alloy, or a lower density of intermolecular dislocations [Prosviryakovet al.,2020].

#### CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding this article.

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