PREPARATION, CHARACTERIZATION AND PROPERTY EVALUATION OF CRYOGENICALLY SOLIDIFIED MMC'S

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Abstract - *Materials are selected for a given application is mainly based on the material's properties. Most engineering structure is required to carry loads, so the material property of greatest interest is its strength. Strength alone is not always enough. Some engineering structures are required light weight as in aircraft. The purpose of the project is to study above mechanical properties of Aluminum LM13 with zirconium oxide in cryogenic condition by varying zirconium oxide with 2, 4, 6 and 8% particulate using sand casting process with different chilling thickness 15mm and 20mm. An improved mechanical property of composites makes them very useful for various applications in many areas from technological point of view. The microstructure and hardness of the fabricated composites were analyzed and reported.*

***Key Words*: Aluminum LM13, zirconium oxide, Sand casting, Cryogenic solidification, Mechanical properties testing.**

1. INTRODUCTION

A composite material is a material consists of two or more constituents. The constituents are combined at microscopic level and not soluble each other. Generally, a composite material is composed of reinforcement such as fibbers, particles/particulates, flakes embedded in a matrix (metals, polymers). The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix. When designed properly, the new combined material exhibits the better strength than would each individual material.

A metal matrix composite (MMC) is composite material with at least two constituent parts, one being a metal necessarily, the other material may be a different metal or another material, such as a ceramic or organic compound. When at least three materials are present, it is called a hybrid composite.

Metal matrix composites (MMCs) usually consist of a low-density metal, such as aluminium or magnesium, reinforced with particulate or fibers of a ceramic material, such as silicon carbide or graphite. Compared with unreinforced metals, MMCs offer higher specific strength and stiffness, higher operating temperature, and greater wear resistance, as well as the opportunity to tailor these properties for a particular application. However, MMCs also have some disadvantages compared with metals. Chief among these are the higher cost of fabrication for high-performance MMCs, and lower ductility and toughness. Presently, MMCs tend to cluster around two extreme types. One consists of very high performance composites reinforced with expensive continuous fibers and requiring expensive processing methods. The other consists of relatively low-cost and low-performance composites reinforced with relatively inexpensive particulate or fibers.

The cost of the first type is too high for any but military or space applications, whereas the cost/benefit advantages of the second type over unreinforced metal

1.1 Materials used

The raw materials used in this work are

- Alluminium alloy LM13 (Matrix)
- Zirconium oxide (Reinforcement)
- Chill box

A) Aluminum alloy LM13

LM13 (EN 1706 AC-48000)-Aluminium casting alloy. This alloy conforms to BS1490:1988 LM13. Casting are standardized in the perception treated (TE) condition, solution treated, artificially aged and stabilized (TF7) condition and the fully heat treated (TF) condition.

Figure 1.1: Aluminium alloy LM 13

Table -1.1: Chemical composition of aluminium alloy LM13

Aluminium alloy	LM13
copper	0.7-1.5
Magnesium	0.8-1.5
Silicon	11.0-13.0
Iron	0.8
Manganes	0.5
Nickel	2.0-3.0
Zinc	0.1
Lead	0.1
Tin	0.1
Titanium	0.1

B) Zirconium oxide:

Zirconium oxide (ZrO2), sometimes known as zirconia (not to be confused with zircon), is a white crystalline oxide of zirconium. Its most naturally occurring form, with a monoclinic crystalline structure, is the mineral baddeleyite. A dopant stabilized cubic structured zirconia, cubic zirconia, is synthesized in various colours for use as a gemstone and a diamond simulant.

Figure 1.2: Zirconium oxide

Table 1.2: Properties of Zirconium Dioxide

Chemical formula	ZrO_2
Molar mass	123.218 g/mol
Appearance	White powder
Density	5.68 g/cm^3
Melting point	2,715 ^{o}C (4,919 ^{0}F; 2,988 K)
Boiling point	4,300 ^{o}C (7,770 ^{0}F; 4,570 K)
Solubility in water	negligible
Solubility	Soluble in HF, and H_2SO_4
Refractive index	2.13

Table 1.3: Thermochemistry of Zirconium oxide

Standard molar entropy	50.3 J K^{-1} mol^{-1}
Standard enthalpy of formation	-1080 KJ/mol

C) Chill box

Figure 1.3: chill box

The chill box is made up of mild steel with one side brazed with copper metal; it is used in sand casting while pouring molten metal (Al LM13+Zr02) in order to maintain uniform solidification). There are two chill box is used for solidification purpose with copper metal thickness 15mm and 20mm respectively.

2. METHEDOLOGY

Fabrication (Composite material)

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Preparation of the test specimen

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Conduct the test on prepared specimen

⇩

Result and Discussion

⇩

Conclusion

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Fabrication

- Aluminium LM13 ingot of required quantity are weighted and placed in a graphite crucible.

Figure 2.1: Aluminium alloy LM13 and Zirconium oxide weighing

- The furnace top is closed refractory material and furnace is switched on and heated to 8000C till 2 hour and allowed to stabilize it for 15 min.

Figure 2.2: shows furnace and stirrer

- Preheated Zirconium dioxide particles added to molten aluminium alloy.
- Stirrer is to be immersed about 3/4 of molten alloy and the rotation speed of stirrer is 500rpm. The stirrer is coated by ceramic and graphite.
- After stirring, the Hexachlorophene tablet is applied to molten metal for degassing aluminium alloys and also grain refining.
- After applying tablet wait 10 minutes then, flux is applied to molten metal. Flux is used for the prevention of non-metal inclusion, oxides, absorption of hydrogen gas to prevent consumption of magnesium.
- Passing the liquid nitrogen (-196 0C) before pouring the molten metal mixture to create cryogenic effect in copper end chill block.
- The chill box is placed at any one side of sand cavity, this allows the molten metal to allow uniform cooling and solidification of metal.
- First the chill box is filled with liquid nitrogen then the molten metal, the liquid nitrogen is poured continuously till it absorbs the full heat.

Figure 2.3: Passing of liquid nitrogen into the chill box and molten metal into the cavity

- Pouring the molten metal mixture in to the sand mould. At the same time liquid nitrogen was also circulated.

Figure 2.4: The solidified composite is separated by breaking the mould and placed according to weight percentage.

- After pouring the cast is allowed to solidify at room temperature. Once mould has been cooled cast has been taken out.
- Aluminium lm13 metal matrix composites with Zirconium dioxide reinforcement of 2%, 4%, 6% and 8%by weight were cast, by the above mentioned procedure.

2.1 Preparation of test specimen

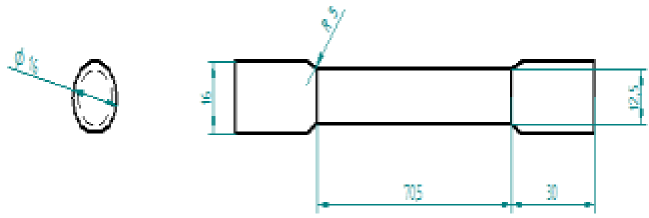
- COMPOSISTION OF SAMPLES

The proposed samples and their compositions are given in the table

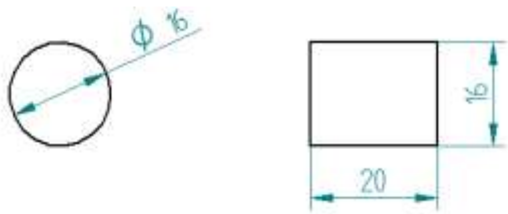
Table 2.1.1: Composition of metal matrix composite samples

SPECIMEN	ALUMINIUM LM 13 %	ZrO_2
A	98	2
B	96	4
C	94	6
D	92	8

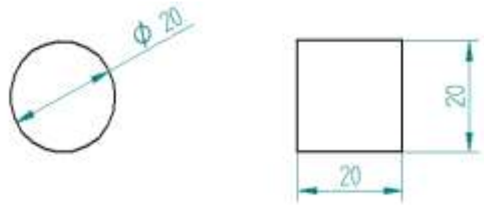
- **Tensile test specimen**



- **Compression test specimen**



- **Hardness test specimen**



A) TENSILE TEST

Tensile testing is most often carried out at a material testing laboratory. The most common testing machine used in tensile testing is the universal testing machine. This type of machine has two crossheads one is adjusted for the length of the specimen and the other is driven to apply tension to the test specimen.

Table 2.1.2: Tensile test results

Al LM13+2%ZrO_2			
Specimen	**Ultimate load in KN**	**Ultimate strength in N/mm²**	**Modulus of elasticity in Gpa**
Without chill thickness	8.86	72.18	21.24
15mm Chill thickness	21.14	155.55	23.24
20mm Chill thickness	23.01	157.25	24.17

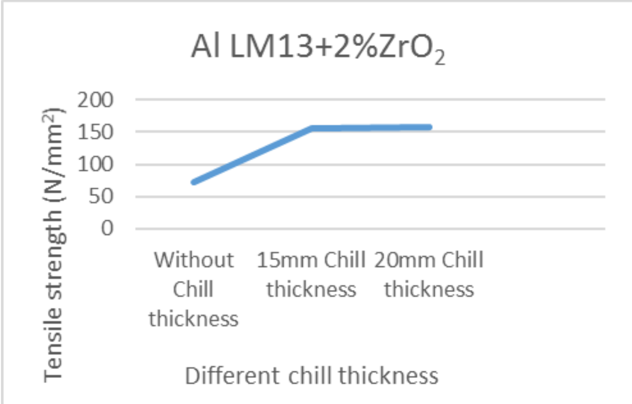


Chart 2.1.1: Effect of Cryogenic solidification on tensile strength

Figure indicates the effect of cryogenic solidification on tensile strength of the composites of Al LM13+2%ZrO_2. As the chill thickness increases, the tensile strength of the composite increases. The maximum tensile strength of 157.25N/mm² is obtained.

Table 2.1.3: Tensile test results

Al LM13+4%ZrO_2			
Specimen	**Ultimate load in KN**	**Ultimate strength in N/mm²**	**Modulus of elasticity in Gpa**
Without chill thickness	8.22	73.20	16.27
15mm Chill thickness	20.13	159.78	20.41
20mm Chill thickness	21.65	163.77	22.74

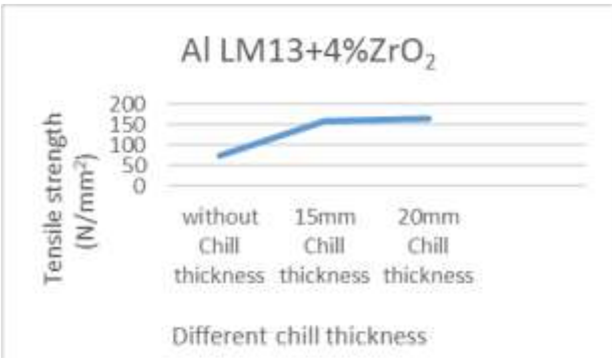


Chart 2.1.2: Effect of Cryogenic solidification on tensile strength

Figure indicates the effect of cryogenic solidification on tensile strength of the composites of Al LM13+4%ZrO_2. As the chill thickness increases, the tensile strength of the composite increases. The maximum tensile strength of 163.77N/mm² is obtained.

Al LM13+6%ZrO_2			
Specimen	**Ultimate load in KN**	**Ultimate strength in N/mm²**	**Modulus of elasticity in Gpa**
Without chill thickness	8.07	64.78	22.79
15mm Chill thickness	18.03	127.44	24.75
10mm Chill thickness	20.88	143.55	23.89

Table 2.1.4: Tensile test results

Al LM13+8%ZrO_2			
Specimen	**Ultimate load in KN**	**Ultimate strength in N/mm²**	**Modulus of elasticity in Gpa**
Without chill thickness	9.81	79.94	8.01
15mm Chill thickness	14.01	121.14	18.76
20mm Chill thickness	15.20	123.86	21.07

Table 2.1.5: Tensile test results

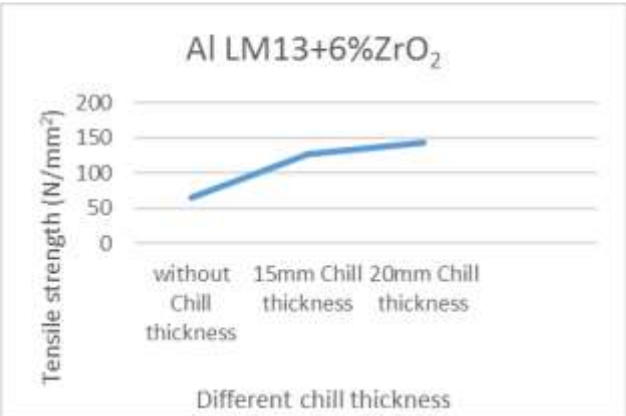


Chart 2.1.3: Effect of Cryogenic solidification on tensile strength

Figure indicates the effect of cryogenic solidification on tensile strength of the composites of Al LM13+6%ZrO_2. As the chill thickness increases, the tensile strength of the composite increases. The maximum tensile strength of 143.55N/mm² is obtained

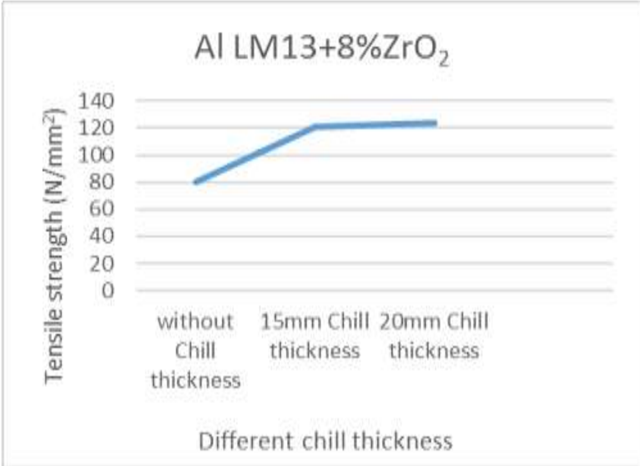


Chart 2.1.4: Effect of Cryogenic solidification on tensile strength

Figure indicates the effect of cryogenic solidification on tensile strength of the composites of Al LM13+8%ZrO_2. As the chill thickness increases, the tensile strength of the composite increases. The maximum tensile strength of 123.86N/mm² is obtained.

B) HARDNESS TEST

Hardness is a characteristic of a material, not a fundamental physical property. It is defined as the resistance to indentation, and it is determined by measuring the permanent depth of the indentation.
Hardness values of samples were measure using brinell hardness testing machine

Specifications: -

Parameter		**Description**
Ball diameter	-	5mm
Applied load	-	250 Kg

Table 2.1.6: Hardness test

Al LM13+2%ZrO_2	
Specimens	**Brinell Hardness number**
Without chill thickness	79.15
15mm Chill thickness	146.58
20mm Chill thickness	164.59

Chart 2.1.5: Effect of Cryogenic solidification on hardness

Figure shows the effect of cryogenic solidification on hardness of the composites of Al LM13+2%ZrO_2.As the chill thickness increases, the hardness of the composite increases. The maximum hardness number is 164.59 obtained from 20mm chill thickness.

Table 2.1.7 Hardness test results

Al LM13+4%ZrO_2	
specimens	**Brinell Hardness number**
Without chill thickness	93.57
15mm Chill thickness	198.01
20mm Chill thickness	209.47

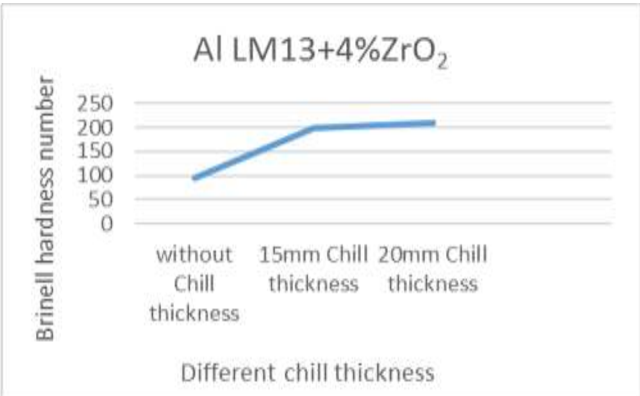


Chart 2.1.6: Effect of Cryogenic solidification on hardness

Figure shows the effect of cryogenic solidification on hardness of the composites of Al LM13+4%ZrO_2.As the chill thickness increases, the hardness of the composite increases. The maximum hardness number is 209.47 obtained from 20mm chill thickness.

Table 2.1.8 Hardness test results

Al LM13+6%ZrO_2	
Specimens	**Brinell Hardness number**
Without chill thickness	98.75
15mm Chill thickness	205.75
20mm Chill thickness	217.27

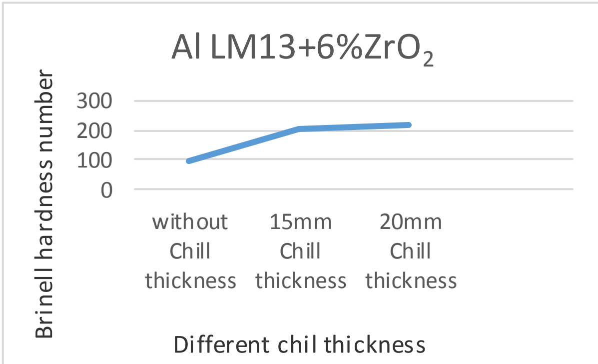


Figure 2.1.7: Effect of Cryogenic solidification on hardness

Figure shows the effect of cryogenic solidification on hardness of the composites of Al LM13+6%ZrO_2.As the chill thickness increases, the hardness of the composite increases. The maximum hardness number is 217.27

Table 2.1.9 Hardness test results

Al LM13+8%ZrO_2	
Specimens	**Brinell Hardness number**
Without chill thickness	101.24
15mm Chill thickness	246.88
20mm Chill thickness	274.47

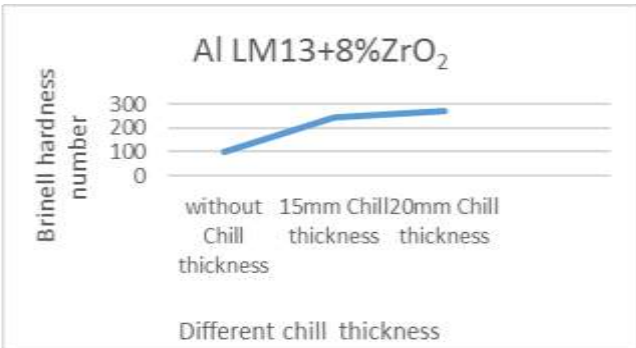


Chart 2.1.8: Effect of Cryogenic solidification on hardness
Figure shows the effect of cryogenic solidification on hardness of the composites of Al LM13+8%ZrO_2. As the chill thickness increases, the hardness of the composite increases. The maximum hardness number is 274.47 obtained from 20mm chill thickness.

C) COMPRESSION TEST

Compression testing is a very common testing method that is used to establish the compressive force or crush resistance of a material and the ability of the material to recover after a specified compressive force is applied and even held over a defined period of time. The compression test specimens are machined according to the design. The obtained values are tabulated to plot graph.

Table2.1.10: Compression test results

Al LM13+2%ZrO_2				
Specimen	**Ultimate load in KN**	**Deformation in mm**	**Ultimate stress in N/mm²**	**Ultimate strain**
Without chill thickness	79	2.6	0.38	0.13
15mm Chill thickness	87	2	0.46	0.10
20mm Chill thickness	91	1.95	0.48	0.097

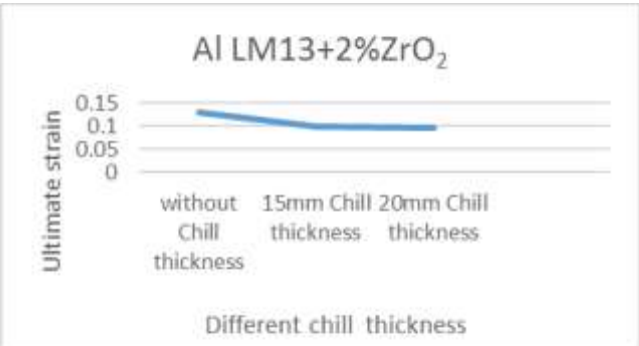


Chart 2.1.9: Effect of Cryogenic solidification on compression strength

As the chill thickness increases, the compression strength increases and deformation of the composite material decrease, it can withstand more load. The maximum withstand load is 91KN.

Table 2.1.11: Compression test results

Al LM13+4%ZrO_2				
Specimen	**Ultimate load in KN**	**Deformation in mm**	**Ultimate stress in N/mm²**	**Ultimate strain**
Without chill thickness	77	2.3	0.38	0.115
15mm Chill thickness	90	1.95	0.44	0.097
20mm Chill thickness	93	1.8	0.46	0.09

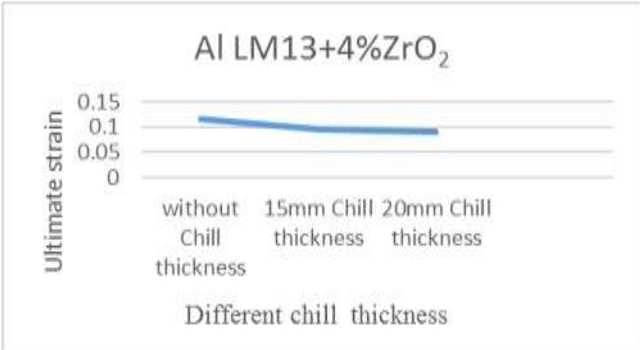


Chart 2.1.10: Effect of Cryogenic solidification on compression strength

Figure indicates the effect of cryogenic solidification on compression strength of the composites of Al LM13+4%ZrO_2. As the chill thickness increases, the compression strength increases and the deformation of the composite material decrease, and it can withstand more load. The maximum withstand load is 93KN.

Table 2.1.12: Compression test results

Al LM13+6%ZrO_2				
Specimen	**Ultimate load in KN**	**Deformation in mm**	**Ultimate stress in N/mm²**	**Ultimate strain**
Without chill thickness	81	2.3	0.40	0.115
15mm chill thickness	90	1.95	0.44	0.097
20mm chill thickness	95	1.8	0.47	0.09

Chart 2.1.11: Effect of Cryogenic solidification on compression strength

Figure indicates the effect of cryogenic solidification on compression strength of the composites of Al LM13+6%ZrO_2. As the chill thickness increases, the compression strength increases and the deformation of the composite material decrease, and it can withstand more load. The maximum withstand load is 95KN.

Table 2.1.13: Compression test results

Al LM13+8%ZrO_2				
Specimen	**Ultimate load in KN**	**Deformation in mm**	**Ultimate stress in N/mm²**	**Ultimate strain**
Without chill thickness	82	2.4	0.40	0.12
15mm chill thickness	91	1.95	0.45	0.097
20mm chill thickness	96	1.85	0.47	0.092

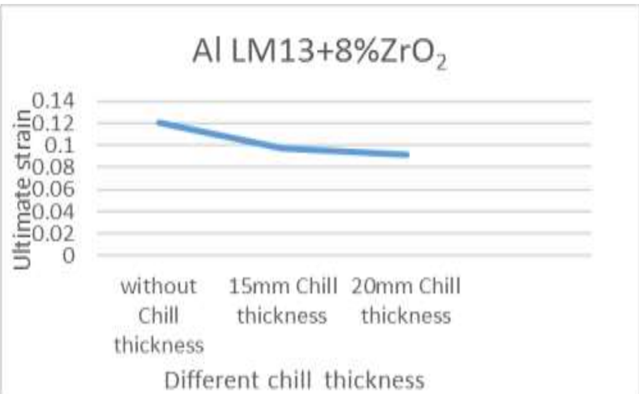


Chart 2.1.12: Effect of Cryogenic solidification on compression strength

Figure indicates the effect of cryogenic solidification on compression strength of the composites of Al LM13+8%ZrO_2. As the chill thickness increases, the compression strength increases and the deformation of the composite material decrease, and it can withstand more load. The maximum withstand load is 96KN.

D) EVALUATION OF MICROSTRUCTURE

To check the porosity, particle size and concentration of reinforcement microstructural analysis is carried out. And also SEM analysis was carried out for the samples. The following figure shows the SEM images of the samples of alluminium alloy LM13 and reinforced with different percentage of weight ratio.

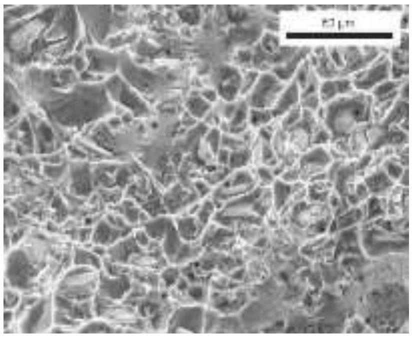
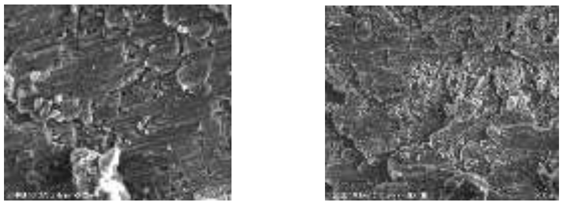
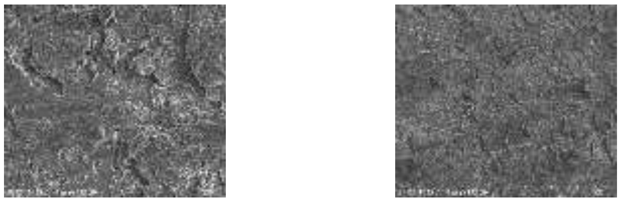


Figure 2.5: SEM Fractogrphic image of Al Metal Matrix Alloy (LM13).



(a) (b)

Figure 2.6: Al LM 13 Metal matrix reinforced with 2 weight percentage of ZrO2 without cryogenically solidified.

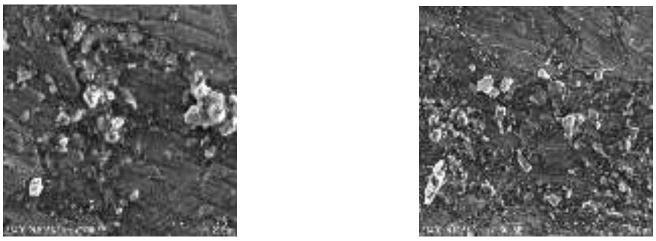


(c) (d)

Figure 2.7: Al Metal LM13 matrix reinforced with 2 weight percentage of ZrO2 with cryogenically solidified.

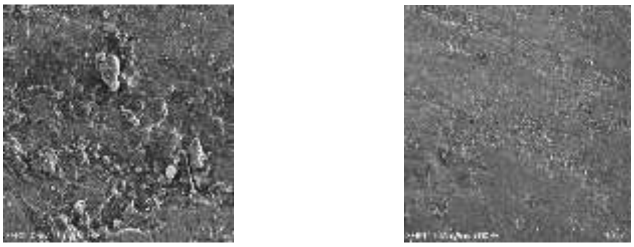
Figure 2.6: (a) SEM micrograph of aluminium alloy LM13 showing cells (b) Higher magnification micrograph showing cells and cell wall there is a more pores and coarse.

From figure 2.7: (c) SEM micrograph of the LM13 2wt% ZrO2 showing cell wall and (d) higher magnification micrograph of the cell wall showing distribution of ZrO2 particle in the Al alloy LM13.



(a) **(b)**

Figure 2.8: Al Metal LM13 matrix reinforced without 4 weight percentage of ZrO2 without cryogenically solidified.



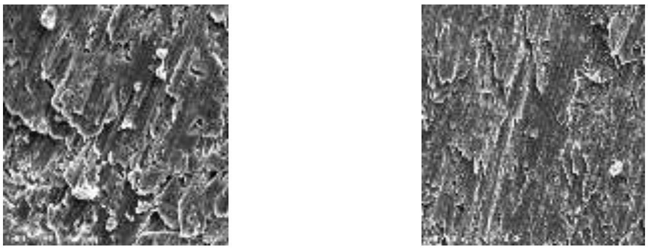
(c) **(d)**

Figure 2.9: Al Metal LM13 matrix reinforced with 4 weight percentage of ZrO2 with cryogenically solidified.

From figure 2.8: SEM analysis showed irregular shape of type A pores, Likewise, solidification shrinkage is not a likely source since the pores are in the proximity of the wall's thinnest section and copper chills. Likely explanation of this kind of porosity is gas entrainment process.

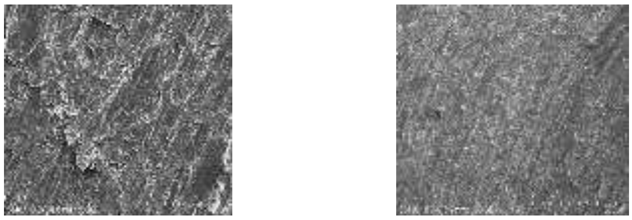
It explains both the irregular morphology of the pore's inner surface and the porosity location.

Where as in figure 2.9 which is cryogenically solidified composites, here we can absorb the uniform distribution of 4% ZrO2 and has less pores and coares.



(a) **(b)**

Figure 2.10: Al Metal LM13 matrix reinforced with 6 weight percentage of ZrO2 without cryogenically solidified.



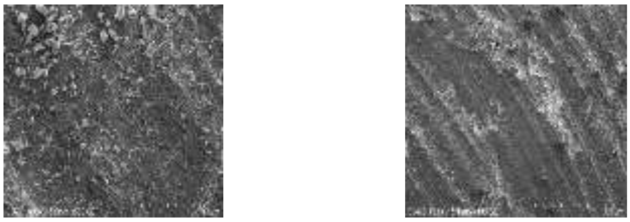
(c) **(d)**

Figure 2.11: Al Metal LM13 matrix reinforced with 6 weight percentage of ZrO2 with cryogenically solidified.

Figure 2.10: (a) we can observe irregular shapes and pores, (b) Higher magnification micrograph showing cells and less pores compared to (a). Figure 2.11: (c) here we can absorb the uniform distribution of 4% ZrO2 and has less pores and the ZrO2 uniformly distributed in (d).

(a) **(b)**

Figure 2.12: Al Metal LM13 matrix reinforced with 8 weight percentage of ZrO2 without cryogenically solidified.



(c) **(d)**

Figure 2.13: Al Metal LM13 matrix reinforced with 8 weight percentage of ZrO2 with cryogenically solidified.

Figure 2.12: The fracture mode is found to be of cleavage type due to excessive distribution of ZrO2 particulates. These particulates under stress are prone to fracture thus reducing the ductility of the composite and also decreasing in hardness and strength of the composite considerably. But in case of Figure 2.13 the distribution of zirconium oxide is uniform, under the stress are prone to withstand the stress, thus increase in ductility, hardness of the composites with increase in weight percent ratio with different chill thickness.

3. CONCLUSIONS

The metal matrix composites were successfully fabricated by sand casting and stir casting method and are cryogenically solidified by liquid nitrogen using different chill thickness (without chill, 15mm and 20mm). From the analysis the following conclusion can be drawn.

- The tensile strength of the composites increases by increase in weight fraction of zirconium oxide.
- The tensile strength of the composites increases with different chill thickness on cryogenically solidification.
- The hardness of the composites increases by increase in weight fraction of zirconium oxide.
- The hardness of the composites increases with different chill thickness on cryogenically solidification.
- The compression strength of the specimen increases with increase with increase in weight fraction of zirconium oxide and chill thickness.
- From the SEM analyses undoubtedly confirmed different causes of the porosity formation although the pores have similar dimensions. The shape and chemical composition of the pores inner surfaces indicates that pores are formed because of air entrainment during filling stage.

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