VANADIUM CARBIDE REINFORCED WITH ALUMINIUM METAL MATRIX THROUGH POWDER METALLURGY ROUTE

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CERTIFICATE

I hereby certify that the work being presented in the dissertation entitled "VANADIUM CARBIDE REINFORCED WITH ALUMINIUM METAL MATRIX THROUGH POWDER METALLURGY ROUTE" in partial fulfilment of the requirement of the award of the Degree of master of technology and submitted to the Department of Mechanical Engineering of Lovely Professional University, Phagwara, is an authentic record of my own work carried out under the supervision of (Name Of Supervisor, Designation) Department of Mechanical Engineering, Lovely Professional University. The matter embodied in this dissertation has not been submitted in part or full to any other University or Institute for the award of any degree.

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ABSTRACT

Aerospace material aluminium (2024) used to prepare composite with Vanadium Carbide(VC). In a muffle furnace with a temperature of 600°C sintering process is carried out properly without any inert gas atmosphere. With different percentage of Vanadium Carbide(VC) with aluminium(Al) like 2 wt% VC, 4 wt% VC, 6 wt% VC, 8 wt% VC, 10 wt% VC, 12 wt% VC, 14 wt% VC, 16 wt% VC in which it enhance the mechanical property of the sample .Hardness of sample increase with increasing percentage of amount reinforcement along with in a similar manner density of sample increases. From XRD of sample we get that absence of intermetallic bond between Aluminium (A) and Vanadium Carbide (VC) we got defect free microstructure.

Keywords:

Metal-matrix composites (MMC), Particle-reinforced composites, Powder processing, Sintering.

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CHAPTER 1

1.1 INTRODUCTION

Always composite material is very much useful for everyday life .Aluminium metal matrix (AMCs) have numerous applications in various field like aerospace, military and automotive industries. Beyond that Aluminium is the most abundant element in the Earth, it comes in third number after silicon and oxygen. AMCs mostly used because of its high toughness, strength and modulus, low coefficient of thermal expansion and good wear resistance. Also mechanical property depends on morphology, size, volume fraction and distribution of reinforcement along with chemical compatibility which has been discussed by numerous researchers. Powder metallurgy route is solid route and it is most effective route than other liquid route because in powder metallurgy route uniform distribution of reinforcement which has been influences the final properties. This tends to improvement on the strength of boundary of particle-matrix.

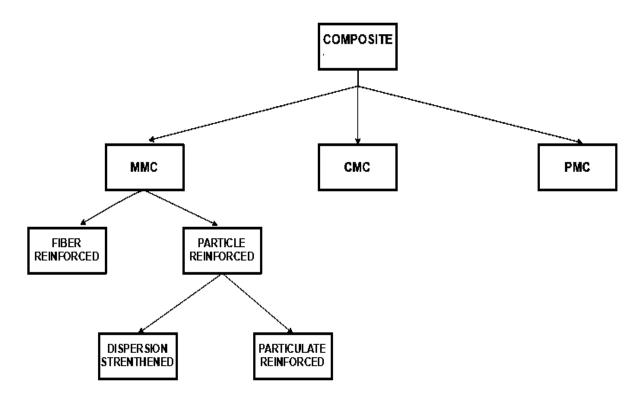


Figure 1. Types of composite

Various composite material types leads to various composite fabrication like Metal Matrix Composite (MMC), Ceramic Matrix Composite (CMC) and Polymer Matrix Composite (PMC). In which we choose MMC as aluminium is our base metal because in MMC minimum we required an metal along with other constituent. With base matrix MMC we require reinforcement so one is fibre so the composite called fibre reinforced composite and other particle reinforced composite. Next we choose particle reinforced composite, In particle reinforced composite particle may be different shape and size like cylindrical, spherical, angular etc. Particle reinforced composite subdivide in to dispersion strengthened and particulate reinforced. As our reinforcement is VC is it is particulate reinforced composite. Because dispersed strengthened particle size is less than 0.25 µm.

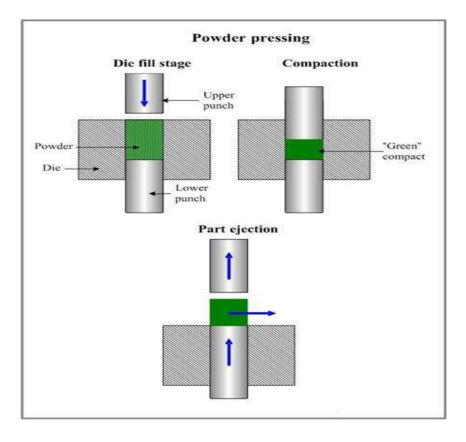


Figure 2. Powder pressing

In powder metallurgy technique we have series of process like compacting, sintering. Compacting leads to how sintering to be done like we know that there are two types of sintering solid state sintering and liquid state sintering, solid state diffusion and bonding of particle takes place in solid state sintering. In liquid state sintering we have to add a liquid forming powder that is called binder and heating up to that temperature where it forms liquid, so it spreads and helpful to bonding of particle and densification. Compacting can be done various machine like Universal testing machine (UTM) and compression testing machine. The compression testing machine is used for apply the load to a specimen up to its limit. The compression testing machine apply load up to 1000 KN.

In compaction process lubricant is necessary to removal of material after compaction. Numerous types of lubricant are used in compacting like wax, carbon, copper, nickel and zinc sterate or otherwise we called it zinc soap. Excess lubrication also leads to burning of sample in sintering process. Compacting is very critical operation to give final shape and also various level of uniformity also determined. So various factor that we take in to consideration while compacting i.e., proper load, rate of load along with dwell time. Along all these factor we have to notice about design of die which can bear the desired amount of load. Ununiformed transmitted pressure leads to distortion of specimen. In sintering particles welds with each other and finish part produced which is very much strong. After sintering post sintering also required because changes in dimension in die so post sintering is helpful to better dimensional accuracy in some cases repressing also required and it tends to increases the density of material. As density increases its leads to strength and mechanical properties increases. If we need further improves in mechanical properties we will goes through re-sintering.

Aluminium 2024 generally have excellent machining characteristic and at the machining time at high speed it produces fine quality of chips and the basic percentage of cupper present in 2011 aluminium i.e.(5-6)%. Along with bismuth(0.2-0.6)%, iron(0-0.7)%,lead(0.2-0.4)%, silicon(0-0.4)% and zinc(0-0.3)% respectively. Also aluminium 2011 grade used to manufacture of atomizer, hose part, screw machine products, tube fittings, pipe stems, automotive trim, ordnance.[4]beyond that aluminium 2011 have poor corrosion properties so less researcher take it for their research purpose. To eradicate the corrosion we have taken vanadium carbide used as reinforcement. Absorbers, transparent and opaque are three material categories for microwave interaction.

Vanadium carbide (VC) is our ceramic reinforcement used because it improves the corrosion resistance of base metal matrix. In aluminium 2024 when we added vanadium carbide it reduces the corrosion behaviour of base metal matrix. Along with VC other mechanical properties which enhance the properties of base metal matrix like higher bending strength, higher micro hardness, wear resistance, erosion resistance and tensile strength. And it also decreases the difference between thermal expansion coefficient of base matrix and reinforcement.VC has many applications such as high temperature structural materials.

Wear resistance, hardness and strength improve by ceramic reinforcement and improvement depends on uniformity and amount of reinforcement distribution along with strength of matrix-particle boundary. Mechanical properties of reinforcement also considered for reinforcement use. Most of researcher find out the reaction between matrix and reinforcement which leads to destruction of mechanical properties which the composite obtained on the other hand others get the better result because of interfacial reaction between matrix and reinforcement.

Carbides of transition metal such as TiC, HfC, TaC and VC are important commercially because of its high wear resistance and hardness. Mostly Binary carbide metals are used because of its high micro hardness. Due to formation of intermetallic compounds (Al-Ta, Al-Nb, Al-Mo and Al-V) reduce the gap between thermal coefficient of expansion of matrix and reinforcement. Al3V, Al3Ti, Al3Nb are transition metal aluminides always shows high performance due to high mechanical properties. Al3V is the resultant of vanadium Carbide and Aluminium it leads to FCC crystal structure is higher than the aluminium and vanadium carbide. [1]

Conventional sintering carried out in muffle furnace. Different temperature up to below its melting point sintering should be done. It is an electric furnace which is used for heating of material by avoiding inclusion of gas fly ash and combustion product. Front loading box type of oven is used for sintering of the product.

In muffle furnace the sintering of the samples are carried out then further used for different testing like micro hardness, density, optical microstructure, SEM, XRD. In micro hardness the samples hardness measured at microscopic level. By proper indentation leads to we get

the better result. For optical microstructure we need to mirror finish the sample to get better result. SEM gives us result on surface morphology or any intermetallic compound formation on the composite. While analyse XRD peaks gives desired result of amount of reinforcement in that MMC.

1.2 TERMINOLOGY

MMC-It is a type of composite material it is made of minimum two constituent and at least one is a metal.

Particle reinforced composite-It is a secondary material which is reinforced in primary base matrix which has different shape and size.

Particulate reinforced composite-It is a type of particle whose size is less than 0.25 micron which reinforced in the base matrix.

1.3 RATIONALE AND SCOPE OF THE STUDY

- 1. In this research work we justify about why aluminium 2024 grade is an aerospace metal which has various application in automotive industry.
- 2. Vanadium Carbide less used by researcher because of its high cost but its improve the mechanical properties of base matrix.
- 3. Micro hardness and density of sample increases adding VC with aluminium metal matrix.

1.4 OBJECTIVES OF STUDY

Our main research is studying various properties that enhance by reinforcing agent.

- 1. The overall objective is to improve mechanical properties of aluminium 2024 grade by enhancing its hardness and density.
- 2. Mechanical property enhance by various reinforcement.
- 3. To study the effect of high pressure and temperature in compacting and sintering process.
- 4. To study different sintering process in detail along with different parameters that affects in fabrication.

CHAPTER 2

2.1 REVIEW OF LITRETURE

The research work done earlier on composite material fabricated by powder metallurgy are given below-

Ehsan Ghasali et al. (2016) used Vanadium Carbide (VC) used as reinforcement because of its extreme hardness, high wear resistance, high temperature strength along with high corrosion resistance. Moreover VC is better absorber of radiation comes from microwave in microwave sintering. A rectangular bar shape specimen is prepared by after 250 MPa of uniaxial pressing except in spark plasma sintering. Because in spark plasma sintering (SPS) after mixing directly used it, there is no pre-processing requirement for the powder.

Three types of sintering basically used here i.e. conventional, microwave and spark plasma sintering. Author discus about three factor i.e. density, bending strength and micro hardness. After further testing and inspection author found density is more of in spark plasma sintering then corresponding to microwave sintering and we got low density in conventional sintering. Similarly bending strength and micro hardness of material in spark plasma sintering also more than particle sintered in microwave sintering, and least in conventional sintering. Identification of phase carried out in XRD. With the help of Archimedes' Principle bulk density find out after sintering.

Aluminium 1056 grade with mesh size 250 reinforced with VC particle size less than 200nm. And formation of intermetallic phase Al3V with Al4C3, AlV2. Time and temperature required in microwave sintering and conventional sintering is more than spark plasma sintering. Low particle size tends to proper agglomeration. In overall some defects occur in SPS i.e. porosity due to weak mechanical properties. [1]

M. Lieblich et al.(2013) discussed about wear behaviour of dual particle size of reinforcement in aluminium 6061 graded metal matrix. Two size of particle is used for their inspection of wear behaviour one is particle size less than 3 microns and other is 10-45 microns. There are three types of mixing method, wet blending, rotatory cube and planetary ball milling. It clarify that wear resistance increases with decrease in particle size i.e. 3 microns MoSi2 reinforced have better wear resistance.

Wear resistance depends upon two factor i.e. intrinsic factor and extrinsic factor. In intrinsic factor size, shape, volumes, distribution, hardness, friction. In extrinsic factor type of test includes applied load, sliding speed.

Reinforcement Mosi2 have high thermal stability corresponding to tribolayer or we can say mechanically mixed layer (MML) .Three types of layer author discuss i.e. Tribolayer, plastically deformed layer and non-affected region due to surface modification loss of volume is less in sliding wear, as a result in friction coefficient increases. Moreover author specify that tribolayer protect the practically deformed region.[2]

Z.Y. Liu et al. (2010) Investigated in powder metallurgy process mixing is properly done through high ball milling to charge ratio (BCR) it leads to improvement of homogeneity due to proper distribution of SiCp. Which enhance the tensile strengths of composite material. Size ratio of aluminium to SiCp (PSR). Uniformity of SiCp leads to improvement in extrusion. Critical volume fraction analyze quantitatively. Model shows that improving homogeneity by small PSR and large deformation ratio of aluminium matrix, by decreasing PSR and increasing BCR extrusion occur. [3]

Katsuyoshi Kondoh et al. (2008) examined about erosion resistance of AlN dispersoids along with cavity formation in aluminium, conventional process and in situ synthesis process are method of preparation along with a equipment called magnetostrictive-vibration through which evaluation is done. Erosion resistance improves by in situ synthesized due to AlN bonded very well with aluminium metal matrix. AlN cause poor resistance and easily removed from the work piece because less coherence with aluminium metal matrix. Porosity leads to cavitation in the composite after sintering. Wear behaviour also increase the porosity occur due to high pressure. [4]

J. Corrochano et al. (2007) analysed that 10 percentage by weight of alumina used in aluminium metal matrix fabricated by powder metallurgy. Air tight furnace with cold wall along with alumina linings a single crystal alumina whisker (Al2O3w) are produced in prepilot scale by vapour-liquid-solid, c-axis alpha alumina fibres of single crystals are produced. In high and room temperature tensile properties investigated. Then author conclude that Al2O3 have impressive mechanical properties in room temperature also single crystal attained strong bonding with base metal matrix.[5]

Linlin Yuan et al. (2016) investigated about hot rolling and solution treatment in AlB2 reinforced aluminium metal matrix fabricated by powder metallurgy.(7-12)% by weight boron content in aluminium metal matrix. Upon heat treatment and hot rolling mechanical properties and micro structure changes in composite which is investigated by scanning electron microscope(SEM) and in tensile test. Scratch morphology, wear behaviour and friction coefficient which is investigated by scratch test in pure aluminium and aluminium composite. As a result author concluded that wear and hardness properties higher in reinforced composite than unreinforced composites. [6]

Chuandong Wu et al. (2016) evaluated their study for mechanical behaviour and microstructure of aluminium (7075) metal matrix by adding reinforcement of different particle size. Three particle size are taken in to consideration of B4C particles i.e. (2µm, 4.2µm and 56.9µm). Agglomeration of fine B4C reinforcement occur better than that coarse reinforcement particle with constant volume fraction, along with composite with low particle size high fracture strength and yield strength. Quantitatively fine B4C particles have high value of strain gradient strengthening which leads decreases the dislocation between particles. Different particle leads to different fracture mechanism in the composites.[7]

Cunguang Chen et al. (2015) presented a review hexagonal boron nitrite reinforced with pure aluminium metal matrix fully densified through powder metallurgy route. Structural characteristics and powder morphologies are verified through Scanning electron microscope (SEM), X-ray diffraction (XRD). Liquid phase sintering tends to high density of the composite checked by Brinell hardness and corresponding to compressive behaviour also measured. Different grain characteristics shown by different granularity as a result of this recrystallization of original grains (35µm) and interface is very sharp. Fracture strain and compressive strength both increase with decrease in aluminium particle size. Author concluded that Al particle size 2µm exhibited high density, compressive strength, hardness as compare to 35µm Al powder. [8]

X.Z. Kai et al. (2013) represented that stress concentration and porosity tends reinforcement agglomerations, deformation is always takes in to consideration when particulate is reinforced in metal matrix.32% by weight of B4C mixed with Aluminium

metal matrix uniformly fabricated by flake powder metallurgy. More space required to B4C with aluminium flake powder as compare to spherical aluminium powder due to higher volume of flake powder as compared to aluminium powder. That's the reason author choose fake powder metallurgy rather than conventional powder metallurgy its leads to uniform distribution of B4C particle and consolidate the composite. Al2O3 nano skin is also present in aluminium powder so in conventional powder metallurgy it tends to fractured along with dispersed in to the matrix during consolidation and it tends to higher strain hardening rate in the time of deformation. At last author concluded that flake powder metallurgy of aluminium composite exhibits higher tensile strength, more stronger and ductile than conventional powder metallurgy process. [9]

P. Ashwath et al. (2014) investigated that that SiC, graphene and Al2O3 common reinforcement used in aluminium metal matrix. Reinforcement percentage and effect of ball milling effect the mechanical properties of aluminium metal matrix composite the particle of SiC and Al2O3 is 10 microns and grapheme is 10 nano microns. Mostly 550 °C is the sintering temperature with finished in 10-15 minutes. 650 MPa pressure required to better compacting to achieve high density. Incorrect heating rate and improper amount of reinforcement leads to crack formation in our green compact. To overcome this defect author directed to use reinforcement 10% by wt. or less than that. [10]

Anil Kumar Bodukuri et al. (2016) explain that high strength to weight ratio reason behind that SiC and B4C used here reinforcement in aluminium metal matrix fabricated by powder metallurgy. After proper ball milling sintering is to be done at required temperature. Three types of combination of reinforcement to matrix material is to be taken by author. Micro hardness increase by increase the percentage of B4C content. [11]

Ehsan Ghasali et al. (2016) studied that 15% by weight SiC and 7% by weight TiC used as reinforcement in Pure aluminium matrix and 1056 alloy aluminium matrix fabricated by conventional and microwave sintering methods.750 and 650°C sintering temperature required for both micro wave and conventional sintering in a graphite bed up to 1 hour, resulting highest micro hardness, bending strength and relative densities of 1056 aluminium in microwave sintering at 750 °C. Crystalline phase have inspection through XRD and

uniform distribution of particle shown by SEM in both microwave and conventional sintering. [12]

M. Sobhani et al. (2016) investigated that behaviour of resistance in growth in crack of micro and nano aluminium composite in which 20% by weight of TiO2 is present. Composite prepared by alumina and titania powder through sintering. A secondary phase Al2TiO5 formed by using micro and nano sized TiO2 powders. Identification-fracture method use to determine toughness curve. Compare to monolithic alumina nano and micro composite have high toughness. Micro cracking and bridging were two toughening mechanism of aluminium composite. [13]

Heguo Zhu et al. (2011) explain about wear behaviour in high temperature of aluminium metal matrix along with microstructural evaluation reinforcing ZrO2-B due to formation of Al-ZrO2-B. With decrease in Al3Zr in composite it leads to increase in B/ZrO2 mole ratio.Al3Zr diminished when mole ratio of B/ZrO2 reached at 2 and the resultant outcomes consisting ZrB2 and alpha Al2O3particles. At 20N load and 373K temperature the wear behaviour increases and reach to maximum value until the sliding velocity decreases. At 473 K the wear behaviour decrease further increases constantly the sliding velocity. When Molar ratio of B/ZrO2 increases it leads to friction coefficient and wear rate decreases and in other hand deformation increases. Wear in high temperature is a limiting factor in aluminium matrix composite. [14]

Lin He et al. (2009) investigated that Vanadium carbide using as reinforcement in aluminium matrix in situ by using commercial amorphous carbon and ferrovanadium. Spark plasma sintering is used to sinter Vanadium and carbon at 650- 700°C and solid phase diffusion between V and C corresponding to carbonization reaction occur very easily. Firstly increases of density and then with increasing temperature decreases, at 1050°C maximum density obtained for 5 min. Up to 900 °C hardness increase continuously then increases slowly and corresponding maximum hardness obtained at 1150 °C. Vanadium carbide contain V8C7 are (1-3) micro meter along with iron particles are homogeneously mixed and distributed then corresponding to 1050 °C sintering is done. [15]

Ehsan Ghasali et al. (2014) explained about aluminium reinforced with B4C composites were prepared by microwave sintering of composition of B4C in Al 10wt%, 15wt% and 20 wt% and sintering temperature is about 650°C, 750°C, 850°C and 950°C. The different percentage of reinforcement leads to changes in mechanical properties of base matrix. The maximum values compressive strength (330 ± 10 MPa) and bending (238 ± 10 MPa) were measured samples prepared at 750 and 950°C respectively. Vicker hardness is 112HV for Al-20 wt% B4C which is sintered at 850°C. Formation of intermetallic compound i.e, Al3BC at 850°C which is investigated in XRD. Uniform distribution of reinforcement in base matrix which is shown in SEM micrograph. [16]

R.M. Mohanty et al.(2008) represented that 25 wt% Boron carbide (B4C) reinforced with aluminium metal matrix of aluminium 1100 sintered at 873K then further Micro structure and mechanical properties of investigated. Existence of porosity find out by Dark-field image. Hardness of material increases at 11 fold and flexible strength decreases formation of intermetallic compound with decreases the amount of reinforcement the fracture strength increases. If we increases the percentage of reinforcement it leads to massive damage to the grain boundary through pull out the particle by generating shear forces. Author's main objective is to reducing porosity by increasing interfacial bonding. [17]

V.Umasankar et al. (2013) report on an investigation about theatrical density and strength of sample during sintering. Here author described about micro hardness and density of sample increases with increasing amount of reinforcement and compacting pressure. By indenting in static ball they got that fracture strength increases by increasing compacting pressure and increasing amount of reinforcement. [18]

R. Narayanasamy et al. (2008) experimented on fabrication of silicon carbide in powder metallurgy route with effect on different particle size on base matrix. Composition of silicon carbide vary on (0-20)%.Different particle size of 50 μm,65μm and 120 μm is to be analyse for workability. Further investigation on formability stress index, strain hardening, indexing value and strength coefficient are most efficient desired outcomes. All are to relate with amount of reinforcement and different particle size of the reinforcement. [19]

C. Antony Vasntha Kumar et al.(2016) carried out experiment on rutile (TiO2) and silicon carbide (SiC) reinforced in aluminium metal matrix to form hybrid composite

through powder metallurgy route. Various percentage of TiO2 i.e,0%,4%,8%, and12% blended with 15% SiC along aluminium. Wear behaviour of composite find out by pin-on-disc by dry sliding wear condition. Uniform distribution of reinforcement is observed by Scanning Electron Microscope (SEM), X-ray diffraction (XRD), Energy dispersive spectroscope (EDS). Micro hardness and wear resistance increase by increase the percentage of TiO2 content. Micro hardness and wear resistance increases by increasing percentage of TiO2, high dislocation density and hardness is determined by SEM along with oxide layer on the sample detected by EDS. Due to oxide layer formation the contact area between the surfaces reduces therefore less loss on the material. [20]

P. Ravindran et al.(2013) studied that SiC reinforced with aluminium based graphite fabricated through powder metallurgy route. The hybrid composite formed lubricating itself by the help of graphite. Dry sliding and tribological behaviour is investigated by author. With adding SiC wear loss of composite decreases along with compacting pressure increases wear resistance increases with hardness. Low wear loss obtained also in high sliding speed. Tribologycal behavior of 20 wt% Gr with 5 wt% SiC better than other hybrid composition. Combination of hard reinforcement (SiC) and soft reinforcement (Gr) we got better result in wear behaviour which leads to various applications in automotive and aerospace industry. [21]

CHAPTER 3

3.1 EQUIPMENT, MATERIALS AND EXPERIMENTAL SETUP

3.1.1 MATERIALS

1. MATRIX MATERIAL

Aluminium metal matrix with grade 2024 and particle size of 44 μm used for fabrication which is procuring from parshwamanimetals metal Mumbai.

2. REINFORCEMENT

Vanadium Carbide used here one is less than 2µm which is procuring from sigma Aldrich.

3. LUBRICANT

Zinc sterate used as lubricant during compacting of powder for removal of specimen after compacting.

3.1.2 EXPERIMENTAL SETUP

1.COMPRESSION TESTING MACHINE

The compression testing machine is used for apply the load to a specimen up to its limit. The compression testing machine apply load up to 1000 KN.



Figure 3. Compression testing machine

3.2.2 MUFFLE FURNANCE

It is an electric furnace which is used for heating of material by avoiding inclusion of gas fly ash and combustion product. Front loading box type of oven is used for sintering of the product.



Figure 4 Muffle furnace

In this muffle furnace the refractory brick shown in the figure, above this brick all the samples are placed in ascending order of the percentage of sample of Vanadium Carbide. So after that close the chamber, make sure that the chamber is properly closed from the outside. Proper precaution should be taken while removal of material from the chamber so use gloves to remove the material.

CHAPTER 4

4.1 RESEARCH METHODOLOGY

The methodology follow to achieve our goals will be:

- 1.) To the study the recent research papers Aluminium metal matrix and Vanadium carbide from the trusted sources to acquire valuable data and studied the research paper according to year and making tabulation of reinforcement with aluminium matrix.
- 2.) And collecting data of various aluminium grades and for reinforcement.
- 3.) To study a series of experiments in the field of composite in powder metallurgy processes to about property enhancement.

Finally, the result obtained from both the sources will be combined and refined for the feasible and concrete end results.

CHAPTER 5

5.1EXPERIMENTAL PROCEDURE

5.1.1 PREPARATION OF COMPOSITE

MMC fabricated by Aluminium 2024 with particle size 44 μm and VC of particle size less than 2 μm through powder metallurgy route, series of process which are doing all fabrication process as follows-

1. BLENDING

Al 2024 with different different percentage of reinforcement i.e. VC blended in a mortar and pestle up to 5 to 10 min.



Figure 5. Mortar and pestle

.

2.COMPACTING

Compacting is how much pressure is needed to material is fully densify. Compaction in a die is the conventional method mostly used now a day. Rigid die press the powder in

mechanical or hydraulic press. Density achieved up to 90% after compacting. A friction force opposes the pressure while compacting of powder.

Uniformity after compacting we get by-

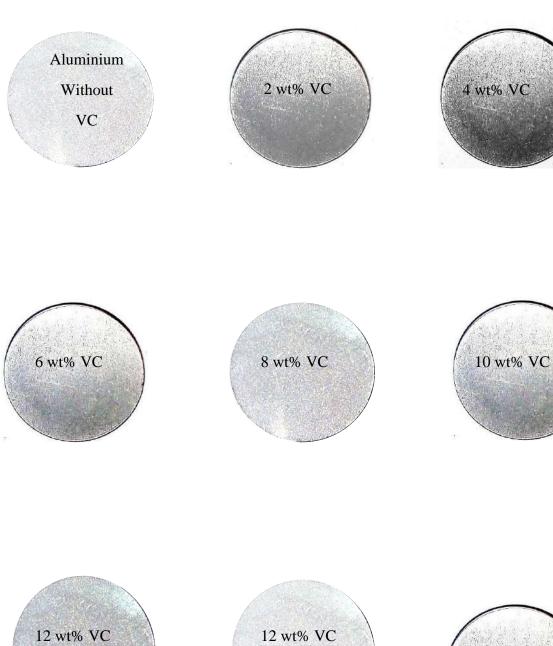
- 1. Proper compacting technique
- 2. Tool type
- 3. Types of material used for pressing
- 4. Lubricant

Movement of individual tool leads to proper compacting like lower punch, upper punch and die. Without use of lubricant leads to variation of density upper portion and lower portion specimen. In upper portion we get high density and in lower portion obtained less density because friction in die wall prevent from uniform pressure distribution.

Compacting carried out in a die of diameter 15.9 mm and with a pressure of 250MP.

In compression testing machine we compacted the powder in 49.65 KN load without dwell time.

After compaction the samples are shown given below-



5.1.3 SINTERING

Consolidation of powder in to a desired structure in a thermal process at atomic level is call sintering. Bonding tends to improved mechanical, electrical and magnetic properties. Solid state diffusion in single phase is most efficient and accurate in sintering.

Sintering is carried out in a muffle furnace a temperature up to 600°C with increasing 10°C per min. All samples are place at one times (i.e. nine sample along with its 2 replica) so that all samples get same sintering atmosphere up to one hour.

while removal of material from the furnace. Different percentage of reinforcement looks different colour after sintering.

CHAPTER 6

RESULTS AND DISCUSSION

6.1 DENSITY

Density tends to mass for unit volume. Density is calculated normally by measuring the samples diameter and height in micrometre. After calculating we have observed that we get different density value before sintering and after sintering. Density increase after sintering we get that One important thing that with increasing percentage of VC density of sample also increases.

Table 1. Density

VC(%) in AMC	Density of sample before sintering	Density of sample after sintering
0% VC	2.44	2.44
2%VC	2.35	2.38
4%VC	2.43	2.49
6%VC	2.42	2.47
8%VC	2.41	2.44
10%VC	2.42	2.45
12%VC	2.53	2.57
14%VC	2.43	2.50
16%VC	2.23	2.52

6.2 MICROHARDNESS

Resistance to a proper indentation we called as the hardness. Which shows that deformation and resistance by acquiring proper strength. In Vicker hardness test indenter made up of diamond which is in square pyramidal shape of apex angle 136°. We can apply load up to 10-15 sec to the specimen resulting a rectangular-shaped indention impression. From which we get d1 and d2. From d1 and d2 we get the hardness value.

Micro hardness of sample find out in microscopic level and it is a method used to find out the hardness of material or resistance to penetration in to the material. Micro hardness of sample increases by increasing percentage of VC in AMC.

Table 2. Micro hardness

VC(%) in AMC	Micro hardness
0% VC	38HV
2%VC	38HV
4%VC	42HV
6%VC	44HV
8%VC	53HV
10%VC	50HV
12%VC	54HV
14%VC	59HV
16%VC	64HV

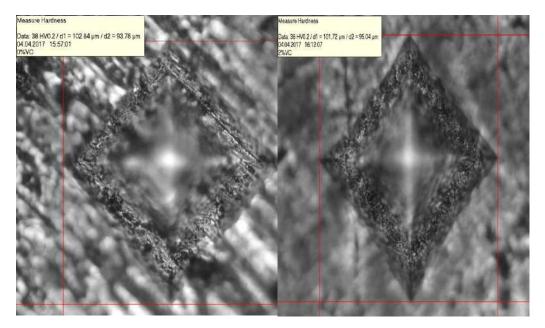


Figure 7. Micro hardness of aluminium

Figure 8. Micro hardness of $\ 2 \ wt\%$ VC

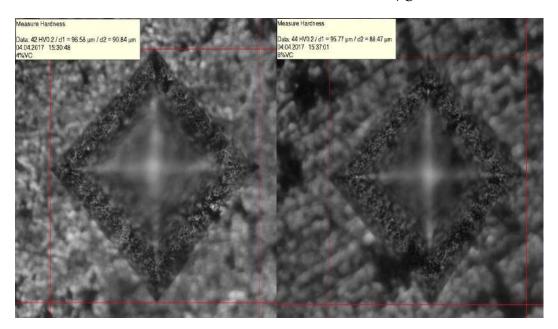


Figure 9. Micro hardness of 4. wt%

Figure 10. Micro hardness of 6. wt%

Figure shown here about the micro hardness of sample by analysing data the micro hardness value increase as we further increase the reinforcement percentage. In aluminium sample the micro hardness is 38HV and it further increase up to 44 HV in 6% wt reinforcement of the sample.

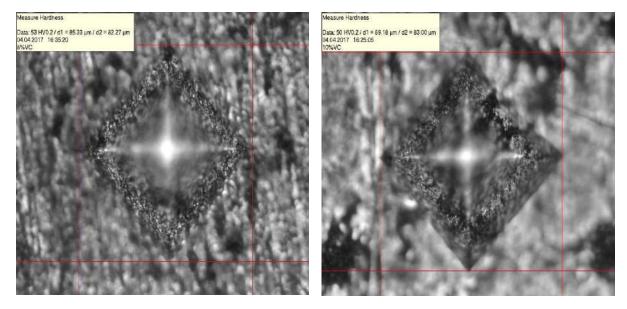


Figure 11. Micro hardness of 8 wt% VC

Figure 12. Micro hardness of 10 wt% VC

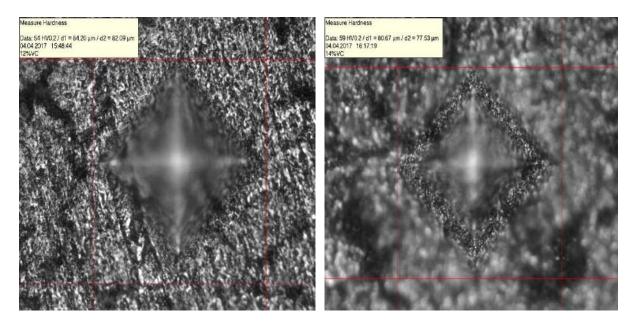


Figure 13. Micro hardness of 12. wt% VC

Figure 14. Micro hardness of 14 wt% VC

Similar case arises also here with increasing the percentage of reinforcement micro hardness of the sample also increases. We should note that for proper micro hardness proper defect free surface is required.

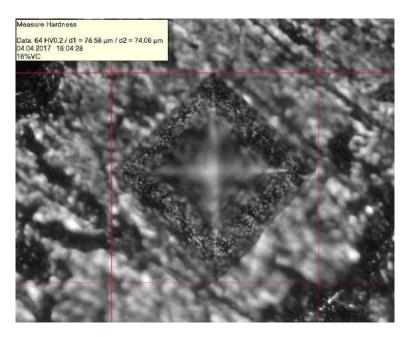


Figure 15.Micro hardness of 16 wt% VC

We got highest micro hardness value in 16 wt% reinforcement of the sample i.e. 64 HV. So Vanadium Carbide increase the micro hardness of the composite sample.

6.3 SEM

3D quality image are produced by electron are scattered from back and generate remarkable images. Florescent pattern are produced which is generated in X ray from semi qualitative chemical analysis. Many objective lenses are assembled by illuminating object in dark field by transmitting and reflecting the work. Its remove the limitation of microscope by examining rough and irregular object most efficiently. From SEM we can find out about whether metal is poorly-sintered or well-sintered, We get unsintered particle boundaries in poorly sintered materials in small pores and angular pore edges are located in particle grain boundaries.

In scanning electron microscope scanning is carried out focused electron beam after that we get the desired image of the object. Different resolution of image is taken by me to find out desired result e.g. Aluminium(Al), Vanadium Carbide(VC) and any other formation of intermetallic compound like Al3V.

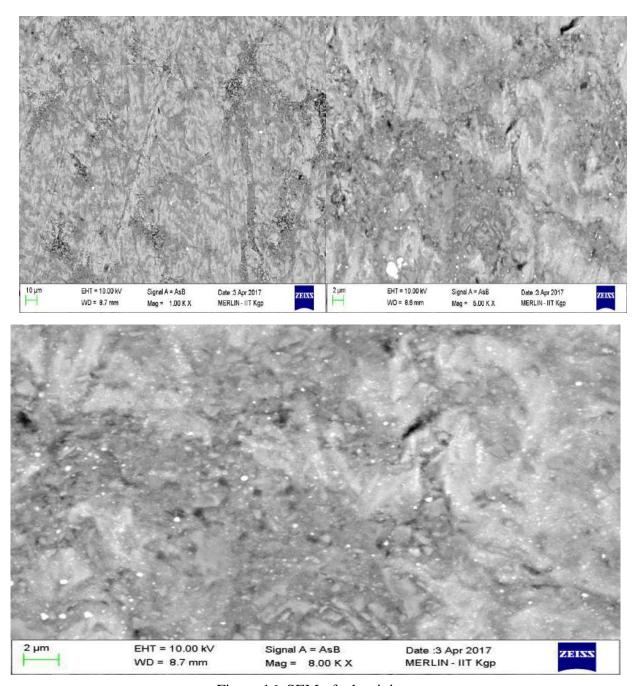


Figure 16. SEM of aluminium

SEM micro graph taken in to consideration of the samples that magnification of 1 KX, 5KX and 8 KX samples we get aluminium composition in the sample.

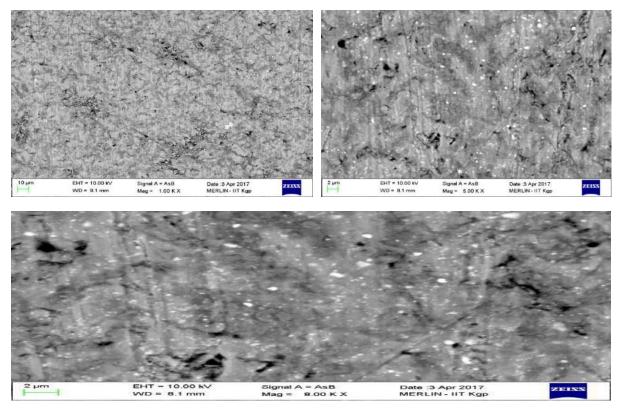


Figure 17. SEM of VC 2% by wt

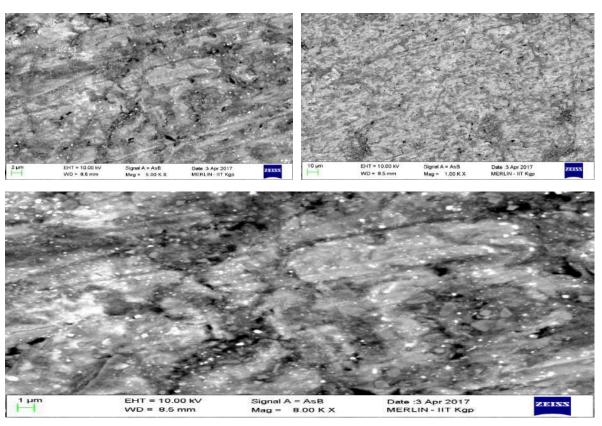
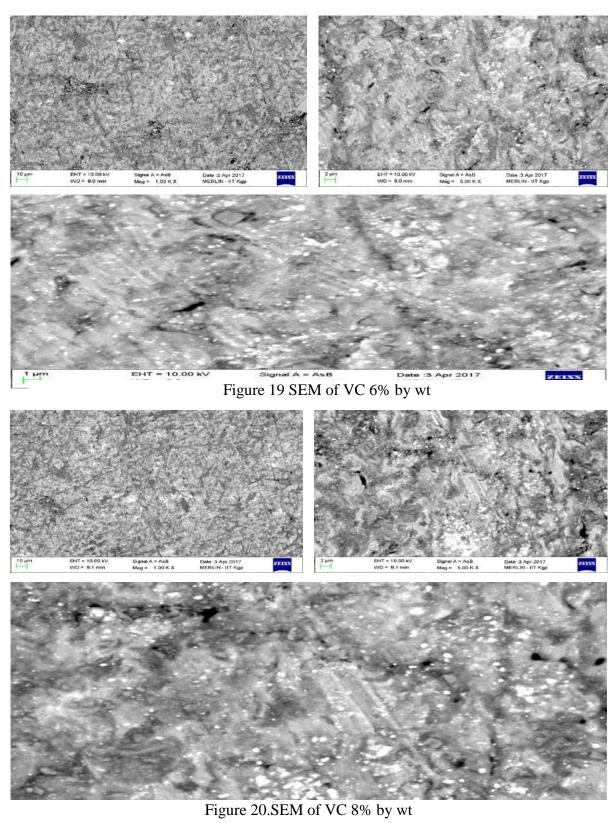
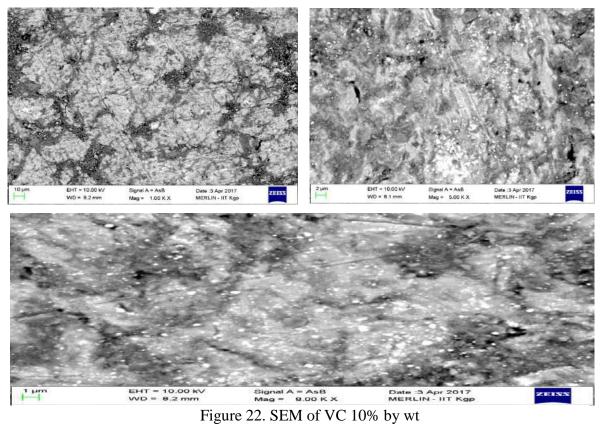


Figure 18. SEM of VC 4% by wt





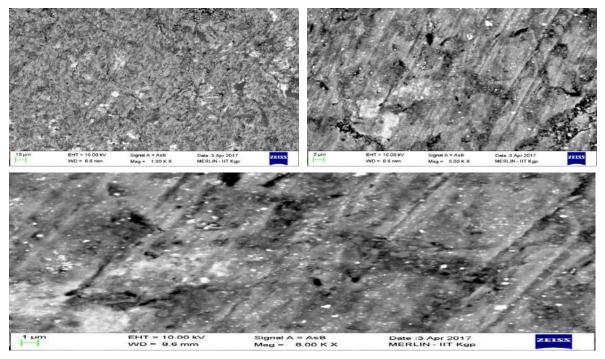


Figure 23.SEM of VC 10% by wt

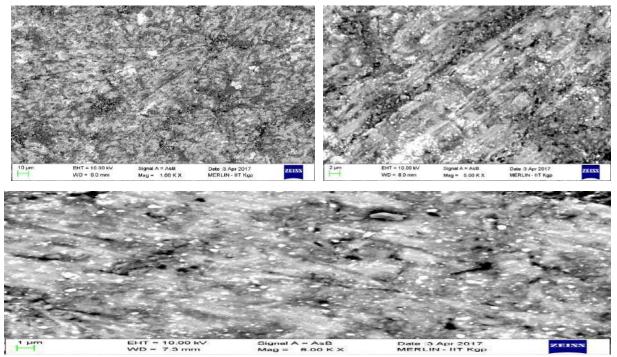


Figure 25.SEM of VC 14% by wt

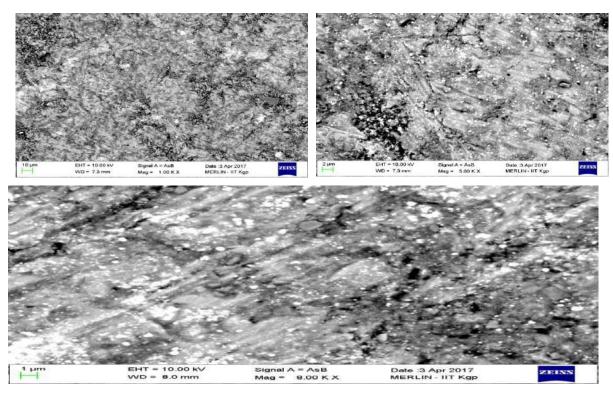


Figure 26.SEM of VC 16% by wt

From SEM we concluded about the morphology of the sample white sports are Vanadium Carbide and clear area are aluminium. We have successfully avoided the Intermetallic compound formation i.e. reaction between base metal and reinforcement, which leads to porosity on the metal.

6.4 OPTICAL MICROSTRUCTURE

6.4.1 SAMPLE PREPARATION

6.4.1.1 Grinding

Using emery paper of 220,500,1000 grid size along with water act as coolant which placed on automated grinding wheel which rotate rpm of 300.after grinding specimen are cleaned ultrasonically in alcohol.

6.4.1.2 Polishing

Polishing operation carried out using 1µm diamond polishing sprey. Unetched samples have porosity, pore, shape and size, foreign particle inclusion.

6.4.1.3 Etching

it is performed by immersing the specimen in a suitable echant. It's observed by different phases, grain size and alloying. Eaching is unnecessary if specimen is reflect and absorb the incident light. Echant used in polishing is Keller's reagent which content 90% water, 1.5% hydrochloric acid, 2.5% nitric acid, 1% hydrofluoric acid. Before keep the sample under the microscope we have to clean it properly in ethanol.

Specimen is mounted on metallurgical microscope. Magnification start at low x100 then further increase up to our requirement.x100, x500 and x1000 magnification image are captured.



Figure 27. Microstructure of aluminium

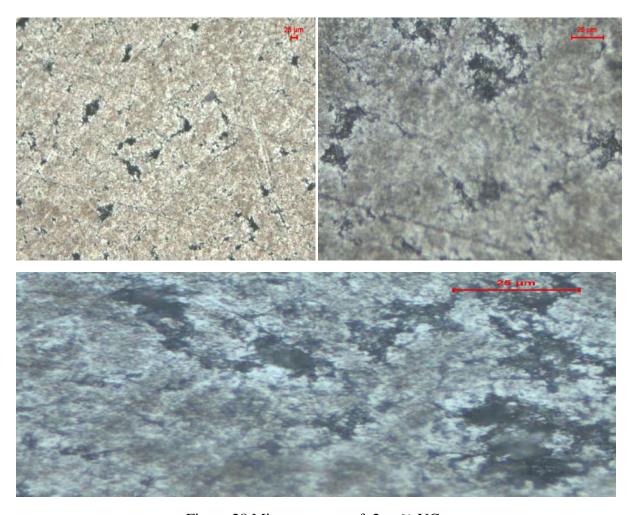


Figure 28.Microstructure of 2 wt% VC

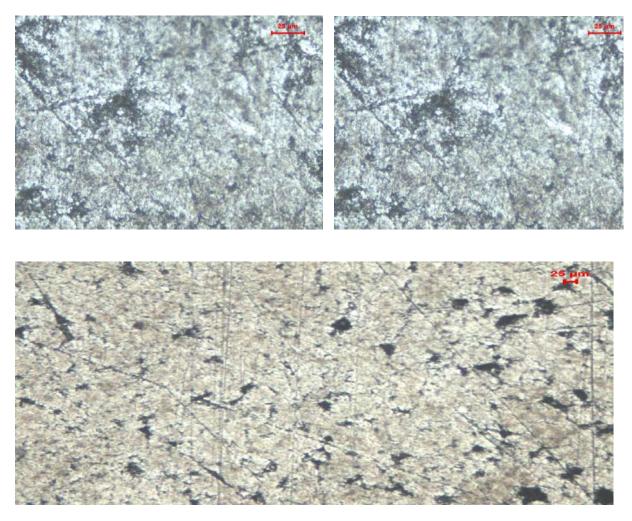


Figure 29.Microstructure of 4 wt% VC

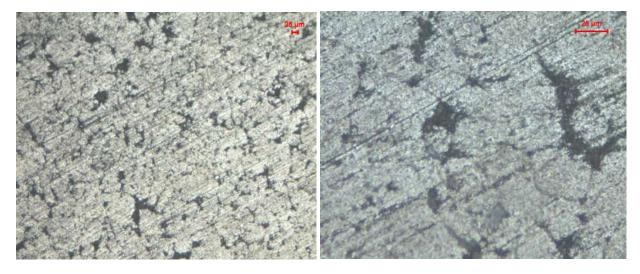


Figure 30.Microstructure of 6 wt% VC

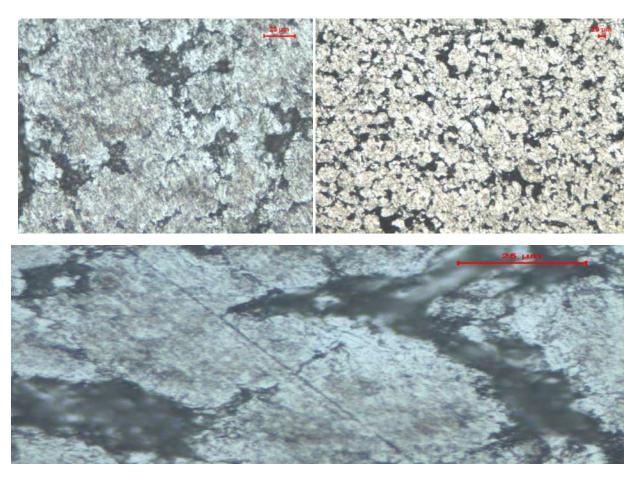


Figure 31.Microstructure of 8 wt% VC



Figure 32.Microstructure of 10 wt% VC

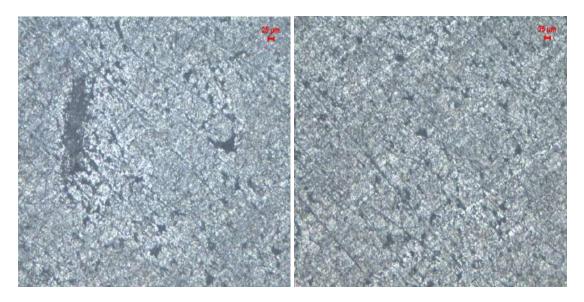


Figure 33.Microstructure of 12 wt% VC

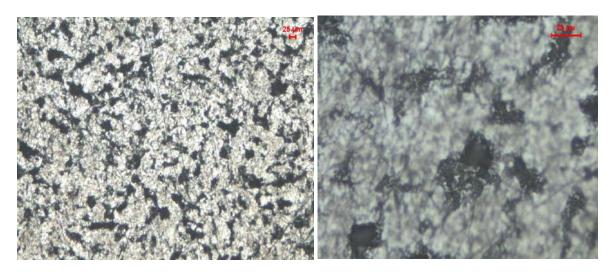


Figure 34.Microstructure of 16 wt% VC

By inspecting optical microstructure of sample white portion are aluminium 2024 and black portion are Vanadium carbide. Intermetallic compound formation by stoping the reaction between base metal and reinforcement.

6.5 XRD

It is technique used for phase identification of crystaline material and also provide the inormation of unit cell dimentions. Interference pattern are produced by x-ray beam impose on acrystalline solid. Usally all its wavelength identical. In a rotating holder the sample is placed properly. Diffraction occurs according to Bragg equation:

$$d = \lambda/2\sin\theta$$

After analyze properly we get the peaks of graph which has been ploted by origin pro software. Aluminium to Vanadium carbide composition we got from different peaks as shown below.

X-ray diffractometers consist of three basic elements: an X-ray tube, a sample holder, and an X-ray detector.

X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. These spectra consist of several components, the most common being K_{α} and K_{β} . K_{α} consists, in part, of $K_{\alpha 1}$ and $K_{\alpha 2}$. $K_{\alpha 1}$ has a slightly shorter wavelength and twice the intensity as $K_{\alpha 2}$. The specific wavelengths are characteristic of the target material (Cu, Fe, Mo, Cr). Filtering, by foils or crystal monochrometers, is required to produce monochromatic X-rays needed for diffraction. $K_{\alpha 1}$ and $K_{\alpha 2}$ are sufficiently close in wavelength such that a weighted average of the two is used. Copper is the most common target material for single-crystal diffraction, with CuK_{α} radiation = 1.5418Å. These X-rays are collimated and directed onto the sample. As the sample and detector are rotated, the intensity of the reflected X-rays is recorded. When the geometry of the incident X-rays impinging the sample satisfies the Bragg Equation, constructive interference occurs and a peak in intensity occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor.

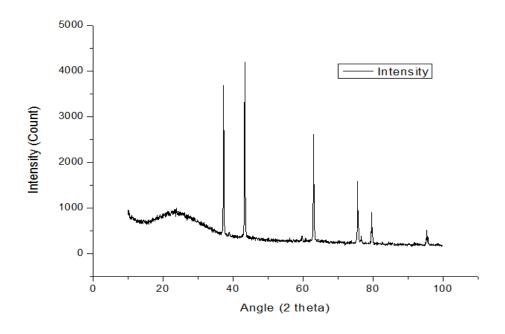


Figure 35.XRD of aluminium

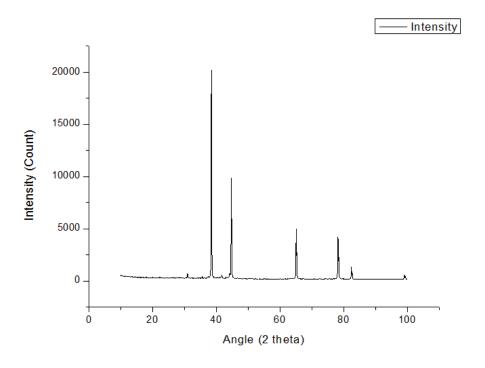


Figure 36. XRD of 2 wt% VC

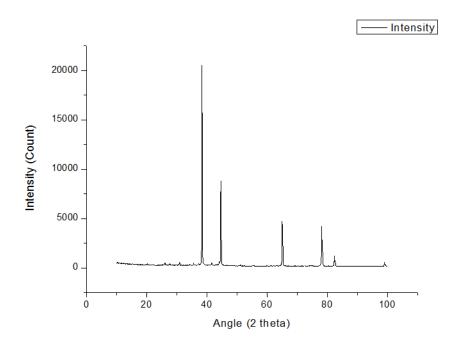


Figure 37.XRD of 4 wt% VC

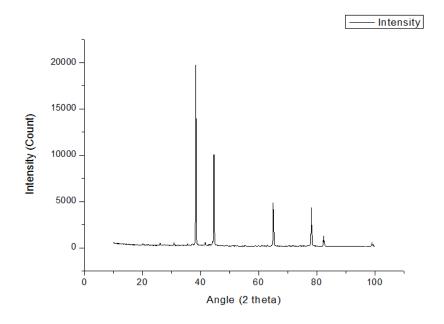


Figure 38.Figure 44.XRD of 6 wt% VC

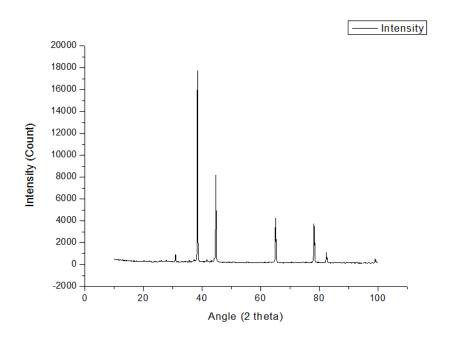


Figure 39. XRD of 8 wt% VC

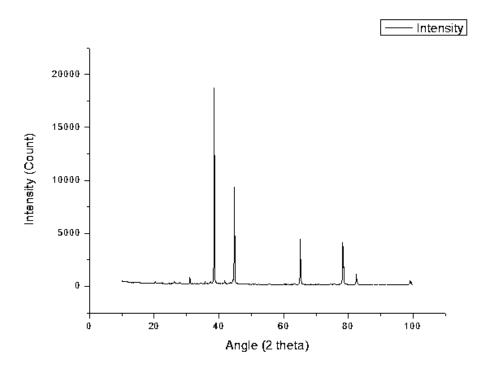


Figure 40. XRD of 10 wt% VC

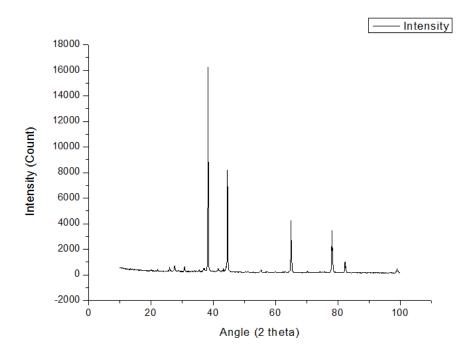


Figure 41.XRD of 12 Wt% VC

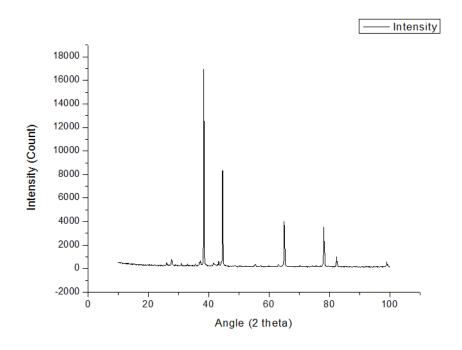


Figure 42.XRD of 14 wt% VC

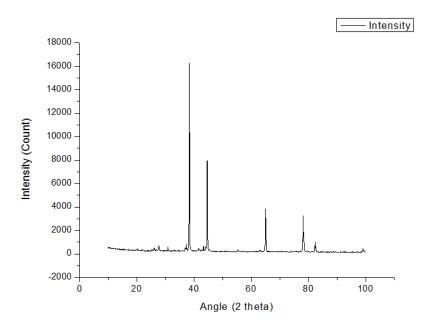


Figure 43. XRD of 16 wt% VC

After analyse the peaks of the graphs we concluded that aluminium is in the highest and Vanadium Carbide at the bottom point and due to conventional sintering their no reaction between the base matrix and reinforcement. So there is no intermetallic compound formed in the composite. The main advantage of XRD it is a non destructive testing with no sample prepatration required.

CHAPTER 7

CONCLUSION AND FUCTURE SCOPE

- 1. By numerous studies we get that increasing percentage of VC micro hardness of sample increases.
- 2. Density of sample consequently increases by increasing VC percentage.
- 3. We got best result on sample by analysing density and micro hardness of sample after loading proper compacting pressure along with uniform distribution reinforcement in base metal matrix.
- 4. SEM shows the Morphology of the sample and we clarify that there is no intermetallic bond form in the sample.
- 5. From the peaks of XRD in the graph we got the amount of reinforcement in different sample as we increasing reinforcement peaks vary and there is no sign of intermetallic bond formation and reaction between the samples.
- 6. From optical micro structure we got the best result on morphology of sample.
- 7. There is huge scope in automotive and aerospace industry because of its extreme hardness and high density.

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