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Preparation and Investigation on Properties of Cryogenically Solidified Nano Metal Matrix Composites

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ABSTRACT
In the present work, AL-alloy containing 12% silicon (LM 13) matrix nano composites were fabricated in sand moulds by using copper end blocks of copper end chill thickness 10 &15 nm with cryogenic effect . The size of the reinforcement (NanoZro$_2$) ranges from 50-80nm 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, hardness and fracture toughness. Micro structural studies of the fabricated Nano Composites indicate that there is uniform distributions of reinforcements in the matrix materials (LM 13). An increasing trend of hardness, UTS & fracture toughness has been observed. The best results have been obtained at 12 wt %. The results were further justified by comparing two copper end chill thickness 10 &15 mm. Finally the Volumetric Heat Capacity of the cryo-chill is identified as an important parameter which affects mechanical properties.

***Keywords* -** Cryo-chill, Cryogenic effect, Copper end chills, Fracture toughness, Volumetric Heat Capacity.

I. INTRODUCTION

During the last decade, numerous researchers have investigated for the development of Nano Metal Matrix Composites. It is found that Al- alloy Nano Metal Matrix Composites play an important role than Al alloy Matrix Composites especially in the field of automobile, aerospace & military industries. Because of the improvement in mechanical properties like high specific strength, good wear resistance, fracture resistance, thermal and electrical properties. As a result, their applications are extended into the fields of automotive, aerospace & military industries. Al alloy based matrix Nano Composites are also advanced materials. These materials are fabricated by reinforcing with Nano sized hard particles/ceramics to get tailored properties. So far, Nano sized particles used in Al based Matrix Nano Composites are SiC, TiC, Al_20_3, TiCr, Boron carbide, Zirconia etc to strengthen the composites(1-4). Al alloy containing 12% silicon are widely used in internal combustion engines parts such as pistons, cylinder blocks, cylinder heads since it has high resistance to wear, corrosion & thermal conductivities but exhibit poor tribological property & fracture toughness character. Al alloy based Matrix Composites freeze over a wide temperature range and are difficult to feed into a mould cavity during solidification (3, 5). The dispersed porosity caused by the pasty type of solidification of wide freezing range alloy castings can be effectively reduced by the use of Cryogenic chilling during sand mould casting. Many researchers have used the application of chills in the production of Metal Matrix Composites during solidification of castings. But cryogenic chilling on solidification is more effective because the shrinkage problems and pasty zones in the sand moulds during solidification can be reduced or avoided.

Among various methods for the production of MMCs or AMCs or NMMCs, stir casting method is one of the simple, economical & useful method. In this method, uniform distribution of dispersoid in the metal matrix can be achieved (8) since ZrO_2 is in Nano size.

In the present work, the main aim was to fabricate Nano Metal Matrix Composites by combining the desirable properties of the Al alloy (LM – 13) and the reinforcement Nano ZrO_2 by using stir casting method. Adding the reinforcement in the melt of LM – 13 in the order of 3 wt%. To achieve best results during solidification of the molten mixture, copper end chill blocks were used in the sand moulds. For cryogenic effect, liquid nitrogen (at -80°C) was passed in the passage provided in the chill blocks.

II. EXPERIMENTATION

In this research nano – ZrO_2 particles were reinforced in Al alloy (LM13). 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 1 shows chemical composition of LM13.

Table 1: Chemical composition of LM13.

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

III. FABRICATION OF THE NANO COMPOSITE

The required amount of LM13 was placed in a graphite crucible and heated in a resistance furnace at around 760 ^{0}C in an inert atmosphere for about 50 minutes for complete melting. Preheated (up to 320 ^{0}C) nano ZrO_2 particles of 3Wt% were introduced evenly into the molten metal (LM13) 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 480 rpm to get the uniform distribution of the reinforcement. This process was repeated for 6, 9, 12 & 15Wt% 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 fig1 with copper end chill 10mm 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 Fig2. Before Pouring and during pouring of the molten mixture, liquid nitrogen was passed into the passage provided in the copper end chill block of 10mm chill thickness. The above procedure was repeated for 6, 9, 12 & 15 Wt% additives. 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 thickness of 15mm.

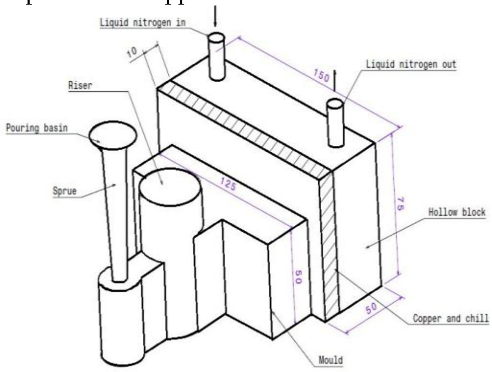


Fig. 1: Sand mould with Copper end chill with arrangements for passing liquid nitrogen

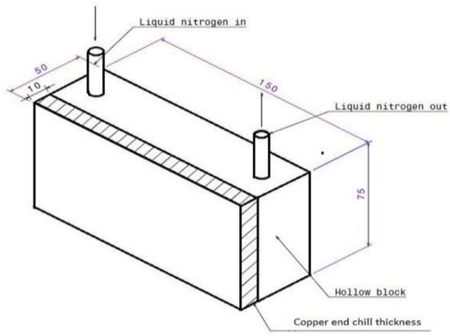
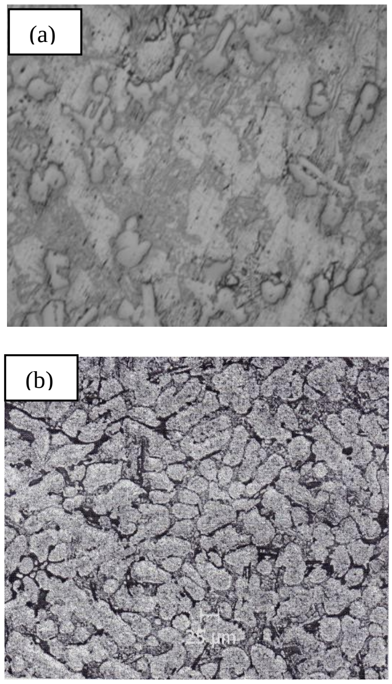


Fig. 2: Copper end chill in a steel hollow block (Brazed) for passing liquid nitrogen

IV. MICROSTRUCTURAL CHARACTERIZATION



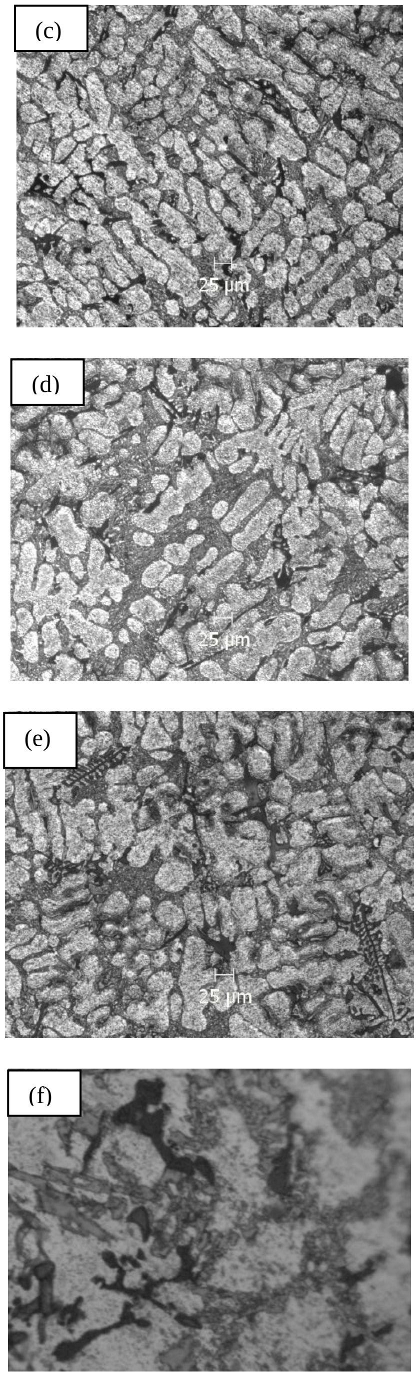


Fig. 3a: LM13 Al alloy ingot, Fig. 3b: 3 Wt% of Nano ZrO_2, Fig. 3c: 6 Wt% of Nano ZrO_2, Fig. 3d: 9 Wt% of Nano ZrO_2, Fig. 3e: 12 Wt% of Nano ZrO_2 & Fig. 3f: 15 Wt% of Nano ZrO_2. Micro Structure taken at 100X magnification.

It is observed from the photomicrographs that the molten material of CNMMC solidified under cryogenic effect. This results in high rate of heat transfer and rapid cooling of the hot melt in CNMMC's samples. Hence the critical size of the solidified melt is reduced and a greater number of nuclei are generated causing a finer microstructure. In addition to the rapid cooling of the melt, the stirring action of reinforcement particles do not have time to settle down due to the density difference between matrix material and the reinforcement and this results 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 effect. These two factors lead to improved mechanical properties of the CNMMC's. Thus the strong bonding between the nano ZrO_2 and the matrix material causes more effective load transfer. Fig3a shows microstructure of the matrix material (LM 13 alloy).

Microstructure characteristics of hydraulically pressed CNMMC's are discussed in terms of distribution of reinforcement and matrix – reinforcement interfacial bonding (Fig3b, 3c, 3d & 3e). Using 10 and 15mm thick copper end chill revealed uniform distribution of the reinforcement with very limited clusters, good reinforcement – matrix material interfacial integrity, improved grain refinement. At the same time, due to gravity of nano ZrO_2 associated with parameters such as good stirring action in the molten stage of LM13, 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. Fig.3f shows the non-uniform distribution of the reinforcement when 15 Wt% of nano $ZrO_2$was added.

V. HARDNESS AND STRENGTH TEST

Vickers micro hardness testing were conducted after the microscopic study on all the CNMMC's specimen using metsuzawa MXT50 digital hardness tester using 25 gf indentation load in accordance with ASTM E18-94 standards. Copper was selected in this investigation since it has high thermal conductivity and calculated VHC was high. The results of micro hardness test conducted on all hydraulically pressed CNMMC's samples revealed an increasing trend in matrix material with increase of reinforcement up to 12Wt% for both the chill thickness of 10mm and 15mm. Results of hardness measurement revealed that increase in the nano ZrO_2 content and the chill thickness leads to a significant increase in the hardness. Because of the presence of nano ZrO_2 particles in the matrix material lead to higher resistance to the localized deformation during indentation and the cryogenic effect. Fig4 and Fig5 show the nature of the graph for copper end chill thickness 10mm and 15mm respectively. UTS are higher for all the CNMMC's as compared against the molten matrix material (LM13). When the reinforcement content nano ZrO_2 increases from 3Wt% to 12Wt% for both size of the copper end chill

(10mm and 15mm 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.
Equation for calculating Volumetric Heat Capacity. Volumetric Heat Capacity of the chill material (Copper) is given by,

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

Where,
V=Volume of the chill,
ρ = Density of the chill material (8.96 gm/cm^3),
C_p = specific heat of the chill material (0.448 J/Kg-K).

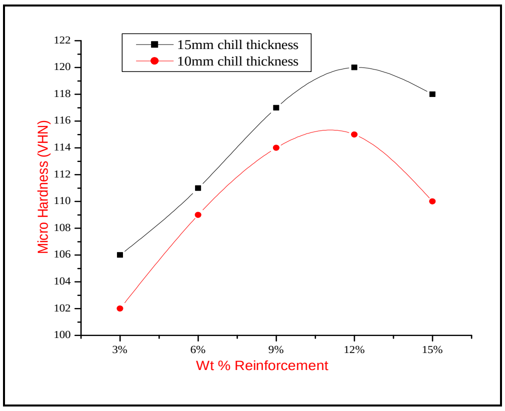


Fig. 4: Graph showing Wt % of reinforcement v/s Vickers Micro Hardness Test

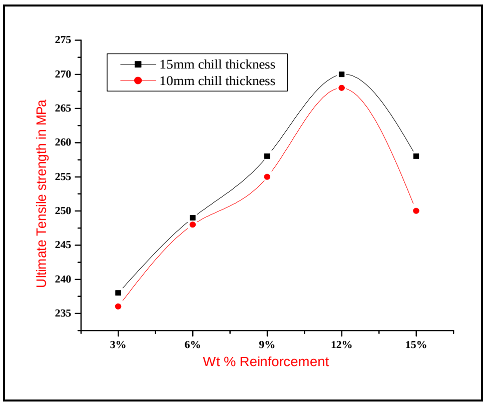


Fig. 5: Graph showing Wt % of reinforcement v/s UTS

VI. Fracture Toughness

Fracture toughness tests were conducted on all CNMMC's 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 which was pre-cracked by fatigue loading.

The fracture toughness of the CNMMC's using 10 and 15 mm copper ends and reinforcement content is shown in fig 6. Comparing these results, it can be seen that increasing the reinforcement content and cryo-chilling seems to have an effect on fracture toughness of the cast nano composites. Fracture toughness graph indicates that it increases from 3 wt% to 12 wt% of the reinforcement in the increasing trend.

It is also observed that CNMMC's containing 12 wt% using 15 mm copper end chill thickness invariably has the highest fracture toughness. Further it is observed that fracture toughness value decreases when 15 wt% reinforcement was present.

These results show that the matrix material (LM13) 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 CNMMC's compared with the monolithic alloys. The mechanism which controls the variation of fracture toughness of the CNMMC's are dependent on microstructure and strain range.

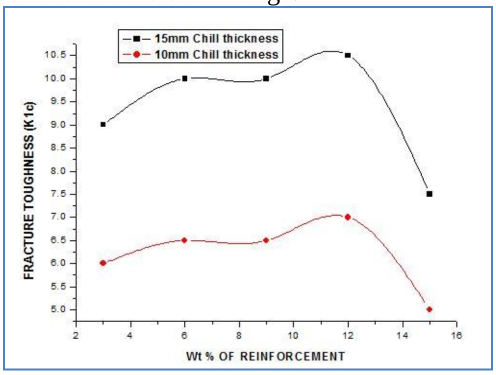
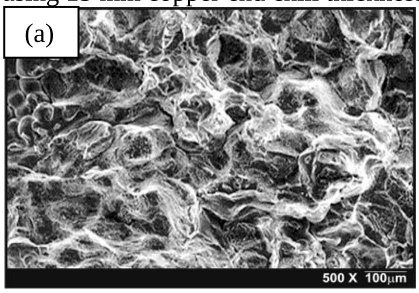


Fig 6: Graph shows the Wt% of reinforcement v/s Fracture toughness (K1c).

VII. Fracture Surface Analysis

SEM fractographs fig 7 (a-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 CNMMC's show transition for ductile mode of failure to cleavage mode because of presence of nano ZrO_2 particles. Fig (e) shows the fractographs of matrix alloy (LM13) cryogenically chilled using 15 mm copper end chill thickness.



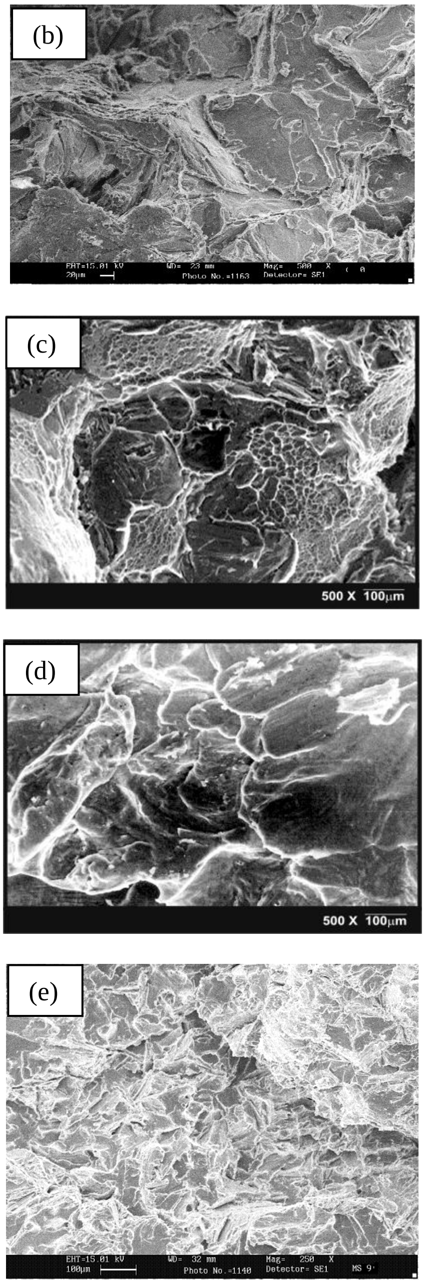


Fig 7a,b,c & d: Fractographs of cryogenically solidified nano metal matrix composites 3 Wt. % to 12 Wt. % of reinforcement using 15 mm copper end chill thickness at 500X magnification and scale 100 µm. Fig 7e factograph of cryogenically solidified metal matrix material (LM13).

VIII. Conclusion

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

- Microstructure of cryochilled nanocomposites are finer than that of the matrix material and the interfacial bonding between the matrix material and the reinforcement is stronger of the cryochilled nanocomposites.
- Mechanical property characterization of composite cast using 15mm and 10mm thick copper chill block containing 12Wt% reinforcement revealed that the presence of nano ZrO_2 particulates in cryochilled matrix significant improved hardness and strength. 15 mm copper end chill has the highest hardness and strength.
- Further addition of reinforcement in the metal matrix, mechanical properties reduces. As the Chill thickness increases the mechanical properties of the CNMMC's also increases. When 15 Wt% reinforcement was added, non-uniform distribution was observed.
- Fracture toughness also increases from 3 wt% to 12 wt% of adding the reinforcement. 15 mm copper end chill has the highest fracture toughness.
- Fractography analysis revealed that fracture behaviour of FCC structured Al matrix alloy was changed from ductile mode of fracture to cleavage mode because of the presence of nano ZrO_2.

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