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A Novel, Simple and Cost Effective Al A356/Al₂O₃ Nano-composite Manufacturing Route with Uniform Distribution of Nanoparticles

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The present study aims at developing A356/Al₂O₃(np) nano-composite with a novel and cost effective method for attaining homogeneous dispersion of Al₂O₃ nanoparticles in the Al matrix. In the current research, Al micro powders distributed on aluminum foil and then Al₂O₃ nanoparticles ultra-sounded in ethanol and sprayed on Al substrate. Subsequently, the sandwich foil was added to molten A356 during stirring. The nano-composite was characterized through scanning electron microscopy (SEM) for microstructural evaluations. Also, mechanical properties of A356/Al₂O₃(np) nano-composite was measured through compression and hardness tests. By this novel method, an Al based (A356) nano-composite with 2 wt. % Al₂O₃ nanoparticles was obtained which shows 26.79, 95.6 and 43 % enhancement in yield stress, compression strength and hardness, respectively, as compared with the pure alloy processed under the same condition.

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1. INTRODUCTION

A356 is a hypoeutectic Al-Si alloy with excellent casting and machining characteristics. Typically, this alloy is used in castings for aircraft parts, pump housings, impellers, high velocity blowers and structural castings where high strength is require. Preliminary investigations show that introducing nano ceramic particles (such as Al₂O₃, SiC, B₄C, SiO₂) to A356 has a beneficial effect on optimizing strength–ductility relationship in these alloys [1]. Generally, metal matrix nano-composites (MMNCs) exhibit more outstanding properties over metal matrix composites (MMCs) and are assumed to overcome the shortcoming of MMCs such as poor ductility, low fracture toughness and machinability [2].

It has been reported that with a small fraction of nano-sized reinforcements, MMNCs could obtain comparable or even far superior mechanical properties than MMCs. Based on the state of matrix in which MMNCs are fabricated, processing routes for MMNCs

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are categorized into three types: 1-solid state processing that include powder metallurgy, high-energy milling [3-8], severe plastic deformation [9, 10], 2- liquid state processing that include stirring [11-17], infiltration [18-21], rapid solidification [22-24], as well as some in-situ fabrication such as liquid-gas bubbling [25, 26] and 3liquid-solid processing such as compo-casting and semisolid forming [27]. Liquid state processing such as stirring is usually energy-efficient and cost-effective. Moreover, it can produce bulk composites in large quantities. Wide selection of materials, better matrixparticle bonding and easier control of matrix structure are other important advantages of this route. In spite of several advantages of reinforcing ceramic nanoparticles, the fabrication of MMNCs using liquid state processing remains a big challenge for materials scientists. To obtain the expected property, nanoparticles must be effectively distributed in metal matrix.

Due to the small size and large specific surface area (surface to volume ratio) of nanoparticles that cause poor wetting, it is extremely difficult to distribute nanoparticles uniformly in metal matrix [28], especially in the case of alumina ceramic in the molten aluminum.

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TABLE 1. Chemical composition (wt.%) of commercial A356 aluminum alloy measured by quantometer (Emission Spectrometer) and standard composition.

	Si	Fe	Cu	Mn	Mg	Zn	Ti
bought Ingot	6.8	0.19	0.09	0.01	0.35	0.02	0.07
Stansard	6.5-7.5	< 0.20	< 0.20	< 0.10	0.25-0.45	< 0.10	< 0.20

Al₂O₃ particles with high specific stiffness, superior high temperature and excellent mechanical properties are used as inert ceramic reinforcement phases in the AMCs. The poor wetting is because of the presence of a film oxide at the surface of the aluminum. The wettability is a complex phenomenon that depends on the factors such as geometry of interface, process temperature, soaking time, and it determines the quality of bonding among the systems [29]. One successful route to overcome this challenge is utilizing ultrasonic cavitation in stir process. However, even though the ultrasonic cavitation process has been shown to be effective, it is difficult to utilize this technology for industrial applications in that the volume of castings are limited to the power of the ultrasonic source [2].

According to literature survey done by authors, recently, in order to overcome agglomeration of nanoparticles in conventional stir casting and better distribution of the reinforcement particles throughout the matrix, various techniques have been introduced that include reactive wetting and stir mixing [30], stir and compo-casting processes [31], combination of mechanical mixing and hot extrusion [28-32], combination of semisolid-state mechanical mixing and liquid-state ultrasonic processing [28] and preparation of nano-composite master powders using ball milling and incorporating them utilizing vortex method [33].

In the current research, a novel and simple method is designed for the mass production of $A356/Al_2O_3$ (np) nano-composite. Nano sized Al_2O_3 particle reinforced aluminum matrix composites were fabricated using combination of ultrasonic, spraying and conventional stir casting technique that assure the uniform dispersion of the nanoparticles.

2. MATERIALS AND METHODS

In the current work, AMS A356.0 aluminum alloy matrix composites reinforced with nano-size alumina particles were produced by novel method. Al₂O₃ nanoparticles (US Research Nanomaterials, Inc, US3008) used in this research had 99% purity, spherical morphology and average size of 80 nm. Also, for evaluation of effectiveness of the designed distribution method in case of SiO₂ nanoparticles, amorphous silica nanoparticles (US Research Nanomaterials, Inc, US34-38, 20-30nm) were used for fabrication of A356/SiO₂ nano-composite, too. Al powder (purity 99%, 45-100

micron) was purchased from Mahor Powder Metallurgy Co. Table 1 shows chemical composition (wt. %) of the commercial A356 aluminum alloy measured by quantometer (Emission Spectrometer) and standard composition.

Flowchart of manufacturing process is shown in Figure 1. Step one was dispersion of nanoparticles in ethanol using ultrasonic bath (60 minutes) in order to break up the initial Al_2O_3 or SiO_2 nano-powder clustering; step two was spraying of nanoparticle-ethanol mixture on the surface of a thin Al powder layer (about 200-300 micron thickness) that distributed on aluminum foil substrate and then sandwiching the mixture (see Figure 2(a)). In step three, sandwich foils (containing Al_2O_3 nanoparticles and Al powders) were cut in appropriate parts (see Figure 2(b)) and added into molten alloy during stirring. The stirring was continued for 15 min to produce homogenous mixture. The pouring temperature and speed of graphite impeller was selected 6700 °C and 700 rpm, respectively.

The porosity volume percent of the A356/Al₂O₃ nano-composites was determined by comparing the measured density (by the Archimedean method of weighing small pieces cut from the composite first in air and then in water) with that of their theoretical density (calculated using the rule of mixture) [34, 35]. Distribution of nanoparticles in A356 matrix was investigated by scanning electron microscopy (SEM) (model VP1450, LEO, Germany). All the samples were gold-sputtered before SEM observation. Mechanical tests of A356/Al₂O₃ composites were performed by compression strength and hardness measurement. Compression tests were carried out on a Zwick Z250 testing machine at room temperature with the rate of 0.5 mm/min. Hardness tests were done 5 times for each specimen with KOOPA UV1 testing machine and the averages of results were reported.

3. RESULTS AND DISCUSSION

Figure 3 demonstrates the microstructure of A356/0.4 wt% SiO_2 (np) composites at different magnifications. As mentioned above, we used the silica and alumina nanoparticles for investigation of ability of new method in the case of uniform nanoparticles distribution.

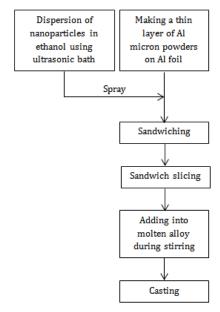


Figure 1. Flowchart of manufacturing steps.

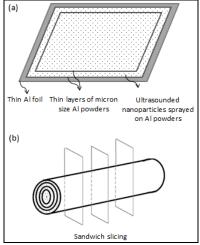


Figure 2. Graphical scheme of (a) nanoparticle-ethanol mixture sprayed on the surface of a thin Al micron powder that distributed on aluminum foil substrate and (b) sandwiching the foil and slicing it.

When sandwich slices are added into the melt, Al powders and foil start to dissolve in the molten matrix and gradually release nanoparticles into the melt. As indicated in Figure 3 (a), formation of silicon blades in A356 matrix is observable.

Figure 3(b) shows homogenous dispersion of silica nanoparticles between silicon blades. Higher magnification reveals homogenous dispersion of silica nanoparticles in the matrix (Figure 3(c)), although some small clusters remain in the microstructure (see Figure 3(d)).

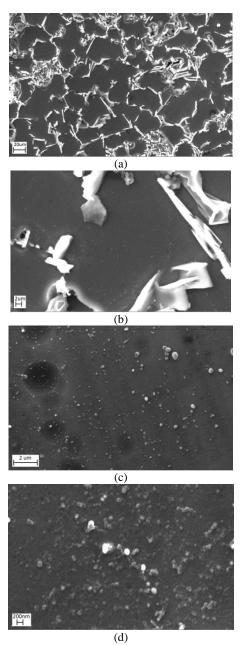


Figure 3. SEM images of microstructure of A356/0.4wt% $SiO_2(np)$ nano-composite at different magnifications: (a), (b) formation of silicon blades in A356 matrix and (c), (d) homogenous dispersion of silica nanoparticles between blades

Figure 4 shows another SEM image of this sample. It reveals good distribution of particles and very low agglomeration in the nano-composite reinforced with 0.4 wt% SiO_2 , fabricated by this process. Therefore, according to Figures 3 and 4 we can say the designed method in this research has a good ability for production of uniform nano-composite.

Figure 5 ((a)-(h)) shows the SEM images of microstructure of A356 and nano-composite samples at different magnifications. A356 alloy has the intermetallic phase that have incorporated at grain boundaries. In fact, since iron is dissolves in molten aluminum and considering that with reducing the temperature solubility of iron reduced, therefore the iron tends to combine with other elements and forms the intermetallic phases. In the absence of Si and Mg, the intermetallic phases are formed Al₃Fe and Al₆Fe. But in the presence of Si and Mg (Table 1) Al₈FeMg₃Si₆ can be produced. Another phase that may be formed in the presence of Mn is Al₁₅(Fe,Mn)₃Si₂ [36]. The coarse and continuous iron-rich intermetallic phases mentioned in Figure 5 (a), (c), (e) and (g), generally because of their color and typical morphology as dashed rectangular are easily detectable in the microstructure. In addition, uniform distribution of Al₂O₃ nanoparticles in nanocomposite samples were confirmed by SEM micrographs at higher magnifications. These particles can be seen in QBSD SEM images of A356/Al₂O₃ nanocomposite, which are absence in A356, sample (see Figure 5(b)). According to the SEM images, utilizing the current method can create a uniform distribution of Al₂O₃ nanoparticles in A356 matrix (see Figure 5 (d) and (f)). However, as can be seen in Figure 5 (h), the increase of weight percentage of Al₂O₃ to 3%wt causes the agglomerated zone of nanoparticles. The increase in volume fraction of Al₂O₃ affects the grain size and morphology of intermetallic phases as well as nanoparticles dispersion [37, 38].

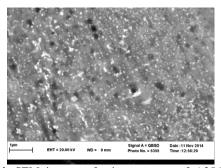
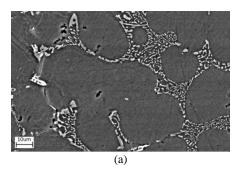
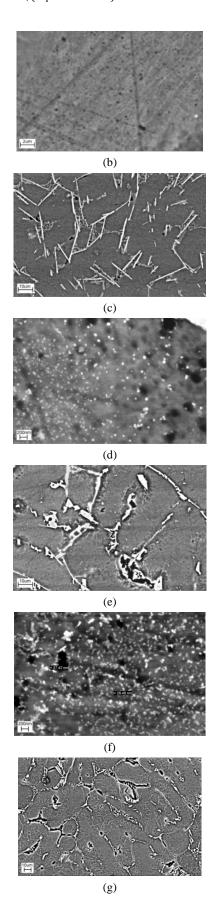


Figure 4. SEM images of microstructure of A356/0.4wt% $SiO_2(np)$ nano-composite





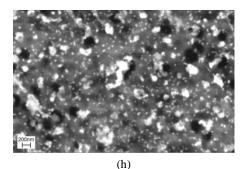


Figure 5. SEM images of microstructure of A356 and nanocomposite samples at different magnifications. (a), (b): A356, (c), (d): A356/ 1 wt% Al_2O_3 , (e), (f): A356/ 2 wt% Al_2O_3 (g), (h): A356/ 3wt% Al_2O_3

The compressive stress-strain curves of nano-composite samples are provided in Figure 6. Table 2 shows the yield stress and compressive strength of nano-composite samples. Addition of alumina nanoparticles has a significant impact on the mechanical properties of the samples.

As shown in Table 2, samples with 1, 2 and 3 wt. % Al_2O_3 nanoparticles have a higher yield strength and compressive strength compared to A356. According to results of compression tests presented in Table 2, compression strength of samples with 1, 2 and 3 wt. % Al_2O_3 nanoparticles have 34.6, 95.6 and 97.5 % enhancement, respectively, compared to A356 matrix alloy. Different theories can be considered for investigation of strengthening mechanism of Al_2O_3 nanoparticles.

The strengthening mechanisms responsible for the yield strength enhancement include Orowan strengthening, grain size refinement due to the Hall-Petch effect, creation of dislocations to accommodate thermal expansion mismatch between the reinforcement and the matrix, and the loadbearing effect of the reinforcement.

Among them, Orowan strengthening plays a decisive role in enhancing the yield strength of MMNCs due to the nanoscale dimension of reinforcing particles. These nanoparticles impede the movement of dislocations through dislocation bowing and looping effects. The bowing of dislocations between the nanoparticles builds up dislocation loops around each nanoparticle. On the other hand, Orowan strengthening is insignificant in the MMCs reinforced with microparticles, because the reinforcing particles are coarse and thus the interparticle spacing is large [4, 38]. Comparison of stress-strain curve of nano-composite with 2 and 3 wt. % Al₂O₃ nanoparticle (see Figure 6) and also compression strength of them (see Table 2) indicate that addition of Al₂O₃ nanoparticles up to 2 wt. % did not have significant effect on strength of nano-composite fabricated using this method. According to literature

survey done by authors, the main reason for this manner is porosity. The porosity content of nano-composites is shown in Table 3. The results show that the amount of porosity increases with increasing nano alumina. This is due to the effect of low wettability and agglomeration at high content of reinforcement and pore nucleation at the matrix- Al₂O₃ interfaces. Furthermore, decreasing liquid metal flow (increasing viscosity) associated with the particle clusters led to the formation of porosity. Such observation has been reported in the literature [32, 39, 40]. Finally, according to the results of SEM images, compression strength and considering the cost of Al₂O₃ nano-powder, quite significant improvement in strength is noted when 2 wt.% nano- Al₂O₃ for stir-casting is added. Figure 7 shows the influence of nanoparticle content on hardness of nano-composite samples. According to the data presented in the Table 4, addition of 1, 2, and 3%, alumina nanoparticles, respectively increased the hardness values about 22, 43, 67% relative to control sample (A356 alloy).

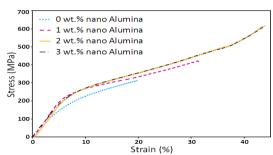


Figure 6. Compression of stress-strain curves of A356 alloy and $A356/Al_2O_3(np)$ nano-composite

TABLE 2. Yield stress and compressive strength of nanocomposite samples

Sample	Yield strength (MPa)	Compression strength (MPa)
A356	199.87	313.56
A356/1 wt. % Al ₂ O ₃	242.55	422.14
A356/2 wt.% Al ₂ O ₃	253.43	613.35
A356/3 wt.% Al ₂ O ₃	250.36	619.34

TABLE 3. The porosity percent of nano-composite

Sample	Porosity (%)
A356	1.43
A356/1 wt. % Al ₂ O ₃	1.93
A356/2 wt. % Al ₂ O ₃	2.27
A356/3 wt. % Al ₂ O ₃	4.71

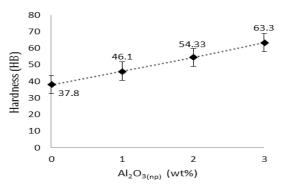


Figure 7. Influence of nanoparticle content on hardness of samples

TABLE 4. Influence of nanoparticle content on hardness of nano-composites.

Sample	Average hardness (HB)	Enhancement relative to A356
A356	37.8	0
A356/1 wt. % Al ₂ O ₃	46.1	22%
A356/2 wt. % Al ₂ O ₃	54.3	43%
A356/3 wt. % Al ₂ O ₃	63.3	67%

Generally, the effect of nanoparticles on the increasing of hardness is mainly due to grain refinement, Hall–Petch mechanism, and particle strengthening effects which act as obstacles to the motion of dislocations [30]. Since hardness is defined as resistance to deformation, therefore, as shown in Table 4, increasing nanoparticles weight percentage limits the deformation of matrix and hardness increases. This behavior can contribute to reinforcement of particle distance. Increasing the weight percentage of particles limits the ability of plastic deformation of matrix and causes the higher hardness of composite.

3. CONCLUSION

We report a novel and simple manufacturing route, a combination of ultrasonic, spraying and conventional stir casting to produce A356/Al₂O_{3(np)} nano-composite. The results reported in this work provide a simple promising way to solve the difficult challenges encountered in homogenous dispersion of oxide nanoparticles in metal matrix nanocomposite manufactured through solidification processing. According to SEM images, reinforced particles were well distributed in the matrix of composites. However, partial agglomeration was observed in composites with high content of Al_2O_3 . The A356/2 wt% $Al_2O_{3(np)}$ nanocomposite produced by this method exhibits 26.79% enhancement in yield stress, 95.6% enhancement in compression strength and 43% enhancement in hardness.

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Keywords: A356/Al₂O₃ Nano-composite Homogeneous Dispersion SEM Observations Mechanical Strength در تحقیق حاضر، روشی جدید و مقرون به صرفه جهت تولید نانوکامپوزیت A356/Al₂O₃ برای دستیابی به توزیع همگن نانوذرات Al₂O₃ در زمینه آلومینیوم مورد بررسی قرار گرفت. بدین منظور نخست پودر میکرونی آلومینیوم بر روی فویل Al₂O₃ براکنده و سپس نانوذرات Al₂O₃ در اتانول التراسونیک شده و بر روی زمینه آلومینیومی اسپری گردید. به دنبال آن، عملیات کامپوزیت سازی با افزودن فویل ساندیویچی به مذاب A356 حین هم زدن، انجام شد. از تصاویر میکروسکوپ الکترونی روبشی (SEM)، آزمون فشار و سختی سنجی جهت بررسی نحوه توزیع نانوذرات درون زمینه و خواص مکانیکی محصولات تولیدی استفاده گردیدنتایج نشان داد که در این روش جدید با افزودن ۲ درصد وزنی نانوذرات آلومینا به زمینه، نانوکامپوزیتی حاصل می گردد که دارای تنش تسلیم، استحکام فشاری و سختی به ترتیب ۲۹٫۷۹، ۲۹٫۷۹ و ۳۶٪ بهبود یافته نسبت به آلیاژ خالص تحت شرایط تولید یکسان می باشد.

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