

Microstructure and Mechanical Behaviour of Al6061-ZrB₂ In-situ Metal Matrix Composites

R Vasanth Kumar¹, R Keshavamurthy² and Chandra S Perugu³

¹ Department of Mechanical Engineering, Christ University, Bangalore, Karnataka India

² Department of Mechanical Engineering, Dayananda Sagar College of Engineering, Bangalore, Karnataka, India

³ Department of Materials Engineering, Indian Institute of Science, Bangalore, Karnataka, India

vasanth.kumar@christuniversity.in

Abstract. Aluminium matrix composites processed through in-situ molten reaction has emerged as an alternative for eliminating defects existing in ex-situ reinforced metal matrix composites. Development of composites through in-situ method using inorganic salts via liquid metallurgy route is the most widely accepted technique. In the present work, Al6061-ZrB₂ in-situ composites have been developed through in-situ reaction of Al-10%Zr and Al-3%B master alloys in Al6061 alloy. Study of microstructure and mechanical properties of in-situ reinforced ZrB₂ in Al6061 alloy have been carried out. Composite exhibited grain refinement and improved the mechanical properties of Al6061 alloy. Ductility of composite is reduced with increase in content of ZrB₂.

Keywords: Aluminium matrix composite, ZrB₂, In-situ, Master alloy, Mechanical properties.

1. Introduction

Increased demands of current industries for improved material performance have necessitated the use of particulate reinforced metal matrix composites (PMMCs). Aluminium is identified as important material in a manufacturing system for exhibiting light weight with high strength in automobile and aerospace applications [1]. Aluminium metal matrix composites (AMMCs) possess improved properties when compared with monolithic alloys for high strength-weight ratio, high wear resistance and low thermal expansion [2,3]. With this requirement, Al6061 alloy has proved its existence for good workability, good weldability and excellent corrosion resistance [4]. Silicon carbide and aluminium oxides were extensively used as reinforcement during earlier decades of developing AMMCs [5]. Newer trends have been existed in using different reinforcements such as silicon nitride [6,7], boron carbide [8], aluminium oxide [9], titanium oxide [10], titanium boride [1] for fabrication of PMMCs. Among various reinforcements tried out till date, ZrB₂ stands as a popular material with strong covalent bonding with very high melting temperature, superior hardness and strength along with excellent thermal conductivity and thermal shock resistance, to exist as a suitable candidate in critical environments associated with aerospace industry [11]. Composites can be fabricated by two casting methods [1], firstly by adding the powder externally to the melt through ex-situ synthesis



(Eg. Liquid ingot casting and Powder metallurgy) and later by synthesizing the particles within the melt through in-situ synthesis (Eg. Reactive hot pressing, Reactive infiltration, exothermic dispersion and direct melt reaction). Poor wettability, reduced bonding and non-uniform distribution have limited the use of ex-situ castings in engineering applications [12,13]. It has become very essential to eliminate ex-situ synthesis and promote in-situ reactions for meeting the demands of industrial applications [14,15,16]. However, the direct melt reaction process requires agitated time at high temperatures to acquire full incorporation through the response of the additional reactants with liquid metal [17].

Literature has been focused on aluminium composites processed through in-situ technique with different reinforcements in the form of fine particles to investigate the nature and morphology of metal matrix composite and their behaviour. Dinharan et al. [5,18] has developed AA6061 alloy reinforced with ZrB_2 produced by an in-situ reaction between K_2ZrF_6 and KBF_4 salts. Hardness and strength of composite increases by increasing ZrB_2 content in matrix alloy. Ramesh et al. [1] has developed Al6061-TiB₂ composites through in-situ method using Al-Ti and Al-B master alloys. Tensile strength and Ductility increases in composite with higher content of ZrB_2 elements. Moosavian et al. [19] has prepared ZrB_2 /A356 alloy by in-situ technique using Al-15Zr and Al-8B master alloy. Microscopic study, XRD analysis and Tensile test of extruded composite has been investigated before and after T6 heat treatment and compared with matrix alloy. Tensile test reveals that ultimate tensile strength (UTS) and ductility increases compared to matrix alloy with further increase in UTS and decreasing ductility after T6 heat treatment. Songli Zhang et al. [20] has developed Al-ZrB₂ composites with formation of Al_3Zr through in-situ reaction and investigated microstructure at room temperature. Morphologies of ZrB_2 and Al_3Zr elements were in regular tetragonal and hexagonal shapes with a size of about 0.3-0.5 μm . Yutao Zhao et al. [21] has fabricated ($Al_3Zr+ZrB_2$)/Al composites from Al- K_2ZrF_6 - KBF_4 system and investigated the morphology variations of Al_3Zr crystal in composites due to the effect of molten temperature. Effect of temperature is more sensitive on morphology of Al_3Zr particles compared to ZrB_2 element.

In this paper, major focus is on developing light weight material with enhanced mechanical characteristics for automobile and marine applications. Until now, very less information is available on synthesis of Aluminium based composites using Al-Zr and Al-B master alloys via in-situ reaction.

The present work focuses on the preparation of Al6061-ZrB₂ in-situ composite processed by Al-10%Zr and Al-3%B master alloy technique. Al6061 was chosen as matrix alloy owing to its excellent castability, formability and weldability for more application on industrial sector. Further, this alloy can be strengthened by suitable heat treatment [1].

2. Experiments

2.1. Manufacturing of composites

Al6061 alloy, Al-10%Zr and Al-3%B master alloy were used as the raw materials for preparation of composites. Raw materials have been procured from M/s Fenfee metallurgical, Bangalore, India. The weight percentage of Al-10%Zr and Al-3%B Master alloys are reported in Table 1. The chemical composition of raw materials used for fabrication of composite is reported in Table 2.

Table 1. Weight percentage of Al-10%Zr and Al-3%B master alloys.

Sample	Weight % of Master alloys
Sample- X	Al6061 + 10%Al-10%Zr and 30%Al-3%B
Sample- Y	Al6061 + 20%Al-10%Zr and 60%Al-3%B

Table 2. The Chemical composition of Al6061 alloy and Al-10%Zr and Al-3%B Master alloys.

Al6061 alloy		Al-10%Ti master alloy		Al-10%Ti master alloy	
Element	%	Element	%	Element	%
Mg	0.802	Zr	10.2	B	2.9
Si	0.809	Fe	0.25	Fe	0.25
Fe	0.155	others	0.5	others	0.5
Cu	0.355	Al	Balance	Al	Balance
Mn	0.027				
Pb	0.023				
Zn	0.008				
Ti	0.01				
Al	Balance				

Al-6061 alloy, Al-10%Zr and Al-3%B master alloys were melted in graphite crucible up to 850⁰C in a stoichiometric ratio using 6KW electrical resistance furnace. The composite melt was maintained for duration of 30mins for completion of in-situ reaction. The melt was agitated by stirring intermittently for every 10mins using mechanical stirrer to ensure uniform mixing of in-situ products. After removing the slag, commercially available Hexachloroethane tablets were used for degassing the molten alloy followed by pouring into preheated metal die.

2.2. Characterization

The composites were cut into different samples of required size for conducting optical microstructure study, SEM, XRD and Tensile tests. The sample was polished using standard metallographic technique and etched with Kellers reagent. The microstructure of the polished specimen was captured using ZEISS metallurgical microscope. SEM study was carried out on a metallographically polished sample using JSM 840a Jeol scanning electron microscope. Further, samples were characterized by XRD and diffraction patterns were recorded. Microhardness test was performed on a polished specimen. Values were recorded at five different locations and average value was considered. Tensile tests for matrix alloy and composites were carried out on an Instron 30KN load Universal testing machine as per ASTM E8M-01 standard.

An average of three results was considered as strength of each material. Ductility was also calculated to analyze the ability of the samples for subjecting it to the secondary process.

3. Results and Discussion

3.1. Microstructure

Figure1-3 shows the optical micrographs of Al6061 alloy and Al6061-ZrB₂ In-situ metal matrix composites. A microscopic image clearly indicates the distribution of irregular plate shaped ZrB₂ particles throughout the matrix alloy. Grain refining action of ZrB₂ particles are clearly observed in the composite.

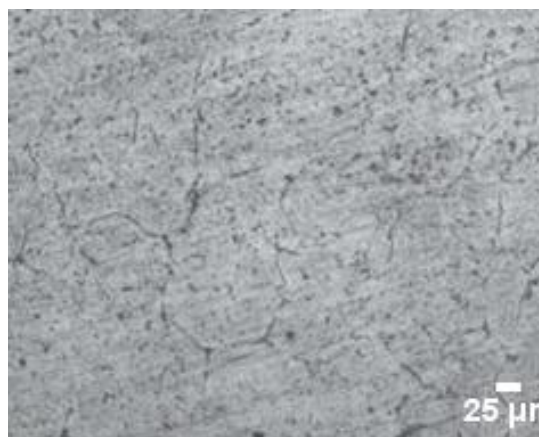


Figure 1. Photographs of Al6061 alloy.

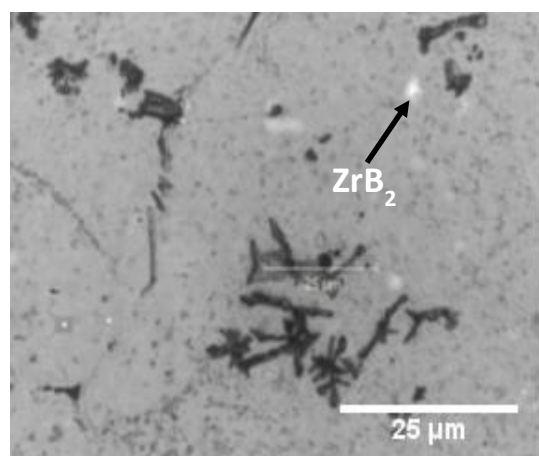


Figure 2. Photographs of Al6061-ZrB₂ in-situ composite: (a) Sample X.

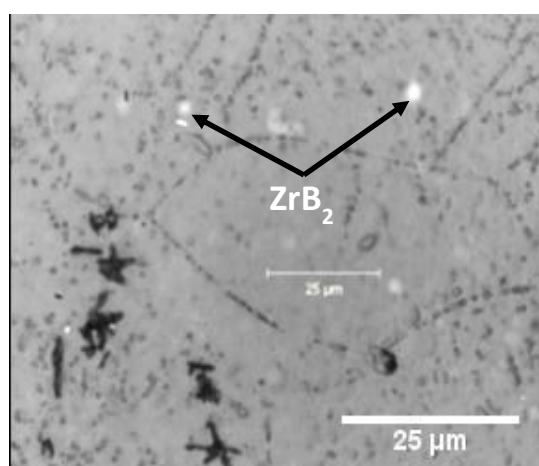
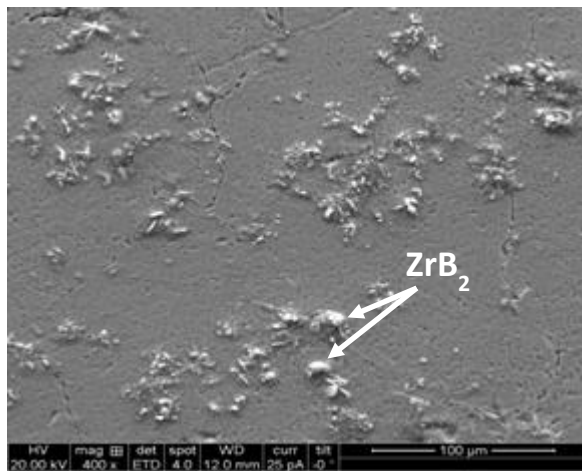
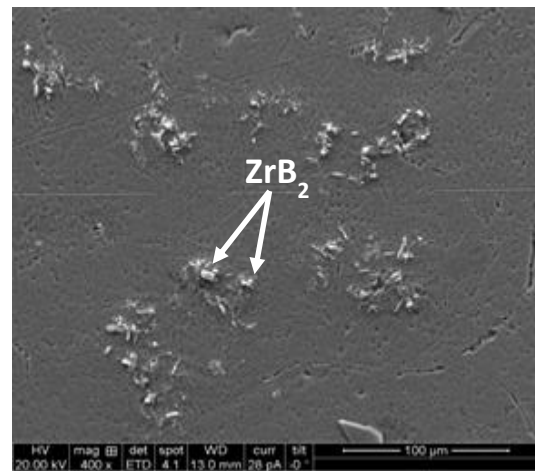


Figure 3. Photographs of Al6061-ZrB₂ in-situ composite: (a) Sample Y.



Sample X



Sample Y

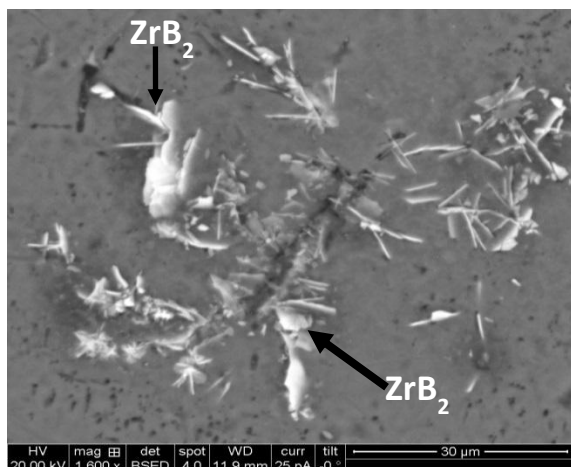
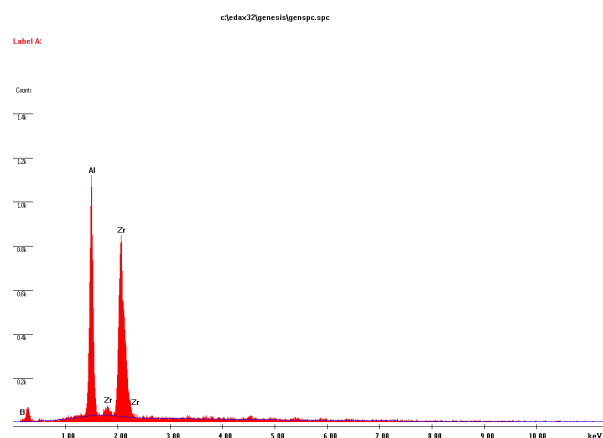
Figure 4. SEM images of Al 6061-ZrB₂ in-situ composite.**Figure 5.** SEM of Al6061- ZrB₂ composite.**Figure 6.** EDAX of Al6061- ZrB₂ composite.

Figure 4-5 shows the SEM micrographs of Al6061- ZrB₂ and Figure 6 shows EDAX analysis of Al6061- ZrB₂ composite. SEM images clearly show the morphology of ZrB₂ particles and EDAX analysis indicates the presence of Zirconium and Boron element.

Due to formation of ZrB₂ particles inside the melt, chances of oxidation of element are avoided. These particles free from oxide surfaces exhibits improved interfacial properties in the composites. Thus, non-oxidized composites reduce the wettability angle between matrix alloy and ZrB₂ casting and resulting in excellent wettability [5]. It is known that uniform distribution of reinforced elements in matrix alloy is mainly due to solidification process in composite. Reduction of wettability angle between ZrB₂ particle and melt resists the movement of ZrB₂ particles during solidification. Subsequently, the ZrB₂ particles are seen inside the grain without entering to the grain boundaries.

3.2 X-ray diffraction analysis

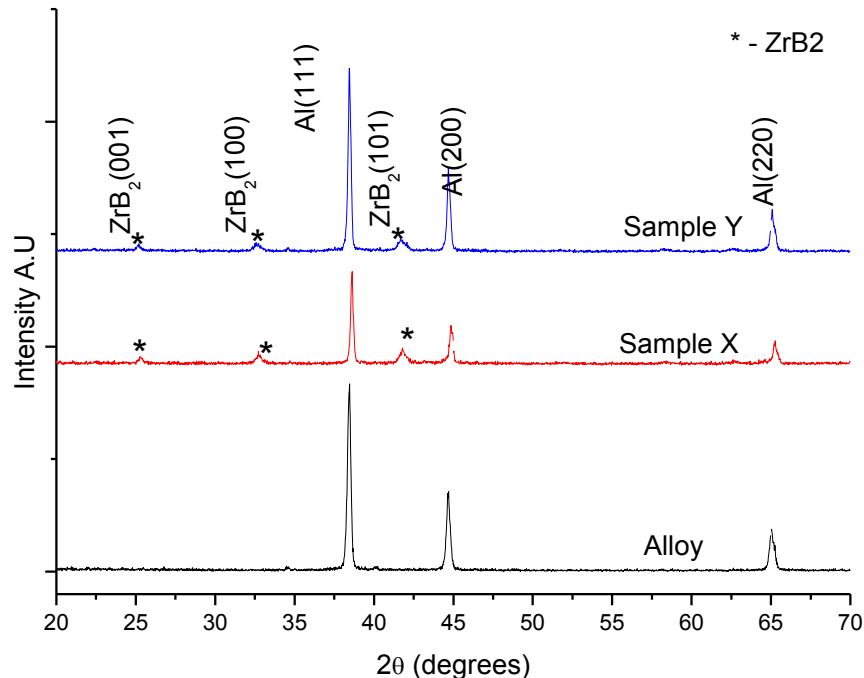


Figure 7. XRD results of fabricated PMMCs by in-situ method.

Figure 7 shows X-ray diffraction pattern of Al6061-ZrB₂ composites. XRD peaks confirm the formation of ZrB₂ particles in the developed composites.

3.3 Grain Size Analysis

Figure 8 shows the grain size of Al6061 alloy and Al6061- ZrB₂ In-situ composite. It is observed that grain size of the composite has been reduced due to the presence of ZrB₂ particles [1]. ZrB₂ itself acts as a nucleating agent for grain refinement in composite [22]. Adding of inoculant particles into the melt results with potential composite developing the heterogeneous nucleation with the presence of solutes [1]. Solute has an ability to undercool the zone constitutionally within the interface [22]. Further, undercooling initiates the nucleation process with activation of nucleants in the region of the interface. This results with constraining of grain growth leading to effective refinement in grains [23]. In the present work, Zirconium act as a solute and ZrB₂ acts as nucleating agent initiating heterogeneous nucleation within the melt. Grain refinement is exhibited due to the presence of solute and nucleating agent. Thus, Zirconium elements have triggered the grain refining action due to constitutional undercooling in the vicinity of the interface. Further, the presence of boron element is also an attribute for grain refinement [24].

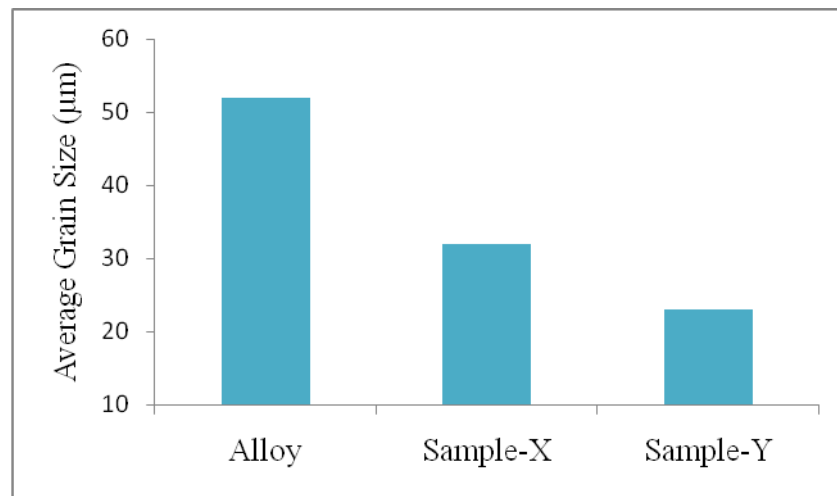


Figure 8. Grain size of samples.

3.4 Microhardness

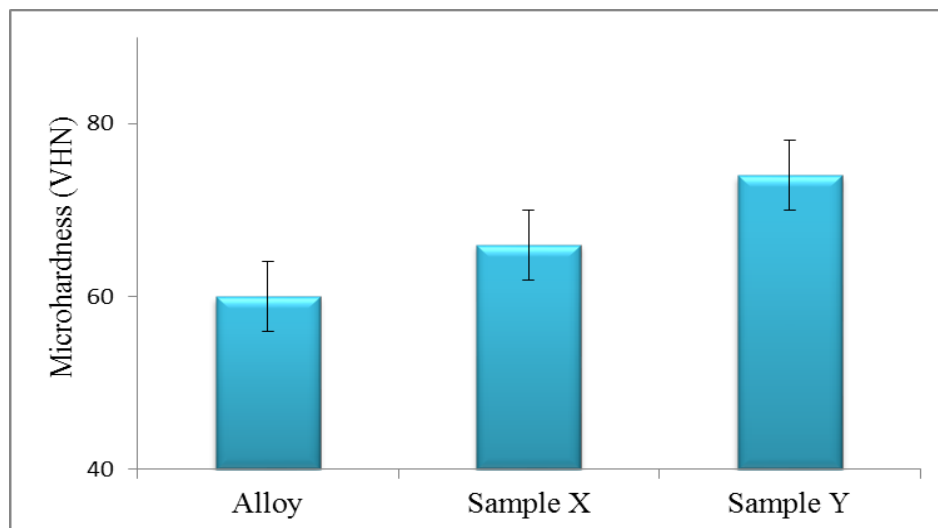


Figure 9. Microhardness of Al6061 alloy and Al6061-ZrB₂ in-situ composite.

Figure 9 shows the variation of microhardness in Al6061 alloy and Al6061-ZrB₂ in-situ composite. It is observed that the presence of ZrB₂ particles has improved the microhardness of composite compared with unreinforced alloy. During the process of solidification in cast composites, ZrB₂ elements may lead to increase in dislocation density [5]. Since, in-situ technique is adopted; uniform bonding between matrix and reinforcement exists in composite. This is helpful in enhancing the bulk properties of composite. In-situ process -ceramic particles bears the load and offers the resistance [1]. In this work, ZrB₂ acts as a load bearing element and takes maximum load for plastic deformation by increasing the hardness [5]. ZrB₂ being a ceramic material allow material to flow without undergoing deformation, but when it exceeds critical value it causes fracture without further deformation. According to Hall-Petch equation, Hardness can be improved by reduction in grain size [25]. Thus, grain refinement can be an effective cause for enhancing the hardness in ZrB₂ composite. Further, microhardness of composite increases with increase in ZrB₂ particles. Microhardness increases by 10% and 24% respectively in increased content of ZrB₂ in-situ composite.

3.5 Tensile behaviour of composites

Figure 10 and Figure 11 shows the variation of yield strength and ultimate tensile strength of Al6061 alloy and Al6061-ZrB₂ in-situ composite. It is observed that increase in ZrB₂ particles has resulted in an increase in tensile strength of the composite. Further, it is also found that increase in ZrB₂ content has increased the ultimate tensile strength with the reduction in % of ductility [5]. The presence of hard ZrB₂ particles, processing of composite through in-situ method and grain refinement may be the attribute for improvement in tensile strength of composites [25]. Fine particle size in composite has always associated with improved mechanical properties.

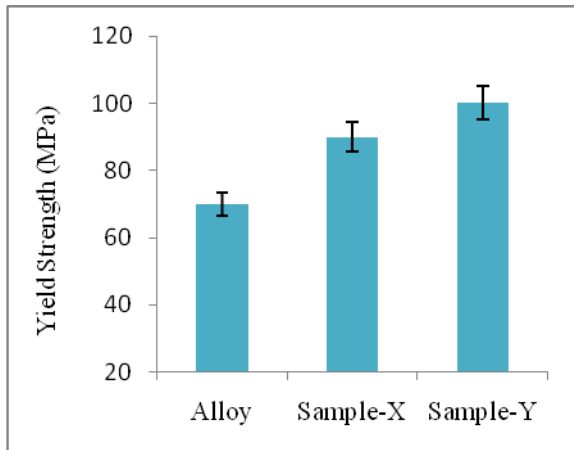


Figure 10. Variation of Yield strength.

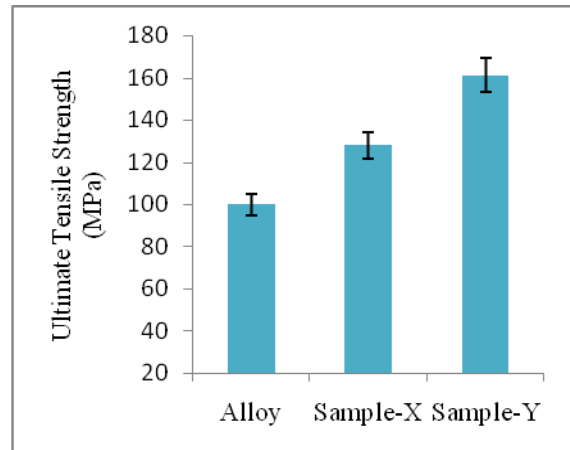


Figure 11. Variation of Ultimate Tensile Strength.

Figure 12 shows the variation of ductility of Al6061 alloy and Al6061-ZrB₂ in-situ composite. It is observed that composite has exhibited with the decrease in ductility with an increase in ZrB₂ content in matrix alloy. Reduction in ductility with an increase in reinforcement may be due to the presence of hard ZrB₂ particles and grain refinement [25]. Grain refinement reduces the ability of structural alloys for ductile-brittle transition. Ductility decreases by 6% and 11% respectively in a composite prepared with an increase in ZrB₂ content.

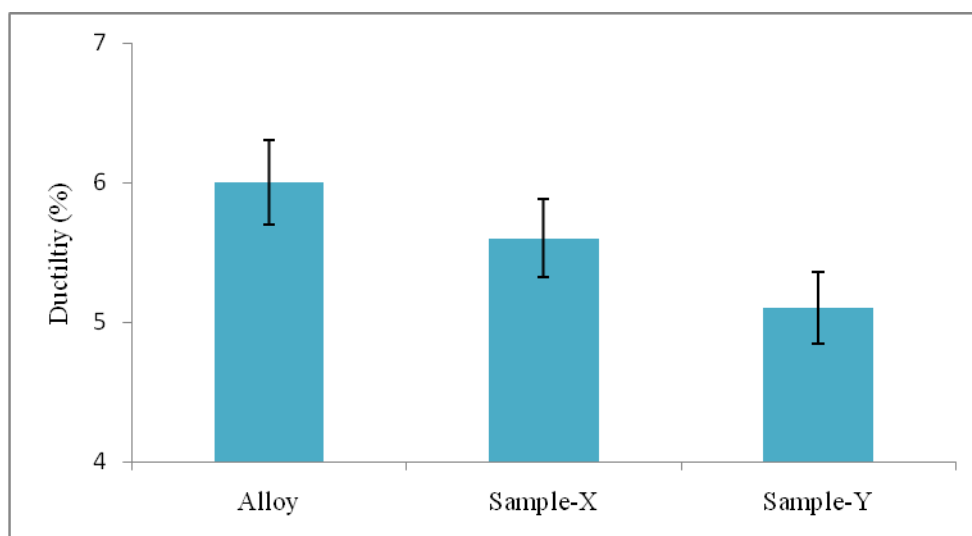


Figure 12. Variation of ductility.

4. Conclusion

Al6061-ZrB₂ in-situ composites were successfully developed through liquid metallurgy using Al6061 alloy and Zirconium and Boron master alloy as reactive elements. Microstructure shows the uniform distribution of reinforced ZrB₂ particles with good bonding between matrix and reinforcement.

Appreciable grain refinement has been observed in composite. Microhardness, yield strength and ultimate tensile strength of composites are higher than matrix alloy. Reduction in ductility of the composite is due to effective grain refinement.

Acknowledgements

The authors wish to express their sincere gratitude to Department of Materials Engineering, Indian Institute of Science, Bangalore for extending the facilities to carry out this investigation.

References

- [1] Ramesh C S, Pramod S, Keshavamurthy R, 2011, *Materials Science and Engineering:A*, A study on microstructure and mechanical properties of Al6061-TiB₂ in-situ composites, **528**, 4125-4132
- [2] Kang Y C, Chan S L I, 2004, *Materials Chemistry and Physics*, Tensile properties of nanometric Al₂O₃ particulate-reinforced aluminium matrix composites, **85**, 438-443
- [3] Zhao Y T, Sun G X, 2001, *Journal of Materials Science Letters*, In situ synthesis of novel-composites in the system Al-Zr-O, **20**, 1859-1861
- [4] Ramesh C S, Keshavamurthy R, Channabasappa B H, Pramod S, 2009, *Materials & Design*, Influence of heat treatment and slurry erosive wear resistance of Al6061 alloy, **30(9)**, 3713-3722
- [5] Dinaharan I, Murugan N, Siva Parameswaran, 2011, *Materials Science and Engineering:A*, Influence of in situ formed ZrB₂ particles on microstructure and mechanical properties of AA6061 metal matrix composites, **528**, 5733-5740
- [6] Ramesh C S, Keshavamurthy R, Channabasappa B H, Abrar Ahmed, 2009, *Materials Science and Engineering:A*, Microstructure and mechanical properties of Ni-P coated Si₃N₄ reinforced Al6061 composites, **502**, 99-106
- [7] Ramesh C S, Keshavamurthy R, Channabasappa B H, Pramod S, 2010, *Tribology International*, Friction and wear behaviour of Ni-P coated Si₃N₄ reinforced Al6061 composites, **43**, 623-634
- [8] Thomas MF Lillom, 2005, *Materials Science and Engineering:A*, Enhancing ductility of Al6061 + 10wt% B₄C through equal-channel angular extrusion processing, **410-411**, 443-446
- [9] Singh J, Alpas A T, 1995, *Scripta Metallurgica et Materialia*, Experimental investigations towards optimization of the parameters for loss quantities in A356/Al₂O₃ Nanocomposites, **32**, 1099-1105
- [10] Ramesh C S, Anwar Khan A R, Ravikumar N, Savanprabhu P, 2005, *Wear*, Prediction of wear coefficient of Al6061-TiO₂ composites, **259**, 602-608
- [11] Sonber J K, Murthy T S R C, Subramanian C, Kumar S, Fotedar R K, Suri A K, 2011, *International Journal of Refractory Metals and Hard Materials*, Investigation on synthesis of ZrB₂ and development of new composites with HfB₂ and TiSi₂, **29**, 21-30
- [12] Kumar S, Chakraborty M, Subramanya Sarma V, Murty B S, 2008, *Wear*, Tensile and wear behaviour of in situ Al-7Si/TiB₂ particulate composites, **265**, 134-142
- [13] Min Zhao, Gaohui Wu, Logtao, Jiang, Zuoyong Dou, 2006, *Composite Part A: Applied Science and Manufacturing*, Friction and wear properties of TiB₂P/Al composite, **37**, 1916-1921
- [14] Shorowordi K M, Laoui T, Haseeb A S M A, Celis J P, 2003, *Journal of Materials Processing Technology*, Microstructure and interface characteristics of B₄C, SiC and Al₂O₃ reinforced Al matrix composites: a comparative study, **142(3)**, 738-743

- [15] Yang B, Chen G, Zhang J, 2001, *Materials & Design*, Effect of Ti/C additions on the formation of Al_3Ti of in situ TiC/Al composites, **22**, 645-650
- [16] Zhang Y, Ma N, Wang H, Le Y, 2005, *Material Letters*, Effect of Fe on grain refining of pure aluminium refined by Al_5TiB master alloy, **59**, 3775-3778
- [17] Sadanandam J, Bikshamaiah G, Gopalakrishna B, Suryanarayana S V, 1992, *Journal of Materials Science Letters*, Effect of different reinforcements on the thermal expansion of 2124 aluminium metal-matrix composites, **11**, 1518-1520
- [18] Dinharan I, Murugan N, 2014, *Journal of Cast Metals Research*, Microstructure and some properties of aluminium alloy AA6061 reinforced in situ formed Zirconium diboride particulate stir cast composite, **27(2)**, 115-121
- [19] Moosavian H, Emamy M, Arani M M, Mahboubi S, 2013, *Key Engineering Materials*, The study of Microstructure and Tensile properties of an In-situ A356- ZrB_2 Metal matrix composites, **553**, ISSN: 1662-9795, 29-33
- [20] Songli Zhang, Yutao Zhao, Gang Chen, Xiaonong Cheng, 2007, *Journal of Materials Processing Technology*, Microstructure and dry sliding wear properties of in situ $(\text{Al}_3\text{Zr} + \text{ZrB}_2)/\text{Al}$ composites, **184**, 201-208
- [21] Yutao Zhao, Songli Zhang, Gang Chen, Xiaonong Cheng, 2007, *Materials Science and Engineering:A*, Effects of molten temperature on the morphologies of in situ Al_3Zr and ZrB_2 particles and wear properties of $(\text{Al}_3\text{Zr} + \text{ZrB}_2)/\text{Al}$ composites, **457**, 156-161
- [22] Mark Easton, David StJohn, 1999, *Mettallurgical and Materials Transaction A*, Grain refinement of Aluminum alloys: Part II-Confirmation of, and a mechanism for, the solute paradigm, **30**, 1625-1633
- [23] Mark Easton, David StJohn, 1999, *Mettallurgical and Materials Transaction A*, Grain refinement of Aluminum alloys: Part I. the nucleant and solute paradigm- a review of the literature, **30(6)**, 1613-1623
- [24] Ramesh C S, Abrar Ahmed, Channabasappa B H, Keshavamurthy R, 2010, *Materials & Design*, Development of Al6063- TiB_2 in situ composites, **31**, 2230-2236
- [25] Niels Hansen, 2004, *Scripta Materialia*, Hall-Petch relation and boundary strengthening, **51(8)**, 801-806
- [26] Feng C Y, Froyen L, 2000, *Journal of Materials Science Technology*, In situ synthesis of Al_2O_3 and TiB_2 particulate mixture reinforced aluminium matrix composites, **35**, 837-850
- [27] Hong S H, Chung K H, 1995, *Materials Science and Engineering:A*, Effects of vacuum hot pressing parameters on the tensile properties and microstructure of SiC – 214 Al composites, **194**, 165-170
- [28] Wood J V, Davis P, Kellie J L F, 1993, *Journal of Materials Science Technology*, Properties of reactively cast aluminium – TiB_2 alloys, **9**, 833-840
- [29] Seyed Reihani S M, 2006, *Materials & Design*, Processing of squeeze cast Al6061-30vol% SiC composites and their characterization, **27**, 216-22
- [30] Parvin R, Assadifard R, Safarzadeh P, Sheibani S, Marashi P, *Materials Science and Engineering:A*, Preparation and mechanical properties of SiC- reinforced Al6061 composite by mechanical alloying, **492**, 134-140
- [31] Zhang Xinghong, Qu Qiang, Han Jiecai, Han Wenbo, Hong Changqing, 2008, *Scripta Materialia*, Microstructural features and mechanical properties of ZrB_2 -SiC-ZrC composites fabricated by hot pressing and reactive hot pressing, **59**, 753-756
- [32] Guo Shu Qi, Yang Jenn Ming, Tanaka Hidehiko, Kagwa Yutaka, 2008, *Composites Science and Technology*, Effect of thermal exposure on strength of ZrB_2 - based composites with nano-sized SiC particles, **68**, 3033-3040
- [33] Narendra Kumar, Rakesh Kumar Gautam, Sunil Mohan, 2015, *Materials & Design*, In-situ development of ZrB_2 particles and their effect on microstructure and mechanical properties of AA5052 metal matrix composites, **80**, 129-136