

# Synthesis and Characterization of Al-SiO<sub>2</sub> Composites

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## Abstract

The paper presents studies aimed at producing aluminum matrix composites reinforced with amorphous silica particles. The possibility of producing composites of the Al-SiO<sub>2</sub> (5 wt. %) system using intensive mixing casting and semi-solid metal casting methods has been established. Semi-solid metal casting followed by squeeze casting demonstrated the highest efficiency. The potential of using magnesium as a surface-active additive to remove oxygen from the surface of the dispersed particles and improve the mechanical properties of the composite material during heat treatment has been demonstrated. It has been found that the resulting composites exhibit a uniform distribution of dispersed silica particles throughout the metal volume. Furthermore, they possess hardness, corrosion resistance, and specific gravity values that exceed those of the original aluminum alloy. Composites produced using the developed technique may find application in various transportation engineering fields, as well as in aviation and aerospace industries.

## 1. Introduction

The development of composite materials, consisting of a metal matrix and reinforcing elements distributed within it, is one of the most significant areas of research and development in modern metallurgy and material science [1-19]. In many cases, only composites can meet the demands of new technologies, which are characterized by more demanding operating conditions, such as increased loads, higher speeds, higher temperatures, aggressive environments, and reduced weight [5,7].

Among the various types of composites available, those with an aluminum matrix have gained the most widespread use. This is because aluminum matrix composites exhibit increased strength, reduced specific gravity, and an advantageous combination of several mechanical and operational characteristics [8,9].

Today, there are various technologies available for producing aluminum-matrix composites. The main ones include powder metallurgy, dispersion, molten metal infiltration, and various types of casting techniques. Intensive stir casting is the most accessible and widely used technology for creating composite materials. This process involves introducing strengthening particles into an aluminum melt followed by mechanical or electromagnetic agitation.

However, one of the disadvantages of this technique is the tendency of the particles to agglomerate due to their poor wettability. Several studies have shown that one of the most cost-effective methods for creating aluminum-matrix composites is semi-solid metal casting. This technique involves the metal being in a solid-liquid state between the liquidus and solidus temperatures. This is because in the solid-liquid phase, the aluminum melt has an increased viscosity, which helps with the mixing of dispersed particles.

There are several variations of this process, including thixocasting, rheocasting, and thixomolding [17,18]. To reduce porosity and achieve the desired microstructure of the finished product, injection molding may be necessary. Most studies on aluminum-matrix composite production focus on the effect of various ceramic particles, such as Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, MgO, SiC, and carbon nanotubes, on the properties of the aluminum matrix [16].

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Despite the growing interest in aluminum matrix composites, the overwhelming majority of research focuses on the use of costly reinforcing phases – silicon carbide, aluminum oxide, carbon nanoparticles, as well as silicon dioxide, whose market price varies from \$550 to \$870 per ton depending on the manufacturer. This significantly limits the potential for large-scale industrial application of such composites due to their high production cost.

Conversely, the potential for effectively utilizing cheap and widely available industrial by-products, particularly amorphous microsilica from silicon production waste, as a reinforcing phase has been insufficiently studied [4,5,28–30]. The key issue hindering its application is the extremely poor wettability of  $\text{SiO}_2$  particles by the aluminum melt, leading to their agglomeration, non-uniform distribution within the matrix, and defect formation, which adversely affects the mechanical properties of the final material. Existing methods for producing Al- $\text{SiO}_2$  composites often fail to provide the necessary control over microstructure and properties.

Thus, the present study aims to address this existing gap by developing and optimizing a resource-efficient technology for producing Al- $\text{SiO}_2$  composites using silicon production waste. This technology is designed to overcome the challenge of dispersing particle wettability and to yield a material with a homogeneous microstructure and enhanced performance characteristics.

The aim of the work was to develop a technology for producing composites of the Al- $\text{SiO}_2$  system with a content of reinforcing phase particles up to 5 wt. % using intensive mixing casting and semi-solid metal casting methods, as well as to evaluate the effect of  $\text{SiO}_2$  particles on the microstructure and properties of the obtained composites.

## 2. Research Methods

To conduct laboratory studies on the production of composites using amorphous silica as a base metal, hypoeutectic aluminum-silicon alloy AlSi7 with a following content of major impurities, wt. %: Si – 7, Fe – 0.19, Mg – 0.25, Mn – 0.1, Cu – 0.05, Zn – 0.07, and Ga – 0.001, was used. Amorphous microsilica was obtained from the gas purification system of JSC "Silicon" (Shelekhov, Russian Federation), after which it was enriched using the flotation method [3]. The amorphous microsilica, a by-product from the gas purification system of JSC "Silicon", had a broad initial particle size distribution ranging from 0.1 to 300  $\mu\text{m}$ , with a median particle size (D50) of approximately 45  $\mu\text{m}$ . To obtain a more refined and uniform fraction, the raw microsilica was enriched by flotation. The enriched fraction, with a median particle size (D50) of 60  $\mu\text{m}$ , was then subjected to ultrasonic treatment (using a UZDN-2T ultrasonic disperser) in acetone for 30 minutes at a power of 500 W and a frequency of 22 kHz to deagglomerate the particles and remove surface contaminants. Subsequently, the particles were washed with distilled water and dried at 120°C for 2 hours. Finally, to remove residual moisture and activate the surface, the dried powder was heat-treated at 250°C for 1 hour in a muffle furnace before introducing it into the melt. The average size of the silicon dioxide particles used was 60  $\mu\text{m}$ .

To improve the wetting properties of microsilica particles and prevent agglomeration, before introducing them into the aluminum melt, they were subjected to ultrasonication in acetone and then washed with distilled water. After drying, to reduce thermal shock and promote better melt infiltration microsilica particles were heat treated at a temperature of 200–300°C [1]. To address the wetting issue, in addition to preliminary heat treatment of microsilica, alloying the melt with magnesium was carried out [10]. Magnesium in an amount of 1% by weight was added to the melt in the form of a magnesium ligature MG-90.

Two methods were used to produce aluminum composites:

- i. casting with intensive mechanical mixing followed by gravity casting.
- ii. semi-solid metal casting followed by squeeze casting.

The original aluminum alloy was remelted through continuous casting for objective comparison of the microstructure, physical, and mechanical properties.

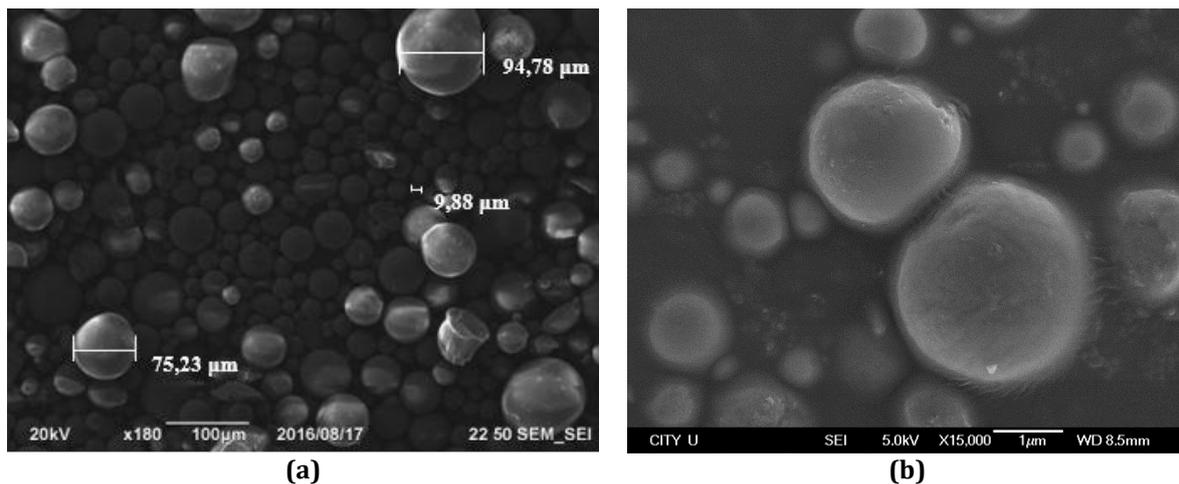
During casting with intensive mixing, microsilica particles were introduced at a temperature of 730 °C. In the semi-solid metal casting process,  $\text{SiO}_2$  was introduced into the melt between the solidus (585 °C) and liquidus (615 °C) temperatures of the matrix alloy, which was in a semi-solid state. The pouring stages were conducted above the liquidus temperature of aluminum alloy (730 °C).  $\text{SiO}_2$  particles heated to 200–300 °C were added to the aluminum melt at a rate of 5 grams per minute. During this process, the molten metal was stirred using a rotary stirrer at 200 revolutions per minute. The final step was the squeeze casting of metal using a 25 T hydraulic press. After that, the ingots were heat-treated at 500 °C for 14 hours followed by quenching in warm water (70 °C) and subsequent precipitation or aging at 165 °C for eight hours. The T6 heat treatment process was applied to both the unreinforced alloy and the composite. This process consisted of solution treatment (525 °C for twelve hours) followed by quenching in warm water and aging at 165 °C.

To analyze the phase composition of samples, X-ray diffraction was used with an XRD-7000 diffractometer manufactured by Shimadzu. Samples were analyzed in the range between 10 and 70 degrees. The microstructure of alloy samples (in secondary and backscattered electron modes) was examined using a JIB-4500 multibeam scanning electron microscope produced by JEOL equipped with an X-Max energy-dispersive detector by Oxford

Instruments (UK). Additionally, to analyze the microstructure of obtained aluminum-silicon alloy samples, metallographic studies were conducted using an Olympus GX-51 optical microscope (Japan). Hardness was measured using a Zwick Brinell hardness tester with a 2.5 mm diameter ball and a force of 62.5 kg. The test was performed at room temperature, and hardness measurements were taken at ten different locations on each sample to obtain an average value and standard deviation. The corrosion properties of initial aluminum and obtained composites were studied using an electrochemical workstation in potentiodynamic mode with a three-electrode cell. In this cell, the initial aluminum-silicon alloy and obtained Al-SiO<sub>2</sub> composite acted as working electrodes. Platinum served as an auxiliary electrode, and calomel acted as a reference electrode. Each potentiodynamic polarization test was repeated three times to ensure reproducibility of the results.

### 3. Results and Discussion

Fig. 1 presents scanning electron microscope (SEM) images of microsilia particles, which exhibit a spherical shape. It is clear that the size of these SiO<sub>2</sub> particles spans a broad range, with smaller particles adhering to the surface of larger particles due to the high surface energy exhibited by the latter (Figure 1b).



**Fig. 1** SEM images of microsilia particles

Fig. 2a presents the microstructure of the initial pre-eutectic AlSi7 alloy, which is characterized by the presence of dendrites formed by a solid solution of silicon in aluminum ( $\alpha$ -Al), as well as eutectic  $\alpha$ -Al+Si structures located in the interdendritic spaces.

The AlSi7 alloy obtained by die casting demonstrates a fine-grained structure with an average grain size of 15  $\mu$ m, as well as the absence of shrinkage defects and gas porosity.

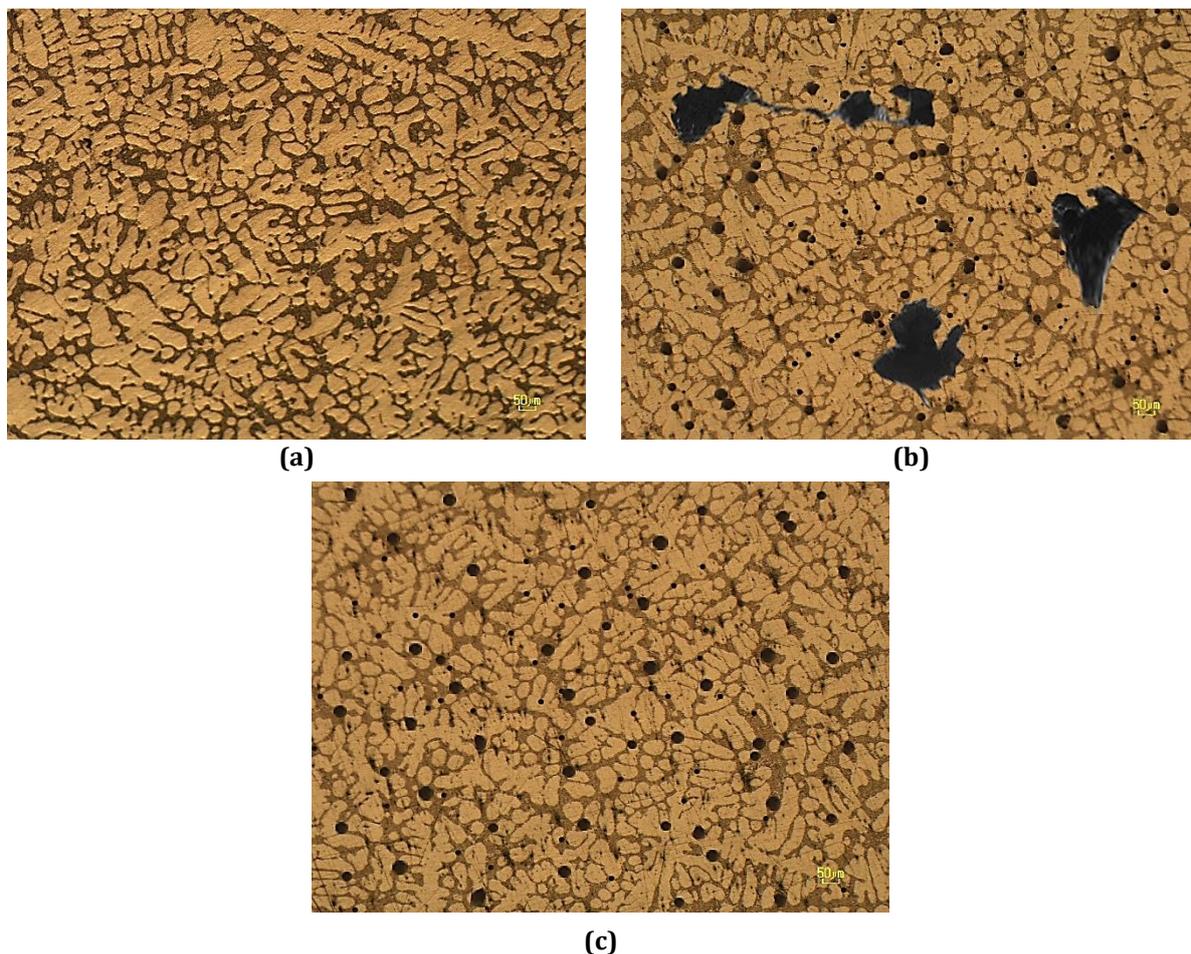
The AlSi7 alloy produced by die casting exhibits a fine-grained microstructure with an average grain size of approximately 15  $\mu$ m, as well as a lack of shrinkage flaws and gas porosity.

The microstructure of the Al-SiO<sub>2</sub> composite material, which was produced through intensive mixing and casting, followed by pouring at a temperature of 720 °C, resulted in the aggregation of microsilia particles and the development of regions with shrinkage porosity (Fig. 2b). This suggests a high level of aggregation of silica particles, which is directly proportional to an increase in casting temperature.

Fig. 2c shows the microstructure of the Al-SiO<sub>2</sub> composite obtained by semi-solid metal casting at 600 °C with intensive mechanical mixing and subsequent squeeze casting to ensure uniform distribution of microsilia particles throughout the composite volume, reduce grain size, and eliminate shrinkage porosity. Due to squeeze casting process, which allows the metal to be processed as a solid-liquid suspension, there is a high degree of distribution of dispersed reinforcing particles and prevention of their agglomeration.

Semi-solid casting was performed in the temperature range between the liquidus and solidus, where the alloy containing primary precipitates of the dendritic phase  $\alpha$ -Al was maintained at a temperature of 590 °C. This contributed to a uniform distribution of SiO<sub>2</sub> particles throughout the volume of the matrix alloy that could not have been achieved through intensive stirring of the molten metal.

To objectively assess the influence of the processing route and the introduction of dispersed SiO<sub>2</sub> particles on the aluminum matrix microstructure, a quantitative grain size analysis was performed according to the standard ASTM E112 method. The analysis results are presented in Table 1.



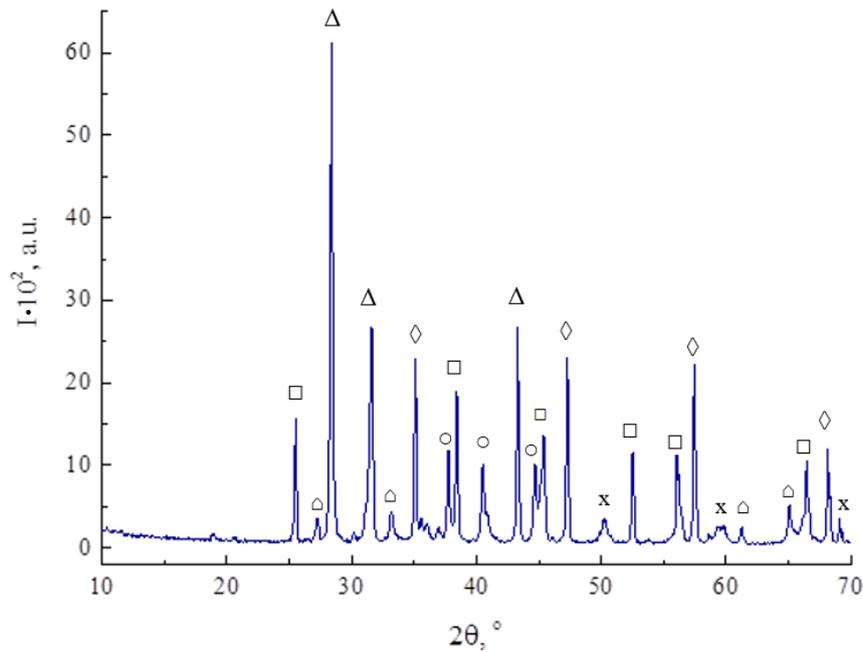
**Fig. 2** Microstructures: (a) of the original aluminum alloy AlSi7; (b) of the composite obtained by casting with intensive mechanical mixing; (c) of the composite obtained by the method of semi-solid metal casting

**Table 1** Quantitative grain size analysis

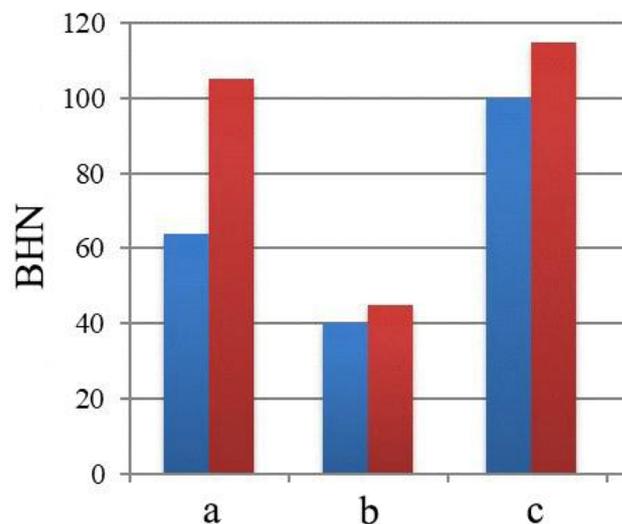
Sample	Processing Route	Average Grain Size, $\mu\text{m}$	Standard Deviation, $\mu\text{m}$
1	Base AlSi7 alloy (cast)	14.8	$\pm 2.1$
2	AlSi7 + 5% SiO <sub>2</sub> (stir casting)	13.5	$\pm 3.5$
3	AlSi7 + 5% SiO <sub>2</sub> (semi-solid casting + squeeze casting)	8.2	$\pm 1.3$

The diffraction pattern of the sample obtained from the composite material contains peaks corresponding to aluminum, silicon, and SiO<sub>2</sub> (Fig. 3). The highest intensity peaks in the X-ray diffraction pattern correspond to metallic aluminum ( $2\theta = 28.7^\circ, 32.4^\circ, 43.5^\circ$ ), crystalline silicon ( $2\theta = 35.1^\circ, 47.4^\circ, 57.5^\circ, 68.4^\circ$ ) as well as silicon dioxide ( $2\theta = 25.5^\circ, 38.1^\circ, 45.3^\circ, 52.7^\circ, 57.3^\circ, \text{ and } 66.5^\circ$ ) and aluminum oxide ( $2\theta = 37.6^\circ, 41.9^\circ, 44.8^\circ$ ). XRD analysis has also revealed the presence of two intermetallic compounds in the composite: MgAl<sub>2</sub>O<sub>4</sub> and Mg<sub>17</sub>Al<sub>12</sub>. These compounds were formed due to the addition of magnesium to the original aluminum-silicon alloy. Furthermore, it has been confirmed that magnesium in the composite enhances the wettability between the aluminum matrix and reinforcing microsilica particles by forming spinel MgAl<sub>2</sub>O<sub>4</sub>, which in turn purifies the structure of the composite from oxides. Additionally, the intermetallic compound Mg<sub>17</sub>Al<sub>12</sub> contributes to improving the mechanical properties of the composite during heat treatment.

The data obtained allowed us to determine that the SiO<sub>2</sub> content in the composite corresponds to the planned value of 5 wt. %. This confirms the feasibility of controlled introduction of the desired number of dispersed particles into the aluminum matrix. Studies have demonstrated that the addition of magnesium during composite production enhances the wettability of the dispersed particles. However, to prevent the formation of the Al<sub>3</sub>Mg<sub>2</sub> intermetallic compound, which reduces the strength of composites, magnesium addition to the aluminum matrix must be limited to 2 wt. %.



**Fig. 3** Diffraction pattern of the Al-SiO<sub>2</sub> composite in the 2θ range of 10–70° (Δ – Al; □ – amorphous SiO<sub>2</sub>; ◇ – Si; ○ – Al<sub>2</sub>O<sub>3</sub>; x – MgAl<sub>2</sub>O<sub>4</sub>; ◻ – Mg<sub>17</sub>Al<sub>12</sub>)



**Fig. 4** Hardness: (a) of the original aluminum alloy AlSi7; (b) of the composite obtained by casting with intensive mechanical mixing; (c) of the composite obtained by the method of semi-solid metal casting

The results of the study on the Brinell hardness of specimens of cast aluminum alloy AlSi7 and composites based on it, produced using two different manufacturing processes, are presented in Fig. 4.

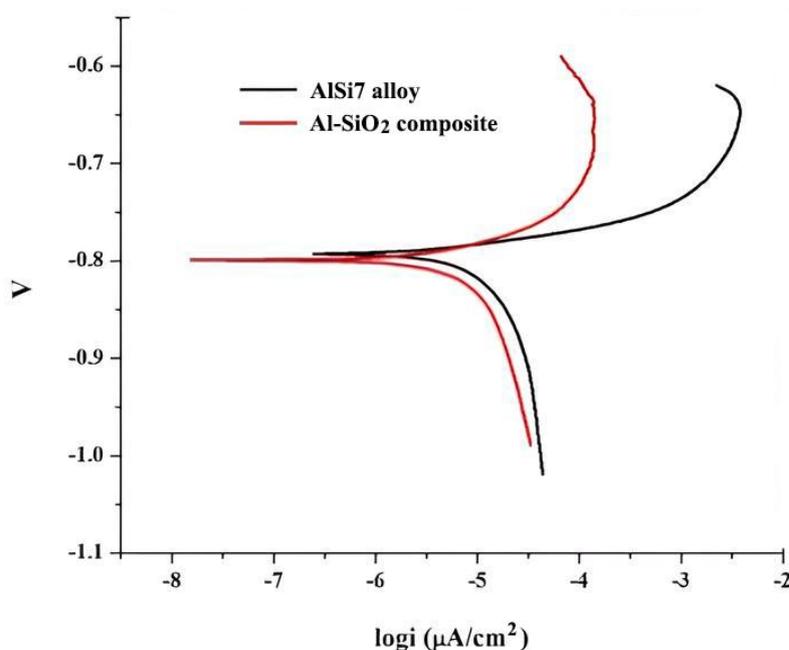
It is evident that the addition of amorphous microsilia particles into cast aluminum-silicon alloy, if uniformly dispersed, can increase its hardness. SiO<sub>2</sub> particles in the aluminum matrix act as crystallization nuclei and promote grain refinement. The difference in thermal expansion coefficients between microsilia and the alloy matrix leads to mismatches in deformations at the interface, acting as barriers to dislocation motion. The hardness of composites depends directly on the method of production and the degree of particle distribution. The composite produced through casting with intensive mixing, even after heat treatment, demonstrates a significant decrease in hardness compared to base AlSi7 alloy. This is attributed to the high degree of microsilia particle agglomeration and, consequently, shrinkage porosity formation. The composite manufactured through semi-solid metal casting followed by squeeze casting exhibits a hardness value that exceeds that of the original alloy. This is

due to better microsilica distribution in the metal volume, as well as a reduction in grain size resulting from crystallization under pressure and the presence of numerous crystallization centers.

The increased compressive strength of the composite material is due to the resistance of microsilica particles to deformation stress in the matrix. Plastic flow in the matrix is limited due to the closer spacing between the microsilica particles, which prevents the buildup of dislocations and, as a result, leads to strain hardening in the composite.

In addition to hardness, the compressive strength of the composites was evaluated. The original AlSi7 alloy exhibited a compressive strength of  $510 \pm 15$  MPa. The composite produced by intensive stir casting showed a reduced strength of  $435 \pm 20$  MPa, which correlates with the observed agglomeration of silica particles and increased porosity. In contrast, the composite manufactured via semi-solid metal casting followed by squeeze casting demonstrated a significantly higher compressive strength of  $656 \pm 18$  MPa. This 28% increase compared to the base alloy is attributed to the uniform distribution of  $\text{SiO}_2$  particles, grain refinement, and the elimination of shrinkage defects due to the applied pressure during solidification. The fracture strain of the optimal composite was measured at 4.2%, indicating a good combination of strength and limited ductility typical for particle-reinforced composites.

In addition to its high strength, the composite produced by semi-solid metal casting demonstrates improved corrosion resistance compared to the original AlSi7 alloy (Fig. 5).



**Fig. 5** Curves of potentiodynamic polarization of the initial aluminum alloy AlSi7 and the composite obtained by the method of semi-solid metal casting

This is due to the formation of interphasic products in the composite, which is obtained through the reaction between aluminum, magnesium, and microsilica. These compounds, such as  $\text{MgAl}_2\text{O}_4$ ,  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ , act as protective oxide layers on the surface of the material, reducing the tendency for corrosive effects.

Investigation of the density of the samples revealed that the value for the composite produced by semi-solid metal casting was  $2.47 \text{ g/cm}^3$ , while the original alloy had a density of  $2.64 \text{ g/cm}^3$ .

To contextualize the significance of the improvements achieved, the mechanical and corrosion properties of the developed composite were compared with those reported in recent literature for similar Al- $\text{SiO}_2$  composites. As summarized in Table X, the composite produced via semi-solid casting in this work demonstrates a competitive advantage.

The achieved hardness of 100 HB represents a significant improvement over the values reported by Malek & Abderraouf [4] for Al- $\text{SiO}_2$  composites fabricated via stir casting (50-60 HB) and is comparable to the high-end values achieved by Sree Manu et al. [8] using a specialized compocasting method (80-90 HB). This indicates that our semi-solid casting route combined with squeeze casting is highly effective for producing high-strength composites.

More notably, the enhancement in corrosion resistance is substantial. The observed positive shift in corrosion potential ( $\sim 50$  mV) and the reduction in corrosion current density (by a factor of  $\sim 1.5$ ) outperform the results typically achieved by simple stir-cast composites, where poor particle distribution often creates localized galvanic

cells [6]. This superior performance is attributed to the uniform distribution of  $\text{SiO}_2$  particles and the formation of a stable, protective layer of  $\text{MgAl}_2\text{O}_4$  spinel and  $\text{SiO}_2$ , which mitigates pitting corrosion—a common issue for aluminum matrix composites in chloride-containing environments. While direct quantitative comparisons of corrosion rates are complex due to differing test conditions, the relative improvement observed in this study is among the highest reported for Al-based composites reinforced with silica-based particles [3].

Therefore, the developed manufacturing route not only successfully incorporates low-cost amorphous silica waste but also yields a composite with properties that meet or exceed those of composites produced using more conventional and expensive reinforcements or methods.

**Table 2** *The comparison of developed composite mechanical and corrosion properties with data reported in different research aimed to obtain Al-SiO<sub>2</sub> composites*

Reference	Reinforcement	Processing Method	Hardness (HB)	Corrosion Resistance Notes
Current research	5 wt.% amorphous $\text{SiO}_2$	Semi-solid + Squeeze Casting	100	~ 1.5x improvement in corrosion current density
Malek & Abderraouf [4]	$\text{SiO}_2$	Stir Casting	50-60	Moderate improvement
Sree Manu et al. [8]	Microsilica	Modified Compocasting	80-90	Not reported
Pattnayak et al. [3]	Rice Husk Silica	Stir Casting	~70	Slight improvement

#### 4. Conclusion

The studies conducted in this work have made it possible to determine the feasibility of producing Al-SiO<sub>2</sub> composite systems using intensive stirring casting and semi-solid metal casting techniques. The semi-solid metal casting technique, followed by squeeze casting, demonstrated the highest efficiency. It was possible to produce a composite with a reinforcement phase particle content of 5 weight percent and their uniform distribution throughout the matrix alloy. The microstructure of the obtained composite shows a reduction in grain size and the absence of shrinkage porosity. The investigation into the effect of magnesium on the wetting of dispersed microsilica particles revealed its positive impact on the removal of oxygen from the surface of the dispersed particles, preventing additional oxide formation through the formation of the intermetallic compound  $\text{MgAl}_2\text{O}_4$ , as well as improving the mechanical properties of the composite material during heat treatment due to the formation of  $\text{Mg}_{17}\text{Al}_{12}$ . It has been found that the resulting composite exhibits a 50 % increase in hardness (to 100 HB) and a 6 % reduction in density (to 2.47 g/cm<sup>3</sup>) compared to the original AlSi7 alloy (70 HB, 2.64 g/cm<sup>3</sup>). Potentiodynamic studies also revealed a positive shift in corrosion potential of ~50 mV and an approximately 1.5-fold decrease in corrosion current density, indicating a significant improvement in corrosion resistance. Therefore, it can be inferred that the presented approach for producing Al-SiO<sub>2</sub> systems is effective, as it enables the creation of composites with superior strength, corrosion resistance, and low porosity.

It was discovered that the resulting composite exhibits hardness (656 MPa) and corrosion resistance that surpass similar characteristics of the initial alloy. Therefore, it can be inferred that the presented approach for producing Al-SiO<sub>2</sub> systems using intensive mixing and semi-solid metal casting processes is effective, as it enables the creation of composites with superior strength, corrosion resistance, and low porosity.

Composites produced using the developed technique possess a combination of properties (specific strength, corrosion resistance) that makes them promising candidates for applications in automotive and other transportation engineering fields. However, to firmly assess their potential for use in aviation and aerospace industries, further research is necessary, encompassing evaluations of tensile strength, fracture toughness, and resistance to cyclic loading (fatigue endurance).

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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:** Kuz'min M.P., Kuz'mina M.Yu.; **data collection:** Kuz'mina A.S.; **analysis and interpretation of results:** Kuz'min M.P., Kuz'mina M.Yu. Kuz'mina A.S.; **draft manuscript preparation:** Kuz'min M.P., Kuz'mina M.Yu. Kuz'mina A.S. All authors reviewed the results and approved the final version of the manuscript.

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