PREDICTION OF ADHESIVE STRENGTH, DEPOSITION EFFICIECNY AND WEAR BEHAVIOUR OF PLASMA SPRAY COATING OF LOW GRADE MINERAL ON MILD STEEL AND COPPER SUBSTRATE BY SOFT COMPUTING TECHNIQUE

THESIS SUBMITTED
IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE DEGREE OF
MASTER OF TECHNOLOGY

In
Metallurgical & Materials Engineering

By

MANISH BAGWAN

DEPARTMENT OF METALLURGICAL & MATERIALS ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY,
ROURKELA, INDIA
MAY, 2013

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Prof. S C Mishra

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Declaration

I hereby declare that, the work which is being presented in this thesis entitled “Prediction of Adhesive Strength, Deposition Efficiency and Wear Behavior of Plasma Spray Coating of Low Grade Mineral on Mild Steel and Copper Substrate by Soft Computing Technique” in partial fulfillment of the requirements for the award of M.Tech degree, submitted to the Department of Metallurgical & Materials Engineering, National Institute of Technology, Rourkela, is an authentic record of my own work under the supervision of Prof. S.C. Mishra. I have not submitted the matter embodied in this thesis for the award of any other degree or diploma to any other university or Institute.

Date : May 2013

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CERTIFICATE

This is to certify that, the thesis entitled "Prediction of Adhesive Strength, Deposition Efficiency and Wear Behavior of Plasma Spray Coating of Low Grade Mineral on Mild Steel and Copper Substrate by Soft Computing Technique" being submitted to the National Institute of Technology, Rourkela by Mr. Manish Bagwan, Roll no. 211MM1359 for the award of M.Tech degree in Metallurgical & Materials Engineering, is a bonafide record of research work carried out by him under my supervision and guidance.

The candidate has fulfilled all the prescribed requirements. The Thesis which is based on candidate's own work has not been submitted elsewhere for award of any degree.

In my opinion, the thesis is of standard required for the award of M.Tech degree in Metallurgical & Materials Engineering.

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Manish Bagwan

CONTENTS

Abstract

Currently emerging technologies contains some of the most prominent ongoing advances, innovations and developments in a variety of engineering field to advance surface property by using modern technology. Because for higher productivity and efficiency across the entire spectrum of manufacturing and engineering industries has ensured that most modern day components/ parts are subjected to day by day increasing harsh environments in routine operations. All the Critical industrial components of the machines are, therefore, prone to more rapid degradation as the parts fail to withstand the aggressive operating conditions and this has been affecting the industry's economy to a very high extent. The prime objectives are to develop essential surface properties with an economical process. Today the investigation explores the coating potential of industrial wastes. Fly-ash emerges as a major waste from thermal power plants. It mainly comprises of oxides of iron, aluminium, titanium and silicon along with some other minor constituents. Fly-ash premixed with illmenite and quartz which are minerals of low cost available in plenty are excellent for providing protection against resistant to erosion and abrasive wear.

In this wide research world Plasma spraying is gaining acceptance for development of quality coatings of various materials on a wide range of substrates. Use of the industrial wastes of such kind as coating material minimizes the plasma spray coating deposition cost, which posed to be the major obstacle to the wide spread purpose due to high cost of the spray grade powders.

Fly-ash+quartz+illmenite (weight percentage ratio: 55:25:20) is deposited on copper and mild steel substrates by atmospheric plasma spraying, at operating power levels ranging from 10 to 20kW and after that characterization of the coatings is carried out. The properties/ quality of the coatings depend on the operating condition, process parameters and materials used. The plasma spraying process is controlled by interdependent parameter, co-relations and individual effect on coating characteristics. The size of the particles of raw material used for coating is characterized using Malvern Instruments a Laser particle size analyzer. Coating interface adhesion strength is calculated using a method of coating pull, confirming to ASTM C-633 standard. Deposition efficiency is a key factor that determines the techno economics of the process, evaluated for the deposited coatings. Coating thickness of the polished cross section is measured, using an optical microscope.

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Chapter 1

INTRODUCTION

- Research Background
- Objectives of Research

Chapter 1
Introduction

1.1 RESEARCH BACKGROUND

Currently emerging technologies contains some of the most prominent ongoing developments, innovations and advances in various engineering field to improve surface property by using different type of modern technology. Because for higher efficiency and productivity across the entire spectrum of engineering and manufacturing industries has ensured that most modern day components are subjected to increasingly harsh environments. The critical components of industries are, therefore, prone to more rapid degradation as the components fail to withstand the severities of aggressive operating conditions and this has been taking a heavy toll of the economy of industries. In large number of cases i.e. by impervious environments and also by high relative motion between mating surfaces, extreme temperatures, corrosive media and cyclic stresses, the accelerated deterioration of components and their ultimate failure has been traced to damage of the components/ materials. So, now a day, it is necessary to develop research on new materials for fabrication. So this study's mission is to develop quality assurance in coating surfaces and systems. Furthermore, the study is concerned with the enhancement of the life time and the products quality as well as with failure analysis and damage prevention of new coating components and materials. As a result of the above, the concept of incorporating engineered surfaces by various surface modification techniques, capable of combating the accompanying degradation phenomena like fatigue, corrosion and wear to improve components performance, durability and reliability has gained increasing acceptance.

Treatment of Surface is an established provider of advanced material processing and coating technologies for a wide range of applications in the Aerospace, Oil & Gas, Semiconductor, missile, power, electronic, biomedical, Automotive, textile, petroleum, machine tools, construction industries, petrochemical, chemical, steel, power and cement. The development of the suitable high performance coating on the component fabricated using a suitable higher mechanical strength alloy/alloy, offers a promising method of meeting both the surface and property requirements of almost all imagined applications. Protective coatings are deposited as a barrier between surfaces of the component and the aggressive environment that it is exposed to during operation is now globally acknowledged to be an attractive means to significantly hamper the actual component by acting as the first line of defence. Along with the traditional one, the newer surfacing techniques are exceptionally suited to modify a wide range of engineering properties. There are several properties that can be modified by adopting the surface engineering approach include electrochemical, electrical, electronic, magnetic/acoustic, tribological, mechanical, thermo-mechanical, biocompatible and optical properties.

Surface treatments typically are adding material, removing material or chemically varying the surface. Surface treatments are widely used in most industries/ firms for providing improved surface properties of component of the machines. Some categorized surface treatments are:

a) Anodizing

It is an electrolytic passivation process used to increase the thickness of the natural oxide layer on the metal part surfaces. In this process the part to be treated forms the anode electrode of an electrical circuit. Some typical anodizing processes are Sulphuric acid anodizing, Phosphoric acid anodizing, Chromic acid anodizing, Organic acid anodizing, etc. on the galvanic series Aluminium metal is on the anodic side. Its position is similar to magnesium and zinc, i.e. it gets readily oxidized. The oxide on aluminium is naturally corrosion resistant, abrasion resistant, very hard, an insulator and very tenacious. In natural form the oxide film thickness on aluminium is not more than 0.50 microns.

b) Electroplating (galvanizing)

Electroplating is often also called electro-deposition. Electro-deposition is a process of producing coating metallic, on a surface by the action of electric current. The deposition of a metallic coating on an object is achieved by applying a negative charge on the object to be coated and immersing it into a solution with a salt of the metal to be deposited on the surface (i.e., the object to be plated is made the cathode of an electrolytic cell). The metallic ions in the salt carry a positive charge and are thus deposited to the object as they reach the negatively charged object which is to be electroplated.

c) Cladding

Application of one material over another to provide a layer/ skin intended to control the permeation of weather elements or for aesthetic purposes. Cladding is not necessarily providing a waterproof condition. This control element may only serve to safely direct water or wind in order to control run-off and prevent permeation into the main structure.

d) Diffusion coating (Nitriding, Phosphating, Carburizing, Cyaniding, Boronizing, Chromizing, etc.)

This Process is a thermally activated high temperature oxidation / wear resistance/ corrosion coating for iron, cobalt and nickel based metals which are at severe operating conditions. It provides a chemically bonded firm coating which acts as a diffusion barrier against oxygen and other elements into the substrate.

e) Polymer Film

Among the wide range of insulating material with implanted metal nanoparticles, thin or thick insulating layers have raised special interest. Thin film Polymers are especially suitable as host materials for nanoparticles, while their physical properties and chemical structure can be very different.

f) Shot Peening

A cold working process used to produce a compressive residual stress layer and modify metals mechanical properties. It involves impacting a surface with shot (ceramic particles, round metallic or glass) with forces sufficient to create plastic deformation on metal surfaces. While peening a surface spreads plastically, causing change in the surface mechanical properties. It is often called for in aircraft repairs to relieve tensile stresses built up in the grinding process and replace them with beneficial compressive stresses. Depending on the shot quality, part geometry, part material, shot material, shot intensity, shot coverage and shot peening can increase fatigue life up to 1000%.

g) Lubricants

Failure mechanisms include high adhesion, high contact resistance, contact erosion and melting/ shorting. But by addition of lubricant these failure mechanisms can be hinder much more time. For example, it was found that the rollers with lubricating coating resulted in lowest boundary friction than the rollers with the hardest DLC coatings.

h) Flame hardening

In this process heat is applied by a high temperature flame followed by water quenching jets. It is usually applied from medium to large size components such as sprockets, slide ways of machine tools, bearing surfaces of shafts, large gears, axles, etc. In flame hardening a defined surface area directly impinged by an oxy-gas flame. The result of the hardening is controlled by following four factors, i.e. (i) the duration of heating; (ii) the design of the flame head; (iii) the composition of the metal being treated and (iv) the target temperature to be reached.

i) Thermal spraying

These technologies are specializes in coating solutions using leading edge technology and equipment for Flame Sprayed Coatings, Plasma Spraying, Electric Arc Spraying and High Velocity Oxy Fuel Coatings.

j) Induction hardening

This process is a non-contact heating technique which utilizes the principle of electromagnetic induction to produce heat inside the work piece surface layer. When a conductive material is placed into a strong alternating magnetic field electrical current can be made to flow in the steel thereby creating heat due to the I2R losses from the material. The current generated flows primarily in the surface layer, the depth of this layer being uttered by the frequency of the alternating field, the heat time, the permeability of the material, the surface power density and the diameter of the bar or material thickness.

From all of the above techniques, thermal spraying is popular for its durability, wide range of applicability and adhesion of coating with the substrate. Surface modification technologies have grown promptly, both in terms of finding better solutions and in the number of technology variants available, to offer a wide range of cost and quality.

Surface engineering has been developed largely on account of the fact that it is a discipline of science and technology that is being gradually relied upon to meet all the key modern day technological requirements: material savings, environmental friendliness, enhanced efficiencies, etc. [1, 2]. The selection of coating material is a vital factor which needs to be considered carefully in relation to the substrate and its application method. The selection of wrong coating material can, not only affect the long term reliability but can cause enormous difficulties with both processing and costs. While the ‘Material Considerations’ section below is very important to finding the correct coating and is also important to find a coating chemistry which meets specific application requirements. One of the most critical decisions of a Process Engineer is the selection of the correct choice of coating material (lacquer). Criteria for selection must be based on answering many questions [2], which includes:-

- What is to be protected against? (e.g. moisture/ chemicals/ wear/ environment)
- What temperature range at which the equipment/ parts are operated?
- For an instance, does it need to match the coefficient of expansion of the component?
- How much the coating material resist to the expected attack?
- How can the material be reworked easily once applied?
- How fast the material be applied and dried?
- What procedure and equipments are required to achieve the suitable coating quality (repeatability and uniformity)?
- What is the Price of the coating material and cost of the process?
- What are the chemical, physical and electrical requirements for the coating material itself?
- On which substrate, material is to be coated? (In account of range of adhesion strength/ coating deposition/ coating thickness etc.)
- Electrical, chemical, and mechanical compatibility with the parts and substances to be coated?
- Quality of the material supplier (different fly-ash composite material manufacturers will not make equal quality of material).

Answers will determine the appropriateness of a particular Process, production, material and commercial issues.

Thermal spraying techniques are surface modification process in which melted (or heated) materials are sprayed onto a substrate surface. The feedstock (coating precursor) is heated by electrical (plasma or arc) or chemical means (combustion flame). Plasma spraying process is a thermal spraying technique, which is a relatively specialized high temperature industrial process that utilizes electrically generated plasma to heat and melt the feedstock material. The process is capital intensive and requires significant electrical power. Deposition thickness gives a wide range from a few micrometers (µm) up to several millimeters by use of a variety of feedstock materials, including metals and ceramics. The feedstock material is normally presented to the plasma torch typically, in the form of a powder or wire. This feedstock melts rapidly within the plasma gun, where the typical operating temperature is ~10,000°C (18,000°F). This technique is mostly used to produce coatings on different structural materials and provides protection against corrosion, erosion and wear, high temperatures, to change the appearance and also improves electrical properties of the surface. Plasma spraying is extensively used in a wide range of industries like aerospace, nuclear energy as well as conventional industries like chemicals, textiles, plastics and paper to develop a suitable surface coating to improve the component life span at operating environment mainly wear resistant coatings in crucial components.

During the last decade, a large number of investigations have been carried out for new development of plasma spray coating material by using industrial waste and low-grade ore [3]. Flyash is a finely divided powder generated as a solid waste in huge quantities in thermal power plants. A small fraction of flyash is used in the development of high value products. New ways of utilizing flyash are being explored in order to minimize the plant wastage and provide a safeguard to the environment. Flyash is a fine powder which can be used as refractory material in industry. Now-a-days flyash composite has a number of useful applications [4]. However, increase in the demand of its applications; have led to the development of new fly-ash composite coatings. According to recent investigations composite fly-ash coatings can obtain high corrosion resistant, in addition to increased wear resistance. Some of these recent reports concerning the development and surface properties of this type of fly-ash composite coatings are presented below.

This investigation describes about processing of plasma spray coatings and characterization/evaluation of substrate surface properties i.e. microstructure, adhesion strength, deposition efficiency, thickness, hardness and wear resistance. Fly-ash+quartz+illmenite with weight percentage ratio 55:25:20 is deposited on mild steel and copper substrates by atmospheric plasma spraying. Spraying carried out at various operating power levels ranging from 10 to 20 KW. Coating-substrate interface adherence strength is evaluated using coating pull out method. Deposition efficiency is an important factor that determines the techno economics of the process which is evaluated for the deposited coatings. Hardness measurement is done on the polished cross section of the samples using Leitz Micro-Hardness Tester. Coating thickness is measured on the polished cross-sections of the samples, using an optical microscope. Coating surface & interface morphology is studied with Scanning Electron Microscope. Erosion wear behaviours of these coatings are studied by “Air Jet Erosion Test Rig”.

1.2 OBJECTIVES OF RESEARCH

The objectives of the present investigation/ experiment are as follows:

- To explore the coating potential of fly ash+quartz+illmenite on metal substrates by plasma spraying.
- To develop plasma sprayed coating from fly ash+quartz+illmenite on metal substrates and to find out deposition efficiency, thickness and wear properties etc.
- Micro-structural characterization (surface and interface morphology) to evaluate the soundness of the coatings.
- Measurement of adhesion strength and hardness of the coatings.
- Sustainability of the coatings against wear and erosion with solid particle erosion test.
- To analyze the experimental results using some statistical techniques so as to identify the significant factors/interaction parameter set by which one can get better plasma surface property.

Chapter 2

Literature Survey

- Introductory Statement
- Surface engineering
- Techniques of surface modification
- Thermal spraying
- Plasma spraying
- Industrial applications of plasma spraying
- Wear
- Types of wear
- Symptoms of wear
- Recent trends in material wear research
- Wear resistant coatings
- Utilization of fly ash as wear resistant coatings
- Erosion wear of ceramic coatings

Chapter 2
Literature Survey

2.1 INTRODUCTORY STATEMENT

The literature survey of the broad subject of this chapter describes namely the development of surface modification technology, which explains various coating techniques specially plasma spraying, their characteristics and the raw spray materials. It gives a short account of the coating deposition process by plasma spray technique. Under various conditions, the performances of wear resistant coatings have been reviewed critically. It also presents a review of the wear i.e. Symptoms of wear, types of wear and recent trends in metal wear research together with erosion wear behaviour of ceramic coatings.

2.2 SURFACE ENGINEERING

In a broad diversity of industry the role of surface coating have been imparted its increasingly importance because longer service life and higher energy efficiency are expected more strongly for various industries. Thermal spraying holds an inimitable position in the spectra of surface modification technologies as it can provide thick coatings over ~100µm over a large area at a very high application rate compared with other coating processes such as CVD, PVD and electroplating [11]. Surface engineering can be outlined as: "the treatment of the surface regions and surfaces of a material to let the surface to attain functions that are distinct from those functions demanded from the material (bulk material)" [12]. A difficult decision for an engineer, when selecting materials for structural components in the modern high technology field such as space power, nuclear, oil exploration etc. operating in the extremely hostile environment of temperature, pressure, gas flow and corrosion media. The properties desired at the surface are different from those at the bulk of the components. Hence lead to the use of surface coating. Erosion, wear, corrosion, fatigue and creep can cause environmental degradation of the surface over time. Surface engineering involves varying the properties of the surface to ease the degradation. It can be accomplished by making the surface robust to the environment so that it will be used [13]. Surface Engineering is the name of the discipline - surface modification technique is the philosophy behind it.

Surface treatment may be required to:

- Improve resistance to indentation and wear, erosion, (wear surfaces of machinery, and rolls, shafts, cams, and gears slide ways in machine tools).
- Reduce adhesion (electrical contacts).
- Improve thermal insulation.
- Improve corrosion & oxidation resistance (sheet metals for automotive or other outdoor uses, gas turbine components, and medical devices).

- Control friction (sliding surfaces on tools, bearings, dies and machine ways).
- Improve stiffness and fatigue resistance (bearings and multiple-diameter shafts with fillets).
- Improve lubrication (surface modification to retain lubricants).
- Rebuild surfaces on worn components (worn tools, dies, and machine components).
- Improve surface roughness (appearance, dimensional accuracy, and frictional characteristics).
- Impart decorative features, colour or special surface texture.
- Increase product life span.

By fulfilling the above criterion, surface engineering techniques are being used in the steel, aerospace, textile, petroleum, petrochemical, cement, power, automotive, missile, power, biomedical, electronic, machine tools, chemical and construction industries. Approximately all types of materials including ceramics, metals, composites and polymers can be coated on similar or dissimilar materials. To explain the matter some example can be taken. A) Tungsten carbide cobalt composite is extensively used as cutting tool material, and is well known for its wear resistance and high hardness. If a thin coating of TiN is applied on to the WC-Co insert, its capability increase significantly [13]. B) The surface of erratic stainless steel modified with Mo by laser treatment. It is found that, pitting potential increased by approximately 130 mV than that of untreated stainless steel [14]. In fact a cutting tool, is subjected to a high degree of abrasion and TiN is capable of resisting abrasion. Moreover TiN is brittle, but the relatively tough core of WC-Co composite protects it from fracture. Therefore through surface modification process we bring together two (or more) materials by the appropriate method and take advantage of the qualities of both [16].

2.3 TECHNIQUES OF SURFACE MODIFICATION

Today a large number of commercially available technologies are present in the industrial scenario. An overview of such technologies is presented below (Fig 2.1):

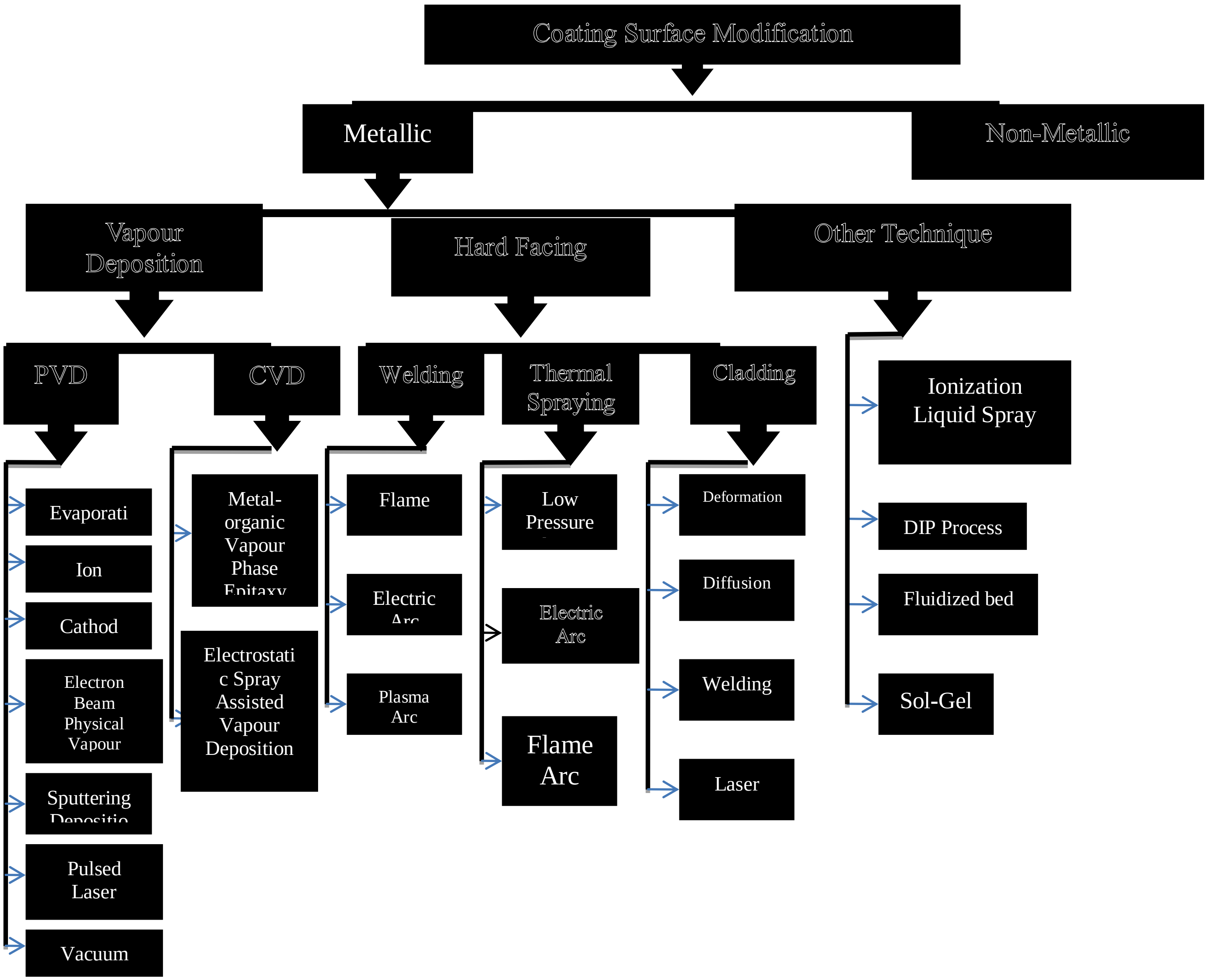


Figure 2.1 Summarized plot of surface modification techniques.

2.4 THERMAL SPRAYING

In the early 1900s, M.U. Schoop was the first scientist to explore the possibility that a stream of metallic particles formed from molten metal might be used to produce coatings. But the thermal spraying technologies expanded in the 1970s due to development of thermal plasmas, and the increasing demand of high temperature and wear resistant materials and coating systems [17]. In the 1990s thermal spraying was demandingly available and had become a standard tool for improving surfaces in about all industries. Thermal spraying is the use of a material (consumable) to a substrate by melting into droplets and impinging the molten droplets on a substrate to form a continuous/ pulsed coating [18]. Thermal spray consumables can be alloys, metallic, ceramic or polymeric substances. Some material can be sprayed it can be melted by the heat source employed and does not undergo degradation during heating [19]. The spray techniques that have been used to deposit coatings for protection against aggressive atmosphere are listed below, as reviewed in Heath et al. [20]:

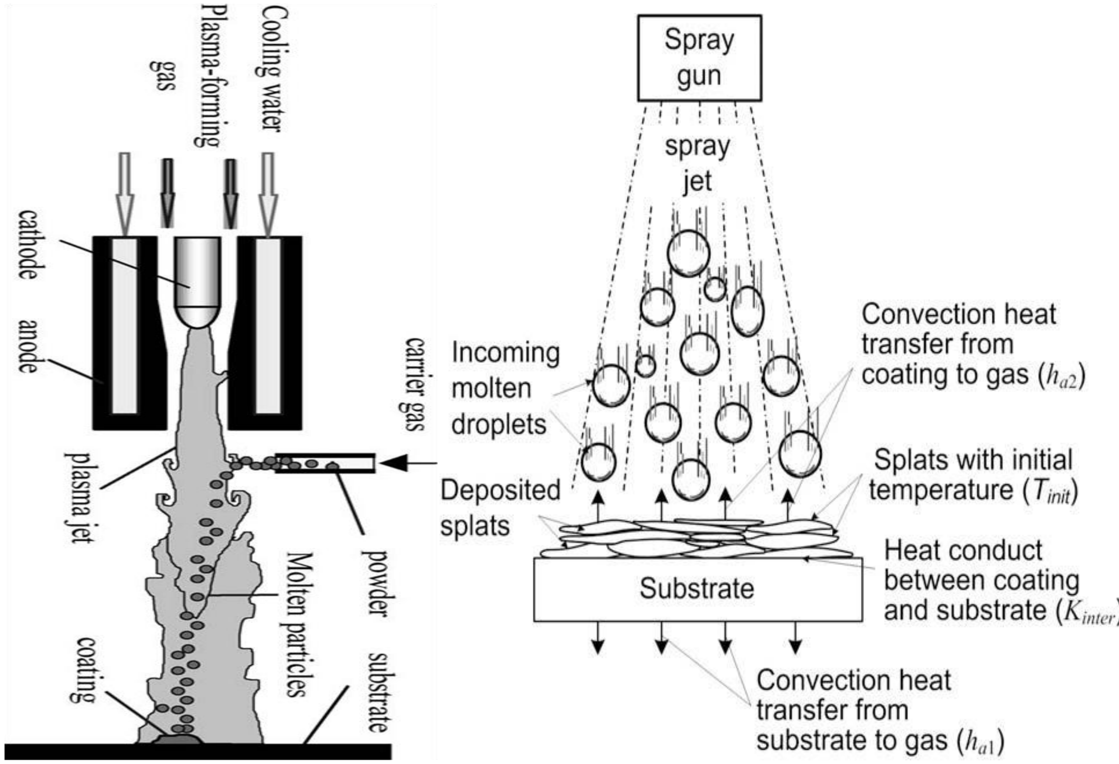
- Flame spraying with a wire or powder particles.
- Electric arc wire spraying
- Plasma spraying
- High Velocity Oxy-Fuel (HVOF) spraying
- Spray and fuse

These processes are basically differentiated from each other on the basis of particle speed, flame temperature and spray atmosphere [21]. Thermal spray technology is uniquely important to an ever-increasing engineering community, for its (i) improved spray footprint definition versus wide spray beam; (ii) high throughput versus competitive techniques; (iii) significantly improved process control; (iv) lower cost-per-mass of applied material, together with overall economical economics. Thermal spray coatings have been formed for at least 50 years, but the last decade has seen essential revolution in the capability of the technology to produce truly high performance coatings of a great range of materials on many different substrates [22].

The varieties of advantages of thermal spraying technology listed are as follows:

- Choice of wide variety coating materials ceramics, cermets, metals, alloys, and carbides.
- Thick coatings can be useful at high deposition rates.
- Coatings are mechanically bonded to the substrate - can often spray coating materials which are metallurgically incompatible with the substrate, e.g. materials with high melting point than the substrate.
- Components can be sprayed with little or no pre- and post- heat treatment and component distortion is minimal.
- Parts can be rebuilt quickly and at low cost and usually at a fraction of price of a replacement.
- By using a premium material for the thermal spray coating, the components coated can live longer than new parts.
- Thermal spray coatings can be applied both manually and automatically.

The main principle behind thermal spraying is to melt material feedstock (wire or powder) to accelerate the melt to impact on a substrate where rapid deposit builds and solidification up occur. Therefore a heat source and a means of accelerating the material are required. This is presented in Fig 2.2 [23].



(a) **(b)**

Figure 2.2 (a) Schematic diagram showing plasma spraying and **(b)** Schematic physical-thermomechanical description of plasma spray process.

The nature of bonding at the coating-substrate interface depends on metallurgical/ mechanical bonding. This is significant feature of thermal spraying. Another aspect of thermal spraying is that the substrate surface temperature seldom exceeds 2000C. Stress related distortion problems are also not so considerable. The spraying action is achieved by the rapid energy transfer of combustion gases to the molten droplets or by a separate supply of compressed air. There are two essential ways of generating heat required for melting the consumables. They are (i) combustion of a fuel gas and (ii) high energy arc processes [24, 25], categorize in Table 2.1. Processes available for thermal spraying have been developed specifically for a purpose and fall into two categories-high and low energy processes. The key processes and their energy sources are shortened in Table 2.2 [26].

Table 2.1 Basic way to generate heat for melting spray powder.

Thermal Spray Process	Gas combustion process	Oxy-fuel/wire
		Oxy-fuel/powder
		Detonation gun
		HVOF
	Arc process	Electric Arc
		Plasma Arc

Table 2.2 Thermal spraying processes.

Processes		Energy sources	Different Nomenclature
Low energy process	Flame spraying	Chemical	Oxyfuel gas-powder spraying
			Oxyfuel gas-wire spraying
			metallizing
	Arc spraying	Electrical	Electric arc spraying
			Twin-wire arc spraying
			metallizing
High energy process	Plasma spraying	Electrical	Atmospheric plasma spraying
			Vacuum plasma spraying
			Low pressure plasma spraying
			Water stabilized plasma spraying
			Inductive plasma spraying
	Detonation flame spraying	Chemical	D-gun
	High velocity oxyfuel spraying	Chemical	HVOF spraying
			High velocity oxygen fuel spraying
			High velocity flame spraying
			High velocity air fuel

2.5 PLASMA SPRAYING

Plasma spraying is one of the most widely used thermal spraying technique which finds a lot of applications due to its versatility of spraying a wide range of materials from metallic to non metallic and hence more suitable for spraying of high melting point materials like refractory ceramics material, cermets etc [27,28]. A schematic diagram of plasma spray process is shown in Fig 2.3. This process is part of thermal spraying, in which finely divided metallic and non-metallic materials are deposited in a molten or semi-molten state on a prepared substrate [29]. In the fifties, the plasma torches were developed to test materials at high enthalpies for simulated vehicles. Subsequently in the late fifties and early sixties, the first attempts were reported using plasma torches for spraying of primarily refractory materials. Almost any material can be used for plasma spraying on almost any type of substrate. This flexibility is almost certainly one of the major reasons for the rapid development of this technology [30]. The high temperatures enable the use of coating materials with very high melting points such as ceramics, cermets, alloys and refractory. Materials can be processed as long as there is a temperature difference of at least 300K between the melting temperature and decomposition or evaporation temperature [31]. Among other key features of plasma spraying are the formation of microstructures with equiaxed grains fine and noncolumnar the ability to produce homogeneous coatings that do not change in composition with thickness and length of deposition time, the ability to process materials in virtually any environment (e.g., air, reduced-pressure inert gas, high pressure, under water) [32]. Applications for plasma spraying include corrosion, erosion, temperature and abrasion resistant coatings and production of monolithic and near net shapes, which at the same time take advantage of the rapid solidification process. Powder of glassy metals can be plasma sprayed without changing their amorphous characteristics. High temperature superconductive materials have also been deposited by the plasma spray technique. A new application of plasma spraying is in producing hydroxyapatite coatings onto the stems of orthopaedic endoprostheses [33].

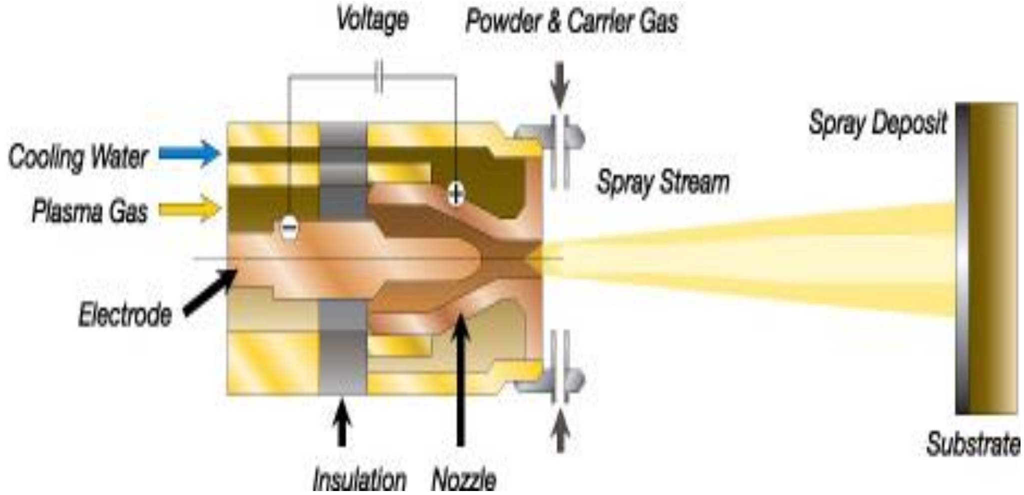


Figure 2.3 conventional plasma spray process.

In this technique an arc is created between tungsten cathode and a copper anode. Generated plasma gas is is forced to pass through the annular space between the electrodes. The gas undergoes ionization in the high temperature environment resulting plasma, while passing through the arc. In the plasma arc temperature can be as high as 2,000°C to 20,000°C (as shown in Fig 2.4) and is capable of melting whatever thing.

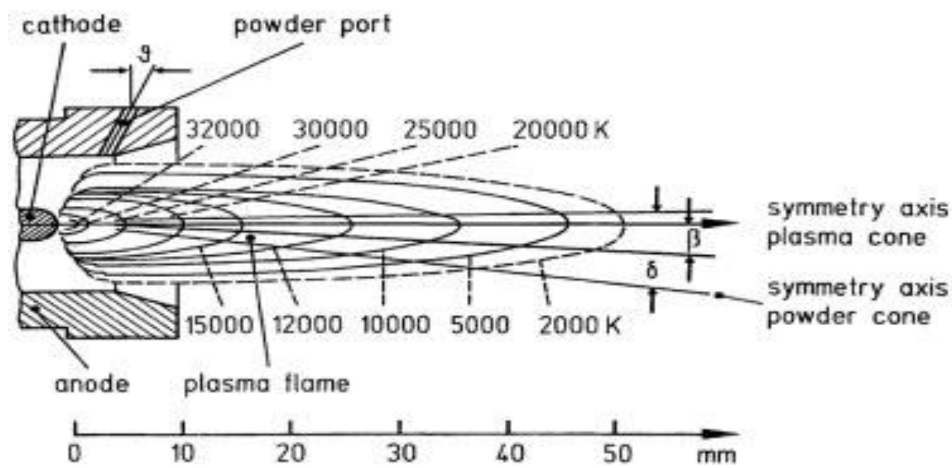


Figure 2.4 Temperature Distribution and geometry of the plasma jet.

The ionization is achieved by collisions of the neutral molecules of the gas with electrons of the arc. The plasma protrudes out of the electrode encasement in the form of a flame. Electrodes are water cooled. The raw coating material in the powdered form is poured into the flame with necessary feed rate. The powders melt immediately by gain of plasma energy and momentum and then rush towards the target to form a thin deposited layer. In this way the coating builds up layer by layer [34]. Elaborate cooling arrangement is required to protect the spray system from surplus heating. The equipment consists of the following modules [35]*:*

- **The plasmatron:** It is the device in which the plasma reaction takes place and which houses the electrodes and. It has the shape of a gun and it is connected to the water cooled power supply cables, gas supply hose and powder supply hose.
- **The power supply unit: U**sually plasma arc works in a low voltage (40-70 Volts) and high current (400-800 Amps), DC ambient. The available AC power of 3 phases, 440Volts must be transformed and rectified to costume the reactor. It is taken care of by the power supply unit.
- **The powder feeder:** The powder is kept inside a hopper. An individual gas line directs the career gas that fluidizes the powder and carries it to the plasma arc. Accurately the flow rate of the powder can be controlled.
- **The coolant and water supply unit:** It circulates water into the plasmatron, the power cables and the power supply unit. Units capable of supplying refrigerated water are also available.
- **The control unit:** Important functions (gas flow rate control, current control etc.) are performed by the control unit. It also comprises of relays and solenoid valves and other interlocking arrangements essential for safe running of the equipment. For e.g. an arc can only be started if the coolant supply is on and water pressure and flow rate is adequate.

An arrangement of plasma spraying equipment is shown in Fig 2.5.

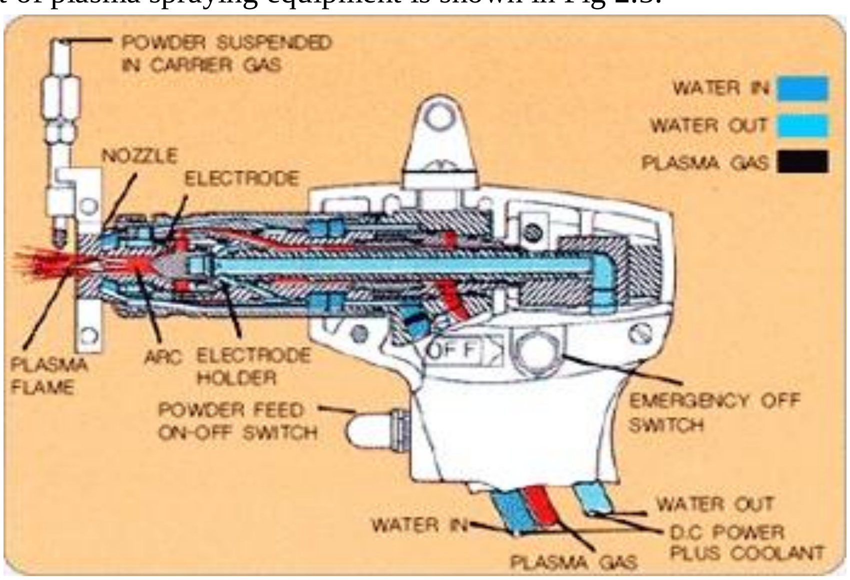


Figure 2.5 Plasma Spraying arrangement.

The main advantages of the plasma spraying process have been presented by Heath et al. [36] and others as follows:

1. Very flexible in coating material selection and optimization for specific resistance to corrosive environments and particle abrasion/erosion.
2. Coating systems (multi-layer or functionally graded) can be used.
3. Unique alloys and microstructures can be obtained with thermal spraying which are not possible with a wrought material. These consist of continuously graded composites and corrosion resistant amorphous phases.
4. Costs of the coating solution are normally significantly lower than those of a highly alloyed bulk material; thermal spray coatings are especially interesting for their cost/performance ratio.
5. Thermal spray coatings additionally offer the potential of on-site application and repair of components, given a sufficient ease of access for the sprayer and his equipment.
6. Microstructure with fine, equiaxed grains and without columnar boundaries are formed.
7. Produces deposits that do not change in composition with thickness (length of deposition time).
8. Can change from depositing a metal to a continuously varying mixture of metals ceramics (i.e. functionally graded materials).
9. High deposition rates (>4kg/h).
10. Fabricates freestanding forms of virtually any material or any materials combination.
11. Process materials in virtually any environment e.g. high or reduced inert gas pressure, air, etc.

However, thermal spraying is favoured to achieve optimum results. Amongst the thermal spray coating processes, plasma spraying is reported to be a flexible technology that has been successful as a reliable cost-effective solution for many industrial issues by Fauchais et al. [37]. The high temperatures of plasma spray processes allows the deposition of coatings for applications in areas of wear protection liquid and high temperature corrosion and also special applications for thermal, biomedical purposes and electrical. Plasma-sprayed metallic coatings are used in high-temperature applications e.g. diesel engines, aircraft engines and land-based gas turbines to protect the component from oxidation and corrosion [38].

2.5.1 Requirements for Plasma Spraying

Roughness of the substrate surface:

Surface roughness of substrate provides better mechanical interlocking. Better is the surface roughness, better is the adhesion strength. Generally by grit blasting technique a rough surface is created. The grits are kept inside a hopper and compressed air is supplied at the bottom of the hopper. The compressed air stream into a hose takes the grits float and ultimately directed to an object kept in front of the exit nozzle of the hose. The grits used for this purpose highly angular in nature, are irregular in shape, and made up of hard material like silicon carbide alumina etc. Small craters are created upon impact on the surface by localized plastic deformation and finally yield a very rough and highly worked surface. The roughness obtained is resolute by shot blasting parameters, i.e. shot size, material, shape and air pressure, between nozzle and the job, stand-off-distance angle of impact, substrate material etc [39]. The effect of shot blasting parameters on the adhesion of plasma sprayed alumina has been studied [40]. Mild steel serves as the substrate material. The surface roughness and the parameters listed above are proportionally with adhesion. A major time lapse between shot blasting and plasma spraying causes a marked decrease in bond strength [41].

Bond coat:

Materials like ceramic cannot be sprayed directly onto metals because of large difference between their thermal expansion coefficients (α). Ceramics have a much lower value of "α" and hence undergo much less shrinkage as compared to the metallic base to form a surface in compression. When compressive stresses exceed a certain limit, the coating gets removed. To alleviate this problem of appropriate material, usually metallic of in-between a value is plasma sprayed onto the substrate followed by the plasma spraying of ceramics. Bond coat may render itself of use for metallic topcoats as well. Molybdenum is a typical example of bond coat of metallic topcoats. Molybdenum reacts very well to the steel substrate and develops a somewhat rough top surface ideal for the topcoat spraying. The choice of bond coats always depends upon the application, for example, in wear purpose, an alumina and Ni-Al top and bond coats combination are used [42]. In thermal barrier application, CoCrAlY or Ni-AI bond coat [43] and zirconia topcoat are accepted. Ceramic coatings are subjected to hertzian loading deform elastically and the metallic substrate deforms plastically. During acceptance, elastic improvement in the coating takes place, whereas for metallic substrate a permanent set has previously taken place. Owing to this elastoplastic inequality the coating tends to spall off at the interface. A bond coat can reduce this inequality as well.

Cooling water:

Distilled water was used for cooling function. Normally a little volume of distilled water is recirculated into the gun and it is cooled by an exterior water supply from a large tank. Sometime water from a large exterior tank is pumped directly into the gun [44].

2.5.2 Process Parameters in Plasma Spraying

In plasma spraying parameters are interrelated with each other, which determine the degree of particle melting, deposition efficiency and adhesion strength of the powder. Deposition efficiency is the ratio of amount of powder deposited on substrate to the amount fed to the gun as raw material. A detailed listing of these parameters and their effects are reported in the literature [45]. Some important parameters and their roles are listed below:

Arc power:

It is the electrical power drawn by the consumable/non-consumable arc. The power is injected into the plasma gas, which obtains energy from plasma stream. Part of the energy of power is dissipated as radiation and also by the gun cooling water. Arc power shows the mass flow rate of a given powder that can be effectively melted by the arc with appropriate contact time. Deposition efficiency progresses to a certain extent with an increase in arc power, since it is related with an enhanced melting of particles [46]. However, increasing power beyond a certain limit may not cause a substantial enhancement. On the contrary, a complete particle melting is achieved; a higher temperature of gas may prove to be harmful. Because at some point vaporization may take place, which lowers the deposition efficiency.

Plasma gas:

Generally Nitrogen or Argon doped with about 10% Hydrogen or Helium is used as plasma gas. The main constituent of the mixture of gas is called primary gas and the minor is called the secondary gas. The unbiased molecules are subjected to the electron bombardment resulting in their ionization. Both enthalpy and temperature of the gas increase as it absorbs energy. Since nitrogen and hydrogen are diatomic gases, they first undergo division followed by ionization. Thus, the need for higher energy input to enter into the plasma state is needed. This energy rises the enthalpy of the plasma. On the other hand, the mono-atomic plasma gases, i.e. helium or argon, approach a much higher temperature in the normal enthalpy range. Good heating capability is expected from them for such required high temperature [47]. In addition, hydrogen followed by helium has a very high specific heat, and therefore is capable of attaining very high enthalpy. When argon is doped with helium a quite narrow spray cone is obtained which is especially useful for spraying on small targets.

Carrier gas:

Usually the primary gas itself is used as a carrier gas. The flow rate of the career gas is an important factor. If the flow rate is very high then the powders might escape the hottest region of the jet and a very low flow rate cannot convey the powder effectively to the plasma jet (as shown in Fig 2.6) [45].There is an optimum flow rate for each powder at which the fraction of unmelted powder is minimum and hence the deposition efficiency is maximum.

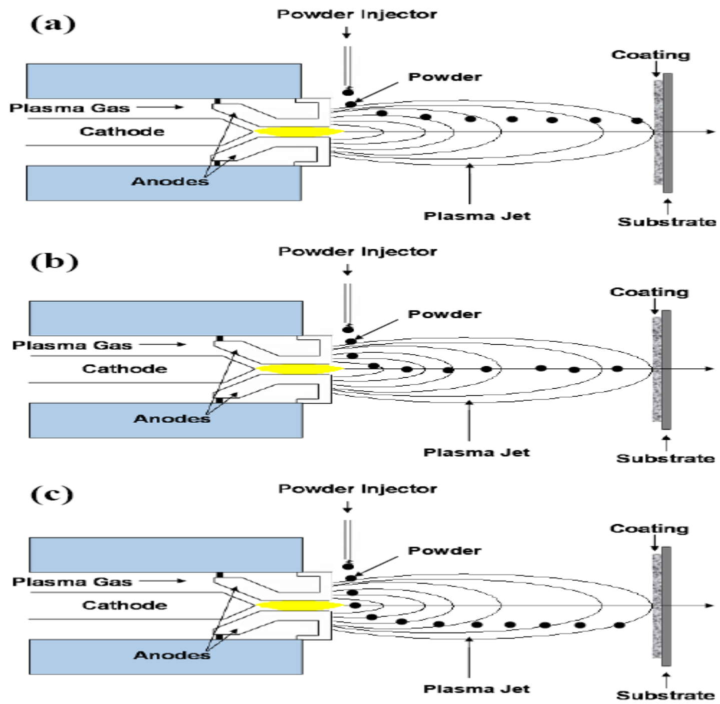


Figure 2.6 Carrier Gas Flow Rate a) too low b) correct c) too high [35].

Mass flow rate of powder:

Ideal mass flow rate for each powder has to be determined. Spraying with a lower mass flow rate keeping all other conditions constant results in under utilization and slow coating buildup. On the other hand, a very high mass flow rate may give rise to an incomplete melting resulting in a high amount of porosity in the coating. The un-melted powders may bounce off from the substrate surface as well keeping the deposition efficiency low [45].

Powder related variables:

These variables are powder size, shape and size distribution, phase composition, processing history etc. They constitute a set of extremely important parameters. For example, in a given situation if the powder size is too small it might get vaporized. On the other hand a very large particle may not melt substantially and therefore will not deposit. The shape of the powder is also quite important. A spherical powder will not have the same characteristics as the angular ones, and hence both could not be sprayed' using the same set of parameters [48].

Stand-0ff-distance (Spray Distance):

It is the distance between the tip of the gun and the substrate surface (shown in Fig 2.7). A long distance may result in freezing of the melted particles before they reach the target, whereas a short standoff distance may not provide sufficient time for the particles in flight to melt and may erode the substrate surface [41, 45]. A larger fraction of the un-melted particles go in the coating owing to an increase in stand-off-distance.

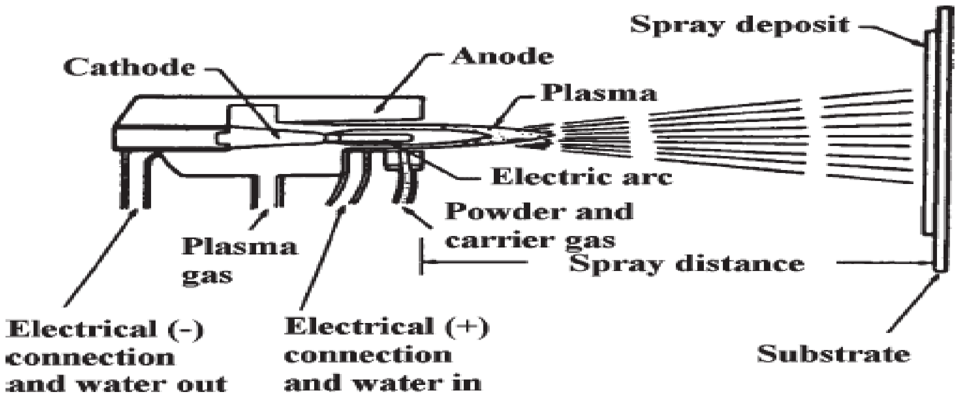


Figure 2.7 Schematic presentation of electrode arrangement and spraying distance.

Spraying angle:

Angle is one of an important factor during spraying. It is responsible for splat formation. Different angle chosen by considering different material (ductile/ brittle substrate). The influence of spraying angle on the cohesive strength of chromia, zirconia 8wt% yittria and molybdenum has been investigated, and it has been found that the spraying angle does not have much influence on the cohesive strength of the coatings [49]. SEM examination of splat morphologies obtained from impacting droplets at substrate with different angle of (a) 0°, (b) 10°, (c) 20°, (d) 30°, (e) 40°, (f) 50°, (g) 60° are shown in Fig 2.8.

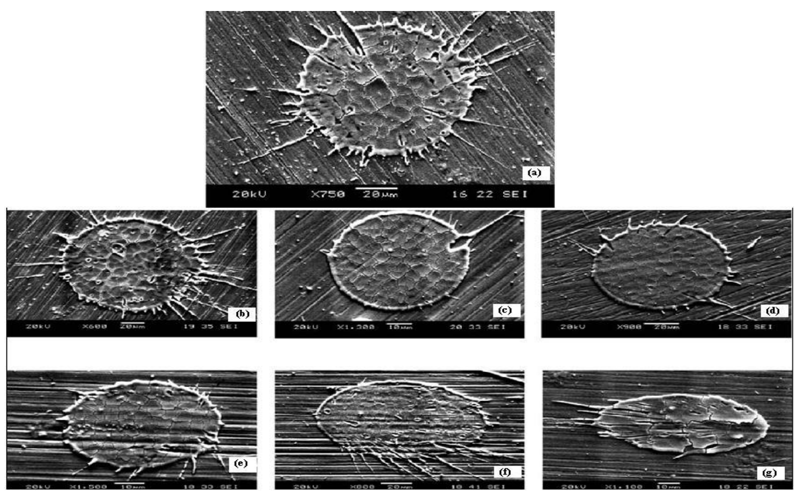


Figure 2.8 SEM examination of splat morphologies obtained from impacting droplets at substrate inclinations of (a) 0°, (b) 10°, (c) 20°, (d) 30°, (e) 40°, (f) 50°, (g) 60° [41].

Substrate cooling:

During a continuous spraying, the substrate might get heated up and may develop thermal stresses related distortion accompanied by a coating peel-off. This is especially true in situations where thick deposits are to be applied. To reduce the substrate temperature, it is kept cool by an auxiliary air supply system. In addition, the cooling air jet removes the unmelted particles from the coated surface and helps to reduce the porosity.

Angle of power injection:

Coating Powders can be injected perpendicularly, coaxially or obliquely in to the plasma jet. The residence time of the powders material will vary with injection angle for a given carrier gas flow rate. The residence time will influence the degree of melting of a given powder. For example, to melt high melting point materials a long residence time needed and hence oblique injection may prove to be better. The angle of injection is found to influence the cohesive strength and adhesion strength of the coatings [19].

2.5.3 Mechanism of Coating Formation in Plasma Spraying Process

Plasma spray is formed by the impact of a stream of particles from nozzle striking the substrate surface, the major controlling factors which influenced by the structure of a particular coating are the velocity, temperature and size distribution of the incident particles. Ideally all the surface striking particles would be completely molten. Unmolten particles may bounce off reducing the deposition efficiency and partly melted particles are incorporated within the deposit modifying its microstructure and properties [50]. Coatings are formed by the build up of successive layers of molten droplets which flatten and solidify on impact to give lamellar microstructure. When a liquid droplet strikes the surface at low velocity, it flattens to a disc (shown in Fig 2.9) [51] which then come to the equilibrium shape of spherical cap to form a cone and the spreads again to the final equilibrium shape determined by the static surface tension forces (shown in Fig 2.10). At high impact velocities the thin sheet of liquid becomes unstable and disintegrates at the edge into many small droplets i.e. splashing occurs. Its cooling rate then rapidly increases by conduction from molten particle to surface of the substrate. The cooling rates achieved are of the order of 10^6-10^7 $Ksec^{-1}$ [52]. P. Fauchais and co-worker also investigated on coating generation and predicted a model for calculating the splat-quenching rate [28]. It was observed that some metastable phases are formed during cooling, like γ-alumina rather than α-alumina which was explained on the basis of nucleation kinetics, i.e. γ- alumina was easily nucleated because of lower interfacial energy between crystal and the liquid and at sufficiently rapid cooling rates, the metastable form is retained at room temperature [53, 54]. Mechanical behavior of the coatings is limited to the degree of contact between the lamellae within the coatings (cohesion strength) and between the lamellae and the substrate (adhesion strength) rather than the nature of bonds in regions of good contact. This study was made on alumina coatings. The low apparent area of contact may be due to entrapped gases and other asperities between the impinging droplet and the substrate [55]. So surface grit blasting and cleaning of the substrates is necessary for better bonding between coating and the substrate [56].

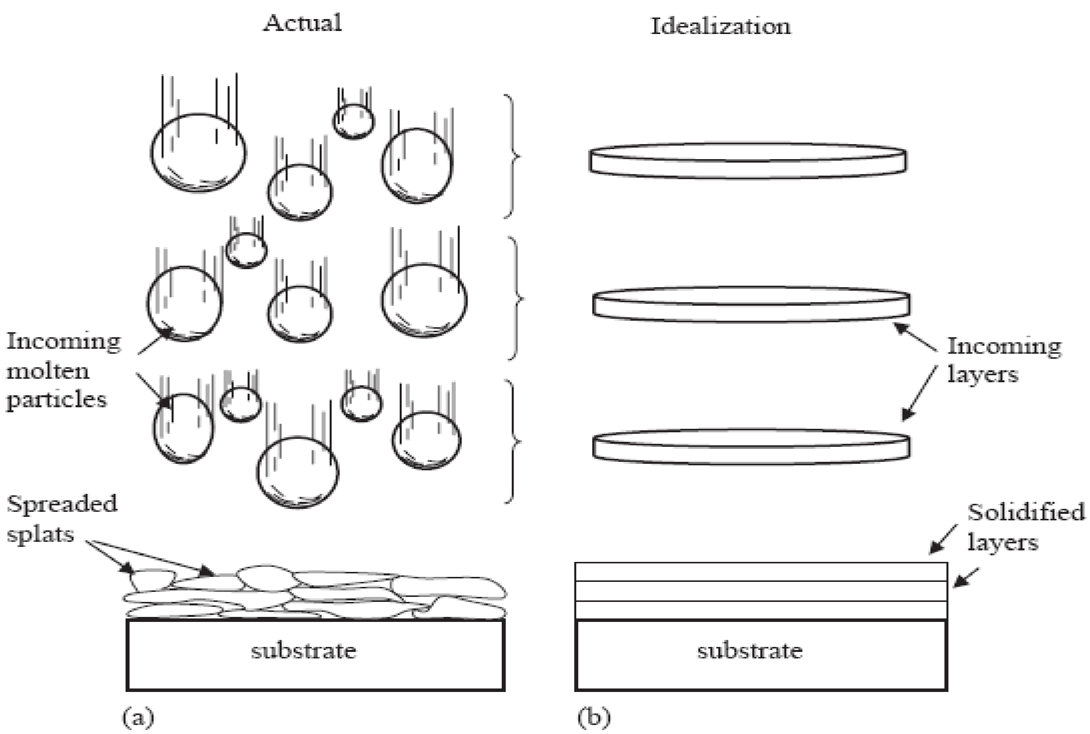


Figure 2.9 Schematic of the (a) physical plasma spray process and (b) its idealization for modelling [15].

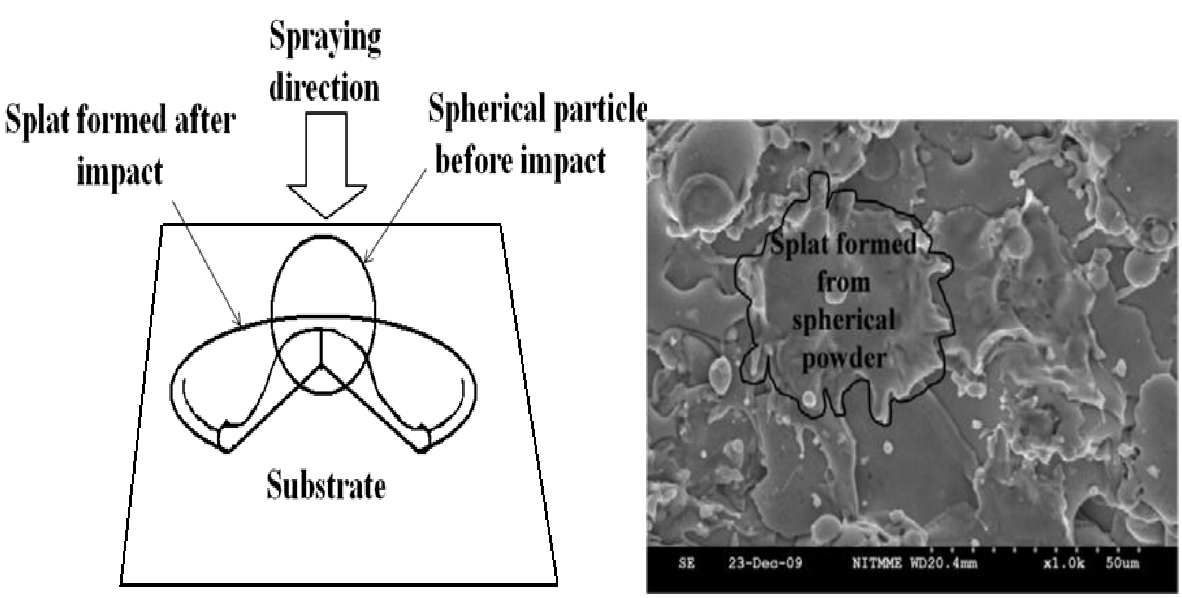


Figure 2.10 Splat formations after the impact of the spherical powder during spraying.

2.6 INDUSTRIAL APPLICATIONS OF PLASMA SPRAYING

There has been a gradual increasing in the number of applications of plasma sprayed coatings. Availability of hardware and adaptability of the technique are the main cause of this growth. This technique has been successfully applied to a wide range of industrial technologies from aerospace industry to biomedical industry [51]. Some of the typical applications are given below:

Steel Industry

In the steel working industry plasma surfacing roller used to handle very heavy thermal loads of hot steel. In additional, slag from steel production must be reckoned with, and in zinc production, corrosive attack from the molten zinc. Plasma coating have been qualified for use on both new parts and repair applications in steel production equipments [28, 49].

Aircraft Industry

Aircraft jet engine parts are subjected to serve mechanical, thermal stresses and chemical. A jet engine has a number of construction nodes where plasma coating is employed with much success in order to protect them. Some typical plasma sprayed parts are face of the blower box, compressor box and disc, fuel nozzles, guide bearing, blades, combustion chambers etc. [6]. Plasma spray is used to replace hard chromium plate is that of aircraft landing gear components.

Automotive industry and the production of combustion engines:

Plasma sprayed coatings used in automotive industries endure higher working pressure and temperature with improve in good friction properties, wear resistance, resistance against burn-off and corrosion due to hot combustion products and resistance against thermal loading. Some of the several applications developed for the automotive industry at the Slovak Academy of Sciences (SAV) in Bratislava are spraying torsion bars with aluminium coatings against corrosion. The plasma spraying technology is introduced in the production of gearshift forks for gearboxes in fiat car factory and on the critical parts of big Diesel engines [48]. The cylinder bores of the engines are coated by means of a special rotating plasma gun manipulator, which can apply the coating to the interior of the small bores with a wear resistant surface.

Medical Industry

For strong and durable anchoring of orthopaedic implants such as artificial hip joints, surface finish is of great importance. Plasma spray coatings applied using the vacuum, are purposely sprayed with a much fissured surface that allows the bone to grow into it. There are plasma coatings that act as a biocompatible titanium coating or bioactive hydroxy apatite coatings, which actively accelerates the growth of the natural bone into the surface of the prosthesis [16, 26].

Gas Turbine industry

Sprayed coatings are used in both stationary and flight gas turbines in many different places and for many different functions [36].

Electrochemical industry:

In the electromechanical and computer industries the electrically conductive Al, Cu, W and the semi-conductive and insulating ceramic layers are widely used. Some contacts of electrodes, e.g. the spark gaps of nuclear research equipment, are produced of massive tungsten. Modern electrodes can replace such electrodes with a sprayed tungsten coating about 0.5mm thick. This electrode ensures short- time passages of 300,000A current with a life of several hundred switching [45, 37].

Hydraulic industry

The range of possible applications in this field of Hydraulic machines is very extensive, mainly in water power plants, in production and work of pumps, where many parts are subjected to combined effects of wear, corrosion, erosion and cavitations [42].

Rolling mills

The wear resistant coatings are used in Rolling mills and pressing shops to renovate the heavy parts of heavy-duty machines whose replacement would be very costly [43]. Several applications in this field are presented herewith:

- Rolling strand journals being repaired by giving a coating layer of stainless steel. Blooming roll mill journal renovated with a NiCrBSi layer.
- Gears of rolling mill gearbox being renovated by a wear resistance coating.
- Conveyer rollers in plate production with zirconia based refractory coatings.
- To repair a rolling mill slide and the plungers of a forging press a hard wear resistance is applied.

Foundry mills

Heat resistant plasma coating is widely used for foundry and metallurgical equipment where molten metal or very high temperatures are encountered. This equipment includes the sliding plugs of steel ladles with alumina or zirconia coatings. Oxygen tubes, cast iron moulds in continuous casting of metals are also employed plasma spraying [23].

Chemical plants:

The base metal of machine parts is subjected to different kind of wear and corrosion continuously in contact with chemical reagents. In these cases plasma coatings are applied to protect the base metal. They can be used for various bearing surfaces, tubes, burners, blades, shafts, parts of cooling equipments etc [9].

Textile industry:

For the first time, Czechoslovakia textile industry employed Plasma spraying technique. Plasma spraying has replaced the conventional technologies of anodization, chrome plating and chemical surface hardening. Advantages of this technique are for different critical machinery parts**:** Different thread guiding & distribution rollers, distribution plates, ridge thread brakes, driving & driven rollers, tension rollers, thread brake caps, gallets, lead-in bars etc. High wear resistance coatings are required on machinery parts which are in contact with synthetic fibers. For this purpose especially Al_2O_3+ 3% TiO_2, Cr_2O_3, WC + Co, Al_2O_3 + 13% TiO_2 are applied [21, 17]. These coatings with hardness ranging from 1800 - 2600 HRV are extraordinarily dense, have high wear resistance and provide excellent bonding with the substrate. Plasma spraying has following advantages in textile industries:

- Replacement of worn out parts is minimized and hence reduces the idle times.
- Physical and mechanical properties of fibers are improved.
- Revolution speed of these lighter parts can be increased.
- Shelf life of the textile machinery parts with plasma sprayed coating last 5 to 20 times longer than parts coated by chrome plating or another classical technique.
- Economic savings are realized considerably by substituting heavy steel or cast iron parts with aluminium or durable ones with wear- resistant coatings.

Paper and printing industry:

The machinery in the paper and printing industry is usually quite large and is subjected to considerable wear from the sliding and friction contact with the paper products. Affected machinery parts are typically Paper drying rolls, filters, sieves, roll pins, printing rolls, tension rolls and other parts of printing machines. Spraying of oxide layers carried out for its economical solution.

Here oxide layers composed of Al_2O_3 with 3 to 13 % additions of TiO_2, Cr_2O_3 or MnO_2 are applied. Cast iron rolls are typically first sprayed with NiCr 80/20, 50 μm thick and then over it 0.2mm thick Al_2O_3 + 13% TiO_2 layer is coated [27]. The special advantages are mentioned below:

- Ensures corrosion resistance of rolls i.e. the base metal
- Resistance of oxide layers against printing inks extends the life of machine parts
- Production cost is reduced considerably
- Coating resulted to the so-called "orange peel" phenomena, surface finishing obtainable that prevents paper foil, dyes etc. from sticking and allows their proper stretching.

Glass industry:

Molten glass rapidly wears the surface of metal when that comes in contact with it. In order to protect the metal tools, plasma sprayed coatings are made on to it [37].

2.7 WEAR

Wear is a natural process of a material in which damage/deterioration of its surface occurs. Wear may be defined as damage to the solid surface caused by the removal or displacement of material by the mechanical action of a contacting solid, liquid or gas which occurs as a natural consequence when two surfaces in relative motion interact with each other. Widely varied wearing conditions causes wear of materials. It may be due to surface damage or removal of material from one or both of two solid surfaces in a sliding, rolling or impact motion relative to one another. In 1940 Holm [47] starting from the atomic mechanism of wear, calculated the volume of substance worn over unit sliding path. Different types of wear (Tribology) have been investigated by different researcher by taking different materials [45-38]. In these investigations various wear theories are taken in which Physico-Mechanical characteristics of the materials and the physical conditions (i.e. the resistance of the rubbing body and the stress state at the contact area) are taken in to consideration. Wear of metals depends on many variables for which wear investigation programs must be planned systematically. It should be understood that the real area of contact between two solid surfaces compared with the apparent area of contact is invariably very small, being limited to points of contact between surface asperities. The load applied to the surfaces will be transferred through these points of contact and the localized forces can be very large. Wear is not an intrinsic material property but characteristics of the engineering system which depend on load, temperature, speed, hardness, the environmental conditions and presence of foreign material [27]. During relative motion, material on contacting surface may be removed from a surface, may result in the transfer to the mating surface, or may break loose as a wear particle. The wear resistance of materials is related to its microstructural characteristics may take place during the wear process and hence it seems that in wear research emphasis is placed on microstructure [38].

2.8 TYPES OF WEAR

In most basic wear studies dry friction has been investigated where the problems of wear have been a primary concern to avoid the influences of fluid lubricants. Dry friction is defined as friction under not intentionally lubricated conditions but it is well known that it is friction under lubrication by atmospheric gases, especially by oxygen [89]. A fundamental scheme to classify wear was outlined by Burwell [9], include five distinct types of wear, namely (1) Abrasive (2) Adhesive (3) Erosive (4) Surface fatigue (5) Corrosive.

2.8.1 Abrasive Wear

Abrasive wear or abrasion account for most failures in any industrial equipment, which can originate from of the two rubbing surfaces, rubbing against each other. It can defined as the wear that is caused by the displacement of material from a solid surface due to hard particles or protuberances sliding along the surface and cutting grooves on the softer surfaces. In sliding mechanisms, abrasion can arise from the existing asperities on one surface (if it is harder than the other), from the generation of wear fragments which are repeatedly deformed and hence get work hardened for oxidized until they became harder than either or both of the sliding surfaces, or from the adventitious entry of hard particles, such as dirt from outside the system. Two body abrasive wear occurs when one of the harder surface cuts material away from the second of less harder. Abrasives can act as in grinding where the abrasive is fixed relative to one surface or as in lapping where the abrasive tumbles producing a series of indentations as opposed to a scratch. According to the recent tribological survey, abrasive wear is responsible for the largest amount of material loss in industrial practice [19].

2.8.2 Adhesive Wear

Adhesive wear can be defined as wear due to localized bonding between contacting solid surfaces leading to material transfer between the two surfaces or the loss from either surface [22]. In this wear it is necessary for the surfaces to be in intimate contact with each other. To obstruct for adhesion wear it is necessary for two surfaces to held apart by lubricating films, oxide films etc. Fretting wear is also this type of wear mechanism.

2.8.3 Erosive Wear

Erosive wear can be defined as the progressive loss of original material from a solid surface due to mechanical interaction between that surface and a fluid, a multi-component fluid, or impinging liquid or solid particles [ASTM G40-99] [33]. When the angle of impingement is small, the wear produced is closely similar to abrasion. When the angle of impact is normal to the surface, more loss of material occurs. The erosion mechanism depends on the material (brittle / ductile). So erosion has been divided into brittle and ductile erosion. Ductile materials fail as a result of impacting particles causing localized plastic flow that exceeds the critical strain to failure in the local areas. When the erodent particles in either gas or liquid carrier fluid strikes the surface of a ductile material, they initially extrude thin microplatelates of the base material from craters which are formed at the sites of impacts and then the platelates are then further flattened. After a small number of particles have impacted the same localized area, the extruded platelates would have been strained to their critical strain and fracture of portions of the platelate will occur [94]. The mechanism of erosion of brittle materials (i.e. ceramic type materials) is considerably different. Brittle materials are removed by a cracking and chipping mechanism. Here erosion occurs by the propagation and intersection of cracks produced by the impacting particles. The dense, columnar grain, outer scale cracks are chipped away, while the small equiaxed grains of the inner scale initially form hertzian cone cracks or ring cracks. Subsequently, at latter times, increased loading leads to increasing number of ring cracks leading to chipping away of the inner scales [34,25].

Erosion modes can be subdivided according to the erosive medium as:

- ❖ **Solid particle erosion:** It is the removal of material by repeated impact of tiny solid particles in gaseous or liquid medium [16]. In this erosion, a series of particles strike and rebound from the surface and cause a force on the material due to their deceleration. This erosion depend on velocity, size, amount of erodent particle

- ❖ **Liquid impingement erosion: W**hen small drops of liquid are striking on the surface of a solid at a high speed (~1000m/s) and very high pressures are experienced, exceeding the yield strength of most materials, this erosion occurs. Thus plastic deformation or fracture may result from a single impact and repeated impacts may lead to pitting and erosive wear. Here liquids need not contain particles to damage to a solid surface [47].

- **Cavitation erosion:** Cavitation is defined as the formation and subsequent collapse of cavities or bubbles within liquid. Cavitation erosion is the mechanical damage of a solid surface caused by thee cavities or bubbles collapsing either at or near the surface [28].

- **Slurry erosion: when erosion occurs by** a mixture of solid particles in a liquid with a high velocity, then it can be called as slurry erosion [39].

- **Erosion-Corrosion:** This is basically erosion enforced by corrosion mechanism. Corrosion may enhance the erosion rate through preferential dissolution or it may inhibit erosion through the formation of a passive film. Erosion-corrosion interaction is the potential synergy between the two processes. When metal alloys are exposed to the combined degradation mechanisms of corrosion and small solid particle erosion, combinations of the loss mechanisms of ductile and brittle materials occur, but primarily the brittle behaviour [54].

2.8.4 Surface Fatigue Wear

Wear arises from material fatigue is called as surface Fatigue Wear. This wear became dominant, when a solid is subjected to cyclic loading involving tension and compression above a certain critical stress. Repeated loading causes the generation of micro cracks at the site of a pre-existing point of weakness and leads to join of micro-void and form the crack. When crack reaches the critical size, it changes its direction to emerge at the surface, and thus flat sheet like particles is detached during wearing. The number of stress cycles required to cause such failure decreases as the corresponding magnitude of stress increases [10].

2.8.5 Corrosive Wear

Thermodynamically unstable metals with oxygen to form an oxide and gradually develop scales or layers on the surface. These layers are very weak or unprotected which allows gradual degradation of metal on the surface. Deterioration also cause by the effects of the atmosphere, acids, gases, alkalis, etc. This type of wear creates pits and perforations and may eventually dissolve metal parts [11].

2.9 SYMPTOMS OF WEAR

Wear is a characteristic of the system & its surrounding and is influenced by many parameters. So it is necessary to understand the wear mechanism to protect the metal. In Laboratory scale investigations, individuals of tribo-systems are carefully control and study the effects of different variables on the wear behaviour of the coating. The data generated through such research under controlled conditions may help in correct interpretation of the results. A summary of the appearance and symptoms of different wear mechanism is indicated in Table 2.3 [102] and the same is a systematic approach to diagnose the wear mechanisms.

Table 2.3 Different wear mechanism, symptoms and surface appearance.

Type of wear	Symptoms	Appearance of worn out surface
Abrasive	Presence of chip-out of surface	Grooves
Adhesive	Metal transfer s prime symptoms	Seizure, catering rough and torn-out surfaces
Erosion	Presence of abrasives in the fast moving fluid and short abrasion furrows	Waves & Troughs
Fatigue	Presence of surface or subsurface cracks accompanied by pits and spalls	Sharp and angular edges around pits
Corrosion	Presence of metal corrosion products	Rough pits and depressions
Delamination	Presence of subsurface cracks parallel to the surface with semi-dislodge or loose flakes	Loose, long and thin sheet like particle
Impacts	Surface fatigue, small sub-micron particles or formation of spalls	Fragmentation, peeling and pitting
Fretting	Production of voluminous amount of loose debris	Roughening, seizure and development of oxide ridges
Electric attack	Presence of micro-craters or a track with evidence of smooth molten metal	Smooth holes

2.10 RECENT TRENDS IN MATERIAL WEAR RESEARCH

Most of wear researches carried out in the 1940's and 1950's were conducted by metallurgical and mechanical engineers to generate data for the protective structural materials of different motor drive, trains, bearings, brakes, bushings and other types of moving mechanical assemblies [3]. It became apparent during the survey that wear of metals was a prominent topic in a large number of the responses regarding some future priorities for research in tribology. Much of the wear research conducted over more than past 50 years is in ceramics, polymers, composite materials and coatings [4]. Now-a-days this type of research are in rapid progress in different country in different part of the world.

2.11 WEAR RESISTANT COATINGS

Today a variety of materials, e.g., carbides, oxides, metallic, etc. are available commercially for protecting the metal surface. The wear resistant coatings can be classified into the following categories: [12]

(i) Carbides: WC, TiC, ZrC, Cr_2C_3, SiC, etc.

(ii) Oxides: Cr_2O_3, Al_2O_3, TiO_2, ZrO_2 etc.

(iii) Metallic: NiCrAlY, Triballoy etc.

(iv) Diamond

The choice of a material depends on the application. However, the ceramic coatings are very hard and hence can provide more abrasion resistance than their metallic counterparts.

2.11.1 Carbide Coatings

For wear and corrosion applications WC is very popular among all carbides [105]. The WC powders are clad with a cobalt layer. During spraying the cobalt layer undergoes melting and upon solidification form a metallic matrix in which the hard WC particles remain embedded. Spraying of WC-Co involves a close control of the process parameters such that only the cobalt phase melts without degrading the WC particles. Such degradation may occur in two ways: one is Oxidation of WC leading to the formation of $CoWO_4$ and WC_2 [6] and another one is dissolution of WC in the cobalt matrix leading to a formation of brittle phases like CoW_3C which embrittles the coating [7]. An increase in the spraying distance and associated increase of time in flight leads to a loss of carbon and a pickup of oxygen. As a result the hardness of the coating decreases [8]. An increase in plasma gas flow rate reduces the dwell time and hence can control the oxidation to some extent. However, it increases the possibility of cobalt dissolution in the matrix [9]. The other option to improve the quality of such coating is to conduct the spraying procedure in vacuum [7]. Some carbides like TiC, TaC and NbC are provided along with WC in the cermet to improve upon the oxidation resistance, hot strength, and hardness. A coating of Cr_3C_2 with Ni-Cr alloy cladding is known for its excellent sliding wear resistance, superior oxidation and erosion resistance, but its hardness is lower than that of WC. After spraying in air, Cr_3C_2 loses carbon and transforms to Cr_7C_3. Such transformation generally improves hardness and erosion resistance of the coating [10].

2.11.2 Oxide Coatings

Metallic coatings and metal containing carbide coatings sometime are not suitable in high temperature environments in both wear and corrosion applications due to formation of oxidation or decarburization. In such case the material of choice can be an oxide ceramic coating, e.g., $Al_2O_3.Cr_2O_3$, ZrO_2, TiO_2 or their combinations [11]. However, a high wear resistance, and chemical and thermal stability of these materials are counterbalanced by the disadvantages of low values of thermal expansion coefficient, thermal conductivity, mechanical strength, fracture toughness and somewhat weaker adhesion to substrate material. The thickness of these coatings is also limited by the residual stress that grows with thickness. Therefore, to obtain a good quality coating it is essential to exercise proper choice of bond coat, spray parameters and reinforcing additives.

2.11.3 Metallic Coatings

Metallic coatings can be easily applied by flame/plasma spraying or welding techniques making the process very economical. Metallic wear resistant materials are classified into three categories:

(i) Cobalt based alloys

(ii) Nickel based alloys

(iii) Iron based alloys

The common alloying elements in a cobalt-based alloy are Cr, Mo, W and Si. The microstructure is constituted by dispersed carbides of M7C3 type in a cobalt rich FCC matrix. The carbides provide the necessary abrasion resistance and corrosion resistance. Hardness at elevated temperatures is retained by the matrix [12, 13]. The principal alloying elements in Ni-based alloys are Si, B, C and Cr. The abrasion resistance can be attributed to the formation of extremely hard chromium borides. Besides carbides, Laves phase is also present in the matrix [12]. Iron based alloys are classified into pearlitic steels, austenitic steels, martensitic steels and high alloy irons. The principal alloying elements used are Mo, Ni, Cr and C. The softer materials, e.g., ferritic, are for rebuilding purpose. The harder materials, e.g., martensitic, on the other hand provide wear resistance. Such alloys do not posses much corrosion, oxidation or creep resistance. Nickel aluminide is another example of coating material for wear purpose. The prealloyed Ni-Al powders, when sprayed, react exothermically to form nickel aluminide. This reaction improves the coating substrate adhesion. In addition to wear application, it is also used as bond coat for ceramic materials. NiCoCrAlY is an example of plasma sprayable superalloy. It shows an excellent high temperature corrosion resistance and hence finds application in gas turbine blades. In addition, it serves as a bond coat for zirconia based thermal barrier coatings [14].

2.11.4 Diamond Coatings

In some industrial application diamond films are commonly produced by CVD, plasma assisted CVD, laser ablation technique and ion beam deposition [13, 16]. Such coatings are used in electronic devices and ultra wear resistant overlays. The limitation of the aforesaid methods is their slow deposition rates. The DIA-JET process involving DC Ar/H2 plasma with methane gas supplied at the plasma jet is capable of depositing diamond films at a high rate [17]. However, the process is extremely sensitive to the process parameters. Deposition of diamond film is also possible using an oxy-acetylene torch [18]. One significant limitation of a diamond coating is that it cannot be rubbed against ferrous materials, owing to a phase transformation leading to the formation of other carbon allotropes [19]. Diamond films are tested for the sliding wear against abrasive papers, where wear progresses by micro fracturing of protruding diamond grits. The process continues till the surfaces become flat and thereafter wear progresses by an interfacial spalling. Therefore, the life of the coating is limited by its thickness [20].

2.12 UTILIZATION OF FLY ASH AS WEAR RESISTANT COATINGS

Fly ash is the industrial waste in all iron and steel industries. Fly ash is a finely divided by-product with particle size varying from 0.5 to 100 μm. It is refractory and abrasive in nature as it contains hard oxide materials. The effective utilization of these wastes not only decreases environmental pollution, but also produces high value-added products [21, 22]. The chemical composition of fly ash is not constant everywhere due to the nature of different product in different industries. However, its main constituents are silicon dioxide (SiO2), aluminium oxide (Al2O3) and iron oxide (Fe2O3), and are hence a suitable source of aluminium and silicon. In previous time, silica and alumina-silicate bricks have been preferred as refractory materials in many industrial applications due to their high wear resistance and high load bearing capacity at high temperatures. During the last decade, although a large number of investigations have been carried out for development of plasma spray ceramic coatings. Fly ash is a substitute for high cost conventional spray powder in high performance industrial coatings [23]. It can be utilized to develop ceramic coatings on metal substrate. In this context, Mishra et al. has been investigated some experiments on fly-ash composite to spray on different type of metal by plasma spraying [24-28]. Buta Singh et.al have investigated the fly ash coating obtained by shrouded plasma spray process on carbon steel and found it to be effective to increase the oxidation and salt corrosion resistance of the given carbon steel [29]. Fly-ash composite coatings, such as fly-ash+zinc coatings [30] and fly-ash+ Na-geopolymer [31] have been extensively studied. The properties of fly ash has been studied by Tiwari and Saxena and coatings that were developed have shown improved corrosion and abrasion resistance and also better resistance to chemicals (5% Na2CO3 , 1% NaOH, and 2% H24 [32].

2.13 EROSION WEAR OF CERAMIC COATINGS

When equipments/ parts are operated in industrial hostile environment involving two or more damage modes such as corrosion-erosion or corrosion-wear; many coatings perform poorly due to the synergistic action of wear and corrosion [33]. Considerable efforts have been continuously made to develop high-performance coatings that can resist corrosive wear encountered in various industries such as oil, petroleum, mining, sand and chemical industries [34]. Now-a-days Plasma sprayed coatings (one of a Thermal spray technique) are used as thermal barriers and abrasion, erosion or corrosion resistant coatings in a wide variety of applications owing to their high hardness and high temperature performance [35]. The high temperatures of plasma spray processes permit the deposition of coatings for applications in areas of liquid and high temperature corrosion and wear protection and also special applications for thermal, electrical and biomedical purposes [36, 37]. Superalloys can be used as protective coatings [38, 39]. The results show that the relationship between the erosion mass loss and the erosion time is linear, the coatings hold a maximum erosion rate at 60^{o} impact angle, and the relation between the erosion rate and the impact speed is an exponential function. The erosion tests were carried out by a stream of alumina particles with an average size of 50μm at 70 m/s, carried by an air jet with impingement angle of 90^{o}.

Chapter 3

Experimental set up and Methodology

- Introduction
- Development of coatings
- Characterization of feedstock
- Characterization of coatings
- Erosion wear behaviour of coatings

Chapter 3
Experimental set up and Methodology

3.1 INTRODUCTION

This chapter explains about procedure of different experimental processes used to prepare the coatings and to characterize. Before preparation of coating, some basic process required for substrate material i.e. size measurement of coating powder, Grit blasting & cleaning of substrate. After plasma spraying, the coated materials have been subjected to a series of characterization test i.e. microstructural characterization of the surfaces and microstructural characterization of substrate-coating interface,

3.2 DEVELOPMENT OF COATINGS

3.2.1 Preparation of Powder

Fly ash of 55 weight percentages premixed with 25 weight percentage of quartz and 20 weight percentage of illmenite are mechanically milled in a FRITSCH-Planetary ball mill for 3 hours to get a homogenized product. There are 4 numbers of zirconia balls of 20gm and 20 numbers of zirconium balls of 2gm present in the planetary ball mill. The powders obtained were sieved by the help of a Roto-Tap Sieve Shaker Machine by using Laboratory test sieves (ISO R565). Required size of powder mixture for spraying are collected.

3.2.2 Preparation of Substrate

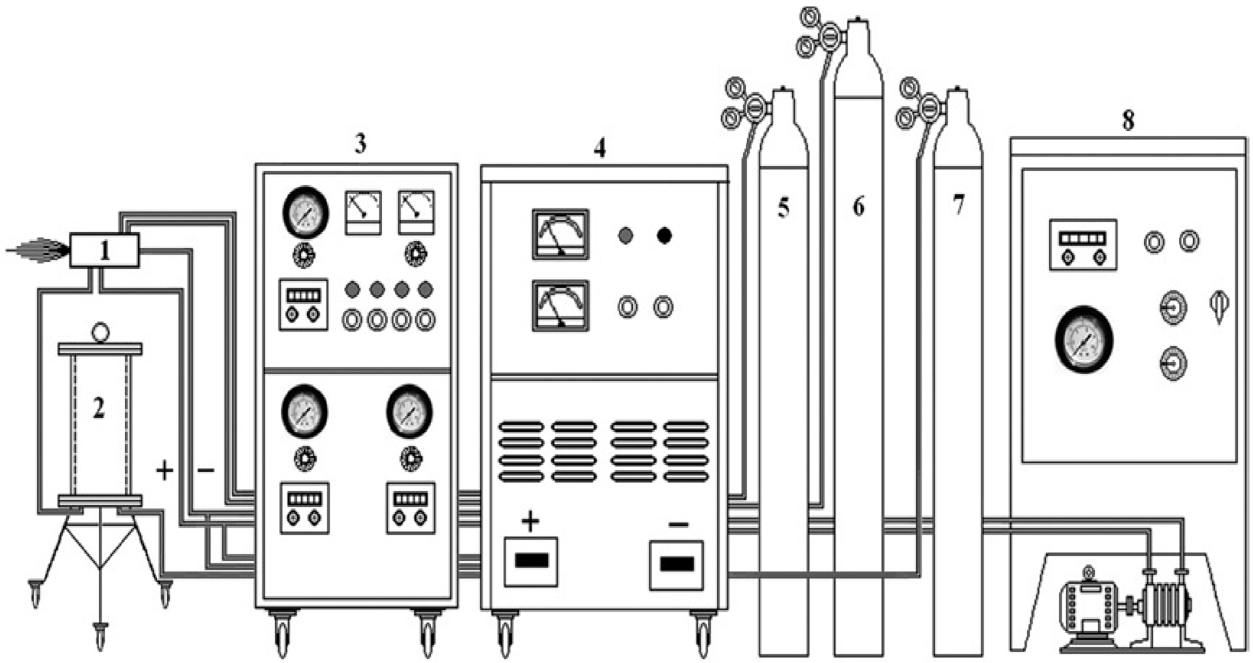
Two Commercially available metals: Mild Steel and Copper have been chosen as substrate materials. These substrates were circular disc having dimension of 1 inch diameter and 3mm thickness. The specimens were grit blasted at a pressure of 3 kg/cm^2 using alumina grits (grit size of 60). Stand-off-distance was kept between 120-130 mm for blasting. Surface roughness of the substrates was approximately 5 Ra. The grit blasted specimen surface was cleaned with acetone in an ultrasonic cleaning unit. Plasma spraying was immediately carried out after cleaning.

3.2.3 Plasma Spray Coating Deposition

The plasma spray deposition was done at the Laser and Plasma Technology Division, Bhaba Atomic Research Center, Mumbai. 40kW DC non-transferred arc mode Conventional atmospheric plasma spraying (APS) set up was used. In plasma torch input power level was varied from 10kW to 20 kW, by controlling the voltage, gas flow rate and the arc current. The powder injection was external from the nozzle and directed towards the plasma. Argon and hydrogen plasma mixture gas used as carrier gas. The powder feed rate of 13 gm/min was kept constant, using a turntable type volumetric powder feeder. A four stage closed-looped centrifugal pump (water cooling) used for cooling the system, regulated at a pressure of 10kg/cm^2 supply.

The typical arrangement of the plasma spray equipment and schematic diagram of the plasma spraying process are shown in Fig 3.1. The equipment consists of the following units:

- ✓ Plasma spraying equipment
- ✓ Control console
- ✓ Powder feeder
- ✓ Power supply
- ✓ stand-off-distance of torch
- ✓ Torch cooling system (water)
- ✓ carrier gas supply
- ✓ Hoses, cables, gas cylinders and accessories



1.Plasma torch 2.Powder feeder 3.Control console 4.Plasma power source
5. Ar gas cylinder 6.H_2 gas cylinder 7.N_2 gas cylinder 8. Cooling tower

Figure 3.1 General arrangement of plasma spraying equipment.

Argon is taken as the primary plasmagen gas and nitrogen as the secondary gas. The powder mixtures are deposited at a spraying angle of 90°. The properties of the coating products are dependent on the spray process parameters. The operating parameters during coating deposition are listed in Table 3.1.

Table 3.1 Operating parameters during coating deposition.

Operating parameters	Values
Plasma arc current(amp)	260-500
Arc voltage (volt)	40-44
Torch input power(kW)	10,13,17,20
Plasma gas(argon) flow rate(IPM)	28
Secondary gas(N2) flow rate(IPM)	3
Carrier gas(Ar) flow rate(IPM)	12
Powder feed rate (gm/min)	13
Torch to base distance(TBD)(mm)	100

3.3 CHARACTERIZATION OF FEEDSTOCK

3.3.1 Particle Size Analysis

The particle sizes of the raw materials used for coating (fly ash+quartz+illmenite powder) were characterized using Laser particle size analyzer of Malvern Instrument (PSA).

3.3.2 Compositional Analysis

The compositional analysis of fly-ash was done by wet chemical analysis method [43].

3.4 CHARACTERIZATION OF COATINGS

3.4.1 Scanning Electron Microscopic Studies

By using JEOL JSM-6480 LV scanning electron microscope (SEM), microstructure of raw powder and plasma sprayed coated specimens were studied. The surface morphology as well as the coating-substrate interface morphology of all coatings was observed under the microscope. Here SEM mostly using the secondary electron imaging.

3.4.3 Evaluation of Coating Interface Bond Strength

Coating adhesion strength measure by using a special type jig (Fig 3.2) which was fabricated. Cylindrical mild steel dummy samples (length 25 mm, top and bottom diameter 12 mm) were prepared. By punching the surfaces of the dummies were roughened. These dummies were then fixed on top of the coating with the help of a polymeric adhesive. After mounting the dummy on the jig, ultimate tensile stress has been measured by pull-out method (Fig 3.3). This coating pullout test was carried out using the Instron 1195 set up at a crosshead speed of 1 mm/minute. the reading (of the load) at the time of the torn-off moment of coating from the specimen corresponds to coating adhesive strength, was recorded. A typical test set up during testing is shown in Fig 3.4. The test was performed as per ASTM C-633 [44].

Figure 3.2 Jig under the test. **Figure 3.3** Specimen under tension.

Figure 3.4 Adhesion test with Instron 1195 UTM.

3.4.4 Evaluation of Coating Deposition Efficiency

Deposition efficiency can be defined as the ratio of the weight of coating deposited on the substrate to the weight of the expended feedstock [45]. Weighing method is used to measure this. Specimen weighing was done by using a precision electronic balance with ±0.1 mg accuracy [46]. Each specimen have been weighed before and after coating deposition. Gc known as the difference is the weight of coating deposited on the substrate. Gp is the weight of expended feedstock which can be calculated from the powder feed rate and time of deposition. The deposition efficiency (η) is then calculated using the equation:

$$\eta = \left(\frac{Gc}{Gp} \times 100\right)\%$$

3.4.5 Coating Thickness Measurement

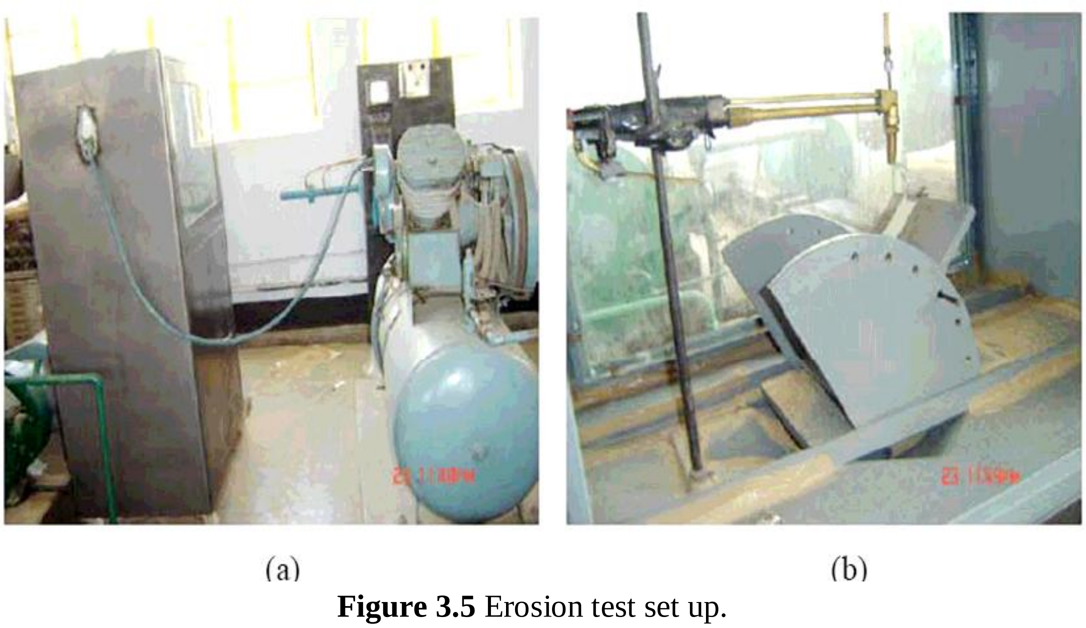
To measure the thickness of coated material on the substrate, specimen cross section was polished and measured using an optical microscope. Five readings were taken on each specimen and the average value is reported as the (mean) coating thickness.

3.4.6 Hardness Measurement

The coated samples were transversally sliced to form small specimens which containing coating deposition. Coating cross-sections were mounted and polished. Vickers hardness measurement was made using Leitz Micro hardness Tester equipped with a monitor and a microprocessor based controller, with a load of 50 Pa (0.419N) and a loading time of 20 seconds. About four or more readings were taken on each sample and the average value is reported as the data point.

3.5 EROSION WEAR BEHAVIOUR OF COATINGS

Solid particle erosion is usually simulated in laboratory by one of two methods: one is "sand blast" process where particles impacted onto a stationary target and are carried in an air flow and the second one is "whirling arm" process where through a chamber of falling particles in which the target is spun. In the present investigation, sand blast type erosion apparatus with angular erodent is used (Fig 3.5). The test is conducted as per ASTM G76 standards [48]. Erosion Test carried out with varying particle sizes, velocities, incidence angles, and particles fluxes in order to generate quantitative data on materials and to study the mechanisms of damage.



(a) (b)

Figure 3.5 Erosion test set up.

In this work, the jet erosion test rig used whose schematic diagram shown in Fig 3.6 employs a 300 mm long nozzle of 300 mm long and 3 mm bore. The mass flow rate was measured by conventional method. Particles were fed from a simple hopper under gravity into the groove. The measurement of velocity of impact is done using double disc method [49]. This nozzle size permits a wider range of particle types to be used in the testing, allowing better simulations of real erosion conditions.

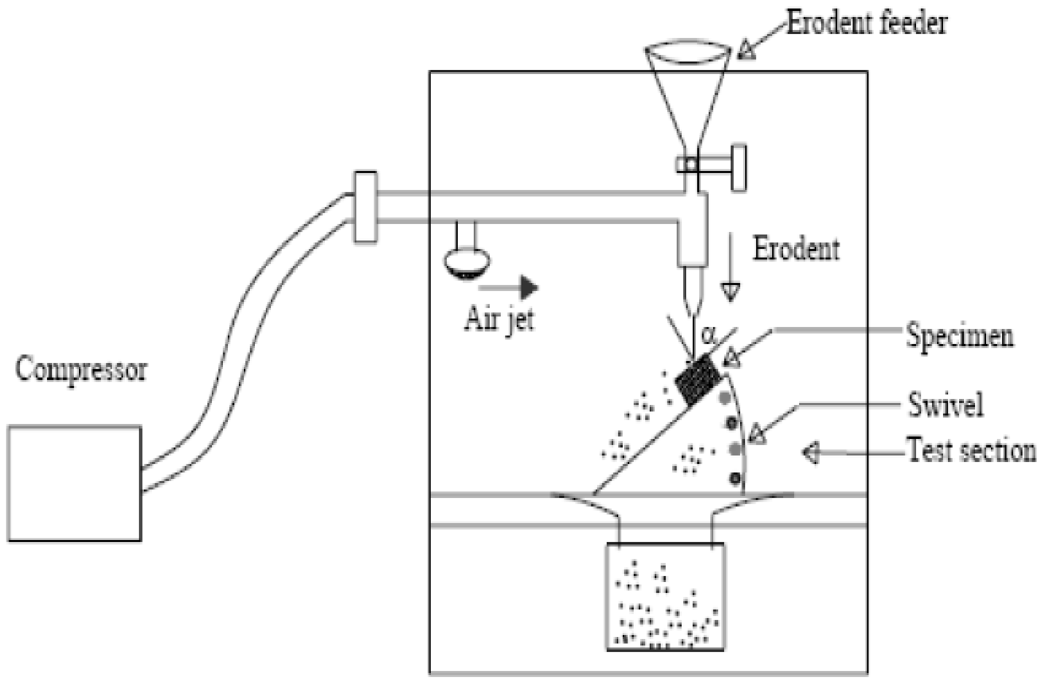


Figure 3.6 Schematic diagram of erosion test rig.

Some of the type of this test set up is:

- Different nozzles may be accommodated: provide stability to change the particle plume dimensions and velocity range.
- Vertical traverse for the nozzle: provide nozzle with variable to target stand-off-distance, hence influencing the size of the eroded area.
- Large test chamber with sample mount (typical sample size 25mm X 25mm) can be angled to the flow direction: by tilting the sample stage, the angle of impact of the particles of the material can be changed in the range of 0°-90° and this will influence the erosion process.

The erosion wear test were carrying out by varying angles (i.e. at 30°, 45°, 60°, 90°), standoff distance (100 mm t0 140 mm), pressure (1 bar to 3 bar), time (60 sec to 180 sec). The erosion wear rates were obtained with silicon carbide erodent of 130 μm size. Erosion rate defined as the coating mass loss per unit erodent mass was calculated.

Chapter 4

Results and Discussion

- Introduction
- Characterization of Coating Material
- Characterization of coatings
- Coating performance
- Discussion

Chapter 4
RESULTS AND DISCUSSION

4.1 INTRODUCTION

Thermal plasma spray coatings are deposited on two different metal substrates i.e. Mild Steel and Copper, using fly-ash+quartz+illmenite as the coating material. Characterizations of the coatings ware done and the tribological performances of the coating were investigated. The results of various tests are presented and discussed in this chapter.

4.2 CHARACTERIZATION OF COATING MATERIAL

4.2.1 Particle Size Analysis

The particle size distribution of fly ash+quartz+illmenite powder just before spraying was characterized by using Laser particle size analyzer of Malvern Instruments. The particle size distribution of the feedstock is represented in Fig 4.1. The particles are found to be in the range of 40 to 100 micron. Maximum volume fractions of the particles are in the range of 50 micron.

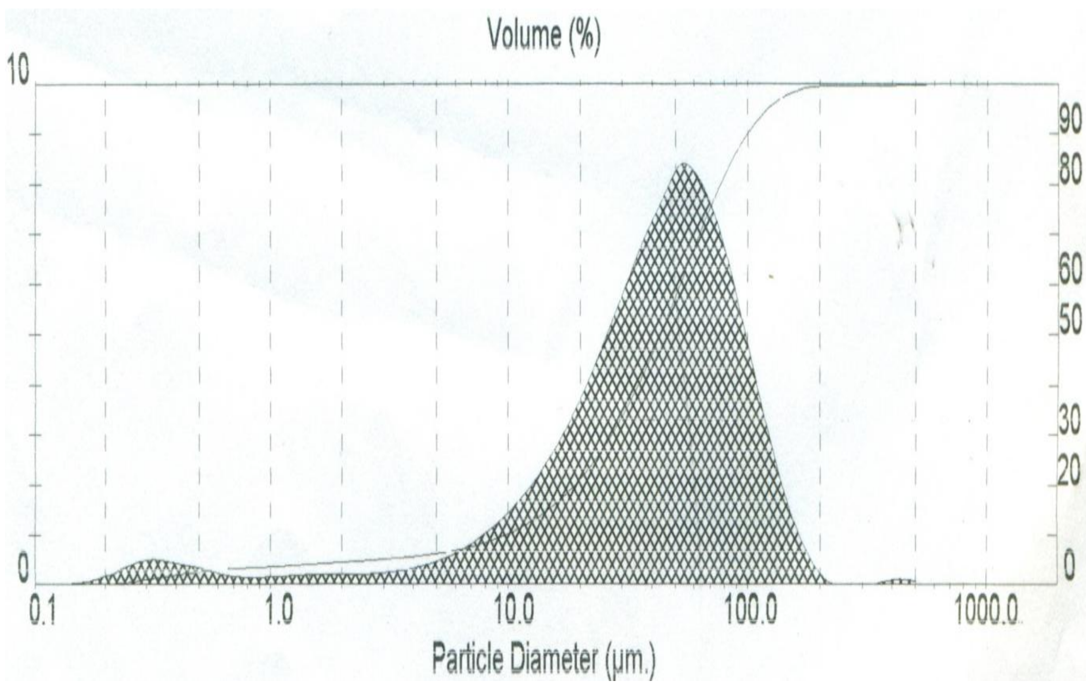


Figure 4.1 Particle size analysis of fly-ash+quartz+illmenite spray coating feedstock.

4.2.2 Chemical Composition Analysis

Fly-ash is collected from Rourkela Steel Plant (CPP-1), Rourkela. The chemical composition analysis of major constituents of fly ash is given in Table 4.1. To this material, quartz powder (SiO2) and illmenite ($FeTi_2O_3$) has been added (20 wt% each) to prepare the feedstock.

Table 4.1 Chemical composition of fly-ash.

Compounds	**Percentage**
SiO2	58.00
Al_2O_3	29.30
Fe_2O_3	5.60
CaO	1.05
TiO_2	1.70
MgO	1.25
P_2O_5	0.25
K_2O	0.84
Na_2O	0.61
SO_3	0.41
H_2O	0.20

4.2.3 Morphology of powder/raw material for coating

SEM micrographs of fly ash+quartz+illmenite feedstock prior to coating are shown in Fig 4.2. It is observed that the Particles are of irregular in shape and of varied sizes. Some particles are elongated type and some are multifaceted.

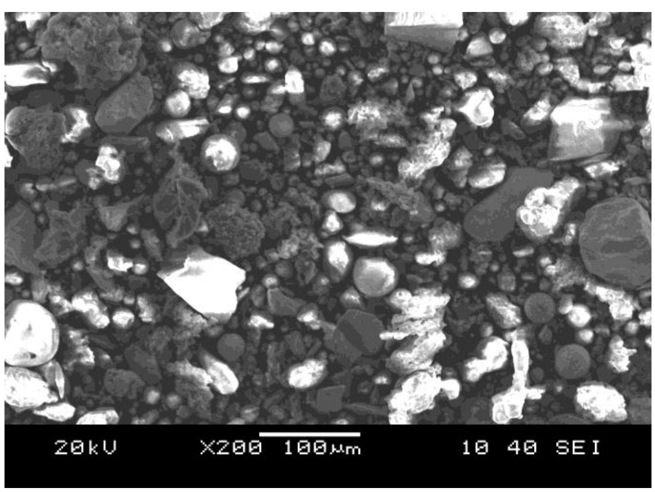


Figure 4.2 SEM micrographs of fly ash+quartz+illmenite raw powder prior to coating.

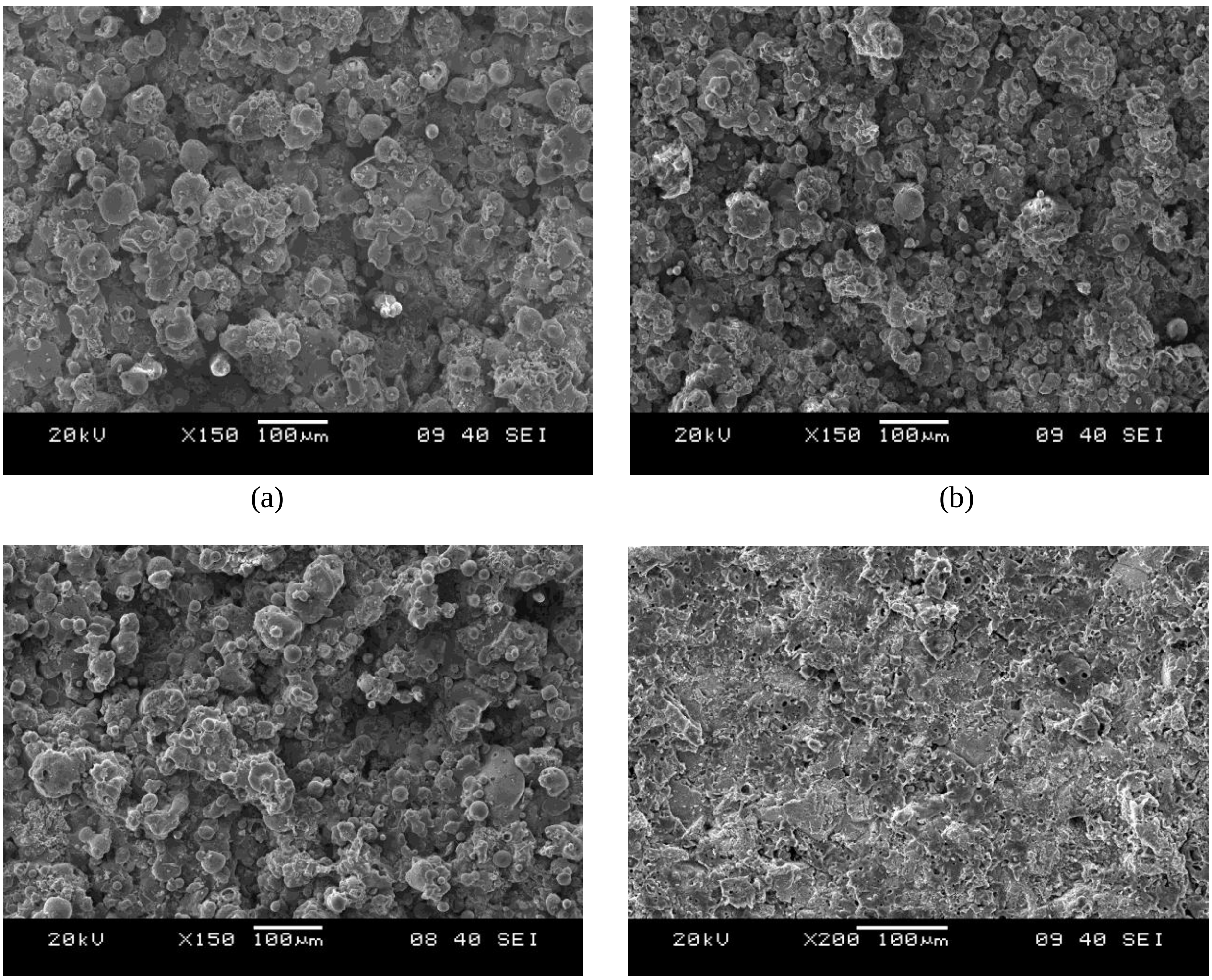
4.3 CHARACTERIZATION OF COATINGS

The coatings are deposited with atmospheric plasma spraying and the following characterizations are carried out.

4.3.1 Microstructural Study of the Coatings

Surface Morphology:

The interface adhesion of the coatings depends on the coating morphology and inter-particle bonding of the sprayed powders. SEM micrographs of fly ash+quartz+illmenite coating surfaces are shown in Fig 4.3.



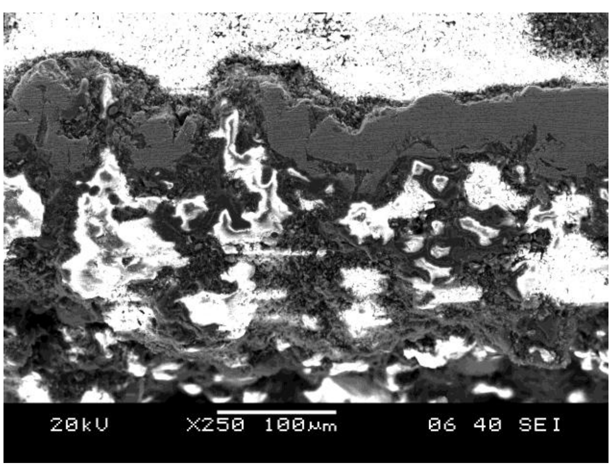
(a) (b) (c) (d)

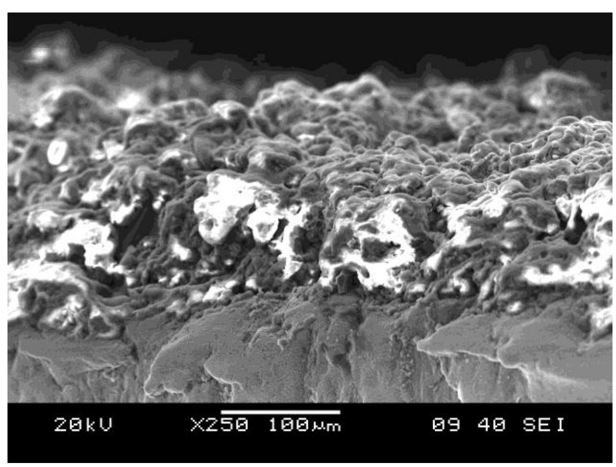
Figure 4.3 Surface morphology of fly ash+quartz+illmenite coatings deposited on mild steel Substrates, at **(a)** 10KW **(b)** 13KW **(c)** 17 KW **(d)** 20KW power level.

The coating deposited at 10KW power level (Fig 4.3(a)) on Mild Steel substrate, shows a uniform distribution molten/semi molten particle, which have agglomerated to form laths. More amount of cavitations observed at this lower power level. Some open pores are found on the inter particle boundaries and at triple particle/ grain junctions. These may have originated due to the inadequate flow of molten particles during their solidification. The coating deposited at 13KW and 17 KW (Fig 4.3(b) &(c)), a different morphology is obtained i.e. spheroidal splats are found, indicating complete melting of the particles during in-flight traverse through jet of plasma. A smaller amount cavitations observed in inter-granular boundaries. At coating made at 20 KW power level (Fig 4.3(d)); more thermal energy is obtained by the particles, so during solidification from molten state, they agglomerate/ combine to form splats i.e. flattened region is formed. A very less amount of cavitations is observed here. This is the reason for better interface adhesion of the coating onto the substrate, which increases the adhesion strength. This morphology so obtained shows a uniform distribution of molten/ semi molten particles; hence no more increase in adhesion strength.

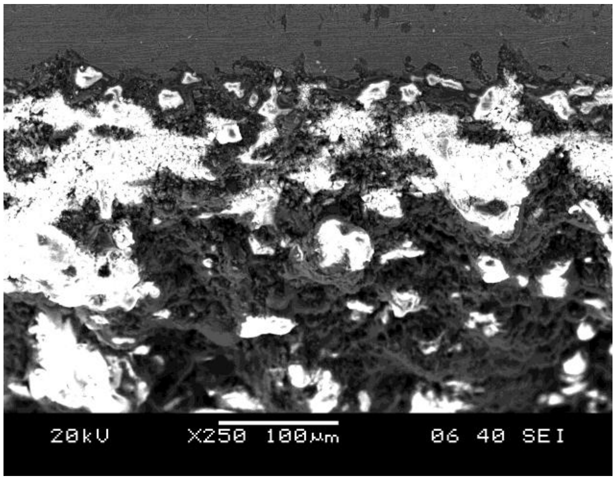
Interface Morphology:

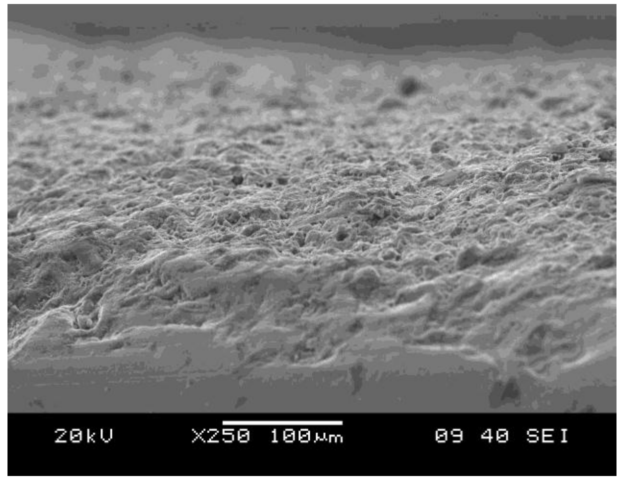
The surface morphology of the coatings cannot predict how will be the interior (layer deposition) structures. So, the polished cross-sections of the samples were examined under scanning electron microscopy and are shown in Fig 4.4. From these micrographs approximate better result can be found. Fig 4.4(a) shows the coating deposited at 10 KW. Hear it is found that lamellar structure present with cavitations at the coating-substrate interface. Very small diameter splats are found with inter-splat voids. Some semi-molten powder particles found. So here, there is no expectation for good adhesion strength. Fig 4.4(b) shows the coating at 13 KW power level. Less number of voids are present than that of 10 KW deposition. Here, Splats are globular, larger in dimension and equi-axed type. Fig 4.4(c) shows deposition at 17 KW power level. Here there is a negligible amount of voids, which are not easily visible. Here better inter-particle boundary match leads to better bond strength throughout the length of coating. In Fig 4.4(d), 20 KW power level used for coating deposition, in which spheroidal particles observed with larger diameter splat. Here inter-layer gaps are found. This is due to more vaporization of molten particle during spraying. Other than the mechanical interlocking of the sprayed coating with the metal substrate, some metallurgical bonding might have occurred at the interface which is evident from the presence of some inter-diffusion zones.





(a) (b)





(c) **(d)**

Figure 4.4 Interface morphology of fly ash+quartz+illmenite coatings deposited on mild steel Substrates, at **(a)** 10KW **(b)** 13KW **(c)** 17 KW **(d)** 20KW power level.

4.3.2 Coating Deposition Efficiency

It is defined as the ratio of the weight of coating deposited on the substrate to the weight of expended feedstock. The weighing method is conventional method used widely to measure this [4]. It can be described by the following equation

$$\eta = \left(\frac{Gc}{Gp}\right) \times 100\,\% \quad \text{------------------------------} \quad \textbf{(4.1)}$$

Where, η is the deposition efficiency.

Gc is the weight of coating deposited on the substrate.

Gp is the weight-expended feedstock.

The deposition efficiency is a measure of the fraction of the powder that is deposited on the substrate. Deposition efficiency depends on many factors that include the input power to the plasma torch, material properties, such as melting point, particle size range, heat capacity of the powder being sprayed and standoff distance (torch to substrate distance) etc. For a given standoff distance and given material with specific particle size, torch input power appears to be an important factor for the deposition efficiency. Deposition efficiency values of fly ash+quartz+illmenite coating made at different operating power levels on mild steel and copper substrates are given in Table 4.2.

Table 4.2 Deposition efficiency of fly-ash+quartz+illmenite coatings.

Substrate Type	Coating Material	Power level(KW)	Deposition efficiency (%)
Mild Steel	Fly-ash+quartz+illmenite	10	23
Mild Steel	-do-	13	32
Mild Steel	-do-	17	41
Mild Steel	-do-	20	49
Copper	-do-	10	27
Copper	-do-	13	34
Copper	-do-	17	42
Copper	-do-	20	47

The variation of deposition efficiency of fly-ash+quartz+illmenite coatings with operating power level on mild steel substrate is shown in Fig 4.11. The deposition efficiency is increased in a sigmoidal fashion with the torch input power. Plasma spray deposition efficiency of a given material depends on its melting point, thermal heat capacity, rate of dissipation of heat at substrate and particle size of the sprayed powder etc. The coating deposition efficiency is significantly influenced by the input power to the torch.

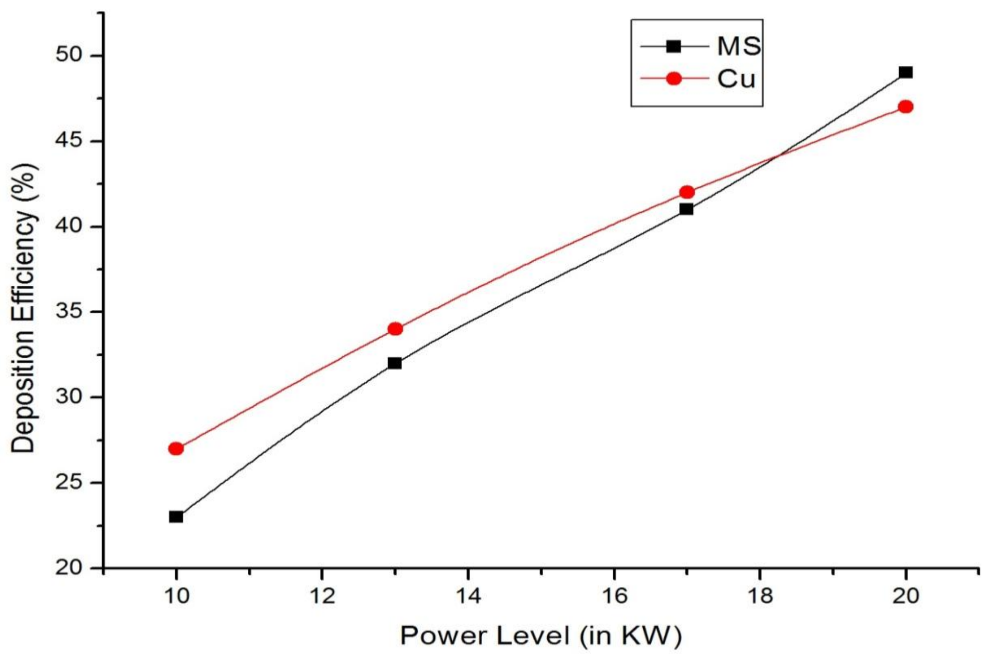


Figure 4.5 Variation of deposition efficiency of fly-ash+quartz+illmenite coatings at different power levels.

At lower power level, the plasma jet temperature is not high enough to melt the entire feed powder that enters the plasma jet. On increasing the power level, plasma temperature and enthalpy is increased, thus melting a larger portion of the powder feed. Thus the spray efficiency increases with increases with increasing input power to the plasma torch. On increasing the power level of the torch to a certain limit, temperature of the plasma becomes high enough leading to vaporization/dissociation of the feedstock. Thus there is not much increase in deposition efficiency. Because molten particle form a vaporized layer just before impact on the substrate surface which leads to formation of inter-layer voids [42, 43].

4.3.3 Coating Thickness

Coating thickness of fly-ash+quartz+illmenite was measured on the polished cross-sections of the samples, using an optical microscope. The thickness values obtained for coatings deposited at different power levels for Mild steel and copper substrates are presented in Table 4.3. Each data point on the curves is the average of at least four readings. Form the above data, it is found that maximum thickness of 310 µm found for mild steel. For copper maximum thickness is 355 µm, which is greater than mild steel substrate, may be due to difference in thermal conductivity of the substrates.

Table 4.3 Thickness values of fly ash+quartz+illmenite coatings on mild steel substrates.

Substrate Type	Coating Material	Power level (KW)	Coating Thickness (µm)
Mild Steel	Fly-ash+quartz+illmenite	10	255
Mild Steel	-do-	13	310
Mild Steel	-do-	17	290
Mild Steel	-do-	20	265
Copper	-do-	10	313
Copper	-do-	13	335
Copper	-do-	17	299
Copper	-do-	20	300

The variation of thickness values with torch input power for Mild steel and copper substrates is presented in Fig 4.6. From this figure it is observed that maximum coating thickness of ~ 310 µm is obtained at 13 KW power level on mild steel substrates which decreases gradually with the increase in input power to the plasma torch. Similar situation obtained for copper substrate and the maximum coating thickness of ~ 350 µm at 13 KW power level. In case of thermal spray of oxide coatings developed by APS technique, particle deposition is influenced by the input power to the plasma torch. With the increase in power level, the plasma density increases leading to rise in enthalpy and thereby the particle temperature. Hence more number of particles gets melted during in-flight traverse through the plasma jet. When these molten species hit the substrate, get flattened and adhere to the surface forming big splats. Hence, although there is decrease in coating thickness but a dense coating is formed.

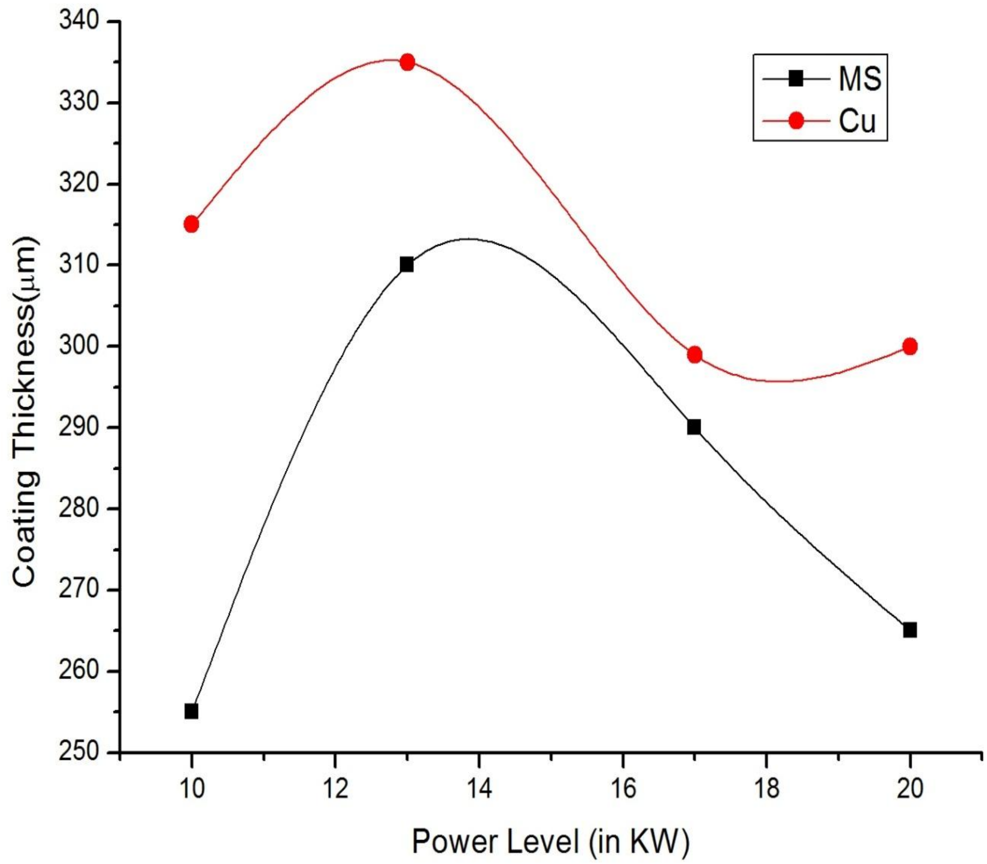


Figure 4.6 Variation of coating thickness of fly ash+quartz+illmenite coatings at different power levels.

4.3.4 Hardness of the Coatings

To measure hardness of the coating, microscopic observation of the polished cross section of the coatings under optical microscope is taken. Coating hardness measurement is carried out with Leitz Micro-Hardness Tester using 50Pa (0.419N) load, are summarized in Table 4.4. Each data point is the average of four observations. From this table, it is observed that there is an increase in coating hardness with the increase in plasma power level. This may be due to the formation/transformation of compounds viz. silica and alumina etc. to their allotropic forms and their compositional variations during spray deposition with the increase in input power to the plasma torch. Variation of coating hardness with respect to power level is given in Fig 4.7.

Table 4.4 Hardness on the coating cross section for the coatings deposited at different power levels.

Substrate Type	Coating Material	Power level(KW)	Avg. Hardness (H_V)
Mild Steel	Fly-ash+quartz+illmenite	10	529.12
Mild Steel	-do-	13	580.31
Mild Steel	-do-	17	638.65
Mild Steel	-do-	20	585.26
Copper	-do-	10	585.53
Copper	-do-	13	600.25
Copper	-do-	17	645.87
Copper	-do-	20	593.56

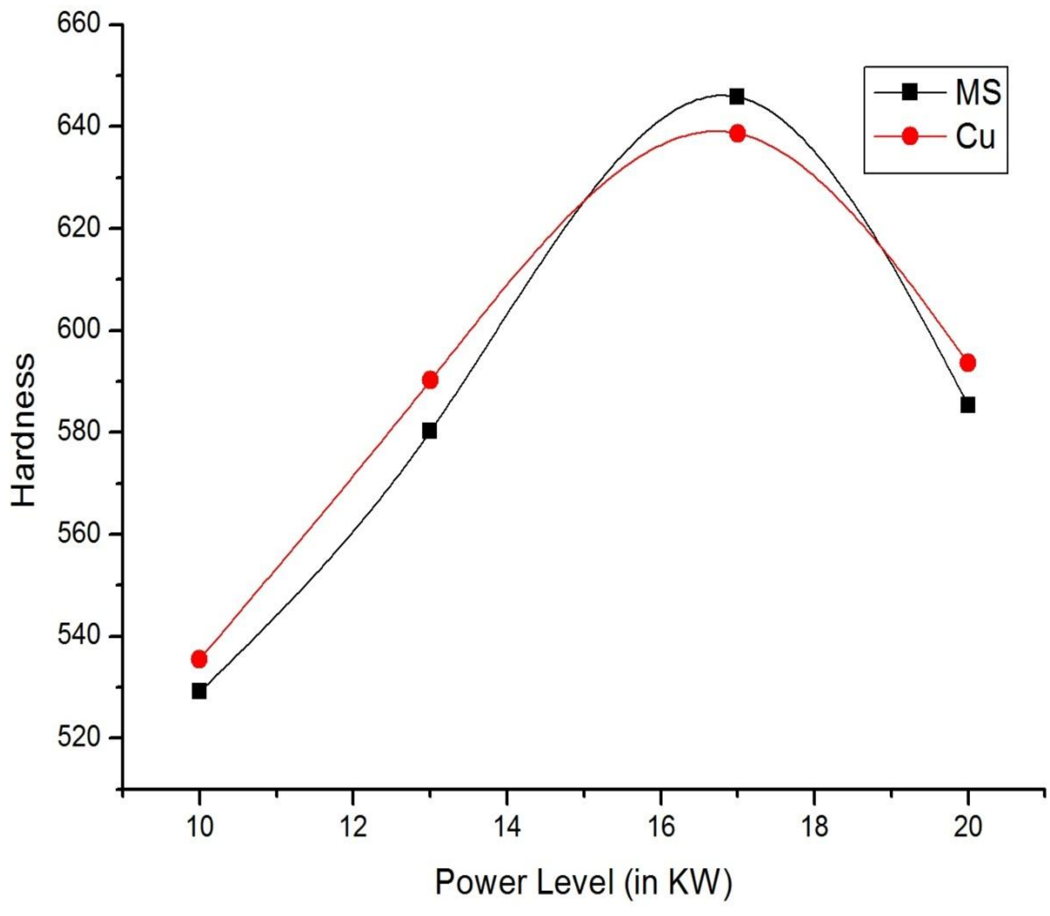


Figure 4.7 Variation of coating hardness of fly ash+quartz+illmenite coatings at different power levels.

4.3.5 Coating Adhesion Strength

Coating adhesion tests have been carried out by many investigators with various coatings [45]. The fracture mode is adhesive if it takes place at the coating-substrate interface and that the measured adhesion value is the value of practical adhesion, which is strictly an interface property and depending exclusively on the surface characteristics of the adhering phase and the substrate surface condition. From the microscopic point of view, adhesion is due to physicochemical surface forces (i.e. Vander-walls, Covalent, ionic etc.), which are established at the coating-substrate interface. In the present investigation, evaluation of coating interface bond strength is done using coating pull out method confirming to ASTM C-633. The variation of adhesion strength with operating power level for Mild Steel and Copper substrate is shown in Fig 4.10. Each data point is the average of five tests. It is found that, with increase in operating power level there is an increase in adhesion strength up to a certain level of operating power of the torch. It is found that for different type of substrate, the magnitude of adhesion strength is different. For Mild Steel the strength has varied from 3.5MPa to 6.66MPa, the maximum of 6.66MPa at 20KW power level. For Copper substrate this value ranges from 3.00MPa to 6.32MPa. Deposition efficiency is improved to a certain limit with increase in arc power, as it is associated with an enhanced particle melting and hence the adhesion strength. As the operating power level is increased, a large fraction of particles attain molten state as well as the velocity of the particles also increase. Therefore there is better probability for splat formation i.e. forms a lamellar structure and hence better mechanical inter-locking of molten particles/splats on the substrate leading to increase in adhesion strength [45]. But with increase in torch input power, fragmentation and vaporization of the particles increases. There is also a greater chance to fly off of smaller particles and results almost no further increase in adhesion strength of the coatings [40].

Table 4.5 Adhesive Strength of the coating deposited at different power levels.

Substrate Type	Coating Material	Power level(KW)	Adhesive Strength (MPa)
Mild Steel	Fly-ash+quartz+illmenite	10	3.30
Mild Steel	-do-	13	5.00
Mild Steel	-do-	17	6.40
Mild Steel	-do-	20	6.66
Copper	-do-	10	2.80
Copper	-do-	13	5.20
Copper	-do-	17	6.20
Copper	-do-	20	6.40

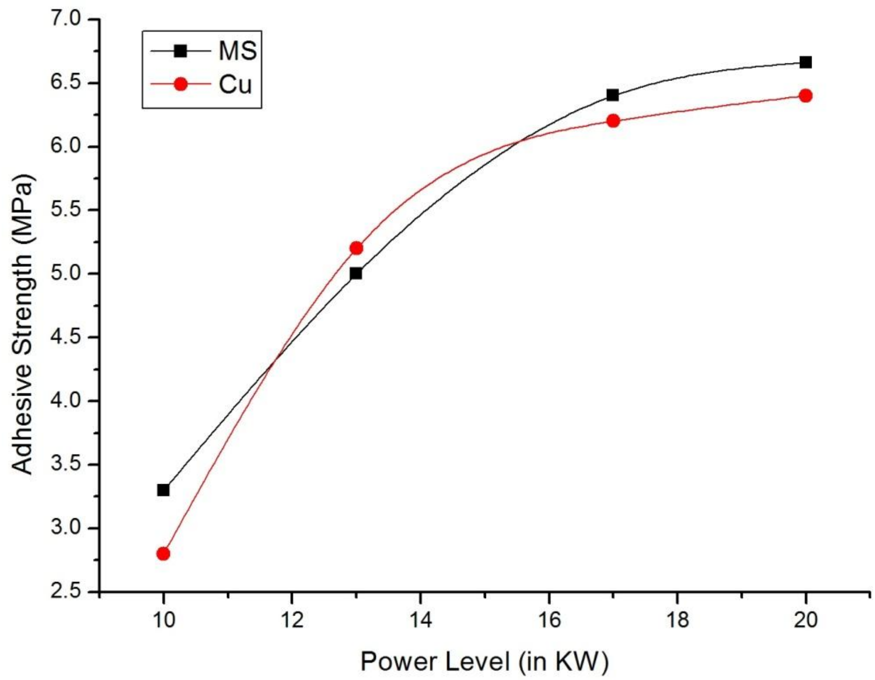


Figure 4.8 Comparison of adhesion strength of mild steel and copper with respect to power level.

Again by comparing adhesion strength of Mild Steel against Copper (Fig 4.8), it is found that at 10KW, 17 KW and 20KW power level adhesion strength of Mild Steel is greater. Coating made after 20 KW power level, the adhesion strength i.e. Mild Steel and Copper substrates tends to decrease. The low values of adhesion strength may be due to difference in coefficient of thermal expansion of substrate and coating material and/or formation of pores, cracks, voids in the coating and along coating-substrate interfaces [45].

4.3.6 ANN Prediction of Adhesion Strength

Artificial Neural Network analysis (ANN) is package software [9], which simulates the functional norm of the human brain. ANNs are moderately new modelling technique composed of a large number of related elements which are operating in parallel. They can be used to solve problems that are difficult for human reasoning or for conventional techniques. ANN calculation is quite faster than other finite element modelling calculations [52]. In this process, the parameters are inter-connected by means of weights which are numbers, translating the strength of neuron connections. Input parameters are taken as number fluxes which guide the network structure and help in obtaining the output pattern. ANN is based on a training procedure to decrease the error between ANN response and experimental response for a given set of input variables by considering neuron number and weight updates. ANN bibliography is very rich with learning models, like the popular back propagation and the quick propagation, the Hebbian algorithm, the ADALINE model or the Kohonen learning rule and among the other models [51]. Plasma spraying is considered as a non-linear problem with respect to its variables: either operating conditions or material. To obtain functional coatings exhibiting desired properties and combinations of processing parameters are to be planned. These combinations are different by the influence on the coating characteristics and properties. In order to control the process of spraying, the challenges nowadays are to make out parameter correlations, interdependencies and individual effects on coating characteristics. To optimize these interrelated effects, a robust method i.e. ANN is used. Different ANN structures (I-H-O) (shown in Fig 4.9) with varying number of neurons in the hidden layer are tested at constant learning rate, error tolerance, cycles, momentum parameter, slope parameter and noise factor. Based on least error principle, one structure, shown in Table 4.7, is selected for training of the input and output data. The rate of learning is varied up to 0.002 during the training of the input-output data. Neuron number of the hidden layers is varied and in the optimized structure of the network, this number is found to be 8 for adhesion strength. The number of cycles selected during training is high enough so that the ANN models could be rigorously trained.

Table 4.6 Input parameters selected for training (Coating adhesion strength).

Input Parameters for Training	Values
Error tolerance	0.003
Learning parameter(ß)	0.002
Momentum parameter(α)	0.002
Noise factor (NF)	0.001
Maximum cycles for simulations	1,00,00,000
Slope parameter (£)	0.6
Number of hidden layer neuron	8
Number of input layer neuron (I)	5
Number of output layer neuron (O)	1

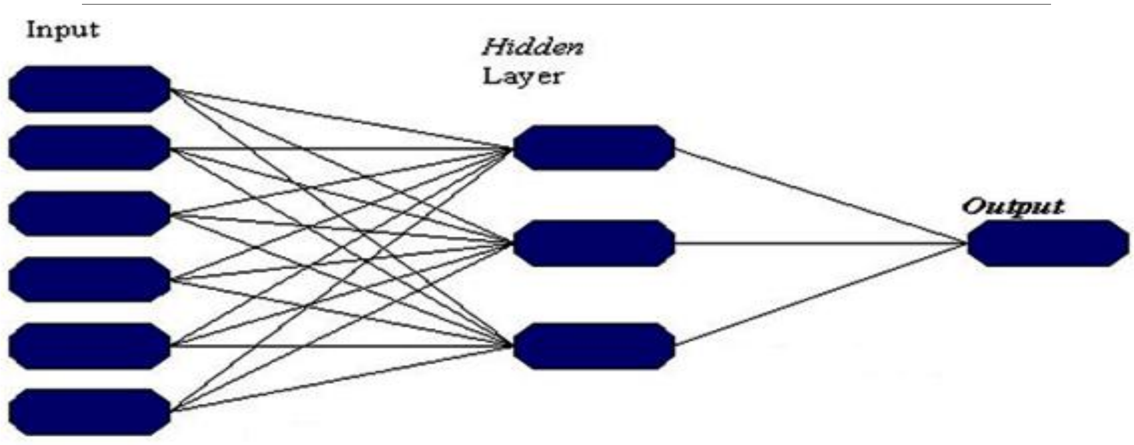


Figure 4.9 The basic architecture of the feed forward neural network with accompanying equations that describe the transfer functions between layers.

Predicted adhesion strength compare with experimental results based on different feed rate

The prediction value of neural network was tested with 12 data sets from the original process data. Each data set contained inputs such as torch input current and an output value i.e. Adhesion strength was returned by the network. Fig 4.10 presents the comparison of predicted output values for coating adhesion strength with those obtained experimentally for both cupper and Mild Steel substrate, which is done at constant 12 gm/mm powder feed rate and 100mm torch to base distance with change in power level. The predicted results show good agreement with experimental observations, which assure to find out the adhesion strength by taking different parameters for future work. Here it is clearly observed that the predicted plot goes in the same way as that of experimental i.e. by increasing power level the adhesion strength increases up to a certain limit and no increase in adhesion strength with further increasing power level. Again for confirmation by changing feed rate to 18 gm/min and torch to base distance 140 mm, the plot for both Cupper and Mild Steel changed (shown in Fig 4.18) with good agreement between predicted value and experimental value.

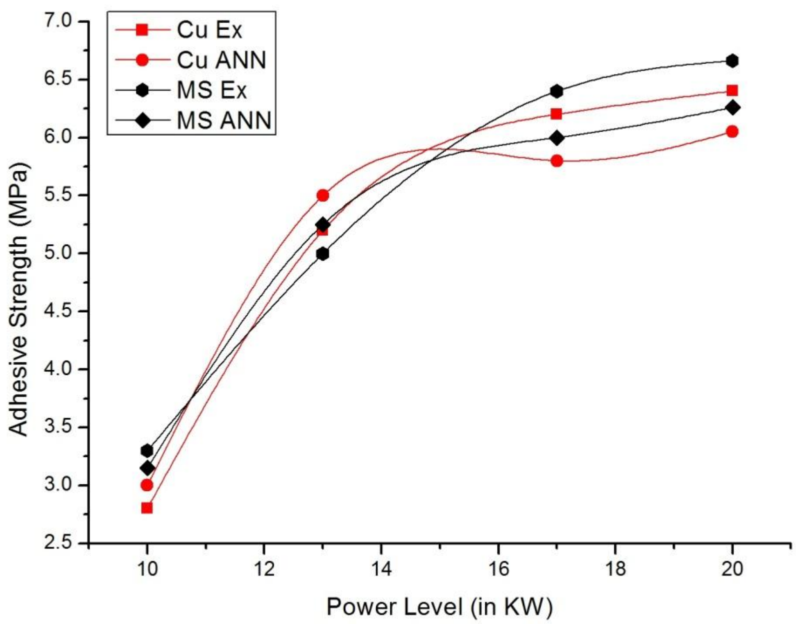


Figure 4.10 Comparative plot of experimental and ANN predicted values of adhesion strength of fly-ash+quartz+illuminite on Copper & Mild Steel substrate (Plasma spray at 12 gm/min feed rate and 100 mm torch to base distance).

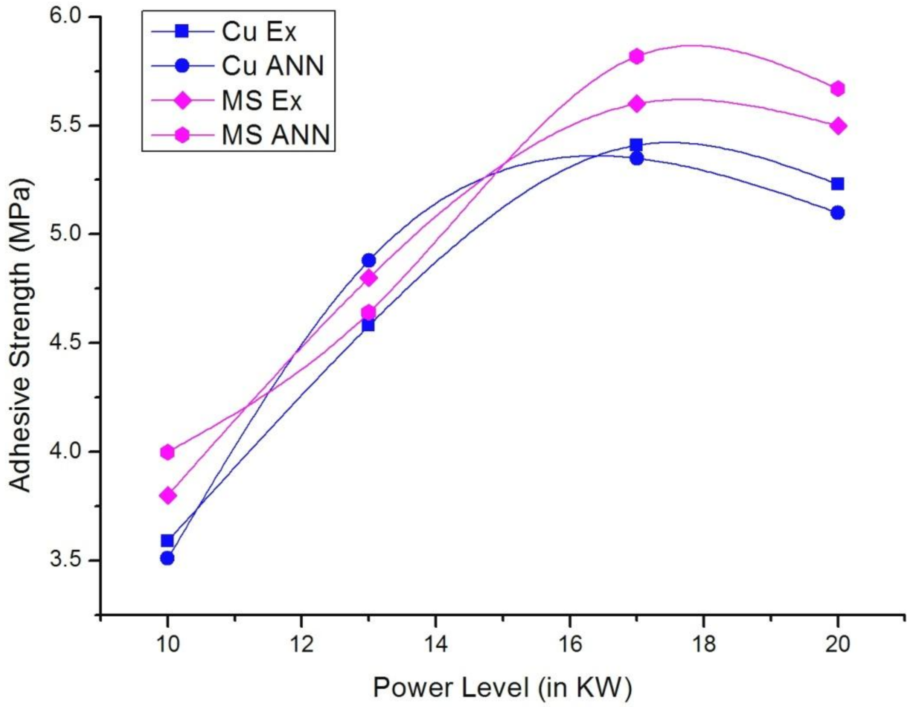


Figure 4.11 Comparative plot of experimental and ANN predicted values of adhesion strength of fly-ash+quartz+illuminite on Copper & Mild Steel substrate (Plasma spray at 18 gm/min feed rate and 140 mm torch to base distance).

Comparison between Mild Steel & Copper Substrate in Account of ANN Predicted Adhesion Strength Results

In the Fig 4.11, it is clear that the adhesion strength increases with respect to power level from 10KW to 17 KW and further increase in power level there is no change in adhesion strength. It reveals that for 12 gm/min feed rate with 40µm powder size and 100mm torch to base distance, one should choose ~17 KW power level for better spray coating. If greater than 17 KW power level choose, then there will be loss of process efficiency [9]. Here it can know that, there is decrease in adhesion strength, by increasing power level to 20 KW. The reason is due to higher thermal energy at 20 KW, there is more vaporization of powder generate for which splats are connected before deposited at the surface of the substrate and there are many cavity formed at interlayer space. But it is found that the surface roughness at 20 KW is very less as comparison to 17 KW, because some particles are deposited from their vaporized condition. From the prediction plot in fig 4.12, the adhesion strength of mild steel always greater than that of copper.

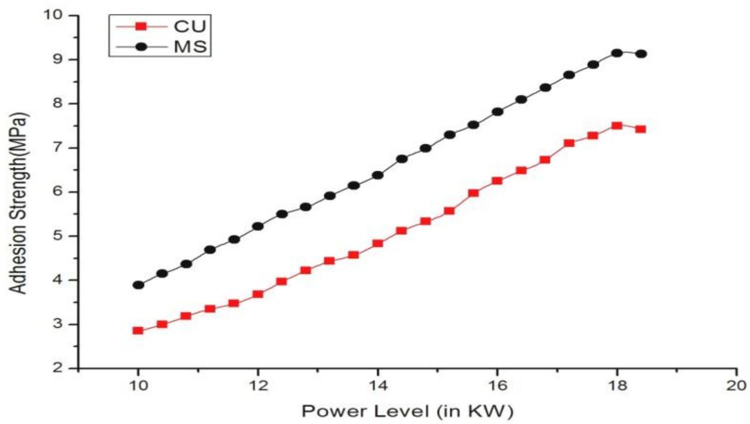


Figure 4.12 Predicted adhesion strength of Copper & Mild Steel substrate with respect to different power level (Plasma spray of fly-ash+quartz+illuminite at 12 gm/min feed rate and 40µm powder size, 100mm torch to base distance).

Prediction results based on powder particle size

In case of cupper, at 12 gm/min feed rate and 100mm torch to base distance, it is observed from Fig 4.13 that, higher is the powder size, lower is the adhesion strength and there is nearly uniform increase in adhesion strength for each plot. From ANN calculation by choosing this set of parameter with 40µm powder size, the adhesion strength for cupper substrate will be 5.48MPa at 20 KW power level. But in case of Mild steel as shown in Fig 4.13, at 12 gm/min feed rate and 100mm torch to base distance, the increment value of adhesion strength is very low in between 10KW to13KW and then uniformly increases up to 17 KW power level. Higher adhesion strength will be 9.00MPa for mild steel with 40µm powder size. From Fig 4.13 & 4.14 plots, it can be seen that the plasma spray will give better result at ~17KW to 17 KW power level.

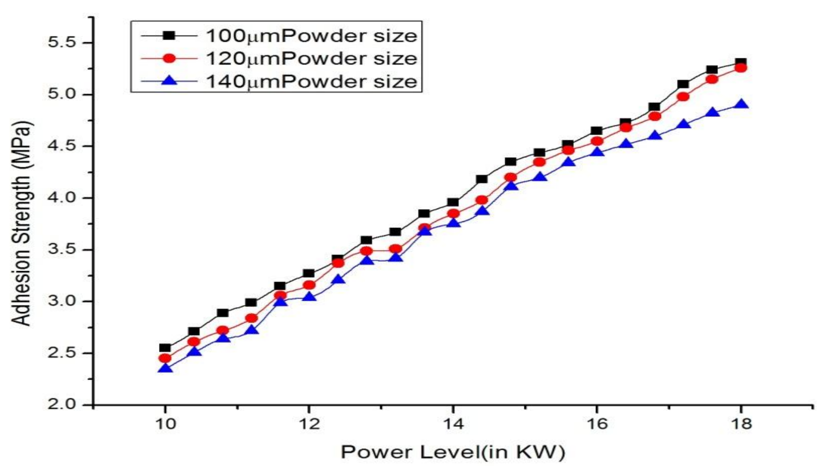


Figure 4.13 Predicted adhesion strength vs Power level for Copper by change in size of powder (Plasma spray of fly-ash+quartz+illuminite at 12 gm/min feed rate and 100 mm torch to base distance).

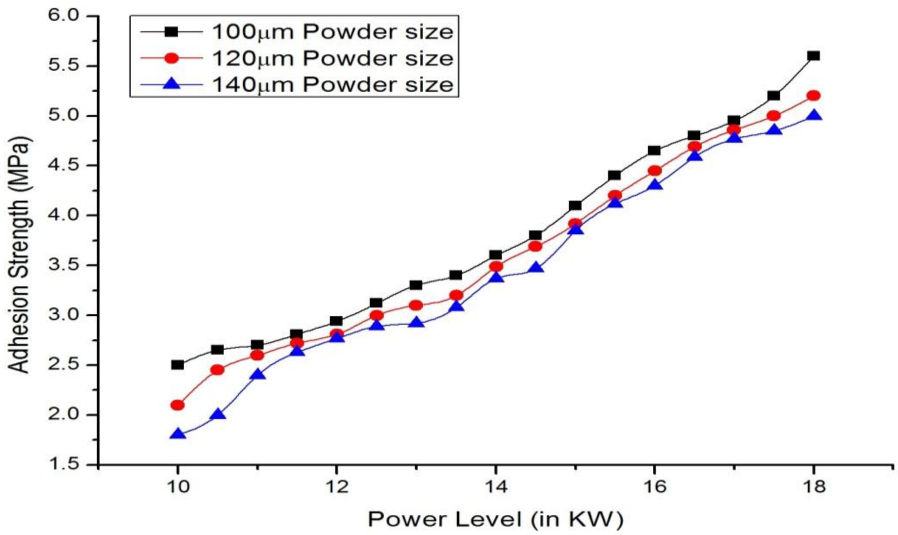


Figure 4.14 Predicted adhesion strength vs Power level for Mild Steel by change in size of powder (Plasma spray of fly-ash+quartz+illuminite at 12 gm/min feed rate and torch to base distance 100mm).

Prediction results based on torch to base distance

The adhesion strength decreases by increasing the torch to base distance, which is clearly observe in Fig 4.15. For smaller powder size (30 µm), the adhesion strength is better than that of higher powder size (70 µm, 90 µm and 120 µm), at 17 KW power level and 12 gm/min feed rate. In case of cupper, for 50 µm powder size the highest adhesion strength 6.66 MPa will be achieve at 50mm torch to base distance. The Fig 4.16 (for mild steel) gives the same idea for coating by different particle size with respect to torch to base distance.

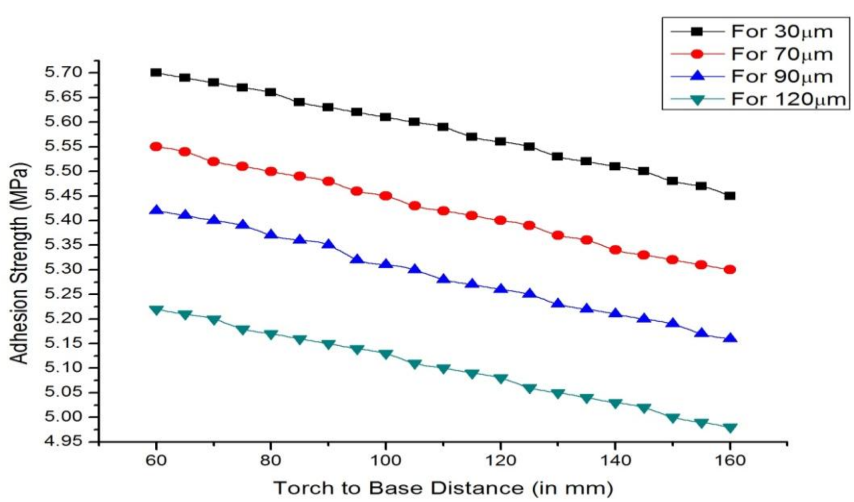


Figure 4.15 Predicted adhesion strength vs torch to base distance for Copper by change in size of powder (Plasma spray of fly-ash+quartz+illuminite at 17 KW power level and 12 gm/min feed rate).

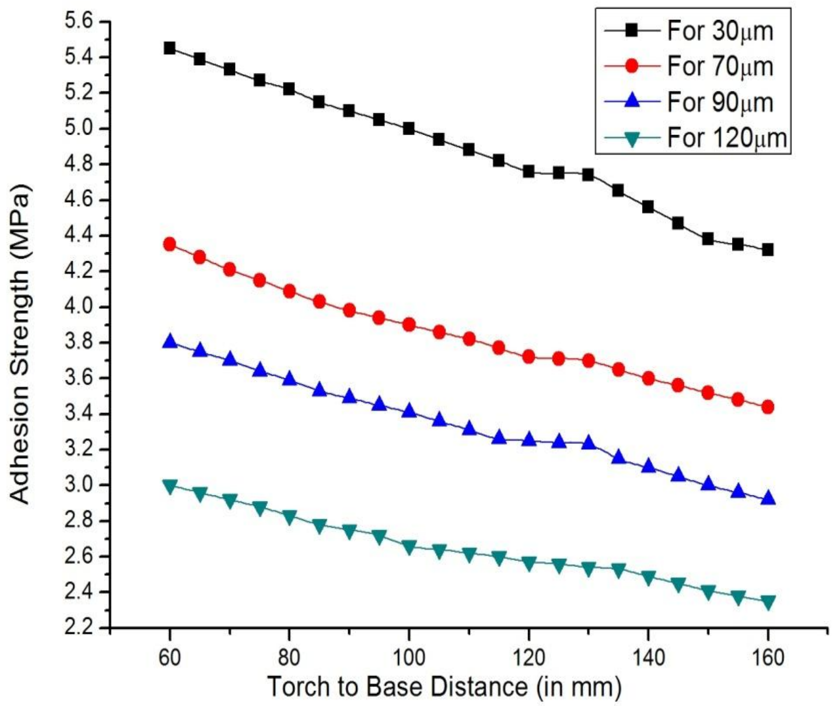


Figure 4.16 Predicted Adhesion strength vs torch to base distance for Mild Steel by change in size of powder (Plasma spray of fly-ash+quartz+illuminite at 13KW power level and 12 gm/min feed rate).

4.4 EVALUATION OF COATING PERFORMANCE

4.4.1 Erosion Wear Behaviour of Coatings

Erosion wear is a nonlinear process with respect to its variables: that are materials and operating conditions. To obtain the best functional output of coatings exhibiting selected in-service properties, the right combinations of operating parameters are to be known. These parameter combinations normally differ by their influence on the erosion wear rate i.e. coating mass loss. Statistical methods are commonly used to improve the quality of a product or process. Such methods enable the user to define and study the effect of every single condition possible in an experiment where numerous factors are involved. In case of plasma spray coatings encountering such situations, no specific model has been developed and thus the study of their erosion behaviour has been mostly based on experimental data [23, 32]. The less erosion wear rate is one the main requirements of the coatings developed by plasma spraying. In order to achieve certain values of erosion rate accurately and repeatedly, the influence parameters of the process have to be controlled accordingly. Since the number of such parameters in plasma spraying is too large and the parameter-property correlations are not always known.

4.4.2 Microstructural Investigation of Erodants and Eroded Surfaces.

The morphology of the erodent (i.e. SiC) is shown in Fig 4.17. It can be visualized that silicon carbide particles are having multiple angular facets and sharp edges. This might be the cause of fast rate of removal of material through chipping/ plowing of the surface when silicon carbide is used as erodent, thereby leading to higher amount of material loss (i.e. cumulative mass loss). Hence erosion rate is higher. So there is negligible crack initiation/formation and major chipping portions are observed on the eroded surfaces.

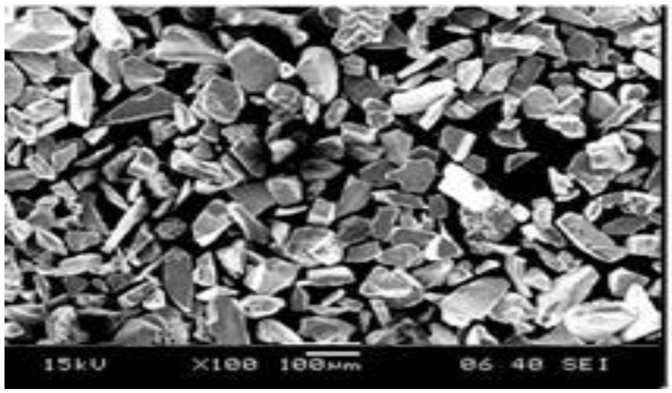
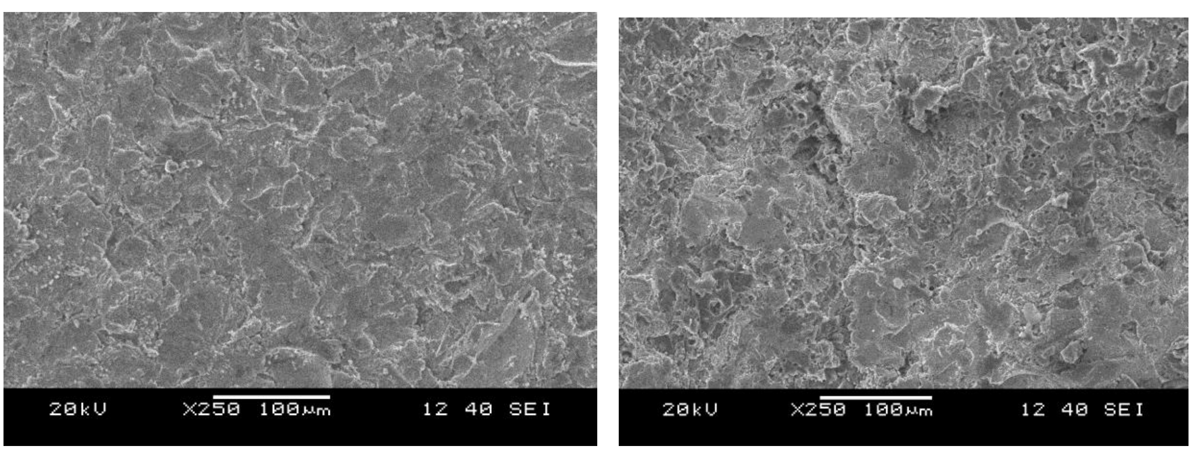


Figure 4.17 Surface morphology of SiC particles.

Morphology of Eroded Surfaces

Surface morphology of fly ash-quartz coating eroded with SiC erodent at different impact angles are shown in Fig 4.18.



(a) **(b)**

Figure 4.18 SEM micrographs of eroded surfaces of coatings deposited at 17 KW at angle of impact **(a)** 30° **and (b)** 90° using SiC as the erodent.

Fig. 4.18 (a) and Fig 4.18 (b), show the surface morphology of worn surfaces, when the erodent is impacted at 30° and 90° respectively. Some small cracks are observed and spread along splats boundaries. At 30° angle of impact, there are many angular grooves occurs. At 90° angle of impact, sharp vertical groves are seen which is due to dominating effect of perpendicular component of the force of impingement of the erodent. In general it can be stated that when hard and multifaceted erodent i.e. SiC is used, deeper groves are produced and plastically flow regions are found and also limit the surface crack propagation.

4.5 DISCUSSION

In this investigation, it is found that the adhesion strength increases up to a certain limit (i.e. up to 17 KW) then further increasing in power level there is no significant change in adhesion strength. Similar trend is also seen in case of deposition efficiency which is a measure of the amount of material deposited per unit surface area. It is observed that, the coating thickness acquires a maximum at 13 KW input power and then decreases. Variation of coating hardness with power level reveals that, there might be formation/transformation of allotropic forms and their compositional variations during spray deposition. To assess the suitability of these coatings for tribological applications, solid particle erosion wear behaviour is studied. With increase in impact angle erosion wear increases and attains maximum at 90°. The angle of impact determines the relative magnitude of the two components of the impact velocity namely, the component (i.e. perpendicular and tangential) normal to the surface and parallel to the surface. The normal component determines/is responsible for the lasting time of impact (i.e. contact time) and the load. The product of this contact time and the tangential (parallel) velocity component determines the amount of sliding that takes place. The tangential velocity component provides a shear loading to the surface in addition to the normal load of the normal velocity component. Hence, on changing the angle the amount of sliding that takes place also changes and also the nature and magnitude of the stress system changes. Both of these aspects influence the way a coating/material wears. Statistical modelling/analysis techniques viz. ANN, can be very much useful to predict the experimental data with conducting least number of experiments; and also can predict experimental results beyond the parameter domains used in the experimentations.

Chapter 5

Conclusions

Chapter 5
Conclusions

The conclusions drawn from the research are as follows:

- Fly ash, the waste generated from thermal power plants may be eminently coatable on metal substrates when mixed with quartz and illmenite, the low grade mineral ore, employing atmospheric plasma spraying technique.
- Such coatings possess potential coating characteristics such as good hardness, adhesion strength, etc.
- Operating power level of the plasma torch influences the coating adhesion strength, deposition efficiency and coating hardness to a great extent. The coating morphology is largely affected by the plasma input power.
- Adhesion strength of the coatings varied with the plasma power level and the kind of substrate on which coatings are deposited. Maximum adhesion strength of 6.66 MPa on mild steel substrate and 6.40 MPa on copper substrate are obtained.
- Coating adhesion is better in case of mild steel substrate than that of copper substrate.
- A maximum deposition efficiency of 49 % could be achieved for mild steel and 47 % for copper substrates and the deposition efficiency has increased in a step up fashion with the increase in torch power input.
- Due to phase transformations and inter-oxide formation during plasma spraying, changes in coating characteristics such as hardness etc. are observed.
- The coatings developed in this work are much harder than substrate metals on which they are deposited. Hence these coatings can be recommended for tribological applications.

- Maximum erosion of the coatings took place at an impact angle of 90^0.

- Microstructure of the eroded surfaces suggests that, when hard and multifaceted erodent i.e. SiC is used, deeper groves are produced and plastically flow regions are found and also less amount of surface crack propagation.

SCOPE FOR FUTURE WORK

The present work opens up a wide area for future investigators to explore the possibility of development of new fly-ash composite materials to ascertain further improvement in coating properties with considering cost of coating material.

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