

PAPER • OPEN ACCESS

## Effect of ZrO<sub>2</sub> Addition on Mechanical Properties and Microstructure of Al-9Zn-6Mg-3Si Matrix Composites Manufactured by Squeeze Casting

To cite this article: Muhammad Syarifudin *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **517** 012001

View the [article online](#) for updates and enhancements.



**IOP | ebooks™**

Bringing you innovative digital publishing with leading voices to create your essential collection of books in STEM research.

Start exploring the collection - download the first chapter of every title for free.

# Effect of ZrO<sub>2</sub> Addition on Mechanical Properties and Microstructure of Al-9Zn-6Mg-3Si Matrix Composites Manufactured by Squeeze Casting

Muhammad Syarifudin, Elisabeth Nadya Hale, Bondan Tiara Sofyan

Department of Metallurgy and Materials Engineering, Faculty of Engineering, Universitas Indonesia, Kampus UI Depok 16424, Indonesia

**Abstract.** Steel is used because of its high strength and toughness, but it has high density, therefore lighter material with comparable toughness is developed. One alternative is aluminum matrix composite with zirconia (ZrO<sub>2</sub>) as the reinforcement with high fracture toughness. Al-9Zn-6Mg-3Si (wt. %) composites were developed with addition of 2.5, 5, and 7.5 vol. % ZrO<sub>2</sub> through squeeze casting. To improve toughness, the composite was solution treated at 450 °C for 1 h, then aged at 200 °C for 1 h. Materials characterization included Optical Emission Spectroscopy (OES), Rockwell B hardness testing, impact testing, fractography analysis, microstructure analysis using Optical microscope (OM) and Scanning Electron Microscope (SEM) / Energy Dispersive X-Ray Spectroscopy (EDS), as well as X-Ray Fluorescence (XRF). The results showed that the more ZrO<sub>2</sub> particles, the higher porosity and the lower the hardness and the impact values, both in as-cast condition and after ageing at 200 °C at 1 h.

## 1. Introduction

Steel which is known to have high strength and high toughness is commonly used in several applications. Still, the high specific mass of steel (7.85 g/cm<sup>3</sup>) makes it less effective. Therefore, metal matrix composites (MMC) with light matrix and high toughness reinforcement are developed as alternative materials.

Aluminium 7xxx contains zinc, magnesium, and copper as its main alloying elements. It is suitable to be used as composite matrix considering its low density along with superior strength and toughness compared to the other aluminium alloy series. Zirconia (ZrO<sub>2</sub>) is used as the reinforcement to promote the fracture toughness and strength due to its value of fracture toughness of 13 MPa.m<sup>-0.5</sup>, Young Modulus of 190 GPa, and hardness of 1250 HV [1].

Zirconia may increase the strength of the aluminium matrix composite with two mechanisms, the first one being grain refinement and the second one being the increment of dislocation density as a result of the difference in thermal expansion coefficient between ZrO<sub>2</sub> and aluminium. Previous research conducted by Madhusudhan [2] showed that the addition of up to 8% ZrO<sub>2</sub> particles in Al-8Zn-3Mg-2Cu alloy increased the hardness and strength. However Kharizma [3] explained that higher addition of ZrO<sub>2</sub> on Al-13.1Zn-6.1Mg-6.7Si-1.4Cu alloy, resulted in higher porosities due to the low wettability of ZrO<sub>2</sub> particles in molten aluminium [4].

The presence of porosity will lower the strength and hardness of the aluminium matrix composite, therefore squeeze casting should be used to enhance heat transfer through the contact between the molten metal with the pressurized hydraulic plate. It will result in fine grained products with minimum porosity and shrinkage. Yang [5] concluded that squeeze casting will reduce the solidification time of the molten aluminium that improved mechanical properties. Another research by Shi et. al [6] proved that the optimum pouring temperature for squeeze casting method of aluminium alloy is 690~700 °C that produced the highest tensile strength and elongation, since the grains are not overly grown. This research



studies Al-9Zn-6Mg-3Si composites with addition of 2.5, 5, and 7.5 vol. % ZrO<sub>2</sub> particles produced by squeeze casting. Further ageing at 200 °C for 1 h aimed to increase toughness and hardness without significantly embrittling the materials. Since ageing can generate second phases which are intermetallic compounds, specifically MgZn<sub>2</sub> in Aluminium 7xxx. These intermetallic compounds may appear in different shapes including needle-like, oval, or even Chinese-script which increase the toughness of the aluminium composites.

## 2. Materials and Method

This study used Al-9Zn-6Mg-3Si (wt. %) alloy as the matrix and ZrO<sub>2</sub> as the reinforcement, with the variation of 2.5, 5, and 7.5 (vol. %). Input materials were Al, Al-20Si, Zn and Mg ingots that were melted in a crucible furnace at 850 °C. Degassing was conducted by using argon gas for 60 seconds. Zirconia particles were previously heated at 1100 °C and then added to the molten metal while stirred for 30 s. The molten composite was poured in 170x70x55 mm<sup>3</sup> metal mould and then squeezed at 76 MPa for 10 min. The composites were then homogenized at 450 °C for 24 h. Table 1 presents the obtained composition of the aluminium composites tested by Optical Emission Spectroscopy (OES) Thermo Scientific ARL 2000.

Rockwell hardness test were conducted in accordance to ASTM E18 with three indentation for each measurement. Charpy impact test referred to ASTM E23 standard and metallographic standard preparation was used with 0.5 % HF etchant. Microstructures were obtained by using Optical Microscope (OM), SEM-EDS and analyzed by ImageJ Software. X-Ray Fluorescence (XRF) Portable X-Spectra, and X-Ray Diffraction (XRD) tests were also performed.

The samples were then solution treated at 450 °C for 1 h followed by water quenching and aged at 200 °C for 1 h. The aged samples were also characterized with the same methods.

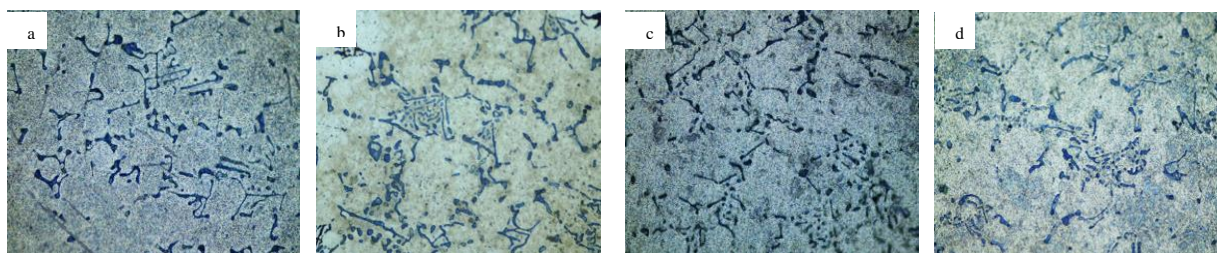
**Table 1.** The actual composition of the aluminium composite

Element	Composition (wt. %)			
	Composite A (0 % ZrO <sub>2</sub> )	Composite B (2.5 % ZrO <sub>2</sub> )	Composite C (5 % ZrO <sub>2</sub> )	Composite D (7.5 % ZrO <sub>2</sub> )
Zn	9.66-9.72	9.98-10.36	9.69-9.78	10.17-10.36
Mg	5.27-5.39	9.66-10.51	4.55-4.61	4.66-5.07
Si	2.65-2.67	2.49-2.51	2.64-2.66	2.57-2.58
Al	<i>Bal.</i>	<i>Bal.</i>	<i>Bal.</i>	<i>Bal.</i>

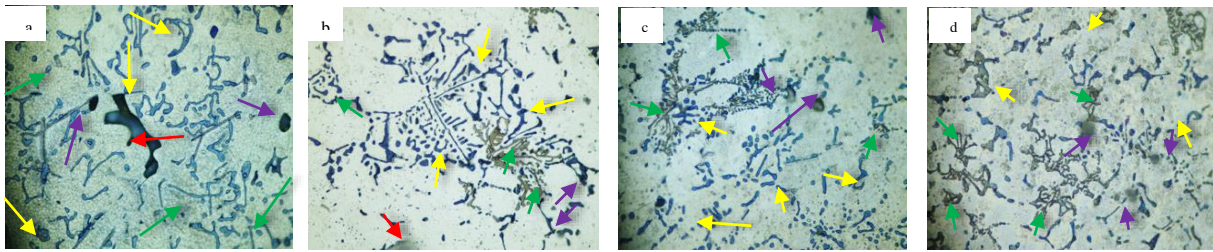
## 3. Results and Discussion

### 3.1 Microstructural Characterization

Figs. 1 and 2 show that with higher ZrO<sub>2</sub> particles content, the more porosities found in the microstructures in both as-cast and aged Al-9Zn-6Mg-3Si composites, which are indicated by arrows in the microstructure. In Fig. 2 the yellow arrows show the silicon interdendritic phases and the green arrows point to chinese script structures which are the intermetallics. Meanwhile the red and purple arrows are shrinkages and porosities, respectively, that increase by the addition of ZrO<sub>2</sub> particles. The porosities appear to be much larger as the ZrO<sub>2</sub> content higher.

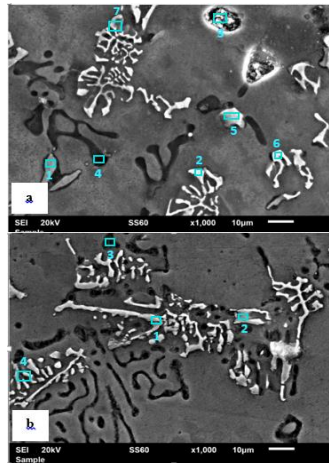


**Figure 1.** Microstructure of as-cast Al-9Zn-6Mg-3Si with (a) 0, (b) 2.5, (c) 5, and (d) 7.5% ZrO<sub>2</sub> particles.



**Figure 2.** Microstructure of aged Al-9Zn-6Mg-3Si with (a) 0, (b) 2.5, (c) 5, and (d) 7.5% ZrO<sub>2</sub> particles. Yellow, green, red and purple arrows indicate silicon, chinese script, shrinkages and porosities, respectively.

SEM micrographs of Al-9Zn-6Mg-3Si composites with 0 and 2.5 % ZrO<sub>2</sub> particles are displayed in Fig. 3 while Table 2 presents EDS characterization at complementary positions. In both figures, EDS examination did not show any content of ZrO<sub>2</sub> particles. However, in Table 3, which presents XRF characterization in the composite with 2.5 vol. % ZrO<sub>2</sub>, Zr element is detected, proving the ZrO<sub>2</sub> reinforcement is there. This difference take place as a result of uneven ZrO<sub>2</sub> distribution, for some parts of ZrO<sub>2</sub> particles gathered in a porosity and the others is settled down inside the crucible.



**Figure 3.** SEM micrographs of the aged Al-9Zn-6Mg-3Si composite with (a) 0 and (b) 2.5 vol % ZrO<sub>2</sub> particles

**Table 2.** Microanalysis at points shown in Fig. 3(a)

Point	Element (% at.)							Phase
	Zn	Mg	Si	O	C	Fe	Al	
1	10.89	-	-	-	-	-	89.11	Al
2	7.41	14.66	10.92	8.62	-	0.18	58.21	FeSiAl <sub>8</sub>
3	10.43	-	-	3.31	-	-	86.26	Al <sub>2</sub> O <sub>3</sub>
4	10.09	2.43	-	4.72	-	-	84.53	MgZn <sub>2</sub>
5	8.51	6.64	3.88	4.72	-	-	76.25	Mg <sub>2</sub> Si
6	5.01	2.02	5.04	4.7	15.66	16.65	50.01	Mg <sub>2</sub> Si
7	6.45	-	1.98	6.32	23.04	8.12	54.13	FeAl <sub>3</sub>

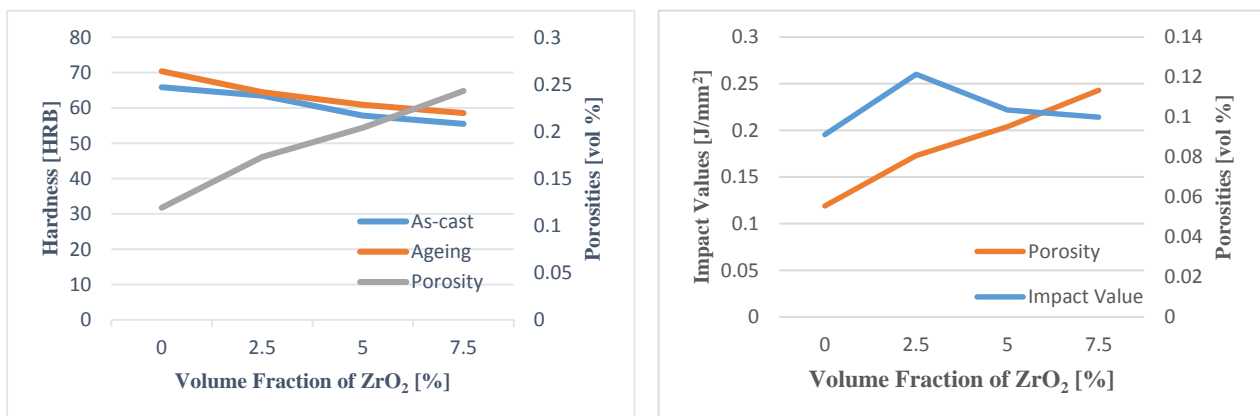
**Table 3.** Microanalysis at points shown in Fig. 3(b)

Point	Element (% at.)							Phase
	Zn	Mg	Si	O	C	Fe	Al	
1	4.72	3.92	4.94	6.15	12.94	9.65	57.49	Mg <sub>3</sub> Zn <sub>3</sub> Al <sub>2</sub>
2	4.41	10.21	5.54	5.64	11.1	8.36	54.75	MgZn <sub>2</sub>
3	1.36	46.49	25.47	5.43	-	-	21.25	Mg <sub>2</sub> Si
4	6.6	-	1.69	5.05	16.49	6.28	63.99	FeAl <sub>3</sub>

The chinese script morphology is proven to be the intermetallic  $Mg_2Si$  and  $FeSiAl_8$  phases (points 2, 5, 6 in Fig. 3(a) and point 3 in Fig. 3(b)). Other intermetallics were  $Mg_3Zn_3Al_2$  (point 1 in Fig. 3(b)) and  $MgZn_2$  (point 4 in Fig. 3(a) and point 2 in Fig. 3(b)). According to Hirsch, et. al [7], these intermetallic phases have a great effect in the formation of porosity since they will reduce metal fluidity and enhance the chance of micro porosity formation.

### 3.2 Mechanical Properties

The hardness of the Al-9Zn-6Mg-3Si composites with 0, 2.5, 5, and 7.5 %  $ZrO_2$  reinforcement is shown in Fig. 4. Overall, Fig. 4 indicates a decrease in the hardness along with the increase in  $ZrO_2$  content, both in the as-cast and aged condition. Samples in aged condition possessed higher hardness values, this happens because after the ageing treatment, precipitation hardening mechanism occurred.



**Figure 4.** Effects of  $ZrO_2$  on, a) the hardness and b) impact values of Al-9Zn-6Mg-3Si composites in as-cast condition and after ageing at 200 °C for 1 h.

These results are affected greatly by the presence of the porosity in the matrix. To confirm the amount of porosities in the samples quantitative analysis was conducted and the results are presented in Fig.4. The porosities are larger and in higher amount with the addition of  $ZrO_2$  particles. These results are in line with previous work [8], which found that addition of  $ZrO_2$  for more than 5% in Al-9Zn-6Mg-3Si composites diminished the strengthening effects due to the presence of porosities. With the increasing porosity, it is confirmed in Fig. 4 that the hardness value of the composites which contains more  $ZrO_2$  particles is lower than the composites with the less  $ZrO_2$  particles. This explains the great difference between the actual hardness and theoretical hardness calculated with the rules of mixture shown in Table. 4 that indicates the effect of the porosities is very dominant compared to the strengthening effect of reinforcement and ageing.

**Table 4.** Calculated results of theoretical and actual as-cast and aged composites hardness

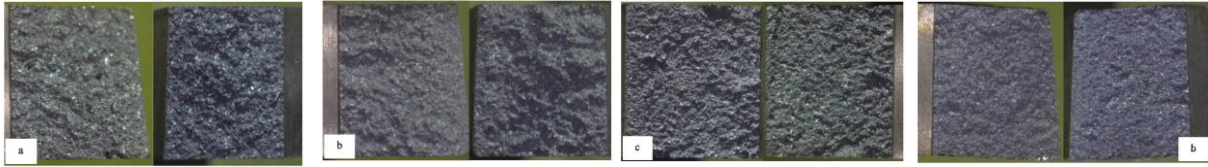
Volume Fraction of $ZrO_2$ [vol. %]	Actual Hardness [HRB]	Theoretical Hardness without porosity [HRB]
0	65.85	65.93
2.5	63.45	92.98
5	57.82	120.03
7.5	55.47	147.08

Volume Fraction of $ZrO_2$ [vol. %]	Actual Hardness [HRB]	Theoretical Hardness without Porosity [HRB]
0	70.37	70.56
2.5	64.45	97.50
5	60.87	124.43
7.5	58.53	151.37

The result shown in Fig. 4 displays an overall decrease in the impact value. This supports the research conducted by Aqida, et. al [9] that the presence of porosity will cause a reduction in the toughness of the materials. This happens because the porosities are the crack initiation and propagation path when the load is given so the energy absorbed is lower.

The fractography images of the Al-9Zn-6Mg-3Si composites in Fig. 5 show brittle fractures in all 0, 2.5, 5, and 7.5 vol% ZrO<sub>2</sub>.



**Figure 5.** The macro fractography impact testing result of Al-9Zn-6Mg-3Si composites with (a) 0, (b) 2.5, (c) 5, and (d) 7.5 vol% ZrO<sub>2</sub> particles.

#### 4. Conclusions

The study of ZrO<sub>2</sub> reinforcement addition with variation of 0, 2.5, 5, and 7.5 vol% in Al-9Zn-6Mg-3Si composite resulted in the following conclusions :

- 1) Addition of ZrO<sub>2</sub> particles correlated with formation of porosities so that reduced the hardness and impact values of the composites.
- 2) Ageing at 200 °C for 1 h increased the hardness of the composites which may be due to the formation of precipitates.
- 3) The ZrO<sub>2</sub> particles were not distributed evenly because of the poor wettability with the molten Aluminium resulting in the formation of porosity in the composite.
- 4) Special treatment for the ZrO<sub>2</sub> particles is needed to reduce porosity including addition of wetting agent.

#### 5. Acknowledgement

The authors are grateful with the provision of research funding through *Hibah Publikasi Internasional Terindeks untuk Tugas Akhir* (PITTA) 2018 from Universitas Indonesia with contract no. 2401/UN2.R3.1/HKP.05.00/2018.

#### 6. References

- [1] J. Park, *Bioceramics: Properties, Characterizations, and Applications* (2008) USA: Springer Science-Business Media, LLC.
- [2] M. Madhusudhan, G. J. Naveen, K. Mahesha, Mechanical Characterization of AA7068-ZrO<sub>2</sub> reinforced Metal Matrix Composites . *Mat. Today Proceedings*. 4 (2017) 3122-3130.
- [3] R.K. Agrista, *Studi Pengaruh Penambahan Penguat ZrO<sub>2</sub> Terhadap Karakteristik Komposit Al-13.1Zn-6.1Mg-6.7Si-1.4Cu Hasil Squeeze Casting Untuk Aplikasi Balistik (Study of ZrO<sub>2</sub> Reinforcement Particles Addition on Characteristics of Al-13.1Zn-6.1Mg-6.7Si-1.4Cu Composites Manufactured by Squeeze Casting for Ballistics Application)*. (2014). Skripsi. Depok: Universitas Indonesia.
- [4] S.A. Khorramie, M. A. Baghchesara, D. P. Gohari, Fabrication of aluminum matrix composites reinforced with Al<sub>2</sub>ZrO<sub>5</sub> nano particulates synthesized by sol–gel auto-combustion method. *Trans. Nonfer. Met. Soc. China*. 23 (2013) 1556-1562.
- [5] L.J. Yang, The effect of solidification time in squeeze casting of aluminium and zinc alloys. *Mat. Proc. Tech*. 192-193 (2007) 114-120.
- [6] W. Shi, , Squeeze Casting Process and Mould Design for 2A12 Aluminum Alloy Shell. *Spec. Cast. And Nonfer. Alloys*. 38 (2018) 629-631.
- [7] J. Hirsch, B. Skrotzki, G.Gottstein, *Aluminium Alloys, Their Physical and Mechanical Properties* (2002) New Jersey : John Wiley & Sons, Inc.

- [8] H. Abdizadeh, M.A. Baghchesara, Investigation on mechanical properties and fracture behavior of A356 aluminum alloy based ZrO<sub>2</sub> particle reinforced metal-matrix composites. *Cer. Int.* 39 (2013) 2045-2050.
- [9] S.N. Aqida, M.I. Ghazali, J. Hashim, Effects of porosity on mechanical properties of metal matrix composite: an overview. *Jur. Tek.* 40 (2004) 17-32.