

**Experimental investigation on Mechanical and Metallurgical Properties of
Titanium Based Composite Used for Aerospace Applications**

A Dissertation

Submitted in partial fulfilment of
requirements for the award of degree of

Master of Engineering

in

Production Engineering

Submitted by

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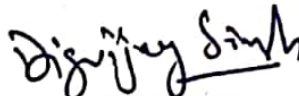
July, 2019

Certificate

I hereby declare that the work done in the Dissertation entitled "Experimental investigation on Mechanical and Metallurgical Properties of Titanium Based Composite Used for Aerospace Applications" submitted towards partial fulfillment of requirement for award of degree of Master of Engineering in Production Engineering, Thapar Institute of Engineering and Technology, Patiala, is an authentic record of the work carried out by me under the supervision and guidance of Dr. Vinod Kumar Singla, Associate Professor, Mechanical Engineering Department, Thapar Institute of Engineering and Technology, Patiala.

The matter embodied in this report has not been submitted in part or full to any other university or institute for the award of any degree.

Dated: 13/9/19


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This is to certify that above declaration made by the student concerned is correct to the best of our knowledge and belief.


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Dedicated to
My loving
Parents ...

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ABSTRACT

Titanium matrix composites have important and wide applications in the aerospace industries due to light weight and strength. The current work was focused on powder metallurgy of reinforced titanium matrix composite with graphene. Ti-6Al-4V alloy and graphene powders were used as matrix and reinforcement respectively. The wt. % of Ti-6Al-4V alloy and graphene powders were 99.5% and 0.5% respectively. Spark plasma sintering technique was used for fabrication of composite. The mixture of powder sintered at 1100°C at an applied load of 60 MPa under 6 Pa vacuum with heating rate of 150 °C/min and holding time of 15 min was used. The microstructural observation was performed on SEM and optical microscope. The SPS process leads to high densification of composite within short time and produces good surface. The hardness of the composite improved significantly by adding reinforcement.

Keywords: Titanium matrix composite, Graphene, Microstructure, Hardness, Spark Plasma Sintering.

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3.

ABBREVIATIONS

MMCs -	Metal Matrix Composites
TMCs -	Titanium Matrix Composites
ND -	Nano Diamond
CNT -	Carbon Nano Tube
VGCF -	Vapour Growth Carbon Fibers
SPS -	Spark Plasma Sintering
RM -	Reactive Milling
wt.% -	weight percentage
GNSs -	Graphene Nano Sheets
GNPs -	Graphene Nano Platelets
YS -	Yield Strength
UTS -	Ultimate Tensile Strength
XRD -	X-Ray Diffraction
SLGO -	Single Layer Graphene Oxide
SEM -	Scanning Electron Microscope
DC -	Direct Current
VH -	Vickers Hardness
Ra -	Roughness average
Rz -	Average maximum height of the peak
Rq -	Root mean square roughness

CHAPTER 1

INTRODUCTION

1.1 METAL MATRIX COMPOSITES

Conventional material does not have combination of different mechanical properties such as strength, stiffness, toughness, density etc. Current engineering applications require materials that are stronger, lighter and less expensive. To overcome this problem and to meet the demand of latest technology we need composites material like metal matrix composites which provides the combination of different properties. A good example is the current interest in the development of materials that have good strength to weight ratio suitable for automobile applications where fuel economy with improved engine performance is becoming more critical. Metal matrix composites are fast replacing conventional metallic alloys in so many applications as their use have been extended from predominantly aerospace and automobile to defense, marine and sports.

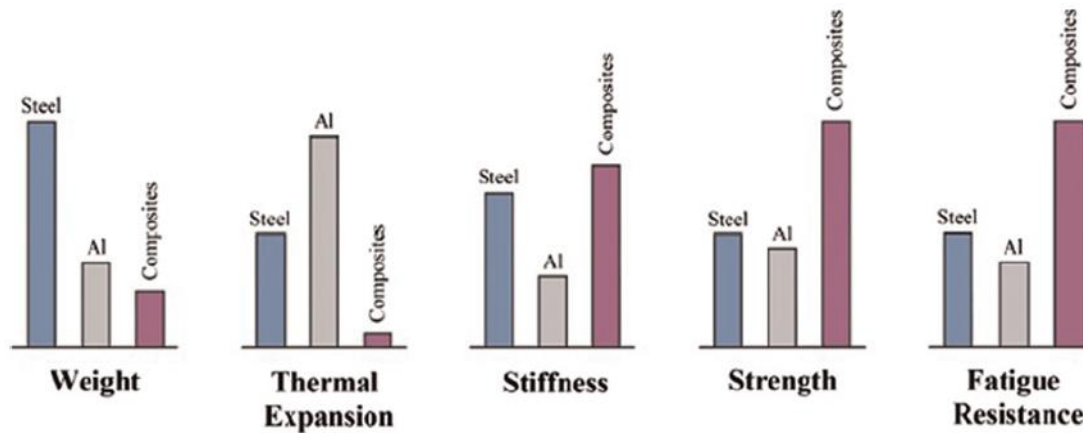


Figure 1.1: Comparison of Properties: Metals vs Composites (Source: M Haghshenas, 'Metal-Matrix Composites, DOI: 10.1016/B978-0-12-803581-8.03950-3)

Metal matrix composite provides such combination of properties such as high specific strength, low coefficient thermal expansion and high thermal resistance, good damping capacities, superior wear resistance, high specific stiffness and satisfactory levels of corrosion resistance.

Advantages of MMCs over metals:

1. High thermal conductivity

2. Good radiation resistance
3. High strength and stiffness
4. Low moisture absorption
5. Higher electrical conductivity
6. Food process ability
7. Good fire resistance

But there are some disadvantages of MMCs over metals:

1. High cost of production
2. Immature technology
3. Complex fabrication process[1]

A metal matrix composite is a composite material in which minimum two materials is present. In which one material is metal necessarily or other is a may be a metal or non metal such as organic compound, ceramics etc. When there is more than two elements are present then the composite is known as hybrid composites [2].

Metal matrix composites are formed by reinforcing a reinforcement material into matrix metal. Generally reinforcement material surfaces are coated to prevent the reaction between reinforcement and matrix metal. For example, carbon fibers are used in aluminium matrix for making light weight and high strength composites. But carbon reacts with aluminium and make a hard and brittle material so to prevent this carbon fibers are coated with nickel or titanium boride.[2] Matrix is a material with specific mechanical and physical properties such as formability, thermal conductivity, and ductility. While on the other hand reinforcement is a hard substance with high strength, low density, low thermal expansion and high stiffness [1].

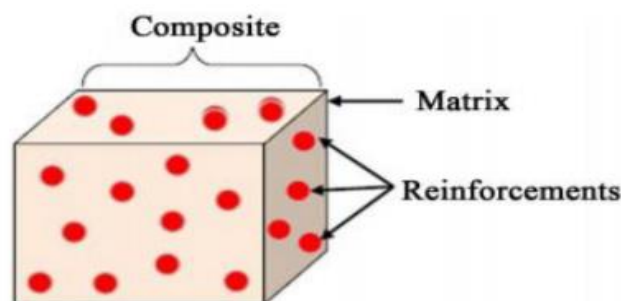


Figure 1.2: Composite material (Source: Mechanical Characteristics and Novel Applications of Hybrid Polymer Composites- A Review)

1.2 MATRIX

The matrix is a material in which reinforcement is added. Matrix is monolithic and completely continuous. Matrix is generally a lighter material like magnesium, copper, aluminium, titanium etc which provide support to the reinforcement and for high temperature applications cobalt and their alloys are used commonly [2].

1.3 REINFORCEMENT

Reinforcement is a material which is reinforced or embedded into the matrix. Reinforcement material we use is generally stronger and harder than the matrix material. Reinforcement material not only used to reinforcing into the composite but it can also change the physical properties of the composite such as wear resistance, density, strength, thermal conductivity or friction coefficient [2]. Reinforcements are of two types:

- 1.3.1 **Discontinuous reinforcement:** these reinforcements are generally isotropic so can be easily worked with standard working techniques such as forging, rolling etc. And can be machined with conventional techniques.
- 1.3.2 **Continuous reinforcement:** these use fibers such as carbon fibers therefore are anisotropic in nature. Most commonly examples of continuous reinforcement are silicon carbide and alumina [2].

1.4 CLASSIFICATION OF MMCs

MMCs can be classified on the basic of matrix metal we used and can be classified as follows:

- 1.4.1 Aluminium based composites
- 1.4.2 Magnesium based composites
- 1.4.3 Titanium based composites
- 1.4.4 Copper based composites
- 1.4.5 Super alloy based composites

1.4.1 Aluminium based composites

Aluminium based composites are the most commonly used composites in aerospace and automotive industries. The reason behind this is the properties of aluminium based composites and these properties are higher strength, good stiffness, low density, higher temperature capabilities, controlled thermal expansion, higher wear resistance. In aluminium

based composites one part is aluminium or aluminium such as AlSi, AlCu, AlSiMg alloys which is known as matrix and the other part is embedded into matrix known as reinforcement such as SiC, Al₂O₃, C, B, B₄C, AlN, and BN. [1]

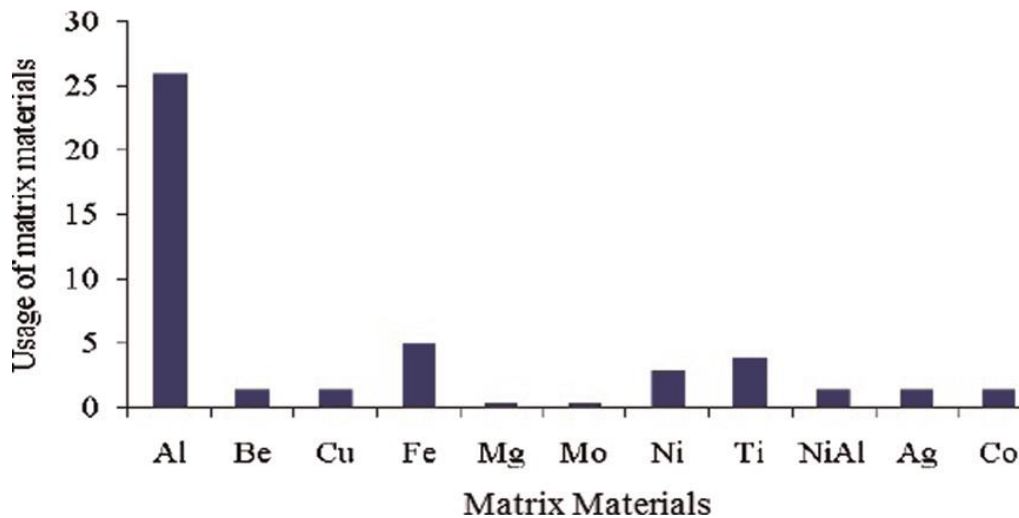


Figure 1.3: Usage volume of different matrix materials in MMCs (Source: A. A. Adebisi, M. A. Maleque, M. M .Rahman, ‘Metal matrix composite brake rotor: Historical development and product life cycle analysis’, *International Journal of Automotive and Mechanical Engineering* 4, 471-480, 2011,[3])

The advantages of reinforced aluminium over the simple or non reinforced aluminium are as follows:

- High strength
- High stiffness
- Low density (or light weight)
- Better higher temperature properties
- Controlled thermal expansion coeff.
- Better thermal management
- Higher electrical conductivity
- High wear resistance
- High damping properties

In recent years, aluminium composites are in different engineering sectors due to its various structural, non structural and functional applications which increase the use of aluminium based composites in these sectors including economic and environmental benefits.

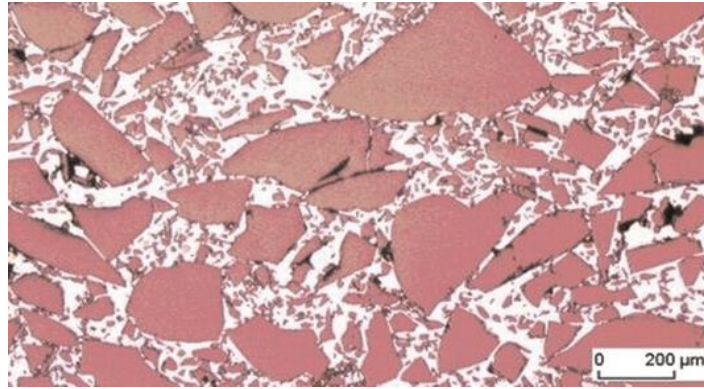


Figure 1.4: SiC-particle reinforced aluminium-matrix composite material (SiC-volume content: 70%) (Source: Sorensen, J., 2011. Great Potential (Still) for Metal Matrix Composite Structures, CPS Technologies Blog, <http://www.alsic.com> [4])

The major profit for using aluminium composites in transportation sectors is their low fuel consumption, less noise pollution and less airborne emission [1].

1.4.2 Magnesium based composites

For high demand of lower weight and high performance material there is a need to increase study of magnesium based composites. The metal matrix composites made by using magnesium or magnesium alloys such as Mg-Al are used for light weight engineering material and for different military and aerospace application. Magnesium based composites are used for various automotive application such as for making gears, connecting rods, shaft, piston, rotor, gearbox bearing etc. One disadvantage of using magnesium MMCs is its high cost of production.

The most commonly used matrix material for magnesium based composite in automotive industries are Mg-Al alloys such as AM60 and AZ91. That's why they are the most studied matrix for magnesium based MMCs. Other matrix materials for matrix materials for magnesium composites are used rarely such as pure magnesium, Mg-Ag-Re alloy, Mg-Li alloy. Ceramics are most commonly used reinforcements for magnesium based MMCs due to its various properties such as low density, high hardness, high strength, high modulus of elasticity, high thermal stability. But with various properties it also has some limitations such as less ductility, less wettability, less compatibility. When such properties required then SiC is most popular due to its high wettability and stability with magnesium as compared to others.

1.4.3 Titanium based composites

For years titanium-based composites were mainly considered aerospace materials; however, titanium is progressively more being considered for non-aerospace, industrial applications, such as automotive, industrial and consumer application. The high specific properties (e.g., strength/density, stiffness/density, and modulus/density), creep resistance, and corrosion resistance of titanium, make it good for many applications. The addition of hard particles reinforcement in matrix to produce a titanium-matrix composite is also a very useful method for increasing wear resistance properties.

The additive particles selected as reinforcements of TMCs include: Cr_3C_2 , TiC, TiN, TiO_2 , Si_3N_4 , SiC, Ti, TiB, Al_2O_3 , Zr_2O_3 , R_2O_3 , (R stands for rare earth element), Ti_3Al , or TiAl, and Ti_5Si_3 , intermetallic compounds.

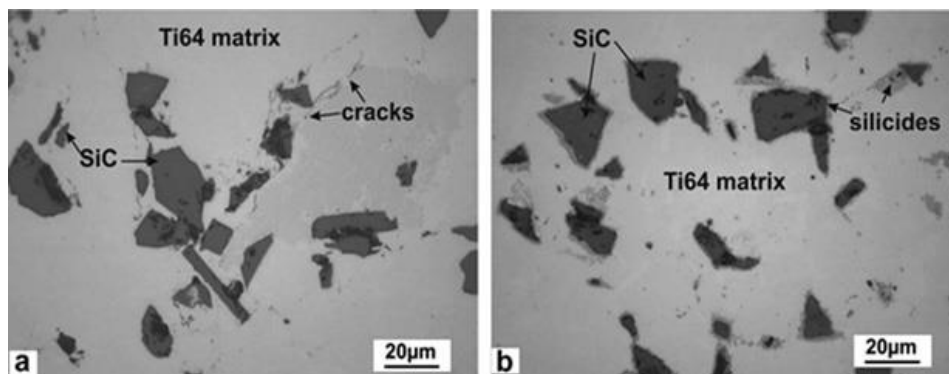


Figure 1.5: Microstructures of a SiC reinforced titanium matrix composite (Sources: Bewlay, B.P., Jackson, M.R., Zhao, J.C., Subramanian, P.R., 2003. A review of very-high-temperature Nb-Silicide-based composites. Metallurgical and Materials Transactions 34A, 2043–2052. [5])

Boron (B) particles and carbon nano particles such as nano diamond (ND), carbon nano tube (CNT), and vapour-growth carbon fibers (VGCF) are also effective single elements to form reinforcing compounds.

1.4.4 Copper based composites

When Al_2O_3 is used as reinforcement material in copper matrix then unique properties can be achieved such as high thermal conductivity, high electrical conductivity, high strength and superb resistance to annealing. Copper has high thermal conductivity and SiC, CuSiC have low coefficient of thermal expansion that's why there composites are good solution for

thermal applications. When density, thermal conductivity and coefficient of thermal expansions are required than CuSiC based metal matrix composites are used.

Copper silicon carbide has good thermo mechanical properties and higher conductivity. They also have less density, good thermal conductivity, good machining capabilities and low coefficient of thermal conductivity in comparison to copper. But while manufacturing the CuSiC composites the main challenge is to prevent the reaction between copper and silicon carbide due to which its thermal conductivity changes.

1.4.5 Super alloy based composites

Super alloys are mainly used in applications of gas turbine for making the blades of turbine. By increasing the strength of material and high operating temperature, the performance of material is increased therefore the overall operating cost is reduced. In recent years in the area of super alloy based composites there is a high progress for developing Nb-silicide-based composites. For very high temperature applications such as in jet engines, rockets these composites are very useful. For example the melting point of silicide based composites is approx 1750°C.

Another important super alloy composite for high temperature applications is ceramic-fibre reinforced super alloys. These composites are produced with a very slow rate or on demand and unit cost of these composites are very high. For industrial purpose these composites are not suitable due to its high cost [1].

1.5 PROPERTIES OF METAL MATRIX COMPOSITES

1.5.1 Physical properties

The most common physical properties of MMCs are density, thermal conductivity and coefficient of thermal expansion. Composites are multifunctional materials which can combine both physical as well as mechanical properties. Physical properties of composites depend on the addition of reinforcement in matrix. Aluminium silicon carbide composites is a example in which ceramic increases the elastic modulus of the MMC without affecting its density. It is observed that specific axial thermal conductivity of MMCs is usually greater than copper and aluminium.

1.5.2 Mechanical properties

By adding reinforcement to the different metals and alloys their mechanical properties can be improved such as greater strength and stiffness as well as high wear resistance. By adding brittle and hard reinforcement particles into the metal matrix give strength to the metal. The main mechanical properties of metal matrix composites are strength, modulus of elasticity, fatigue, elongation, creep which is discussed as follows.

Strength

Adding of reinforcement into the matrix can increase both yield strength and ultimate tensile strength. Predicting the strength of metal matrix composites in its constituent phase is very difficult. Sometimes it can be increased very dramatically. By using continuous fibers, as reinforcement material into the matrix, highest increase in modulus and strength can be achieved.

Modulus of elasticity

The stiffness of a material is directly proportional to the modulus of elasticity which can be increased by adding reinforcement to the matrix. Type and volume fraction of reinforcement into the composite are the main factors which affect the elasticity of the MMCs.

Fatigue

By adding reinforcement into the matrix the fatigue life of composite also increases. The fatigue resistance of unreinforced material is less than the fatigue resistance of reinforced composites when loaded in tension. At aging temperature the strength of matrix decreases with results in decrease of fatigue resistance.

Elongation

Elongation in composites is less than the matrix alloy. The main factors which affect the ductility of the composites are type, size, nature, microstructure and distribution of particles of reinforcement into the matrix.

Creep

The addition of short-fiber reinforcement into matrix increases the creep strength. The whisker-reinforced MMCs are more creep resistance than particulate MMCs [1].

1.6 APPLICATIONS OF METAL MATRIX COMPOSITES

Application of MMCs can be divided into aerospace and non-aerospace categories. For aerospace application low density, low thermal expansion and conductivity, high strength and stiffness is required most not the cost but performance. And for non aerospace applications cost and performance both can affect.

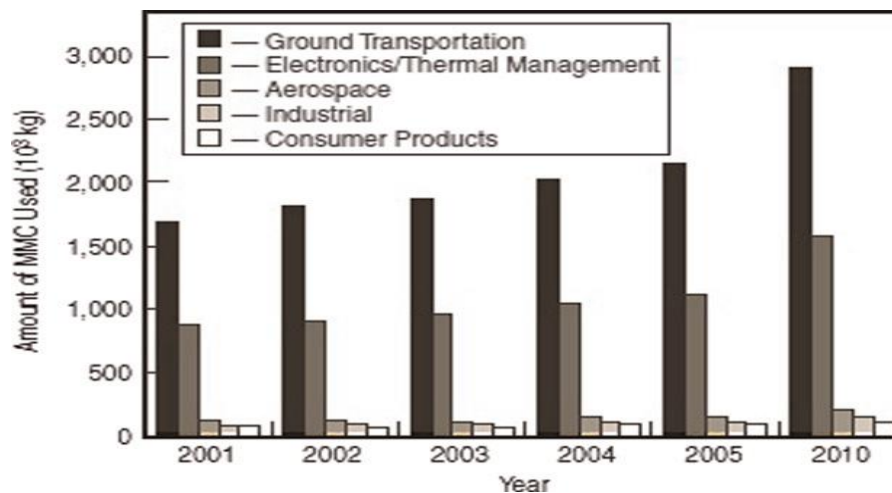


Figure 1.6: The use of MMCs in different market sectors (Source: [1])

1.6.1 Aerospace applications

Due to high specific stiffness and high strength metals matrix composites are used in a very large scale in aerospace industries because it increases the performance of the aircraft. They are also used in military and commercial aircraft. For military applications continuous fiber composites are used because they have high strength, high stiffness and high fatigue resistance. In F-16 aircraft aluminium doors are replaced with SiC reinforced composite [1].

Boeing 787 uses MMCs in their airframe and structure more than any other Boeing commercial planes.

1.6.2 Automotive applications

MMCs are used in different automotive applications. By using MMCs 50% weight of engine is reduced over cast iron without increasing engine size. In automotive market cost is considered to be low as much as possible. Therefore mainly aluminium composites are used in this market. About 57% weight reduced in connecting rod without increase of cost.

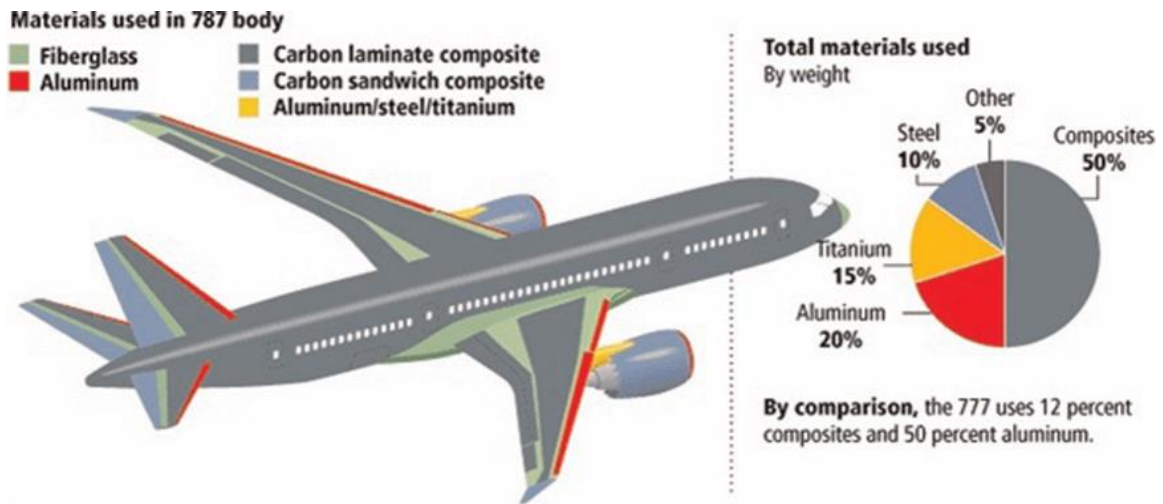


Figure 1.7: Percentages of materials used in Boeing 787 aircraft (Source: Holley, R., 2013. *The Great Metal Tube in the Sky. Material Strategies, Innovative Applications in Architecture*, <https://arch5541.wordpress.com>. [6])



Figure 1.8: Brake drums and brake rotors made of MMC (Source: Chawla, N., Chawla, K.K., 2013. *Metal–Matrix Composites, seconded*. New York: Springer Science.)

1.6.3 Electronics applications

In electronic, new integrated circuits generate more heat than the previous one therefore thermal fatigue occurs. This problem can be solved by using MMCs by matching coefficient of thermal expansion.

1.7 REINFORCEMENT MATERIALS USED IN COMPOSITES

There are mainly three types of reinforcement used in composite, namely, fibers, fabric particles and whisker. In fibers one axes is very long when compared to other two axis that may be circular or nearly circular. Particles do not have any defined shape and orientation. Whiskers have defined shape but very small in length as well as diameter when compared to fibers. Figure 1.9 show different types of reinforcement.

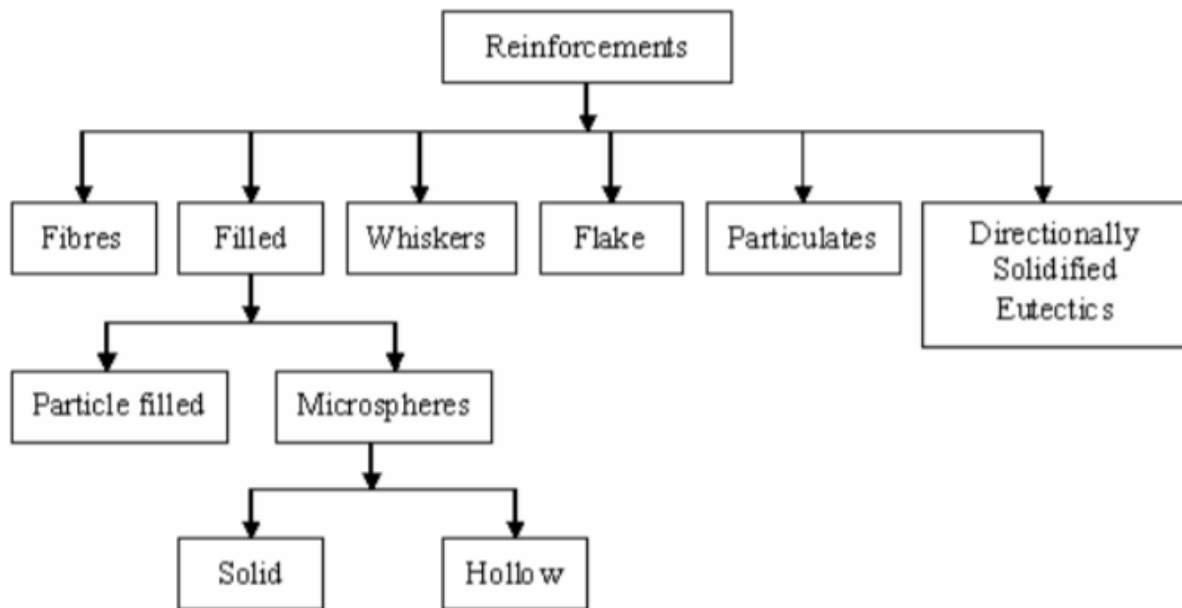


Figure 1.9: Different types of reinforcement used in composites (Source:

https://nptel.ac.in/courses/Webcourse-contents/IISc-BANG/Composite%20Materials/pdf/Lecture_Notes/LNm1.pdf)

Reinforcement gives strength to the composite which we required for different applications. But they also give heat resistance, resistance to corrosion, conduction and rigidity to the composite. Reinforcement gives strength to the composite resist the failure of matrix that means a composite must have minimum ductility and brittle as much as possible. [8]

1.7.1 Fibers

Fibers transfer desired strength to the matrix and enhance their properties as desired. Length, shape, orientation and composition of the fibers are responsible for the performance of fiber composite. Strength of the composite is indicated by the orientation of the fibers and the strength of composite is highest when the fibers are oriented in longitudinal direction. Great performance of the longitudinal fibers is obtained when the load is applied in the direction of fibers. A very little change in angle of loading decreases the strength of composite significantly.

Organic and inorganic fibers are used in composites. Organic fibers have high flexibility, high elasticity and low density while inorganic fibers have high thermal stability, high modulus and greater rigidity than organic fibers. Examples of fibers are silicon carbide fibers, glass fibers, high silica and quartz fibers, metal fibers, graphite fibers, alumina fibers, multiphase fibers, boron fibers etc. [8]

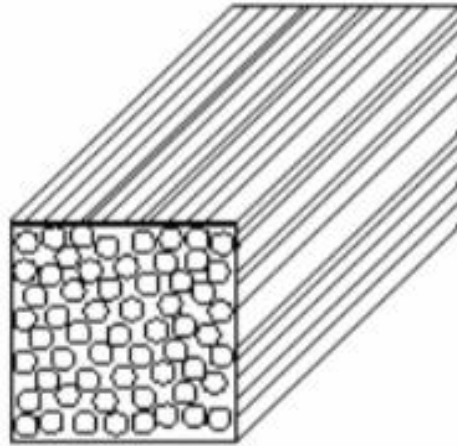


Figure 1.10: Long fiber reinforced composite (Source: [8])

1.7.2 Whiskers

Whiskers are single crystal with nearly zero defects. Whiskers are short fibers with different cross section made up from copper, iron, graphite, silicon carbide etc. Generally whiskers are 3 to 55 μm in length and have strength of 700 MPa. Combination of metals and whiskers give strength to the system at higher temperature. Ceramic whiskers are very good for low weight structure due to high specific modulus and high specific strength.

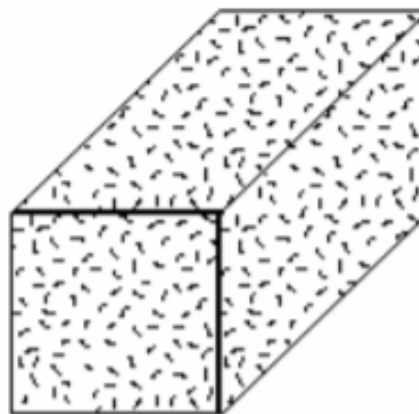


Figure 1.11: Short fiber reinforced composite (Source: [8])

1.7.3 Flakes

Flakes are generally used in place of fibers because they are densely packed and also due to lower cost and easy to produce. But they also don't have control to the final product and shows defects in end product. Glass flakes leads to cracks on the edges which weakens the final product. For structural applications flakes have more advantages than fibers. Flakes

have high modulus of elasticity than fibers and relatively economical to produce and handled in small quantity. [8]

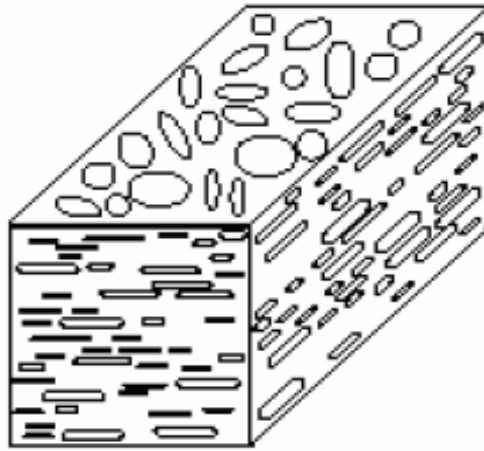


Figure 1.12: Flakes composite (Source: [8])

1.7.4 Filler Composites

These composites can be made by adding filler material to the plastic matrix which can increase the properties of composite and also increases the strength and reduces weight. The filler particles are irregular in shape and have polyhedrons, short fibers or spheres type geometry.

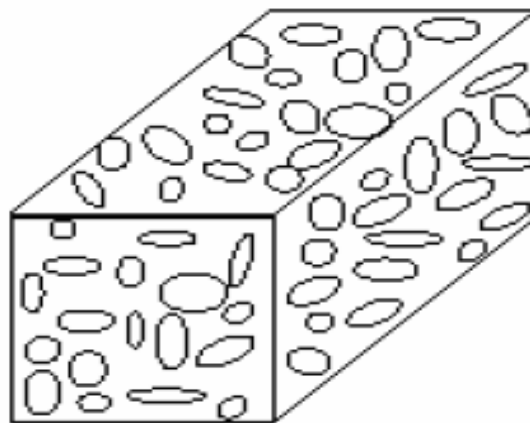


Figure 1.13: Filler Composites (Source: [8])

The advantages of filler are they can increase stiffness, thermal resistance, abrasion resistance, stability, strength.

1.7.5 Particulate Reinforced Composites

In this type of composite the particles give strength to the system. The strength of the composite depends on the particle's size, diameter, and volume fraction of reinforcement. Matrix material also influences the properties of the composite. [8]

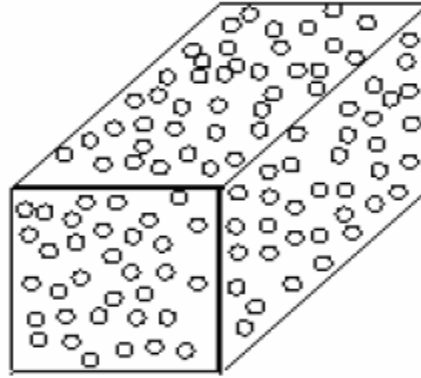


Figure 1.14: Particulate Reinforced Composites (Source: [8])

1.8 GRAPHENE

Graphene is a two dimensional carbon allotrope. It is in form of hexagonal lattice in which atoms are present on each vertex. It has different types of properties. Graphene is 100 times stronger than steel. It is a good conductor of heat and electricity and also almost transparent. It was produced for centuries in small quantities for making of pencils and other applications. But in past years after many researches were done and knowing its new properties it was produced in large scale. Due to which graphene global market reached to \$9000000 by 2012. It is mostly used for the purpose of research and development in electronics, composites and semiconductors. [9]

1.8.1 Properties of Graphene

1. Graphene is a good conductor of electricity.
2. It has high thermal conductivity approx $5300 \text{ Wm}^{-1}\text{K}^{-1}$.
3. It is the strongest material which has high mechanical properties.
4. It has tensile strength of 139 GPa and stiffness (young's modulus) of 1 TPa.
5. It has fracture toughness of about $4\text{MPa}\sqrt{\text{m}}$. [9]

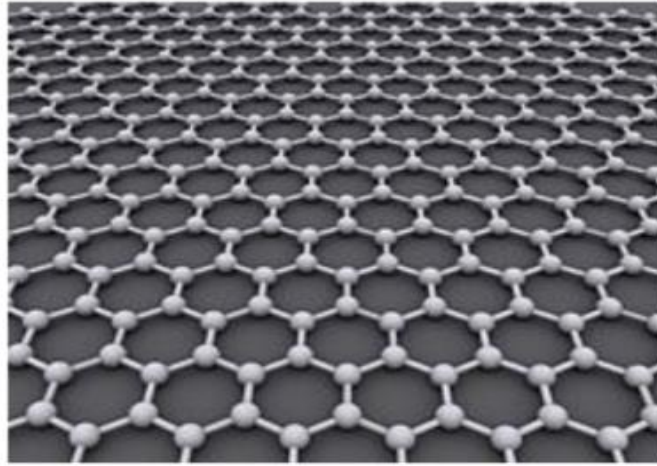


Figure 1.15: Hexagonal Structure of Graphene (Source: [9])

1.8.2 Applications of Graphene

- 1. Biomedical:** Graphene has different biomedical applications such as tissue engineering, smart implants, targeted drug delivery etc.
- 2. Composites and coating:** Due to its mechanical properties such as strength, conductivity, light-weight, flexibility graphene is used as reinforcement for making composites.
- 3. Electronics:** Due to various electronic properties it is used for making different types of electrical products such as semi-conductors, transistors, touch screen of phones and tablets.
- 4. Energy:** Graphene is used in batteries for fast charging and reduce weight of the batteries. It is also used in solar panel.
- 5. Sensors:** Graphene is used for making ultra sensitive sensors and can detect very tiny particles. [10]

CHAPTER 2

LITRETURE SURVEY

In this chapter we shall be studying different research paper on composite to know more about the composites, different fabrication techniques and there process parameters to decide the process and material for present thesis work.

2.1 FABRICATION OF METAL MATRIX COMPOSITES

Prakash et al. [11] prepared the composite by powder metallurgy by adding 0, 5 and 10 weight percent of Boron Carbide (B_4C) ceramic particles as reinforcement and Ti-6Al-4V as matrix. It was found that by increasing B_4C content into the composite density decreases, hardness increases and corrosion resistance capability also increases. It was also found that Ti-6Al-4V composite had high wear resistance when compared to Ti-6Al-4V alloy.

Thotsaphon et al. [12] prepared the composite by spark plasma sintering by using pure titanium as matrix and carbon nanotubes (CNT) as matrix. It was found that mechanical properties such as tensile properties and elongation improved significantly by adding the CNTs to the pure titanium. By scanning electron microscope fracture surfaces of tensile specimens were analyzed and found a uniform distribution of CNT particles in the composite.

Chao et al. [13] prepared the composites by powder metallurgy method by using ZrO_2 as reinforcement and pure titanium as matrix material. It was found that by adding ZrO_2 mechanical properties of composite increased and compressive strength of 1380 MPa (570 MPa more than pure Ti and good plasticity was found out.

Poletti et al. [14] used SiC as reinforcement and titanium as matrix material and fabricated by powder metallurgy. It was found that densities found to be nearly 100% to the theoretical, no reaction zone and better particle distribution oriented in extrusion direction.

Junqiang et al. [15] used TiB, TiC and Nd_2O_3 as reinforcement particles reinforced into the Ti matrix metal by powder metallurgy technique. It was found that the reinforcement particles were distributed uniformly into the matrix. TiB grows in needle shape while TiC and Nd_2O_3 grow in equiaxed shape. It was also found that addition of NdB_6 reduce grain size and porosity of composites.

Ensieh et al. [16] used the SiC nanoparticles as reinforcement and titanium as matrix material and fabricated by powder metallurgy method it was found that by increasing SiC weight percent in composites relative density also increases. It was also found that energy absorption of composites increases with increasing SiC nanoparticles into composite.

Yong et al. [17] studied the effect of Nd (Neodymium) rare earth metal on three different titanium alloys, namely, Ti-4.5Al-6.0Mo-1.5Fe, Ti-6Al-1Mo-1Fe and Ti-6Al-4V. It was found that addition of Nd metal increases the density of titanium alloys slightly (maximum 1%). It was also found that there was little increment in tensile strength but elongation increased significantly.

Liu et al. [18] fabricated Graphene Nano Sheets/TiC bulk composites using SPS from GO colloid and TiC powders. The addition of GNSs results in grain refinement of TiC matrix and fully stops the TiC grain growth. The flexural strength and Vickers hardness of GNSs/TiC composites have been enhanced as compared to monolithic TiC due to their finement of matrix grains.

Li et al [19] fabricated the composite by reinforcing bulk graphene into aluminium matrix. It was found that stiffness, tensile strength and elongation of composite slightly improved when compared to the matrix aluminium metal.

2.2 EFFECT ON MECHANICAL PROPERTIES OF COMPOSITES

Li et al. [20] used multiwall carbon nanotubes as reinforcement and pure titanium as matrix and fabricated by powder metallurgy method. It was found that there was 143.6% and 80.7% increase in yield strength and ultimate tensile strength respectively when compared to pure titanium. It was also found that ductility increases when reinforcement content increases.

Carrasco et al. [21] used Ni₃Al as reinforcement and aluminium as matrix metal. It was found that there was a great advantage of the extrusion process over casting method. The composite was stable at 300 °C upto 1000 hr no interdiffusion between aluminium and Ni₃Al found.

Isabel et al. [22] used boron carbides and amorphous boron particles as reinforcement material and titanium grade 1 powder as matrix material and fabricated by direct hot pressing. It was found that the smaller the particle better the composite behaviour. It was also found

that there was direct relationship between decrement of elongation and increment of reinforcement concentration.

Thotsaphon et al. [23] used low cost carbon black as reinforcement and titanium (Ti) powders (sponge and fine Ti) as matrix material fabricated by spark plasma sintering. The mechanical properties improved by adding small amount of low cost carbon black (0.07-0.16 mass %). The YS of the extruded sponge and fine TMC were increased to 70.0 and 291 MPa and tensile strength increased to 67 and 231 MPa, respectively when compared to pure titanium.

Zheng et al. [24] used B_4C particle as reinforcement and Al2024 alloy as matrix material and fabricated by powder metallurgy method. The fracture strength of 100% milled Al2024 powder composite was 670 MPa. It was found that when the unmilled Al2024 powder volume fraction increased from 10% to 40% the compression strength decreased from the room temperature compression strength decreased from 1115 MPa to 739 MPa and at 50% volume fraction of unmilled Al2024 powder decreased to 580 MPa.

Geng et al. [25] prepared the composite by synthetic reaction using TiB and Nd_2O_3 as reinforcement and titanium as matrix material. It was found that TiB grows in needle shape and Nd_2O_3 grows in dendrite or sphere shape.

Joshi et al [26] prepared Ti-TiO₂ composite powders by reactive milling (RM). It was found that the composite powder offer the great high temperature and high strength properties to the system. It was also found that the hardness increases with increase in milling time.

Kondoh et al. [27] prepared the titanium matrix composite by SPS technique by adding carbon nanotubes as reinforcement and titanium as matrix. It was found that the mechanical properties of composite improved by the addition of CNTs. By the addition of 0.35 mass % of CNTs tensile strength and yield strength are increased by 157 MPa and 169 MPa respectively.

Singh et al. [28] prepared the composite by adding 2, 5, 10 and 20 wt. % of TiB₂ powder as reinforcement and titanium as matrix by vacuum sintering method. It was found that the while increasing wt. % of reinforcement relative density decreases and the hardness of composite increases significantly.

2.3 EFFECT ON CHANGING WEIGHT PERCENT OF REINFORCEMENT

Mahesh et al. [29] used titanium nitride (TiN) as reinforcement and aluminium (Al) as matrix material and fabricated by powder metallurgy technique. 5, 10, 15 weight percentage of TiN particles in composites were used. The powder was compacted at two different pressures 250 MPa and 300 MPa and sintered at two different temperatures 400°C and 500°C. It was found that hardness, density and compressive strength of composite increases with increase in reinforcement content and the surface roughness decreases with increase in amount of reinforcement, compacting temperature and sintering temperature.

Li et al. [30] prepared the composites by spark plasma sintering by 0.1-0.4 wt% of carbon nanotubes (VGCFs) and graphite (Gr) reinforced into the titanium matrix. It was found that mechanical properties of composite increases when VGCF/Gr content increase from 0.1-0.4 wt%. YS and UTS of Ti 0.4wt% of VGCF was increased from 40.4% and 11.4% when compared to pure Ti and those are 30.5% and 2.1% for Ti-0.4 wt% Gr.

Bhatt et al. [31] used aluminium as matrix and magnesium with four different weight percent (0.5, 1, 2.5 and 5) and SiO₂ constant at 5 weight percent as reinforcements fabricated by spark plasma sintering. It was found that Mg completely dissolved into Al matrix and also decreases in particle size by XRD observation. It was also found that hardness of nanocomposites is two times higher than microcomposite.

Dash et al. [32] used Al₂O₃ with different volume fraction (5, 10 and 15 volume %) as reinforcement and copper as matrix material and fabricated by spark plasma sintering with different sintering atmosphere (N₂, H₂ and Ar). It was found that maximum Vickers hardness value found out 60, 75 and 80 at Cu-15% Al₂O₃ in N₂, Ar and H₂ atmosphere. It was also found that Cu-Al₂O₃ composite show poor mechanical properties in N₂ and Ar atmosphere while compared to H₂ atmosphere.

Maseko et al [33] prepared the Ti based binary composites by incorporating ZrB₂ into titanium matrix using SPS technique. It was found that with 10 weight percent of ZrB₂ composite shows highest hardness when compared to 5 and 15 weight percent of ZrB₂ composite.

Babatunde et al [34] prepared the Ti-Ni-ZrO₂ composites by spark plasma sintering technique. It was found that there were no pores and cracks in the composite with uniform distribution of Ni. It was also found that with increase of ZrO₂ content from 5 to 10 volume percent Vickers hardness also increases.

Hu et al [35] fabricated metal matrix composite by reinforcing single layer graphene oxide (SLGO) in titanium matrix using laser sintering method. Microstructure and component of composite were studied by SEM, XRD, EDS and Raman spectroscopy. SLGO-Ti composite showed high value of hardness and modulus when compared to laser sintered Ti. The Vickers hardness values of 1wt%, 2.5 wt% and 5 wt% SLGO-Ti composite were 630VH, 742VH and 509VH respectively.

Savalani et al [36] prepared the composites by laser cladding technique. 5, 10, 15 and 20 wt. % of CNTs were used as reinforcement and Ti as matrix metal. It was concluded from the study that mechanical properties were improved by increasing the CNTs content. It was also found that the composites formed had high hardness and high wear resistance.

Bustamante et al. [37] fabricated the composite by adding 0.25, 0.5 and 1.0 wt % of graphene nanoplatelets (GNPs) into aluminium powder matrix by mechanical alloying. Composites fabricated were microstructurally characterized with Raman spectroscopy, SEM and XRD. It was found that hardness of the composite increased by increasing milling time at 2 hour sintering time.

2.4 EFFECT OF CHANGE IN TEMPERATURE ON MECHANICAL PROPERTIES OF MMCs

Mu et al. [38] prepared the composites by 1.8% volume fraction of grapheme nanoplatelets as reinforcement and pure titanium as matrix by powder metallurgy techniques. The mechanical properties are compared for different ball milling time (20 min and 2.5 hour) and for different hot pressing temperature (900°C, 1150°C, and 1300°C). It was found that 27.3% increase in UTS when compared to pure titanium under 20 min ball milling. Elongation increased with temperature. It was also found that 51.5% increase in UTS under 2.5 hr ball milling and 1300°C hot pressing temperature when compared to pure titanium.

Feng et al. [39] prepared the composites by spark plasma sintering using Ti-4.0Fe-7.3Mo alloy as matrix and TiB as reinforcement at three different sintering temperatures (800°C,

1000°C and 1200°C). It was found that the composite sintered at 1000°C shows maximum values of relative density, flexural strength, Young's modulus and moderate fracture toughness which were 99.6%, 1007 MPa, 146 GPa and 8.64 MPa.m^{1/2} respectively.

Xiangbo et al [40] reinforced TiB particles into titanium matrix by spark plasma sintering method at 950 to 1250°C. It was found that by increasing sintering temperature relative density of composites also increases. It was also found that at 1250°C composite showed highest relative density, bending strength and fracture toughness.

Mu et al [41] fabricated the composite by reinforcing multi layer graphene (MLG) into pure titanium using spark plasma sintering technique followed by hot rolling. It was found that composite formed showed good tensile strength of 1050 MPa. It was also found that when rolling temperature increased from 1023 K to 1223 K the volume fraction of titanium carbide increased from 2.5 to 12.3 higher than MLG.

2.5 EFFECT ON MECHANICAL PROPERTIES OF COMPOSITES FABRICATED USING VARIOUS PROCESSES

Kumar et al. [42] prepared the composites by three different techniques, namely, hot isostatic pressing, spark plasma sintering and vacuum sintering. TiB with 20% and 40% fraction as reinforcement and titanium as matrix material were used. It was found that mechanical properties such as elastic modulus, microhardness and Poisson's ratio are different with different techniques and also change by volume fraction of TiB content.

Saeid et al. [43] prepared the composites by two different methods one was press and sintering and other was spark plasma sintering by using TiB as reinforcement and Ti-6Al-4V alloy as matrix material. It was found that the two main defects found in press and sintering method which was porosity and clustering of TiB but these were not found in spark plasma sintering. It was also found that at 1100°C all the B₄C additive powder reacts completely but at 900°C some unreacted B₄C particles were found.

Kondoh et al. [44] prepared the composite by SPS and hot extrusion by using 1, 2 and 3 weight% of CNTs as reinforcement and Ti as matrix. It was found that the mechanical properties of composites significantly improved by adding small amount of CNTs. It was also found out that the coarsening and growth of Ti grain never occurred.

Cao et al. [45] fabricated the composite by reinforcing graphene into Ti6Al4V matrix by using hot isostatic pressing (HIP). The mechanical properties and microstructure were studied by tensile tests, SEM and optical microscope. It was found that by reinforcing graphene to Ti6Al4V strength of the composite significantly improved without losing its ductility.

2.6 RESEARCH GAPS FROM LITERATURE REVIEW

1. From literature survey it also observed that very less work was done on titanium MMCs and titanium alloys. These alloys have various applications in aerospace, automobile, marine and commercial industries and also titanium withstands at high temperature and titanium has a high strength to weight ratio therefore titanium as matrix material for our composites was chosen.
2. Weight plays a vital role in the usage of Titanium based hybrid composites therefore there is be a need of developing light weight hybrid composites which can fulfil the need of industrial applications.
3. There is a need of developing such material which can withstand at high temperature.

CHAPTER 3

OBJECTIVES AND METHODOLOGY

3.1 OBJECTIVES OF PRESENT WORK

1. Fabrication of Ti metal matrix composites by reinforcing graphene using spark plasma sintering method.
2. To study the mechanical properties such as micro structure of composite.
3. To determine the microhardness and surface roughness value of composite.

3.2 MATERIAL SELECTION

A lot of work was done on aluminium and magnesium alloys, therefore titanium alloy (Ti-6Al-4V) was used as a matrix material and graphene was used as a reinforcement material in composite.

Titanium was used in present work due to following reasons:

1. Ti-6Al-4V alloy has high strength and high toughness.
2. They are light weight.
3. They withstand at high temperature and good electrical properties.
4. They have wide range of applications in aerospace, automobiles, defence, etc.

Due to mechanical properties such as strength, conductivity, light-weight, flexibility graphene is used as reinforcement for making composites. Graphene was used in present work due to its following properties:

1. Graphene is a light weight material.
2. It is the strongest material having high tensile strength.
3. It is a good electrical conductor.
4. Graphene withstand very high temperature.
5. Graphene has wide range of applications in biomedical, composites, sensors, solar energy, etc.

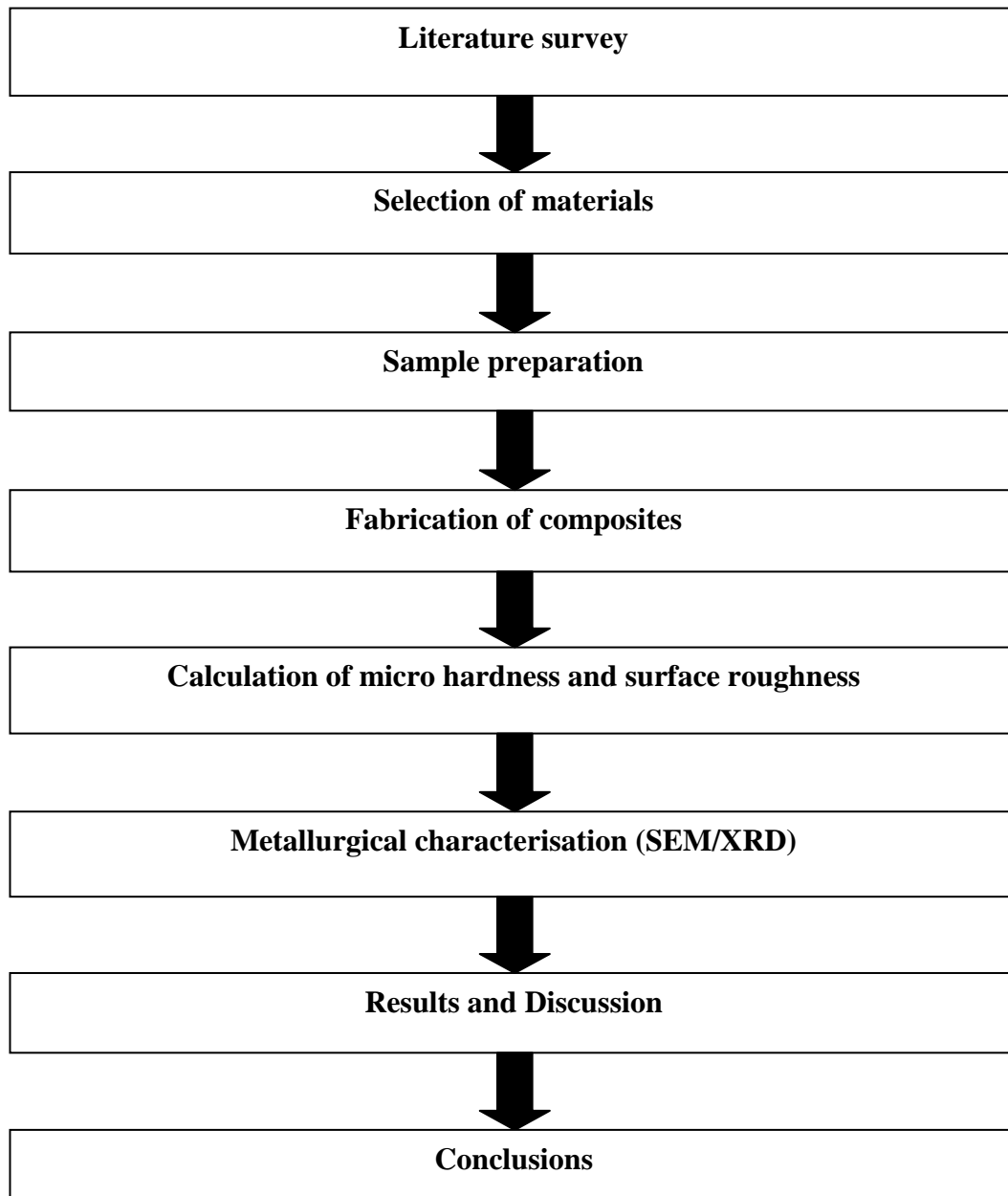


Figure 3.1: Flow chart of work plan

Table 3.1: Composition of Ti-6Al-4V alloy

Alloy	Ti	Al	V	Fe	O	C	N	H
Ti-6Al-4V	87.6%	5.5%	3.5%	≤0.4%	≤0.02%	≤0.08%	≤0.05%	≤0.015%

Table 3.2: Properties of Ti-6Al-4V alloy

S. No	PARAMETERS	VALUE
1.	Density	4.429- 4.512 g/cm ³
2.	Ductility	0.05- 0.18
3.	Hardness	3370- 3730 Mpa
4.	Melting temperature	1178- 1933 K

Table 3.3: Properties of Graphene

S. No	PARAMETERS	VALUE
1.	Density	2.267 g/cm ³
2.	Melting point	3652- 3697 °C
3.	Boiling point	4200 °C
4.	Thermal conductivity	119- 165 W/m/K

3.3 EXPERIMENTAL SETUP

Ti-6Al-4V powder (Parswami metals Mumbai, India) and graphene (Shilpa enterprises, India) were used as the raw material for preparing the composite. The average particle size of Ti-6Al-4V powder is about 20 microns and graphene is 5 microns. Mixtures of both powders were prepared by using 99.5 weight% of Ti6Al4V and 0.5 weight% of graphene powder in Oscar Ultrasonicator Setup (available at TIET patiala) for 5 hours at 10 sec pulse time ON or 2 sec pulse time OFF using acetone.

SPS setup (CSIR-National Physical Laboratory, Delhi) with current on/off ratio 12:2 was used for preparing the composite. The mixtures were sintered under 6 Pa vacuum. The specimen was sintered at sintering temperature of 1100°C, at an applied load of 60 MPa. The heating rate of 150 °C/min and holding time of 15 min was used. The composite was prepared for characterization and testing for microhardness and surface roughness.



Figure 3.2: Ti6Al4V-Graphene composite prepared using SPS process

3.4 EQUIPMENTS USED

Oscar ultrasonicator was used for mixing the powders using acetone. Spark plasma sintering setup was used for fabrication of composite. Surface roughness and micro hardness test were performed on the composite. Scanning Electron Microscope (SEM) was used for surface morphology. XRD machine was used for crystalline grain size. The equipments used for present work and measurements are given below:

3.4.1 Oscar ultrasonicator

Oscar ultrasonicator was used for preparing the mixture of the Ti-6Al-4V powder and graphene powder. The 0.5 wt% graphene was wet blended with Ti-6Al-4V alloy powder in ultrasonic bath for 5 hour. This facility was available at Thapar Institute of Engineering Technology, Patiala.



Figure 3.3: Oscar ultrasonicator (Courtesy: TIET, Patiala)

3.4.2 Spark Plasma Sintering Set Up

Spark plasma sintering technique was used for preparing the composite sample. In this method, pulsed DC current was passed through the Die (graphite) or conducting powder. Powder was heated internally below its melting point so that the internal bonds were formed due to diffusion. As the heat was passed internally, so the process is very fast as compared to conventional hot pressing technique. This facility was available at CSIR-National Physical Laboratory, Delhi. Diameter of graphite Die at their facility was 12.7 and 20 mm and heating rate of 50 - 350°C/minute. Load depends on the size of composite.



Figure 3.4: Spark Plasma Sintering Setup (Courtesy: CSIR-National Physical Laboratory, Delhi)

3.4.3 X-Ray Diffraction

X-Ray Diffraction analysis was done by using panalytical X-Pert Pro diffractometer ($\theta - 2\theta$) equipped with Cu-K α radiation ($\lambda = 1.5418 \text{ \AA}$). XRD was used for phase identification of material and give information about unit cell. This facility was available at Thapar Institute of Engineering Technology, Patiala.



Figure 3.5: X-RAY Diffraction (Courtesy: SAI Lab, TIET, Patiala)

3.4.4 Micro hardness tester

Micro hardness was measured to determine the penetration resistance of the composite. Measurements were done on a Computer Interfaced Micro Hardness Tester (Model: MVH2), available at Thapar Institute of Engineering Technology, Patiala. The micro hardness measurement is dependent on the diameter of indentation produced on the samples. Indentation was done with a diamond cone indenter at a constant load of 100gm/mm² for a dwell period of 20 sec. Quantimet software with 40 MP cameras was used for focusing images.



Figure 3.6: Micro hardness tester Model: MVH2 (Courtesy: Advanced Measurement Lab, TIET, Patiala)

3.4.5 Surface Roughness Tester

Surface roughness was measured using the Mitutoyo model SJ-400, Germany. The equipment uses the stylus method of measurement, has profile resolution of 12 nm and measure roughness up to 100 μm .

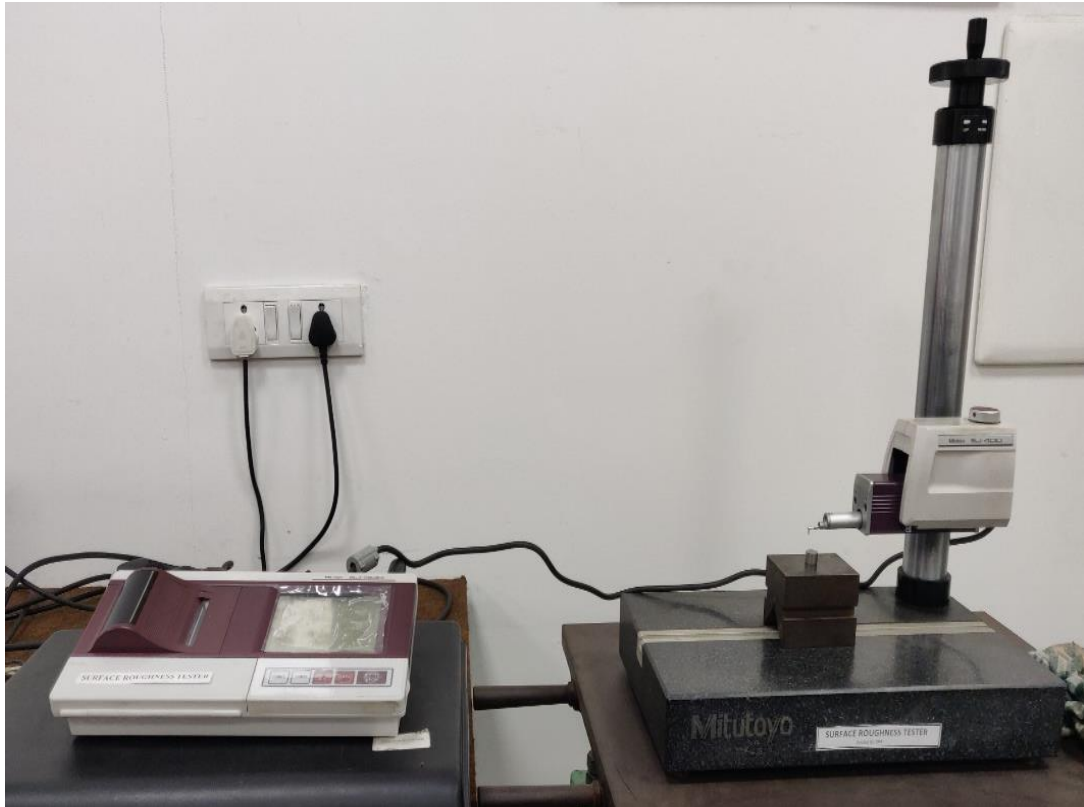


Figure 3.7: Surface Roughness Tester SJ-400 (Courtesy: Metrology lab TIET, Patiala)

3.4.6 Scanning Electron Microscope

In SEM, high energy electron focus beam was used for generating different variety signals. This gives the information about sample morphology, chemical composition, and crystalline structure. The SEM used for this study is a highly accurate and precise instrument (Make: JSM-6510LV, JEOL Ltd, Tokyo, Japan) for fast characterization and imaging of fine structures. It has magnification range from 5–300,000 X (printed as a 128 mm x 96 mm micrograph). This facility is available at SAI Labs, TIET, Patiala.



Figure 3.8: Scanning Electron Microscope (Courtesy: SAI Lab, TIET, Patiala)

CHAPTER 4

RESULTS AND DISCUSSION

4.1 DENSIFICATION

Theoretical density

Density of Ti-6Al-4V alloy - 4.429 g/cm³

Density of graphene - 2.267 g/cm³

Density of composite made by 99.5% Ti-6Al-4V alloy and 0.5% graphene = 99.5% of 4.429 g/cm³ + 0.5% of 2.267 g/cm³ = 4.40685 + 0.01134 = 4.41819 g/cm³

Calculated density

Weight of composite - 8.716 g

Volume of composite - 2 cm³

Density of composite - 4.3581 g/cm³

Porosity of composite

$$P = \left[\frac{(\rho_t - \rho)}{\rho_t} \right] * 100\% = 1.36\%$$

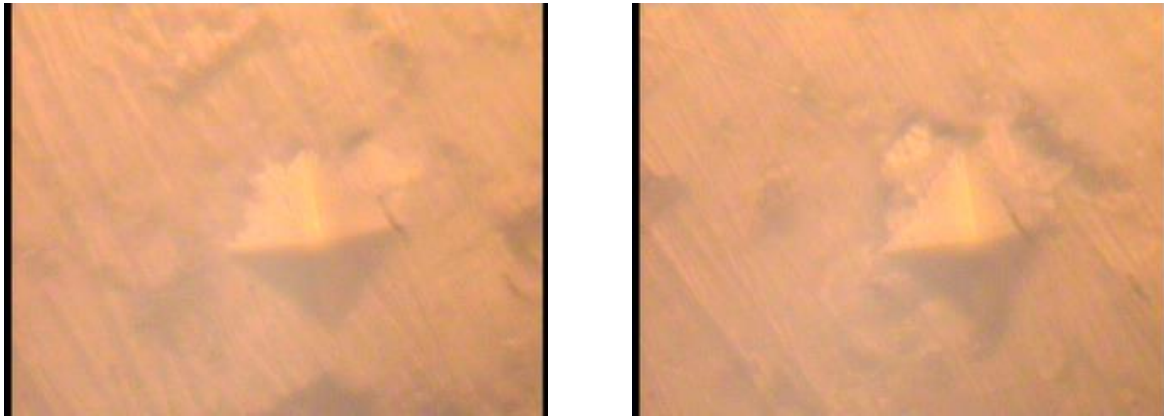
Where, ρ_t is theoretical density and ρ is density of composite.

Densification

$$\frac{\text{Calculated density}}{\text{Theoretical density}} = \frac{4.3581}{4.41819} = 0.98639 = 98.64 \%$$

It was found that after reinforcing graphene into Ti-6Al-4V alloy, the density of composite was reduced. It was also found that the composite formed was highly dense. Therefore, it was observed that the spark plasma sintering technique is very good technique for making the composite.

4.2 RESULT OF MICROHARDNESS



a) Indentation 1

b) Indentation 2

Figure 4.1: Indentation for measuring microhardness

Table 4.1: Microhardness of composite

Microhardness	Indentation 1	Indentation 2
Vickers hardness value	968.8289	978.1223

Microhardness of the composite was measured on Micro hardness testing machine Model: MVH2. The average Vickers hardness values measured of the Ti6Al4V-graphene composite was 973.4756 VH and the theoretical Vickers hardness value of Ti6Al4V alloy was 396 VH. Therefore it was found that there was 245% increase in microhardness after adding graphene to the Ti6Al4V alloy.

4.3 RESULT OF SURFACE ROUGHNESS

Table 4.2: Surface roughness value of composite

Parameter	Reading 1	Reading 2	Reading 3
Ra(μm)	1.64	1.25	1.44
Rz(μm)	9.8	8.1	8.0
Rq(μm)	2.04	1.63	1.75

Where, Ra - Roughness average, Rz - average maximum height of the peak and Rq - Root mean square roughness.

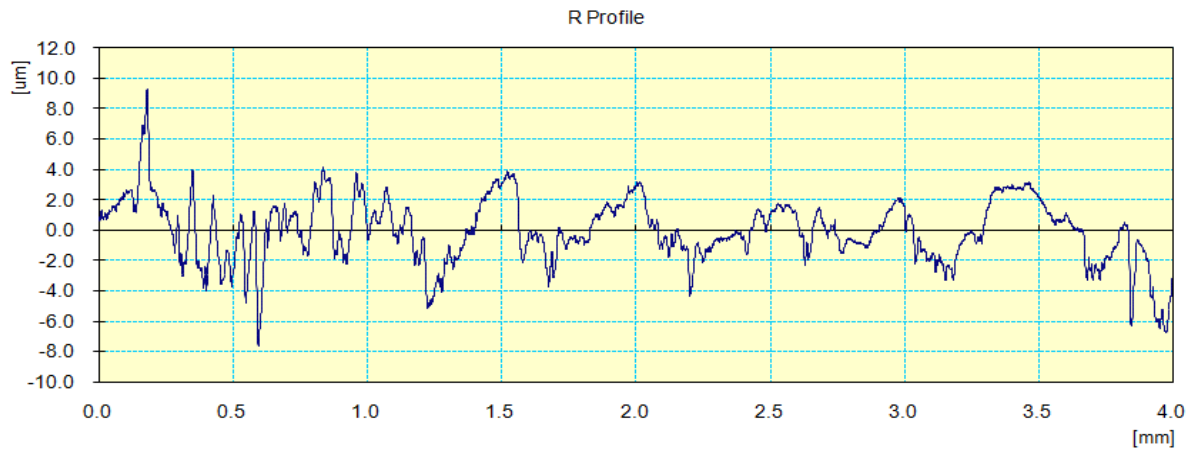


Figure 4.2: Roughness profile for reading 1

Three readings were recorded for the composite sample. The average measured surface roughness for the composite was found to be 1.44 μ m. From that it was concluded that the spark plasma sintering technique produces good surface finish.

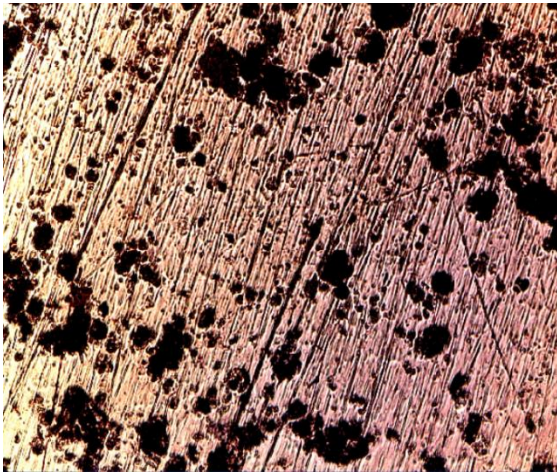
4.4 RESULTS FOR XRD

XRD analysis was done to study crystallographic deformation in composite. Six peaks were taken from the result to analyze the sample. Ti6Al4V-Graphene composite sintered at 1100 $^{\circ}$ C.

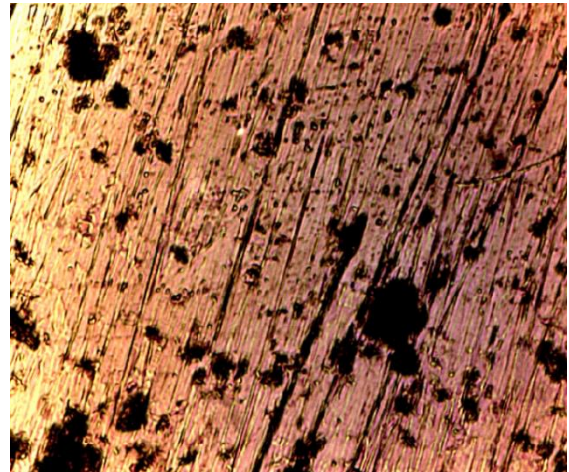
Table 4.3: Crystallite size of Ti6Al4V-Graphene composite investigated at different machining conditions from XRD

	Position 2 θ (deg)	Height (counts)	FWHM 2 θ (deg)	Area (counts*2 θ)	Integral breadth (area/height) β_{exp} (degree)	Crystallite size (nm)
1	38.7912	4881.3	0.2558	1847.87	0.378	40
2	42.085	3562.85	0.1468	333.48	0.093	
3	63.3468	523.24	0.2303	178.27	0.340	
4	78.5202	1161.13	0.1092	253.59	0.218	
5	81.6191	439.57	0.307	199.68	0.454	
6	93.332	181.89	0.3744	136.2	0.748	

4.5 RESULTS FOR OPTICAL MICROSCOPE



(a)



(b)

Figure 4.3: Optical image of composite (a) x100 (b) x200

Figure 4.4 (a) and (b) shows the optical images of Ti6Al4V-graphene composite sintered at 1100°C. To investigate microstructure of the sintered composite SEM observation were done at high magnification.

4.6 RESULTS FOR SEM

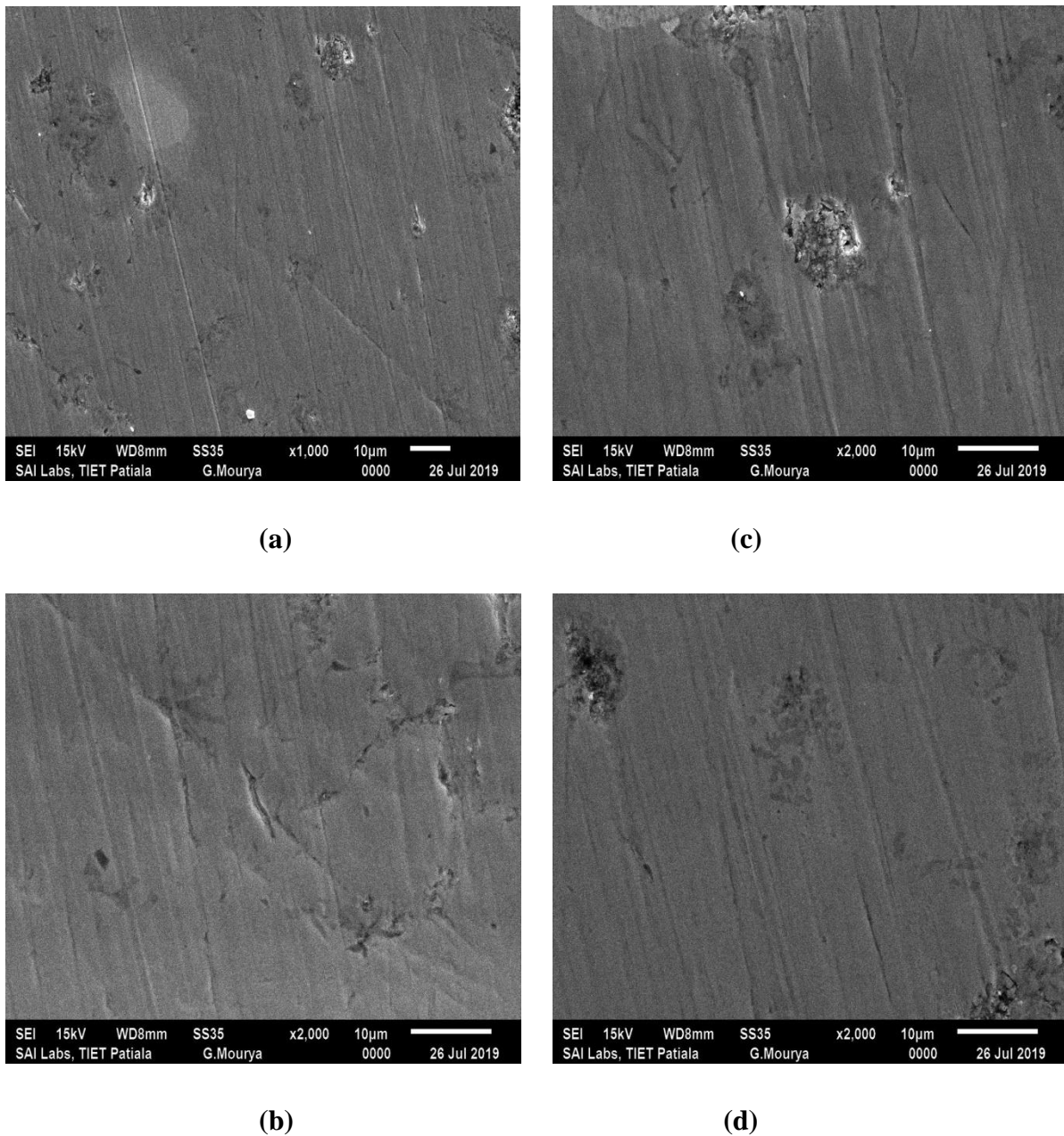


Figure 4.3: SEM images of Ti6Al4V-graphene composite sintered at 1100°C through SPS process (a) x1000 (b) x2000 (c) x2000 (d) x2000

The SEM images of the Ti6Al4V-graphene composite sintered at 1100°C were showed in Figure 4.3 (a), (b), (c) and (d). From the figure 4.3 (a) it was observed that there were very fine and small cracks on the surface of prepared samples. No porosity was observed. It was also seen that there was a fine cluster of some metallic compound. Then from figure 4.3(b) no crack was visualize on the surface. Small porosity was observed. The surface obtained was very smooth and no blow hole or pin hole formation was observed. From figure 4.3(c) it was concluded that there was formation of cluster

of some oxides and carbides. These salts were visualized in form white molten layer deposited on the surface. From figure 4.3(d) some black spots were visualized on the surface of prepared sample this may be due to burning of some ingot elements present in the sample. However no measure cracks were seen on the surface of sample. No major porosity was found on the surface of samples.

CHAPTER 5

CONCLUSIONS

On the basis of work done on Ti6Al4V-Graphene composite mechanical and microstructural properties were studied and following conclusions were made:

1. Ti6Al4V-graphene composite was fabricated successfully by using spark plasma sintering technique.
2. Ti6Al4V with 0.5 weight percent graphene sintered at 1100°C fabricated successfully.
3. It was concluded that the SPS technique is very good for producing highly dense composite with very less porosity
4. It was concluded that the hardness of the composite increased significantly by adding graphene when compared to Ti6Al4V alloy.
5. SPS technique produces good surface finish product.

5.1 FUTURE SCOPE OF WORK

1. A lot of work can be done by changing the reinforcement and matrix material.
2. A lot of work can be done on same reinforcement and matrix material by using different fabrication techniques and by varying different process parameters.

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