

Influence of Al₂O₃ Nano-dispersions on Mechanical and Wear Resistance Properties of Semisolid Cast A356 Al Alloy

Ahmed Y. Shash, Amer E. Amer and Moataz El-Saeed

Abstract The present investigation studies the prospects of using nanoparticles as reinforcement ceramic powders to gain improved performance of A356 Al cast alloy. Alumina nano-powder of 40 nm size was stirred into the A356 matrix with different fraction ratios ranging from (0, 1, 2 and 4 wt%) in a mushy zone (600 °C) using a constant stirring time for one minute. To evaluate the results, the alloys were further characterized by various tribological and mechanical characterization methods. The results showed higher strength values with improved ductility when compared to the monolithic alloy under the same casting conditions. Also, the wear resistance has been positively enhanced as the amount of the Al₂O₃ nano-particles addition increases from 1 to 4 wt% leading to a decrease in the weight loss ranging from 5.5 to 4.0 mg, respectively. The Scanning Electron Microscopy of the fracture surface and the wear surface revealed the presence of nanoparticles at the interdendritic space of the fracture surface and was confirmed with an EDX analysis of these particles.

Keywords Nano-metal matrix composites • Al₂O₃ nano-powders • Wear resistance • Mushy zone • Mechanical stirring

A.Y. Shash (✉)

Mechanical Design and Production Department, Faculty of Engineering,
Cairo University, Giza, Egypt
e-mail: ahmed.shash@cu.edu.eg

A.E. Amer

Mechanical Design and Production Engineering Department, Beni Suef University,
Beni Suef, Egypt
e-mail: aeid958@yahoo.com

M. El-Saeed

Department of Mechanical Engineering, Akhbar El-Youm Academy, 6 of October City,
Giza, Egypt
e-mail: mo3taz.elsaeed@gmail.com

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1 Introduction

History is often marked by the materials and technologies that reflect human capabilities and understanding. Many time scales begins with the stone age, which led to the Bronze, Iron, Steel, Aluminum and Alloy age, as improvements in refining and smelting took place and science made it possible to move towards finding more advanced materials. Composite structures have shown universal savings of at least 20 % over metal counterparts and a lower operational and maintenance cost [1]. Aluminum based alloys and metal matrix composites (MMCs) exhibit attractive tribological and mechanical properties such as a high specific modulus, good strength, long fatigue life, superior wear resistance and improved thermal stability, which allow these alloys to have numerous applications in the aerospace, automobile and military industries. As the data on the service life of composite structures is becoming available, it can be safely said that they are durable, maintain dimensional integrity, resist fatigue loading and are easily maintainable and repairable. Composites will continue to find new applications, but the large scale growth in the marketplace for these materials will require less costly processing methods and the prospect of recycling [2] will have to be solved [3].

Composite materials are emerging chiefly in response to unprecedented demands from technology due to rapidly advancing activities in aircrafts, aerospace and automotive industries. These materials have low specific gravity that makes their properties particularly superior in strength and modulus to many traditional engineering materials such as metals. As a result of intensive studies into the fundamental nature of materials and better understanding of their structure property relationship, it has become possible to develop new composite materials with improved physical and mechanical properties [4–6]. These new materials include high performance composites such as polymer matrix composites, ceramic matrix composites and metal matrix composites etc. Continuous advancements have led to the use of composite materials in more and more diverse applications. The importance of composites as engineering materials is reflected by the fact that out of over 1600 engineering materials available in the market today more than 200 are composites [6].

For most applications, a homogeneous distribution of the particles is desirable in order to maximize the mechanical properties [5]. In order to achieve a good homogeneous distribution of a particle in the matrix, the process parameters related with the stir casting method must be studied [4, 5]. So that it is essential to study the influence of stirring speed and stirring time on the distribution of particles in MMC.

In the Prabu [7] study, the stirring speeds were set at 500, 600 and 700 rpm and the stirring times were set at 5, 10 and 15 min for this study.

Many researchers have claimed enhanced properties for these produced composites relative to those produced by reinforcing with micro-particles.

Therefore the aim of this research work is to improve the mechanical and tribological properties of the A356 aluminum alloy using ceramics Al_2O_3 nano particles.

2 Experimental Procedures

The experimental work carried out through this scientific study consists of the following three stages:

- (a) Production of new NMMC alloys.
- (b) Identification of the mechanical and wear resistance properties.
- (c) Characterization of the new material.

2.1 Materials Produced

The hypoeutectic alloy A356 was used as a base metal for the produced material having the chemical composition shown in Table 1. The material used for reinforcement was 1, 2, and 4 % by weight Al₂O₃ ceramic nano-particles with constant particle size of 40 nm, the description of which is given in Table 2.

2.2 Equipments Used

2.2.1 Melting Furnace

An electric resistance furnace was designed and constructed for approaching this research work for preparing the NMMCs. It consists of a lift out ceramic crucible of max. 2 kg, a heating system, and is connected to a stirring mechanism with a

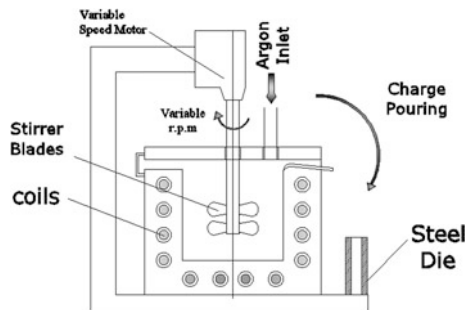
Table 1 Chemical composition (in wt%) of A356 cast Al-Si

Alloy	Chemical composition (wt%)							
	Al	Si	Mg	Fe	Cu	Pb	Zn	Mn
A356	Bal.	7.44	0.3	0.27	0.02	0.022	0.01	Nil

Table 2 Properties of Al₂O₃ reinforcement powders

Reinforcement	γ -Al ₂ O ₃
Density (solid) (g/cm ³)	3.95
Crystal structure	FCC
Appearance	White solid
Young's modulus (GPa)	380
Average size (nm)	40
Melting point	2054 °C

Fig. 1 Schematic apparatus used for preparing the NMMCs



3000 rpm max. rotating speed motor and adjustable height with a control unit of up to 1200 °C connected to a thermocouple for controlling the stirring temperature Fig. 1.

2.2.2 Metallic Mould

The melt was poured into a mild steel mould, in which the casted samples were in a 24 mm diameter as shown in Fig. 2.

2.3 Melting Methodology and Approach

A charge of 0.5 kg of the A356 alloy was introduced to the crucible and heated up to the melting temperature (640 °C). The melt was degassed and shielded with argon before pouring after reaching the liquid state to prevent oxidation of the molten metal. The melt was subsequently brought down to the semi-solid state by around 605 °C and hence the Al_2O_3 nano-powders were preheated to 700 °C and then added to the melt simultaneously with mechanical stirring for 1 min at 1500 rpm. The fabrication conditions of the composites prepared in this investigation are

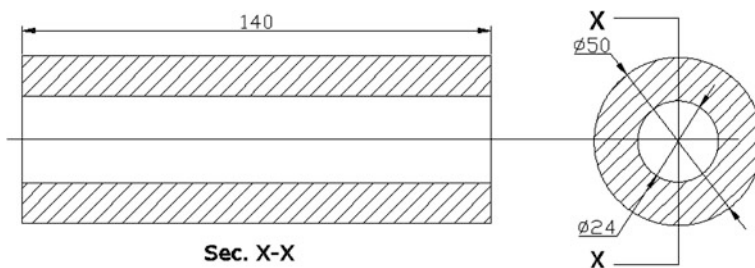


Fig. 2 Schematic drawing for the mould used

Table 3 List of produced alloys and fabrication conditions

Melt no.	Additions	Stirring (rpm)	Pouring temp. (semi-solid)
Melt 1	A356	1500	605 °C
Melt 2	A356 + 1 % Al ₂ O ₃	1500	605 °C
Melt 3	A356 + 2 % Al ₂ O ₃	1500	605 °C
Melt 4	A356 + 4 % Al ₂ O ₃	1500	605 °C

summarized in Table 3. Cast samples were poured into the prepared mould without additions and with additions of the different investigated Al₂O₃ percentages.

2.4 Mechanical Properties

Mechanical properties, mainly tensile strength, ductility, hardness and wear resistance, were determined in the as-cast conditions for the investigated NMMC samples.

2.4.1 Tensile Test

The tensile tests were conducted on round tension test specimens of diameter 5.02 mm and gage length 25.2 mm using a universal testing machine according to DIN 50125. The elongation percentage and ultimate tensile strength were calculated. The results were based on the average of three samples taken from each melt.

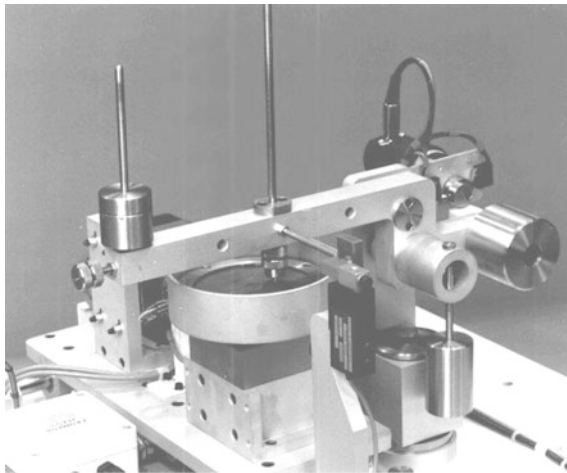
2.4.2 Hardness Test

The hardness tests were conducted on Rockwell hardness testing machines in the Faculty of Engineering, Cairo University, using a ($\frac{1}{16}$)" diameter hardened steel ball and a 62.5 kg applied load. The reported results are the average of three readings for each case.

2.4.3 Wear Test

A PLINT TE 79 Multi Axis Tribometer Machine was used for measuring friction force; friction coefficient and wear rate for NMMC manufactured materials, as illustrated in Fig. 3, in which a standard specimen with a diameter of 8 and 20 mm length as a computerized pin on disc machine used for friction and wear testing of materials is loaded vertically downwards onto the horizontal disc.

Fig. 3 PLINT TE 79 multi axis tribometer machine



2.5 Material Characterization

2.5.1 Microstructural Evolution

Representative sections from the cast samples were cut into 3 pieces: the 1st from the top, the 2nd from the middle and the 3rd from the bottom. Samples were wet grounded on a rotating disc using silicon carbide abrasive discs of increasing fineness (120, 180, 220, 320, 400, 600, 800, 1000 and 1200 grit). Then they were polished using 10 μm alumina paste.

2.5.2 Optical Microscopy (OM)

The microstructure examination was carried out using an OLYMPUS DP12 optical metallurgical microscope, equipped with a high resolution digital camera for the investigation of the microstructure.

2.5.3 Scanning Electron Microscope (SEM)

The surface topography and the fracture characteristics were studied using SEM to understand the fracture mechanism and also to detect the favorable sites for particle incorporation by using a JSM-5410 Scanning Electron.

The JSM-5410 scanning electron microscope is a high-performance multipurpose SEM with a high-resolution of 3.5 nm, and EDXS (energy dispersive X-ray spectrometer). Its automated features included Auto Focus/Auto Stigmator, and Automatic Contrast and Brightness. The EDS makes the JSM-5410 expandable from morphological observations to multi-purpose high-resolution elemental analysis.

3 Results and Discussions

3.1 Mechanical Properties of the NMMC

Table 4 illustrates the mechanical properties (tensile strength, elongation in %, and hardness) of the produced castings with reinforced Al₂O₃ nanopowder.

As can be seen from Fig. 4, as the wt% fraction of Al₂O₃ nanopowder increases the UTS increases reaching 195 MPa, until a value of 2 wt% of Al₂O₃. Beyond this weight fraction, the UTS decreases as the wt% increases.

As shown in Fig. 5, increasing the weight fraction of Al₂O₃ has no visible effect on ductility until reaching 1 wt%, then with increasing the wt% beyond 1 wt% the ductility increases. The ductility of NMMC increases by about 40 % at 2 wt% of Al₂O₃ nanopowder. At 4 wt% fraction, the ductility reaches to its minimum values; due to the agglomeration of the dispersed particles in the NMMC, while its hardness at this weight fraction increases by about 30 % as shown in Fig. 6. The presence of the ceramic phase increases the hardness of the alloys and hence, reduces the ductility of the composites in comparison with the matrix alloy.

Substantial increases in strength, along with good ductility, have been observed in a number of alloys with multiphase nanoscale microstructures.

Table 4 Mechanical properties (tensile strength, elongation in % and hardness) NMMC using Al₂O₃ Nanopowder

Melt no.	Additions	UTS (MPa)	Elongation %	Hardness RB
Melt 1	A356	155	5	57
Melt 2	A356 + 1 % Al ₂ O ₃	170	5	61
Melt 3	A356 + 2 % Al ₂ O ₃	195	6.8	72
Melt 4	A356 + 4 % Al ₂ O ₃	163	4.2	73

Fig. 4 The effect of wt% fraction of Al₂O₃ nanopowder on the ultimate tensile strength of MMC at 1500 rpm stirring speed at a semi-solid-state (600 °C)

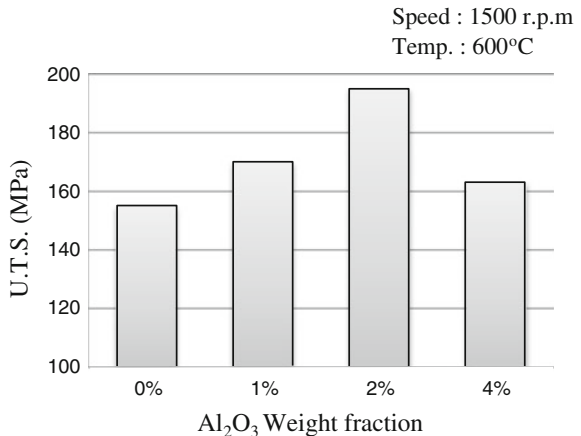


Fig. 5 The effect of wt% fraction of Al_2O_3 nanopowder on elongation % of MMC at 1500 rpm stirring speed when in a semi-solid state (600°C)

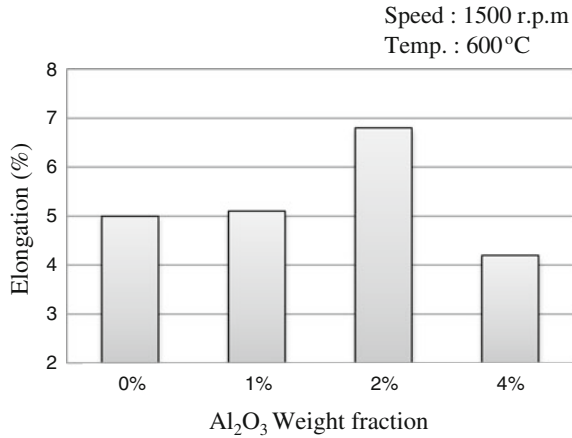
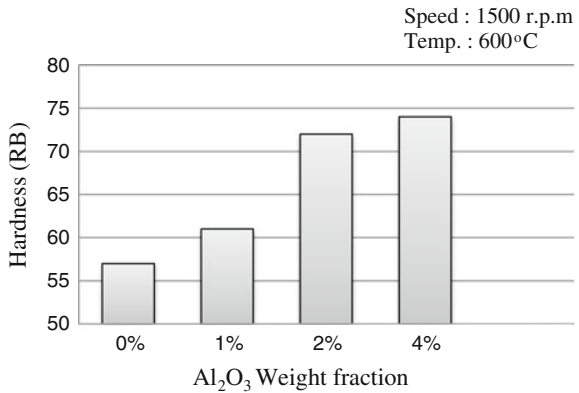


Fig. 6 The effect of wt% fraction of Al_2O_3 nanopowder on the hardness of MMC at 1500 rpm stirring speed when in a semi-solid state (600°C)



These properties originate from the fine distribution of globular particles in an A356 matrix on a nanometer scale, where the globular particles act as strength bearing components, while the A356 matrix supplies ductility. The existence of a crystalline approximant phase at the interface between the particles and the FCC Al matrix improves interfacial bonding between the different phases, thus important for the combination of high strength and good ductility without failure at the interface [8].

The wear tests were then performed with the following parameters: velocity = 0.8 m/s, time = 1200 s and load = 10 N.

The average wear results of A356 samples reinforced with 0, 1, 2 and 4 wt% Al_2O_3 nanopowder are shown in Table 5.

As was expected from the results shown in Table 5 that the wear resistance increases as the weight percentage of the reinforced nano particles increases.

Table 5 The average wear results of A356 samples reinforced with 0, 1, 2 and 4 wt% Al₂O₃ nanopowder

Sample no.	Additions	Weight loss mg	Friction coefficient
1	A356	3.9	0.4
2	A356 + 1 % Al ₂ O ₃	5.5	0.430
3	A356 + 2 % Al ₂ O ₃	4.5	0.385
4	A356 + 4 % Al ₂ O ₃	4.0	0.361

The wear resistance results were evidently confirmed by increasing the hardness and decreasing the friction coefficient values as the wt% of the nano dispersions increases.

3.2 Microstructure Evolution

Figure 7a, b shows the optical microstructure of the base matrix A356 alloys reinforced with 2 wt% fraction of Al₂O₃ nano-powder. The microstructures of the two castings show that the phases are uniformly distributed. It also clearly show a morphological change in the microstructures. In the base matrix sample, the microstructure is dendritic whereas in the other rheocast samples, the primary dendrites are fragmented due to mechanical stirring.

As the structure contains good amount of eutectic phases it should give a range of mechanical properties when mechanical stirring processed [9]. This was clear in the mechanical properties of alloys reinforced with nano-powder using mechanical stirring.

The SEM illustrated in Fig. 8 shows a typical fracture surface for a specimen reinforced with a 2 wt% fraction of Al₂O₃ using the best conditions of stirring speed

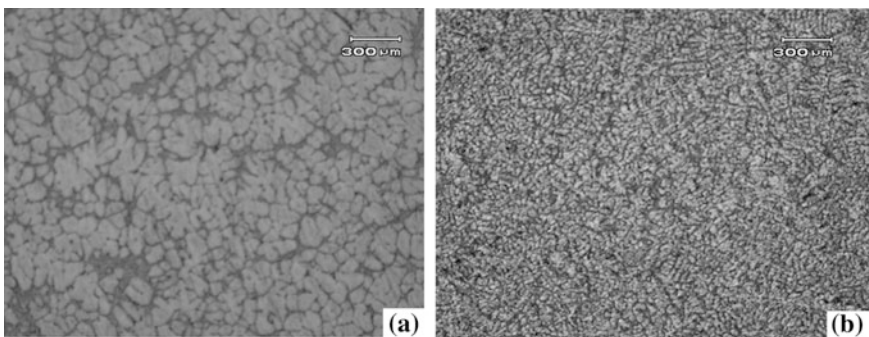
**Fig. 7** The optical microstructure of **a** base matrix A356 alloys. **b** reinforced with 2 wt% fraction of Al₂O₃ nanopowder

Fig. 8 The SEM of the fracture surface specimen reinforced with 2 wt% fraction of Al_2O_3 nano-powder

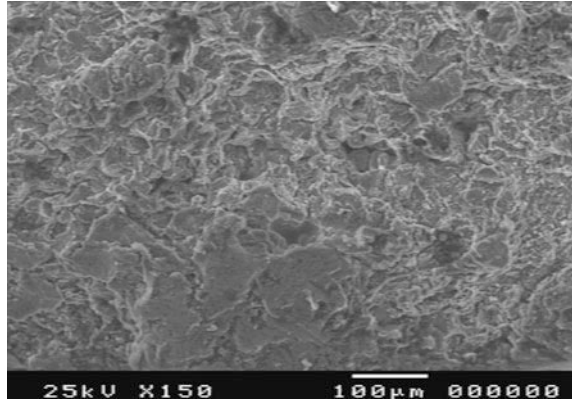
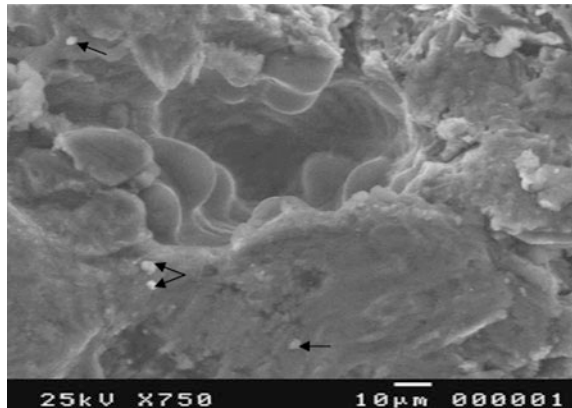


Fig. 9 SEM fracture surface containing agglomerated particles



and temperature. Fracture surface shows mixture of dendrites and globular structures. Figure 9 represents the fracture surface containing agglomerated particles.

The agglomeration could happen by the particles reinforced inside the matrix during the melting stage. Moreover, these agglomerated particles were not homogeneously distributed inside the matrix. Al_2O_3 agglomerated particles have size of about $3\ \mu\text{m}$ attached in the interdendritic space and in matrix. The EDX analysis shown in Fig. 10, has evidently confirmed that these are Al_2O_3 particles though a strong reflection from the matrix was inevitable. The specified analysis of the EDX and the percentage of O, Al and Si are illustrated in Table 6. It is clear that the high percentage of the Oxygen and Aluminum, confirms the presence of Al_2O_3 nano-powders in the matrix.

The samples subjected to wear tests have been examined using SEM. Figure 11a, b clearly show that the Al_2O_3 nano-particles distributed on the surface of samples with 1 and 4 wt% Al_2O_3 addition subjected to wear, respectively.

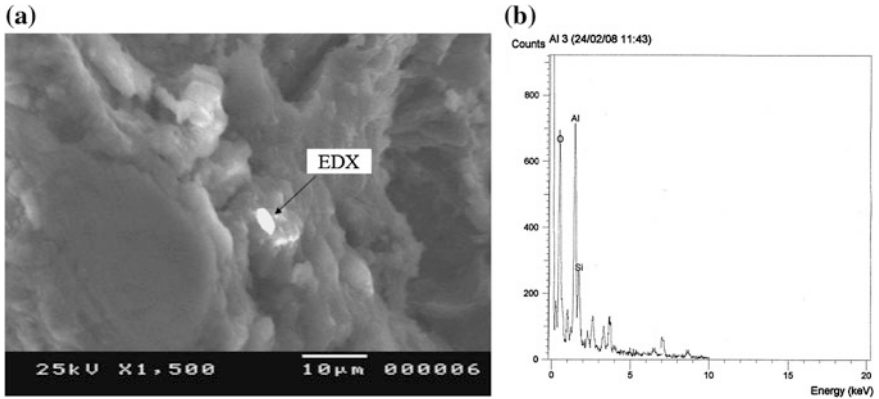


Fig. 10 a SEM containing Al₂O₃ agglomerated particles, b EDX of Al₂O₃ agglomerated particles

Table 6 Specified analysis of the EDX of Al₂O₃ agglomerated particles

Label	Range (keV)	Gross	Net	Total %
O Ka	0.407–0.668	5459	3149	38.1
Al Ka	1.327–1.628	5942	4222	51.1
Si Ka	1.648–1.888	2347	891	10.8

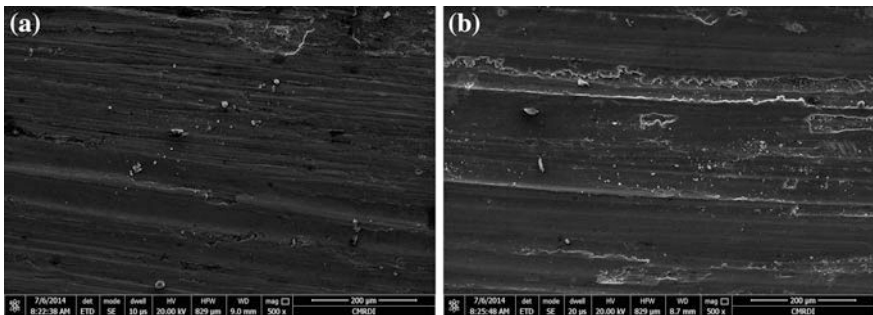


Fig. 11 SEM for wear surface of a 1 wt% Al₂O₃ and b 2 wt% Al₂O₃ nano-particles

4 Conclusion

The nano-composites manufactured using the semi-solid route exhibited better mechanical properties when compared with those prepared using the liquid metallurgy route. Also, a high mixing speed is required in order to obtain good distribution of the particles reinforced and introduce it inside the matrix as introducing the reinforced Al₂O₃ particles to the A356 matrix in the semi solid state is difficult

due to the higher density of the matrix at this state, in which the alloys stirred with 1500 rpm exhibits the best tensile strength and elongation.

The A356 matrix alloy reinforced with 2 wt% fraction of Al_2O_3 nano-powder has the best mechanical properties at conditions of 1500 rpm stirring speed at semi solid state temperature 600 °C. The wear resistance increases as the weight percentage of the reinforced nano-particles increases. The wear resistance results were evidently confirmed by increasing the hardness and decreasing the friction coefficient values as the wt% of the nano-dispersions increases.

In the base matrix sample without stirring, the microstructure is dendritic whereas in the other rheocast samples, the primary dendrites are fragmented due to mechanical stirring which explains the improvement in the mechanical properties. Analysis using both scanning electron microscope (SEM) and high magnification shows evidence for the possibility of incorporating and entrapping nano-sized particles within the interdendritic interface developing during the solidification of the dispersed alloys.

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