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# Microstructure and Some Properties of Aluminum-Silicon Matrix Composites Reinforced by Alumina or Chromia

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### Abstract

In this work, yttrium oxide particles (powder) reinforced AL-Si matrix composites (Y2O3/Al-Si) and Chromium oxide particles reinforced AL-Si matrix composites (Cr2O3/AL-Si) were prepared by direct squeeze casting. The volume percentages of yttrium oxide used are (4, 8.1, 12.1, 16.1 vol %) and the volume percentages of the chromium oxide particles used are (3.1, 6.3, 9.4, 12.5 vol. %). The parameters affecting the preparation of Y2O3/Al-Si and Cr2O3/AL-Si composites by direct squeeze casting process were studied. The molten Al-Si alloy with yttrium oxide particles or with chromium oxide particles was stirred again using an electrical stirrer at speed 500 rpm and the molten alloy was poured into the squeeze die cavity. The pouring temperature that used for all castings is (700)°C. The required squeeze pressure, 53 MPa, was then applied for 30 seconds at a delay time of 5. The die temp is (200°C). The Y<sub>2</sub>O<sub>3</sub>/Al-Si composites and Cr<sub>2</sub>O<sub>3</sub>/Al-Si composites produced by squeeze casting have more microstructure refinement, higher hardness and lower wear rate than the unreinforced alloy.

Keywords: Composite, Microstructure, Al-Si, Yttria, Alumina

### 1. Introduction

The squeeze casting technique produces parts with reduced porosity levels because of the high pressure applied during solidification and due to efficient liquid feeding obtained by moving the ram to compensate for the freezing contraction. The cooling rate of the casting can be increased by applying high pressure during solidification due to increase in thermal conductivity [1, 2].

Metal matrix composites (MMCs) of metallic alloys reinforced with ceramics particles are designed to offer high specific mechanical properties that are highly desirable in various applications and provide a relatively novel way of strengthening metals [3,4].

Aluminum/Silicon alloys are known for their high strength to weight ratio, good castability, low thermal expansion and high corrosion resistance. These properties have led to their application in all types of internal combustion engines as pistons, cylinder blocks and cylinder heads. Al-Si matrix composites are composed of Al-Si alloy matrix, and reinforcement. Reinforcements added to Al-alloy matrices include intermetallic compounds, ceramic phases such as oxides, carbides and nitrides as well as refractory metals[5].

Particle reinforced composites are characterized by particles having a diameter greater than 1  $\mu$ m with a volume fraction of 5 to 40%. Particle reinforced composites show isotropic stiffness improvements with generally lesser degradation of fracture properties than that of whisker reinforced MMCs [1].

Etter et al. [6] also examined graphite particles with Al-Si alloy prepared by squeeze casting, but with different Si content. They concluded an increase in the mechanical strength and fracture toughness by using this process and with Si addition.

Long et al. [7] showed an increase in the mechanical properties (stiffness, hardness, and fatigue strength and wear resistance) before and after heat treatment of 60wt? SiC particles /Al-Cu-Mg-Ag alloy. This was related directly to the addition of SiC particles and squeeze casting process.

Kiourtsidis et al. [8] showed a decrease in the dendritic size of 8-24 vol % SiC particles/2024 Al alloy with increasing the SiC volume fraction. Also they studied the corrosion behavior of that composite, and they found no galvanic interaction between SiC and the matrix, instead there is an anodic reaction which is independent of the SiC volume fraction.

The aim of this work is to study the preparation of  $Y_2O_3/Al$ -Si and  $Cr_2O_3/AL$ -Si composites by direct squeeze casting process.

### 2. Experimental Procedures 2.1. Materials and Methods

The composite materials used in this work are Al-Si eutectic alloy with yttrium oxide and with chromium oxide particles as reinforcements. A standard aluminum-silicon eutectic alloy type 1725 DIN [9] was used in this work as the matrix of the composites. This alloy has good bearing properties, good fluidity and low coefficient of thermal expansion with a density of 2.68 g/cm<sup>3</sup>. It is a common alloy used in the production of pistons [10,11]. Table (1) shows the chemical composition of the alloy. The analysis was done by using the atomic absorption apparatus at the Ministry of Science and Technology in Baghdad, Iraq.

Table	1.
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Chemical composition of the A119-1725 Al-Si alloy.							
Si	Fe	Cu	Mn	Mg	Zn	Ti	Al
12.1	0.65	0.83	0.2	0.27	0.45	0.02	Bal.

Chromium oxide powder with particle size  $(50-75) \mu m$  and yttrium oxide powder with particle size  $(50-75) \mu m$  were used as reinforcements. The particle size was measured by sieving method. The density of the Chromium oxide powder and yttrium oxide powder are  $(3.404 \text{ and } 2.65 \text{ gm/cm}^3)$  respectively as determined by taking the weight and volume of a green packed powder in a cylindrical form and using the formula [12]:

 $\rho = m/V \qquad \dots (1)$ 

Where  $\rho$  is the density gm/ cm<sup>3</sup>, m = mass (gm), V= volume (cm<sup>3</sup>) .

The squeeze casting system consists of hydraulic press, squeeze die and auxiliary equipment. A vertical hydraulic press is used (with a ram 70 mm in diameter) to apply a pressure of (1-100) Kg/cm<sup>2</sup> in a perpendicular direction to the squeeze casting die. The squeeze punch was made from hot forming steel, and designed with a hole in its upper part to be fixed to the ram with two steel bolts. The lower part of the ram is a rectangular in shape with a semicircle ends which is used to squeeze the molten alloy. The die is horizontal and consists of two parts joined by four high temperature resistant steel bolts. This design allows the final squeezed composite casting to be removed from the die easily. Figure (1) shows the punch and the die.



Fig.1. The Punch and Die Used in Squeeze Casting.

The standard Al-Si alloy was melted in an electrical furnace. A calibrated K- type thermocouple is used to measure the temperature with an accuracy of  $\pm 1^{\circ}$ C. A box type electrical heating furnace (Carbolite Co, England) was used to heat the reinforcement particles.

The standard Al-Si allov was melted using an electrical furnace at the required temperature using alumina crucible. The experiments were done at the University of Technology. The chromium oxide particles (3.1, 6.3, 9.4, 12.5 vol. %) or Yttrium oxide particles (4, 8.1, 12.1, 16.1 vol %) were wrapped in a high purity Al foils and heated to 300°C for 15 minutes in the heating furnace. The heated aluminum foil with the chromium oxide particles or with the yttrium oxide particles were then added to the molten Al-Si alloy and the mixture was stirred for 3 minutes using an electrical stirrer (type RW20-JANKE & KUNKEL Co, Germany) at speed 500 rpm. Then the molten Al-Si alloy with chromium oxide particles or with yttrium oxide particles was put again in the melting furnace. The squeeze casting die was then preheated to the required preheating temperatures (200)°C. The preheated die was then placed on the hydraulic press table. The molten Al-Si alloy with chromium oxide particles was

stirred again using an electrical stirrer at speed 500 rpm and the molten alloy was poured into the squeeze die cavity. The pouring temperatures that used for all castings are  $(700)^{\circ}$ C. The required squeeze pressure was then applied for 30 seconds at a delay time of 5 seconds and allowing for solidification. The hydraulic pressures that are used for all castings are (53) MPa. After that the solidified composite casting was removed from the die.

The Al-Si squeeze cast composites were cut into rectangular samples with dimensions  $1 \times 1 \times 0.5$ cm<sup>3</sup>. Each specimen was wet ground with silicon carbide papers 1000 grit using grinding machine (Struers DAP-5, Denmark) and then polished on polishing clothes using  $5.0 \,\mu\text{m}$  and  $0.3 \,\mu\text{m}$ alumina suspension sequentially. Each specimen was then washed with water and alcohol, and etched by a dilute HF acid (1<sup>'</sup>/. distilled water and 99<sup>'</sup>/. HF). The microstructure was examined by an optical microscope (100WCARLZEISS JENA, Germany) with a digital camera type (Smartic with 5 Mega pixels resolution).

### 2.2. Density measurements

Two types of density were calculated as follows:

### 1) Theoretical Density Calculation

The following mixture formula was used to calculate the theoretical density of the composites [13]:

$$\rho_{\rm c} = \rho_{\rm m} f_{\rm m} + \rho_{\rm r} f_{\rm r} \qquad \dots (2)$$

where

- $\rho_c$ : composite density
- $\rho_m$ : matrix density

 $f_m$ : matrix volume fraction

 $\rho_r$ : reinforcement density

f<sub>r</sub> : reinforcement volume fraction

### 2) Practical Density Calculation

The practical density  $(\rho_p)$  can be calculated from formula (1)

### 2.3. Porosity

The porosity of the AL-Si composite was calculated for all specimens using the following formula [14]:

$$P = [1 - (\rho_p / \rho_c)] * 100 \qquad \dots (3)$$

Where: P= porosity %  $\rho_p$  = Practical density  $\rho_c$ = Composite density

### 2.4. Microhardness Test

The microhardness of each specimen was measured by using Vickers microhardness apparatus provided by the Materials Engineering Department, University of Technology. Vickers hardness number was calculated by the following equation [15]:

Hv = 
$$1.8544 [P / (d_{av})^2]$$
 ...(4)

where:

P : The applied load = 1 Kg

 $d_{av}$ : The average diameter of the two diagonal length of the rhombus indentation

For each specimen three hardness measurements were taken and the average hardness was calculated.

## 2.5. Wear Test

Dry sliding wear tests were conducted for the graphite particles reinforced Al-Si matrix composite specimens on the pin-on-disc apparatus. A cylindrical wear specimen was used with a diameter of 10 mm and a length of 20 mm. The specimen slides on a carbon steel disc with a hardness of 38 HRC.

Wear rates were calculated from the following formula [8]:

Wear rate (Wr) = 
$$\Delta w / 2\pi Rn\rho_{pr}t$$
 ...(5)  
And

 $\Delta \mathbf{w} = \mathbf{w}_1 - \mathbf{w}_2 \qquad \dots (6)$ 

where:

Wr : Wear rate  $(cm^{3}/cm)$ 

 $w_1$ : Specimen weight before the wear test (gm).

 $w_2$ : Specimen weight after the wear test (gm).

R : The distance from the center of the specimen to the center of the steel disc = 7 cm.

n : The number of cycles for the steel disc =512 rpm.

 $\rho_{pr}$ : The practical density of the composite specimens (gm/cm<sup>3</sup>).

t : The sliding time (20 minutes).

The applied load used in this test was 10 N.

# 3. Results and Discussion 3.1. Microstructure

Figure (2) shows the microstructure of gravity cast commercial Al-12<sup>7</sup>.Si alloy and Figure (3) shows the microstructure of squeeze cast Al-

12% Si alloy. It consists of  $\alpha$ -aluminum dendrites (white) surrounded by eutectic phase and primary silicon particles (light gray). The microstructure in Figure (3) is refined due to the effect of squeeze conditions in the faster cooling rate and hence smaller eutectic and dendrites.



Fig.2. Unreinforced Al-Si Alloy.

Fig.3. Squeeze Cast Al-Si Alloy



Fig.4. 2wt<sup>½</sup> Y<sub>2</sub>O<sub>3</sub>/Al-Si composite.

The microstructures of the Y<sub>2</sub>O<sub>3</sub> reinforced Al-Si matrix composites and Cr<sub>2</sub>O<sub>3</sub> reinforced Al-Si matrix composites are shown in Figures (4-7) and (8-11) respectively. The microstructure reveals that there are small discontinuities and a reasonably uniform distribution of  $Y_2O_3$ particulates and more uniform distribution of the Cr<sub>2</sub>O<sub>3</sub> particles. The ceramic phase is shown as dark phase. The quantity and distribution of reinforcement particles differ from figure to figure since they depend primarily on the composite preparation before applying squeeze casting process. The microstructures of these MMCs show that the primary aluminum tends to avoid

Fig.5. 4wt<sup>½</sup> Y<sub>2</sub>O<sub>3</sub>/Al-Si composite

the discontinuous phase and nucleates in the interstices between particles. Primary aluminum crystallized from the melt does not nucleate on the particle surface, because of surface thermodynamics and solute. The primary aaluminum grows by rejecting solute in the melt, while the reinforcing particles tend to restrict diffusion and fluid flow; therefore,  $\alpha$ -aluminum tends to avoid it [16,17]. It can be seen from these figures that the squeeze pressure are capable of producing good bonding between the particles and the matrix alloy. This is concluded from the absence of any cavities that can result from the pull out of these particles during the specimen

preparation process for the microstructure examination.

Increasing the volume fraction of the  $Y_2O_3$  particles and  $Cr_2O_3$  particles have led to more

refinement in the microstructure as can be seen in Figures (7) and (11) respectively which is in agreement with the findings of Long [18].



Fig.6. 6wt? Y2O3/Al-Si composite.



Fig.7. 8wt<sup>½</sup> Y<sub>2</sub>O<sub>3</sub>/Al-Si composite



Fig.8. 2wt<sup>7</sup>/. Cr<sub>2</sub>O<sub>3</sub>/Al-Si composite

Fig.9. 4wt<sup>7</sup>/ Cr<sub>2</sub>O<sub>3</sub>/Al-Si composite



Fig.10. 6wt<sup>½</sup>Cr<sub>2</sub>O<sub>3</sub>/Al-Si composite.

Fig.11. 8wt<sup>7</sup>/Cr<sub>2</sub>O<sub>3</sub>/Al-Si composite

### 3.2. Density and Porosity

Table (2) shows the values of density and porosity for the unreinforced alloy and the squeeze cast alloy and composites. It is clear that the density of the  $Y_2O_3/Al$ -Si composites is lower than that of the unreinforced alloy since  $Y_2O_3$ particles have lower density than that of the Al-Si alloy, while the density of the  $Cr_2O_3/Al$ -Si composites is higher than that of the unreinforced alloy since  $Cr_2O_3$  particles have higher density.

Figure (12) illustrates the relationship between density and volume fraction of the  $Y_2O_3$  particles. This figure shows that the density decreases slightly with increasing particle volume fraction. Also the practical values of density is less than the theoretical ones due to the presence of shrinkage

and gas porosities as confirmed in Table (2) and Figure (13).

Figure (14) illustrates the relationship between density and  $Cr_2O_3$  particles volume fraction. It shows that the density increases with increasing the volume fraction since  $Cr_2O_3$  have higher density as mentioned before. Figure (15) illustrates the porosity versus  $Cr_2O_3$  volume fraction and it shows the decreasing in porosity with the volume fraction. This is due to the tendency of the particles to segregate and to the increasing of the cooling rate of the casting which can produce more shrinkage porosity. This result indicates that the used squeeze pressure was not enough to eliminate or reduce such porosity at the higher used volume fraction.

Table 2,

Density and Porosity of the Alloy and the Composites Squeeze Cast at 700°C Pouring Temperature, 200°C Die Temperature and 53 MPa Squeeze Pressure for 30 sec.

Material	Theoretical Density (gm/cm <sup>3</sup> )	Practical Density (gm/cm <sup>3</sup> )	Porosity (%)
Al-Si alloy	2.68	2.644	1.3
2wt <sup>7</sup> /Y <sub>2</sub> O <sub>3</sub> /Al-Si composite	2.6788	2.58	3.7
4wt <sup>7</sup> .Y <sub>2</sub> O <sub>3</sub> /Al-Si composite	2.6776	2.49	7
6wt <sup>7</sup> /Y <sub>2</sub> O <sub>3</sub> /Al-Si composite	2.6764	2.42	9.6
8wt <sup>7</sup> /Y <sub>2</sub> O <sub>3</sub> /Al-Si composite	2.6752	2.39	10.7
2wt <sup>7</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al- Sicomposite	2.7	2.646	2
4wt <sup>7</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al- Sicomposite	2.726	2.658	2.5
6wt <sup>7</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al- Sicomposite	2.75	2.68	2.6
8wt <sup>7</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al- Sicomposite	2.771	2.689	3



Fig.12. Density Versus Y<sub>2</sub>O<sub>3</sub> Volume Fraction.



Fig.13. Porosity Versus Y<sub>2</sub>O<sub>3</sub> Volume Fraction.



Fig.14. Density Versus Cr<sub>2</sub>O<sub>3</sub> Volume Fraction.



Fig.15. Porosity Versus Cr<sub>2</sub>O<sub>3</sub> Volume Fraction.

### 3.3. Hardness

Table (3) shows the values of Vickers microhardness for the squeeze cast unreinforced alloy and the composites. This table shows that the composites have higher hardness than the Al-Si alloy due to the effect of squeeze casting and particles addition in increasing the cooling rate and refining the microstructure. Figures (16 and 17) illustrate the relationship between hardness and particles volume fraction for theY2O3/Al-Si composites and Cr<sub>2</sub>O<sub>3</sub>/Al-Si composites respectively. Figure (16) shows that the hardness is maximum at 8 volume percentage of  $Y_2O_3$ . Figure (17) shows that the hardness is maximum at a 7.6 volume percentage of  $Cr_2O_3$ . The lower value of hardness at these percents is due to the segregation of the particles which may result from composite preparation during casting, and the higher amount of porosity at the higher used volume fraction. This is also consistence with wear rate test. The minimum wear rates for theY2O3/Al-Si composites and Cr2O3/Al-Si also occur at a volume percentage of 8 and 7.6 for both types of composites respectively.

### Table 3,

Microhardness of the Alloy and the Composites Squeeze Cast at 700°C Pouring Temperature, 200°C Die Temperature and 53 MPa Squeeze Pressure for 30 sec.

Materials	Vickers Microhardness No.
Al-Si alloy	69
2wt <sup>/</sup> Y <sub>2</sub> O <sub>3</sub> /Al-Si composites	117
4wt <sup>/</sup> Y <sub>2</sub> O <sub>3</sub> /Al-Si composites	72.4
6wt <sup>/</sup> Y <sub>2</sub> O <sub>3</sub> /Al-Si composites	129
8wt <sup>?</sup> Y <sub>2</sub> O <sub>3</sub> /Al-Si composites	69.3
2wt?Cr2O3/Al-Si composites	98.7
4wt?Cr2O3/Al-Si composites	86.2
6wt <sup>2</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al-Si composites	104
8wt <sup>2</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al-Si composites	80.1



Fig.16. Hardness Versus Y<sub>2</sub>O<sub>3</sub> Volume Fraction.



Fig.17. Hardness Versus Cr<sub>2</sub>O<sub>3</sub> Volume Fraction.

### 3.4. Wear Rate

Table (4) shows the values of wear rate for the squeeze cast unreinforced alloy and the composites. It shows that the composites have lower wear rate than that of the unreinforced alloy due to the squeeze casting and particles effect on increasing the hardness and refining the microstructure.

Figures (18 and 19) illustrate the relationship between wear rate and particles volume fraction for the $Y_2O_3$ /Al-Si composites and Cr<sub>2</sub>O<sub>3</sub>/Al-Si composites respectively. From these figures and table (4), it can be seen that the minimum wear rates for the $Y_2O_3$ /Al-Si composites and Cr<sub>2</sub>O<sub>3</sub>/Al-Si also occur at a volume percentage of 8 and 7.6 respectively. This is in agreement with the results of microhardness in which higher hardness had led to lower wear rate.

### Table 4,

Wear Rate of the Alloy and the Composites Squeeze Cast at 700°C Pouring Temperature, 200°C Die Temperature and 53 MPa Squeeze Pressure for 30 sec.

Material	Wear rate (cm <sup>3</sup> /cm) *10 <sup>-9</sup>
Al-Si alloy	13.53
2wt <sup>2</sup> /Y <sub>2</sub> O <sub>3</sub> /Al-Si composites	7.66
4wt <sup>2</sup> Y <sub>2</sub> O <sub>3</sub> /Al-Si composites	8.74
6wt <sup>/</sup> Y <sub>2</sub> O <sub>3</sub> /Al-Si composites	7.44
8wt <sup>2</sup> Y <sub>2</sub> O <sub>3</sub> /Al-Si composites	13.01
2wt <sup>2</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al-Si composites	6.8
4wt <sup>7</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al-Si composites	7.44
6wt <sup>2</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al-Si composites	5.2
8wt <sup>2</sup> /Cr <sub>2</sub> O <sub>3</sub> /Al-Si composites	9



Fig.18. Wear Rate Versus Y<sub>2</sub>O<sub>3</sub> Volume Fraction.



Fig.19. Wear Rate Versus Cr<sub>2</sub>O<sub>3</sub> Volume Fraction.

### 4. Conclusions

- 1) The Al-Si alloy produced by squeeze casting has refined microstructure and a density close to the theoretical one.
- The Y<sub>2</sub>O<sub>3</sub>/Al-Si composites and Cr<sub>2</sub>O<sub>3</sub>/Al-Si composites produced by squeeze casting have more microstructure refinement, higher hardness and lower wear rate than the unreinforced alloy.
- The density decreases slightly with increasing particle volume fraction for Y<sub>2</sub>O<sub>3</sub>/Al-Si composites.
- 4) The density increases with increasing the volume fraction for  $Cr_2O_3/Al$ -Si composites.

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# التركيب المجهري وبعض الخصائص للمواد المتراكبة نوع الالمنيوم- سليكون المقواة بأوكسيد الايتريوم او أوكسيد الالومينا

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### الخلاصة

تم در اسة تحضيرمادة متراكبة ذات اساس من سدبيكة الألمنيوم-سد ليكوزة واة بدفائق اوكسديد الكروم (الألمنيوم-سليكون/اوكسيد الكروم) و دقائق اوكسيد الايتريوم (الالمنيوم- سليكون/ اوكسيد الايتريوم) بطريقة السباكة بالعصر المباشر. تم اضافة كسور حجمية مختلفة من دقائق اوكسيد الكروم) و دقائق (% 2.10 , 9.4 , 12.5 ) وكذلك اضافكسور حجمية هختلفة من اوكسيد الايتريوم (% 2.1 , 10.1 , 12.1 , 16.1 ) اجريت عملية خلط منصه بهر سبيكة الالمنيوم- سليكون/ اوكسيد الايتريوم او منصهر سبيكة الالمنيوم- سليكون/ اوكسيد الايتريوم (% 2.1 , 10.1 , 10.1 ) اجريت عملية خلط منصه بهر سبيكة الالمنيوم- سليكون/ اوكسيد الايتريوم او منصهر سبيكة الالمنيوم- سليكون/ اوكسيد الكروم ستخدام خلط كبر بائي وبسرعة ٥٠٠ دورة سبيكة الالمنيوم- سليكون/ اوكسيد الايتريوم او منصهر سبيكة الالمنيوم- سليكون/ اوكسيد الكروم ستخدام خلط كبر على وبسرعة ٥٠٠ دورة لمقية ق منابيكة الالمنيوم- سليكون/ اوكسيد الايتريوم او منصهر سبيكة الالمنيوم- سليكون/ اوكسيد الكروم ستخدام خلط كبر كبورية المنورة ليقية ق م استعمال المتغيريك مثل ضد خط العصدر (53MPa) رجة حرارة الصدب (2°700) زمن الكبس (.30sec) زمين التريث (.5sec) ودرجة حرارة القالب ( C) 200°C) . تم فحص البنية المجهرية للمواد المتراكبة ، الصلادة ومعدل البالظهرت النتائج حصول تنعيم في البنية المجهرية في السد بائك المقواة مقارنة مع السبيكة غير المقواة . كما اظهرت النتائج زيادة في الصلادة وانخفاض في معدل البلى في السبائك المقواة مقارنة مع السبيكة غير المقواة .