

Full Length Research Paper

Fracture behavior of cryogenically solidified aluminum-alloy reinforced with Nano-ZrO₂ metal matrix composites (CNMMCs)

Joel Hemanth

Rajiv Gandhi Institute of Technology, R. T. Nagar Post, Hebbal, Bangalore-560032, Karnataka, India.
E-mail: joelhemanth@hotmail.com.

Accepted 30 May, 2011

Fracture behavior of LM-13 aluminum alloy reinforced with different wt.% of Nano-ZrO₂ particles, solidified under various shallow cryogenically cooled chills (at -80°C), cast using DMD (disintegrated melt deposition) technique followed by hot extrusion were investigated. The size of the particles dispersed varies from 50 to 80 nm and amount of addition varies from 3 to 15 wt.% in steps of 3 wt.%. Microstructural studies of the Nano-composite developed indicate that there is uniform distribution of the reinforcement in the matrix and mechanical properties reveal that presence of Nano-ZrO₂ particles as dispersoid (up to 12 wt.%) and chill thickness (25 mm) has improved significantly the strength, hardness and fracture toughness with slight reduction in ductility. Fractography of the specimens showed that the fracture behavior of matrix alloy has changed from ductile intergranular mode to cleavage mode of fracture. Finally heat capacity of the shallow cryo-chill is identified as an important parameter which affects mechanical properties.

Key words: Composites, mechanical, fracture, Nano, chill, shallow cryogenic, microstructure.

INTRODUCTION

It is indeed to measure the strength of material to estimate the toughness property, because strength is usually measured by the deformation area which is influential on the property of the material (Bowden and Tabor, 1964). Reports of measurement of mechanical properties at cryogenic temperature are few and the properties at these conditions are unknown fully (Petty, 1960; Oku and Usui, 1971). But recently several researches have been carried out on ferrous and non ferrous materials to fill the gap (Bensely et al., 2006; Huang and Liao, 2003). Hence it is important that the mechanical properties of the composite developed are evaluated at cryogenic temperatures. In particular, the fracture properties of this composite need to be investigated in detail (Ogata et al., 2002; Ono et al., 2002; Yuri et al., 2002), since fatigue fracture has been found in automobiles and it is considered to cause the failure of many automobile and structural components. And it has been also reported that sub-surface crack initiation is associated with the microstructure rather than specific defects such as inclusions (Umezawa et al., 1990). Since sub-surface crack initiation tends to occur at low stress amplitudes and high numbers of cycles,

whereas fatigue crack initiation at the specimen surface occurs at high stress amplitudes to reduce the fatigue properties in the longer life region (Murakami, 2002; Shiozawa et al., 2002; Nishijima and Kanazawa, 1999). A high level combination of strength and toughness is required for, components for space application components which need to sustain large fatigue forces (Suemune et al., 1998). On the other hand Nano-structured materials inherit relatively large interfacial energy (Gleiter, 2000). Recently mechano-synthesis of Cu-AL₂O₃ Nano-composite has been reported by Seung and Wang (2005).

As aerospace technology continues to advance, there is a rapidly increasing demand for advanced materials with high mechanical and thermal capabilities for such ultra high applications (Opeka et al., 2004). Its application also stretched to automobile, electronic and computer industries to replace the existing materials including plastics (Luo, 1995). The early 1990s are considered to be the renaissance for Al as structural material due to environmental concerns, increasing safety and comfort levels. A significant improvement in the properties of Al alloys, reduced fuel consumption because of light weight

Table 1. Chemical composition of matrix material (Al-alloy LM 13).

Elements	Zn	Mg	Si	Ni	Fe	Mn	Al
% by wt	0.5	1.4	12	1.5	1.0	0.5	Bal

and hence it has made huge demand from automobile industry (Saravanan and Surappa, 2000; Hassan and Gupta, 2002). This growing requirements of materials with high specific mechanical properties with weight savings has fueled significant research activities in recent times targeted primarily for further development of Al based composites (Singh and Tsai, 2003; Guy, 1967; Gupta et al., 2000). A recent industrial review revealed that there are hundreds of components from structural to engine in which aluminum alloy is being developed for variety of applications (Yamamoto and Sasamoto, 2000). It is also predicted that for Al alloys the demand increased globally at an average rate of 20% every year (Awasthi and Wood, 1988). It is noticed that the limited mechanical properties (strength and hardness) of Al and its alloys adversely affect its applications in automobile and aerospace industries (Lai, 2000; Wang and Yang, 2002). This remains one of the major concerns in its fabrication to suit its application in recent days.

Search of open literature indicates that, so far number of Al based MMCs including chilled MMCs (Hemanth, 2003, 2005, 2007, 2008) are being developed but no work has been done in this field. Hence the present research is undertaken to fill the void and to investigate the integrated properties of Al-alloy/ ZrO₂ CNMMCs. Among all the reinforcements used in Al based composites only Nano size particulates has shown their potential superiority in improving mechanical properties, microstructure with noticeable weight savings (Lloyd, 1994).

Disintegrated melt deposition (DMD) technique (Tham and Cheng, 1999; Zhang and Guo, 2002) has been chosen in this research which remains an attractive choice among all the solidification processing routes due to its capability of bringing together the advantages of both spray processing and conventional casting. Accordingly, the primary aim of the present investigation is to synthesize Al alloy based composite reinforced with Nano-ZrO₂ particulates using DMD technique. The composites thus obtained were hot extruded and characterized for their micro structural, mechanical, thermal and electrical properties. Particular emphasis is placed to study the effect of presence of Nano particulates and its increasing amount on the microstructure and mechanical response of standard LM 13 Al alloy.

EXPERIMENTAL PROCEDURE

In this research Nano-ZrO₂ particles dispersed in Al alloy (chemical composition of LM 13 the matrix material are shown in Table 1), fabricated by DMD technique, solidified under cryogenically cooled chills followed by hot extrusion were investigated. Copper end chills

of thickness 5, 10, 15, 20 and 25 mm in which arrangements were made to circulate liquid nitrogen (at -80°C) to study the effect of heat capacity on mechanical and microstructural behavior (arrangement shown in Figure 1). The size of the Nano-ZrO₂ particles dispersed varies from 50 to 80 nm and the amount addition varies from 3 to 15 wt.% in steps of 3 wt.%. The primary processing consists of synthesis of monolithic and Nano-sized ZrO₂ reinforced Al composites (Al/Nano-ZrO₂) containing five different weight % of ZrO₂ was carried out using DMD technique. Synthesis of the composite involved heating of Al alloy in a graphite crucible up to 720°C using resistance furnace to which the preheated reinforcement (to 250°C) was added and stirred well by an impeller which rotates at 450 rpm to create vortex to get uniform distribution of the reinforcement. This treated Al alloy containing Nano-ZrO₂ particles were made to solidify under the influence of cryo-cooled chills of different thickness set in AFS standard dry sand mold. In secondary processing, the deposited monolithic and Nano sized ZrO₂ in Al matrix are extruded in a hydraulic press at 260°C.

Properties of reinforcement material

Properties of the reinforcement (Nano-ZrO₂) are as follows: Density: 2.6 gm/cc, melting point: 1860°C, UTS: 425 MPa, VHN: 150, Young's Modulus: 98 GPa., Size: 50 to 80 nm, Density: 2.6 gm/cc (Nano-ZrO₂ was supplied by Nano structured and Amorphous Materials, Inc, USA)

Testing procedure

Microstructural characterization were conducted on polished CNMMC specimens (etched with Keller's reagent) using OLYMPUS metallographic microscope to investigate morphological characteristics of grains, reinforcement distribution and interfacial integrity between matrix and the reinforcement. Hitachi S4100 field emission scanning electron microscope was used to analyze the fractured surfaces. Vickers microhardness measurements were made on polished specimens using Matsuzawa MXT50 digital hardness tester using 25 gf indentation load in accordance with ASTM E18-94 standard. Tension tests were performed on American Foundry men Society (AFS) standard tensometer specimens in accordance with ASTM E8M-01 standard using Instron 8516 machine. Fracture toughness tests were performed using a closed loop INSTRON servo-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. All the above tests were conducted at an ambient temperature of 28°C.

RESULTS AND DISCUSSION

Results of the investigation on mechanical properties reveal that adding reinforcement content up to 12 wt.% enhances mechanical properties and addition above this limit deteriorates mechanical properties. Also, 25 mm thick chill has pronounced effective on mechanical properties

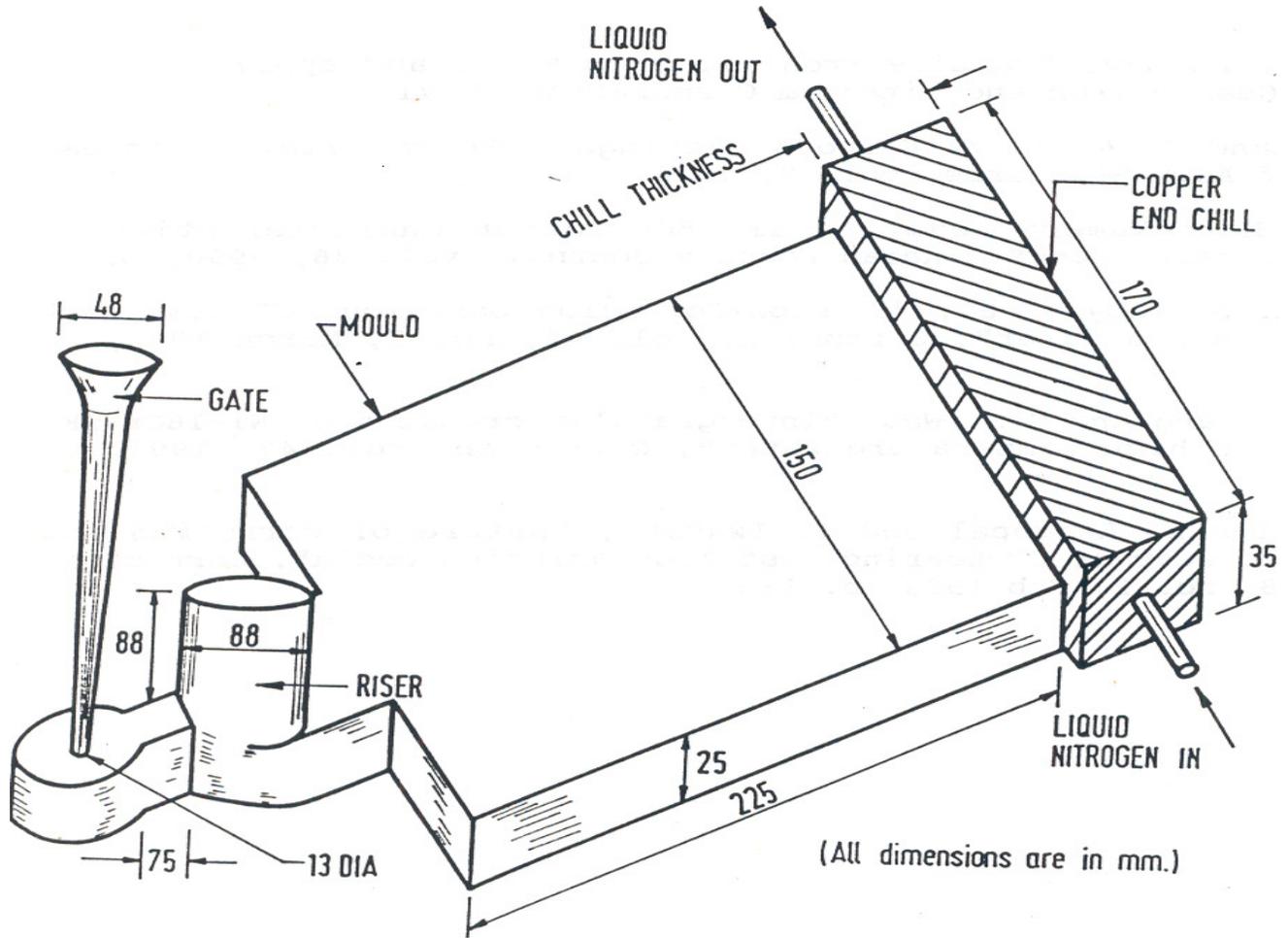


Figure 1. Experimental set up (AFS standard mold containing cryogenic end chill block).

because of its high volumetric heat capacity. Hence present discussion is focused mainly on CNMMCs containing 12 wt. % dispersoid cast using 25 mm thick cryo-chill block.

For all the tests specimens were taken from the chill end, because it is observed that, farther away from the chill the specimen is taken, the lower are the mechanical properties. This could be because the rate of chilling is lower, when the specimen is farther away from the chill.

Synthesis of CNMMCs

Synthesis of Al –alloy based CNMMCs was successfully accomplished by DMD process followed by cryo-chilling and hot extrusion. The condition prevailed during melting, processing, dispersion, deposition and solidification under cryo conditions (as mentioned in experimental procedure) was instrumental in the prevention of micro porosity, segregation or agglomeration of reinforcement due to effect of its gravity has indicated its suitability of stirring and the realization of good solidification

conditions during deposition to produce sound chilled castings. The results, in essence, indicate the feasibility of the DMD process along with cryo chilling followed by hot extrusion act as a potential fabrication technique for Nano-ZrO₂ composites.

Cryogenic effect on solidification

The optical photomicrographs in Figures 2, 3 and 4 show that the matrix microstructure of cryogenically chilled Nano composites is finer than that of the unchilled matrix alloy. Figures 2 and 3 show microstructure of un chilled as cast composite and extruded composite containing 12 wt.% reinforcement and Figure 4 shows microstructure of CNMMC containing 12 wt.% dispersoid cast using 25 mm thick chill. When the melt of the CNMMC solidifies under cryogenic conditions, the sub-zero temperature of the chill and the hot melt come in contact experiences severe super-cooling. This results from a high rate of heat transfer and rapid cooling of the melt in chilled CNMMC samples. Hence, the critical nucleus size of the

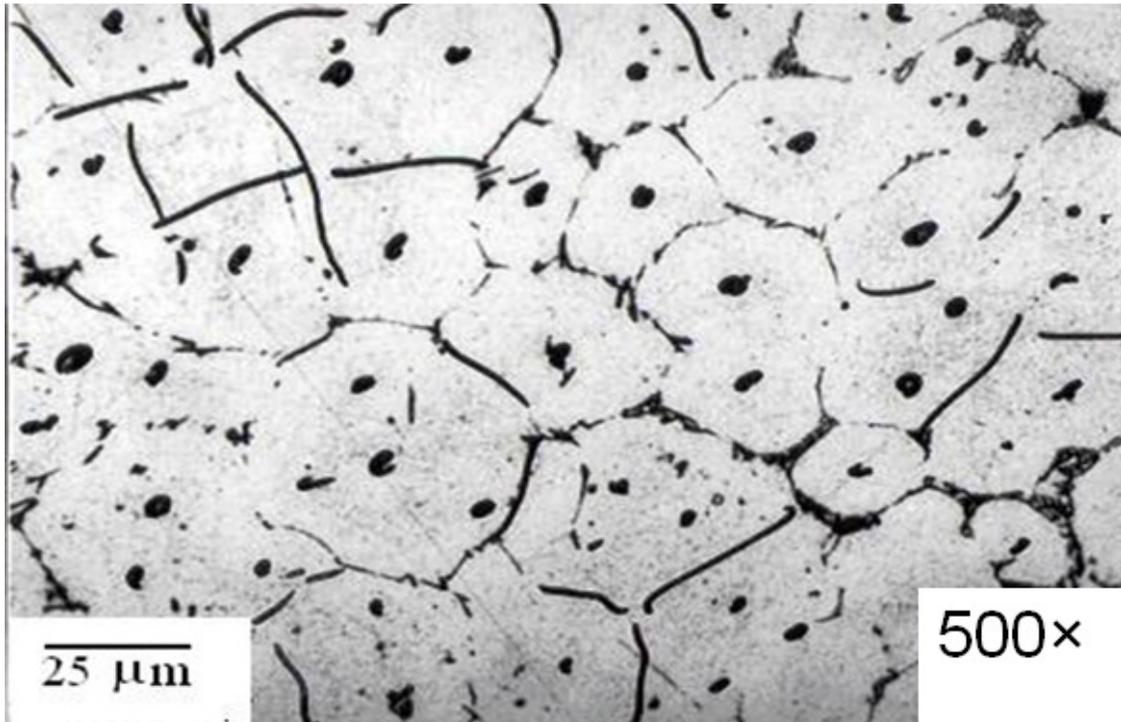


Figure 2. Optical microstructure of as cast un chilled Nano-composite containing 12 wt.% reinforcement (500 × magnification).

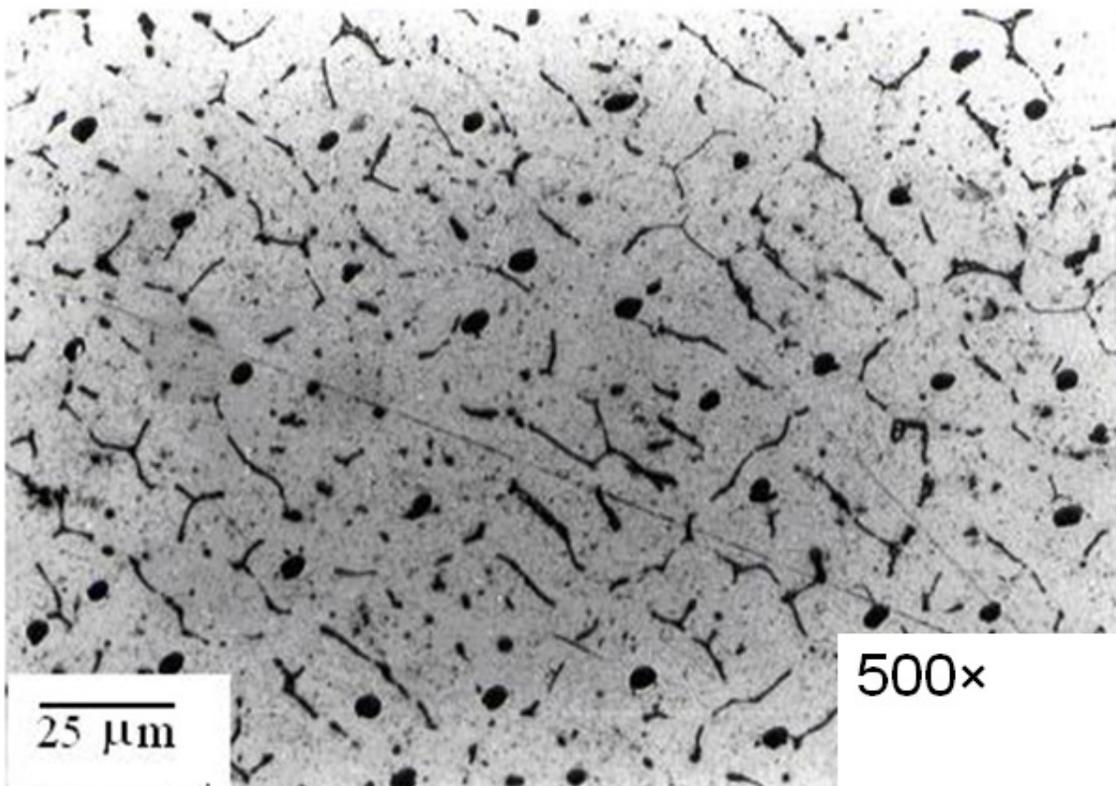


Figure 3. Optical microstructure of extruded un chilled Nano-composite containing 12 wt.% reinforcement (500 × magnification).

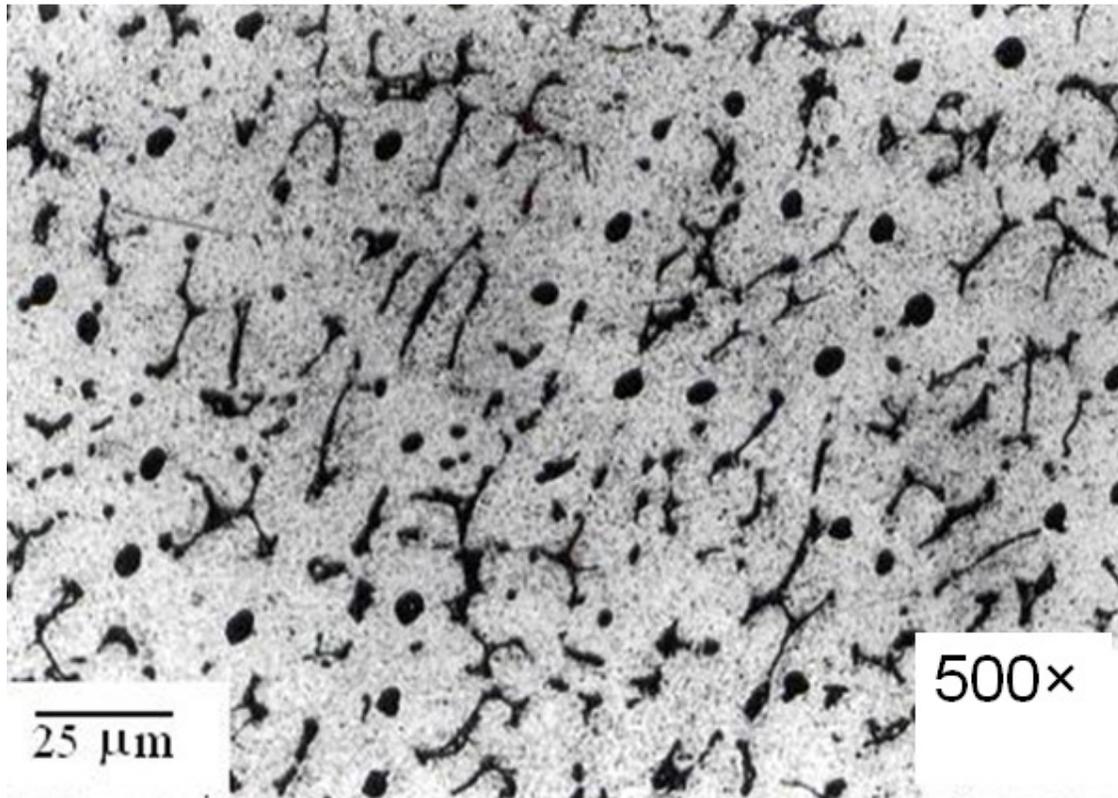


Figure 4. Optical microstructure of extruded CNMMC containing 12 wt.% dispersoid cast using 25 mm thick cryo-chill (500 × magnification).

solidified melt is reduced and a greater number of nuclei are generated, causing a finer microstructure. Additionally, because of the rapid cooling of the melt and stirring the dispersoid particles do not have time to settle down due to density differences between the matrix melt and the dispersoid, and these results in a more uniform distribution of Nano-ZrO₂ particles in the matrix. The cryogenic effect during solidification causes stronger bonding between the matrix and the dispersoid. This may be attributed to the fact that the wettability between the particles and the matrix was improved with cryogenic effects. This uniform distribution of particles and the finer matrix structure and chilling (Figure 4) lead to improved mechanical properties of the CNMMCs as compared with the un-chilled composite (Figures 2 and 3). Thus the strong bonding between the dispersoid and the matrix causes more effective load transfer. This in turn reduces the possibility of pullout of particulates from the matrix. Hence, any failure of the CNMMC would more likely be due to particle failure rather than particle pullout from the matrix.

Micro structural characterization

Micro structural characteristics of extruded cryo-chilled

Nano-composites are discussed in terms of distribution of reinforcement and reinforcement matrix interfacial bonding. Microstructural studies conducted on the extruded Nano-composites containing 12 wt.% dispersoid cast using 25 mm thick chill revealed uniform distribution of the reinforcement with limited extent of clusters, good reinforcement-matrix interfacial integrity, significant grain refinement with minimal porosity (Figure 4). This is due to the gravity of ZrO₂ associated with judicious selection of stirring parameters (vortex route), good wetting of pre heated reinforcement by the matrix melt. Metallography studies of the extruded samples revealed that the matrix is recrystallized completely. Grain reinforcement in case of Nano-composites can primarily be attributed to capability of Nano-ZrO₂ particulates to nucleate Al grains during directional solidification and restricted growth of recrystallized Al grains because of presence of finer reinforcement and chilling. Interfacial integrity between matrix and the reinforcement was assessed using scanning electron microscope of the fractured surfaces to analyze the interfacial de-bonding at the particulate-matrix interface. Here also the result revealed that a strong bond exists between the interfaces as expected from metal/oxide systems (Gilchrist, 1989).

Micro and macro tests reveal that, high rate heat transfer During solidification (cryo-chilling) of the composite in this

Table 2. Mechanical properties (strength and hardness) of CNMMCs and matrix alloy cast using cryo chill of thickness 25 mm.

Property (wt% ZrO ₂)	Hardness (HV)	0.2% Yield strength (MPa)	UTS, (MPa)	Ductility (%)
3	111	127	245	17.7
6	118	135	253	16.4
9	129	161	262	15.9
12	138	186	277	14.1
15	131	171	268	13.2
Matrix alloy (LM 13)	58	120	130	18

investigation leads to strong bonding of the dispersoid and the matrix. This may be one of the main reasons for increase of strength, hardness and fracture toughness of the composite developed. The result of micro structural studies of CNMMCs however did not reveal presence of any micro-pores or shrinkage cavity. After the secondary process (extrusion) also, there was no evidence of any microstructural defects

Mechanical properties (hardness and strength)

The results of micro hardness test conducted on extruded CNMMCs samples revealed an increasing trend in matrix hardness with increase reinforcement content up to 12% and chill thickness up to 25 mm (Table 2 for mechanical properties). Results of hardness measurements revealed that an increase in the reinforcement content and chill thickness leads to a significant increase in the hardness and can be attributed primarily to: presence of harder ZrO₂ ceramic particulates in the matrix, a higher constraint to the localized deformation during indentation due to their presence and reduced grain size due to chilling. In ceramic-reinforced composite, there is generally a big difference between the mechanical properties of the dispersoid and those of the matrix. This result in incoherence and a high density of dislocations near the interface between the dispersoid and the matrix. Precipitation reactions are accelerated because incoherence and the high density of dislocations act as heterogeneous nucleation sites for precipitation.

UTS is higher for all the CNMMCs as compared against the matrix alloy. It is further seen that for CNMMCs the values of these parameters increase as the dispersoid content increases up to 12 wt. %, beyond which the trend reverses. Conversely, percentage elongation (measure of ductility) decreases monotonically as dispersoid content increases. Table 2 shows the UTS near the chill end for different composites cast using cryogenic chills of different thicknesses. As the chill thickness increases, UTS also increases, confirming that the heat capacity of the chill significantly enhances the UTS. Results of the ambient temperature tensile tests also revealed significant improvement in 0.2% yield strength (YS) (Table 2) for CNMMCs developed with little adverse affect on ductility.

Increase in tensile strength (reinforcement up to 12%)

is also attributed to increase in grain boundary area due to grain refinement, built of thermal stresses (due to cryo-chilling) at the interface due to different coefficient of thermal expansion (CTE) and effective transfer of applied tensile load to the uniformly distributed well bonded reinforcement (strength of ceramic materials lies much higher than metallic materials (Srinivasan and Gupta, 1999) as the UTS of the composite developed is 46% higher than that of the matrix alloy. Under the applied stress, increasing the amount of grain boundaries acts as obstacle to the dislocation movement and end up with dislocation pile up at the grain boundary region (Reed, 1964). Again, multi-directional thermal stress induced during processing easily starts multi-gliding system under applied stress so that dislocations were found developing and moving in several directions. These multi-glide planes agglomerate under the applied stress forms grain boundary ledges. As applied load increases, these ledges act as obstacle to dislocation movement resulting in pile-ups. The coupled effect of these two obstacles lead to increase in the strength of the composite (Hemanth, 2002).

Fracture studies conducted on fractured tensile specimens indicate that the change in fracture mode from ductile to a typical cleavage mode could not improve ductility of the Nano-composite due to presence of ceramic reinforcement. As compared with the matrix (Al alloy) ductility, the ductility of the CNMMCs are reduced (by 78.33% for 12 wt.% addition of reinforcement) due to presence of Nano-ZrO₂ particles, which is a contrary effect.

Fracture toughness

Fracture toughness tests performed on 3 point bend (containing chevron notch) specimen which was pre cracked by fatigue loading. The manner in which stress response varies with cycles and plastic-strain amplitude is an important feature of the low-cycle fatigue process. The cyclic stress required for pre-cracking the specimen provide useful information pertaining to the mechanical stability of the intrinsic micro structural features during reverse plastic-straining, coupled with an ability of the material to distribute the plastic strain over the entire volume, are the two key factors governing the cyclic response of a material.

Table 3. Fracture toughness of CNMMCs and matrix alloy.

Copper chill block thickness (mm)	Fracture toughness (K_{IC}) (MPa) \sqrt{m} (near the chill block end)				
	3 wt% dispersoid	6 wt% dispersoid	9 wt% dispersoid	12 wt% dispersoid	15 wt% dispersoid
25	11	13	14	16	12
20	9	11	12	13	10
15	9	10	11	11	08
10	6	7	7	08	06
05	04	05	05	06	06

Fracture toughness of the matrix alloy LM 13 is 3.8 MPa \sqrt{m} .

Effect of dispersoid content on fracture toughness

The fracture toughness of the composite chilled using different type of chills and dispersoid content are shown in Table 3. Comparing these results, it can be seen that increasing the reinforcement content and chilling seems to have an effect on the fracture toughness of the material. It is also observed that CNMMC containing 12 wt. %, cast using 25 mm thick chill block invariably has the highest fracture toughness. Further it is observed that, at reinforcement content beyond 12 wt. %, the values register a decrease in fracture toughness

Fracture toughness results of the cryo-chilled Nano-composite indicate that presence of hard ZrO_2 has a pronounced effect and it increases as dispersoid content increases up to 12 wt.%. However, it is not customary to assume that reinforced materials are weaker than unreinforced materials since in most cases it is quite reverse. The results of this study have also shown that, as reinforcement content increases along with the chilling and extrusion process, the matrix becomes dense, stronger and accommodate the reinforcement rigidly and this could lead to a greater strength and fracture toughness of the composite compared with the monolithic alloys. The mechanisms which control the variation of fracture toughness of chilled Al-Nano- ZrO_2 composites are dependent upon both microstructure and strain range. The possible micro mechanisms controlling the fracture behavior during cyclic loading are ascribed to the following synergistic influences (Hemanth, 2002, 2009):

- Load transfer between the soft aluminum matrix and the hard Nano- ZrO_2 particle reinforcement.
- A pre-existing high dislocation density in the aluminum matrix caused by the presence of the hard, brittle ZrO_2 particles.
- Hardening effect arising from constrained plastic flow and tri-axiality in the aluminum matrix is due to the presence of Nano- ZrO_2 reinforcement and cryo-chilling. As a result, the particles resisting the plastic flow of the matrix develops an average internal stress or back stress
- Dislocations arising from competing influences of back stresses in the plastically deforming composite matrix is due to plastic relaxation by the formation of dislocation loops around ZrO_2 particles.

(e) Residual stresses generated and dislocations arising are due to mismatch in thermal expansion coefficients (due to chilling) between the components of the composite, that is, the soft aluminum matrix and the hard reinforcing ZrO_2 .

Fracture surface analysis

Fracture surface analysis revealed different topographies for the composites containing different weight percentage of ZrO_2 particles. Results of the fracture surface analysis conducted on fracture toughness specimens of FCC structured LM 13 Al alloy samples revealed large dimples along with large amount of plastic deformation indicating ductile fracture. The fracture surfaces also exhibit fine and shallow dimples, indicating that the fracture is ductile.

Scanning electron micrographs of the fracture surface of the CNMMCs tested are shown in Figures 5 and 6. Examination of these fracture surface features in SEM at low magnification is to identify the fatigue and final fracture regions, and at higher magnification in the fatigue is region to identify areas of micro-crack initiation and early crack growth, and the over-loaded region to identify the fine scale fracture features. Fracture surfaces revealed different topographies for the composites containing different weight percentage of ZrO_2 particles cast using different type of cryo-chill blocks.

Fracture surface in the case of CNMMCs containing 6 wt.% (Figure 5) reinforcement, cast using 25 mm thick cryo chill block reveal mixed mode fracture and CNMMCs containing 12 wt.% reinforcement revealed cleavage type of fracture (Figure 6) due to the presence of excessive Nano- ZrO_2 particulates. It can also be seen that regions of clustered particles for reinforcement above 12 wt.%, (Figure 7) are sensitive to premature damage in the composite and the large particles seems to be prone to fracture and hence registered a reduction in the fracture toughness value (Table 3). Fracture mode of the matrix alloy which changed from ductile to cleavage type (in case of CNMMC) was dominated with microcrack nucleation and propagation as shown in Figure 6. Scanning electron microscope observations of CNMMCs suggest that, at higher reinforcement content (15 wt.%, Figure 7), void nucleation may also takes place at large precipitates

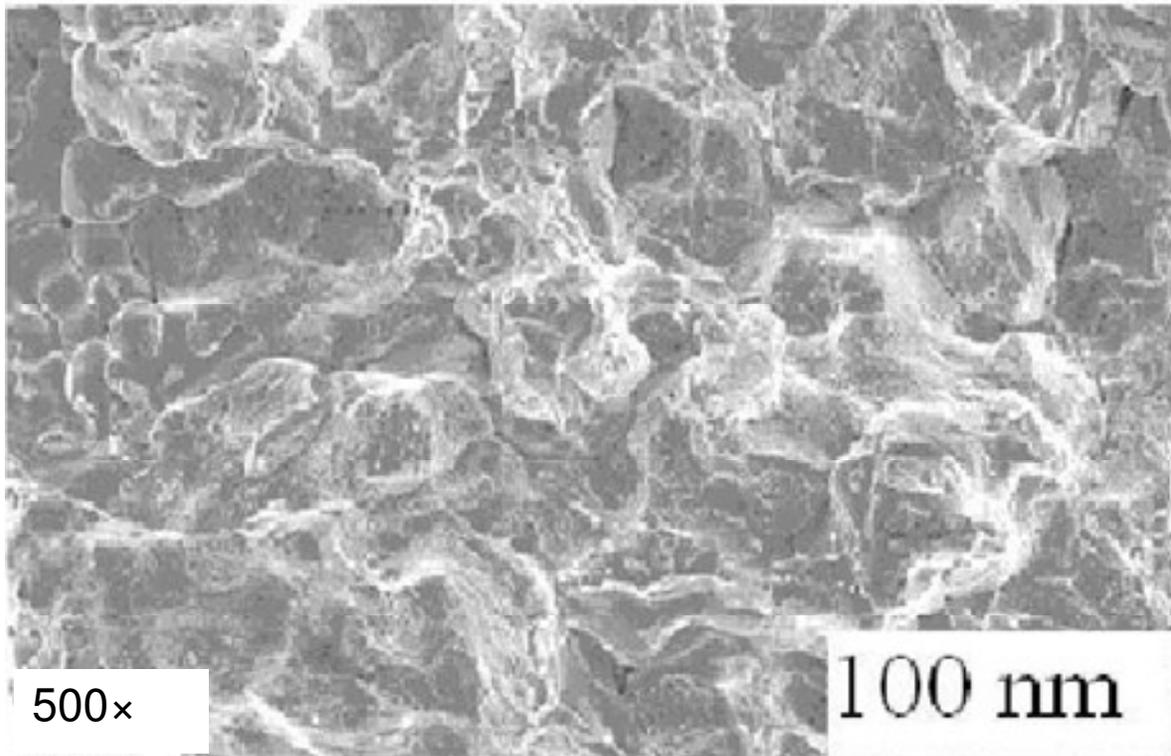


Figure 5. SEM fractograph of CNMMC (6 wt.% ZrO₂) cast using 25 mm thick chill block.

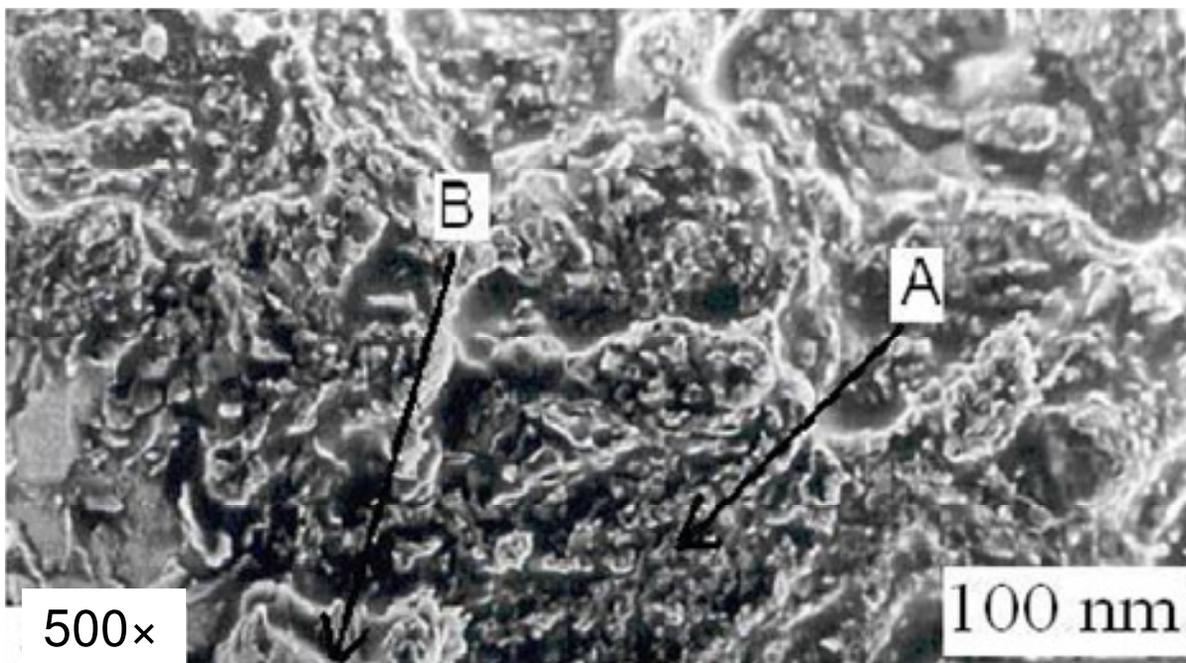


Figure 6. SEM fractograph of CNMMC (12 wt.% ZrO₂) cast using 25 mm chill block.

in addition to the matrix/particle interface, while at lower reinforcement (3 wt.%, Figure 8) rates the fracture seems to occur by breakage of the particles.

Fracture surface analysis of CNMMC containing 9 wt.% dispersoid (Figure 9) exhibit predominantly that fractured particles (dispersoid) and the matrix material with coarse

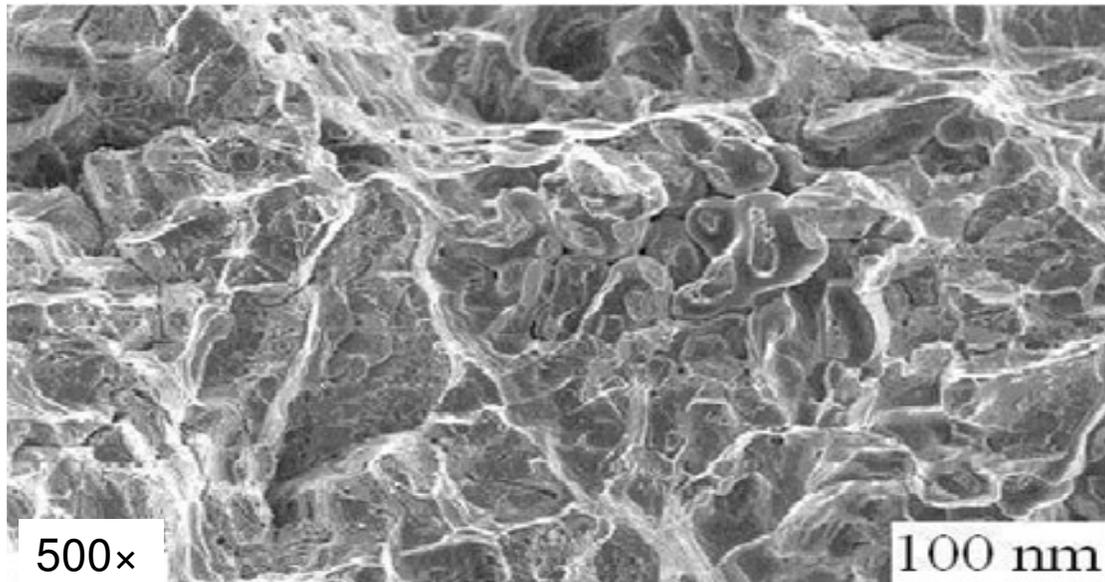


Figure 7. SEM fractograph of CNMMC (15 wt.% ZrO₂) cast using 25 mm chill block.

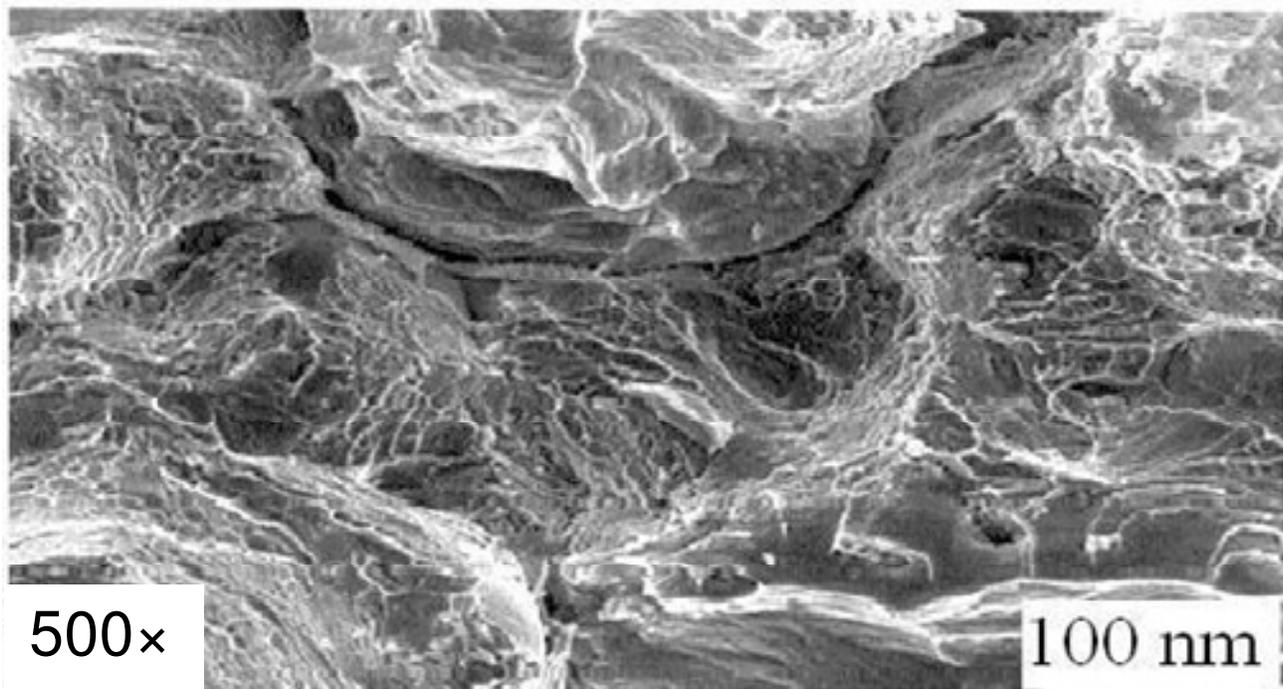


Figure 8. SEM fractograph of CNMMC (3 wt.% ZrO₂) cast using 25 mm chill block.

dimples, suggests that fracture is of mixed mode type. Close examination of the fractured surface indicate that, most dimples were associated with the matrix material (Figure 10). Massive separations of the particle indicate that failure of the material is initiated by fracture of the particles rather debonding between the matrix and reinforcements. SEM fractographs of the cryo-chilled

Nano-composite tested for fracture toughness show a dimpled appearance with a large amount of plastic deformation of the matrix. It was found that the extent of fracture toughness of the composite with increasing the chilling rate as structure becomes fine. Particle fracture was found in some locations (Arrow A in Figure 6) in the particles clusters as well in large particles (Arrow B in

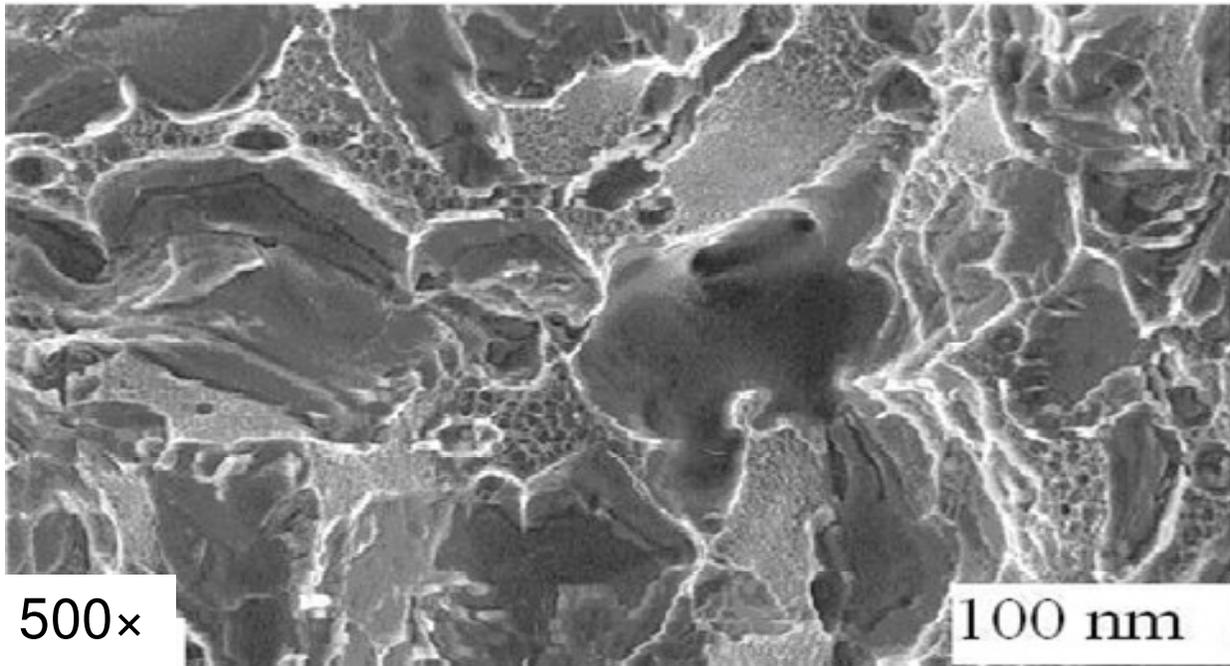


Figure 9. SEM Fractograph of CNMMC (9 wt.% ZrO₂) cast using 25 mm chill block.

Figure 6) present in the matrix somewhat away from the fracture surface, at higher chilling rates. The microstructural features shown in Figure 6 which is a sample containing 12 wt.% dispersoid cast using cryo-chill of 25 mm thick indicates that ZrO₂ particles have fractured during straining and fracture could have occurred in a brittle manner as a result of stress concentrations and propagation of the cracks.

Therefore, cyclic fracture of the un-chilled as cast matrix alloy on a macroscopic and microscopic scale, exhibited ductile fracture with isolated micro-cracks (Figure 5, optical microstructure of Matrix alloy). Observations of CNMMC containing 6 wt.% dispersoid revealed large areas of the fracture surface to be covered with a bimodal distribution of dimples, indicative of mixed mode ductile rupture (Figure 5). A dimple is a half void which form at the coarse second-phase particles, namely the iron and silicon inter-metallic contained in the aluminum matrix. However, growth of the void is limited by competing and synergistic influence of reinforcing particles, chilling effect and the cyclic ductility of the composite microstructure. Finally composite containing 12 wt.% dispersoid showed cleavage indicating that the fracture is towards brittle.

Effect of chilling rate on fracture toughness

The experimental results of the fracture toughness tests done on castings cast using different cryo chill blocks are shown in Table 3. It can be seen that changing the chill thickness seems to have an effect on the fracture

toughness of the material. This implies that increasing the rate of chilling by increasing the heat capacity of the chill block tends to result in an increase in the fracture toughness of the material. Of more significant effect is the temperature of liquid nitrogen (-80°C) and the chill thickness. It can be seen that if all other factors are kept constant, castings cryo-chilled using 25 mm thick copper block invariably has the highest fracture toughness followed by 20, 15, 10 and 5 mm thick cryo-chilled composites in that order. It is also observed that, farther away from the chill the specimen is taken, the lower is the fracture toughness. This could be because, the farther away from the chill the specimen is, the lower is the rate of chilling. This agrees with the deductions made earlier that increasing the rate of chilling tends to result in an increase in the fracture toughness of the material (Hemanth, 2002).

Conclusions

DMD technique coupled with cryo-chilling followed by hot extrusion used for the synthesis Nano-ZrO₂ composites reveal the following: Microstructural analysis showed fine grain refinement, fairly uniform distribution of reinforcement (stirring with vortex route at high speed) with minimal porosity (cryo-chilling effect). Mechanical characterization of composite cast using 25 mm thick cryo chill block containing 12 wt.% dispersoid revealed that the presence of Nano-ZrO₂ particulates in cryo-chilled Al matrix significantly improved hardness (by 42%), strength (by 46%) and fracture toughness (by

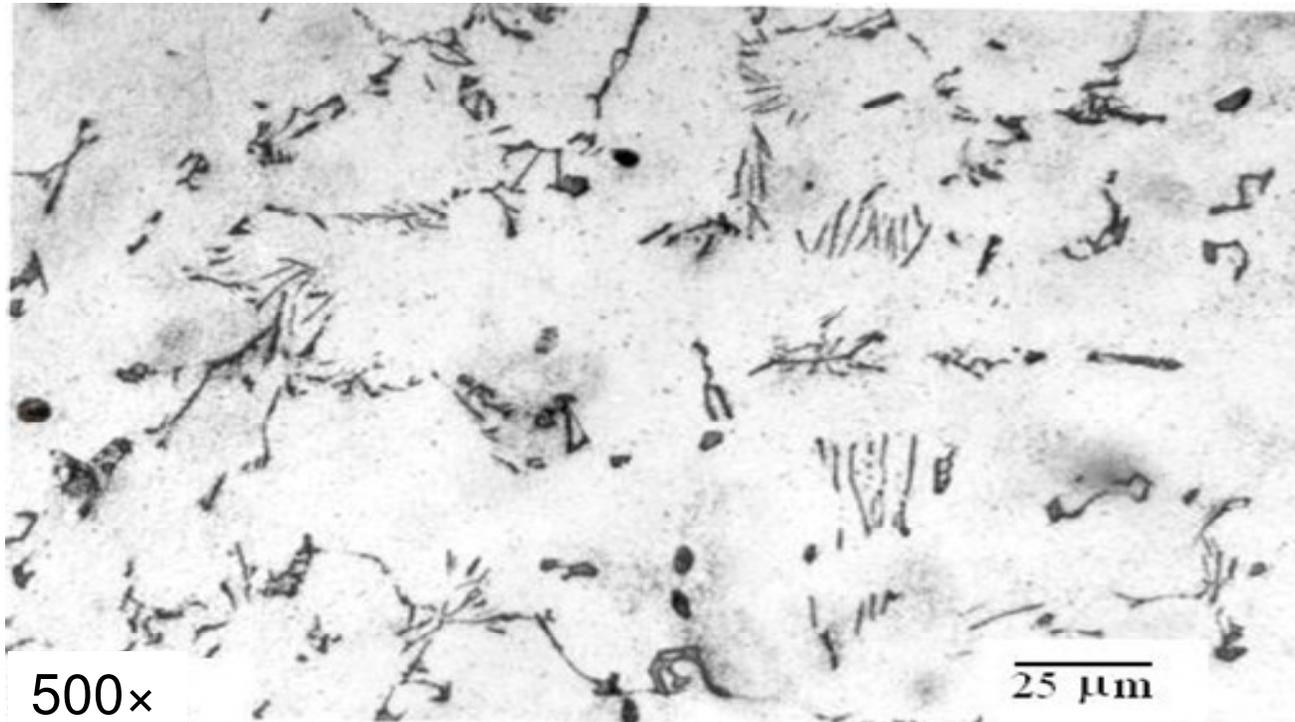


Figure 10. Optical microstructure of matrix alloy (LM 13) (500 × magnification).

23%) but with slight reduction in ductility (by 28%). Fractography analysis revealed that fracture behavior of FCC structured Al matrix alloy was changed from ductile mode of fracture to cleavage mode because of presence of ZrO_2 particulates.

ACKNOWLEDGEMENT

The author wishes to thank Miss. C.J. Sowmya, B.C.A., M.C.A., his colleague for her kind cooperation, help, encouragement and support especially in preparing and embedding the graphics.

REFERENCES

- Awasthi S, Wood JL (1988). 'Mechanical properties of extruded ceramic reinforced Al based composites', *Adv. Ceramic Mater.*, 35: 3449-3458.
- Bowden FP, Tabor D (1964). 'The friction and lubrication of solids. part II, Oxford, UK, Oxford press', 34: 320-331.
- Bensely A, Prabhakaran, Mohan LDA, Nagarajan (2006). G 'Enhancing the wear resistance of case carburized steel (En353) by cryogenic treatment, *Cryogenics*, 45: 747-754.
- Gleiter H (2000). 'Grain refinement of Al-Si alloys', *Acta Mater.*, 48: 1-29
- Guy AG (1967) 'Elements of Physical Metallurgy', Addison-Wesley; USA, pp. 78- 85
- Gilchrist JD (1989). 'Extraction Metallurgy, 3rd Edition, Pergamon press; Great Britain, 8: 148-155.
- Hassan SF, Gupta J (2002). 'Development of high strength Al based composites using elemental Ni particles', *J. Mater. Sci.*, 37: 2467-2477.
- Huang JY, Liao XZ (2003) 'Microstructure of cryogenic treated M2 tool steel, *Mater. Sci. Eng., A*(339): 241-244.
- Hemanth J (2003). 'Effect of high rate heat transfer during casting on the strength, hardness and wear behavior of Al-Quartz particulate MMCs', *J. Eng. Manuf., Part-B*(127): 651-662.
- Hemanth J (2005). 'Tribological behavior of cryogenically treated Al-12%Si alloy/B4C Composites', *WEAR, Elsevier Sc.*, 258: 1732-1745.
- Hemanth J (2007). 'Cryogenic effects during solidification on the wear behavior of Al alloy/glass MMCs', *J. Compos. Mater., Part-A*(38): 395-1402.
- Hemanth J (2008). 'Production, Mechanical and Thermal properties of chilled Al-Quartz (SiO_2p) castable MMC for automotive applications', SAE International World congress paper 2008 no. 2008-01-1093, Detroit, Michigan, USA, pp. 1-8.
- Hemanth J (2002). 'Fracture toughness and wear behavior of Al-boron carbide Particulate composite cast using metallic and non metallic chills' *Mater. Des. Elsevier Sci.*, 23: 41-50.
- Hemanth J (2009). 'Quartz (SiO_2p) reinforced chilled metal matrix composites (CMMCs) for automotive applications, *Mater. Des. Elsevier Sci.*, UK, 30: 323-329.
- Gupta M, Lai MO, Saravananathan D (2000). 'Synthesis, microstructure and property characterization of disintegrated melt deposited Al/SiC composites', *J. Mater. Sci.*, 35: 2155-2164.
- Luo A (1995). 'Processing, microstructure and mechanical properties of cast Mg MMC', *Metallogr. Mater. Trans.*, 26: 2445-2453.
- Lai MO (2000). 'Development of ductile Mg composite material using Ti reinforcement', *J. Mater. Sci.*, 31: 2155-2167.
- Lloyd DJ (1994). 'Particle reinforced Al-Mg metal matrix composites', *Int. Mater. Rev.*, 39: 3-10.
- Murakami Y (2002). 'Metal fatigue: Effects of small defects and non-metallic inclusions', Oxford: Elsevier Sci. Ltd., 35: 123-134.
- Nishijima S, Kanazawa K (1999). 'Fatigue fracture of Engineering Materials. *Struct. Mater. Des.*, 22: 581- 593.
- Oku T, Usui T (1971). 'Neutron irradiation effects on the correlation of flow stress with hardness of some BCC metals', *J. Nucl. Mater.*, 40: 93-103.
- Ogata T, Isawa M, Wu K, Leo F (2002). 'In proceedings of 23rd International symposium on space technology and science, Japan,

45: 658-669.

- Ono Y, Furgi H, Sawada J (2002). 'T Collected abstracts of 2002 Autumn meeting of the Japan Inst. Metals, 12: 89-99.
- Opeka MM, Talmy IG, Zaykoki JA (2004). 'Mechanical properties of ZrB₂ composites', J. Mater. Sci., 32: 5887-5894.
- Petty ER (1960). 'A low temperature inflection in the temperature dependence of hardness of pure metals', J. Inst. Met., 89: 123-129.
- Reed HRE (1964). 'Physical metallurgy principles, 2nd Edition, D. Van Nostrand Company, UK, pp. 56-69.
- Shiozawa K, Lu L, Ishihara S (2002). 'Fatigue fracture of Engg. Material Structures' Mater. Des., 24: 781-789.
- Suemune K, Sakamoto TOT, Ozaki T (1988). 'Manufacture and properties of nitrogen containing Cr-Mn and Cr-Ni austenitic steels for cryogenic use', Adv. Cryo Eng., 34A: 123-132.
- Seung J, Wang H (2005). 'Mechanical and Microstructural properties of Al-ZrO₂ ceramic Composites', Mater. Sci. Eng., 405A: 140-150.
- Saravanan RA, Surappa MK (2000). 'Fabrication and characterization of Al-SiCp particle Composite', Mater. Sci. Eng., 108: 276-285.
- Singh A, Tsai AP (2003). 'Quasicrystal strengthened Mg-Zn-Y alloys by extrusion', Scr. Mater., 49: 417-426.
- Srinivasan TS, Gupta M (1999). 'Microstructure and grain growth behavior of Al alloy MMC processed by disintegrated melt deposition', J. Mater. Eng. Perform., 8: 473-489.
- Tham LM, Cheng L (1999). 'Influence of processing parameters during disintegrated melt deposition processing on near shape synthesis of Al based MMC', Mater. Sci. Technol., 15: 1139-1148.
- Umezawa O, Nagai K, Ishikawa K (1990). 'Microstructure and mechanical behavior of Al based composites, Mater. Sci. Eng., 129: 217-228.
- Wang CR, Yang JM (2002). 'Materials characterization by scanning electron microscopy, Mater. Chem. Phys., 14: 272-286.
- Yamamoto T, Sasamoto IM (2000). 'Extrusion of Al based composites', J. Mater. Sci. Lett., 19: 1053-1064.
- Yuri T, Ono Y, Ogata T (2002). 'Composite structures and properties, J. Jpn. Inst. Metals., 66: 1287-1295.
- Zhang GJ, Guo QG (2002). 'Mechanical and microstructural properties of high strength ceramic based composites', J. Nucl. Mater., 301: 187-195.