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ABSTRACT

Coating is physically bonded with substrate by adherence. So, on successful formation of adherence between coating and substrate, the surface engineering components can works successfully for a longer period of time. So, it is very important to study the bond formation between coating substrate. It is difficult to study the interlayer formation of a coated sample due to low coating thickness. The aim of the present work is to study the interlayer formation between steel substrate developed on the top of it, and a coating comprising of SiC, TiB₂, and TiN.

The study of interlayer can be done in both direct and indirect methods. In the direct approach, the interface region between the coating and substrate needs to be reached by removing the top part of the coating by means of grinding and polishing. This is difficult in case the interlayer is wavy, grinding and polishing of the top part of coating may actually remove some part of interlayer and expose the substrate. This hampers the study. The indirect methods involve studying and sensing properties of the coating, but it is heavily dependent on interpretations.

To study the interlayer of coating and underlying substrate, a metal matrix composite (MMC) is prepared in the powder metallurgical route. In the present work the MMC is prepared by different weight proportion of ceramic powder blend mixed to steel powder. The effect of different parameters such as compaction pressure and soaking time has been studied on composite.

For evaluation of physical and mechanical properties of MMC, physical characterization such as Optical microscopy, x-ray diffraction spectroscopy (XRD) and scanning electron microscopy (SEM), mechanical characterization such as, scratch hardness test, fracture toughness test, friction and wear test are performed.

It is observed form the experiment that mechanical and tribological properties of MMC is optimum at 30wt.% of ceramic in steel matrix and it is also noticed that at compaction pressure 960 MPa and soaking time 30 min MMC shows optimum mechanical and tribological properties.

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ACRONYMS

PMC	:	Polymer Matrix Composites
MMC	:	Metal Matrix Composites
CMC	:	Ceramic Matrix Composites
HIP	:	Hot Isostatic Pressing
LSA	:	Laser Surface Alloying
SHS	:	Self-propagating High -temperature Synthesis
EDS	:	Energy Dispersive Spectroscopy
FE-SEM	:	Field Emission Scanning Electron Microscope
XRD	:	X-ray Diffraction

1.1 Surface engineering

The surface characteristics of engineering material have a significant effect on the serviceability and life of components thus cannot be neglected in design. "Surface engineering" is defined as the branch of science that deals with methods for achieving the desire surface requirement and their behavior in service for engineering components [1].

The surface of any components may be selected on the basis of texture and colorbut engineering components generally demand a lot more than this. Engineering components must perform certain functions completely and effectively under various conditions in aggressive environments.Engineering environments are normallycomplex, combining loading with chemical and physical degradation to the surface of the component.Example of engineering components that working in aggressive environment is cutting tool used in machining process. Tool experience high loads, high speed, friction and high temperature, thesefactors leads to surface wear or the component.Surface coating can help with these circumstances improving the tool surface, not only improves the life of the tool, but also the surface finish.

1.2 Classifications of surface engineering

1.2.1 Changing the surface Metallurgy

Improve properties like wear and fatigue by changing surface metallurgy.

- (a) Localized surface hardening: (Flame, induction, laser and laser beam hardening) [2].
- (b) Laser melting: Improvements wear resistances through grain refinement and the formation of fine dispersion of precipitates on the surface [2].
- (c) Shot peening: Shot peening is the surface enhancement process which produce beneficial compressive stress on metallic surfaces.

(d) This improves fatigue strength and relieves a tensile stress that contributes to stress- corrosion cracking [3,4].

1.2.2 Changing the surface Chemistry

Surface modification process has advantage over coatingprimary because

- I. Usually import internal compressive stresses in near surface region
- II. Will not delaminated off the substrate [5].They include
 - (a) Chemical or electrochemical conversion treatment that produce complex phosphate, chromates/oxide on metal surface.
 - (b) Thermo chemical diffusion that involves the introduction of interstitial elements like C, N, or B in to ferrous alloy surface at elevated temperature.
 - (c) Surface modification by combination of laser beam melting and alloying [6].

1.2.3 Adding a surface layer or coating

This involves an intentional buildup or adding of new layer on metal substratei.e. applicationofcoating or lining. A wide range of processes are used to deposited metal/ceramicand organic (paints or plastics and rubber coating) coating method commonly used are [7].

- a) Organic coating
- b) Ceramic coating
- c) Hot dip metallic coating
- d) Electroplating
- e) Metal or ceramic coating
- f) Cladding (thick metal coating)
- g) Thermo reactive deposition/diffusion process

Among all the above methods of coating deposition, ceramic coating have advantages since ceramic material are noted for their high hardness and good chemical and thermal stability, making their surface properties ideal for number of engineering products which encounter demanding operating condition [7]. The deposition of ceramics phase onto conventional metallic materials results in good combination of bulk toughness and load support with desirable surface characteristics. So, it is discussed here, how surface engineering and surface modification techniques is important for engineering components. However, some time it is found that coating is delaminated from the surface and the components get failed before completion of desire work. Let's take a simple example of daily life, wall paint damage after same period of time because adherence between paint and wall is not proper and it cause to loss the paint from wall surface. Sometime same situation is found in between coating and substrate, so it is necessary to study the mechanical and tribological property of interlayer formed between coating and substrates [7].

1.3 Inter layer formation in coating

Coating is physically bonded with substrate material called adherence. Adherence should be strong enough for the engineering components to works successfully for a longer period of time. So, it is necessary to study the coating substrate adherence or interlayer.

1.3.1 Importance of interlayer formation.

- Importance of coating as a protective surface layer depends on the quality of coating-substrate bonding.
- (b) On successful formation of adherence between coating and substrate, the surface engineering product works successfully for a longer period of time.
- (c) So, it is very important to study the coating-substrate bonding so that the act of surface engineering of a product can be successful.
- (d) Coating should establish physical bonding connection to the substrate material.

1.3.2 Bonding mechanism of coating/substrate

The bonding strength between the coating and the substrate constitutes an important indicator to measure the service performance of the coating system. The

mechanism for the bonding between the coating and the substrate interface has all along been an critical foundation of theoretical research on surface engineering; the difference in coating preparation process has determined the diversified bonding forms of bonding between the coating and the substrate, such as metallurgical bonding, diffusion bond, epitaxial growth, chemical bonding, molecular bonding and mechanical bonding etc. [8].

1.3.3 Types of bonding in coating/substrate

The coating generally exhibits a lamellar structure to which two types of interlayer are as follows:

(a)One is the substrate-coating interface means first lamellar layer next to substrate.

(b)Second inter-splat interface within the coating[9].

The substrate-coating interface bonding determines the coating adhesion, being the resistant to prevent the coating from spalling under service loading. Thus, the adhesive strength must be strong enough not to permit any spalling of coating under service condition [10]. While the inter-splat interface bonding dominates the coating cohesion and there by the coating property [11]. Therefore the characteristic of these two kinds of interfaces have significant effect on the overall properties of coating. Generally speaking the adhesion of the coatingsubstrate arises from chemical bonding, physical geometrical contacts and mechanical interlocking at the interface [11,12]. It has been known for several decades that adhesion between coating and metal substrate is due to chemical or metallurgical bonding through substrate melting induced by molten particle impingement for several typical coating materials. When molten particle with higher melting point impacts on a substrate surface with lower melting point, the thermal interaction between them will cause localized substrate melting. This phenomenon was confirmed experimentally and numerically by the formation of metallurgical bonding when spraying Mo, Ta and W particle on steel substrate [12,13]. It is observed from above literature that chances of formation of metal matrix composite at interface would be more, when high melting point materialdeposited on low melting point substrate.

1.4 Composite materials and its Classifications

A composite is a structural material that consists of two or more combined constituents that are combined at a macroscopic level and are not soluble in each other. One constituent is called reinforcing phase and one in which it is embedded is called the matrix. The reinforcing phase material may be in the form of fibers, particles or flake. The matrix phase materials are generally continuous. Examples of naturally found composites include wood, where the lignin matrix is reinforced with cellulose fibers and bones in which the bone-salt plates made of calcium and phosphate ions reinforce soft collagen.[13]

The roles of matrix in composite materials are to give shape to the composite part, protect the reinforcements to the environment, transfer loads to reinforcements and toughness of material, together with reinforcements. The aims of reinforcements in composites are to get strength, stiffness and other mechanical properties, dominate other properties as coefficient of thermal extension, conductivity and thermal transport. The composites materials are some advantages as:

1.4.1 Advantages of composites are the followings:

- 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

1.4.2 Disadvantages of composites are the followings:

> Cost of materials

- Long development time
- Difficulty manufacturing
- ➤ Low ductility
- ➢ Temperature limits
- Solvent or moister attack
- Hidden damages and damage susceptibility.

1.4.3 Classification

1.4.3.1 Composites are classified by geometry of the reinforcement

- ➢ Particulate,
- ➢ Flake,
- ➢ Fibers filled
- > Whiskers
- Directionally solidified eutectics.

1.4.3.2 Classified by the type of matrix

- Polymer matrix composite
- Metal matrix composite
- Ceramic matrix composite
- Carbon-carbon matrix composite

Polymer matrix composite material is classified as thermoset and thermoplastics. Againthermosets and thermoplastics are divided byepoxy, phenolic polyamide resin, polyester and polyethylene, polystyrene, polyamides, nylons and polypropylene respectively.[14]

Particulate composites consist of particles immersed in matrices such as alloys and ceramics. They are usually isotropic because the particles are added randomly. Particulate composites have advantages such as improved strength, increased operating temp, oxidation resistance, etc. Typical examples include use of Aluminum particles in rubber, silicon carbide particles is aluminum, and gravel sand, and cement to make concrete.

Flake composites consist of flat reinforcements of materials. Typical flake materials are glass, mica, alumina and silver. Flake composites provide advantages such as high out-of- plane, flexural modulus, higher strength and low cost. Howeverflakes cannot be oriented easily and only a limited no. of materialsare available for use.

Fiber composites consist of matrices reinforced by short (discontinuous) or long (continuous) fibers. Fibers are generally anisotropic and examples include carbon and aramides.

Polymer Matrix Composites (PMCs) is the most common advanced composites are polymer matrix composites. These composites consist of a polymer thermoplastic or thermosetting reinforced by fiber (natural carbon or boron). These materials can be fashioned into a variety of shapes and sizes. They provide great strength and stiffness along with resistance to corrosion. The reason for these being most common is their low cost, high strength and simple manufacturing principles.

Metal Matrix Composites (MMCs) as the name implies, have a metal matrix. Examples of matrices in such composites include aluminum, magnesium and titanium. The typical fiber includes carbon and silicon carbide. Metals are mainly reinforced to suit the needs of design. For example, the elastic stiffness and strength of metals can be increased, while large co-efficient of thermal expansion and thermal and electrical conductivities of metals can be reduced by the addition of fibers such as silicon carbide.

Ceramic Matrix Composites (CMCs) have ceramic matrix such as alumina, calcium, alumino silicate reinforced by silicon carbide. The advantagesof CMC include high strength, hardness, high service temperature limits for ceramics, chemical inertness and low density. Naturally resistant to high temperature, ceramic materials have a tendency to become brittle and to fracture. Composites successfully made with ceramic matrices are reinforced with silicon carbide fibers. These composites offer the same high temperature tolerance of super alloys

but without such a high density. The brittle nature of ceramics makes composite fabrication difficult. Usually most CMC production procedures involve starting materials in powder form. There are four classes of ceramics matrices: glass (easy to fabricate because of low softening temperatures, include borosilicate and alumino silicates), conventional ceramics (silicon carbide, silicon nitride, aluminum oxide and zirconium oxide are fully crystalline), cement and concreted carbon components.

Carbon-carbon composites (CCMs) use carbon fibers in a carbon matrix. Carbon-carbon composites are used in very high temperature environments of up to 6000F, and are twenty times stronger and thirty times lighter than graphite fibers.

1.4.4 Constituents of composites

(a) Matrices

The role of matrix in a fiber-reinforced composite is to transfer stress between the fibers, to provide a barrier against an adverse environment and to protect the surface of the fibers from mechanical abrasion. The matrix plays a major role in the tensile load carrying capacity of a composite structure. The binding agent or matrix in the composite is of critical importance. Four major types of matrices have been reported: Polymeric, Metallic, Ceramic and Carbon. Most of the composites used in the industry today are based on polymer matrices. Polymerresins have been divided broadly into two categories: Thermosetting and Thermoplastics. Thermoset is a hard and stiff cross linked material that does not soften or become moldable when heated. Thermosets are stiff and do not stretch the way that elastomers and thermoplastics do. Several types of polymers have been used as matrices for natural fiber composites. Most commonly used thermoset polymers are epoxy resins. Thermoplastics are polymers that require heat to make them process able. After cooling, such materials retain their shape. In addition, these polymers may be reheated and reformed, often without significant changes in their properties [15].

(b) Reinforcements

The role of the reinforcement in a composite material is fundamentally one of increasing the mechanical properties of the neat resin system or reinforcements impart special physical (mechanical and electrical) properties to enhance the matrix properties.

1.5 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 [16]. 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 [17]. 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 [18]. Partly due to economic benefits, research began to evaluate particulates as reinforcements for MMCs, including silicon carbide (SiC), aluminum oxideoralumina (A1₂O₃),

titanium carbide (TiC), titanium diboride (TiB₂) 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 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.

1.5.1 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 as shown in Fig. 1 [19].

Fiber composite materials can be further classified into continuous fiber composite materials (multi- and monofilament) and short fibers or, rather, whisker composite materials as shown in Fig. 2 [20].



Fig. 1 Flowchart of types of metal matrix composites [19].



Fig. 2 Types of metal matrix composite [20].

1.5.2 Fabrication of Metal matrix composite

In general the most common manufacturing MMC technologies 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 performs), but not necessarily to final shape or final microstructure and the secondary processing is the step which obviously follows primary processing, and its aim is to alter the shape and microstructure of the material (shape casting, forging, extrusion, heat treatment, machining) [21-24]. 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
- Vapour state processing
- Plasma /spray deposition
- In situ processing

1.5.2.1 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 [25].

Solid state processing method is followed by three ways:

1. Diffusion bonding

- 2. Deformation processing
- 3. Powder metallurgy

1.5.2.1.1Diffusion 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 tobonding.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 [26].



Fig. 3 Diffusion bonding process [26].

1.5.2.1.2 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.[27]



Fig. 4 Roll bonding process of making a laminated MMC [28].

1.6Powder metallurgy process

1.6.1 Introduction

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 [29].

Powder Metallurgy processing involves the following steps

1.6.2 Steps of powder metallurgy

- Powder production
- > Compaction
- > Sintering

1.6.2.1 Methods for making powders:

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

1.6.2.2 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.

1.6.2.3Sintering:

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 [30]

(a) Surface diffusion- Diffusion of atoms along the surface of a particle.

- (b) Vapour transport- Evaporation of atoms which condense on a different surface.
- (c) Lattice diffusion from surface- atoms from surface diffuse through lattice.
- (d) Lattice diffusion from grain boundary- atom from grain boundary diffuses through lattice.
- (e) Grain boundary diffusion- atoms diffuse along grain boundary.
- (f) Plastic deformation dislocation motion causes flow of matter.

Also one must distinguish between densifying and non-densifying mechanisms. (a)–(c) 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 (d)–(f) are densifying mechanisms atoms are moved from the bulk to the surface of pores thereby eliminating porosity and increasing the density of the sample [31].

1.7 Mechanical and tribological performance of some single phase composites

1.7.1 TiN (Titanium Nitride)

Nitride has many applications because of their high hardness; high wears resistance and sustains high temperature. Titanium nitride among various nitrides is particularly interesting due to its high wear resistance coating for cutting-tool material and high corrosion resistance [32]. It has been used in engineering application for cutting tools to enhance their hardness, wear resistance and corrosion resistance. More recently it has been used for the heads of hip prostheses to improve their wear &fatigue resistance[33]. Titanium nitride can be produced by self-propagating high temperature synthesis (SHS) process. It is thermally stable up to 3300 K and chemically inert to most common acids [34].

1.7.2 SiC (Silicon Carbide)

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 [35]. 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. [36].

1.7.3 TiB₂ (Titanium Diboride)

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 [37]. 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].

1.8 Objective of current work

To study the formation of the interlayer in a coated sample where the ceramic matrix composite coating layer forms a metallurgical bond to the steel substrate and adhere to it. For this we have considered a ceramic matrix composite comprising of SiC, TiB_2 and TiN. The formation of coating takes place through 'In-situ' route with the application of high power continuous wave laser.

$$3TiO_2+2hBN+SiO_2+C=TiB_2+2TiN+SiC+4O_2$$
.....(1)

The governing equation of coating is shown above (Eq. 1)used for the study.Singh et al.[39]work on Ti-B-N coating that coating is developed by laser surface alloying process. The aimed of investigation is determination of the feasibility of formation of in-situ Ti-B-N-SiC system of coating by combining self propagating high temperature synthesis (SHS) and laser surface alloying (LSA) and to investigate the potential of the coating in terms of hardness, wear resistance and anti-friction properties.

1.8.1 Laser surface alloying process (LSA)

Laser surface alloying is laser assisted surface engineering technique that involves melting of a pre-/co-deposited layer of alloying element along with a part

of underlying substrate leading to the formation of alloying zone confined to a very low depth from the top surface within a very short interaction time. Laser surface alloying is usually carried out using a continuous wave (CW) laser beam normal to the substrate. When the laser beam scans the surface, it creates a melt pool. Addition of alloying element to the melt pool is done either as a deposited layer on the substrate, or by feeding powder into the melt pool. Once irradiation stops, solidification occurs due to rapid heat transfer to the bulk. The solidification front progresses rapidly from the bottom towards the surface, at a very high speed (mainly depending on the scanning speed, depth and shape of the melt pool and material properties). Fig 5 show the schematic of laser surface alloying with pre-deposited alloying.

Laser surface alloying is used for increasing the strength of various materials (i.e. steel, cast iron, titanium, nonferrous metals, alloys etc.) with a wide range of elements and their compounds because of the relative ease of introducing them into the zone of alloying [39].



Fig. 5 Laser Surface Alloying

1.8.2 Self-Propagating High Temperature Synthesis (SHS)

Self-propagating high-temperature synthesis in short (SHS) in the SHS, preheating one end of a reactant mixture initiates an exothermic reaction. Once initiated, the reaction becomes self-sustaining and the hot reaction zone propagates through the sample, leaving in its wake the final solid product. It generates sufficientthermal energy to sustain in the form of a reaction (combustion) wave through theextent of the reactants. The wave travels through the reactants, completely converting them to the final product. The specialty of this process is the conversion of chemical energy to thermal energyand once triggered, no further externally applied energy is necessary. Although such reactions are highly exothermic to be self-sustaining, they cannot self-initiate withoutthe addition of external energy. For initiation of SHS reaction, it is required to heat one end of the sample either by thermal radiation or through the use of laser energy orby any other heat source.

1.9Work plan

To simulate the problem situation taking place at the coating substrate interface. It is planned to make compacts of steel powder having the same ceramic phases present in proper stoichiometric proportions as that of the coating mentioned above. After compaction and sintering the sample, physical and mechanical characterizations are carried out thoroughly. The results are compared to the actual coating and its interface with the steel substrate.

Chapter 2

Literature Review

2.1 Process parameter of some composite prepared by Powder Metallurgical process

Erugrul et al. [40] studied the effect of particle size and heating rate of 316L stainless steel by microwave sintering process in hydrogen and argon atmosphere. They preferred higher sintering temperature (above 1200°C) for higher density and reduction of surface oxides formation which enhance corrosion resistance and good ductility of 316L stainless steel.

Reddy et al. [41] did an experiment to observe the effect of SiC particle on Al based MMC. They noticed from their experiment that by using 20% SiC reinforcement on Al alloy based matrix, higher values of modulus of elasticity 68GPa achieved.

Huang et al. [42] did an experiment to observe the effects of TiN nano particles addition on the W–30Cu composites prepared by electroless plating and powder metallurgy route. For metallurgical route of sample making, they had chosen sintering temperature1300°C and soaking time 60 min in a flowing hydrogen atmosphere. They observed that addition of TiN nano particles 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 up to 25 wt. %.

Qureshi et al. [43] developed a theoretical model for the cold die compaction of powder materials. They proposed an expression relating the green density of the compact to the applied pressure. They observed from their experiment that the comparison between experimental and theoretical results shows a remarkable agreement for all the tested conditions. They conclude that the green density distribution will be more uniform for higher initial relative tap density of performs and lubricated powder shows relatively better densification than unlubricated samples. They also demonstrated that with increasing the particle size of the powder yielded better densification.

The densification and grain growth behavior of powder injection molded 316L stainless steelmicro–nanopowders were investigated by **Choi et al. [44]**. They observed the sintered density of the micro-nano powder 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.

Lu et al. [45] prepared (TiB + TiC +Nd₂O₃)/Ti based composites by powder metallurgical route. The composition of powder was selected according to stoichiometric amounts and three types of samples with different NdB₆ contents were prepared. Samples were sintered in a vacuum carbon furnace at 673K temperature. They observed that Reinforcements such as TiB, TiC and Nd₂O₃ are distributed uniformly in the titanium matrix.

Kurgan [46] did an experiment to study the effects of sintering atmosphere on microstructure, sintered density, grain morphology and mechanical properties of AISI 316L stainless steel. Nitrogen and argon were used as the sintering atmosphere. The effects of sintering atmosphere on microstructure of the sintered samples were carried out by using optical microscopy, and scanning electron microscope. They observed from their experiment that sample sintered in nitrogen atmosphere exhibits higher strength and hardness than that sintered in argon atmosphere.

Lis et al. [47] were focused on preparation of stoichiometric Ti_3SiC_2 powders by SHS in the Ti-Si-C system. They placed the powders into a graphite crucible in vacuum using graphite felt and ignited by resistance molybdenum coil. After ignition, a high-temperature front of reaction with temperatures from 1800°C to 2000°C propagated through the stem transformingit into product. They observed that a high chemical activity seems to be one of the characteristic features of ceramic covalent powder synthesized by SHS (combustion) method; such activity is manifested by their improved sinterability. They conclude from their experiment that high sinterability of SHS powders can result from reaction sintering phenomena in presence of inter metallic liquid phase (Ti-Si-C system) or low temperature molten compounds.

Vinoth et al. [48] carried out an experiment for 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 nano composites of AA2024 aluminum alloy matrix reinforced with high weight fractions of nano-metric SiC particles and Graphite particles were produced by blend– press–sinter methodology. Consolidation was carried out at 700Mpa pressure using uniaxial pressing and the Sintering processes was carried out at 530°C for 60 min. 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. They observed that nano composite with 5 wt. % Gr and 10 wt. % SiC showed the highest improvement in tribological performance which confirms that graphite particle was act as an effective solid lubricant for aluminium matrix composite.

Mishra et al. [49] reported the tribological behavior of aluminium alloy Al-6061 reinforced with silicon carbide particles (10% and 15 wt.% SiC) fabricated by stir casting process. The wear and frictional properties of the metal matrix composites was studied by performing dry sliding wear test using a pin-ondisc wear tester. It was evaluated from the results that the sliding distance (62.5%) has the highest influence on wear rate followed by sliding speed (37.5%) and applied load (1.25%). Similarly effect of applied load and sliding distance on coefficient of friction was 85.5% and 13.4% respectively. It is also reported that with increasing SiC content from 10% to 15%, wear resistance of composites increases due to formation a protective layer between the counter bodies.

Bodukuri et al. [50] reported the Al-SiC-B₄C metal matrix composite by powder metallurgy technique and evaluating mechanical properties. Three different combinations of compositions in volume fraction were 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. With increase in percentage of B_4C 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. [51] prepared SiC particulate-reinforced Al composites using powder metallurgy (PM) method and conventional atmospheric sintering. The effect of temperature on the density, hardness, strength, and microstructure of composites was investigated. Detailed failure behavior was analyzed. It was found that the segregation of SiC appeared at higher temperature. The highest microhardness of 80MPa occurred at 700 °C. The strength tended to increase with the increasing temperature due to the formation of Al₂Cu. Both ductile and brittle fracture features were observed.

Wang et al. [52] prepared TiB₂ particulate reinforced magnesium matrix composites by powder metallurgy. Magnesium metal matrix composites (MMCs) reinforced with 10, 20 and 30 vol.% TiB₂ particulates, respectively, were fabricated by powder metallurgy. The microstructure, porosity, hardness and abrasive wear behavior of the composites were evaluated. Micro structural 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 were increased by 41%, 106% and 181%, respectively. The results of abrasive wear tests showed the wear resistance of Mg MMCs is increased with the increasing of the reinforcement volume fraction. This was due to the strong particulate-matrix bonding and high hardness of the TiB₂ particulate.

Kuzenkova et al. [53] reported the sintering response of austenitic (316L) and ferrite (434L) stainless steel consolidated in conventional and microwave furnace. The cylindrical green compacts were prepared by uniaxialy pressure of 600 MPa and dimentions of 16 mm diameter and 6 mm average height. The green
compacts were sintered using conventional and microwave furnace. The conventional sintering of green compacts was carried out in a $MoSi_2$ 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 was provided at 500°C, 700°C and 1000°C. The sintering temperature selected was 1400°C.

Bolzoni et al. [54] did work on the effect of SiC content on the compaction behavior and properties of Al6061/SiC/Gr hybrid composite. In this experiment the powder mixture were cold pressed in six varying pressures (from 250 to 750MPa) and sintered. The results showed the SiC particles decrease the compressibility of the hybrid powders and improves the hardness of composites. The best wear resistance is achieved in the hybrid composite containing 20 vol% SiC particle. Also they reported that the compressibility of aluminum matrix is decreases by increasing the SiC content in the powder mixture. It is also found that composite containing 40 vol% SiC shows lower density due to SiC particles are not deformed and support a part of the applied pressure elastically. Moreover amount of pressure is also wasted because of the friction between particles. Therefore the transferred load to the aluminum (plastic phase) decreases and hence compressibility decreases.

Chapter 3

Experimental Procedure

3.1 Powders for development of MMCs composites

Four types of powders mixtures (for making samples) were used for the experiments. Purity and particles size of powder shown in Table 1.

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

Table 1 Specification of powders used for experiments.

Table 2 Composition of Steel AISI 304

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

Table 2 shows the composition of steel AISI 304.

3.1.1 Mixing

After the selection of powder mixing is done in a ball milling equipment (*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. Powder selected according to the stoichiometric of Eq. (1). And mixed in three different types of composition. As shown in Table 3. While mixing use binder (Zinc stearate) 2-3 wt.% of total powder.

Table 3 Samples Composition

Sample name	Blend of ceramic powder added	
	(in wt% of steel powder)	
S1	20	
S2	30	
\$3	40	

3.1.2 Compaction

After mixing, compaction is done by 25 tons manual pellet press manufactured by *Kimaya engineering* of all three types of mixture powder at different compaction pressure 740, 960 and 1180 MPa. The die set is used (1*3mm dia. Hardened alloy steel evacuable die set*) and height of sample is 6mm.

3.1.3 Sintering

After 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 15min to ensure evaporating of binder which added during mixing. Heating and cooling rate provided is 5° C/min.

3.2 Characterization of samples

The sintered pellets studied with various mechanical, physical and micro structural characterization tests. Physical and microstructure characterization of the pellets were studied using scanning electron microscopy (SEM) and Energy dispersive spectroscopy (EDS) by field emission scanning electron microscope (FESEM) and X-ray diffraction (XRD). Mechanical characterizations were investigated using scratch tester and tribological properties of pellets were evaluated using a linear reciprocating tribometer.

3.2.1 Physical characterization

3.2.1.1 Scanning Electron Microscope (SEM)

The microstructure and morphology of samples was observed in *Zeiss Supra-55* field emission scanning electron microscope (FESEM) on the polished surfacecondition. The sample has been polished with abrasive paper from 800 to 1200 grid and again with a polishing clothwith diamond solution from 1 to $.25\mu$. Cu was selected as target material. The SEM equipped by Energy Dispersion

Spectroscopy (EDS) was used to investigate the chemical composition of microstructures.

3.2.1.2 X-Ray Diffraction (XRD)

X-ray diffraction analysis (XRD) of the sintered pellets observed with Xray diffractometer (*Rigaku Smart Lab* 3 kW). Using Cu-Ka radiation (wavelength). With intensity scanner v/s diffraction angle between 0° to 80° (step size 0.01), voltage of 40KW and 30mA filament current. The data were later analyzed with X'Pert High score software (*Philips Analytical*B.V., Netherlands).

3.2.2 Mechanical characterization

To find out scratch hardness and fracture toughness, scratch tester (*DUCOM made Scratch TesterTR-101*) with Rockwell C diamond indenter (apex angle 120°, tip radius, 200µm, 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 (*FESEM: Zeiss Supra-55*).

3.2.2.1 Fracture Toughness

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 [55]. Fig. 6 and Fig. 7 show schematic diagram of parallelepiped scratching tool and conical scratching tool.

$$K_c = F_{eq} / \sqrt{2pa}$$

$$F_{eq} = F_T \qquad \alpha = 0$$

$$= \sqrt{\left(F_T^2 + \frac{3}{5}F_V^2\right)} \qquad \alpha > 0$$



Fig. 6 Parallelepiped Tool



3.2.2.2 Scratch Hardness

D/2 = Half of scratch width

Scratch tester (*DUCOM made Scratch TesterTR-101*) is used to measure scratch hardness of samples using the following formula [56]. Fig. 8 shows the schematic diagram of scratching by a conical indenter.



Fig. 8 Schematic diagram for evaluating scratch hardness

(Horizontal distance between axis of indenter and centerof formed ridge)

h= Indentation depth from the top surface of the sample + height of ridge

3.3 Linear reciprocating wear test

Linear reciprocating wear tests were carried out using a tribometer (*TR*-201-M3, DUCOM) to investigate friction and wear properties of samples. Cemented carbide (WC-6%Co) balls (φ 6) were used as counter body. The tests were performed with variable loads of 1.3kg, 2.3kg and 3.3kg for different samples. The tests were continued for 15 minute, frequency of sliding is 30Hz and track length of 4mm.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 was measured using *Taylor-Hobson, Surtronic 3+surface profilometer*, Specific wear rate was calculated using thefollowing equation,

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

Here, ω_d is the specific wear rate of the samples in mm³/(N·m), V is the volume of wear track in mm³, d is the sliding distance in m, L is the normal load in N., V wasdetermined 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 nomenclature

Table 4 shows the nomenclature of samples prepared for experiments.

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

Table 4 Samples nomenclature

Chapter 4 Result and Discussion

4.1 Scanning electron microscopy (SEM) analysis

Fig. 9 shows the FESEM micrograph of 20wt.% of ceramic in steel matrix composite sintered at 1400° C and soaking time 30 min. It is seen from the micrograph that reinforcement particles (TiB₂, SiC, and TiN) are uniformly distributed throughout in steel matrix. Fig. 10 shows the EDS spectra of S122 sample. The EDS spectra show the presence of Ti, N, Fe, and Si elements in composites.



Fig. 9 Scanning electron micrograph of sample S122



Fig. 10 Energy dispersive spectroscopy of sample S122 at spectrum 1



Fig. 11 Scanning electron micrograph of sample S222



Fig. 12 Scanning electron micrograph of sample S322

Fig. 11 and 12 shows the SEM micrograph of sample S222 and S322. It is notice from figure that the ceramic particles uniformly distributed in steel matrix and some voids and craters are also seen in the SEM micrograph. The presence voids and craters in the sample due to dislodgments of ceramic particle during the polishing operation. The randomly dispersed pores are also seen in these figures.

4.2 X-Ray Diffraction (XRD) Analysis

XRD analysis of composite with different weight proportion of ceramic powder blend mixed to steel powder has been carried out. The XRD spectra of samples S121, S221 and S321 are shown in Figs. 13, 14, and 15 respectively. The XRD spectrum shows the presence of strong peaks of TiB₂ and TiN. Compatibly weak peaks of SiC have been seen in XRD spectrum due to presence of less amount of SiC in composite. Fe and Cr peaks are also observed in XRD spectrum.



Fig. 13 X-ray diffraction pattern of sample S121



Fig. 14 X-ray diffraction pattern of sample S221



Fig. 15 X-ray diffraction pattern of sample S321

4.3 Analysis of coefficient of friction

4.3.1 Effect of sintering time

Fig. 16-18 shows the graph of coefficient of friction with respect to sliding distance. It is seen from Fig. 16 that the average value of coefficient of friction is 0.45 at soaking time 0 minute. Fig. 17 shows that at sintering time 30 minute, values of coefficient of friction is around 0.60 and in case of soaking time 60 min the average value of coefficient of friction shows 0.45 as shown in Fig. 18. It is seen from the average value of coefficient of friction that at 0 minute soaking time and at 60 minute soaking time the values of coefficient of friction is lower because at 0 minute soaking time leads to under sintering causes lower values of coefficient of friction and athigh soaking time (60 Minute) causes coarsening of particles that leads to decrease in coefficient of friction. At 30 min soaking time shows the maximum values of coefficient of friction.



Fig. 16 Variation of Coefficient of Friction with respect to Sliding Distance of samples S111, S211 and S311, soaking time 0 min. at a temperature of 1400°C



Fig. 17 Variation of Coefficient of Friction with respect to Sliding Distance of samples S121, S221 and S321, soaking time 30 min. at a temperature of 1400°C



Fig. 18 Variation of Coefficient of Friction with respect to Sliding Distance of samples S131, S213 and S331, soaking time 60 min. at a temperature of 1400°C

4.3.2 Effects of compaction pressure

Fig. 19-21 shows the effects of variation of compaction pressure (740MPa, 960MPa and 1180MPa) at a constant soaking time 30 min and sintering temperature of 1400°C. Wear test is performed at a load of 3.3 kg load. It is seen that by increasing compaction pressure density of compact increases resulted improvement in mechanical properties. But it is seen that coefficient of friction is not marginally change due to presence of hard ceramic particle, because the hard ceramic particles are not deformable and they support a part of the applied pressure elastically. Amount of pressure is also wasted because of the friction between particles.



Fig. 19 Variation of Coefficient of Friction with respect to Sliding Distance of samples S121, S221 and S321 at compaction pressure of 740MPa and sintering temperature 1400°C



Fig. 20 Variation of Coefficient of Friction with respect to Sliding Distance of samples S122, S222 and S322 at compaction pressure of 960MPa and temperature 1400°C



Fig. 21 Variation of Coefficient of Friction with respect to Sliding Distance of samples S123, S223 and S323at compaction pressure of 1180MPa and temperature of 1400°C

4.3.3 Effects of wear load

Fig. 22-24 shows the effects of wear load (1.3kg, 2.3kg and 3.3 kg) on coefficient of friction at constant soaking time 60 min and compaction pressure 1180MPa. It is seen from graph that with increasing wear load coefficient of friction increases due to several causes. In environmental condition at lower load the values of coefficient of friction shows lower value because of formation of oxide layer on the top surface of the composite sample. When counter body (cemented carbide ball) come in to the contact of sample surface at lower load, chances of fracture or dislodgements of particle is low that's way coefficient of friction is low but by increasing load coefficient of friction increases because of more engagement of asperities.



Fig. 22 Variation of Coefficient of Friction with respect to Sliding Distance of samples S133, S233 and S333 at wear load of 1.3 kg and temperature of sintering is 1400°C



Fig. 23 Variation of Coefficient of Friction with respect to Sliding Distance of samples S133, S233 and S333 at wear load of 2.3 kg and temperature of sintering is 1400°C



Fig. 24 Variation of Coefficient of Friction with respect to Sliding Distance of samples S133, S233 and S333 at wear load of 3.3 kg and temperature of sintering is 1400°C

4.3.4 Effects of composition

Fig. 25 shows the coefficient of friction of samples S132, S232, and S332 that contained 20, 30 and 40wt.% of ceramics in the steel matrix. It is observed from the values of Coefficient of friction, while increasing ceramic wt.% from 20% to 30% coefficient of friction increases because the composite consist of more wt.% of hard ceramic particles. It is also seen that while further increase in wt.% of ceramic particle coefficient of friction decreases because the excess amount of ceramic particle that leads to nonuniformalty in the matrix causes the reduction in coefficient of friction.



Fig. 25 Variation of Coefficient of Friction with respect to Sliding Distance of samples S132, S232, and S332 that contained 20, 30 and 40wt% of ceramics powder in steel matrix

4.4 Analysis of specific wear rate

4.4.1 Effects of sintering time

Fig. 26-28 shows the specific wear rate with respect to load. It is seen from Fig. 26 and Fig. 28 that the average Specific wear shows higher value at soaking time 0 minute and 60 minute because at 0 minute soaking time leads to under sintering causes lower values of specific wear rate and at high soaking time

(60 Minute) causes coarsening of particles that leads to increase in specific wear rate. At 30 min soaking time shows the minimum values of specific wear rate.



Fig. 26 Variation of specific wear rate with load of samples S111, S211 and S311,

0 min soaking time at sintering temperature 1400° C and compaction pressure

740MPa



Fig. 27 Variation of specific wear rate with load of samples S121, S221 and S321, 30 min soaking time at sintering temperature 1400°C and compaction pressure

740MPa



Fig. 28 Variation of specific wear rate with load of samples S131, S231 and S331, 60 min soaking time at sintering temperature 1400°C and compaction pressure

740MPa

4.4.2 Effects of compaction pressure

Fig. 29 shows the specific wear rate of samples S121, S221, and S321 at different compaction pressure 740MPa, 960MPa and 1180MPa at a constant sintering temperature (1400°C) and soaking time (30 min). It is seen from the Fig. 29 and Fig. 30 that specific wear decreases by increasing compaction pressure from 740MPa to 960MPa 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 Fig. 31 that at 1180MPa 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.



Fig. 29 Variation of specific wear rate with load of samples S121, S221 and S321 at compaction pressure 740MPa and sintering temperature 1400°C



Fig. 30 Variation of specific wear rate with load of samples S122, S222 and S322 at compaction pressure 960MPa and sintering temperature 1400°C



Fig. 31 Variation of specific wear rate with load of samples S123, S223 and S323 at compaction pressure 1180MPa and sintering temperature 1400°C

4.4.3 Effects of composition

From Fig. 32, it is seen that with increasing amount of ceramic from 20wt% to 30 wt% specific wear rate decreases because it contained optimum amount of ceramic particle. Literature also suggest around wt.% of ceramic containing in a composite show lower value of specific wear rate. It is also noticed that sample S331 shows higher specific wear rate while it contained maximum 40wt% of ceramics powder particles because of excess amount of ceramics particles cannot be hold by matrix so it causes the increasing in specific wear rate.



Fig. 32 Variation of specific wear rate with load of samples S131, S231, and S331 that contained 20, 30 and 40 wt% of ceramics powder in steel matrix

4.4.4 Scratch hardness results

Fig. 33-35 shows the values of scratch hardness at different compaction pressure and different sintering time.

Fig. 33 shows the scratch hardness value at 0 minute soaking time. It is seen that scratch hardness values of sample containing 20wt.% of ceramics at different compaction pressure varies from 5.63GPa to 7.58GPa, values of sample containing 30wt.% of ceramics varies from 6.78GPa to 9.45GPa and for sample containing 40wt.% of ceramics is 5.90GPa to 7.81GPa. It is seen that maximum scratch hardness achieved at 740MPa compaction pressure for all three types of sample S111, S211, S311 because after optimum compaction pressure, by increasing the compaction pressure leads to crake formation in composite. It is also observed that S211 shows maximum scratch hardness as compare to S111 and S311 because it contain optimum wt% of ceramics.



Fig. 33 Scratch Hardness of sample S111, S112, S113, S221, S211, S213, S311, S312 and S313 at soaking time 0 min and compaction pressure 740MPa, 960MPa and 1180MPa.



Fig. 34 Scratch Hardness of sample S121, S221, S321, S122, S222, S322, S123, S223 and S323 at sintering time 30 min and compaction pressure 740MPa, 960MPa and 1180MPa.

Fig. 34 shows that scratch hardness of sample containing 30wt.% of ceramics in steel matrix is varies from 9.67GPa to 10.10GPa which is much higher compare to sample containing 20wt.% and 30wt,% ceramics in steel matrix It is seen from Fig. 33 and Fig. 34 that with increasing the soaking time values of scratch hardness increases.





Fig. 35 shows values of scratch hardness at soaking time 60 min. Maximum value of scratch hardness at Fig. 35 shows compatibly lower than the values from Fig. 34 due to coarsening of grain structure.

4.4.5 Fracture toughness

Fig. 36 shows the values of fracture toughness of samples prepared at soaking time 0 min with different compaction pressure such as (740, 960 and 1180) MPa. It is seen that fracture toughness values of sample contain 20wt.% of ceramic blend in steel matrix varies between 3.23MPam^{1/2} to 4.73MPam^{1/2} respectively, for sample contain 30wt.% of ceramic blend in steel matrix values varies between 4.66MPam^{1/2} to 5.95MPam^{1/2} respectively and for sample contain 40wt.% of ceramic blend in steel matrix the values varies between 3.38MPam^{1/2}

to 5.32MPam^{1/2} respectively. It is observed that fracture toughness of sample contain 30wt.% of ceramic blend in steel matrix is maximum at 740MPa compaction pressure because it contained optimum wt% of ceramics powder compare to sample contain 20wt.% and 30wt.% of ceramic blend in steel matrix.

Fig. 37 shows the fracture toughness values of sample S121, S211, S321, S112, S222, S322, S123, S223 and S323 at 30 min. soaking time. It is seen that fracture toughness values shows 7.78MPam^{1/2} for sample containing 30wt.% of ceramics at compaction pressure of 960MPa. A higher value of fracture toughness achieved for samples prepared at 30 min. soaking time compare to the 0 min soaking time.

From Fig. 38 it is seen that sample prepared at soaking time 60 min. fracture toughness decreases compare to 0 min. and 30 min. soaking time because higher soaking time leads to coarsening of grain results decreases in mechanical properties.



Fig. 36 Fracture toughness of sample S111, S112, S113, S221, S211, S213, S311, S312 and S313 at soaking time 0 min. and compaction pressure 740MPa, 960MPa and 1180MPa.



Fig. 37 Fracture toughness of sample S121, S211, S321, S112, S222, S322, S123, S223 and S323 at soaking time of 30 min. and compaction pressure 740MPa, 960MPa and 1180MPa.



Fig. 38 Fracture toughness of sample S131, S231, S331, S132, S231, S332, S133, S233 and S333 at soaking time 60 min and compaction pressure 740MPa, 960MPa and 1180MPa.

Table 5 shows the properties (Coefficient of friction, Specific wear rate, Scratch Hardness, Fracture Toughness) of ceramics composite samples prepared by powder metallurgical method.

Sample nomenclature	Coefficient of friction	Specific wear rate (10 ⁻⁸ mm ³ /N-m)	Scratch Hardness (GPa)	Fracture Toughness (MPam ^{1/2})
S111	0.57	4.75	7.58	3.23
S112	0.53	5.06	5.65	4.73
S113	0.58	4.16	5.63	3.8
S121	0.38	5.88	7.43	3.44
S122	0.5	1.84	7.06	4.13
S123	0.55	6.78	6.3	3.58
S131	0.45	5.67	6.95	4.15
S132	0.65	4.49	5.77	5.05
S133	0.55	4.47	7.93	3.06
S211	0.63	4.31	9.45	5.95
S212	0.58	3.74	6.78	4.56
S213	0.67	5.46	7.32	4.66
S221	0.35	2.22	10.1	6.16
S222	0.6	2.58	9.89	7.78
S223	0.48	6.08	9.67	6.63
S231	0.52	3.76	9.14	4.3
S232	0.55	3.15	7.67	2.6
S233	0.6	3.85	9.69	6.84
S311	0.47	2.65	7.81	3.38
S312	0.47	5.95	5.9	3.83
S313	0.51	7.33	6.99	5.32
S321	0.45	4.01	7.81	2.38
S322	0.4	6.35	8.17	7.14
S323	0.45	4.07	7.64	5.03
S331	0.47	6.62	4.68	2.38
S332	0.4	6.7	7.51	4.66
S333	0.51	3.85	6.51	4.39

Table 5. Mechanical and tribological properties of samples

Sample	Coefficient of friction	Specific Wear rate	Scratch Hardness	Fracture Toughness
		$(10^{-8} \text{mm}^3/\text{N-m})$	(GPa)	$(MPam^{1/2})$
CMC Coating comprises of same phase as in ceramic composite	0.41	2.20	12.61	5.92

Table 6. Properties of CMC Coating comprises of same phase as in ceramic composite

Table 7. Properties of sample S221

Sample nomenclature	Coefficient of friction	Specific Wear rate (10 ⁻⁸ mm ³ /N-m)	Scratch Hardness (GPa)	Fracture Toughness (MPam ^{1/2})
S221	0.35	2.22	10.10	6.16

Table 6 shows the properties (Coefficient of friction, Specific wear rate, Scratch Hardness, Fracture Toughness) of CMC Coating comprises of same phase as in ceramic composite samples.

From Table 7 It observed that sample S221 which contain 30wt.% of ceramics in steel matrix, shows nearly same properties as CMC coating comprises of same phase present in ceramic composite.

Following conclusion drown from the experiments are-

- Sample S221 shows nearly same properties as CMC coating comprises of same phase present in ceramic composite.
- Mechanical properties like scratch hardness and fracture toughness is increases when ceramic wt% increases up to 30% and again decreases when contained of ceramic increases.
- Tribological properties (coefficient of friction and specific wear rate) also seen optimum at 30wt% of ceramic in steel matrix.
- Compaction pressure for better mechanical properties and tribological properties is 960MPa.
- Sintering time at highest temperature 1400°C is 30min shows the better results compared to 0 min and 60 min.

Future Scope

- To find the exact situation as present on coating interlayer samples need to be treated with laser and required to perform more no of experiments.
- To find exact composition between 20wt% and 30wt% more no of samples are required to make between these compositions.
- By varying the amount of ceramic wt% exact mechanical and tribological properties can be finding as presents in coating.

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