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Research Paper

EFFECT OF NANO-ZrO_2 PARTICULATE REINFORCEMENT ON MICROSTRUCTURE AND FRACTURE BEHAVIOR OF CRYOGENICALLY SOLIDIFIED ALUMINIUM ALLOY (LM 13) MMCs

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In the present work, an attempt has been made to develop Al alloy (LM 13) matrix nano composites in sand moulds by using copper end blocks of chill thicknesses of 10, 15, 20 and 25 mm with cryogenic effect. The size of the reinforcement (Nano-ZrO_2) ranges from 50-80 nm being added ranges from 3 to 15 wt. % in steps of 3 wt. %. Cryogenically solidified Nano Metal Matrix Composites were compressed by using hydraulic compression machine. Specimens were prepared according to ASTM standards and tested for their strength, microstructure and fracture toughness. Micro structural studies of the fabricated Nano Composites indicate that there is uniform distribution of reinforcements in the matrix (LM 13). An increasing trend of UTS and fracture toughness has been observed. The best results have been obtained at 12 wt%. The results were further justified by comparing four copper end chill thicknesses of 10, 15, 20 and 25 mm. Finally the Volumetric Heat Capacity of the cryo-chill is identified as an important parameter which affects tensile strength and fracture toughness.

Keywords: Cryogenic effect, Volumetric heat capacity, Fracture toughness, Copper end chills, Cryo-chill

INTRODUCTION

Aluminium alloy based Metal Matrix Composites are widely used in the automobile and aerospace industries because of their improved strength, stiffness and structural rigidity and increased wear resistance over unreinforced Aluminium alloys (Suresh *et al.*, 2003; and Braradeswaran, 2011).

The Nano-Technology has drawn much attention of the researchers to create Nano Composite in the present situation. Aluminium alloy based composites reinforced with nano

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size particles like Graphite, Al_2O_3, Ilmenite ($FeTiO_3$) Silicon carbide, Titanium carbide, ZrO_2, etc., to strengthen the metal matrix. A search of open literature indicates that reinforcement of nano sized particles in the metal matrix such has Aluminium, Magnesium etc results in the improvement of tensile strength, hardness, fracture toughness and wear resistance (Hassan and Gupta, 2007; and Mendoza *et al.*, 2008; and Rasidhar *et al.*, 2013).

Among all the reinforcements used in Aluminium alloy based composites only Nano sized particulates has shown there potential superiority in improving microstructure and mechanical properties with noticeable weight savings (Lloyd, 1994; Hassan and Gupta, 2007; and Rasidhar *et al.*, 2013). Thermally stable ceramic particulates such as ZrO_2 is expected to form a good bonding at metal-reinforcement interface in composite processed using solidification route (Gilchrist, 1989; and Eustathopoulos *et al.*, 1999). For speeding up the directional solidification, cryogenic effect was employed.

Speciality in this investigation is to study the effect of nano-ZrO_2 particulates and its increasing amount/addition on the microstructure and the mechanical response of Al alloy (LM 13). Among various fabrication techniques for composites, stir casting is one of the inexpensive and simple casting route used by many researchers (Sajjadi *et al.*, 2011; and Rasidhar *et al.*, 2013). Solidification of nano composite castings were obtained in sand moulds using copper end chills with the help of cryogenic effect. Thus the solidified composite castings are called Metal Matrix Composites (MMCs).

Different size copper end chill thicknesses of 10, 15, 20 and 25 mm were used to study the Volumetric Heat Capacity (VHC) (Seah and Hemanth, 2007; and Joel Hemanth, 2009). Copper material is selected in this chilling technique since it has high thermal conductivity.

Table 1: Calculated VHC of Copper End Chills of Thicknesses 10, 15, 20 and 25 mm

S. No.	Size of the Chill in mm^3	VHC in J/K
1.	150×75×25	1128.96
2.	150×75×20	903.16
3.	150×75×15	677.37
4.	150×75×10	451.58

VHC is given by the equation, $VHC = V \times ° \times C_p$ Joules/Kelvin.

where: V = Volume, ° = Density (8.96 gm/cm^3) and C_p = Specific heat of the chill material (0.448 J/Kg-K).

EXPERIMENTAL PROCEDURE

In this research nano ZrO_2 particles were reinforced in Al alloy (LM 13). The size of the nano ZrO_2 particles varies from 50-80 nm dispersed in the matrix from 3 to 15 wt. %, in steps of 3 wt. %. Table 2 shows chemical composition of LM 13.

Table 2: Chemical Composition of LM 13

Elements	Cu	Zn	Mg	Si	Ni	Fe	Mn	Pb	Sn	Ti	Al
Wt%	0.7	0.5	1.4	12	1.5	1.0	0.5	0.1	0.1	0.2	Balance

FABRICATION OF THE NANO COMPOSITE

Stir casting technique was used to melt Al alloy since it is simple and economical technique. The required amount of LM 13 was placed in a graphite crucible and heated in a resistance furnace at around 780 °C in an inert atmosphere for about 45 minutes for complete melting. Preheated (up to 310 °C) nano ZrO_2 particles of 3 wt. % were introduced evenly into the molten metal (LM 13) by using special feeding attachment, during which the molten metal was well agitated by a mechanical impeller specially fabricated to create vortex motion and degassing powder was added to avoid the formation of blow holes. The speed of the impeller was maintained at 460 rpm to get the uniform distribution of the reinforcement. This process was repeated for 6, 9, 12 and 15 wt. % of nano ZrO_2.

The molten nano composite material was next poured into a foundry sand mould prepared according to AFS standards. And the mould size is (125 × 50 × 35) mm. For Cryogenic effect, copper end chill block of size (150 × 75 × 50) mm was placed adjacent to other end of the mould as shown in the Figure 1 with copper end chill 10 mm in which arrangement was made to circulate the liquid nitrogen. Copper end chill in steel hallow block for passing liquid nitrogen is shown separately in the Figure 2. Before Pouring and during pouring of the molten mixture with 3 wt. % of nano ZrO_2, liquid nitrogen was passed into the passage provided in the copper end chill block of 10mm thickness. The above procedure was repeated for 6, 9, 12 and 15 wt. % reinforcement. The cryogenically solidified nano metal matrix composites were compressed in a hydraulic press for obtaining the perfect flatness on both sides of rectangular cast block. The above same procedure was repeated for copper end chill thicknesses of 15, 20 and 25 mm.

Figure 1: Experimental Arrangement for Casting Nano Metal Matrix Composites

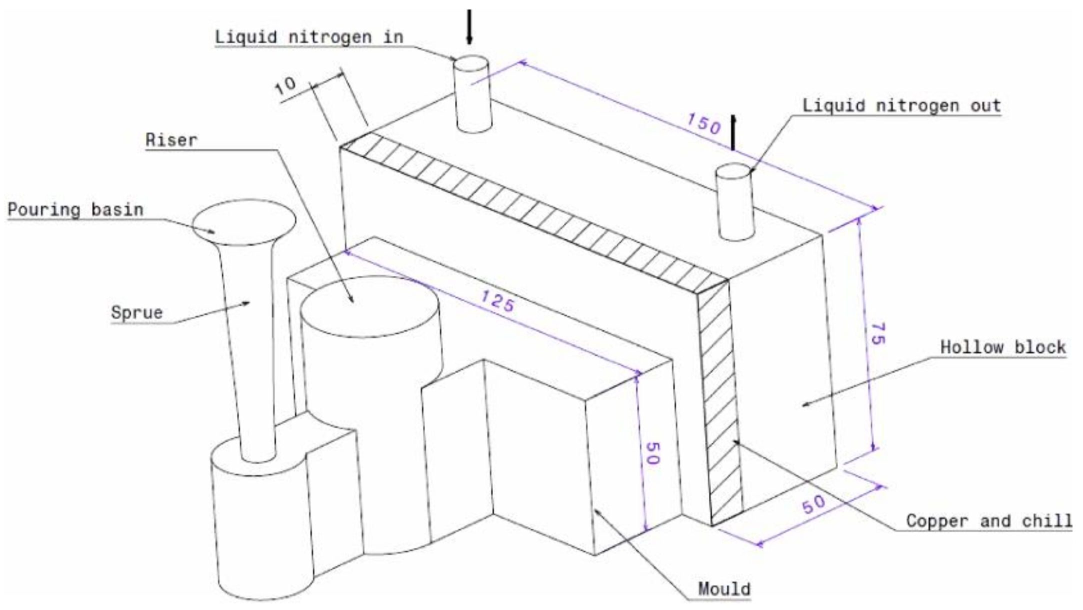
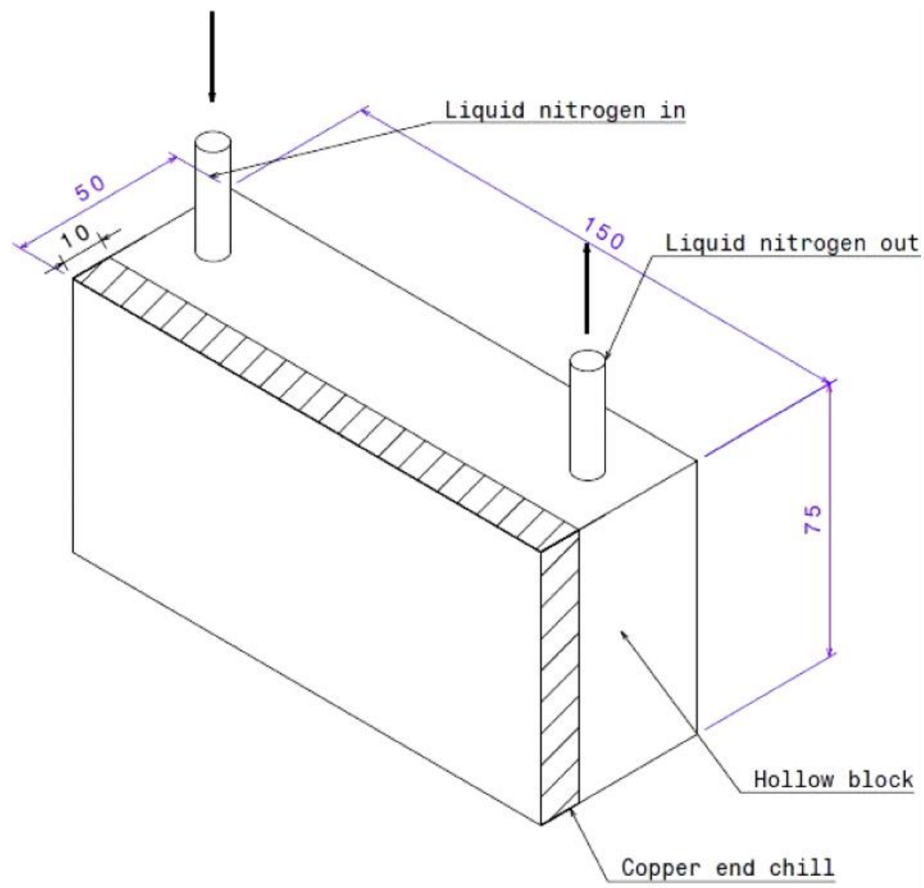


Figure 2: Copper End Chill in a Steel Hollow Block (Brazed) for Passing Liquid Nitrogen



TESTS CARRIED OUT

Micro Structural Examination

Microstructure characterization was conducted on all the polished specimens with Keller's reagent using OLYMPUS metallographic microscope to investigate morphological characteristics of grains, reinforcement distribution and interfacial integrity between matrix material and the reinforcement.

It is observed from the above set of photomicrographs that the molten material of MMC solidified under cryogenic condition suffers a severe super cooling. This results in high rate of heat transfer and rapid cooling of the hot melt in MMCs samples. Hence the critical size of the solidified melt is reduced and a greater number of nuclei are generated

Figure 3: Al alloy (LM 13) ingot at 400 X Magnification

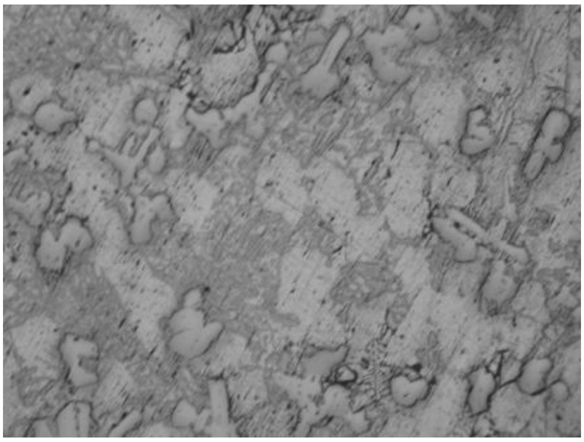
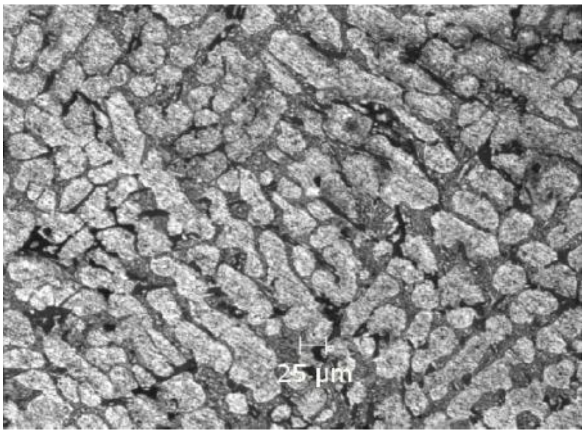
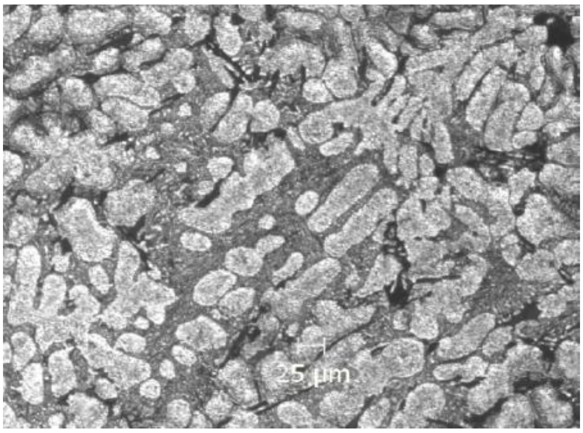


Figure 4: Micro Structure Taken at 400 X Magnification

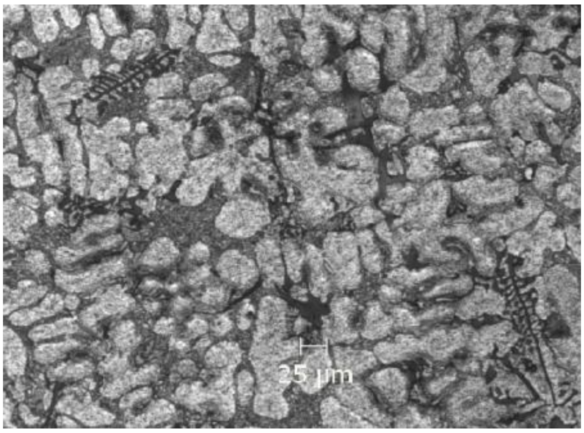
(a) 3 wt. % of Reinforced MMC



(b) 6 wt. % of Reinforced MMC



(c) 9 wt. % of Reinforced MMC



(d) 12 wt. % of Reinforced MMC

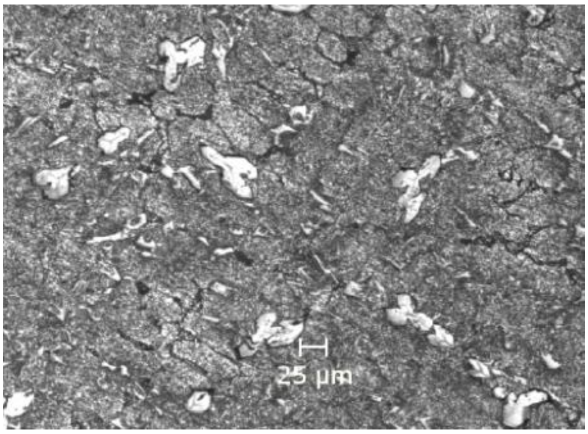
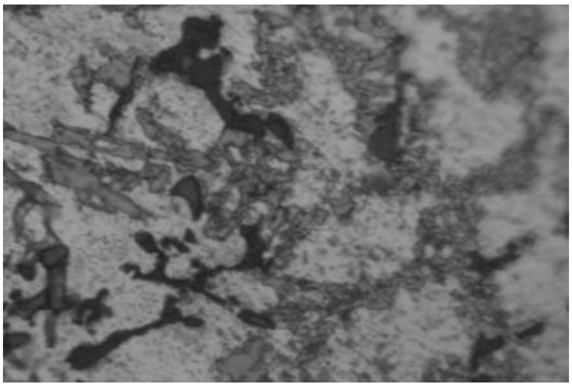


Figure 5: Optical Microstructure of Cryo Chilled Nano Composite Containing 15 wt. % Reinforcement Microstructure Taken at 400 X Magnification



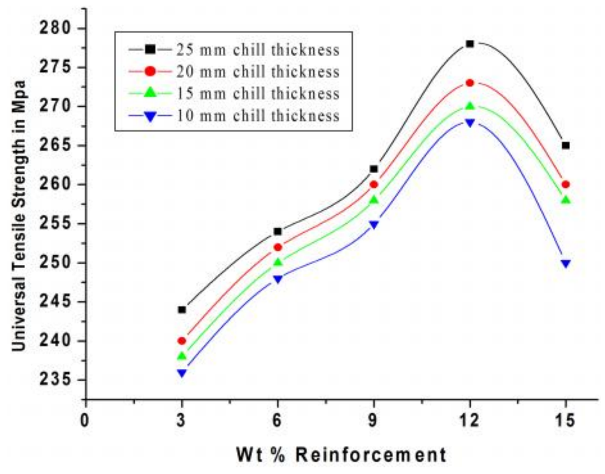
causing a finer microstructure. In addition to the super cooling of the melt, the stirring action of reinforcement segregated reinforcement particles do not have time to settle down due to the density difference between matrix material and the dispersoid and this result in more uniform distribution of nano ZrO_2 particles in the matrix material. The cryogenic effect during solidification causes stronger bonding between the matrix material and the reinforcement. This shows the wettability was good between the particles and the matrix material with the cryogenic cooling. These two factors lead to improved mechanical properties of the MMCs. Thus the strong bonding between the dispersoid and the matrix material causes more effective load transfer. Figure 3 shows microstructure of matrix material (LM 13).

Microstructure characteristics of hydraulically pressed MMCs are discussed in terms of distribution of reinforcement and matrix-reinforcement interfacial bonding (Figures 4a, 4b, 4c and 4d). Using 25 mm thick copper end chill reveal uniform distribution of the reinforcement with very limited clusters, good reinforcement-matrix material interfacial integrity, improved grain refinement with minimum porosity. At the same time, due to gravity of nano ZrO_2 associated with parameters such as good stirring action in the molten stage of LM 13, good wetting of the preheated nano ZrO_2 by the melt of the material. Metallographic studies of the hydraulically pressed samples revealed that the matrix material is fully recrystallized. Figure 5 shows the non-uniform distribution of the reinforcement when excess 15 wt. % of reinforcement was added.

Universal Tensile Strength (UTS) Test

Figure 6 shows that UTS are higher for all the MMCs as compared against the molten matrix material (LM 13). When the reinforcement content nano-ZrO_2 increases from 3 wt. % to 12 wt. % for all four sizes of the copper end chills (10, 15, 20 and 25 mm thick) beyond which the trend reverses. The chill thickness increases UTS values also increases, conforming that the heat capacity of the chill significantly enhances the UTS values. UTS of the unchilled LM 13 is 135 M Pa. It is also observed that MMCs containing 12 wt. % using 25 mm copper end chill thickness invariably has the highest tensile strength.

Figure 6: Graph Showing wt% of Reinforcement V/s UTS Values



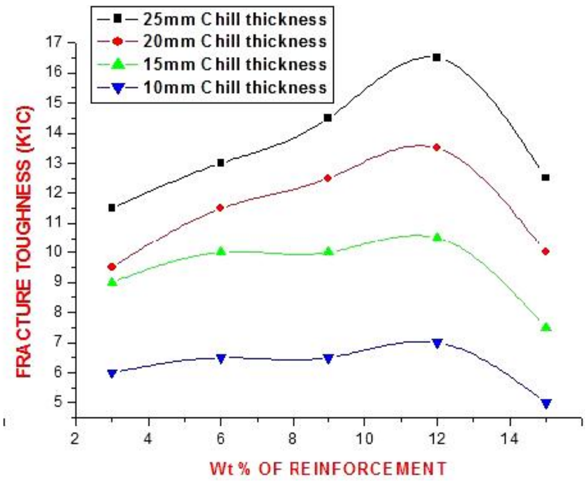
Fracture Toughness Test

Fracture toughness tests were conducted on all MMCs by using a closed loop INSTRON servo hydraulic material testing system. This method involves 3-point bend testing (in accordance with ASTM E399 1990 standard) of machined specimen which was pre-cracked by fatigue loading.

The fracture toughness of the MMCs using 10, 15, 20 and 25 mm copper ends and reinforcement content are shown in Figure 7 and indicates the values increasing from 3 wt. % to 12 wt. % of the reinforcement in the increasing trend. Comparing these results it can be seen that increasing the reinforcement content and cryo-chilling seems to have an effect of fracture toughness on the cast nano composites.

It is also observed that MMCs containing 12 wt. % using 25 mm copper end chill thickness invariably has the highest fracture

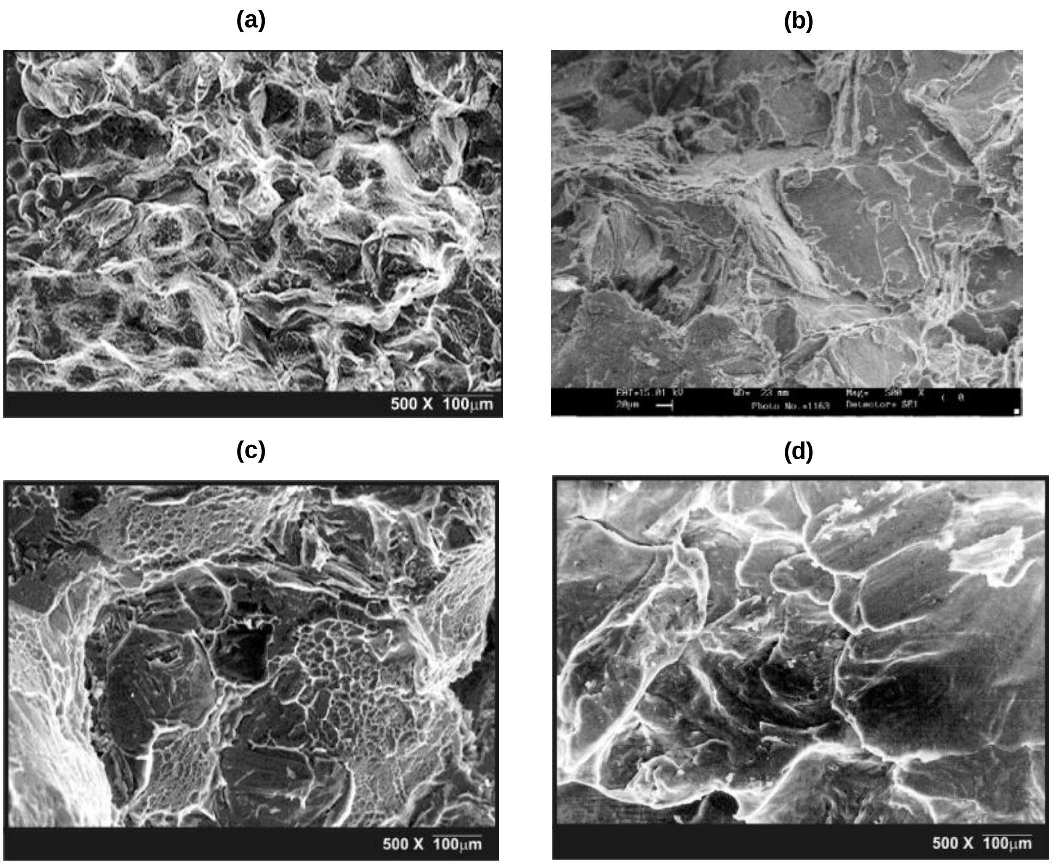
Figure 7: Graph Showing wt. % of Reinforcement V/s Fracture Toughness Values



toughness. Further it is observed that fracture toughness value decreases when 15 wt. % reinforcement were present.

These results show that the matrix material (LM 13) dense, stronger and accommodate the reinforcement (nano ZrO_2) rigidly. There was a strong bonding between the matrix material and reinforcement (up to 12 wt. %) and this could lead to a greater strength and fracture toughness of the MMCs compared with the monolithic alloys. The mechanism which controls the variation of fracture toughness of the MMCs dependent on microstructure and strain range. Fracture toughness of the unchilled LM 13 is 4.0 M Pa√m.

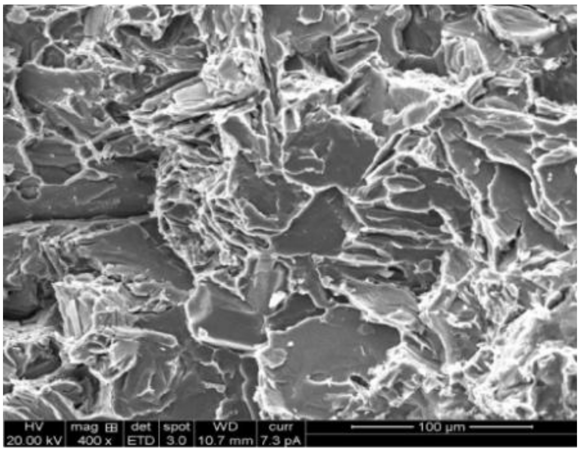
Figure 8 – a, b, c and d: Fractographs of Cryogenically Solidified Nano Metal Matrix Composites 3 wt. % to 12 wt. % of Reinforcement Using 25 mm Copper End Chill Thickness at 500 X Magnification and Scale 100 µm



Fracture Surface Analysis

SEM fractographs Figure 8 (a, b, c and d) shows ductile fracture and dimple formation when 3 wt. %-12 wt. % reinforcement was added. Large areas of the fracture surface were covered with biomodal distribution of dimples, indicative of ductile rupture. However, fracture of MMCs show transition for ductile mode of failure to cleavage mode because of presence of nano ZrO_2 particles. Figure 9 Factograph of MMCs with 15 wt. % reinforcement (nano ZrO_2) at 400 X magnification.

Figure 9: Factograph of MMCs with 15 wt. % Reinforcement (Nano ZrO_2) at 400 X Magnification



DISCUSSION

Several researchers have fabricated Aluminium Metal Matrix Composites (AMMCs) and Nano Metal Matrix Composites (NMMCs) by reinforcing with different dispersoids in various melting and solidification routes. The author has studied a few researches and included in the present paper. Hassan and Gupta (2007) had fabricated NMMC. Mechanical properties revealed that the presence of nano ZrO_2 reinforcement lead to significant improvement in the hardness and tensile strength. Residhar *et al.* (2013) were fabricated and investigated on properties of Ilmenite ($FeTiO_3$) based Al-Nano composites by stir casting process. In their research work also test results revealed that the presence of nano ZrO_2 reinforcement lead to significant improvement in the strength and hardness of Nano Metal Matrix Composite. Kushal Kumar *et al.* (2013) fabricated using Al alloy based MMC reinforced with ZrO_2 particulates. In their research work also, test results revealed that the presence of nano ZrO_2 reinforcement lead to significant improvement in the strength and fracture toughness of Nano Metal Matrix Composite. In this present investigation, the test results of strength and fracture toughness of the nano metal matrix composite revealed significant improvement. Strength and fracture toughness of the cryogenically chilled nano metal matrix composite are significantly dependent on the wt. % of reinforcement and rate of chilling (depends on the thickness of the chill material used). The strength and fracture toughness of the fabricated composite are superior to the unchilled matrix material. There is no benefit in increasing the wt. % of reinforcement content beyond 12 wt. % since it will result in a deterioration of these properties.

CONCLUSION

Stir casting technique and cryogenic chilling used for the fabrication of nano ZrO_2 composites reveals the following:

- Microstructures of cryochilled nano-composites are finer than that of the matrix material.
- The interfacial bonding between the matrix material and the reinforcement is stronger of the cryochilled nano-composites.

- Tensile strength characterization of composite for different thicknesses of copper chill block containing 12 wt. % reinforcement revealed that the presence of nano ZrO_2 particulates in cryochilled matrix significant improvement in the tensile strength.
- Fracture toughness also increases from 3 wt. % to 12 wt. % of adding the reinforcement for all the four chills.
- Strength and fracture toughness was improved by 48.56% and 25% respectively with 12 wt. % of reinforcement cast with 25 mm copper end chill.
- Further addition of reinforcement in the metal matrix universal tensile strength and fracture toughness reduces. As the chill thickness increases the strength of the MMCs also increases.
- At 15 wt. % of reinforcement non-uniform distribution was observed.
- Fractography analysis revealed that the fracture behaviour of FCC structured Al matrix alloy has changed from ductile mode of fracture to cleavage mode because of the presence of nano ZrO_2.

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