

Microstructure and Mechanical Properties of sintered Al 2024 boron fiber- Graphite hybrid MMCs.

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Abstract—Developing a new material for different application is becoming a biggest challenge in the field of material science. Researcher are mainly focusing on Aluminium alloy since it has very good strength to weight ratio and other mechanical properties. In the present study Aluminium 2024 alloy is used as a matrix to develop new composite material. To improve the properties of aluminium 2024 Boron fibre and Graphite-based particles are used as a reinforcement. Which helps in developing new hybrid metal matrix composite material. There are different techniques to develop Metal matrix composite (MMC). In this study Powder metallurgy (P/M) technique is used to develop Al 2024 boron fibre MMCs and Al 2024 boron fiber- graphite hybrid MMCs. Four different weight fractions of reinforcements are used with Al 2024 alloy. 4% and 8% wt.% Boron fiber and 1% of Graphite has been added during fabrication. Die has been designed and fabricated with D3 die steel. ANSYS analysis is carried out to ensure safe working of the die. Optical microscope and SEM analysis was carried out to ensure the uniform distribution of particles. XRD analysis is carried out to ensure the composition of the reinforcement particles. The compacting of powders has been done at 550 MPa and sintered in electric muffle furnace at 500°C for 90 minutes. Microstructure and SEM analysis is carried out to investigate the distribution of reinforcement on matrix. It has been found that the reinforcement is distributed uniformly in the matrix. Effects of content (wt. %) on the physical and mechanical properties of composites were determined by measuring the density, hardness and Compression strength values. Both theoretical and actual density has been calculated for MMCs. Also, Porosity has been determined and it has been found that as the reinforcement addition increases porosity also increases Microhardness carried out for the MMCs. It has been found that addition of Boron fibre as reinforcement Microhardness increased. Compression test was also carried out and it has been concluded the addition of boron fibre has an effect on the compressive strength but addition Graphite does not contribute much to the compressive strength. The MMCs with 8% wt. Boron fibres has highest compressive strength among the prepared MMCs

Keywords—Al 2024, Boron fiber powder, SEM, XRD, Powder metallurgy.

1. INTRODUCTION

Composite is a multiphase material that has a significant proportion of all properties of all phases such that better combinations of properties are realized. The phases must be chemically different and separated by a distinct interface. Most composites are created to improve combination of mechanical properties like stiffness, toughness, ambient and high temperature strength. Mechanical properties of a composite depend on the type and arrangement of reinforcement (fibres or particles) embedded in matrix of another material. In matrix-based structural composites, the matrix serves two paramount purposes viz., binding the reinforcement phases in place and deforming to distribute the stresses among the constituent reinforcement materials under an applied force. Solids that accommodate stress to incorporate other constituents by providing strong bonds for the reinforcing phase are potential matrix materials [1]. Al_2O_3 , $\text{Al}_2\text{O}_3+\text{SiO}_2$, B, C, SiC, Si_3N_4 , NbTi, Nb₃ reinforcements are used as continuous fibres and Al_2O_3 , SiC, SiO₂, vapor grown carbon fibres are used as short fibres and SiC, Al_2O_3 , TiC, B_4C , WC are used as particulates. The ultimate strength of the composite material is highly dependent on the reinforcement material and is the result of synergy between the matrix and the reinforcement. The matrix forces the load sharing by the reinforcement and thereby strengthens the composite [2] Aluminium is highly targeted by aerospace and automotive industry due to its advantages of low density, ability to get strengthened by precipitation, good corrosion resistance, high thermal and electrical conductivity and damping capacity. Aluminium matrix composites are being increasingly finding applications



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in automotive, electronic packaging, armours and sporting goods since 1920. Though it is usually reinforced with Al_2O_3 , SiC and C but other reinforcements like SiO_2 , B, BN, B_4C are also used. They are very attractive when compared to unreinforced alloys on account of its isotropic properties especially with discontinuous reinforcements and lower processing costs [3]. Aluminium metal composite with SiC and the effect various process parameters have been studied. It has been observed that the compacting pressure and reinforcement percentage have higher impact than the sintering temperature on micro hardness and density. And also observed that higher reinforcement and compacting pressure enhance the failure load while sintering temperature and time exert constrained influence. Experimentally proven that It is possible to achieve nearer to 95% theoretical density for 10% reinforcement with 550 MPa sintered at 500 °C for 1h.[4] effect of alumina particle size, sintering temperature and sintering time on the properties of Al– Al_2O_3 composite reveals that It has been concluded that as the particle size of alumina is reduced, the density is increased followed by a fall in density. In addition, at low particle size, the hardness and yield strength and compressive strength and elongation to fracture were higher, compared to coarse particles size of alumina. The highest hardness was 76 HB in specimens containing average particle size of 3 μm sintered at 600 °C for 45 min. Further increase in sintering time to 90 min results in a reduction in hardness to 59 HB.[5] Al based Al_2O_3 and SiC particle reinforced composite materials by P/M method. Tribological properties of these composite materials were investigated by wearing with 10 N load and 50 rpm on a pin-on-disc wear test rig at dry conditions. PM specimens were manufactured by pressing at 360 MPa pressure and by sintering at 600 °C for 0.5 h. Abrasive and adhesive wear tracks decreased for particle reinforced Al specimens due to particle addition and better wear resistance property Wear loss of particle reinforced Al specimens decreased about 1.5– 2 times. [6] in this present study Al 2024 powder is used as a matrix and boron fiber and graphite powder is used as a reinforcement, which is fabricated by powder metallurgy.

2. MATERIALS AND METHODOLOGY

Aluminium 2024 powder, Boron fiber powder and graphite powder has been procured from Leo chemicals, Bengaluru. Zinc stearate (Die wall lubricant) and Wax powder (Binder) has been procured from Sridevi chemicals, Bengaluru.

Chemical composition of Al 2024 powder as given in table 2.1

Table:2.1 Chemical Composition of Al 2024

Element	Cu	Mg	Fe	Mn	Si	Cr	Zn	Al
Content Wt %		1.8	0.5	0.25	0.5	0.25	0.2	Balance

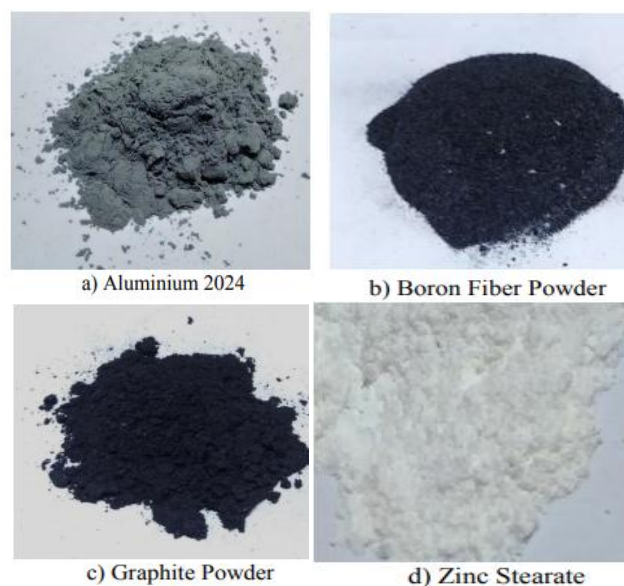
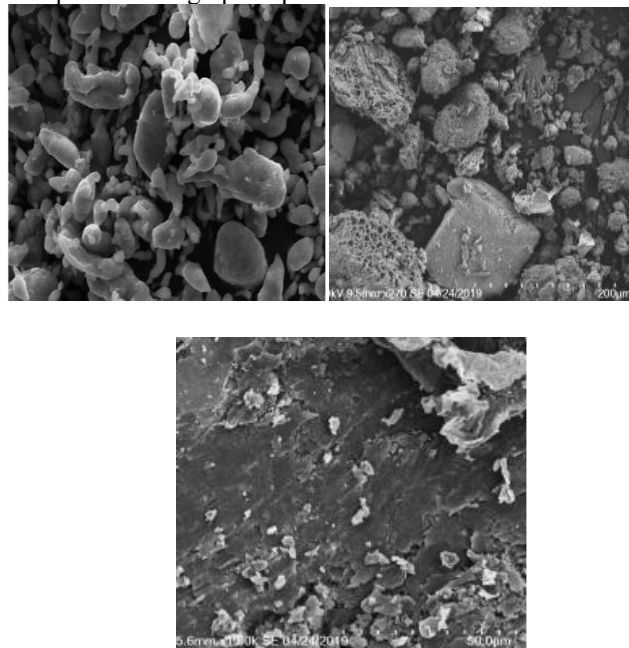


Figure 2.1.(a-d) Matrix and Reinforcements

Figure 2.1 shows Al 2024 powder which is in the range of 5 to 10 microns, boron fiber powder of grain size 5 microns and graphite mesh size of 2 to 5 microns which is used as matrix and reinforcement.

Scanning electron microscope was used to analyse the particles size. Figure 2.2 shows the SEM images of Al



3. FABRICATION OF POWDER COMPACTION DIE

Before going to fabricate the composite material, the die is needed for compaction process which is one of the major steps in powder metallurgy. Depending upon the work structure of the die, design has been carried out considering all the work-conditions. The following characteristics are taken for consideration in design situation,

- Chemical composition and mechanical properties of material selected
- Maximum working load
- Green compact size for the specimen dia 15 mm design of die shown in **Fig.3.1**

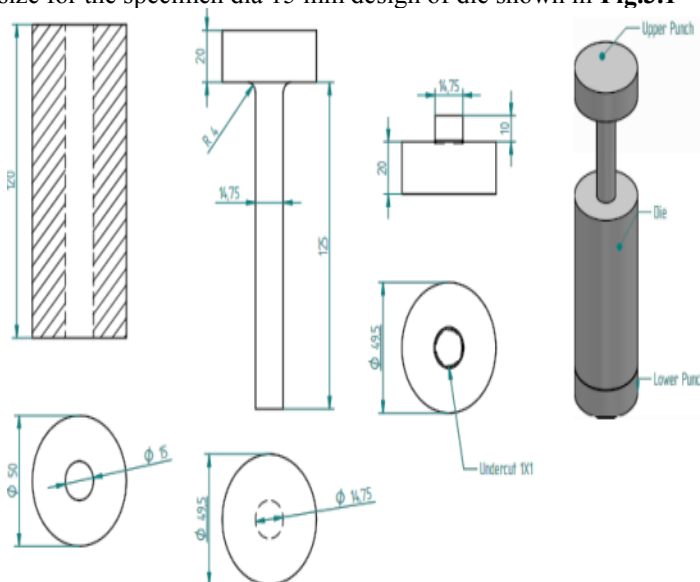


Figure.3.1. Design of Metal die

D3 steel raw material has been procured from SONI STEEL, Bengaluru. The following consideration are made while designing the die.

- ### 1. Working Load

1) Compaction Area= 176.71 mm^2) compaction pressure required to get 95% of theoretical Density=500 MPa. Working load= $176.71 \times 500 = 88.357 \text{ kN}$

2. Circumferential stress in the Thick Cylinder (Die) According to lame's equation at inner radius, Circumferential stress will be max. So,

$$(\sigma_{\theta})_{\max} = (r_o^2 + r_i^2) / (r_o^2 - r_i^2)$$

$$(\sigma_{\theta})_{\max} = 500 \times (252 + 7.5^2) / (252 - 7.5^2) = 599 \text{ MPa}$$

Since allowable stress in the material is 1500MPa Factor of Safety= 2.5, Hence design is safe 4.2.3 Ansys Analysis of Compaction Die and Punches.

The following Figs shows the stresses in the compaction die analysed in ANSYS workbench

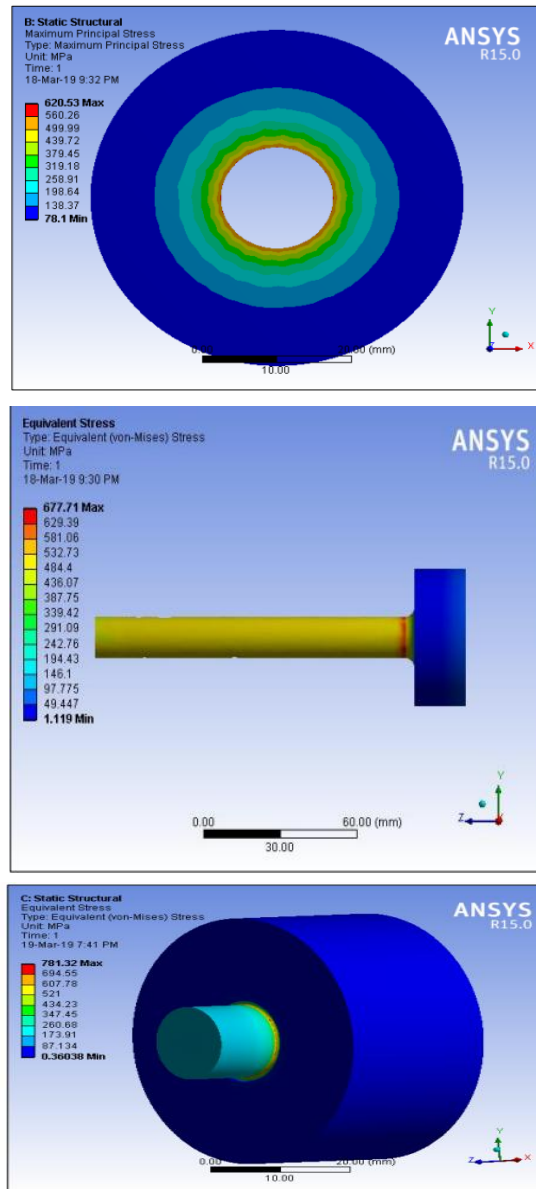


Figure.3.2 stresses in the compaction die analysed in ANSYS workbench.

Fillet and undercut is given to relieve stress concentration. The stresses found in the die is less than the allowable stress hence the die and punches are safe under the application of the working load.

3.2 Fabrication of Composite

As Reinforcement percentage is less, the powders are mixed manually and crushed in the ceramic crucible to get uniform distribution. Then the powder mixture was compacted in a uniaxial hydraulic press to form green compacts. It was a single compaction. The required amount of powder mixture was weighed on an electronic weighing balance and put in a D3 HCHC steel die. Die walls were lubricated with Zinc Stearate (C36H70O4Zn) to ease the compact ejection process. The Al particles were poured into a die 15 mm in diameter. Care was taken

to ensure that the powder was distributed evenly within the die cavity. Hand tapping was induced before compressing to achieve a better tapping density. To achieve high green densities of powders, high compaction stresses are required. In this process, the powder was compacted under the axial stress of 500-550 MPa as per the literature survey. It is very essential to carefully remove the compact from the die to avoid damage. So, an ejector tool is used to facilitate the easy removal of the specimen.

3.3 Sintering

Sintering of powder sequentially involves the establishment and growth of bonds between the particles of powder at their areas of contact and migration of the grain boundaries formed at the bonds. The coated compacts were sintered in an electric muffle furnace at a closely regulated temperature of 500°C for 90 minutes and allowed to get cooled to room temperature in the furnace itself.

4. EXPERIMENTAL PROCEDURE

4.1 Density and porosity

The density of the composite specimens was determined using a high precision digital electronic weighing balance with an accuracy of 0.0001 g. Moreover, another method also employed to measure density. It was measured by measuring the dimensions of the sample using a micrometre and weighing its mass using a balance. Five vertical measurements were made on each sample. Mass was measured on a balance with 4 decimal point accuracy. Procedure 1. Measure the mass of specimen in air and record as M air gms. 2. Then submerge the specimen completely into water and measure the mass and record this value as M water gms. So, it has displaced M air - M water gms of water. Since water has a density of 1 gm/cm³, this implies volume of object = V = M air - M water. 4. Then we can use the equation below to determine the porosity of specimen

$$\text{Porosity} = \frac{(\text{Theoretical density} - \text{Experimental density})}{\text{Theoretical density}} \times 100$$

4.2 Metallographic Analysis

Metallographic analysis offers a powerful quality control as well as an important investigative tool for microstructural study. Examination of the composites microstructures was carried out using SEM analysis to determine the primary characteristics of the specimens. The detailed features of the microstructure were characterized at high magnification using a scanning electron microscope (SEM) with a maximum resolution of 7 nm in a backscattered model and maximum useful magnification of 3,00,000X.

The sample preparation procedure includes sectioning, mounting, grinding, polishing, and etching. To avoid cold working and altering the microstructure, metallographic specimens of sintered preforms were cut with a low speed saw and cooled by a liquid coolant during cutting. Then the preforms were prepared using standard hand polishing using 240, 600, 800 and 1000-grit silicon carbide papers. Specimens were then finish-polished using 1 µm diamond paste suspended in distilled water to obtain mirrorlike surface finish. To expose the microstructural features, the polished specimens were etched with Keller etching agent solution for 10 to 50 seconds. The etching process allows the aluminium matrix to dissolve while leaving the silicon particles elevated above the matrix level. After they were cleaned with water and dried with acetones. The etch-polish-etch procedures were used to attain good microstructure.

Fig. 4.1 show the scanning electron microscope (HITACHI Model SU-3500N).

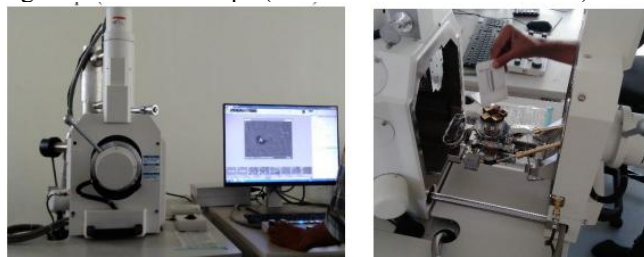


Figure. 4.1 SEM Setup

4.3.XRD ANALYSIS X-RAY DIFFRACTION (XRD ANALYSIS OR XRPD ANALYSIS)

It is a unique method in determination of crystallinity of a compound. XRD is primarily used for identification of crystalline material and identification of different polymorphic forms (“fingerprints”). For XRD analysis the samples are collected in the form of powder while machining.



Figure.4.2 XRD Setup

4.4 Micro hardness and Compression Strength.

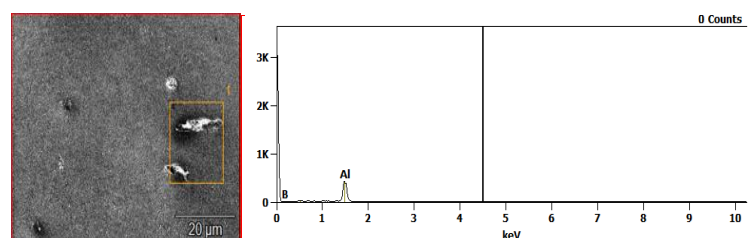
Same specimens used for microstructure analysis are used for hardness study. 500-gram load is applied on the particle for a duration of 15 seconds.

Three readings are taken on each samples and average was taken to find the hardness of the MMCs. Similarly, specimens are machined as per ASTM E9 standard to analyse the compression strength. The specimen of dia 15mm and length of 1 inch is used. deformation such as forging and rolling.

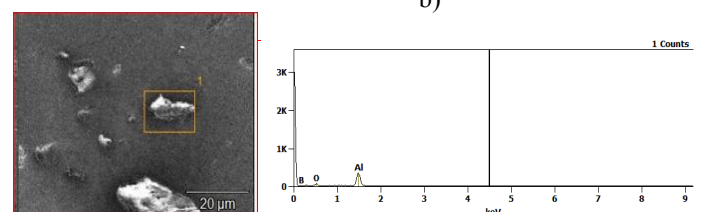
5.RESULTS & DISCUSSION

5.1 Microstructure Analysis using SEM and EDX

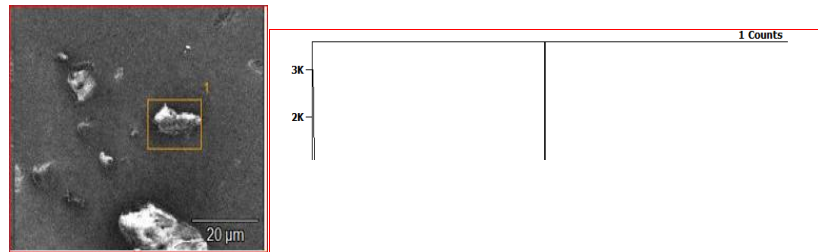
Metallographic analysis offers a powerful quality control as well as an important investigative tool. figure 5.1 shows the SEM images with EDX analysis of Al 2024-b MMCs and Al 2024-B-Gr hybrid MMCs. the analysis of reinforced particles in Aluminium matrix. From the SEM images its observed that, reinforcement particles are distributed uniformly and no clustering or agglomeration of particles were found in the SEM images. This is due to proper blending of powder and reinforcement. Size of the particles are also determined with the help of SEM images. EDX analysis also carried out on the casted sample to confirm the present of reinforcement with the percentage. Figure 5.1 shows the EDX graph, which shows the percentage of Al 2024, boron fibre and graphite. The peak height represents the percentage of reinforcement presents in the composite. As the percentage of reinforcements increases the height of the peak also increased.



a) SEM with EDX of Al 2024 96% and B-4%



b) SEM with EDX of Al 2024 92% and B-8%



c) SEM with EDX of Al 2024 95% and B-4%-Gr-1%

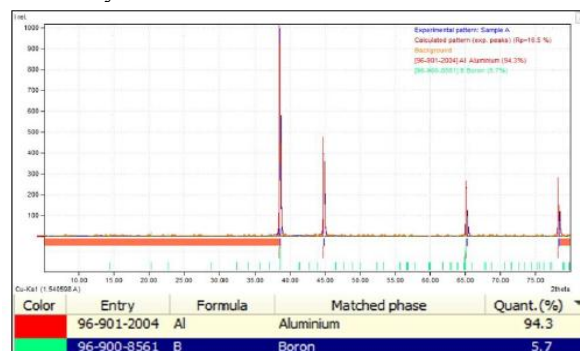
Figure. 5.1 SEM with EDAX of Al 94% BF 6%, 92% & 8%, Al 95% 5% B-1% Gr

5.2 X-ray Diffraction Analysis

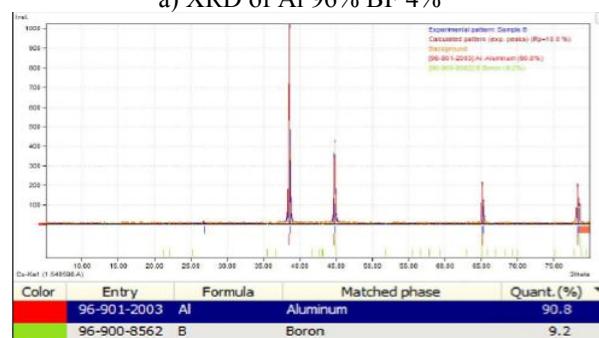
The X-ray diffraction results for the prepared composites are shown in Fig. 5.2. The results indicate the presence of aluminium (in the largest peaks), and the presence of boron fiber particles and carbon, is indicated by minor peaks. A clearly visible carbon peak can be observed in the hybrid composites. The increase in the intensity of the boron peaks with the increasing boron fibre content of the composite is also evident. Fig. 5.3 also shows that there is no oxygen reaction in the samples during the sintering process. The XRD pattern confirmed the presence of Aluminium, boron and graphite particles in the hybrid composite.

The fig. 5.2 a-d show the XRD pattern of composite powders using MATCH 3 software with material ratios

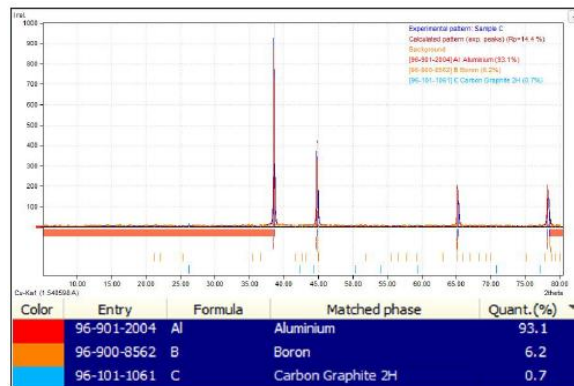
- X axis represents 2θ
- Y axis represents Relative intensity
- The wavelength used for XRD analysis is 1.5405 Å



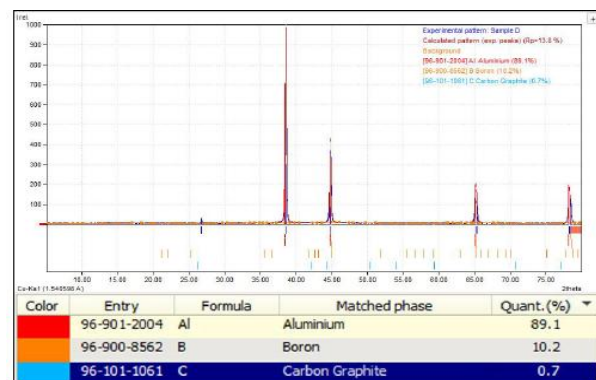
a) XRD of Al 96% BF 4%



b) XRD of Al 92% BF 8%



c) XRD of Al 95%- BF 4%-Gr-1%



d) XRD of Al 91%- BF 8%-Gr-1%

Figure.5.2 (a to d) XRD analysis of Cast Samples

5.3 Density and Porosity Measurement

After the successful casting, the density of the composite is found using Archimedes principle and compare with the theoretical density, which is calculated using rule of mixture. Figure 5.3 shows the theoretical density of different composition MMCs.

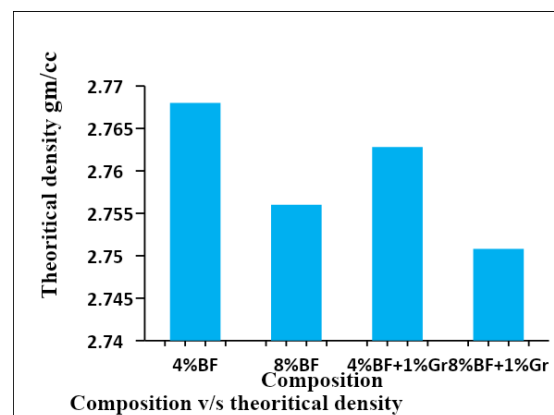


Figure 5.3. Variation of Theoretical density of Al 2024 Boron MMCs and Al 2024-Boron -Gr hybrid MMCs with different weight percentage.

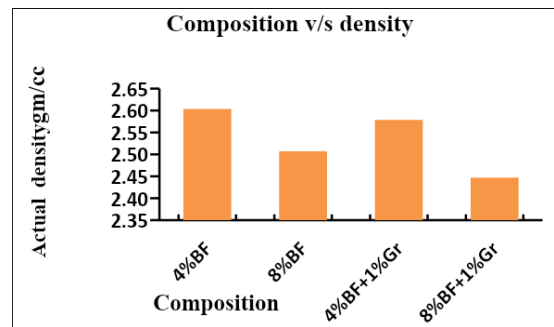


Figure. 5.4 Variation of Experimental density of Al 2024 Boron MMCs and Al 2024-Boron -Gr hybrid MMCs with different weight percentage.

The addition of low-density boron fibre (2.48 gm/cc) and graphite (2.26 gm/cc) which are less dense than the matrix Aluminium 2024 (2.78 gm/cc) are found to be responsible for the decrease in the density of the composite as the reinforcement percentage increased. The composite becomes more brittle in nature as the percentage of reinforcement increased.

5.4 Porosity measurement

Porosity or void fraction is a measure of the void spaces in a material, and is a fraction of the volume of voids over the total volume, as a percentage between 0% and 100%.

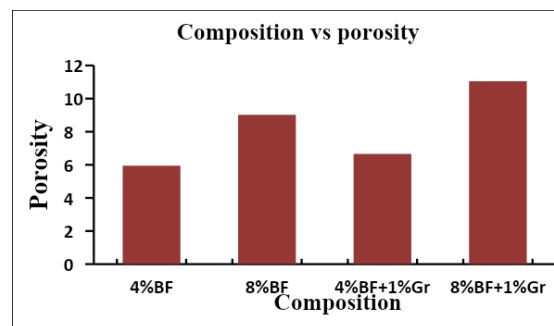


Figure:5.5. Variation of Porosity of Al 2024 Boron MMCs and Al 2024-Boron -Gr hybrid MMCs with different weight percentage

As the reinforcement increases the porosity also increase due to agglomeration of reinforcement and also due to improper bonding of reinforcement with the matrix. The addition of boron fibres increased the porosity majorly by and graphite addition is affected minorly.

5.5 Microhardness

Fig. 5.6 shows the variation of hardness of the Al composite with increased Boron fibre and Graphite content. It can be understood from the Fig. 5.6 that the hardness of the composites was improved with the increase in weight percent of Boron fibre and Graphite reinforcements. The increase in hardness of the hybrid composite is owing to the following reasons (i) High hardness of Boron reinforcement particles. (ii) Uniform distribution of reinforcement in the composites. (iii) Increase in the carbon content due to graphite addition.

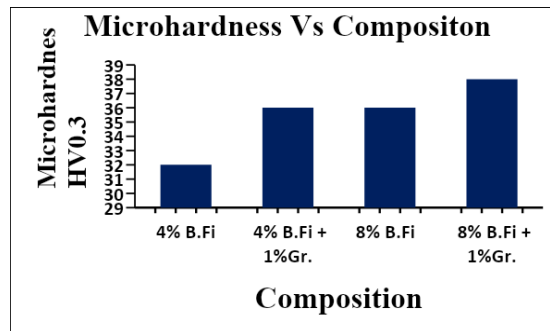


Figure: 5.6 Variation of Microhardness of Al 2024 Boron MMCs and Al 2024-Boron -Gr hybrid MMCs with different weight percentage

5.6 Compression strength

The response of composite (strength) to compression is basically dictated by the percentage of reinforcement and the interface strength. Lin et al. has carried out a theoretical modelling and analysis of plastic deformation of particle reinforced aluminium

composites. It was stated that the plastic deformation of such composites depends on the interfacial strength of the composite. Also, the boundary slip or grain deformation basically depends on the bonding strength.

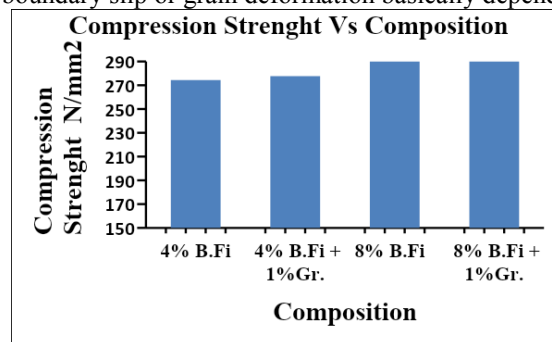


Figure.5.7 Variation of Compressive strength of Al 2024 Boron MMCs and Al 2024-Boron -Gr hybrid MMCs with different weight percentage

Aluminium composite specimens of 15 mm diameter and 25 mm long were prepared as per ASTM standard and tested under direct compression. The ultimate failure load and load Vs deflection were observed and evaluated.

It is observed that as the percentage of reinforcement is increased the compressive strength comes down on account of reduction in ductility due the high hardness of boron fibre and carbon content in the graphite. Load-deformation curves reflects the response of matrix material to compression load. Load Vs deformation curves exhibit that the deformation under compression is elastic plastic deformation and do not exhibit brittle material behaviour.

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6. CONCLUSION

- Aluminium -Boron Fibre, Aluminium-Boron Fibre-Graphite composites in the form of powdeDers have been successfully developed by powder metallurgy (PM) technique.
- The microstructure analysis of the composites indicates that both Boron Fibre and Graphite particles disperse uniformly in the matrix alloy without much agglomeration, and orientate randomly in the matrix.
- Addition Boron Fiber increases hardness and compression strength of the composite. 8% Boron Fibre and 8% Boron Fibres -1% Graphite gives better results.

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