

Development and Property Evaluation of Copper-Chilled Aluminum Alloy Reinforced with Nano-ZrO₂ Metal Matrix Composites (NMMCS)

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Abstract

The present investigation aims at developing aluminum alloy-nano particles composites (NMMCs) in moulds containing copper chill by reinforcing nano-ZrO₂ particulates in aluminum alloy (LM 13) by vortex method. The size of particulates dispersed varies from 90 to 100nm and the amount of addition varies from 3 to 12wt% in steps of 3%. The resultant composites cast using copper chills were tested for their strength, hardness and fracture toughness. Results of the investigation reveal that presence of nano-ZrO₂ particles as dispersoid (up to 9Wt. %) and VHC of the chill used has improved significantly the strength, hardness and fracture toughness with slight reduction in ductility. The strength of the composite developed is highly dependent near to chill end and also on the reinforcement content present in the composite. Increase in the chilling rate and increase in the reinforcement content of the material both results in an increase in UTS and fracture toughness of the developed composite. Fractography of the specimens showed that the fracture behavior of matrix alloy has changed from ductile intergranular mode to cleavage mode of fracture. Microstructural analysis of the developed nano-composite reveals the uniform distribution of the reinforcement in the matrix alloy with significant grain refinement.

Keywords: Nano-ZrO₂; Copper Chill; vortex method; NMMC; UTS; fracture toughness; microstructure.

1. Introduction

Aluminum alloys based composites that freeze over a range of temperature experience difficulty in feeding as the solidification progress [1, 2]. Porosity results in pasty type of solidification which commonly occurs in long range freezing alloys can be effectively treated by judicious location of chills. Chills transfer heat at faster rates and promote direction solidification. There are several investigations on the influence of chills on the solidification and soundness of long freezing range alloy castings [3-10]. The analysis related to copper chilled NMMC is undertaken because from the examination of literature that no data available on copper chilled Nano-ZrO₂ reinforced composite (NMMCs). Conventional metal matrix composites (MMCs) reinforced with ceramic particulates exhibit high strength and elastic modulus near isotropic properties. These materials of special interest because of their ease of fabrication at relatively low cost. Aluminum based matrix have been used as structural materials in the aerospace and automotive sectors in particular. Large ceramic particulates are prone to cracking during mechanical loading, leading to premature failure and low ductility of the composite. Particulate size has a strong effect on the failure mode, strength and ductility of the Al-based composites. Both tensile strength and ductility decrease with increase in particle size [11-17] therefore decreasing the ceramic particulate size can lead to substantial improvements in mechanical performance of MMCs. Eg: enhanced strengthening and reduced particle cracking. Use of nano particulates as reinforcement in metallic materials has inspired considerable research interest in recent years because of the potential development of novel composites with unique mechanical and physical properties. The production of such nano-metal matrix composites can create new technological opportunities and challenges.

1.1 Volumetric Heat Capacity (VHC) of Chill

The rate at which the heat is extracted from the solidifying molten metal in the mould will depend on the size and properties of chill material. The important parameters such as volume, specific heat and density of the chill materials account in evaluating the chill efficiency, which can relate as given below

$$VHC = V \times C_p \times \rho$$

Where V is the volume of the chill, C_p is the specific heat of the chill material, and ρ is the density of the chill material. During solidification with chill in the mold, the heat transfer takes place through the metal-chill interface during initial stages of solidification. Subsequently, an air gap forms between the casting and the chill face due to dissimilar thermal behavior of the chill and the surface of the thin layer of the solidified metal. This air gap results in temporarily delaying the heat transfer. In the final stages, the heat transfer takes place again in direct contact with casting and the chill surface [18]. The thermal properties of copper chill are shown in the Table 1.

Table 1. Volumetric heat capacity of Copper Chill

Chill material	Density g/cc	Specific heat J/Kg K	Thermal conductivity W/mK	VHC for 35mm chill thick
Copper	8.96	0.448	1.022	835.93

2. Experimental Procedure

2.1. Fabrication of copper chilled NMMC

The chemical composition of the aluminum alloy used as the matrix is given in Table 2. In this investigation, nano-ZrO₂ particulates were dispersed in Aluminum alloy by vortex method. The size of the nano-ZrO₂ particulates dispersed varies from 90 to 100 nm and the amount of addition varies from 3 to 12 Wt.% in steps of 3%. The primary process consists of synthesis of monolithic and nano-sized ZrO₂ reinforced Al composites containing four different weight percentage of ZrO₂ was carried out using vortex method. The process involved heating of Al alloy in a graphite crucible up to 750°C to which the preheated reinforcement (up to 450°C) was added and stirred well by an mechanical impeller rotated at 400 rpm to create vortex and to get uniform distribution of the reinforcement in the liquid melt. The process of dispersing the particles is completed within one minute. After the complete injection of the dispersoid in the molten metal, it is poured into a green sand mould containing copper chills. The mould for the plate type of casting (225x150x25mm (AFS standard)) as shown in Fig. 1 were prepared using silica sand with 5% bentonite as binder and 5% moisture and dried in an air furnace. The matrix mixture was poured into the mould at a pouring temperature of 730°C, which was cooled from one end by a copper chill set in the mould as shown in Fig. 1. Ingots were also cast with and without employing copper chill in order to study the effect of chilling on strength, fracture toughness and microstructure of the composite developed. The copper chill dimension remained 170mmX35mmX35mm.

Properties of reinforcement (ZrO₂) are as follows. Density: 8.1 gm./cm³, melting point: 1860°C, UTS: 425 MPa, VHN: 150, young's modulus: 98 GPa (ZrO₂ is supplied by nano structured and Amorphous Materials, Inc., USA).

Table 2 Chemical Composition of Matrix alloy

Elements	Zn	Mg	Si	Ni	Fe	Mn	Al
Wt. %	0.5	1.4	12	1.5	1.0	0.5	Bal

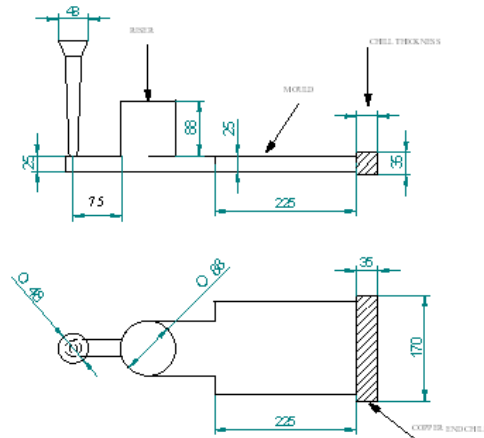


Fig.1. Green Sand mould for Production of NMCs

3. Specimen Selection and Testing Procedure

The specimens were selected near to chill end. The longitudinal axes of these specimens were parallel to the longitudinal axis of the copper chill set during casting. Ageing is carried out before all the test specimens were prepared. The ingots were placed in heattreatment furnace and its temperature is pre-set to $450^{\circ}\text{C} \pm 5^{\circ}\text{C}$ then soaked for four hours followed by ambient cooling.

Micro structural characterization studies were conducted on polished NMMC specimens using OLYMPUS metallographic microscope to investigate morphological characteristics of grains, reinforcement distribution and interfacial integrity between the matrix and enforcement. Brinell hardness measurements were made on the polished specimens. The specimens for the tensile test were prepared according to AFS standard tensometer specimens in accordance with ASTM E8M-01 standard using instron 8516 machine. Fracture toughness tests were performed using a closed loop Instronsevo-hydraulic material testing system. This method involves 3-point bend testing (in accordance with ASTM E 399 1990 standard) of machined specimen which have been pre-cracked by fatigue. Fracture toughness test specimens are shown in the Fig. 2 and 3.

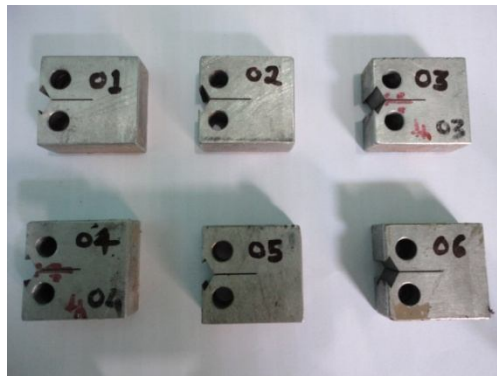


Fig. 2 Photograph of CT Specimens

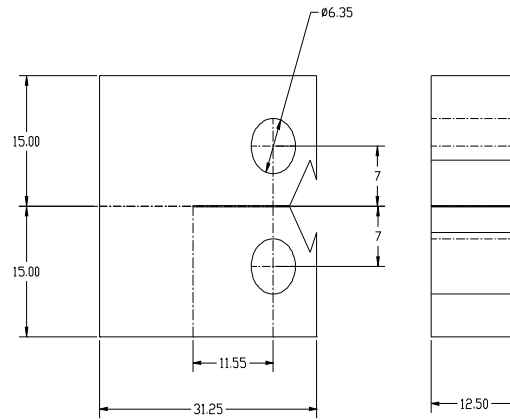


Fig. 3 Dimension of CT Specimen as per ASTM E399-90

4. Results and Discussion

4.1. Synthesis of NMMCs

Synthesis of copper-chilled Al-alloy based NMMCs were successfully accomplished by vortex method. The conditions prevail (as mentioned in sec 2) was instrumental in the prevention of microprosity segregation or agglomeration of reinforcement due to the effect of its gravity has indicated its suitability of stirring and realization of directional solidification conditions during pouring into the green sand molds containing copper chill to achieve sound castings. Thus indicate the feasibility of green sand casting process as a potential fabrication technique for nano-ZrO₂ components.

4.2. Microstructural Characterizations

Microstructural characteristics of copper-chilled NMMCs are discussed in terms of distribution of reinforcement and reinforcement matrix interfacial integrity. Microstructural studies conducted on the as cast copper-chilled NMMCs exhibits uniform distribution of reinforcement (Fig. 4 & 5), good reinforcement matrix interfacial integrity, significant grain refinement. This is due to the judicious selection of stirring parameters (vortex route), good wetting of preheated reinforcement by the matrix melt. During solidification a pasty mass of liquid and solid exists along with the fully solid and fully liquid zones containing nano-ZrO₂ particles. As further solidification progresses, the freezing shrinkage of the molten metal is compensated by flow of pasty mixture of solid and liquid. Hence freezing shrinkage of remaining molten metal and the volume shrinkage due to the solid contraction on the dendrites must be compensated by the flow of liquid metal on the interdendritic channels ensuring perfect bond between the reinforcement and the matrix. Further if the liquid metal gets entrapped between the dendrite arm solidifies without any compensation of liquid flowing, interdendritic shrinkage porosity results in poor bond between the reinforcement and the matrix. Thus for freedom from shrinkage porosity and to ensure good bonding , it is essential to maintain feeding of metal to the regions of last

solidifying liquid and to achieve this condition, it is necessary to set up steep temperature and solidification gradient throughout the solidification process by employing chills.

Metallography studies of the samples revealed that, the matrix is completely refined in case of NMMCs can primarily be attributed to capability of nano-ZrO₂ particulates to nucleate Al grains during solidification and restricted growth of recrystallized Al grains because of presence of finer reinforcement. The results of microstructural characterization of NMMCs however did not reveal presence of any micro pores or shrinkage cavity. Fig. 4 & 5 shows of microstructure of cast NMMC with 6 and 9 Wt.% of reinforcement and fig. 6 & 7 shows microstructure of un-chilled matrix alloy and copper-chilled matrix alloy (LM 13) respectively

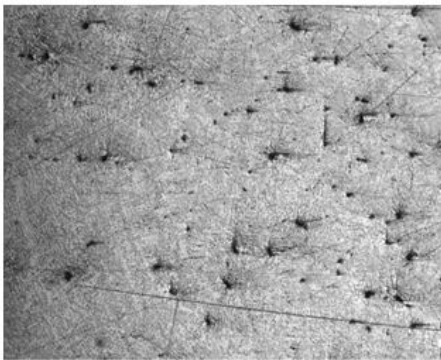


Fig.4. Optical microstructure of Cu chilled NMMCs containing 6 Wt. % of ZrO₂ (500 x magnification)

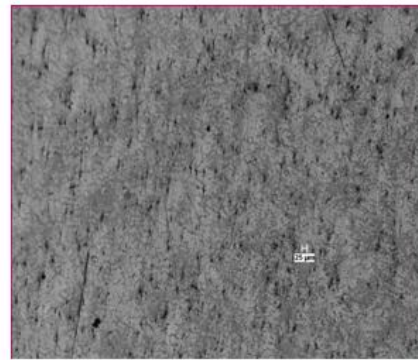


Fig.5. Optical microstructure of Cu-chilled NMMCs containing 9 Wt. % of ZrO₂(500 x magnification)

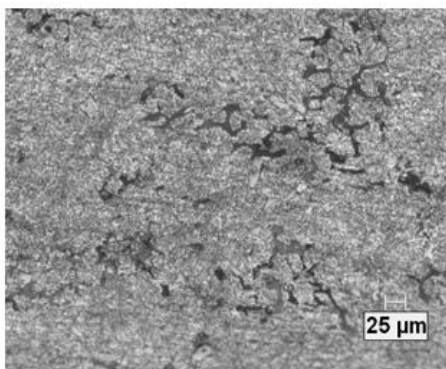


Fig. 6. Optical microstructure of as cast un-chilled matrix alloy (500 x magnification)

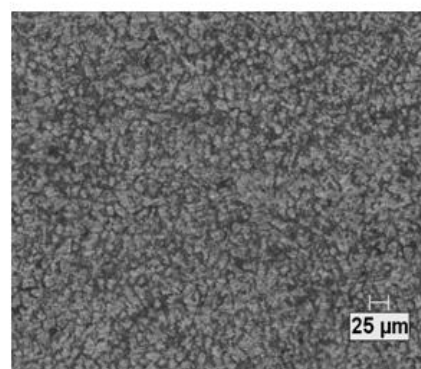


Fig. 7. Optical microstructure of Cu-chilled matrix alloy (500 x magnification)

4.3. Mechanical Properties (hardness and strength):

Results of developed NMMCs on the mechanical properties reveals significant enhancement in mechanical properties with reinforcement addition up to 9 Wt.% and addition above this limit decreases mechanical properties. The Brinell hardness test conducted on developed NMMCs exhibits an increasing trend in matrix hardness with increase in reinforcement content up to 12% (refer Table 3 & Fig. 8). This significant increase in hardness can be primarily attributed to the presence of harder ZrO_2 ceramic particulates which are uniformly distributed in the matrix and fine grain size in the matrix due to chilling. Ambient temperature tensile test results exhibits significant improvement in 0.2% yield strength and ultimate tensile strength (Refer Table 3 & Fig. 9) for developed NMMCs with slight adverse effect on ductility (refer Fig. 10). Increase in tensile strength with reinforcement addition up to 9% can be attributed to increase in grain boundary area due to grain refinement with capability of nano- ZrO_2 particulates to nucleate Al grains during solidification, built up of thermal stresses at the interface due to difference in coefficient of thermal expansion and effective transfer of tensile load on the well bonded uniformly distributed reinforcement in the matrix. Under the influence of applied stress, more number of grain boundaries acts as obstacle to the dislocation movement and end up with dislocation pileup at the grain boundary region [19]. Multi-directional thermal stresses induced during processing easily starts multi-gliding system under applied stress so that, dislocations were found developing and moving in several directions [20]. These multi-guide planes agglomerate under the applied stress forms grain boundary ledges. As the applied load increases these ledges acts as obstacles to dislocation movement resulting in pile-ups. The coupled effect of these two obstacles leads to increase in the strength of the composite [21].

Table 3 Mechanical properties of NMMCs and Matrix alloy (LM 13)

ZrO ₂ (wt.%)	BHN	UTS (MPa)	Ductility %	Fracture Toughness (MPa√m)
Matrix alloy (LM13)	90	130	9	9.00
Matrix alloy Cu-chilled	95	139	10	10.00
3	101	141	8.0	10.28
6	108	160	6.5	10.46
9	119	164	5.0	11.28
12	128	155	4.2	10.36

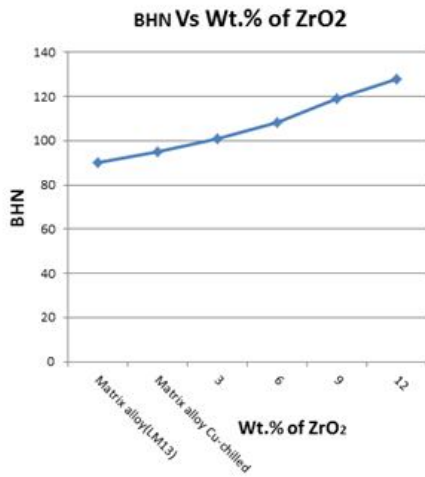


Fig. 8. Plot showing the variation in BHN Vs matrix alloy, chilled matrix alloy, and NMMCs containing various Wt.% of ZrO₂

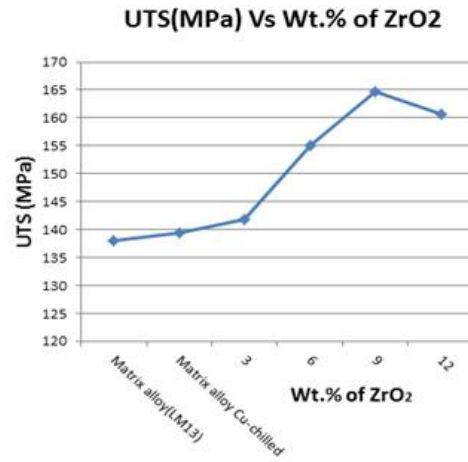


Fig. 9. Plot showing the variation in UTS Vs matrix alloy, chilled matrix alloy, and NMMCs containing various Wt.% of ZrO₂

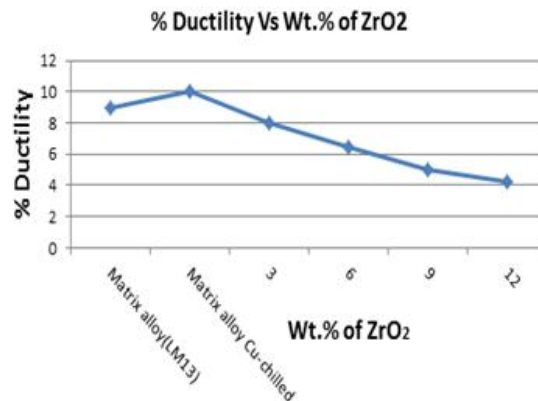


Fig. 10. Plot showing the variation in ductility Vs matrix alloy, chilled matrix alloy, and NMMCs containing various Wt.% of ZrO₂

4.4. Fracture toughness

The results of fracture toughness test done on developed NMMCs are shown in Table 3. From the results, it is observed that increase in the reinforcement content and the volumetric heat capacity of the chill used has a pronounced effect on the fracture toughness of the material. The NMMCs containing 9Wt.% of ZrO₂ has fracture toughness of about 12.8% higher than the unreinforced matrix alloy. Further it is observed that NMMCs having 12Wt.% of ZrO₂ has registered a decrease in fracture toughness. However it cannot be assumed that the materials are weaker than unreinforced materials since in most cases it is quite reverse. The plots of fracture toughness versus Wt.% of ZrO₂ is shown in the Fig. 11.

Fracture surfaces revealed different topographies for the composite containing different weight percentage of nano-ZrO₂ particulates. Close examination of the

fractured surface indicate that, most dimples were associated with the matrix material. Results of fractured surface analysis conducted on fracture toughness specimen of FCC structured Aluminum alloy samples revealed large dimples (ref Fig. 12) along with large amount of plastic deformation, indicating ductile fracture. The fractured surface exhibit predominantly that fractured particles are the matrix material with fine and shallow dimples, indicating ductile fracture. Fracture surface in the case of NMMCs containing 6& 9 Wt. % ZrO_2 (ref Fig. 13, 14) reveal mixed mode fracture and NMMC containing 12wt.% of ZrO_2 revealed cleavage type of fracture (ref Fig. 15) due to the presence of excessive nano- ZrO_2 particulates. It is also noted that clustered particles are sensitive to premature damage in the composite and large particles seems to be prone to fracture and hence registered a reduction in the fracture toughness value.

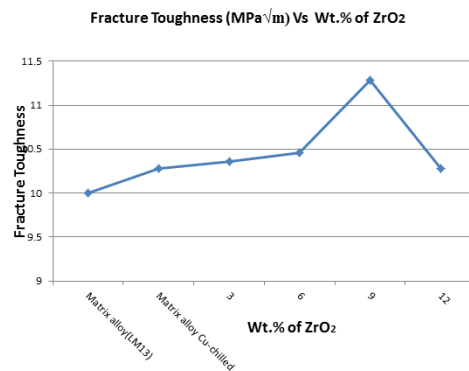


Fig. 11. Plot showing the variation in Fracture toughness Vs matrix alloy, chilled matrix alloy, and NMMCs containing various Wt. % of ZrO_2

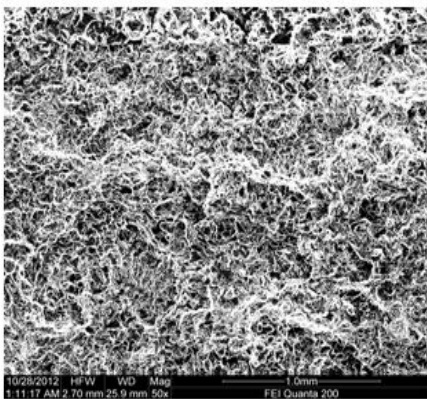


Fig.12. SEM photograph of fractured specimen in fracture toughness test (copper chilled matrix alloy)

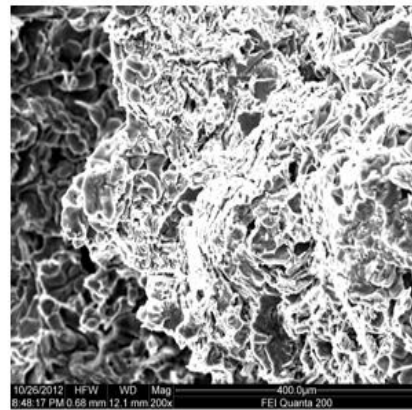


Fig.13. SEM photograph of fractured specimen in fracture toughness test (chilled NMMC containing 6Wt. % of ZrO_2)

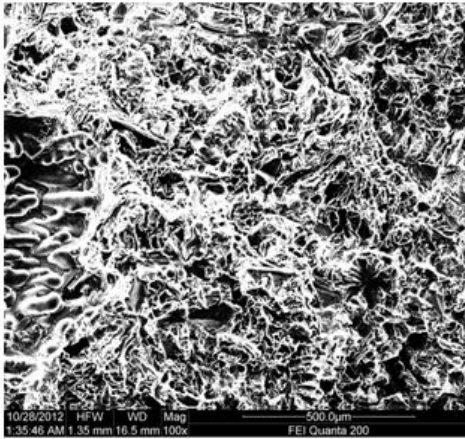


Fig.14. SEM photograph of fractured specimen in fracture toughness test (chilled NMMC containing 9Wt.% ZrO₂)

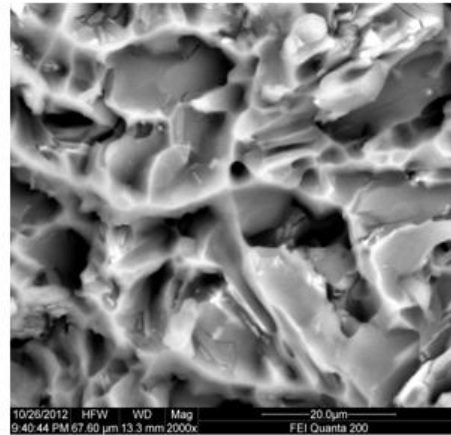


Fig.15. SEM photograph of fractured specimen in fracture toughness test (chilled NMMC containing 9Wt.% ZrO₂)

4. Conclusions

Significant improved in hardness, Strength, fracture toughness with slight reduction in ductility of the aluminum alloy reinforced with nano-ZrO₂ is achieved through VHC of the chill used and also with the addition of nano reinforcement particulates. Microstructural analysis showed fine grain refinement, fairly uniform distribution of reinforcement. Fractography analysis revealed that fracture behavior of FFC structured Al matrix alloy was changed from ductile fracture to cleavage mode because of presence of ZrO₂ particles.

Reference

1. G.P. Reddy, P.K.Br. Foundaryman 69 (1976) 265.
2. X. Cambell, Castings, Butterworth-Heinemann, 1991.
3. W.D Walther, C.M. Adams, H.F. Taylor, Trans. AFS 62 (1954) 219.
4. W.H. Johnson, J.G. Kura, Trans. AFS 67 (1959) 235.
5. R.W. Ruddle, J. Inst. Mater. 77 (1950) 37.
6. R. A. Flinn, H. Kunsman, Trans. AFS 67 (1959) 385.
7. A. Couture, J. W. Meier, Trans. AFS 74 (1966) 164.
8. M.V. Chamberlin, J.G. Mezoff, Trans. AFS 54 (1946) 648.
9. S. Seshan, M.R. Sheshadri, A. Ramachandran, Br. Foundaryman 61 (1968) 339.
10. J.T. Berry, Trans. AFS 78 (1970) 421.
11. Z.Y.Ma, Y. L. Li, Y. Liang, F. Zheng, J. Bi, S. C. Tjong, Mater. Sci. Eng. A 1996, 219, 229.
12. D.C. Jia, Mater Sci, Eng. A 2000, 289, 83.
13. F. Ferkel, B. L. Mordike, Mater Sci, Eng. A 2001, 298, 193
14. Y.C. Kang, S.L. Chan, Mater. Chem. Phys. 2004, 85, 43.
15. L.Lu, M. O. Lai, W. Liang, Compos. Sci Technol. 2004, 64, 2009.

16. S. F. Hassan, M.Gupta, Mater Sci, Eng. A 2005, 392, 163.
17. S.C.Tjong, Z.Y.Ma, Mater Sci, Eng. R 2000, 29, 49.
18. Joel Hemanth, journals of Alloys and Compounds 296(2000) 193-200.
19. R.E. Reed Hill, Physical Metallurgy Principles, 2nd ed. D. Van Nostrand Company, UK, 1964, 56-69.
20. F. Wua. Y.Chen, J.Zhua, Mater. Sci. Eng. A 277 (2000) 143-154.
21. Joel Hemanth, Mater Sci, Eng. A 2009, 110-113

