A Study on the Effects of Laser Glazing on Metal Matrix Composites Fabricated with Carbide-Boride-Nitride Reinforcements

M.Tech. Thesis

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A Study on the Effects of Laser Glazing on Metal Matrix Composites Fabricated with Carbide-Boride-Nitride Reinforcements

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DISCIPLINE OF METALLURGY ENGINEERING & MATERIALS SCIENCE INDIAN INSTITUTE OF TECHNOLOGY INDORE



INDIAN INSTITUTE OF TECHNOLOGY INDORE

CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled **A** Study on the Effects of Laser Glazing on Metal Matrix Composites Fabricated with Carbide-Boride-Nitride Reinforcements in the partial fulfillment of the requirements for the award of the degree of Master of Technology and submitted in the Metallurgy Engineering and Materials science, Indian Institute of Technology Indore, is an authentic record of my own work carried out during the time period from July 2015 to July 2017. Thesis submission under the supervision of Dr. Satyajit Chatterjee, assistant professor, Discipline of Mechanical Engineering. The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other institute.

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This is to certify that the above statement made by the candidate is correct to the best of my/our knowledge.

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ABSTRACT

Metal matrix composites (MMCs), offers high strength, fracture toughness and stiffness than those offered by their polymer counterparts. They can withstand elevated temperature in corrosive environment than polymer composites. Most metals and alloys could be used as matrices and they require reinforcement such as carbides, nitrides and borides etc. materials which need to be stable over a range of temperature and non-reactive too. Titanium, aluminium and magnesium are the popular matrix metals currently in vogue, which are particularly useful for aircraft applications. MMCs used in structural applications are frequently subjected to the corrosive environment, direct normal stresses and shear stresses and this can adversely affect the surface of the MMC may lead to high wear and fracture at the surface, so in order to avoid this we aim to laser glazing on the MMC surface.

The effect of laser glazing on metal matrix composites comprised of steel AISI304 as a metal matrix and TiB₂-TiN-SiC as a reinforcement prepared through powder metallurgical process are studied. Various process parameters are selected to fabricate the MMCs as three weight percentage of blended ceramic mixtures 20 wt.%, 30 wt.% and 40 wt.% along with three soaking times as 0 min, 30 min and 60 min and three compaction pressures 740 MPa, 960 MPa and 1180 MPa. Laser glazing is performed by the help of high power continuous wavelength laser on sintered MMC samples. The evaluation of COF, specific wear rate, fracture toughness and scratch hardness are carried out with the help of linear reciprocating tribometre and scratch tester. SEM micrograph analysis and XRD analysis is also carried out for physical characterization.

By examining the graphs of tribo-mechanical properties of sintered MMCs and laser glazed MMCs, it is observed that the increase in weight percentage of ceramic reinforcement in metal matrix, increase in soaking time and increase in compaction pressure up to an optimum value as 30 wt.% of ceramics content, 30 min. soaking time and 960 MPa compaction pressure the values of COF increases, while decrease in values of specific wear rate and also scratch hardness and fracture toughness values are found to be optimum at the optimum values of process parameters. While improvement in the tribo-mechanical properties are also observed after the laser glazing on the surface of MMCs.

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ACRONYMS

PMCs	:	Polymer Matrix Composites
MMCs	:	Metal Matrix Composites
CMCs	:	Ceramic Matrix Composites
PM	:	Powder Metallurgy
CIP	:	Cold Isostatic Pressing
EDS	:	Energy Dispersive Spectroscopy
FE-SEM	:	Field Emission Scanning Electron Microscope
XRD	:	X-Ray Diffraction
COF	:	Coefficient of Friction

Chapter 1

INTRODUCTION

1.1 Surface engineering

The surface engineering is a multidisciplinary activity intended to tailor the properties of the surfaces of engineering components so that their functions and serviceability can be improved. The surface engineering defines as "treatment of the surface and near-surface regions of a material to allow the surface to perform functions that are distinct from those functions demanded from the bulk of the material" [1]. The desired properties or characteristics of surface-engineered components include:

- Improved corrosion resistance through barrier or sacrificial protection
- Improved oxidation and/or sulfidation resistance
- Improved wear resistance
- Reduced frictional energy losses
- Improved mechanical properties, example enhanced fatigue or toughness
- Improved electronic or electrical properties
- Improved thermal insulation
- Improved aesthetic appearance

These properties can be achieved metallurgically, mechanically, chemically, or by adding a coating.

The bulk of the material or substrate cannot be considered totally independent of the surface treatment. Most surface processes are not limited to the immediate region of the surface, but can involve the substrate by exposure to either a thermal cycle or a mechanical stress. For example, diffusion heat treatment coatings (e.g., carburizing/nitriding) often have high-temperature thermal cycles that may subject the substrate to temperatures that cause phase transformations and thus property changes, or shot-peening treatments that deliberately strain the substrate surface to induce improved fatigue properties. It is the purpose of this subject to review information on surface treatments that improve service performance so that metallurgists, chemists, mechanical engineers, and design engineers may consider surface-engineered components as an alternative to costlier materials [1].

1.2 Surface engineering options and property benefits

1.2.1 Changing the surface metallurgy

There are various techniques to improve the surface properties by changing the surface metallurgy:

- (a) Localized surface hardening (flame, induction, laser and laser and electron beam hardening): It improves the wear resistance through the development of a hard martensitic surface [2].
- (b) Laser melting or laser glazing: It improves wear resistances through grain refinement and the formation of fine dispersion of precipitates on the surface [2].
- (c) Shot peening: Improved fatigue strength due to compressive stresses induced on the exposed surface, also relieves tensile stresses that contribute to stress-corrosion cracking.

1.2.2 Changing the surface chemistry

Surface properties can be enhanced by changing the surface chemistry also, these technique includes:

- (a) Phosphate chemical conversion coatings: Used primarily on steels for enhanced corrosion resistance, increased plating or paint adhesion, and for lubricity (e.g., to increase the formability of sheet metals).
- (b) Chromate chemical conversion coatings: Enhanced bare or painted corrosion resistance, improved adhesion of paint or other organic finishes, and provides the metallic surface with a decorative finish.

- (c) Black oxide chemical conversion coatings: Used for decorative applications, e.g., the "bluing" on steel gun barrels.
- (d) Anodizing (electrochemical conversion coating): Used primarily for aluminum for increased corrosion resistance, improved decorative appearance, increased abrasion resistance (hard anodizing), improved paint adhesion, and improved adhesive bonding (higher bond strength and durability).
- (e) **Carburizing:** Used primarily for steels for increased resistance to wear, bending fatigue, and rolling-contact fatigue.
- (f) Nitriding: Used primarily for steels for improved wear resistance, increased fatigue resistance, and improved corrosion resistance (except stainless steels) [1, 2].

1.2.3 Adding a surface layer or coating

Surface properties can also be achieved by depositing a thin or thick film coating on parent material. The following techniques and their effects are as follows;

- (a) Organic coatings (paints and polymeric or elastomeric coatings and linings): Improved corrosion resistance, wear resistance, and aesthetic appearance.
- (b) Ceramic coatings (glass linings, cement linings, and porcelain enamels): Improved corrosion resistance.
- (c) Slip/sinter ceramic coatings: Improved wear resistance and heat resistance.
- (d) Hot-dip galvanizing (zinc coatings): Improved corrosion resistance via sacrificial protection of steel substrate.
- (e) Electroplating: Depending on the metal or metals being electrodeposited, improved corrosion resistance (e.g., nickel-chromium multilayer coatings, and cadmium and zinc sacrificial coatings), wear resistance (e.g., hard chromium coatings), electrical properties (e.g., copper and silver), and aesthetic appearance (e.g., bright nickel or decorative chromium plating).

- (f) Thermal spraying: Primarily used for improved wear resistance (many coating systems including ceramics and cermets), but also used for improved corrosion resistance (aluminum, zinc, and their alloys) and oxidation resistance (e.g., MCrAlY), thermal barrier protection (partially stabilized zirconia), electrically conductive coatings (e.g., copper and silver), and dimensional restoration.
- (g) Cladding (roll bonding, explosive bonding, hot isostatic pressing, etc.): Improved corrosion resistance.
- (a) Laser cladding: Improved wear resistance.
- (b) Carbide (salt bath) diffusion: Used primarily for steels for improved wear resistance in tooling applications.
- (c) Chemical vapor deposition (CVD): Improved wear (e.g., tools and dies), erosion, and corrosion resistance; also used for epitaxial growth of semiconductors.
- (d) Physical vapor deposition (PVD): Improved wear (e.g., tools and dies) and corrosion resistance, improved optical and electronic properties, and decorative applications [1, 2].

1.3 Composites: introductions, classifications, and applications

A composite material is a combination of two or more chemically different materials with a distinct interface between them. The constituent maintains their separate identities (at least microscopically) in the composite, yet their combination produces properties and characteristics that are different from those of the constituents. One of these constituents forms a continuous phase and is called the matrix. The other major constituent is reinforcement in the form of fibres or particulates that is, in general, added to the matrix to improve or alter the matrix properties. The reinforcement forms a discontinuous phase that is dispersed uniformly throughout the matrix. The reinforcement surface may be chemically treated or coated with a very thin layer to improve wetting of the reinforcement by the matrix as well as to control or enhance interfacial bonding between the reinforcement and the matrix. The surface treatment may also serve to protect the reinforcement surface from degradation by environmental attack, such as by moisture and chemicals, or by adverse chemical reaction with the matrix at high temperature.

The matrix material in the composite may be a polymer, a metal or a ceramic. Depending upon the matrix used, composite materials are classified as ceramic matrix composites (CMCs), metal matrix composites (MMCs), or polymer matrix composites (PMCs). The majority of the composites used commercially are based on polymer matrices; however, both MMCs CMCs are attracting great interest in high temperature applications. Another class of composite is based on a cement matrix. Because of their importance in civil engineering structures, considerable effort is being made to develop cement matrix composites with high resistance to cracking. [1, 2]

1.3.1 Advantages of composites:

- Light weight
- High specific stiffness and strength
- Easy moldable to complex forms
- ➢ Easy bondable
- Good dumping
- Low electrical conductivity and thermal expansion
- ➤ Good fatigue resistance
- Part consolidation due to lower overall system costs
- Internal energy storage and release

1.3.2 Disadvantages of composites:

- Cost of materials
- Long development time
- Difficulty manufacturing
- ➤ Low ductility

- ➢ Temperature limits
- Solvent or moister attack
- Hidden damages and damage susceptibility.

1.3.3 Concept of composite:

Fibers or particles embedded in matrix of another material are the best example of modern-day composite materials, which are mostly structural.

- Laminates are composite material where different layers of materials give them the specific character of a composite material having a specific function to perform. Fabrics have no matrix to fall back on, but in them, fibers of different compositions combine to give them a specific character. Reinforcing materials generally withstand maximum load and serve the desirable properties.
- Further, though composite types are often distinguishable from one another, no clear determination can be really made. To facilitate definition, the accent is often shifted to the levels at which differentiation take place viz., microscopic or macroscopic.
- In matrix-based structural composites, the matrix serves two paramount purposes viz., binding the reinforcement phases in place and deforming to distribute the stresses among the constituent reinforcement materials under an applied force.
- The demands on matrices are many. They may need to temperature variations, be conductors or resistors of electricity, have moisture sensitivity etc. This may offer weight advantages, ease of handling and other merits which may also become applicable depending on the purpose for which matrices are chosen.
- Solids that accommodate stress to incorporate other constituents provide strong bonds for the reinforcing phase are potential matrix materials. A few inorganic materials, polymers and metals have found applications as matrix

materials in the designing of structural composites, with commendable success. These materials remain elastic till failure occurs and show decreased failure strain, when loaded in tension and compression.

Composites cannot be made from constituents with divergent linear expansion characteristics. The interface is the area of contact between the reinforcement and the matrix materials. In some cases, the region is a distinct added phase. Whenever there is interphase, there has to be two interphases between each side of the interphase and its adjoin constituent. Some composites provide interphases when surfaces dissimilar constituents interact with each other. Choice of fabrication method depends on matrix properties and the effect of matrix on properties of reinforcements. One of the prime considerations in the selection and fabrication of composites is that the constituents should be chemically inert non-reactive. Figure 1.1 helps to classify matrices [1-4].



Fig.1.1 Classification of matrix materials [1].

1.3.3.1 Classification of composites

Composite materials are commonly classified at following two distinct levels:

• The first level of classification is usually made with respect to the matrix constituent. The major composite classes include Organic Matrix Composites (OMCs), Metal Matrix Composites (MMCs) and Ceramic Matrix Composites

(CMCs). The term organic matrix composite is generally assumed to include two classes of composites, namely Polymer Matrix Composites (PMCs) and carbon matrix composites commonly referred to as carbon-carbon composites.

- The second level of classification refers to the reinforcement form fiber reinforced composites, laminar composites and particulate composites. Fiber Reinforced composites (FRP) can be further divided into those containing discontinuous or continuous fibers.
- Fiber reinforced composites are composed of fibers embedded in matrix material. Such a composite is considered to be a discontinuous fiber or short fiber composite if its properties vary with fiber length. On the other hand, when the length of the fiber is such that any further increase in length does not further increase, the elastic modulus of the composite, the composite is considered to be continuous fiber reinforced. Fibers are small in diameter and when pushed axially, they bend easily although they have very good tensile properties. These fibers must be supported to keep individual fibers from bending and buckling.
- Laminar composites are composed of layers of materials held together by matrix. Sandwich structures fall under this category.
- Particulate composites are composed of particles distributed or embedded in matrix body. The particles may be flakes or in powder form. Concrete and wood particle boards are examples of this category [1, 4-6].

1.3.3.1.1 Organic matrix composites

Polymer Matrix Composites (PMC)/Carbon Matrix Composites or Carbon-Carbon Composites Polymers make ideal materials as they can be processed easily, possess lightweight, and desirable mechanical properties. It follows, therefore, that high temperature resins are extensively used in aeronautical applications.

Two main kinds of polymers are thermosets and thermoplastics. Thermosets have qualities such as a well-bonded three-dimensional molecular structure after curing. They decompose instead of melting on hardening. Merely changing the basic composition of the resin is enough to alter the conditions suitably for curing and determine its other characteristics. They can be retained in a partially cured condition too over prolonged periods of time, rendering Thermosets very flexible. Thus, they are most suited as matrix bases for advanced conditions fiber reinforced composites. Thermosets find wide ranging applications in the chopped fiber composites form particularly when a premixed or moulding compound with fibers of specific quality and aspect ratio happens to be starting material as in epoxy, polymer and phenolic polyamide resins.

Thermoplastics have one- or two-dimensional molecular structure and they tend to at an elevated temperature and show exaggerated melting point. Another advantage is that the process of softening at elevated temperatures can reversed to regain its properties during cooling, facilitating applications of conventional compress techniques to mould the compounds.

Resins reinforced with thermoplastics now comprised an emerging group of composites. The theme of most experiments in this area to improve the base properties of the resins and extract the greatest functional advantages from them in new avenues, including attempts to replace metals in die-casting processes. In crystalline thermoplastics, the reinforcement affects the morphology to a considerable extent, prompting the reinforcement to empower nucleation. Whenever crystalline or amorphous, these resins possess the facility to alter their creep over an extensive range of temperature. But this range includes the point at which the usage of resins is constrained, and the reinforcement in such systems can increase the failure load as well as creep resistance. Figure 1.2 shows classification of thermoplastics.

A small quantum of shrinkage and the tendency of the shape to retain its original form are also to be accounted for. But reinforcements can change this condition too. The advantage of thermoplastics systems over thermosets are that there are no chemical reactions involved, which often result in the release of gases or heat. Manufacturing is limited by the time required for heating, shaping and cooling the structures [1, 7].



Fig. 1.2 Classification of thermoplastics [7].

Thermoplastics resins are sold as molding compounds. Fiber reinforcement is apt for these resins. Since the fibers are randomly dispersed, the reinforcement will be almost isotropic. However, when subjected to molding processes, they can be aligned directionally.

There are a few options to increase heat resistance in thermoplastics. Addition of fillers raises the heat resistance. But all thermoplastic composites tend loose their strength at elevated temperatures. However, their redeeming qualities like rigidity, toughness and ability to repudiate creep, place thermoplastics in the important composite materials bracket. They are used in automotive control panels, electronic products encasement etc.

Newer developments augur the broadening of the scope of applications of thermoplastics. Huge sheets of reinforced thermoplastics are now available and they only require sampling and heating to be molded into the required shapes. This has facilitated easy fabrication of bulky components, doing away with the more cumbersome molding compounds.

Thermosets are the most popular of the fiber composite matrices without which, research and development in structural engineering field could get truncated. Aerospace components, automobile parts, defense systems etc., use a great deal of this type of fiber composites. Epoxy matrix materials are used in printed circuit boards and similar areas. Fig.1.3 shows the classification of thermoset material.



Fig. 1.3 Classification of thermoset Materials [7].

Direct condensation polymerization followed by rearrangement reactions to form heterocyclic entities is the method generally used to produce thermoset resins. Water, a product of the reaction, in both methods, hinders production of void-free composites. These voids have a negative effect on properties of the composites in terms of strength and dielectric properties. Polyesters phenolic and Epoxies are the two important classes of thermoset resins.

Epoxy resins are widely used in filament-wound composites and are suitable for molding prepress. They are reasonably stable to chemical attacks and are excellent adherents having slow shrinkage during curing and no emission of volatile gases. These advantages, however, make the use of epoxies rather expensive. Also, they cannot be expected beyond a temperature of 140°C. Their use in high technology areas where service temperatures are higher, as a result, is ruled out.

Polyester resins on the other hand are quite easily accessible, cheap and find use in a wide range of fields. Liquid polyesters are stored at room temperature for months, sometimes for years and the mere addition of a catalyst can cure the matrix material within a short time. They are used in automobile and structural applications.

The cured polyester is usually rigid or flexible as the case may be and transparent. Polyesters withstand the variations of environment and stable against chemicals. Depending on the formulation of the resin or service requirement of application, they can be used up to about 75°C or higher. Other advantages of

polyesters include easy compatibility with few glass fibers and can be used with verify of reinforced plastic accoutrey.

Aromatic Polyamides are the most sought after candidates as the matrices of advanced fiber composites for structural applications demanding long duration exposure for continuous service at around 200-250°C [1, 7].

1.3.3.1.2 Metal matrix composites (MMC)

Metal matrix composites, at present though generating a wide interest in research fraternity, are not as widely in use as their plastic counterparts. High strength, fracture toughness and stiffness are offered by metal matrices than those offered by their polymer counterparts. They can withstand elevated temperature in corrosive environment than polymer composites. Most metals and alloys could be used as matrices and they require reinforcement materials which need to be stable over a range of temperature and non-reactive too. However, the guiding aspect for the choice depends essentially on the matrix material. Light metals form the matrix for temperature application and the reinforcements in addition to the aforementioned reasons are characterized by high moduli.

Most metals and alloys make good matrices. However, practically, the choices for low temperature applications are not many. Only light metals are responsive, with their low density proving an advantage. titanium, aluminium and magnesium are the popular matrix metals currently in vogue, which are particularly useful for aircraft applications. If metallic matrix materials have to offer high strength, they require high modulus reinforcements. The strength-to-weight ratios of resulting composites can be higher than most alloys.

The melting point, physical and mechanical properties of the composite at various temperatures determine the service temperature of composites. Most metals, ceramics and compounds can be used with matrices of low melting point alloys. The choice of reinforcements becomes more stunted with increase in the melting temperature of matrix materials [1, 8].

1.3.3.1.3 Ceramic matrix materials (CMM)

Ceramics can be described as solid materials which exhibit very strong ionic bonding in general and in few cases covalent bonding. High melting points, good corrosion resistance, stability at elevated temperatures and high compressive strength, render ceramic-based matrix materials a favourite for applications requiring a structural material that doesn't give way at temperatures above 1500°C. Naturally, ceramic matrices are the obvious choice for high temperature applications.

High modulus of elasticity and low tensile strain those most ceramics possess, have combined to cause the failure of attempts to add reinforcements to obtain strength improvement. This is because at the stress levels at which ceramics rupture, there is insufficient elongation of the matrix which keeps composite from transferring an effective quantum of load to the reinforcement and the composite may fail unless the percentage of fiber volume is high enough. A material is reinforcement to utilize the higher tensile strength of the fiber, to produce an increase in load bearing capacity of the matrix. Addition of high-strength fiber to a weaker ceramic has not always been successful and often the resultant composite has proved to be weaker.

The use of reinforcement with high modulus of elasticity may take care of the problem to some extent and presents pre-stressing of the fiber in the ceramic matrix is being increasingly resorted to as an option.

When ceramics have a higher thermal expansion coefficient than reinforcement materials, the resultant composite is unlikely to have a superior level of strength. In that case, the composite will develop strength within ceramic at the time of cooling resulting in micro cracks extending from fiber to fiber within the matrix. Micro cracking can result in a composite with tensile strength lower than that of the matrix [1, 9].

1.3.3.2 Classification based on reinforcements

Reinforcements for the composites can be fibers, fabrics particles or whiskers. Fibers are essentially characterized by one very long axis with other two axes either often circular or near circular. Particles have no preferred orientation and so does their shape. Whiskers have a preferred shape but are small both in diameter and length as compared to fibers. Fig.1.4 shows the classification based on reinforcements.



Fig. 1.4 Classifications based on reinforcements [10].

Reinforcing constituents in composites, as the word indicates, provide the strength that makes the composite what it is. But they also serve certain additional purposes of heat resistance or conduction, resistance to corrosion and provide rigidity. Reinforcement can be made to perform all or one of these functions as per the requirements.

A reinforcement that embellishes the matrix strength must be stronger and stiffer than the matrix and capable of changing failure mechanism to the advantage of the composite. This means that the ductility should be minimal or even nil the composite must behave as brittle as possible.

1.3.3.2.1 Fiber reinforced composites/fiber reinforced polymer (FRP) composites

Fibers are the important class of reinforcements, as they satisfy the desired conditions and transfer strength to the matrix constituent influencing and enhancing their properties as desired.

Glass fibers are the earliest known fibers used to reinforce materials. Ceramic and metal fibers were subsequently found out and put to extensive use, to render composites stiffer more resistant to heat.

Fibers fall short of ideal performance due to several factors. The performance of a fiber composite is judged by its length, shape, orientation, and composition of the fibers and the mechanical properties of the matrix.

The orientation of the fiber in the matrix is an indication of the strength of the composite and the strength is greatest along the longitudinal directional of fiber. This doesn't mean the longitudinal fibers can take the same quantum of load irrespective of the direction in which it is applied. Optimum performance from longitudinal fibers can be obtained if the load is applied along its direction. The slightest shift in the angle of loading may drastically reduce the strength of the composite.

Unidirectional loading is found in few structures and hence it is prudent to give a mix of orientations for fibers in composites particularly where the load is expected to be the heaviest.

Monolayer tapes consisting of continuous or discontinuous fibers can be oriented unidirectional stacked into plies containing layers of filaments also oriented in the same direction. More complicated orientations are possible too and nowadays, computers are used to make projections of such variations to suit specific needs. In short, in planar composites, strength can be changed from unidirectional fiber oriented composites that result in composites with nearly isotropic properties [1, 10]. Properties of angle-plied composites which are not quasi-isotropic may vary with the number of plies and their orientations. Composite variables in such composites are assumed to have a constant ratio and the matrices are considered relatively weaker than the fibers. The strength of the fiber in any one of the three axes would, therefore be one-third the unidirectional fiber composite, assuming that the volume percentage is equal in all three axes.

However, orientation of short fibers by different methods is also possible like random orientations by sprinkling on to given plane or addition of matrix in liquid or solid state before or after the fiber deposition. Even three-dimensional orientations can achieve in this way.

There are several methods of random fiber orientations, which in a twodimensional one, yield composites with one-third the strength of a unidirectional fiber-stressed composite, in the direction of fibers. In a 3-dimension, it would result in a composite with a comparable ratio, about less than one-fifth.

In very strong matrices, moduli and strengths have not been observed. Application of the strength of the composites with such matrices and several orientations is also possible. The longitudinal strength can be calculated on the basis of the assumption that fibers have been reduced to their effective strength on approximation value in composites with strong matrices and non-longitudinally orientated fibers.

It goes without saying that fiber composites may be constructed with either continuous or short fibers. Experience has shown that continuous fibers (or filaments) exhibit better orientation, although it does not reflect in their performance. Fibers have a high aspect ratio, i.e., their lengths being several times greater than their effective diameters. This is the reason why filaments are manufactured using continuous process. This finished filaments.

Mass production of filaments is well known and they match with several matrices in different ways like winding, twisting, weaving and knitting, which exhibit the characteristics of a fabric [1, 10].

Since they have low densities and high strengths, the fiber lengths in filaments or other fibers yield considerable influence on the mechanical properties as well as the response of composites to processing and procedures. Shorter fibers with proper orientation composites that use glass, ceramic or multi-purpose fibers can be endowed with considerably higher strength than those that use continuous fibers. Short fibers are also known to their theoretical strength. The continuous fiber constituent of a composite is often joined by the filament winding process in which the matrix impregnated fiber wrapped around a mandrel shaped like the part over which the composite is to be placed, and equitable load distribution and favorable orientation of the fiber is possible in the finished product. However, winding is mostly confined to fabrication of bodies of revolution and the occasional irregular, flat surface.

Short-length fibers incorporated by the open- or close-mould process are found to be less efficient, although the input costs are considerably lower than filament winding.

Most fibers in use currently are solids which are easy to produce and handle, having a circular cross-section, although a few non-conventional shaped and hollow fibers show signs of capabilities that can improve the mechanical qualities of the composites.

Given the fact that the vast difference in length and effective diameter of the fiber are assets to a fiber composite, it follows that greater strength in the fiber can be achieved by smaller diameters due to minimization or total elimination of surface of surface defects.

After flat-thin filaments came into vogue, fibers rectangular cross sections have provided new options for applications in high strength structures. Owing to their shapes, these fibers provide perfect packing, while hollow fibers show better structural efficiency in composites that are desired for their stiffness and compressive strengths. In hollow fibers, the transverse compressive strength is lower than that of a solid fiber composite whenever the hollow portion is more than half the total fiber diameter. However, they are not easy to handle and fabricate [1, 10].

1.3.3.2.2 Laminar Composites

Laminar composites are found in as many combinations as the number of materials. They can be described as materials comprising of layers of materials bonded together. These may be of several layers of two or more metal materials occurring alternately or in a determined order more than once, and in as many numbers as required for a specific purpose.

Clad and sandwich laminates have many areas as it ought to be, although they are known to follow the rule of mixtures from the modulus and strength point of view. Other intrinsic values pertaining to metal-matrix, metal-reinforced composites are also fairly well known.

Powder metallurgical processes like roll bonding, hot pressing, diffusion bonding, brazing and so on can be employed for the fabrication of different alloys of sheet, foil, powder or sprayed materials. It is not possible to achieve high strength materials unlike the fiber version. But sheets and foils can be made isotropic in two dimensions more easily than fibers. Foils and sheets are also made to exhibit high percentages of which they are put. For instance, a strong sheet may use over 92% in laminar structure, while it is difficult to make fibers of such compositions. Fiber laminates cannot over 75% strong fibers.

The functional types of metal-metal laminates that do not possess high strength or stiffness are single layered ones that endow the composites with special properties, apart from being cost-effective. They are usually made by pre-coating or cladding methods.

Pre-coated metals are formed by forming by forming a layer on a substrate, in the form of a thin continuous film. This is achieved by hot dipping and occasionally by chemical plating and electroplating. Clad metals are found to be suitable for more intensive environments where denser faces are required. There are many combinations of sheet and foil which function as adhesives at low temperatures. Such materials, plastics or metals, may be clubbed together with a third constituent. Pre-painted or pre-finished metal whose primary advantage is elimination of final finishing by the user is the best known metal-organic laminate. Several combinations of metal-plastic, vinyl-metal laminates, organic films and metals, account for up to 95% of metal-plastic laminates known. They are made by adhesive bonding processes [1, 6-7, 11].

1.3.3.2.3 Particulate Reinforced Composites (PRC)

Microstructures of metal and ceramics composites, which show particles of one phase strewn in the other, are known as particle reinforced composites. Square, triangular and round shapes of reinforcement are known, but the dimensions of all their sides are observed to be more or less equal. The size and volume concentration of the dispersoid distinguishes it from dispersion hardened materials.

The dispersed size in particulate composites is of the order of a few microns and volume concentration is greater than 28%. The difference between particulate composite and dispersion strengthened ones is, thus, oblivious. The mechanism used to strengthen each of them is also different. The dispersed in the dispersionstrengthen materials reinforces the matrix alloy by arresting motion of dislocations and needs large forces to fracture the restriction created by dispersion.

In particulate composites, the particles strengthen the system by the hydrostatic coercion of fillers in matrices and by their hardness relative to the matrix. Three-dimensional reinforcement in composites offers isotropic properties, because of the three systematical orthogonal planes. Since it is not homogeneous, the material properties acquire sensitivity to the constituent properties, as well as the interfacial properties and geometric shapes of the array. The composite's strength usually depends on the diameter of the particles, the inter-particle spacing, and the volume fraction of the reinforcement. The matrix properties influence the behavior of particulate composite too [1, 12].

1.3.3.3 Common categories of composite materials based on fiber length:

Based on the form of reinforcement, common composite materials can be classified as follows:

- 1. Fibers as the reinforcement (Fibrous Composites):
 - a. Random fiber (short fiber) reinforced composites



Fig. 1.5 Short-fiber reinforced composites [13].

b. Continuous fiber (long fiber) reinforced composites



Fig. 1.6 Long-fiber reinforced composites [13].

2. Particles as the reinforcement (Particulate composites):



Fig. 1.7 Particulate composites [13]

3. Flat flakes as the reinforcement (Flake composites):



Fig. 1.8 Flake composites [13]

4. Fillers as the reinforcement (Filler composites):



Fig. 1.9 Filler composites [13].

1.4 Metal matrix composites

Metal matrix composite materials have found application in many areas of daily life for quite some time. The first MMCs were developed in the 1970s for high-performance applications using continuous fibers and whiskers for reinforcement [14] Metal matrix composites (MMCs) combine both metallic properties (ductility and toughness) with ceramic properties (high strength and modulus) possess greater strength in shear and compression and high service temperature capabilities. The extensive use of MMCs in aerospace, automotive industries and in structural applications has increased over past 20 years due to the availability of inexpensive reinforcements and cost effective processing routes which give rise to reproducible properties [15]. These materials are produced insitu from the conventional production and processing of metals. Materials like cast iron with graphite or steel with high carbide content, as well as tungsten carbides, consisting of carbides and metallic binders, also belong to this group of composite materials. For many researchers the term metal matrix composites is often equated with the term light metal matrix composites (MMCs). Substantial progress in the development of light metal matrix composites has been achieved in recent decades, so that they could be introduced into the most important applications. The advantages of MMCs over their monolithic counterparts are well documented and include superior specific strength, stiffness and wear resistance. In the 1970s and 1980s, there was an increase in research and development of MMCs due to their potential improvement at high temperatures. The latter part of the 20th century resulted in research which was not only performance driven, but also involved the economics of MMCs [16]. Partly due to economic benefits, research began to evaluate particulates as reinforcements for MMCs, including silicon carbide (SiC), aluminum oxide or alumina (A1₂O₃), titanium carbide (TiC), titanium diboride (TiB_2) and boron carbide (B_4C) . While not quite having the increased levels of specific strength and stiffness shown by their fiber-reinforced, particulate reinforced MMCs provided significant improvements in such properties when compared with the monolithic materials and their associated processing costs were I significantly less than those associated with continuous reinforcements. This

material group becomes interesting for use as constructional and functional materials, if the property profile of conventional materials either does not reach the increased standards of specific demands, or is the solution of the problem. However, the technology of MMCs is in competition with other modern material technologies, for example powder metallurgy. The advantages of the composite materials are only realized when there is a reasonable cost performance relationship in the component production. The use of a composite material is obligatory if a special property profile can only be achieved by application of these materials. The possibility of combining various material systems (metal-ceramic-non-metal) gives the opportunity for unlimited variation. The properties of these new materials are basically determined by the properties of their single components. The reinforcement of metals can have many different objectives. The reinforcement of light metals opens up the possibility of application of these materials in areas where weight reduction has first priority. The precondition here is the improvement of the component properties. The development objectives for light metal composite materials are

- Increase in yield strength and tensile strength at room temperature and above while maintaining the minimum ductility or rather toughness.
- Increase in creep resistance at higher temperatures compared to that of conventional alloys.
- > Increase in fatigue strength, especially at higher temperatures.
- Improvement of thermal shock resistance.
- Improvement of corrosion resistance.
- Increase in Young's modulus.
- Reduction of thermal elongation.
1.4.1 Classification of metal matrix composites

One classification is the consideration of type and contribution of reinforcement components in particle, layer, fiber and penetration composite materials [17]



Fig. 1.10 Flowchart of types of metal matrix composites [17].

Fiber composite materials can be further classified into continuous fiber composite materials (multi- and monofilament) and short fibers or, rather, whisker composite materials see Fig: 1.5.1(b) [18].



Fig. 1.11 Types of metal matrix composite [18].

1.4.2 Fabrication of metal matrix composite

In general, the most common technologies for manufacturing MMC are divided primarily into two main parts: primary and the secondary. The primary processing is the composite production by combining ingredient materials (powdered metal and loose ceramic particles, or molten metal and fiber preforms), but not necessarily to final shape or final microstructure and the secondary processing is the step that obviously follows primary processing and its aim is to alter the shape and microstructure of the material (shape casting, forging, extrusion, heat treatment, machining) [19-22]. Secondary processing can change the constituents (phase, shape) of the composite. So, MMCs can be made by different ways like

- Solid state processing
- Liquid state processing
- Vapor state processing
- Plasma /spray deposition
- In situ processing

1.4.2.1 Solid-state metallurgical processes

In solid state processing, the reinforcement is embedded in matrix through diffusion phenomena at high pressure and high temperature. It appears crucial monitoring of the diffusion phenomena to avoid the growth of undesirable phases or compounds species on interfaces. The various steps of processing are usually preceded by a "pre-processing" for the purpose of preparing the surface. The most common solid phases are based on powder metallurgy techniques. These typically involve discontinuous reinforcement, due to ease of mixing, blending and effectiveness of densification. The ceramic and metal powders are mixed isostatically, cold compacted and hot pressed to get full density. Then the fully dense compact typically undergoes a secondary operation such as extrusion, forging [23-26].

Solid state processing method is followed by three ways:

- 1. Diffusion bonding
- 2. Deformation processing
- 3. Powder metallurgy

1.4.2.1.1 Diffusion bonding

Diffusion bonding is a common solid-state processing technique for joining similar or dissimilar metals. Inter diffusion of atoms between clean metallic surfaces, in contact at an elevated temperature, leads to bonding. The principal advantages of this technique are the ability to process a wide variety of metal matrices and control of fiber orientation and volume fraction. There are many variants of the basic diffusion bonding process, although all of them involve simultaneous application of pressure and high temperature. Vacuum hot pressing is an important step in the diffusion bonding processes for metal matrix composites. Hot isostatic pressing (HIP), instead of uniaxial pressing, can also be used [27].



Fig. 1.12 Diffusion bonding process [27].

1.4.2.1.2 Deformation processing

Deformation processing can also be used to deform and/or densify the composite material in metal-metal composites. Mechanical processing (swaging, extrusion, drawing, or rolling) of a ductile two-phase material causes the two phases to co-deform, causing one of the phases to elongate and become fibrous in nature within the other phase. The properties of a deformation processed composite depend largely on the characteristics of the starting material, which is usually a billet of a two-phase alloy that has been prepared by casting or powder metallurgy methods. Roll bonding is a common technique used to produce a laminated composite consisting of different metals in layered form. Such composites are called sheet laminated metal-matrix composites.



Fig. 1.13 Roll bonding process of making a laminated MMC [27].

1.5 Powder metallurgy process

1.5.1 Introduction

Because of the difficulty in wetting ceramic particles with molten metal, the powder metallurgy route was the first method developed. Powder Metallurgy may be defined as the art of producing metal powders and using them to make serviceable objects. This method has gained popularity because of the high strength, ductility and toughness that can be obtained by this route. One of the outstanding uses of powder metallurgy is the combination of hard materials in a metallic matrix, which serves as the basis of cemented-carbide products. Moreover, powder metallurgy is more economical than most other manufacturing processes.

The Powder Metallurgy has several attractive features:

- 1. It allows essentially any alloy to be used as the matrix.
- It also allows any type of reinforcement to be used because the reaction between matrix and reinforcement can be minimized by using solid state processing.
- 3. Non-equilibrium alloys can be used for the matrix by utilizing rapidly solidified material. This is particularly important when the composite is to be used for high temperature applications, and rapidly solidified alloys have much better strength at elevated temperatures then conventional alloys [35].
- 4. High volume fractions of reinforcement are possible, thus maximizing the modulus and minimizing the coefficient of thermal expansion of the composite.

Powder Metallurgy processing involves the following steps

1.5.2 Steps of powder metallurgy

- Powder production
- Blending and mixing of powders
- > Compaction
- > Sintering

1.5.2.1 Production and blending of material powders:

Atomization: Produces powders of both ferrous and non-ferrous powders like stainless steel, super alloys and titanium alloy powders.

Reduction of compounds: Production of iron, copper, tungsten, molybdenum.

Electrolysis: for making Cu, iron, silver powders. Powders along with additives are mixed using mixers. Lubricants are added prior to mixing to facilitate easy ejection of compact and to minimize wear of tools; Waxes, metallic stearates, graphite etc.



Fig. 1.14 Schematic diagrams for various steps followed in powder metallurgy [27].

1.5.2.2 Compaction:

Compaction is performed using dies machined to close tolerances dies are made of cemented carbide, die/tool steel; pressed using hydraulic or mechanical presses. The basic purpose of compaction is to obtain a green compact with sufficient strength to withstand further handling operations. The green compact is then taken for sintering.

1.5.2.3 Sintering:

Sintering is essentially a process of bonding solid bodies by atomic forces. The sintering process is usually carried out at a temperature below the highest melting constituent. Sintering occurs by diffusion of atoms through the microstructure. This diffusion is caused by a gradient of chemical potential – atoms move from an area of higher chemical potential to an area of lower chemical potential. The different paths the atoms take to get from one spot to another are the sintering mechanisms.

The six common mechanisms are [28]

- 1. Surface diffusion Diffusion of atoms along the surface of a particle.
- Vapor transport Evaporation of atoms which condense on a different surface.
- 3. Lattice diffusion from surface atoms from surface diffuse through lattice.
- 4. Lattice diffusion from grain boundary –atom from grain boundary diffuses through lattice.
- 5. Grain boundary diffusion atoms diffuse along grain boundary.
- 6. Plastic deformation dislocation motion causes flow of matter.

Also one must distinguish between densifying and non-densifying mechanisms. 1–3 above are non-densifying they take atoms from the surface and rearrange them onto another surface or part of the same surface. These mechanisms simply rearrange matter inside of porosity and do not cause pores to shrink. Mechanisms 4–6 are densifying mechanisms atoms are moved from the bulk to the surface of pores thereby eliminating porosity and increasing the density of the sample [28].

1.6 Properties of some carbide-boride-nitride reinforcements

1.6.1 TiN

Nitrides have many applications because of their hardness, their properties at high temperature and their wear resistance. Titanium nitride among various nitrides is particularly interesting due to its high wear resistance coating for cuttingtool material and high corrosion resistance [29]. Titanium nitride has been used in engineering application for cutting tools to enhance their hardness and wear resistance and corrosion and more recently it has been used for the heads of hip prostheses to improve their wear &fatigue resistance [30].

Titanium nitride can be produced by self-propagating high temperature synthesis (SHS) process [31, 32].

Titanium nitride is thermally stable up to 3300 k and chemically inert to most common acids [33-35].

1.6.2 SiC

Silicon carbide is commonly used in mechanical face seals because of its abrasive resistance, corrosion resistance and thermal shock resistance, moreover sintered SiC is porous and the holes in it act as fluid reservoir, thus maintaining the fluid film and control in porosity results in better frictional performance and thermal shock resistance [36]. SiC composite have recently received particularly interests due to their high specific modulus, high strength and high thermal stability they can be widely used in aerospace, automobile industry such as electronic heat sinks, automotive drive shafts, ground vehicle brake rotors, jet fighter aircraft fins or explosion engine components etc. [37].

1.6.3 TiB₂

Titanium diboride (TiB₂) is a refractory compound that exhibits outstanding feature such as high melting point (2790°C), high hardness (86HRA or 960HV) and high modulus (530 GPa) characteristics [38]. The combination of its excellent properties has made TiB2 increasingly important for a wide range of application in erosive, abrasive, corrosive or high-tem environments [38]

1.7 Laser glazing or remelting

For densify the topmost layer of the coating, resist the top surface from plastic deformation and protect the top surface from corrosive agents, a process is introduced by Hoo et al. known as laser glazing. Laser glazing is process where a focused laser beam is used to melt a very small portion of a component and the melted surface left for rapidly solidification [50]. Problem associated with plasma sprayed coating and sintered sample like delamination, cracking, corrosion and erosion are being eliminated by laser glazing. Laser glazed surface has higher thermal shock resistance. Gahr et al. reported that laser induced remelting and alloying of ceramics composite resulted in a reduction of friction. Laser glazed surface having better mechanical properties like hardness, toughness [51, 52]. Laser-glazing imparts remelting and subsequent solidification of the surface causes lesser porosity on the surface. Laser-glazing of a surface causes improvement in microstructure, grain refinement and wear resistance. Gacsi and Kovacs investigate the laser glazed Al-SiC composites and found significant reduction in porosity of surface layer and denser surface structure with compared to the sintered composite resulted improvement in wear and corrosion resistance. Batista also report the reduction in surface roughness led to the improvement of the hot corrosion resistance of ZrO₂-8%Wt. Y₂O₃ composite coating. Yan Wang et al. studied the effect of laser glazing on the preheated yttria-stablized zirconia (YSZ) coating and concluded the preheating process enhances the quality of the laser glazing coating, since it deepens the glazed layer, widens the molten pool and reduces the cracks by reducing the thermal stresses (caused by cooling rate) [53-56].

1.8 Objective of proposed work

We aim to study the effects of laser glazing on metal matrix composite reinforced with three hard ceramic phases as TiB₂, TiN and SiC fabricated by powder metallurgical route (ex-situ). The stoichiometry of the compositions of reinforcements is selected according to the chemical reaction presented below:

$3TiO_2+2hBN+SiO_2+C=TiB_2+2TiN+SiC+4O_2\uparrow ...(1)$ [31]

1.9 Work plan

To prepare the metal matrix composite consisting of steel as matrix and three hard ceramic phases of carbide-boride-nitride as reinforcements, SiC, TiB₂ and TiN are selected in the proper stoichiometry (as per eq. (1)) with different weight proportions of steel. The powders are mixed and through powder metallurgical compaction and sintering, the composites are fabricated. After formation of the composites, laser glazing is performed on those samples and the effects of laser glazing on the sintered composites are studied and discussed thoroughly.

Chapter 2

LITERATURE REVIEW

2.1 Process parameters of composites prepared by powder metallurgical process

Erugrul *et al.* **[32]** studied the effect of particle size and heating rate of 316L stainless steel by microwave sintering process in hydrogen and argon atmosphere. The study suggests that preferable higher sintering temperature (above 1200°C) for higher density and reduction of surface oxides formation which enhances the corrosion resistance and good ductility of 316L stainless steel.

Bolzani *et al.* **[33]** studied the properties of low-cost powder metallurgy titanium alloy by 430 stainless steel addition. The behavior of powder as well as that of the sintered material are analyzed and compared to that of a master alloy addition Ti-6Al-4V alloy. The produced low-cost titanium alloys show comparable properties of both wrought and powder metallurgical produced titanium alloy. Alloys are fabricated through compaction and sintering. Process parameters are selected as compaction pressure of 700 MPa and maximum sintering temperature of 1400°C, dwell time 15 min at maximum temperature and heating & cooling rate is 10°C/min in the argon atmosphere

Reddy *et al.* **[34]** studied the effect of SiC particle on Al based MMC. It is noticed from the experiments that by using 20% SiC reinforcement on Al alloy based matrix, higher values of modulus of elasticity of 68 GPa is achieved.

Kuzenkova *et al.* **[35]** reported that the dense TiN coating can be produced through sintering from micrometric powder by the conventional sintering at high temperature (above 2000°C) for nano-metric powder sintering temperature is (1300-1500°C).

Huang *et al.* **[36]** did an experiment to observe the effects of TiN nanoparticles addition on the W–30Cu composites prepared by electroless plating and powder metallurgy route. For powder metallurgical route of sample making,

1300°C has chosen as sintering temperature and soaking time of 60 min in a hydrogen atmosphere. It is observed that addition of TiN nanoparticles significantly affected on the microstructure and properties of the W–30Cu composites. A good combination of the compressive strength and hardness of the W–30Cu composite material can be obtained by addition of TiN upto 25 wt.%.

Qureshi *et al.* [37] studied the mechanics of cold die compaction for powder metallurgy. Main objective of this paper is to present a theoretical model for the cold die compaction of powder materials based on the axisymmetric solution of large deformation. The model produces an expression relating the green density of the compact with the applied pressure. The analysis takes into account the internal (restricted movements) coefficient of friction between the particles and the container-compacted powder interface friction. Also, a modified analytical expression for the yield compression stress based on the internal coefficient of friction and the work-hardening of the powder is introduced in the analysis. Experiments are performed on cold die compaction of powder of different particles, having sizes between 45 and 150 m. Comparison between experimental and theoretical results demonstrated remarkable agreement for all the tested conditions.

The densification and grain growth behavior of powder injection molded 316L stainless steel micro–nanopowders are investigated by **Choi** *et al.* [38]. They observed the sintered density of the micro–nanopowder sample increased remarkably in the temperature range of 1000 to 1100 °C due to the sintering effect of nano-powders which increased the number of grain boundaries acting as high material transport path.

Randall M. German [39] reported that at higher sintering temperature, a denser structure is formed with much lower level of porosity. He has given a direct relationship between the sintering temperature and diffusion rate, according to that relationship increase in sintering temperature, rate of diffusion of particles increase and lowers the porosity level, hence better tribo-mechanical properties can be achieved.

Lu *et al.* [40] prepared (TiB₂ + TiC +Nd₂O₃)/Ti composites by powder metallurgical route. The composition of powder is selected according to stoichiometric amounts and three types of samples with different NdB₆ contents are prepared. Samples are prepared in a vacuum carbon furnace and heated electrically with a carbon heater. After holding for 6 h at 1673 K in a vacuum of 5×10^{-3} Pa, the samples are examined and show that Multiple ceramic particulates reinforced titanium matrix composite (TiB₂+TiC+Nd₂O₃)/Ti are ex-situ fabricated successfully by powder metallurgy utilizing the chemical reactions among Ti, NdB₆, B₄C and oxygen in Ti and reinforcements TiB₂, TiC and Nd₂O₃ are found to be distributed uniformly in the titanium matrix and the interface between reinforcements.

Kurgan [41] studied the effects of sintering atmosphere on microstructure and mechanical properties of sintered powder metallurgy 316L stainless steel. In this study, nitrogen and argon are used as the sintering atmosphere for AISI 316L stainless steel compacts. The effects of sintering atmosphere on sintered density, grain morphology and mechanical properties are investigated. The mechanical and microstructural characteristics of the sintered compacts are evaluated using optical microscopy, and scanning electron microscope. The results of this study indicates that the compacts sintered in nitrogen atmosphere exhibits higher strength and hardness than that of the steels sintered in argon atmosphere.

Lis *et al.* [42] studied the reaction sintering phenomena of selfpropagating high-temperature synthesis-derived ceramic powders in the Ti-Si-C system. The investigations are focused on preparation of stoichiometric Ti_3SiC_2 powders by SHS in the Ti-Si-C system. Powdered titanium and silicon elements and low-ash carbon black in stoichiometric 3Ti-Si-2C compositions are homogenized in a ball mill using WC/Co media for 24 h. The mixture is placed into a graphite crucible in vacuum, isolated using graphite felt and ignited by resistance molybdenum coil. After ignition, a high-temperature front of reaction with temperatures from 1800 to 2000°C propagated through the stem transforming it into product and found that high chemical activity seems to be one of the characteristic features of ceramic covalent powders synthesized by SHS (combustion) method. Such activity is manifested by their improved sinterability i.e. possibility of high densification at lower temperature. In known cases, the high sinterability of SHS powders can result from reaction sintering phenomena in presence of an intermetallic liquid phase (the Ti-Si-C system) or low temperature molten compounds.

Vinoth *et al.* **[43]** reported the development and tribological performance of nano-SiC particles on the AA2024 hybrid composites with the addition of nano-graphite. In this experimental study, hybrid nanocomposites of AA2024 aluminum alloy matrix reinforced with high weight fractions of nanometric SiC particles ranging from 5 to 10 wt % and 5% of Graphite particles are produced by blend– press–sinter methodology. Consolidation is done at 700MPa using uniaxial pressing. Sintering procedure is done at 530°C for 60 min. The mechanical properties of the sintered specimens are evaluated by hardness and porosity measurements. The results of the tests revealed that the SiC-reinforced hybrid nano composites exhibited a lower wear loss compared to the unreinforced alloy and Al–Gr composites. The nano-composite with 5 wt. % Gr and 10 wt. % SiC showed the greatest improvement in tribological performance. It confirms that graphite particle is an effective solid lubricant for aluminium matrix composite applied in dry sliding wear condition.

Mishra *et al.* [44] reported the tribological behavior of aluminium alloy Al-6061 reinforced with silicon carbide particles (10% & 15% weight percentage of SiC) fabricated by stir casting process are investigated. The wear and frictional properties of the metal matrix composites are studied by performing dry sliding wear test using a pin-on-disc wear tester and conclude that Sliding distance (62.5%) has the highest influence on wear rate followed by sliding speed (37.5%) and applied load (1.25%) and for coefficient of friction, the contribution of applied load is 85.5%, sliding distance is 13.4% for Al – 6061/ 10% SiC metal matrix composites. And increasing incorporation of SiC (10% & 15%) increases the wear resistance of composites by forming a protective layer between pin & counterface.

Bodukuri *et al.* **[45]** reported the fabrication of Al-SiC-B₄C Metal Matrix Composite by powder metallurgy technique and evaluating mechanical properties in the present work Al- SiC-B₄C metal matrix composite is prepared from sintering of mechanically alloyed powder (ball milling) in powder metallurgy processes. Three different combinations of compositions in volume fraction are chosen namely 90%Al-8%SiC-2%B₄C, 90%Al-5%SiC-5%B₄C and 90%Al 3%SiC-7%B₄C. An attempt has been made to study the characteristics of developed metal matrix composite. As increase in percentage of B₄C the microhardness of the metal matrix composite has increased significantly. The microstructure of the prepared metal matrix composite reveals the uniform distribution of particles in metal matrix.

Wang *et al.* [46] prepared SiC particulate-reinforced Al composites are prepared by powder metallurgy (PM) method and conventional atmospheric sintering and the effect of temperature on the density, hardness, strength, and microstructure of composites are investigated. Detailed failure behavior is analyzed. It is found that the segregation of SiC appeared at higher temperature. The highest micro-hardness of 80MPa occurred at 700°C. The strength tends to increase with the increasing temperature due to the formation of Al₂Cu. Both ductile and brittle fracture features are observed.

Wang *et al.* [47] fabricated TiB₂ particulate reinforced magnesium matrix composites by powder metallurgy magnesium metal matrix composites (MMCs) reinforced with 10, 20 and 30 vol.% TiB₂ particulates, respectively, are fabricated by powder metallurgy. The microstructure, porosity, hardness and abrasive wear behavior of the composites are evaluated. Microstructural characterization of Mg MMCs showed generally uniform reinforcement distribution. As compared with pure Mg, the hardness (HB) values of Mg MMCs reinforced with 10, 20 and 30 vol.% TiB₂ particulates are increased by 41%, 106% and 181%, respectively. The abrasive wear tests show that the wear resistance of Mg MMCs is increased with the increasing of the reinforcement volume fraction. This is due to the strong particulate-matrix bonding and high hardness of the TiB₂ particulate.

Panda *et al.* **[48]** studied the sintering response of austenitic (316L) and ferrite (434L) stainless steel consolidated in conventional and microwave furnace. Steel powders uniaxiallys compacted at 600 MPa into green densities ranging 80% to 82% and make a cylindrical pellet of 16 mm diameter and 6 mm average height. The green compacts are sintered using conventional and microwave furnace. The conventional sintering of green compacts are carried out in a MoSi₂ heated horizontal tubular sintering furnace at a constant heating rate of 5°C/min to ensure uniform temperature distribution during heating, an intermittent isothermal hold for 15-30 min is provided at 500 °C,700°C and 1000°C. The sintering temperature selected is 1400°C.

Soheil mahdavi et al. [49] studied the effects of SiC content on the processing, compaction behavior and properties of Al6061/SiC/Gr hybrid composite. In this experiment the powder mixtures are cold pressed in six different pressures (between 250 MPa and 750 MPa) and sintered. Finally, the produced composites are heat treated and their hardness and wear properties are investigated. Homogenous distribution of the SiC and Graphene particles within the powder mixture and the hybrid composite is clear from SEM images. The results also show that the SiC particles decrease the compressibility of the hybrid powders and improve hardness of composites. Improvement in wear resistance is achieved in the hybrid composite containing 20 vol% SiC particle. Also the study suggests that the compressibility of aluminum matrix decreases by increasing the SiC content in the powder mixture and the 40 vol% SiC contained composite has worst densification behavior. The SiC particles are not deformable and these particles support a part of the applied pressure elastically. Amount of pressure is also isted because of the friction between particles. Therefore, the transferred load to the aluminum (plastic phase) decreases. So compressibility reduces.

2.2 The effects of laser glazing

Gacsi *et al.* [50] studied the microstructural characterisation of aluminium-base ceramic particle reinforced composites produced by powder metallurgy. Powder mixtures containing different quantities (5, 10 wt.%) and grades (F500, F800) of SiC particles as reinforcement constituents are uniaxially cold pressed. Afterwards the green compacts are sintered in a high purity nitrogen atmosphere. Dimensional changes of specimens occurring during sintering are controlled by dilatometry. Porosity measurements are performed on the polished cross-sections of sintered probes by an automatic image analyser. Laser surface treatments are also carried out on the sintered materials. The surfaces of the specimens are remelted using industrial CO_2 laser equipment to achieve compacts with low porosity surface layer. The effect of laser-beam power, of amount and grain size of SiC particles on the remelting process and on the microstructure evolution are investigated and found improvement in wear resistant after laser glazing process with low power laser.

Tsai *et al.* **[51]** studied the effect of laser glazing on plasma sprayed zirconia thermal barrier coatings and reported the existence/formation/occurrence of dense resolidified columnar microstructure in laser-glazed layer and segmented cracks orthogonal to the coating surface. Marked enhancements are observed in the microhardness, surface finish and erosion resistance of the coating surface.

Tondu *et al.* [52] studied the Laser glazing of FeCr–TiC composite coating in which FeCr–TiC powder, manufactured with a self-propagating high temperature synthesis, are deposited using atmospheric plasma spraying onto mild steel substrates. The sprayed composite coatings are very porous and a treatment with a high power CO₂ laser are applied to improve their wear properties. The laser power density of 8 kWcm⁻² and a scan speed of 3 mm s⁻¹ are found to melt the steel matrix of the composite without melting the TiC reinforcement. The temperature of coatings surface at these parameters are determined with a CCD camera to be about 2000°C. The porous nature of the sprayed coatings are evidenced with a scanning electron microscope. Optical microscopic

investigations of the glazed coatings show a sealing of the pores near the coatings' surface. This investigation also revealed a formation of dendrites on laser treatment. X-ray diffraction studies enabled us to confirm that the TiC phase is present in the composite coatings before and after laser glazing. Finally, oscillating wear resistance tests showed that the laser glazing improves the performances of the composites.

Chatterjee et al. [53] studied the effect of laser post-treatment on the properties of nanostructured Al₂O₃-TiB₂-TiN based coatings developed by combined SHS and laser surface alloying and reported that Laser posttreatment leads to formation of a very thin melt layer devoid of porosity and micro-cracks and with no new phase evolution. The microstructure of the melt layer is predominantly columnar. Laser post-treatment results considerable improvement in hardness as substantial densification and compaction at and near the top of the coating take place. There is a significant rise in the wear resistance of the Al₂O₃-TiB₂-TiN coating due to laser post-treatment. Improved wear resistance in the re-melted zone is attributed to the increase in hardness, sealing of micro-porosities, refinement in microstructure, homogenization and development of columnar grains. There is also a significant reduction in the coefficient of friction of the surface during sliding movement against WC-Co counterbody at different sliding speeds and normal loads. The reductions in the coefficients of friction due to laser surface remelting are attributed to higher hardness resulting in lower arc of contact between ball and coating, microstructural homogenization and reduction in surface roughness associated with laser remelting process.

Batista *et al.* [54] investigated the surface laser-glazing effect on atmospheric plasma-sprayed (APS) ZrO_2 –8% WtY₂O₃ thermal barrier coatings (TBCs) which is subjected to a CO₂ continuous wave laser-glazing process in order to generate an external dense layer produced by different processing parameters. For that purpose, different beam scanning speeds and track overlapping are chosen. Surface roughness has been reduced significantly after laser-glazing. Despite the surface crack network, all laser-glazed specimens presented a fully dense and porous free external layer with a columnar

microstructure. Surface cracks along the densified layer are found to have tendency to be oriented in two perpendicular directions, one in the direction of the laser beam travel, the other perpendicular to it. Moreover, the cracks parallel to the beam moving direction are found to be on the overlapping zone, coinciding with the edge of the subsequent track.

Sun *et al.* [55] studied the effect of laser surface remelting on the corrosion behavior of commercially pure titanium sheet. The study reveals that considerable improvement of the corrosion resistance and pitting potential can be achieved by Nd-YAG laser surface remelting. This improvement is attributed to microstructural modification caused by rapid solidification that occurs during the laser surface remelting. One of the very crucial applications of laser glazing process is in the high temperature corrosion resistance and insulating coating for enhancement in the service life.

Reza Ghasemi. *et al.* **[56]** investigated the influence of laser treatment on hot corrosion behaviour of plasma-sprayed nanostructured yittria stabilized zirconia thermal barrier coatings and concluded that laser-glazed TBCs had a hot corrosion resistance higher than that of plasma-sprayed TBCs due to improvement in the surface roughness and the refinement in microstructure and also reducing the specific surface area of the dense glazed layer and improving the stress accommodation through network cracks produced by laser glazing.

Chapter 3 EXPERIMENTAL DETAILS

3.1 Materials

Four different types of powder mixtures have been used for the experiments. The details of the powders mentioned in Table no.3.1.

	Size	Purity	Make
Steel 304	~ 63µm		Shrikrishna
TiB ₂	~ 44µm	99.5%	Alfa Aesar
TiN	~ 10µm		Alfa Aesar
SiC	~ 2µm	99.8%	Alfa Aesar

Table 3.1 Specification of powders used for experiments

Table 3.2 Composition of steel AISI 304

Elements	С	Mn	Si	Р	S	Cr	Ni	Fe
wt.%	0.08	2.00	1.00	0.04	0.03	18.00	8.00	Balanced

3.2 Processing

The steel powder (matrix) and three ceramic powders (reinforcements) are blended and mixed together in the three different stoichiometric proportions as 20%, 30% and 40% (in weight % of steel, Table 3.3) as per chemical reaction depicted at Eq. (1) with calibrated amount of zinc stearate (binder) in ball milling (*Retsch PM 100*) operated at 150 rpm for 2 hours (clockwise and counter clockwise rotation is applied repetitively for a cycle time of 15min) in air without using the balls in the vial. After mixing, the blended mixture is uniaxially cold compacted in a 25 tons manual hydraulic single-acting pellet press (manufactured by *Kimaya engineering*). Three different compaction pressures are chosen as 740 MPa, 960 MPa and 1180 MPa. The die set is used (1*3 mm dia. Hardened alloy steel evacuable die set*) and height of sample is 6 mm. After cold compaction green pellets are sintered in a tubular furnace manufactured by *VB ceramic consultants* at a temperature of 1400°C for three different soaking time period (0, 30 and 60 min) at highest temperature 1400°C. During sintering furnace is evacuated at vacuum level of 10⁻³ mbar. Argon atmosphere maintained inside the tube and provided dwell at 1000°C for 15 min to ensure evaporating of binder which added during mixing. Heating and cooling rate provided is 5°C/min. Thus total 27 samples are prepared (sample nomenclature is given in Table 3.4).

Table 3.3 Samples composition

Sample name	Blend of ceramic powder added (in wt% of steel powder)
S2	20
S3	30
S4	40



Fig. 3.1 Various components used in powder metallurgical prepared MMC samples (a) Ball milling, (b) Cylindrical dies and punches (φ6), (c) Hydraulic pellet press and (d) Muffle furnace.

Laser post treatment (laser glazing) is performed on previously powder metallurgical prepared samples with a high power continuous wave fiber-coupled diode laser (Table 3.4).

Table 3.4 Laser	treatment specifications	and process	parameters
	1	1	1

Laser Type	Fiber laser
Laser Power (kW)	0.320
Power Density (kW cm ⁻²)	1018.6
Spot Dia. (mm)	0.2
Scan Speed (m/min)	4.5
Shielding gas	Argon
Flow rate of gas (Lt/min)	5

3.3 Characterization techniques

The physical and tribological characterization techniques are carried out on both powder metallurgical compacts and laser glazed samples. Physical and microstructure characterization of the samples are studied using scanning electron microscopy (SEM). Energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) are carried out to identify the phases. Tribological charecterizations are carried out using scratch tester and tribological properties of pellets or samples are evaluated using a linear reciprocating tribometer.

3.3.1 Physical characterization

3.3.1.1 Scanning electron microscopy (SEM)

The microstructural and morphological characterizations are carried out on *Zeiss Supra-55* field emission scanning electron microscope (FESEM) on the polished surface condition. The samples are polished with abrasive papers. The chemical compositions of the composites are qualitatively evaluated in elemental mapping mode with the help of energy dispersive spectroscopy (EDS) coupled with FESEM.

3.3.1.2 X-ray diffraction (XRD)

XRD analysis of the composite samples are carried out using a X-ray diffractometer (Rigaku Smart Lab, 3 kW) with nickel-filtered Cu-K α radiation (λ = 0.15406 nm) operated at 30 kV 40 mA. The XRD datas are analysed using the X'PertHighscore Plus[®] analysis package.

3.3.2 Tribo-mechanical characterization

To find out scratch hardness and fracture toughness, a scratch tester (*DUCOM*, *TR-101*) with Rockwell C diamond indenter (apex angle 120°, tip radius, 200 μ m, Blue Star) is used. All tests on scratch tester are conducted in atmospheric condition with temperature and humidity maintained at 24°C and

50% respectively. Width of scratch marks generated at 10N load and 0.1mm/s sliding speed are measured under both light (*Leica DFC295*) and Electron Microscopes (*Zeiss Supra-55*).

3.3.2.1 Evaluation of fracture toughness



Fig. 3.2 Scratch tester machine

The evaluation of fracture toughness (K_c) of the test samples are done by analyzing the scratch data through the procedure proposed by Akono and Ulm [57].

$$K_{c} = F_{eq} / \sqrt{2pa}$$

$$F_{eq} = F_{T} \alpha = 0$$

$$= \sqrt{\left(F_{T}^{2} + \frac{3}{5}F_{V}^{2}\right)} \alpha > 0$$

$$2pa = \left(\sqrt{4tan\phi/\cos\phi d^{3}}\right)$$
For conical Tool
$$= (w + 2d)wd$$
For Parallelepiped Tool





Fig. 3.3 Parallelepiped tool



Fig. 3.3 Conical tool

3.3.2.2 Evaluation of scratch hardness

ridge

Scratch tester is used to measure scratch hardness of samples using the following formula [58].

Scratch hardness =
$$\frac{8F_N}{\pi d^2}$$

$$A_{hc} = \frac{\left\{\pi \left(\frac{D}{2}\right)^2\right\}}{2}$$

 A_{hc} =Load bearing area, α = Cone angle, F_N = Normal load, H_s = Scratch, D/2 = Half of scratch width (Horizontal distance between axis of indenter and center of formed ridge) h= Indentation depth from the top surface of the sample + height of



Fig. 3.4 Schematic diagram for evaluating scratch hardness.

3.3.2.3 Linear reciprocating wear test



Fig. 3.5 Linear reciprocating tribometer.

Linear reciprocating wear tests are carried out using a tribometer to investigate friction and wear properties of samples. Cemented carbide (WC-6%Co) balls (φ 6) are used as counter body. The tests are performed with constant load of 10 N for different samples. The tests are continued for 30 min. and frequency of sliding is 10 Hz and track length of 4 mm. The coefficient of friction (μ) is defined as the ratio of the tangential force (*F*) required to move two surfaces relative to each other to the force pressing these surfaces together (*N*).

$$\mu = \frac{F}{N}$$

The wear track studied using SEM and EDS. The depth of the wear track is measured using *Taylor-Hobson, Surtronic* profilometer. Specific wear rate calculated using the following equation,

$$\omega_d = \frac{V}{dL} 10^{-4}$$

where, ω_d is the specific wear rate of the samples in mm³/(N.m), V is the volume loss in mm³, d is the sliding distance in m, L is the normal load in N., V is determined using the following equation

$$V = l \left[\left(\frac{\pi r^2}{180} \sin^{-1} \frac{b}{2r} \right) - \frac{b}{4} \sqrt{4r^2 - b^2} \right] + \frac{2\pi r^2}{3} \left[r - \frac{1}{2} \sqrt{4r^2 - b^2} \right]$$

Here l is sliding distance in meter and r and b is the ball radius in mm and track width in mm respectively.

3.4 Sample nomenclatures

Serial no.	Weight proportion of ceramic powder blend mixed to steel powder	Soaking time (min)	Compaction pressure (MPa) (Green pellet dia: 13 mm)	Sample nomenclature
1			740	S111
2		0	960	S112
3			1180	S113
4			740	S121
5	20	30	960	S122
6			1180	S123
7			740	S131
8		60	960	S132
9			1180	S133
10			740	S211
11		0	960	S212
12			1180	S213
13			740	S221
14	30	30	960	S222
15			1180	S223
16			740	S231
17		60	960	S232
18			1180	S233
19			740	S311
20		0	960	S312
21			1180	S313
22			740	S321
23	40	30	960	S322
24			1180	S323
25			740	S331
26		60	960	S332
27			1180	S333

Chapter 4 RESULTS AND DISCUSSTION

4.1 Scanning electron microscopy (SEM) analysis

Fig. 4.1 shows the FESEM micrograph of 30 wt.% of ceramic in metal matrix composite sintered at 1400°C for 30 min. FESEM Micrograph reveals the uniform distribution of reinforced particles (TiB₂, SiC, TiN) in steel matrix. The EDS spectra shows in Fig. 4.2 confirms the presence of Ti, N, Fe, Si.



Fig. 4.1 Scanning electron micrograph of sample S122



Fig. 4.2 EDS (Electron dispersive electroscopy) of sample S122



Fig. 4.3 Scanning electron micrograph of sample S222



Fig. 4.4 Scanning electron micrograph of sample S322

SEM micrograph of sample S222 and S322 shows in Fig. 4.3 and Fig. 4.4 also reveals that, the reinforced particles are uniformly distributed throughout the steel matrix and some voids are also noticed in the SEM micrograph which is due to the incompressible hard ceramic particles in steel matrix.

4.2 X-ray diffraction (XRD) analysis

The XRD spectra of samples S122, S222 and S322 are shown in Fig. 13, 14, and 15 respectively. The XRD spectrum shows the presence of strong peaks of TiB₂, TiN, SiC, Fe.



Fig. 4.5 (a), (b) and (c) X-ray diffraction patterns of the top surfaces of sintered three samples S122, S222 and S322.

4.3 Operating conditions used in experiments for friction, wear, scratch hardness and fracture toughness measurement

Counter- Body	WC-6wt.% Co, 6 mm diameter
Frequency	10 Hz
Temp.	25°C (room temp.)
Stroke Length	4 mm
Time	30 min
Load	10 N (constant)
Sliding Distance	160 m

Table 4.1 Operating conditions for linear reciprocating tribometer

Table 4.2 Operating conditions for scratch tester

Type of Indenter	Diamond Rockwell C (Apex angle = 120°)
Type of Loading	10 N (constant)
Stroke Length	4 mm
Scan Speed	0.1 mm/sec

4.4 Analysis of coefficient of friction (COF)

4.4.1 Sintered samples

Fig. 4.6 (a), (b) and (c) shows the variation in coefficient of friction with respect to the 20 wt.%, 30 wt.% and 40 wt.% blended ceramic in steel matrix. It is observed that by increasing the weight percentage of ceramic in MMC the COF increases up to 30 wt.% and as further increase in wt.% of ceramic COF decreases.

It is also observed that at 30 min soaking time the values of COF show higher as compare to the 0 min and 60 min and it is also observed that at 960 MPa, COF shows the higher values as compare to the 740 MPa and 1180 MPa compaction pressure.

4.4.2 Laser glazed samples

In Fig. 4.7 (a), (b) and (c) it is observed that laser glazed samples show the same variation in COF as observed in case of sintered samples in Fig. 4.6 (a), (b) and (c) with respect to the 20 wt.%, 30 wt.% and 40 wt.% composition of blended ceramic in steel. It is observed that at 30 wt.% composition of ceramic samples show the higher values of coefficient of friction as compare to the 20 wt.% and 40 wt.% ceramic content laser glazed samples.

It is also observed that in Fig. 4.7 (a-c) at 30 min soaking time coefficient of friction has higher values as compare to the 0 min and 60 min soaking time. It is also observed that at 960 MPa compaction pressure shows larger values of COF as compare to the 740 MPa and 1180 MPa compaction pressure.

In Fig. 4.7 (a-c) and Fig. (a-c), the trends of COF are discussed for both the samples, sintered and laser glazed samples, at 30 wt.% of ceramic content MMCs show the higher values of COF because the composite consists of more wt.% of hard ceramic. It is also seen that while further increment in wt.% of ceramic particle after 30 wt.%, coefficient of friction decreases because the presence of excess amount of ceramic particle inside the metal matrix leads to nonuniformalty in the matrix causes the reduction in coefficient of friction.



Fig. 4.6 (a), (b) and (c) Variation in coefficient of friction with respect to weight proportions of ceramic in steel, soaking time and compaction pressure (sintered MMCs).



Fig. 4.7 (a), (b) and (c) Variation in coefficient of friction with respect to weight proportion of ceramic in steel, soaking time and compaction pressure (laser glazed MMCs).

It is also seen that at 30 min soaking time higher values of COF is due to optimum soaking time as compared to less values of COF at 0 min soaking time results in under sintering of MMC, hence more porosity remained due to which easily dislodgement of particles, no resistant to wear and COF reduces, while in the case of 60 min soaking time oversintering occurs, causes more coarsening of particles and COF reduces again. It is also seen that as the compaction pressure increases from 740 MPa to 960 MPa, COF increases initially because of density of compact increases then again it decreases as the compaction pressure increases due to the fact that at higher compaction pressure (above optimum compaction pressure) may tend to distort the metal matrix by the presence of hard ceramic particles inside it, this ceramic particle transfers the excess amount of load directly to the metal matrix causes distortion of matrix and less strengthening of MMC. It is also seen that laser glazed samples has higher values of COF compare to the sintered samples it is due to the fact that reduction in porosity inside the MMCs.

4.5 Analysis of specific wear rate

4.5.1 Sintered samples

Fig. 4.8 (a), (b) and (c) shows the variation in specific wear rate with respect to the 20 wt.%, 30 wt.% and 40 wt.% of blended ceramic in steel matrix. It is observed that by increasing the weight percentage of ceramic in MMC the specific wear rate decreases up to 30 wt.% and as further increase in wt.% of ceramic specific wear rate increases.

It is also observed that at 30 min soaking time the values of specific wear rate show minimum as compare to the 0 min and 60 min and it is also observed that at 960 MPa compaction pressure the values of specific wear rate show the minimum values as compare to the 740 MPa and 1180 MPa compaction pressure.



Fig. 4.8 (a), (b) and (c) Variation in specific wear rate with respect to weight proportion of ceramic in steel, soaking time and compaction pressure (sintered MMCs).

4.5.2 Laser glazed samples

In Fig. 4.9 (a), (b) and (c) it is observed that laser glazed samples shows the same variation in specific wear rate as observed in case of sintered samples in Fig. 4.8 (a), (b) and (c) with respect to the 20 wt.%, 30 wt.% and 40 wt.% composition of blended ceramic in steel. It is observed that at 30 wt.% composition of ceramic samples shows the minimum values of specific wear rate as compare to the 20 wt.% and 40 wt.% ceramic content laser glazed samples.

It is also observed that at 30 min soaking time, specific wear rate has minimum values as compared to the 0 min and 60 min soaking time. It is also observed that at 960 MPa compaction pressure shows minimum values of specific wear rate as compare to the 740 MPa and 1180 MPa compaction pressure.
In Fig. 4.10 (d) and (e) is plotted for observing the process parameters more closely, it is observed that at 30 min soaking time has the minimum values specific wear rate as compare to the 0 min and 60 min soaking time.



Fig. 4.9 (a), (b) and (c) Variation in specific wear rate with respect to weight proportion of ceramic in steel, soaking time and compaction pressure (laser glazed MMCs).

In fig.4.7 (e) it is observed that 960 MPa shows minimum value of specific wear rate as compare to the 740 MPa and 1180 MPa compaction pressure.

In Fig. 4.8 (a-c) and Fig. 4.9 (a-c), the trends of specific wear rates are discussed for both the samples, sintered and laser glazed samples. At 30 wt.% of ceramic reinforced MMCs show the minimum values of specific wear rates and it is also found to be optimum amount of ceramic in matrix. Literature also suggests the same. It is also seen that increase in ceramic content above 30 wt.% causes less strengthening of MMC leads to higher specific wear rate, while the increment

in soaking time from 0 min to 30 min specific wear rate decreases up to its minimum values, due to optimum level of particles diffusion inside the matrix results in optimum sintering of MMCs at 30 min soaking time and again the specific wear rate increases as the soaking time crosses its optimum value. At higher soaking time (60 minute) causes coarsening of particles those lead to increase in specific wear rate. It is seen that specific wear decreases by increasing compaction pressure from 740 MPa to 960 MPa because more compaction leads to higher density and better mechanical properties for that reason it is difficult to dislodged particle from the wear track, so by increase in compaction load specific wear rate decrease.





It is observed in at 1180 MPa compaction load specific wear rate increases, due to presence of hard ceramic particles which deformed the matrix and increase the chance for more crack propagation. Higher compaction pressure causes distortion of matrix due to hard reinforced particle causing more crack propagation which is responsible for high specific wear rate.

It is seen from the experiment that laser glazed samples have lower specific wear rates as compared to sintered samples. In case of laser glazed sample a denser, less porous and more hard layer formed due to rapid solidification of laser glazed surface, which restrict the crack propagation which leads to lower value of specific wear rate as compare to sintered samples.

4.6 Analysis of scratch hardness

4.6.1 For sintered samples

Fig. 4.11 (a), (b) and (c) shows the variation in scratch hardness with respect to the 20 wt.%, 30 wt.% and 40 wt.% of blended ceramic in steel matrix. It is observed that by increasing the weight percentage of ceramic in MMC the scratch hardness increases up to 30 wt.% and as further increase in wt.% of ceramic scratch hardness decreases.



Fig. 4.11 (a), (b) and (c) Variation in scratch hardness with respect to ceramic composition, soaking time and compaction pressure (sintered MMCs).

It is also observed that at 30 min soaking time the values of scratch hardness show higher as compare to the 0 min and 60 min and there is no such trend in variation of scratch hardness has been observed with respect to variation in compaction pressure.

4.6.2 Laser glazed samples

In Fig. 4.12 (a), (b) and (c) it is observed that laser glazed samples show the same variation in scratch hardness as observed in case of sintered samples in Fig. 4.11 (a), (b) and (c) with respect to the 20 wt.%, 30 wt.% and 40 wt.% composition of blended ceramic in steel. It is observed that at 30 wt.% composition of ceramic samples show higher values of scratch hardness as compared to the 20 wt.% and 40 wt.% ceramic content laser glazed samples.



Fig. 4.12 (a), (b) and (c) Variation in scratch hardness with respect to ceramic composition, soaking time and compaction pressure (laser glazed MMCs).

It is also observed that at 30 min soaking time scratch hardness has higher values as compared to the 0 min and 60 min soaking time. It is also observed that

at 960 MPa compaction pressure shows larger value of scratch hardness as compared to the 740 MPa and 1180 MPa compaction pressure.

In Fig. 4.11 (a-c) and Fig. 4.12 (a-c) trends of scratch hardness are discussed for both the samples, sintered and laser glazed samples. It is seen that the increased value of scratch hardness at 30 wt.% of ceramic contents due to the formation of more strengthened MMCs at this wt.% as compare to 20 wt.% and 40 wt.% and also at 30 min soaking time, higher values of scratch hardness observed due to optimum value of sintering. After laser glazing the values of scratch hardness is found to be increased due to the formation of super hard, denser and crack free surface layers on the MMCs resulted in restriction to plastic deformations.

4.7 Analysis of fracture toughness

4.7.1 For sintered samples

Fig. 4.13 (a), (b) and (c) shows the variation in fracture toughness with respect to the 20 wt.%, 30 wt.% and 40 wt.% of blended ceramic in steel matrix. It is observed that by increasing the weight percentage of ceramic in MMCs the fracture toughness increases up to 30 wt.% and as further increase in wt.% of ceramic fracture toughness decreases.

It is also observed that at 30 min soaking time the values of fracture toughness show higher as compared to the 0 min and 60 min and there is no such trend in variation of fracture toughness has been observed with respect to variation in compaction pressure.

4.7.2 Laser glazed samples

In Fig. 4.14 (a), (b) and (c) it is observed that laser glazed samples shows the same variation in fracture toughness as observed in case of sintered samples in Fig. 4.13 (a), (b) and (c) with respect to the 20 wt.%, 30 wt.% and 40 wt.% composition of blended ceramic in steel. It is also observed that at 30 wt.%



Fig. 4.13 (a), (b) and (c) Variation in fracture toughness with respect to ceramic composition, soaking time and compaction pressure (sintered

MMCs).



Fig. 4.14 (a), (b) and (c) Variation in fracture toughness with respect to ceramic composition, soaking time and compaction pressure (laser glazed MMCs).

composition of ceramic samples show the higher values of fracture toughness as compared to the 20 wt.% and 40 wt.% ceramic content laser glazed samples.

It is also observed that at 0 min and 30 min soaking time fracture toughness has higher values as compare to the 60 min soaking time. No such trend in variation of fracture toughness has been observed with respect to variation in compaction pressure.

In Fig. 4.13 (a-c) and Fig. 4.14 (a-c) trends of fracture toughness are discussed for both the samples, sintered and laser glazed samples, it is seen that at MMCs having 30 wt.% of ceramic content in matrix have maximum values of fracture toughness as compare to the 20 wt.% and 40 wt.% ceramic contents, at 30 wt.% more strengthened MMCs formed as compare to other wt.%, this more strengthened matrix also allows to restrict the crack propagations, since steel forms more strengthened matrix at the 30 wt.% of ceramic content.

Chapter 5

Conclusion

The effect of laser glazing on tribo-mechanical properties of steel based metal matrix composites are judged in this present study. Besides, the effects of variations in powder metallurgical process parameters are also investigated. A comparative study on laser glazing and sintering is also carried out by examining the graph plotted for various properties. On the basis of this study following points are observed:

- Tribological properties (coefficient of friction and specific wear rate) also seen optimum at 30 wt.% of blended ceramic mixture in steel matrix as compare to other 20 wt.% and 40 wt.%.
- Better mechanical properties and tribological properties are achieved at compaction pressure of 960 MPa against the other compaction pressure of 740 MPa and 1180 MPa.
- 30 min sintering time at constant temperature of 1400°C shows the better results compared to 0 min and 60 min.
- After laser glazing on the sintered MMC samples improvement in tribological properties are observed due to formation of more hard, crack free and denser surface microstructures.

Future Scope

- The study of formation of metal matrix composite (MMC) as an interlayer in ceramic matrix composite (CMC) coating deposited through laser surface alloying (LSA) can also be studied by comparing the results obtained in the present study.
- More number of samples can be prepared by varying in weight percentage of ceramic content near the value of 30 wt.% to see the further effects on tribological properties of MMC.
- Larger dimension MMC specimens can also be prepared by using same powder metallurgical process parameters to evaluate the other mechanical properties like elastic modulus, yield strength etc.
- Other light metals like aluminium and titanium along with other hard ceramic reinforcements can be used to fabricate the metal matrix composites.

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