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Fabrication and characterization of Al-7Si alloy matrix nanocomposite by stir casting technique using multi-wall thickness steel mold

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ABSTRACT

Introduction. The *Al-7Si* is considered one of the key aluminum alloys due to its favorable combinations of casting and mechanical properties. Metal matrix composites (MMCs) reinforced with ceramic particles are widely used in high-tech industries such as military, automotive, aerospace, and electrical engineering. ***The purposes of this study*** are threefold: (1) to investigate the feasibility of producing composite materials based on the *Al-7Si* alloy reinforced with varying amounts of TiO_2 nanoparticles using a stir casting technique; (2) to investigate the effect of mold wall thickness on the microstructure and mechanical properties of the *Al-7Si* alloy during solidification; and (3) to analyze the influence of the reinforcing component content on the mechanical properties and wear resistance of the resulting composite materials. **Methodology.** Metal matrix composite materials based on the *Al-7Si* alloy, containing 0, 2, 4, and 6 wt. % TiO_2 nanoparticles, were fabricated using a stir casting technique. Cylindrical specimens with a diameter of 15 mm and a length of 18 mm were prepared for metallographic and mechanical testing. **Results and discussion.** It is found that the solidification rate increases with increasing mold wall thickness. This leads to an increase in the cooling rate and, consequently, to the formation of a finer-grained structure. The microstructure of the casting demonstrates a change in grain size from fine to coarse when transitioning from the outer surface (adjacent to the inner mold wall) to the inner part of the casting. As a result, the microhardness near the inner mold wall is higher. Density measurements indicate that composites with a higher amount of reinforcing particles exhibit greater porosity. Furthermore, the results of hardness and wear tests reveal that an increase in the TiO_2 particle content leads to increased hardness and a significant reduction in the wear rate of the composite materials.

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Introduction

Metal matrix composites (*MMCs*), particularly those based on aluminum alloys, have garnered increased attention from the automotive and aerospace industries in recent years [1–3]. Among matrix materials for *MMCs*, the *Al-Si* and *Al-Cu* systems have been the most thoroughly investigated [4–7]. With a particular emphasis on tribological properties, the automotive and aerospace industries are increasingly utilizing aluminum alloys reinforced with ceramic particles [8, 9]. Various materials, including carbides, nitrides, borides, oxides, and intermetallics, are widely employed as reinforcing components in aluminum matrix composites [10, 11]. There are three primary approaches to fabricating these composites: solid-state, liquid-state, and semi-solid-state processing methods [12–14].

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For particle-reinforced *MMCs*, the molten metal stirring method is the most often used technique. In order to achieve homogenous mixing, this technique requires careful control of several manufacturing parameters, such as molten metal temperature, stirring time, continuous and uniform rate of reinforcement component addition to the mixture, and stirring speed [13]. The molten metal stirring method offers several advantages, including simplicity, low processing costs, ease of matrix structure control, and the production of near-net shape components. However, a significant challenge encountered when using this method is the poor wettability of ceramic particles, necessitating special measures to ensure adequate bonding between the reinforcing particles and the matrix. Addressing this challenge is essential for achieving high-quality component bonding and improving the overall performance characteristics of the composite materials [3, 9].

TiO_2 is a prominent example of a ceramic material widely used in *MMCs* and possessing significant potential as a reinforcing component for aluminum composites. Aluminum alloys reinforced with TiO_2 are characterized by enhanced hardness, making these materials promising for a wide range of applications, including electronic devices, automotive parts, and aerospace components. The improved mechanical properties of the material provide enhanced stability and durability in demanding operating conditions, such as high temperatures or corrosive environments. It is confirmed that incorporating TiO_2 into aluminum effectively improves the mechanical properties and hardness of aluminum alloys, expanding its application possibilities [4, 15, 16, 17]. In their study, *Nassar* et al. evaluated the mechanical, wear, and structural characteristics of an Al/TiO_2 nanocomposite produced by powder metallurgy. The results demonstrated a uniform distribution of TiO_2 nanoparticles within the *Al* matrix and a low porosity. It is established that with increasing content of nanoscale TiO_2, the yield strength, wear resistance, ultimate tensile strength, and hardness of the nanocomposite increased [2].

It should be noted that TiO_2, like most ceramic materials, exhibits poor wettability with molten aluminum [18–20]. Several methods have been developed to address this issue and improve the wetting of reinforcing particles by the matrix. These include the addition of reaction-active particles, such as magnesium, heat treatment of ceramic particles, and the application of metallic coatings [4, 19]. The cooling rate of a casting is influenced by factors such as mold wall thickness, pouring temperature, and the heat accumulation capacity of the casting mold [21–23]. Increasing the cooling rate has a significant influence on the as-cast structure, leading to grain refinement and modification of the matrix structure [24, 25]. This, in turn, affects the mechanical characteristics, increases the tendency to chill formation, and results in increased hardness, but may reduce strength and impair the machinability of castings. The eutectoid transformation following solidification also influences the microstructure of the as-cast alloy [25, 26].

The purpose of the current study is to investigate the influence of mold wall thickness during the solidification process on the microstructure and mechanical properties of the *Al- 7Si* alloy matrix. In addition, the objective of this study is to develop a comprehensive understanding of the properties and potential of *Al- 7Si* alloy-based composites reinforced with TiO_2 particles for its application in high-stress structural components. ***To achieve these objectives, the following tasks were addressed***:

– the influence of the mass fraction of the hardening component TiO_2 on the density, hardness, and wear resistance of the composites was investigated;

– a metallographic analysis was performed on the microstructure formed in the *Al- 7Si* alloy after casting using multi-wall thickness steel mold;

– the microhardness and average grain size were determined in the obtained alloy samples cast into steel molds with different wall thickness.

Methods

In this study, a hypoeutectic *Al-7Si* alloy was used as the matrix material for the composites. The chemical composition of the alloy is shown in Table 1. TiO_2 particles with the size of about 80 nm were used as reinforcement. Fig. 1 shows the results of qualitative X-ray diffraction (*XRD*) analysis of the TiO_2 nanopowder used as the reinforcing material in this investigation. The mass fraction of TiO_2 nanoparticles

Table 1

Chemical composition of A356 alloy used as the matrix in the *MMCs*

Chemical contents of elements, %							
Al	*Si*	*Mg*	*Mn*	*Cu*	*Zn*	*Ni*	*Fe*
bal.	7.1	0.5	0.01	0.316	0.04	0.01	0.14

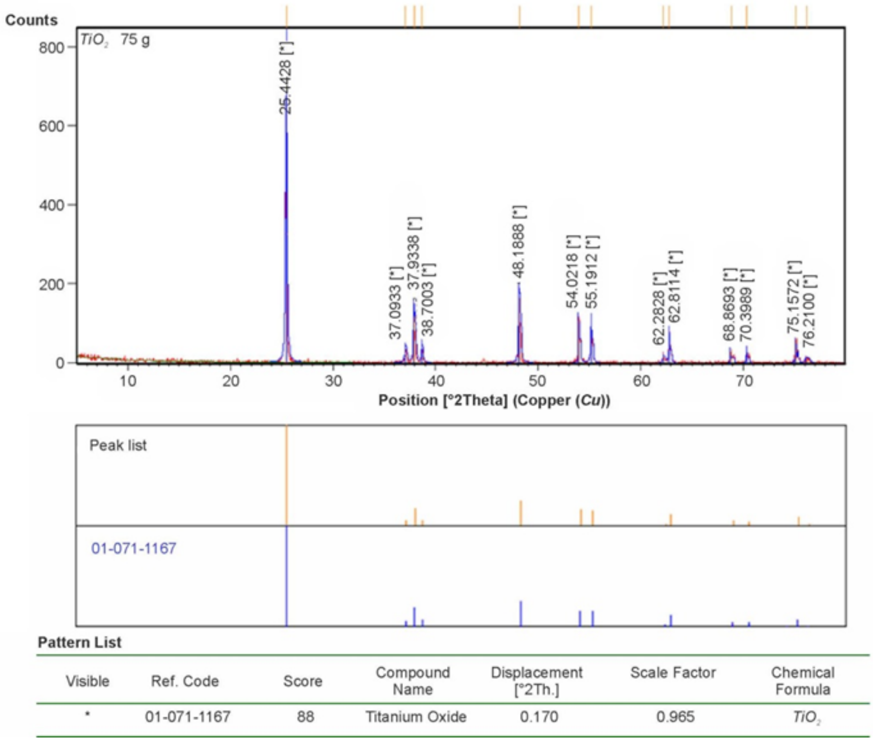


Fig. 1. Result of qualitative *XRD* analysis of nano-titanium oxide (*TiO_2*)

was varied and set to 0, 2, 4, and 6 %. A specially designed and built electric muffle furnace simplified the composite fabrication process. Fig. 2 summarizes the process flow for manufacturing the Al-% Si alloy specimens and nanocomposites. For the experiment, 400 g of the *Al-% Si* alloy was charged into a crucible, which was then placed in an electric resistance furnace. The molten *Al- 7Si* alloy was then cast into low-carbon steel molds with wall thicknesses of 8, 12, and 29 mm to fabricate samples for studying the influence of mold wall thickness on structure and properties.

Nanocomposite samples with an *Al- 7Si* alloy matrix and TiO_2 particles as the reinforcing material were fabricated using a separate steel mold with a wall thickness of 7.5 mm as illustrated in Fig. 2. The TiO_2 particles were introduced into the melt at a constant rate of 5 g/min. After the particle addition was complete, the melt was stirred for 5 minutes at a speed of 450 rpm. To enhance the wettability between the reinforcing component and the matrix, 1 % magnesium was added to the melt. Before being dispersed in the melt, the TiO_2 particles were also preheated at 700 °C for 2 hours to improve its wettability. After fabrication the as-cast specimens were ready for further examination of its mechanical properties and microstructure. The metallographic specimens were subjected to sequential grinding and polishing using *SiC* abrasive papers with grit sizes ranging from 150 to 1,200. To reveal the microstructural components, the samples were etched using a solution that contained 75 ml *HCl*, 25 ml HNO_3, 5 ml *HF*, and 25 ml H_2O.

An optical microscope and a scanning electron microscope (*SEM*) were used at various points on the specimen surface to analyze the microstructure. Grain size determination was performed according to *ASTM E112*. Microhardness was measured on a VHS-1000 microhardness tester using a load of 100 g, utilizing ground and polished specimens. Wear tests were conducted on a pin-on-disc tribometer. The specimens were subjected to loads of 10 N and 20 N while sliding against a 200 mm diameter steel disk at a constant speed of 1 m/s with sliding distances of 350 m and 700 m. The bulk density of *Al- 7Si* matrix composites

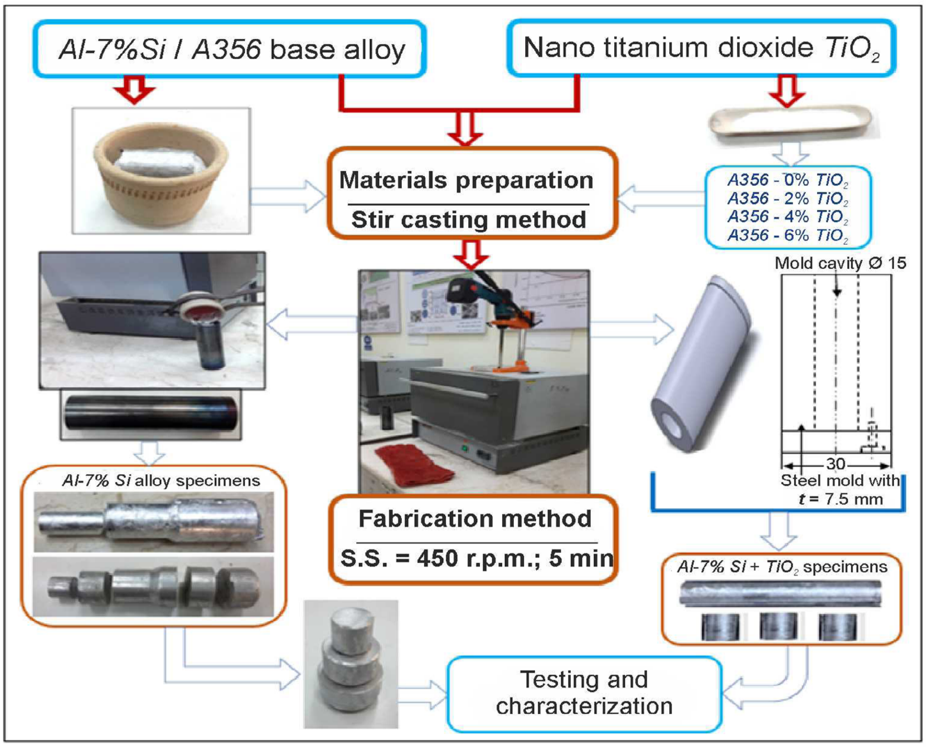


Fig. 2. Flowchart and schematic representation detailing the fabrication sequence of the present study

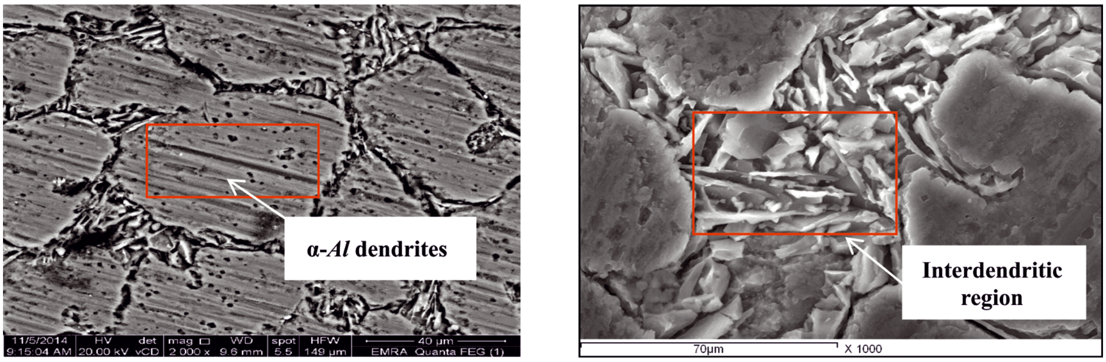
reinforced with TiO_2 particles was determined using the *Archimedes* method. The theoretical density was calculated using the rule of mixtures, based on the mass fraction of TiO_2 particles. The porosity of the composites was then calculated by comparing the experimental and theoretical densities for each specimen. The hardness of the composites evaluated using *Vickers* method.

Results and Discussion

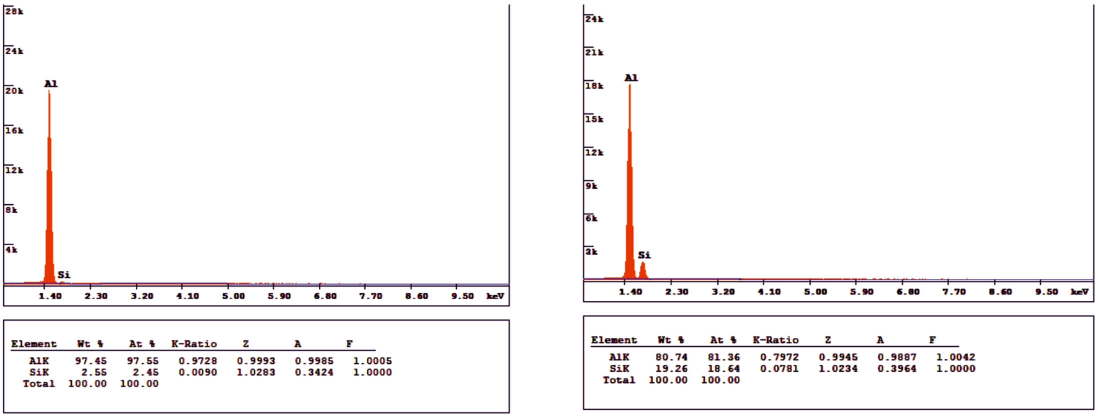
Fig. 3 presents the results of *SEM* and *EDS* analysis of the *Al- 7Si* alloy in the as-cast state. It is evident from Figs. 3, *a* and 3, *c* that the microstructure of the matrix alloy consists of primary **α**-*Al* dendrites and interdendritic areas, which are either shrinkage pores or a *Si*-rich eutectic phase. The abovementioned phases in the microstructure are reflected in the elemental spectra and mass fractions of the elements at various regions displayed in Fig. 3. The microstructure of the *Al- 7Si* alloy matrix with varying mold wall thicknesses and cooling rates is shown in Figs. 4 to 6. It is evident from these figures that as the cooling rate increases, both **α**-*Al* and eutectic silicon get finer.

A stepped mold with wall thicknesses of 29 mm, 12 mm, and 8 mm was used to achieve variable cooling rates during solidification. Mold wall thickness significantly affected the microstructure of the as-cast *Al-7Si* alloy. As can be seen from the presented micrographs, increasing the mold wall thickness leads to a refinement of the alloy's microstructure. This is likely due to the fact that increasing the thickness of the metal mold wall increases the rate of heat extraction and, consequently, the cooling rate. Cooling conditions are known to influence the degree of grain refinement. Rapid solidification promotes the formation of a fine-grained structure with uniform grain distribution, while slow solidification leads to the formation of coarse grains [27].

The effect of mold wall thickness on the grain size in the cast alloy is depicted in Fig. 7. As the mold wall thickness increases from 8 mm to 29 mm, the average grain size at a distance of 9 mm from the outer surface decreases from 63 μm to 34 μm. At a distance of 4 mm from the outer surface, the average grain size decreases from 48 μm to 28 μm. Thus, the solidification rate has a decisive effect on the degree of grain refinement, which explains the effect of mold wall thickness on microstructure and grain size [28–30]. *Kang*

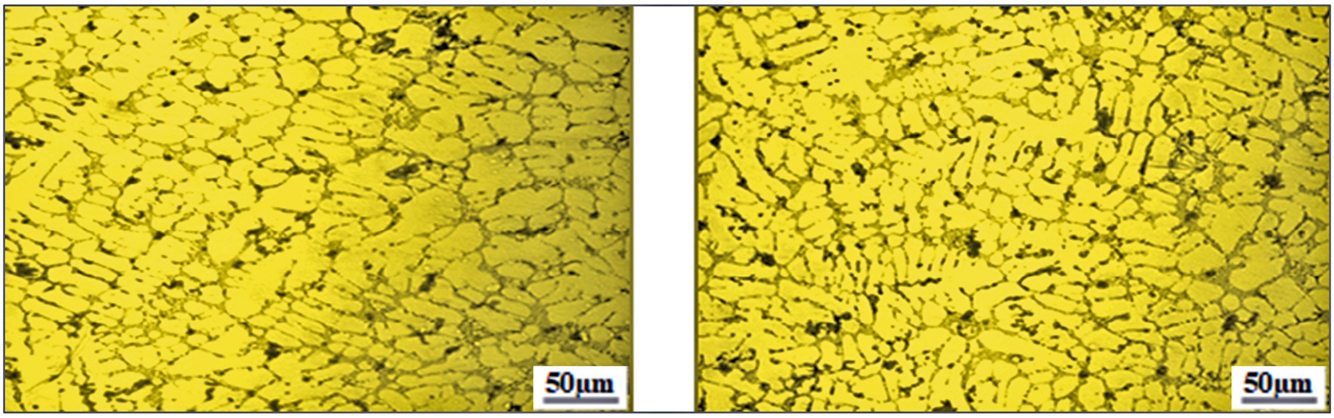


a *b*



c *d*

Fig. 3. SEM/*EDS* analysis on the *Al-Si* alloy-matrix; (*a*), (*c*) microstructure; (*b*), (*d*) *EDS* of α-*Al* dendrites and inter-dendritic regions in (*a*), (*c*), respectively



a *b*

Fig. 4. The microstructure of the casting solidified in 29 mm wall thickness steel mold at: (*a*) distance of 9 mm from mold wall surface; (*b*) distance of 4 mm from mold wall surface

et al. [31] investigated the influence of cooling rate on the mechanical properties and microstructure of *Al-7Si* alloys. According to their findings, a greater undercooling caused by a higher cooling rate, which promotes a reduction in the critical nucleus radius. As a result, the nucleation process is facilitated. However, under high cooling rate conditions, the fine grains do not have sufficient time to grow. Increasing the cooling rate promotes the formation of a finer microstructure. Nucleation occurs more readily with slight undercooling. During surface solidification, numerous nuclei form and grow ahead of the solidification front. An increase in the number of nucleation sites leads to the formation of a finer structure with a larger number of grains. During the solidification of a medium-sized casting, the solidification front moves from the surface to the center. This results in the formation of a homogeneous fine-grained structure throughout the cross-section.

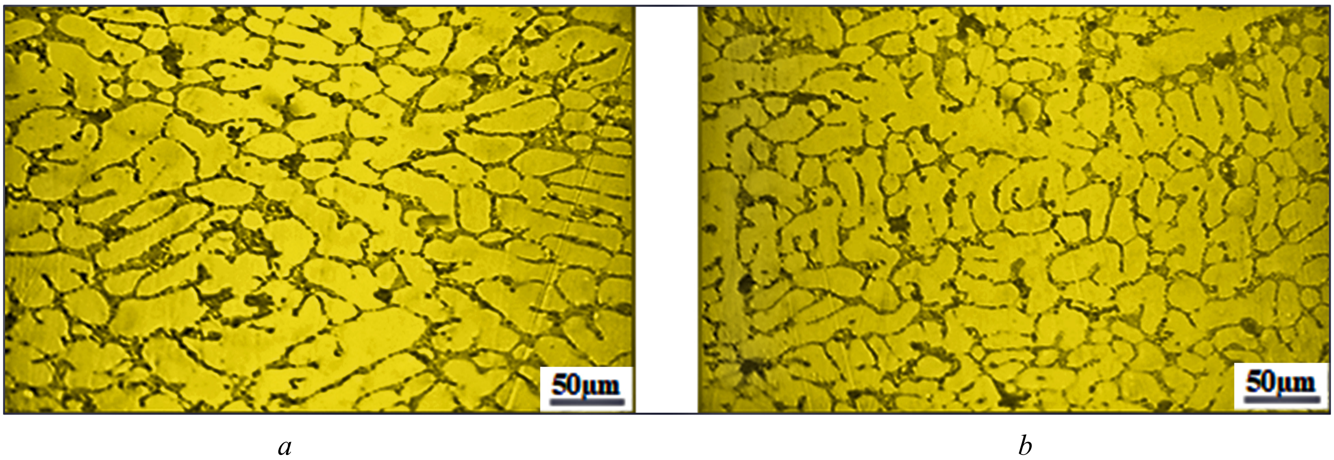


Fig. 5. The microstructure of the casting solidified in 12 mm wall thickness steel mold at; (*a*) distance 9 mm from mold wall surface, (*b*) distance 4 mm from mold wall surface

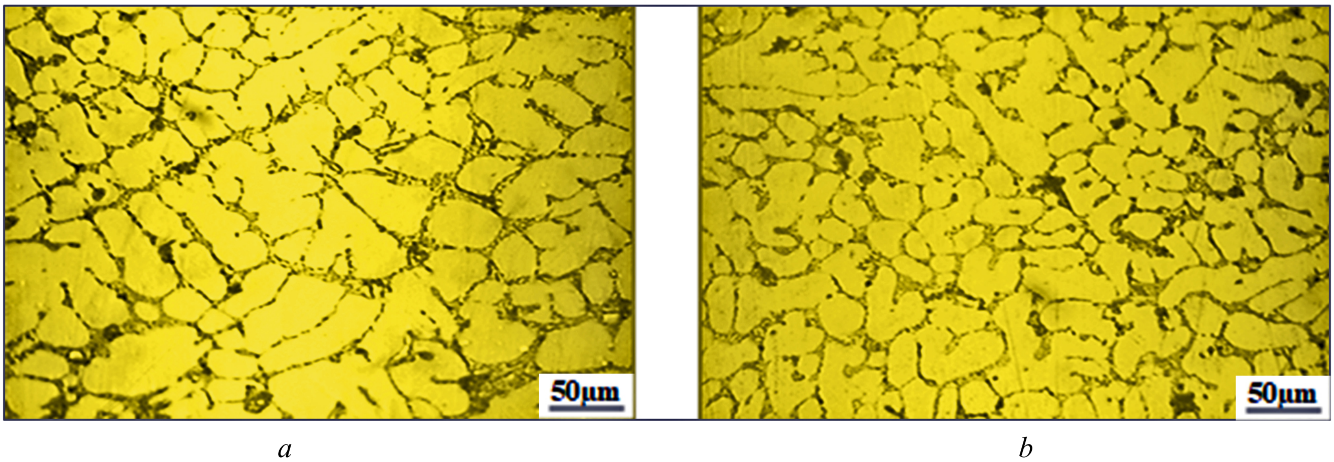


Fig. 6. The microstructure of the casting solidified in 8mm wall thickness steel mold at; (a) distance 9mm from mold wall surface, (b) distance 4mm from mold wall surface

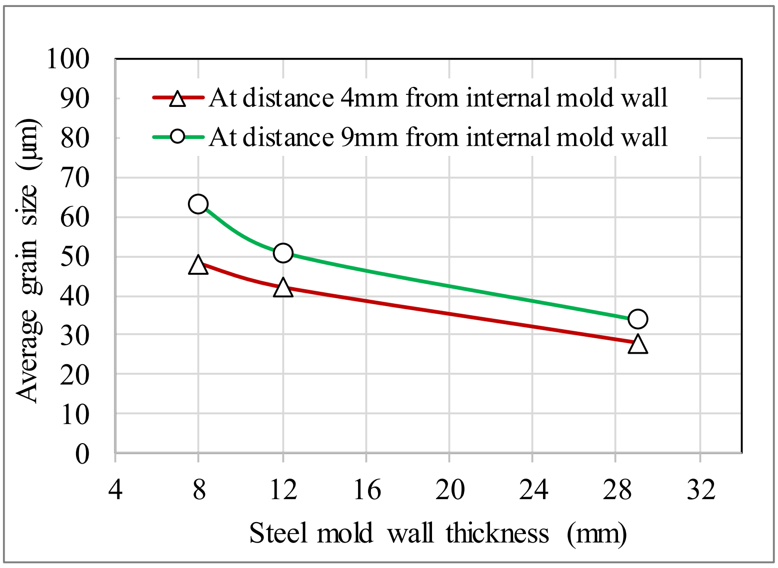


Fig. 7. Average grain size for the castings versus steel mold wall thickness

The microhardness values increase with increasing mold wall thickness due to high cooling rate at the casting surface close to the wall. Fig. 8 shows the relationship between the microhardness values of the castings and the wall thickness of the steel mold. It is evident from this figure that the microhardness of the *Al- 7Si* matrix alloy with a mold wall thickness of 29 mm (close to the mold wall) was 95 HV. At a distance of 9 mm from the inner mold wall, the microhardness decreased to 89 HV. Each value is averaged over four measurements. The casting with a wall thickness of 8 mm had a microhardness of 78 HV close to the mold wall and 74 HV close to the specimen center. As can be seen in Figs. 4 and 6, an increase in average grain size may be the reason for the decrease in microhardness with decreasing wall thickness. Due to the low cooling rate, the microstructure of the alloy obtained in the mold with the minimum wall thickness is characterized by a larger grain size, while in castings obtained in molds with a greater wall thickness, the grain size is smaller due to more intensive heat transfer. Decreasing the cooling rate leads to a deterioration of the alloy's properties and structure, an increase in the dendritic arm spacing (*DAS*), and a reduction in the effectiveness of grain refining and modification processes [32, 33].

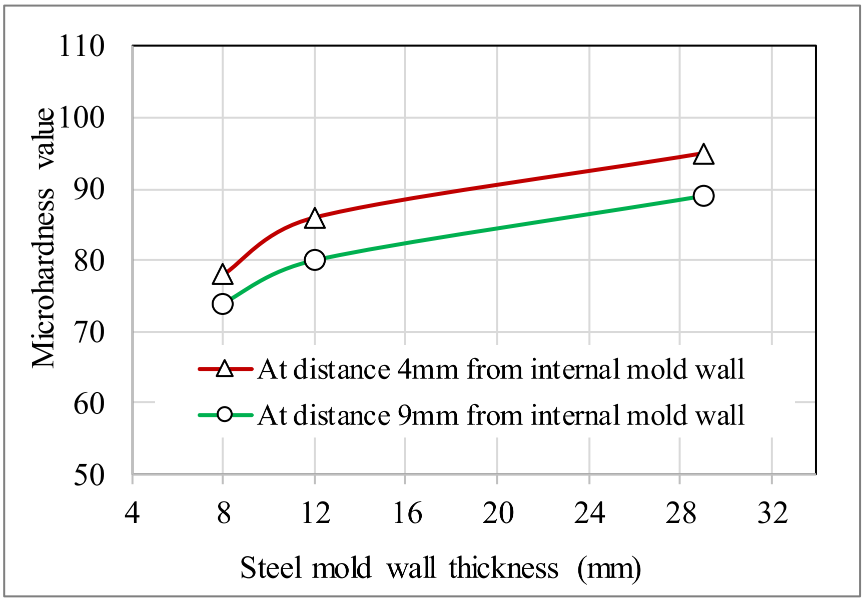


Fig. 8. Microhardness values of the castings versus steel mold wall thickness

The cooling rate also has a significant influence on the solidification rate and structure of the alloy, including the size of the secondary phase particles [34, 35]. According to *Chen* He et al. [36], increasing the cooling rate leads to an increase in the mechanical properties of the alloy, a significant decrease in the average grain size, secondary dendrite arm spacing, width, and volume fraction of the eutectic phase. At high cooling rates, a reduction in grain size and an increase in grain boundary length occur. Increasing the cooling rate leads to a decrease in the size of secondary phase particles, which affects the mechanical properties of the castings [37, 38]. *Kong* et al. [39] investigated the influence of cooling rate on the mechanical properties (hardness and tensile strength) of an aluminum alloy. They noted that the solidification of the alloy is completed in the interdendritic space. At high cooling rates, there is a compression of the **α**-*Al* grains before the dendrites are fully formed, and the number of grain boundaries increases. In contrast to resin sand molds, this leads to the formation of more grain boundaries, a reduction in grain size, and a more uniform distribution of eutectic phases along the grain boundaries. The reduction in the amount of eutectic phases indicates an increase in the concentration of dissolved elements in the solid matrix, which contributes to improving the mechanical properties of the alloy.

Figs. 9 and 10 show the measured porosity and theoretical density of the composites as a function of TiO_2 nanoparticle concentration (in wt.%). Fig. 9 demonstrates the effect of TiO_2 nanoparticle concentration on the density of *Al- 7Si* alloy-based composites. As predicted by the rule of mixtures, the theoretical

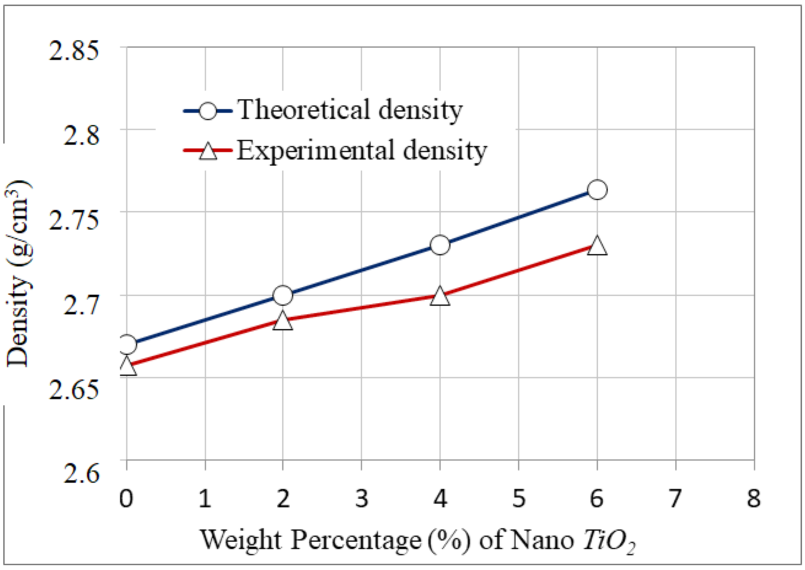


Fig. 9. Theoretical and measured density versus weight percent of reinforcing component

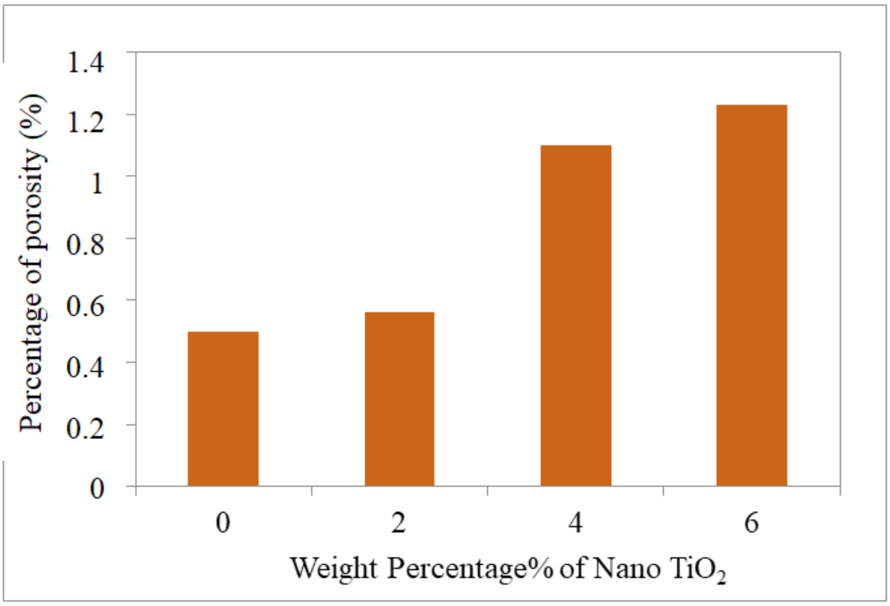


Fig. 10. Percentage of porosity in specimens versus weight percent of reinforcing component

density of the composites increases linearly with increasing concentration of added TiO_2 nanoparticles. Experimentally measured density values are lower than the theoretical ones. Analysis of the density data allowed for the estimation of composite porosity. Fig. 10 illustrates the effect of TiO_2 nanoparticle concentration on the porosity of *Al-7Si* alloy-based composites. It is evident that as the concentration of TiO_2 nanoparticles increases, the porosity of the composites also increases. *MMC* with a TiO_2 nanoparticles content of 2 wt. % had the porosity of 0.56 %, while at 6 wt. %, it increased to 1.23 %. The increase in porosity is associated with the formation of pore nuclei on the surface of the reinforcing particles and its agglomeration at high concentrations [40–42]. *M. Kok* [43] notes that porosity is a common phenomenon in the production of *MMCs* due to the prolonged particle feeding and increased surface area in contact with air, which is due to the small particle size. However, in this study, optimization of process parameters and careful control of wettability improved particles wettability and bonding force between the TiO_2 nanoparticles and the Al-7Si-based alloy, and reduced composite porosity. According to *Mattli* et al. [44], the density of hybrid nanocomposites increases with increasing TiO_2 content. However, because the *Al* matrix (2.7 g/cm³) contained reinforcing additives with higher densities, such as *SiC* (3.21 g/cm³) and TiO_2 (4.23 g/cm³), the density of the hybrid nanocomposites was higher than that of the base aluminum matrix.

Fig. 11 presents the results of the microhardness testing. As the figure shows, the hardness increases with increasing concentration of TiO_2 nanoparticles. Adding 6 wt. % TiO_2 to the *Al- 7Si-based* alloy increased its hardness from 69 HV to 92.5 HV. The higher hardness of the TiO_2 nanoparticles compared to the pure matrix leads to an increase in the hardness of the Al- 7Si-based alloy composite with the addition of these nanoparticles. *Saber* et al. [14] also note that adding nanoparticles to a metal matrix alloy increases the hardness of the nanocomposites. This is because the uniformly distributed nanoparticles act as a reinforcing phase. The *Orowan* mechanism plays a major role in strengthening the composites, especially when the size of the reinforcing components is less than 100 nm [45]. According to this mechanism, the passage of a dislocation line through the ceramic TiO_2 particles leaves a dislocation loop around the non-shearable TiO_2 particles within the matrix. This prevents or decelerates the dislocation motion in the metal matrix alloys. According to *Mahan* et al. [11], adding 5 % TiO_2 increases the *Vickers* hardness by 40 % compared to the original *A2024* alloy. This effect is explained by the solid solution strengthening mechanism, in which TiO_2 particles behave as obstacles to dislocation movement. *Al-Jaafari* [16] also demonstrated that adding 1.5 % TiO_2 nanoparticles to *AA6061* and *AA6082* alloys as reinforcing components increases the *Brinell* hardness number (*BHN*) by 12.1 % for *AA6082* and 32 % for *AA6061*.

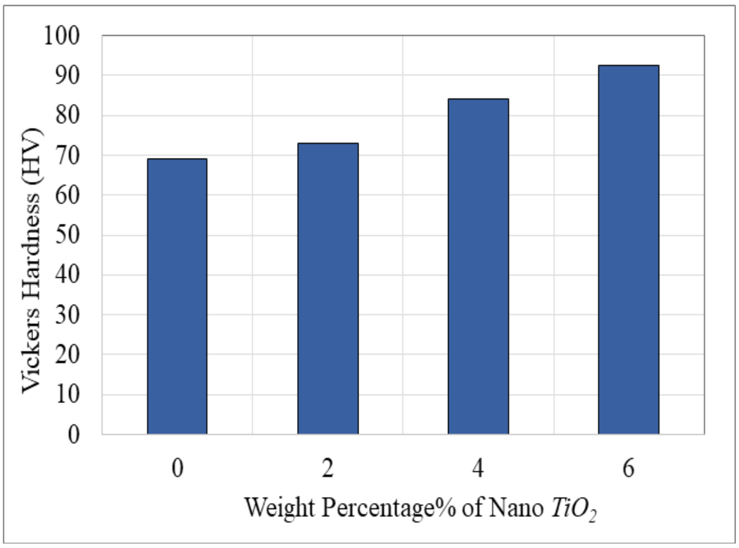


Fig. 11. Effect of TiO_2 wt. % on the hardness of *Al-7Si* matrix composites

The wear test results indicate that the wear rate of the composites increased with increasing test load and decreased with increasing TiO_2 nanoparticle content. As illustrated in Fig. 12, at a load of 10 N and a sliding distance of 350 m, the *Al- 7Si*-based alloy composite with 6 wt. % TiO_2 demonstrated the lowest wear rate (0.017 mm^3/m), while the *Al- 7Si*-based alloy showed the highest wear rate (0.028 mm^3/m). This is explained by the presence of hard particles that increase the overall hardness of the material. A similar trend was observed at a load of 20 N. Fig. 13 demonstrates that the wear rate of the tested samples increased with increasing sliding distance. Thus, the *Al- 7Si*-based alloy (Fig. 12) demonstrated a wear rate of 0.028 mm³/m at a sliding distance of 350 m, while increasing the distance to 700 m (Fig. 13) resulted in a wear rate of 0.039 mm³/m. The same pattern was observed for the fabricated composite materials.

Numerous researchers [4, 5, 7, 8, 15, 46] note that the influence of the applied load increases with the increasing percentage or size of the reinforcing particles. *Walker* et al. [46] explained this phenomenon by the intense abrasive wear of both contacting surfaces, resulting from the use of hard ceramic particles as reinforcement. *Shashi* et al. [15] studied the effect of TiO_2 addition on the wear properties of *AA2014*

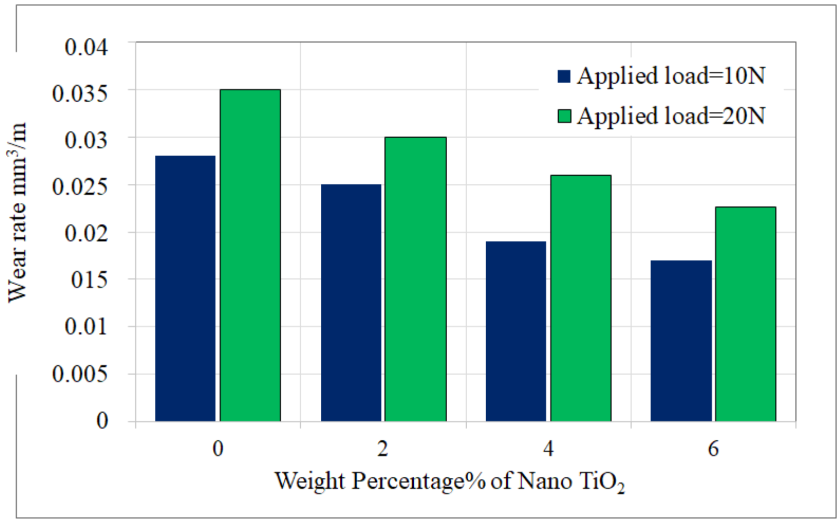


Fig. 12. Effect of *TiO₂* content (%) on the wear rate of *Al- 7Si* matrix composites at a sliding distance of 350 m

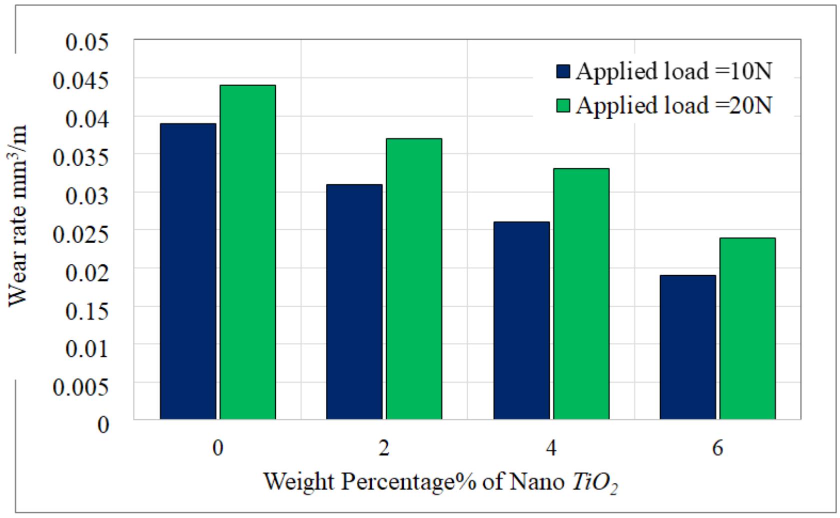


Fig. 13. Effect of *TiO₂* content (%) on the wear rate of *Al- 7Si* matrix composites at a sliding distance of 700 m

and *AA2024* aluminum alloys and discovered an effective method for increasing wear resistance. TiO_2 is a naturally occurring titanium oxide widely used as a pigment in paints, plastics, and cosmetics. When added to aluminum, it increases its durability and hardness by interacting with the metal's crystal lattice. The addition of TiO_2 particles into the aluminum matrix leads to the formation of a composite material with improved wear resistance. The high hardness of the TiO_2 particles makes a significant contribution to increasing the wear resistance of aluminum.

Vinaykumar et al.'s research [8] showed that compared to the *Al6063* alloy, composites based on *Al6063* containing 2, 4, 6, and 8 wt. % TiO_2 demonstrate an increase in wear resistance of approximately 6.25 %, 18.75 %, 37.5 %, and 43.75 %, respectively. *Ganesh Khandoori* [47] and associates investigated the wear behavior of aluminum reinforced with TiO_2 particles (5 %, 10 %, and 15 %) produced by stir casting. The test results unequivocally showed that the mass loss of the samples increases with increasing load, although the wear rate varies. At the same time, the mass loss of the composite decreases with increasing TiO_2 content, which indicates an increase in its wear resistance.

TiO_2 has a *Mohs* hardness of 5.5–6 (out of 10) [48], while aluminum alloys have a *Mohs* hardness of 2.75–3. Thus, the TiO_2 nanoparticles are significantly harder than the *Al- 7Si* matrix. Adding such a hard material to a softer aluminum matrix provides greater wear resistance caused by friction and abrasion.

Dwivedi et al. [15] summarized that the addition of TiO_2 to *AA2014* and *AA2024* aluminum alloys improves its wear resistance. *Kumar* et al. [49] also confirmed that the wear resistance and microhardness of the aluminum matrix increase with increasing TiO_2 content. *Ahamad* et al. [50] explained that the wear resistance of a hybrid composite ($Al\text{-}Al_2O_3\text{-}TiO_2$) increases with an increasing of TiO_2 content, which is related to its lubricating properties.

Conclusion

1. The wall thickness of the casting mold is one of the key parameters determining the solidification rate of the casting. The grains get finer as the cooling rate rises in tandem with the thickness of the mold wall.

2. The microstructure varies with cooling rate during solidification. Slow solidification, characteristic of molds with small wall thicknesses, leads to the formation of a coarse-grained structure. At the same time, a high solidification rate ensures the obtaining of a fine-grained structure with improved mechanical properties.

3. Increasing the mold wall thickness enhances the microhardness of the casting surface by promoting rapid cooling at the casting surface close to the wall.

4. Stir casting was successfully used to fabricate *Al- 7Si* matrix composites reinforced with varying wt.% of TiO_2 nanoparticles. Optimal parameters are found to be as follows: 750 °C pouring temperature, 550 rpm stirring speed, and 5 minute stirring time.

5. Increasing the wt.% of TiO_2 nanoparticles affects the density and porosity of the composites. The theoretical density of the composites increases with the addition of TiO_2 nanoparticles. However, the experimental density values are lower than the theoretical values due to the presence of porosity within the composites.

6. The hardness and wear resistance of the composites are significantly improved by increasing the wt.% of TiO_2 nanoparticles.

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Conflicts of Interest

The authors declare no conflict of interest.