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ORIGINAL ARTICLE

Preparation and characterization of squeeze cast-Al–Si piston alloy reinforced by Ni and nano-Al₂O₃ particles



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Abstract Al–Si base composites reinforced with different mixtures of Ni and nano-Al₂O₃ particles have been fabricated by squeeze casting and their metallurgical and mechanical characterization has been investigated. A mixture of Ni and nano-Al₂O₃ particles of different ratios was added to the melted Al-Si piston alloy at 700 °C and stirred under pressure. After the Al-base-nano-composites were fabricated by squeeze casting, the microstructure and the particle distribution inside the matrix have been investigated using optical and scanning electron microscopes. Moreover, the hardness and the tensile properties of the resulted Al-base-nano-composites were evaluated at room temperature by using Vickers hardness and universal tensile testers, respectively. As a result, in most cases, it was found that the matrix showed a fine eutectic structure of short silicon constituent which appeared in the form of islands in the α -phase around some added particle agglomerations of the nano-composite structures. The tendency of this structure formation increases with the increase of Ni particle addition. As the ratio of the added particles increases, the tendency of these particles to be agglomerated also increases. Regarding the tensile properties of the fabricated Al-base-nanocomposites, ultimate tensile strength is increased by adding the Ni and nano-Al₂O₃ particles up to 10 and 2 wt.%, respectively. Moreover, the ductility of the fabricated composites is significantly improved by increasing the added Ni particles. The composite material reinforced with 5 wt.% Ni and 2 wt.% nano-Al₂O₃ particles showed superior ultimate tensile strength and good ductility compared with any other added particles in this investigation.

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1. Introduction

Aluminum–silicon alloys and their metal matrix composites have found applications in the manufacturing of various automotive engine components such as cylinder blocks, pistons and

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piston insert rings where adhesive wear (or dry sliding wear) is a predominant process (Deuis et al., 1997; Prasad and Krishna, 2011; Chen et al., 1997; Rajnovic and Sidjanin, 2007). However, most Al–Si alloys are not suitable for high temperature applications because tensile and fatigue strengths are not as high as desired in the temperature range of 500– 700 °C (Yasmin et al., 2004). The pistons for high-speed engines are primarily made of aluminum alloys which contain about 11–13% silicon and approx. 1% each of copper, nickel and magnesium (Cheng et al., 2010). Moreover, the strength of aluminum–silicon materials can be increased by locally casting-in ceramic short fibers, thin single crystalline fibers (whiskers), or porous metallic parts (Cheng et al., 2010). These materials are called metal matrix composites (MMCs).

Al-based MMCs, reinforced with ceramics or metallic particles were developed as an alternative to materials with superior strength - weight and strength - cost ratios, high stiffness, and excellent thermal stability, which have great effects on improving wear, creep and fatigue resistance. Selecting reinforcement remains one of the most critical factors in realizing the best properties from the resultant MMCs. The widely used particles for reinforcing Al alloys are alumina (Al₂O₃) and silicon carbide (SiC). Besides their high hardness, they show low density and low cost compared with other reinforcements. In the past two decades, the wear resistance of the aluminum alloys reinforced with Al₂O₃ and SiC in many forms (particles, whiskers and fibers) and sizes has been described by a huge body of publications such as automotive, aerospace and military industries (Mahmoud et al., 2008, 2009, 2010; Mahadevan and Gopal, 2008; Sajjadi et al., 2012).

However, the poor toughness and the extra cost of metal matrix composites relative to aluminum alloys impose serious restrictions on their applications, especially at high volume fractions of the reinforcement (Zhang et al., 2002). Increasing the reinforcement volume fraction of the MMCs can significantly improve the strength and stiffness of the composites on one hand, but it drastically decreases the toughness and ductility on the other hand (Saha et al., 2002). This can be attributed to an increase in the severity of the triaxiality of stress in the matrix, which results in an earlier onset of void nucleation in the matrix and at the particle/matrix interface (Saha et al., 2002). It is widely recognized that the mechanical properties of metal matrix composites (MMCs) are controlled by the type, size, and volume fraction of the reinforcement phase(s), and the nature of the matrix-reinforcement interface. Superior mechanical properties can be achieved when fine, thermally stable and hard reinforcement(s) with good and clean interfacial bonding are dispersed uniformly in the metal matrix (Jiang et al., 2009). Such characteristics can be obtained when the size of the reinforcement phase(s) is reduced to lower than 1 µm. These materials are called "metal matrix nanocomposites" (MMNCs).

"Metal matrix nano-composites" (MMNCs) provide a new family of MMCs that contain particulate reinforcement of particle size ranging from 10 nm to 1 μ m that are dispersed uniformly in the matrix, which possess not only high specific strength and excellent wear properties, but also good ductility and high fracture (Wu and Li, 2010; Silva, 2006; Yang and Li, 2007). Carbon nano-tubes, silica, aluminum oxide, titanium dioxide, zinc oxide, silicon carbide, polyhedral oligomeric silsesquioxanes (POSS) are examples for nano-particle fillers (Yang and Li, 2007). In the last two decades, metal matrix

nanocomposites have witnessed tremendous growth, especially in the automotive industry for their capability to withstand high temperature and pressure conditions (Wu and Li, 2010).

A variety of methods for producing MMNCs on industrial scale have been developed, including powder metallurgy (PM) (Kang and Chan, 2004; Ma et al., 1996), high-energy milling (Sherif and Eskandarany, 1998; He et al., 1998) and severe plastic deformation (Valiev et al., 2000; Alexandrov et al., 1998), which is considered as solid state processing. The major critical problems facing these processes are contamination results from powder preparation and complexity of fabrication steps. Moreover, machining is required to obtain the desired final shape. The other group of the fabrication processes of MMNCs is the liquid-state processing, which includes infiltration techniques. stirring techniques, rapid solidification, as well as some in situ fabrication such as liquid-gas bubbling (Yu, 2010). These processes offer some advantages compared with the solid-state processing such as energy-efficient and cost-effective (Yu, 2010). However, reinforcement non-homogeneous distribution or particle agglomeration in the molten matrix and during solidification, and pore formation are considered the critical problems facing the fabrication of MMNCs by liquid-state processes (Yu, 2010). One of the liquid state processes that can be utilized in producing MMNCs is the Squeeze casting. In this process, the applied pressure and the instantaneous contact of molten metal with the die surface produce rapid heat transfer that yields a porous free casting with mechanical properties approaching the wrought product (Yu, 2010).

Piston is the key part of the engine as it works under high temperature, high pressure, corrosive and wearing conditions while running with high speed (Wu and Li, 2010). The pistons lie at the heart of the internal combustion engine and their reciprocating motion will generate severe stress on the piston crown, sidewall, and the piston's top rings. To reduce the HC emission, the piston top land must be very thin (Lee, 1998). In order to satisfy all these severe conditions, piston materials must have high strength, high toughness and light weight. Several strengthening technologies, such as pressurization have been developed in recent years to strengthen the piston alloys. However, it should be pointed out that the results of these techniques cannot satisfy the recent application requirements (Wu and Li, 2010). Obviously, new techniques are required to increase the strength of the Al piston alloys. In this case, adding hard particles to the Al alloy and decreasing the grain size are considered the optimum solution. By the way, it is important to record here that the most versatile and economical way to produce the AI piston is the conventional casting methods (Lee, 1998).

The present work aimed to obtain a new Al-alloy-based nanocomposite material by squeeze casting of excellent mechanical properties for piston application. The new nano-composite material composed of Al-piston alloy (Hypoeutectic Al–Si alloy) as a matrix. Nickel and nano Al₂O₃ particles are added as reinforcement.

2. Experimental work

In this study, Al–Si alloy of chemical composition listed in Table 1 was used as a matrix material. The microstructure of the matrix shows fully dendrite grain structure as shown in Fig. 1(a). Ni powder with average particle size of $6 \,\mu m$ was

added to the matrix. The matrix alloy was dispersed with Al_2O_3 nanoparticulates having average sizes of 60 nm.

Firstly, the matrix alloy was melted, and the dispersed particles were heated to 400 °C for 30 min before dispersion inside the matrix for improving the interfacial bonding between the matrix and dispersed particles, and to facilitate the distribution of the dispersed particles inside the matrix (Thakur and Dhindaw, 2001). Different amounts of Ni and Al₂O₃ nanoparticulates were added to the melted matrix, stirred and squeeze casted. Table 2 summaries the experimental conditions employed in the present study. Macro- and microstructure observations were performed on all conditions. Scanning electron microscope equipped with EDX analyzer was used to study the distribution of dispersed particles and to identify experimentally the phases that present inside the matrix after squeeze casting. The microhardness of the product was also measured with Vickers hardness tester at 900 g load for 15 s. The tensile properties of the specimens were evaluated using universal tensile testing machine.

3. Results and discussion

3.1. Effect of reinforcing particle addition on the microstructure

Macroscopic appearances of the samples #I, II and III in which only Ni powder is added in different amounts to the Al–Si

Table 2The experimental conditions employed in the presentstudy.

Sample No.	Amount of Ni particles added to the matrix, wt.%	Amount of Al ₂ O ₃ nanoparticulates added to the matrix, wt.%			
#I	5	-			
#II	10	-			
#III	15	-			
#IV	5	2			
#V	5	3			

matrix, in addition to microstructure of Al–Si sample without any additions, are shown in Fig. 1. Generally, in comparison with Al–Si sample, the micrographs in all cases of Ni additions show fine dendrite grain structure distributed inside α -phase. Some porosity was also observed. These dendrite microstructures can be mainly attributed to the high silicon content of the matrix (11.56 wt.%), approaching the eutectic composition (12.6%), and also to the application of pressure during solidification. Moreover, the pressure applied during solidification increases heat transfer between the casting and the die, which reduces porosity in the final casting and also ensures complete filling of the die. The increase of heat transfer increases the cooling rate and as a result a refined microstructure is produced.

Table 1	Chemical composition of the matrix (wt.%).										
Element	Si	Fe	Cu	Mg	Mn	Ni	Ti	Cr	Pb	Al	
Wt.%	11.56	0.53	1.03	0.90	0.18	1.55	0.06	0.04	0.04	Bal.	



Figure 1 Optical micrographs for microstructures of: (a) Al-12% Si piston alloy, (b) piston alloy +5 wt.% Ni, (c) piston alloy +10 wt.% Ni, and (d) piston alloy +15 wt.% Ni.



Figure 2 Optical micrographs for microstructures of: (a) and (b) Al-12% Si piston alloy +5 wt.% Ni +2 wt.% Al₂O₃, and (c) and (d) piston alloy +5 wt.% Ni +3 wt.% Al₂O₃.



Figure 3 SEM micrographs for microstructures of Al-12% Si piston alloy with addition of 5 wt.% Ni composite.

By increasing the added Ni amount, the amount of α -phase was increased, as shown in Fig. 1(c) and (d), in which the fine eutectic structure of short silicon constituent appeared in the form of patches or islands in the α -phase matrix. The tendency of formation of these features increases with Ni addition (compare Fig 1(b)–(d)). The decreasing of the eutectic dendrites and increasing of the α -phase by addition of the Ni powder may be explained as follows: The Al–Si phase diagram could be changed due to the presence of Ni element. The eutectic point could be shifted to another one of higher silicon content. If so, the percentage of α -phase can be increased. This description is not sufficient and more investigations are required to confirm the suggested explanation.

The effect of adding different amounts of nano Al_2O_3 particles, together with 5 wt.% Ni particles, to the matrix is shown in the micrographs of Fig. 2. In general, almost homogenous equiaxed fine grain structures of α -phase were observed. Some clustered reinforcement particles were observed inside the nugget zone as indicated in Fig. 2. These clustering areas were increased with increasing the content of nano Al_2O_3 particles (compare Fig. 2(a) with Fig. 2(c)).

Microscopically, the SEM microstructures observed in the different samples are shown in Figs. 3–7. In case of only Ni addition samples, the Ni particles are distributed in acicular forms inside the Al–Si matrix as shown in Figs. 3–5. In addition, these SEM images show that the Ni particles were dispersed in the aluminum matrix and directly bonded without any cracks or voids between them (See Fig. 4(c) and (d)). The edges of relatively large dispersed Ni particle, as shown in Fig. 4(d), were irregular in shape and have a different color than the interior parts of the Ni particles. The EDS spectra of these areas, as shown in Fig. 5 give atomic content of the Ni of 28 which were the typical compositions of the Al₃Ni while the inner parts were un-reacted Ni element. The formation of



Figure 4 SEM micrographs of microstructures of Al-12% Si piston alloy with addition of 10 wt.% Ni composite.

Al₃Ni in Al – based alloys/reinforced with Ni composites was also reported by other investigators (Mudry and Shtablavyi, 2008). In those works, the structure of Al–Si eutectic with additions of Ni revealed changes corresponding to the formation of chemically ordered microgroups with AlNi and Al₃Ni topology (Mudry and Shtablavyi, 2008).

Regarding the samples that had nano Al_2O_3 particles together with 5% Ni (Samples IV and V), the SEM images, as shown in Figs. 6 and 7, suggest that the nano Al_2O_3 particle distribution in the Al–Si matrix was almost non-uniform. There is an evidence of clustering and agglomeration. This non-uniformity was increased at higher nano Al_2O_3 particle content. On the other hand, the additions of nano Al_2O_3 particles decrease both the matrix grain size to few micrometers and the dendrite arms to few nanometres.

3.2. Effect of reinforcing particle addition on the mechanical properties

The tensile properties of the Al–Si alloy with addition of Ni particle materials are summarized in Figs. 8 and 9. Three

samples were tested for each trial. The average values of ultimate tensile strength and ductility in terms of elongation were calculated. The materials that have Ni content of 5 and 10 wt.% show higher ultimate tensile strength than that of base metal (Al-Si alloy) as indicated in Fig. 8. This can mainly be attributed to the strengthening effect of the reinforcing phases. The Ni particles can be wetted with the molten metal of the piston alloy (base material), and hence, good and strong bonding between the particles and the matrix can be obtained. Consequently, this strong interface can transfer and distribute the load effectively from the matrix to the reinforcement. On the other hand, when the Ni content was increased to 15 wt.%, a small increase in strength was obtained (See Fig. 8). In this case, the reinforcing particles are agglomerated and there is no chance for strong bonding between the particles and the matrix. These results exactly agreed with that obtained in the microstructure section (Section 3.1).

Regarding the ductility in terms of material elongation, the addition of Ni particles (5, 10 and 15 wt.%) to the Al–Si base metal can improve the ductility to reasonable values as clearly



Figure 5 SEM micrograph of Al-12% Si piston alloy with addition of 10 wt.% Ni composite; (a), EDX spectra of red cross in (a); (b).



Figure 6 SEM micrographs of microstructures of Al-12% Si piston alloy with addition of 5 wt.% Ni and 2 wt.% Al₂O₃ composite.



Figure 7 SEM micrographs of microstructures of Al-12% Si piston alloy with addition of 5 wt.% Ni and 3 wt.% Al₂O₃ composite.

shown in Fig. 9. The increase of α -phase percentage and its partial continuity inside the matrix, by the increasing of Ni particles addition, aid in the ductility improvement.

The combined effect of 5 wt.% Ni particles and different nano Al₂O₃ particle addition on ultimate tensile strength and elongation are shown in Figs. 10 and 11. For the ultimate tensile strength (UTS) of the tested specimens, an observable increase was obtained in case of addition of 2 wt.% nano Al₂O₃ particles beside 5% Ni particles. When the nano Al_2O_3 particles were increased to 3 wt.%, the UTS was then decreased as shown in Fig. 10. Fig. 11 shows the same phenomena for ductility; where it increases in case of addition of 2 wt.% nano Al_2O_3 particles and decreases when the nano Al_2O_3 particles becomes 3 wt.%. As reported by Wahab et al. (2009a,b), a good combination of high strength and ductility can be obtained from aluminum based metal matrix composites (MMCs) in a wide area of possible advanced applications. In case of sample V (piston alloy, 5 wt.% Ni



Figure 8 Effect of Ni particle addition on ultimate tensile strength (UTS) of produced composite materials.



Figure 9 Effect of Ni particle addition on engineering strain, ε at fracture of produced composite material.

and 3 wt.% Al₂O₃), the agglomeration or clustering of the added particles and void formation reduces or eliminates their strengthening effect.

The improvement in strength and ductility in case of addition of 2 wt.% nano Al_2O_3 beside 5 wt.% Ni particles may be due to the relatively homogenous structure of the resulted composite materials. In this case, the applied load will transfer to the reinforcement phases (Ni and nano Al_2O_3 particles). In addition, the thermal mismatch between the high expansion aluminum matrix and the low expansion Al_2O_3 ceramic particles is quite high. Also, there is another mismatch between the thermal expansion of aluminum matrix and the Ni-based reinforcing particles. Thus, upon cooling, dislocations form at the reinforcement/matrix interfaces due to the thermal mismatch.

It is very difficult to find the dislocation density for different composites. Generally, the addition of reinforcing materials to the matrix generates boundaries. In the investigated case, there are different boundaries: (1) between the aluminum alloy (matrix) and nickel (reinforcing particles), (2) between the matrix and Al₂O₃ (reinforcing nano-particles), and (3) between the Al₂O₃ and Ni particles. As it is well known in the open literatures (Charles and Arunachalam, 2004; Llovd, 1994; Nan and Clarke, 1996) that during loading of the formed composite, dislocations are generated and accumulated at the produced boundaries. In addition, there are differences between the thermal expansions of the mixed materials. So, during heating, different composite ingredients will expand with different ratios. Upon cooling, dislocations form at the reinforcement/matrix and Al2O3/Ni interfaces due to the thermal mismatch. So, the density of dislocations is increased by the addition of both Al₂O₃ and Ni particles. Due to the differential thermal contraction at the interface between the matrix and



Figure 10 Effect of nano Al_2O_3 particle addition together with 5% Ni particles on ultimate tensile strength (UTS) of produced composite materials.



Figure 11 Effect of nano Al_2O_3 particle addition together with 5% Ni particles on engineering strain at fracture for the produced composite material.

the reinforcements, misfit strain is resulted. The misfit strain and resultant misfit stress, generate dislocations (Charles and Arunachalam, 2004). The additional dislocation density is necessary to accommodate large thermal misfit strains between the particles and the matrix (Lloyd, 1994; Nan and Clarke, 1996).

The generation of dislocation at the reinforcement/matrix interface, due to the thermal mismatch, has been reported and proved by other investigators (Wierzbińska and Sieniawski, 2006; Wahab et al., 2009a,b; Kang and Chan, 2004; Chawla and Shen, 2001; Ranganath et al., 2002). They proved that the thermally induced dislocations lead to strength improvement. As described in Charles and Arunachalam, 2004; Ranganath et al., 2002, the increased dislocation density shares a significant contribution to strengthening of metal matrix.

Regarding the fracture surface of the tensile test specimens, all the samples used in this work were investigated with scanning electron microscope and the results are shown in Fig. 12. Generally, all samples show equiaxed Large and small (fine) dimples in the fracture surfaces, which is considered ductile fracture. The fine dimples were pronounced in samples IV and V, in which nano Al₂O₃ particles are added. This may be due to that the size of the dimple on a fracture surface is governed by the number and distribution of microvoids that



Figure 12 SEM fractographs of (a) piston alloy (without additions), (b) piston alloy +5 wt.% Ni, (c) piston alloy +10 wt.% Ni, (d) piston alloy +15 wt.% Ni, (e) piston alloy +5 wt.% Ni +2 wt.% Al₂O₃, and (f) piston alloy +5 wt.% Ni +3 wt.% Al₂O₃ nanocomposite.

are nucleated. Fine dimples are formed when numerous nucleating sites are activated and adjacent microvoids join (coalesce) before they have an opportunity to grow to a larger size. When the nucleation sites are few and widely spaced, the microvoids grow to a large size before coalescing and the result is a fracture surface that contains large dimples. This explanation agrees with that reported by many investigators (Martin et al., 1996; Sekine and Chen, 1995; Chen and Zhang, 1993). On the other hand, it is important to record here that some porosity was found in all the investigated fracture surfaces.

The economical factor is an important factor to be discussed in this work. The increase of Ni additions increases the weight and cost. For effective and economical piston material, the amount of Ni particles required to strengthen the piston material must be decreased. Therefore, a material of accepted strength and good ductility (i.e. good toughness) can be produced by adding only 5 wt.% Ni particles in addition to 2 wt.% nano Al₂O₃ particles, which has a relatively low density, to the Al-Si alloy. At the same time, it is too difficult to accurately estimate the cost reduction. Alloying elements are considered one of the main factors that affect production cost of any alloy. When the strength of the alloy is increased without adding the expensive alloying elements, the cost will be remarkably reduced. On the other hand, some alloying elements such as copper, which is added for strength improvements, can negatively affect other properties such as corrosion resistance. Moreover, the fabrication process that was used to prepare these materials (stir casting) is considered the most cost-effective process (Michaud, 1993; Zhou and Xu, 1997).

4. Conclusions

In this work, Al–Si piston was dispersed with different contents of Ni particles (5%, 10%, and 15%) and Al_2O_3 nano-particulates (2% and 3% together with 5% Ni particles) through squeeze casting. Macro- and microstructure observations were performed on all conditions. The microhardness of the product was also measured with a Vickers hardness tester. Detailed mechanical properties (Yield strength, tensile strength, elongation, modulus of elasticity) were evaluated by using universal tensile testing machine. The obtained results are summarized as follows:

- The Ni particles were distributed homogenously in acicular forms inside the Al–Si matrix without any cracks or voids between them. The Ni particles reacted with Al matrix forming Al₃Ni intermetallics. Addition of Ni particles decreases the Al–Si matrix grain size and eutectic dendrite arms, but it increases the amount of α -phase.
- The nano Al₂O₃ particle distribution in the Al–Si matrix was almost non-uniform, especially at higher percentage (3%). The additions of nano Al₂O₃ particles together with 5 wt.% Ni particles decrease both the matrix grain size to few micrometers and the dendrite arms to few nanometres.
- The ultimate tensile strength (UTS) of the resulted materials can increase by increasing the Ni content to almost 10%. The highest UTS can be obtained by addition of 5 wt.% Ni and 2 wt.% nano Al₂O₃ particles.
- The addition of Ni particles (5, 10 and 15 wt.%) to the Al–Si base metal can improve the ductility to reasonable values. When the nano Al_2O_3 particles were added by 2 wt. %, the ductility was improved.

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