B. TECH. PROJECT REPORT

On

Deposition of Copper Based Composite Coating using Novel Explosive Spray Coating Method

BY

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DEPARTMENT OF MECHANICAL ENGINEERING

INDIAN INSTITUTE OF TECHNOLOGY INDORE

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Deposition of Copper Based Composite Coating using Novel Explosive Spray Coating Method

A PROJECT REPORT

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

of
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in
MECHANICAL ENGINEERING

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INDIAN INSTITUTE OF TECHNOLOGY INDORE
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CANDIDATE'S DECLARATION

We hereby declare that the project entitled **"Deposition of Copper Based Composite Coating using Novel Explosive Spray Coating Method"** submitted in partial fulfilment for the award of degree of Bachelor of Technology in 'Mechanical Engineering' completed under the supervision of **Dr. Kazi Sabiruddin, Associate Professor, Department of Mechanical Engineering,** IIT Indore is an authentic work.

Further, I/we declare that I/we have not submitted this work for the award of any other degree elsewhere.

21.05.2022

Signature and name of the student(s) with date

Nishit Agrawal & Nikhil Attri

CERTIFICATE by BTP Guide(s)

It is certified that the above statement made by the students is correct to the best of my/our knowledge.

20/05/22

Signature of BTP Guide(s) with dates and their designation

Dr. Kazi Sabiruddin, Associate Professor

Preface

This report on "Deposition of Copper Based Composite Coatings using Novel Explosive Spray Coating Method " is prepared under the guidance of Dr. Kazi Sabiruddin. Through this report we have tried to give a detailed observation on Cu based composite coatings on low carbon steel deposited by novel explosive spray methods. While our work was preliminary focused on characterisation of the deposited coatings, it can be carried forward in multiple dimensions in the future as explained in the future scope.

We have tried to the best of our abilities and knowledge to analyse and explain the content in a lucid manner. We have also added graphs and figures to make it more illustrative.

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Acknowledgements

We wish to thank Dr. Kazi Sabiruddin (Associate Professor, ME) for his kind support and valuable guidance. His dedication towards the subject helped me in pushing boundaries and achieving more. It is through their help and support, due to which I became able to complete the design and technical report.

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Abstract

Micron sized Copper powder is sprayed on steel substrates with the help of a novel explosive spray coating set up developed in the laboratory by burning a pyrotechnic powder mixture (Barium nitrate + Al) in the gun barrel in a controlled manner. A thick Cu-based composite coating is developed on the pre-treated and pre-heated low carbon steels. Different characterisation methods such as XRD, FESEM, Microhardness tests, scratch test etc. are adopted to characterise the deposited coating. A major presence of Cu is noticed near the top surface of the coating. The coating is monolithic in nature with pores, cracks, and amorphous phases present in it. The average hardness of the coating is found to be high due to the presence of hard phases such as $BaAl_2O_4$, Fe_3N, AlN, etc. in it. The adhesion strength of the coating is found to be remarkably high due to the strong mechanical anchorage offered by the substrate asperities towards the coating material and the inter-diffusion of materials between the coating and the substrate at the interface zone.

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Chapter 1
Introduction

An **EXPLOSIVE** is a highly reactive substance that contains a huge amount of potential energy that, when released suddenly, can cause an explosion, usually followed by the generation of light, sound, heat, and pressure. Chemical explosives are classified into two types: 1. detonating or high explosives, and 2. flaming or low explosives High explosives, such as TNT and dynamite, are distinguished by extremely rapid decomposition and the production of high pressures, whereas low explosives, such as Flash, Gun, and Smokeless powders, are distinguished by fast burning and the production of relatively low pressures. The majority of explosives are heat, flame, shock, oxygen, or UV radiation sensitive. Some liquid explosives, such as nitro-glycerine, are used to make propellants, dynamites, and so on. But most of the solid stable Explosives are either used for Military purpose explosion or for fireworks.

Metals have been the preferred material for a variety of applications for millennia due to its durability, adaptability, and strength. However, corrosion is undoubtedly the most common and well-known difficulty that people confront while working with metals. For various metals, multiple treatments have been offered to extend structural lifespan and improve mechanical and electrical properties. Coating metallic surfaces can be done in a variety of ways, each with its own set of drawbacks and advantages. Copper is widely used as a coating material in the fabrication sector.

Copper coating is a significant process because copper has several properties such as:

- High corrosion resistance.
- High wear resistance.
- Excellent thermal and electrical conductivity.
- Exceptionally good adhesion to most base metals.

Some of the most commonly used metal coating methods used across various industries are:

1. Anodizing is a process that encourages the formation of a protective oxide layer on the surface of a metal. The resulting oxide layer forms faster and thicker than it would naturally. Aluminium is the most effective non-ferrous metal for anodizing. The aluminium component is anodized by immersing it in a tank containing an electrolytic solution and a cathode (usually aluminium or lead). Aluminium oxidises and forms a protective barrier when an electrical current is passed through it.
2. Galvanizing is the process of immersing a metal (typically steel or iron) in a bath of molten zinc. When the coated metal is removed, the oxygen and carbon dioxide in the atmosphere react to it to form a protective zinc carbonate layer. Galvanizing offers numerous benefits, making it a popular choice for a wide range of applications. For example, the zinc oxide coating is highly stable and adheres tightly to the metal substrate; it is extremely durable and does not flake off easily.
3. The process of depositing a thin layer of one metal on the surface of another metal is known as electroplating. During electroplating, both metals are immersed in an electrolytic solution. The anode is the metal that will be coated, and the cathode is the metal that will be coated. An electric current is passed through the electrolytic cell, causing metal ions to move from the cathode to the anode and causing the coating to form. Electroplating improves some of the mechanical properties of metals and provides excellent corrosion resistance. Electroplating also produces an eye-catching surface finish, making it ideal for coating jewellery and ornaments.
4. Powder coating, as the name suggests, is the process of coating an object with a powder-based substance. It is an electrostatic process in which the coating particles are electrically charged with the opposite polarity as the part to be coated. Because of the charge difference, the powdered particles adhere to the metal's surface. After that, the coated object is heat-treated in an oven to harden the coating. Powder coatings are known for their long durability and attractive look.

Chapter 2

Literature Review

This chapter gives a detailed information of the research carried out in the past on various methods used in order to achieve copper coating on various substrates

2.1 High-Energy Plasma Arc Spraying

The deposition of coating for a variety of purposes onto equipment and equipment surfaces is in high demand across a variety of manufacturing sectors. The combination of copper-Aluminium circuit breakers with the ability to provide low contact resistance and high tightening torque or high compressive force is a problem in the electrical industry. In this work, it is proposed to use high-energy plasma spraying for reliable copper and aluminium connection by copper plasma spraying on the inner conical aluminium contact surfaces. For this purpose, a unique configuration of a high-energy plasma accelerator was developed, which ensures the production and uniform spraying of Cu material in a single short-time pass (less than one m/s) [1].

2.2 Copper Coating on Graphite

Conditions were optimized to achieve 2.5 m thick continuous copper coatings on 4-200 diameter graphite particles by cementation process using aqueous and supersaturated copper sulphate solutions. To improve the wettability of graphite, both the aqueous and supersaturated solutions processes required activation of the graphite particles by heating at a temperature of 380 °C for one hour and adding 0.2% by volume of glacial acetic acid. The particles were coated in an aqueous solution process (suitable only for graphite sizes 40-200 µm) by suspending them in mechanically stirred 0.2 M Copper Sulphate Pentahydrate solution at 25 °C. For graphite particles ranging in size from 2 to 200 µm, a supersaturated solution process was required. A slurry of two parts activated graphite powder, seven parts Copper Sulphate Pentahydrate, one-part glacial acetic acid, two parts zinc granules, and one part water were used. Graphite particles coated with copper using either method described in this article could be introduced and retained in melts of aluminium base alloys to produce a graphite dispersion in cast aluminium alloys [2].

2.3 Effect of Stand-off Distance and Pressure in Copper Coatings

The result of particle rate on deposition potency and microstructure of copper powder on atomic number 13 alloy substrates victimization depression Cold Spray (LPCS) is here rumoured. For this, experimental substrate-coating systems were invented with varied powder feed rate, cross speed, and atmospheric pressure keeping the compressed gas, substrate temperature, and stand-off distance constant (600 °C, 100 °C, and 5 mm, severally). From the mixture of low feed rates and low cross speed, deposition potency tends to extend. The most deposition potency of thirty-four.8% was obtained for a feed rate of 0.2 g/s, 10 mm/s cross speed, and eight bar of atmospheric pressure.

The pressure and stand-off distance result on coatings was studied by measurement the particle rate with and while not substrate. For this, spraying conditions were varied between 5–8 bar and 5–15 metric linear unit, respectively. Experimental measurements were compared with results from machine fluid dynamics simulation to grasp the result of the spraying parameters on the entire powder size distribution. The very best deposition efficiencies (36–37.5%) were obtained at seven bar atmospheric pressure for spray distances between five and twelve.5 mm. Increasing the pressure to eight bar junction rectifier to a decrease within the deposition potency (6–11%). The tendency of deposition potency appears to match with the in-flight particle rate influenced by the presence of a substrate, which is proof of the bounce-off result. The reduction of the particle rate, alongside the low cross speed appears to get rid of less connected particles by erosion through the peening result [3].

2.4 Characterization of Copper Coatings

Copper is thought to be a suitable and long-lasting corrosion barrier in the design of used nuclear fuel containers for deep geological repositories. The microstructures of cutting-edge copper materials produced by extrusion, a grain boundary engineered electrodeposition technique, and cold spraying were investigated using electron backscattered diffraction. Desirable microstructural properties for pure copper localised corrosion resistance were compiled from the literature, taking into account grain size, grain boundary character distribution, and crystallographic texture. The investigated copper materials were discovered to have favourable microstructures for localised corrosion resistance, specifically a high fraction of special grain boundaries, particularly 3 twins, making them suitable for the given application [4].

2.5 Fabrication of An Explosive Setup for Copper coating.

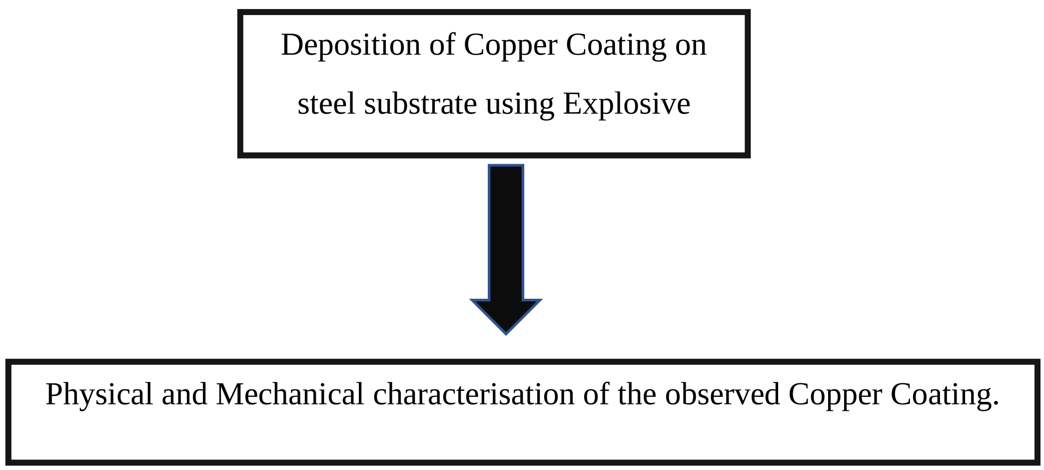
Mayank Sharma developed a feasible and ergonomical setup that used low explosives in order to achieve a Ni-Al coating on the steel substrate. The research has optimised the process parameters, which is perfectly suitable for coating with a good amount of adhesion. It shows the effect of various pre and post-treatment process on the observed coating and adhesion strength of the coated surface is dependent on several factors such as standoff distance, the surface roughness of the substrates, the distance between powders, etc [5].

2.6 Identified Research Gap

Based on the study of the previously done research it has been found that till date no work has been reported on the usage of explosives in the fabrication industry.

2.7 Objectives and Research Methodology

- Using the already designed explosive setup and the optimised process parameters to achieve a good adhesive copper coating on steel substrates.
- Mechanical and physical characterisation of the successful observed Copper coating on steel substrates.



Chapter 3
Materials & Methods

3.1 SETUP:

3.1.1 GUN BARREL –
A partially hollow cylindrical tube is casted using **AISI 1020 Carbon Steel.** Back of the Barrel is hollowed in order to make space for shock absorbing spring and the front cavity is for the placement of both the coating and the explosive powder.

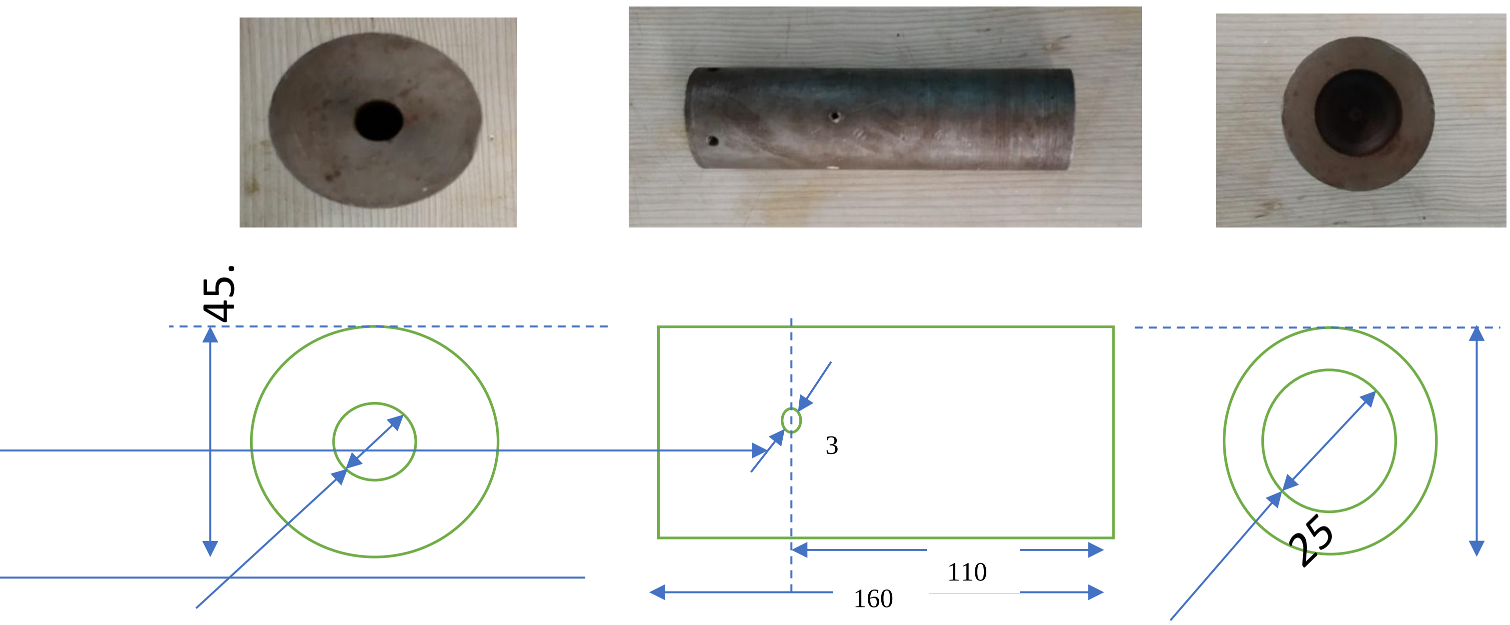


Figure 3.1 Gun Barrel Dimension

3.1.2 CASING –

- 12 holes exist in plate for holding different size of substrate.
- Horizontal slot of 9.50 mm exist in base plate for varying the stand-off distance.
- Two blocks are used for holding barrel.
- Spring Assembly is used of following specifications-

  Internal Diameter – 16 mm

  External Diameter- 24 mm

  Thickness Of Coil – 04 mm
- Detachable joint in between spring and barrel with the help of screw assembly

3.1.3 ELECTRIC CIRCUIT-

Components used and its specifications:

- Spring loader Rocker switch ->12V 3A
- Power Supply Adapter ->12V 3A
- Nichrome wire (Heating Element)**:**
  - **Size**:0.274mm diameter
  - **Resistance:**6.84 Ohms/feet
  - **Melting Temperature:**1500^0C
  - **Composition**:80%Nickel and 20%chromium
- Wire (Power supply)->Copper wire(3mm dia)

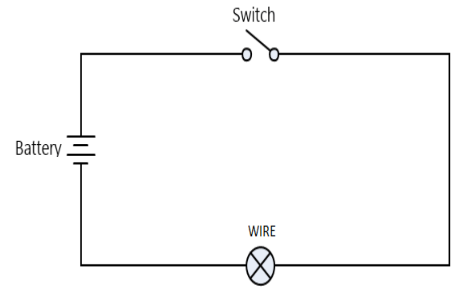


Figure 3.2 Electric Circuit

3.1.4 EXPERIMENTAL SETUP –

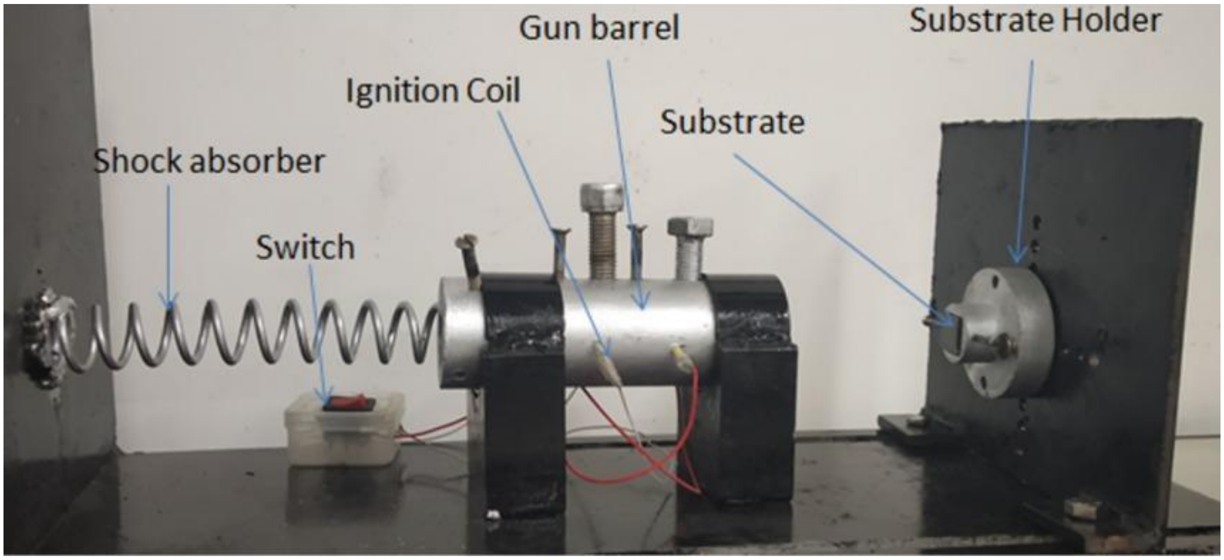
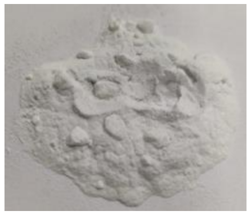


Figure 3.3 Labelled Explosive Setup

3.1.5 MATERIAL CONSUMED –

Copper Powder

Barium Nitrate Powder



Aluminium Powder

3.2 EXPERIMENTAL PROCEDURE –

3.2.1 Substrate Preparation:

Surface Preparation:

Surface preparation is a compulsory first up treatment of a substrate before the application of any type of coating for:

- Removal of Scale, oil, Greece.
- Improvement in adhesion of coating layer with the substrate.

Grit Blasting:

Dry abrasive particles are impinged on the substrate at high speed in the grit blasting technique to clean and change its surface topography. Roughness is generated using alumina oxide grits. For varying values of surface roughness, different process parameters are used. In our study, we have worked with the following grit parameters:

Table 3.1 Grit Blasting Parameters

Substrate	Grit type & size	Blasting pressure (bar)	Standoff distance (mm)	Blasting angle (degree)	Blasting time (sec)	Average Surface Roughness, R_a (μm)
Carbon steel (AISI 1020)	Alumina, 24 Mesh	5	100	90	60	3.5

Preheating the substrate:

Preheating the substrate before the coating deposition is essential for the removal of moisture and surface cleaning for better adhesion and it also helps to reduce the thermal stress with improved coating microstructure. Preheating is carried in the muffle furnace at around 300^0 C for about 15 minutes.

3.2.2 Coating Deposition:

Explosive Material:

A substance that can rapidly breakdown, releasing a significant amount of energy that, if released unexpectedly, can cause an explosion, usually accompanied by the production of heat, sound, and pressure. For the appropriate composition, various chemical powders were combined together. The composition of the explosive powder used in this study is shown below

Table 3.2 Flash Powder Composition

Name – Flash powder	
Elements	**Composition (weight %)**
Pyro (Aluminium Powder)	**33.33**
Barium Nitrate	**66.67**

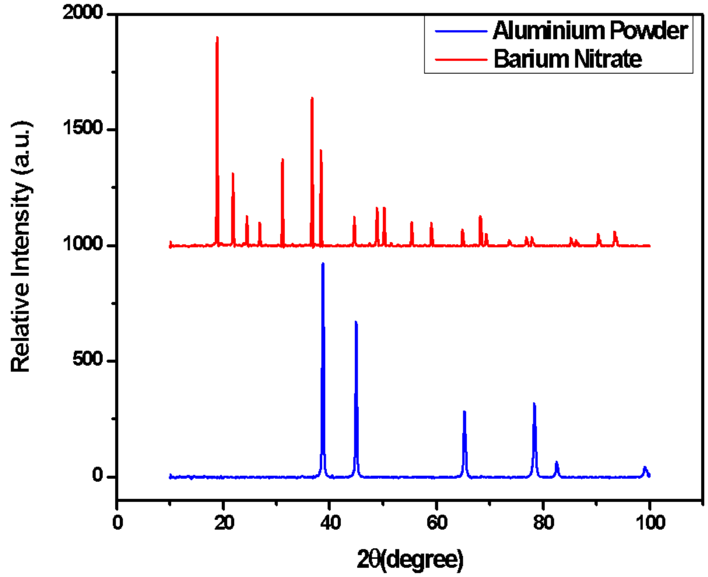


Figure 3.4 XRD of Pure Aluminium and Barium Nitrate

Flash Powder composition consists of a mixture of oxidizer and a metallic fuel, which burns quickly and if confined produces a loud explosion. **Figure 3.4** represents the XRD diffraction pattern of powder used in study. A simplified equation for the flash powder is as follows:

$$\mathbf{10Al + 3Ba(NO_3)_2 \rightarrow 5Al_2O_3 + 3BaO + 3N_2 + Heat}$$

Coating Material:

Copper fine Powder (99.7%) 63.56MW is used as a coating material to be deposited on steel substrate.

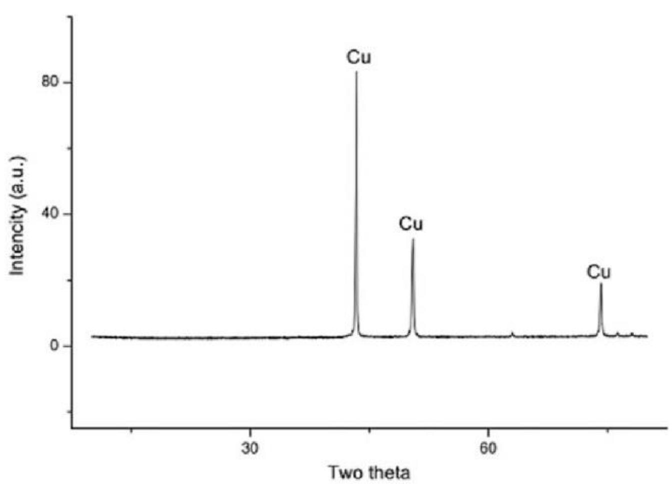


Figure 3.5 XRD of Pure Copper

Selection of Process Parameters :

Process parameters plays a vital role in mechanical and physical properties of the achieved coating surface. In previous studies various experiments were performed in order to study the influence of process parameters such as Standoff Distance, Distance between powders, Ignition system types, Number of ignition points, Surface roughness of the substrates. In our present study we are directly utilising those optimised values in order to achieve efficient coating. Some of these parameters are shown in section view of the gun barrel below:

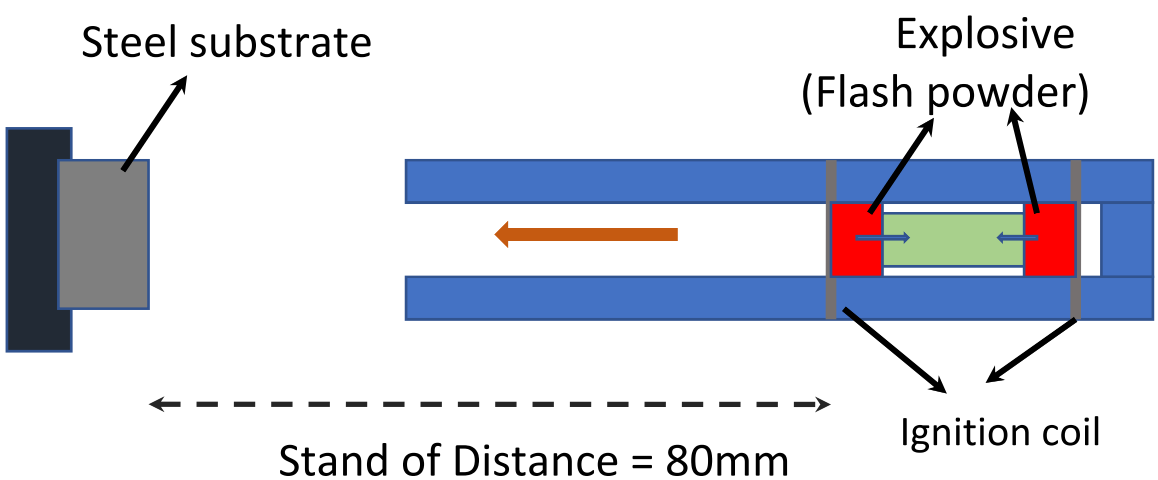


Figure 3.6 Two Point Ignition System

Performing Coating Deposition :

For Depositing Copper powder over the AISI 1020 low carbon steel substrate by using the fabricated coating setup, the used process parameters are mentioned in the table below:

Stand- Off Distance	Quantity of Flash Powder	Quantity of Coating Powder	Ignition Type	Preheat Temperature Substrate	Preheat Temperatur e Powders
80 mm	3 gm	1 gm	Two point	350 ^{0}C	100 ^{0} C

Table 3.3 Process Parameters

3.3 Coating Characterisation:

3.3.1 X – Ray Diffraction:

X- Ray Diffraction (XRD) analysis of the coated surface are done by using an X- Ray Diffractometer by Rigaku Smart Lab with nickel- filtered Cu Kα radiation (λ= 0.1540 mm) operating at 30kV and 40mA. Obtained X-Ray Diffraction data is then analysed for the coating by using X'Pert High score Plus® (Phillips) analysis package.

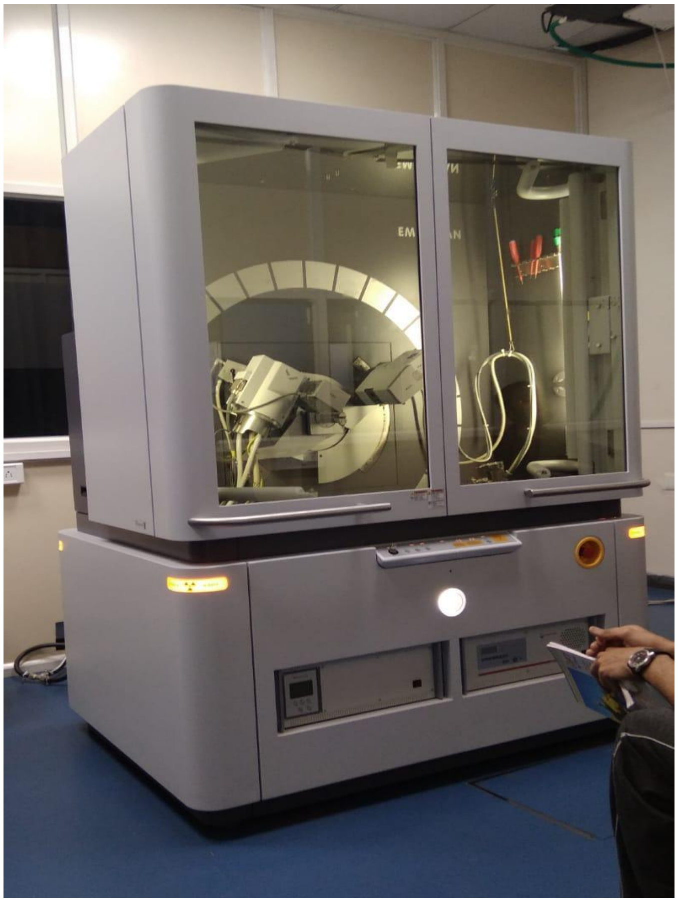


Figure 3.7 X-Ray Diffractor

3.3.2 Microstructure Study:

Field emission scanning electron microscope (FESEM, Zeiss Supra-55) is used for the characterization of the microstructure of the coating and the substrate. The sample is firstly cut, then mounted using black resin in a cylindrical shape on (Buehler SimpliMet 3000), and its cross- section is then polished to attain the desired micro structure of the coating. The polishing is done on (Struers LaboPol 25) in a proper metallographical manner which is starting from the sandpaper of grit size 80 to grit size of 3500 and then polished on the selvyt polishing cloth using diamond paste on it. The cross- sectional images of coating is then analysed visually.

Figure3.8 FESEM

Figure3.9 Polishing Machine

3.3.3 EDS Analysis:

EDS Analysis is done on the coated sample in the cross-section side of coated surface. EDS analysis shows the detailed information on the presence of different elements in the cross-section of the coating.

3.3.4 Coating Roughness Profile Evaluation.

The roughness of the deposited coating is evaluated by Taylor Hobson, Surtronic 25 contact type Stylus Profilometer. This evaluation shows us the average surface roughness (Ra) and the 2D profile of the coating.

Figure3.10 Contact Type Stylus Profilometer

3.3.5 Coating Adhesion Strength Analysis:

Scratch hardness of the coating is performed by Magnum High Load Scratch Tester. The test sample is polished (Ra<=1µm) to obtain scratch width measurement. A gradually increasing normal force is applied by the stylus to the test sample, and the scratch is made by the relative sliding movement of the stylus against the coating surface.

Figure 3.11

Scratch Tester

3.3.6 Microhardness Measurement :

The microhardness values of coating and steel substrate at the cross-section are obtained by Vickers' Hardness Tester VMH -002, Walter. Several indent are made on the cross section at different locations all over coating and substrate cross section and the average values for both are taken for investigation.

Figure 3.12

Vickers' Hardness Tester

Chapter 4
Results

4.1 FESEM micrographs

FESEM analysis of the polished cross section of the coating is shown in the figure 4.1. The analysis of FESEM images of cross section of polished coated sample describes spherical particles distributed all over the coating surface in a uniform manner. By doing EDS analysis we confirmed that these spherical particles were rich in copper and aluminium due to incomplete melting of copper and aluminium which are strongly adhered to the surface of substrate. FESEM micrographs depicts that there were various pores formation and crack development. Major reasons for pores formation can be escaping of gases which were released during the explosive reaction. Another reason for the same could be removal of loosely adhered unmelted copper while polishing the surface. Cracks are observed in the coated layer mainly due to amorphous phases present of Barium Aluminate which are brittle in nature and due intense shock waves and suction waves these cracks are formed. **ImageJ** software is used to evaluate the porosity which was found to be approximately 5.69%.

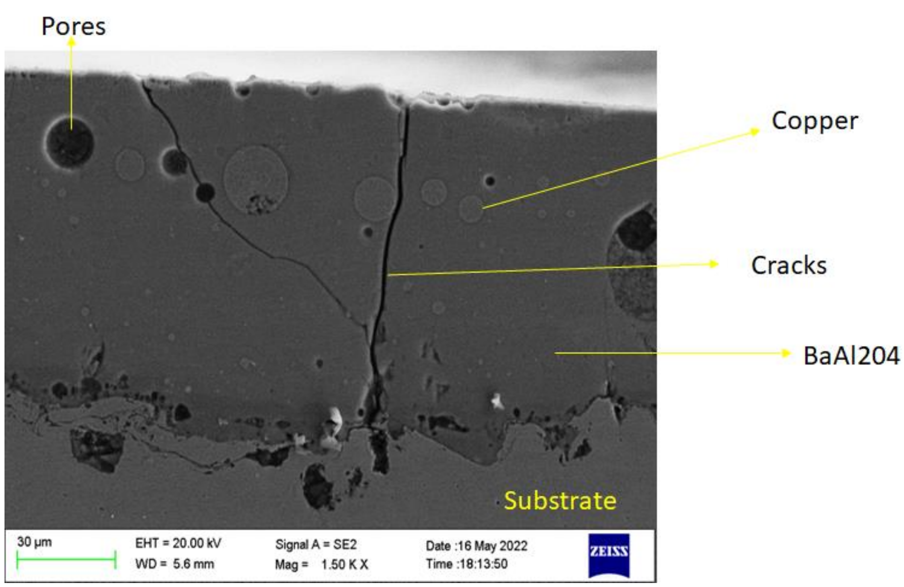


Figure 4.1 FESEM image of coating

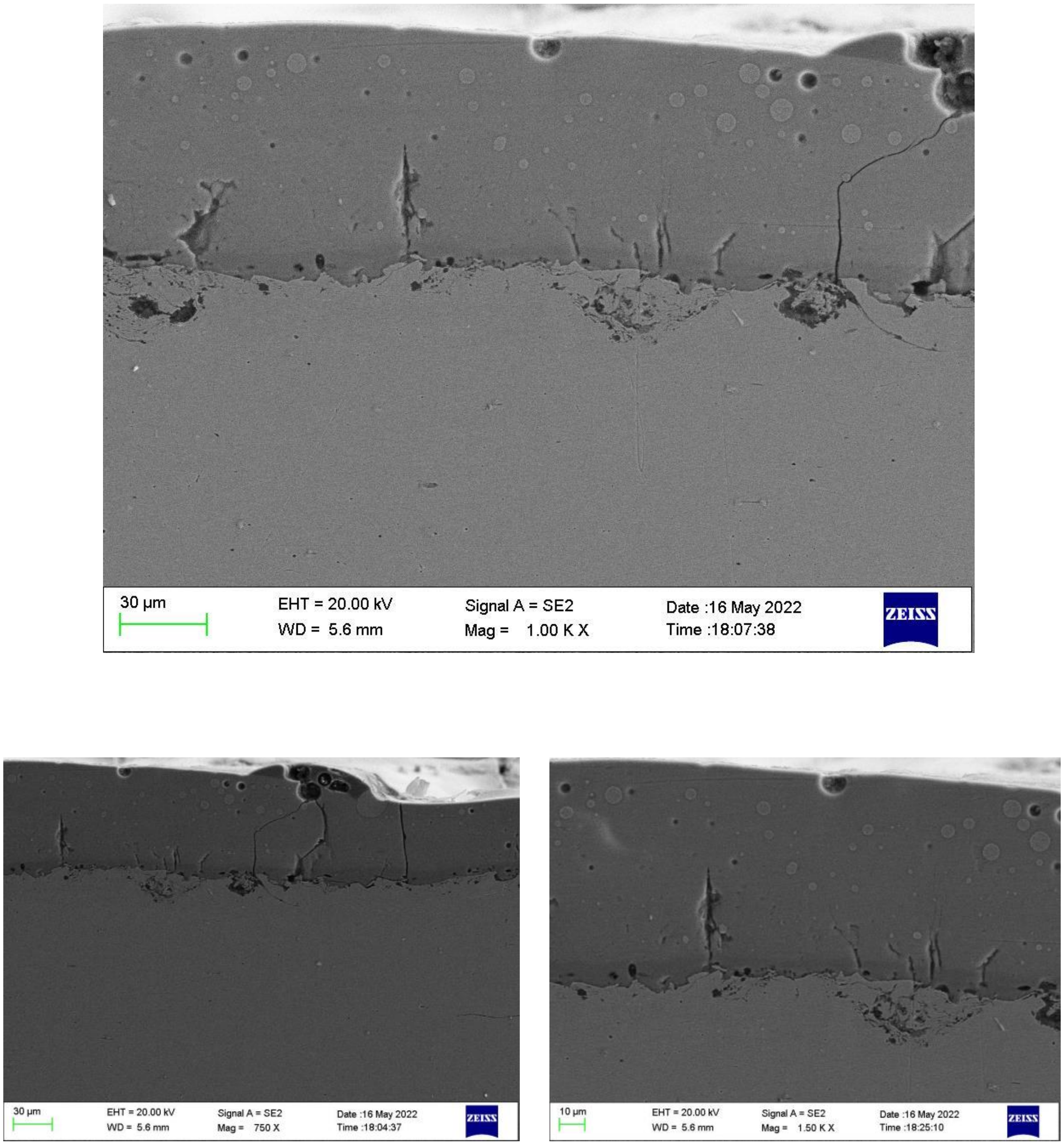


Fig 4.2 FESEM Images

FESEM images for cross section of the coating also give a accurate idea about the thickness of the observed coating. Results show that as it is explosively deposited coating method the thickness is non uniform over the entire substrate. Observed thickness value over the entire cross section of coating is 72.8 ± 18.2 microns.

4.2 EDS analysis

Figure 4.3 shows the EDS analysis of cross section of coated sample. It confirms the presence of copper and barium aluminate along the coating interface. EDS analysis was carried out on various zones which we are referring to spectrum in SEM micrographs. In these zones we found that there was presence of iron, nitrogen, oxygen also.

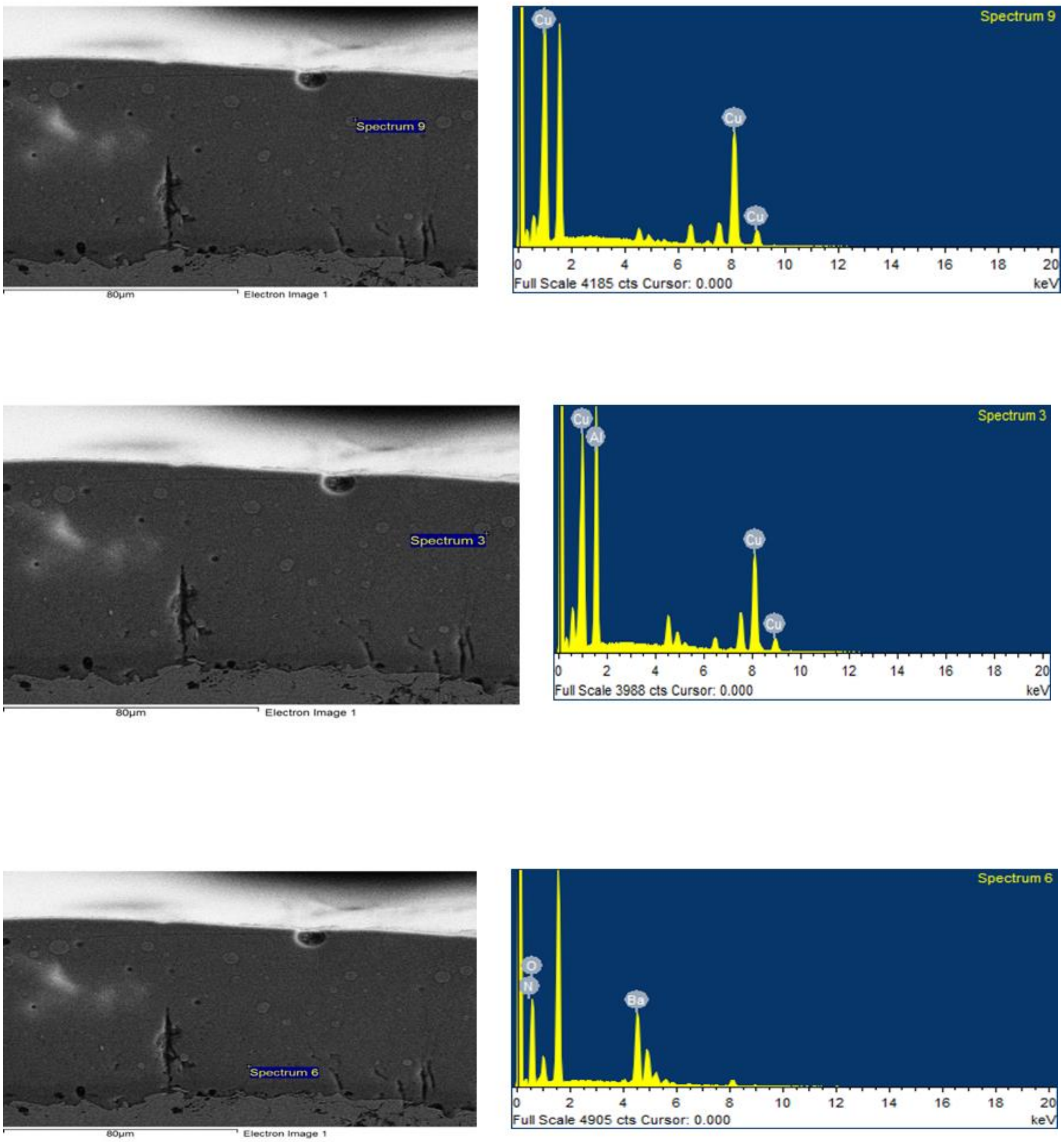


Fig 4.3 EDS Analysis on Coating

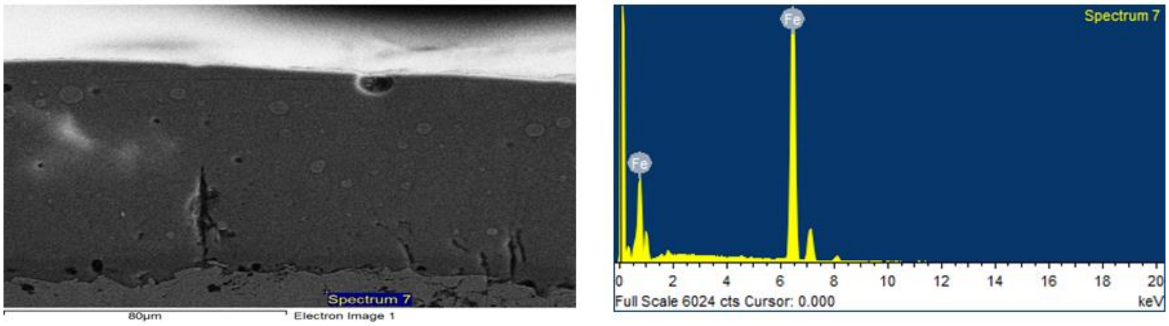
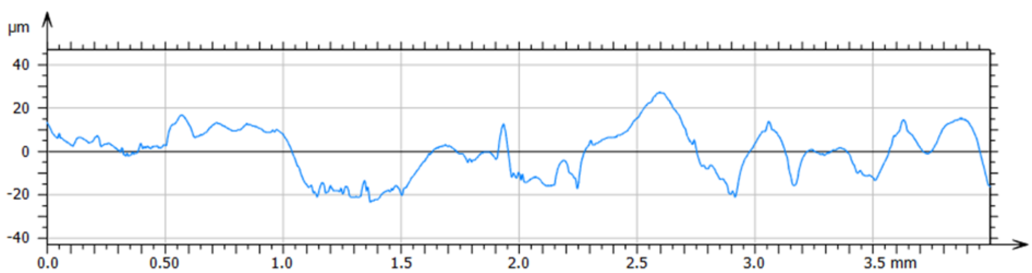


Fig 4.4 EDS Analysis on Substrate

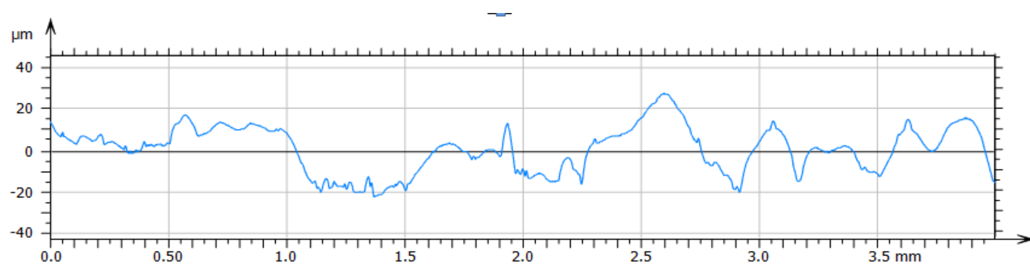
4.3 Average surface roughness (Ra)

Figures 4.5 shows the surface roughness profile of coating on steel substrate. The average value of **Ra** calculated using these graphs was found to be 5.7 ± 1.21µm. We observed that roughness values decreased in comparison to that of before coating grit blasted surface (Ra=8.7µm). It means with proper melting and uniform spreading of copper powder surface roughness improves significantly.



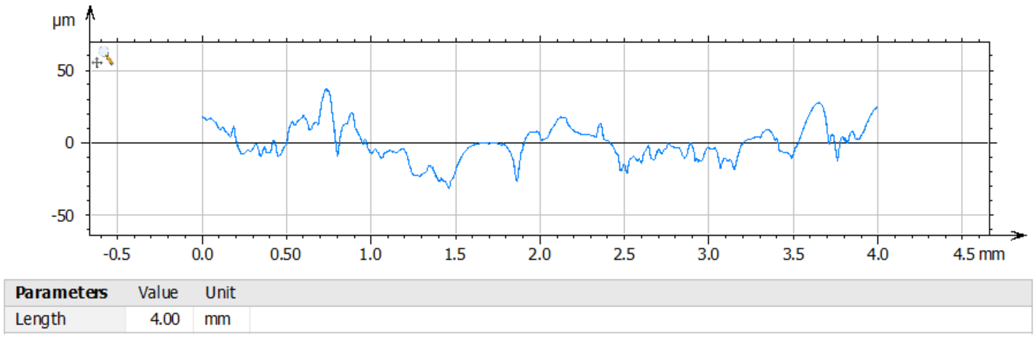
Parameters	Value	Unit
Length	4.00	mm

Ra=5.48um



Parameters	Value	Unit
Length	4.00	mm

Ra=4.68um



Ra=7.02um

Fig 4.5 Roughness Profile

4.4 X-ray diffraction analysis

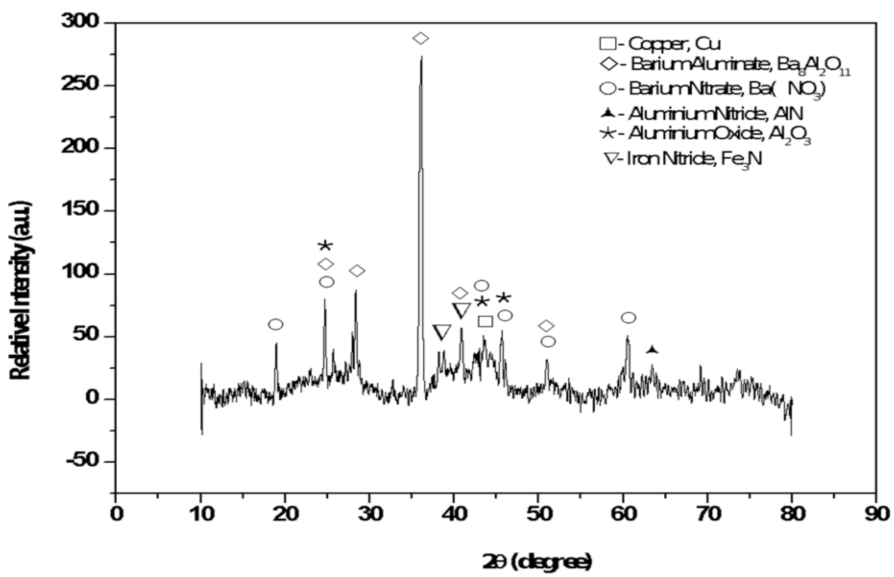


Fig 4.6 X-Ray Diffraction Analysis

Figure 4.6 shows the XRD analysis of a slightly polished coated sample. In the graph we can observe various alloys such as barium aluminate, aluminium oxide, iron nitride, aluminium nitride. By looking at the curvature of the XRD plot we can say that the phases present in the coating are amorphous in nature. The presence of the composites such as Barium Aluminate improves the surface properties of the coating. As some of these alloys are hard in nature, it significantly increases the hardness of the coating.

4.5 Vickers microhardness

4.5.1 Microhardness of substrates

Figure 4.7 shows the plot of microhardness of steel substrates versus distance from the interface. We observed that near the interface of sample hardness value is highest and as we move down from the interface hardness decreases and at a point 120 microns far from the interface hardness value becomes equal to that of substrates before coating. This shows that the region of substrate up to 120 microns is affected by coating or strain hardening takes place up to this point.

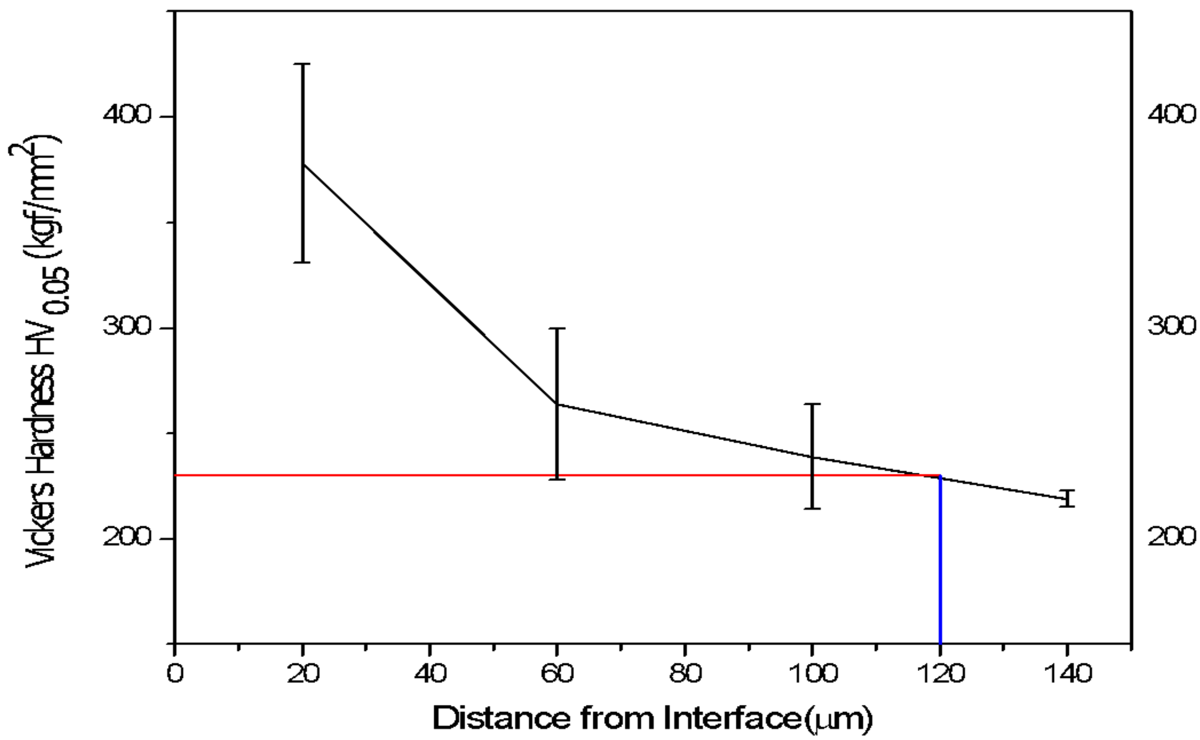


Fig 4.7 Hardness of Substrate

4.5.2 Microhardness of coating

Figure 4.8 shows the curve of coating. Various indents were made at different location of cross section of coating. The hardness value of same is calculated by using microhardness tester with a load of 50g, loading time 4s, dwell time of 10s and unloading time 4s . The microhardness value obtained for the copper coating was around 958HV0.05 whereas for the steel substrate value obtained was around 230 HV0.05. This improvement in hardness value for coating sample is due to the various phases such as aluminium oxide, barium oxide, iron nitride which are hard in nature. The presence of these phases in copper coating sample makes it a composite type of structure.

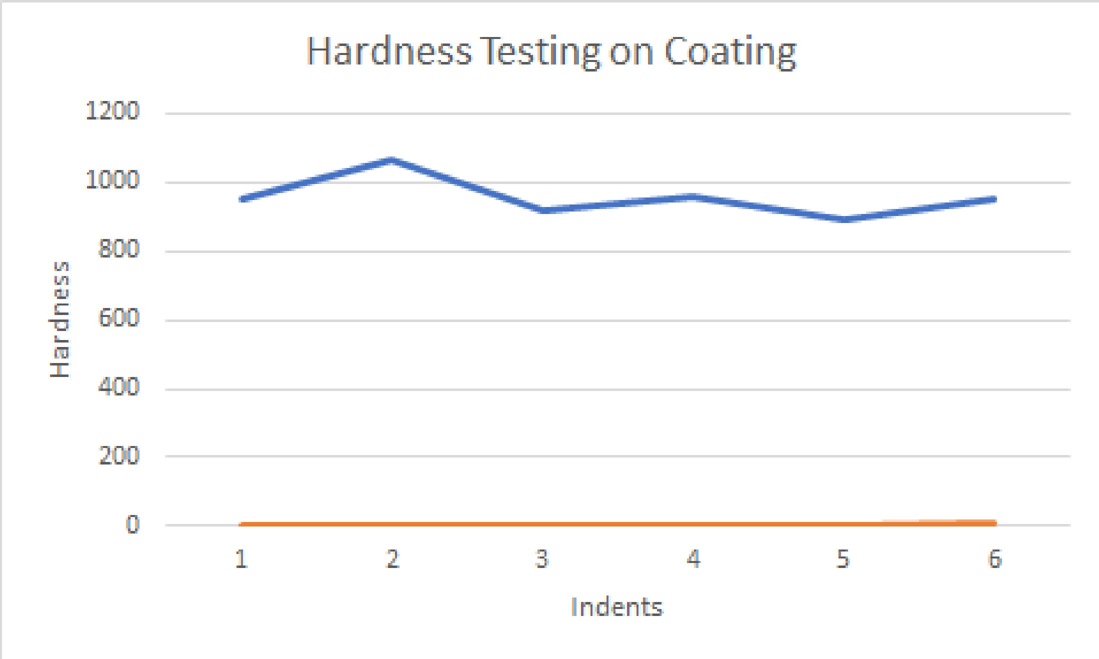


Fig 4.8 Hardness of Coating

4.6 Scratch Adhesion Strength :

High gradually increasing load scratches are performed on the polished top surface of the coating. Fig. X shows the optical microscopic image of four representative scratch marks on the coating obtained from the explosive setup. The complete scratch length of 11 mm, where the applied load is increased from 0 to 165 N, with a load rate of 15 N/mm is shown in the figure below.

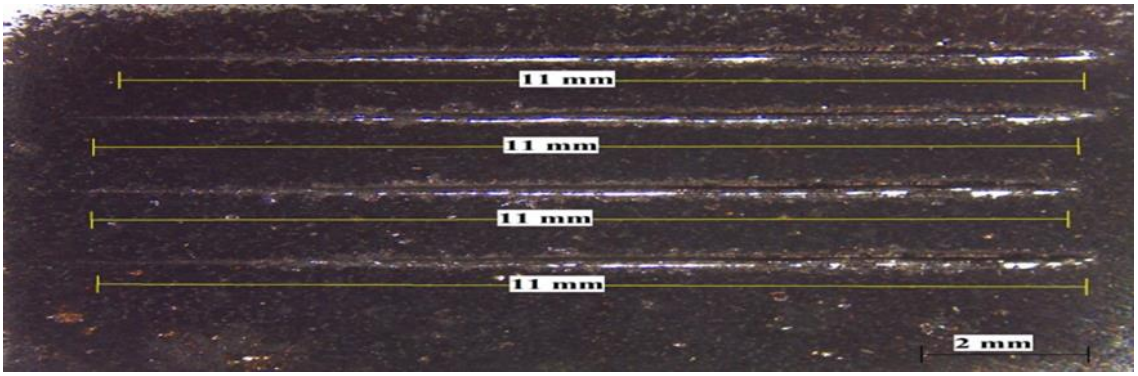


Fig. 4.9. The optical microscopic images of the four scratch marks obtained on the top coated surface.

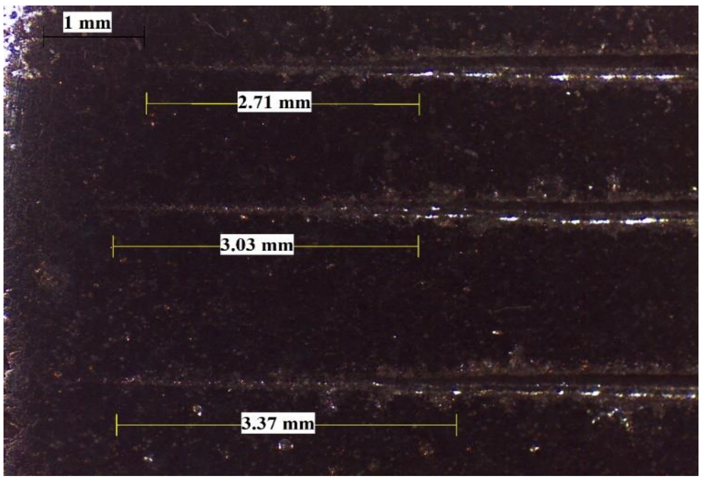


Fig 4.10. Coating Failure Distance

Averaging the failure point distance of each scratch from Figure 4.10, it is observed to be 2.86 ± 0.38 µm. From this the average coating adhesion strength will be calculate and it comes out to be 42.9 ± 5.7 N.

Chapter 6
Conclusions

5.1 Conclusions :

- The coating is consisted of Barium Aluminate, Copper, and some hard nitride phases. By selecting proper process parameters such as (relative quantities of powders, Stand-off distance, multipoint ignition system, etc) it is possible to eliminate the formation of other unwanted phases.

- The coating is found to be strongly adhered to the substrate. It is due to high mechanical anchorage of substrate asperities which has deformed under high shock wave and the interdiffusion of materials between the coating and substrate materials.

- The presence of Barium Aluminate, Al_2O_3 and nitrides has made the coating hard, and the presence of amorphous phases has made it brittle too. Hence, many vertical cracks are noticed to form in the coating.
- Up to 120 µm of depth the substrate is found to be hardened due to the strain hardening effect by high velocity forming of the substrate under the shock wave of the explosion.
- The average roughness of the obtained layer is around 5.7 microns which means the coating is fairly rough and it is possibly due to incomplete melting of the coating powder and presence of the eroded material from the barrel.
- In the cross-sectional image we have observed the deposition of copper is in spherical shape and during the impact most of the original particles are scattered into smaller sizes. Spherical shapes of Cu suggest the partial melting of the Cu powder.
- Due to high shock wave, during the explosive deposition process, interface distortion can be observed using FESEM images of the cross-section, it also leads to the formation of cracks as the coating material is amorphous in nature.
- As it is a super-fast and uncontrolled process, it leads to significant deviation in mechanical and tribological properties over and across the coating.

5.2 Scope of Future Work :

- A standard mechanism can be developed on the setup for making the process continuous.

- There is scope in trying different types of explosives in order to generate more energy in lesser quantities.

- The manufacturing quality of the setup, especially the gun barrel can be improved so that there is no presence of any external eroded material.

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