PRODUCTION OF MONOLAYER GRINDING WHEEL BY NOVEL EXPLOSIVE SPRAY COATING METHOD

M. Tech. Thesis

By

SONALI BRAHMANE



DEPARTMENT OF MECHANICAL ENGINEERING INDIAN INSTITUTE OF TECHNOLOGY INDORE

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by

SONALI BRAHMANE



DEPARTMENT OF MECHANICAL ENGINEERING INDIAN INSTITUTE OF TECHNOLOGY INDORE

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CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled "Production of the monolayer grinding wheel by novel explosive spraying coating method" in the partial fulfillment of the requirements for the award of the degree of MASTER OF TECHNOLOGY and submitted in the Department of Mechanical Engineering, Indian Institute of Technology Indore, is an authentic record of my own work carried out during the time period from August 2020 to May 2022 under the supervision of Dr. Kazi Sabiruddin, Associate Professor, Department of Mechanical Engineering and Indian Institute of Technology (IIT) Indore.

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other institute.

Sonali

Sonali Brahmane

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

Kazi Sabimodin Dr. Kazi Sabiruddin

Sonali Brahmane has successfully given her M.Tech. Oral Examination held on 23rd May 2022.

Zazi Sabimodia

Signature(s) of Supervisor(s) of M.Tech. thesis Date: 23/05/22

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Signature of PSPC Member Dr. Satyajit Chattarjee Date:31/05/2022

sivia

Convener, DPGC Date: 1/05/2022

Apurbeda

Signature of PSPC Member Dr. Apurba Das Date:31/05/2022

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(2002103012)

ABSTRACT

Explosives are generally used for welding and forming in the manufacturing processes. However, it is hard to find work-related coatings where explosive has been used. In the present study, the flash powder is used as an explosive material. This flash powder is coated on a round mild steel surface by using a novel explosive coating setup to fabricate a monolayer grinding wheel. In the first section, the coating is performed on the flat mild steel substrate to achieve optimized process parameters of the explosive coating setup. Process parameters are optimized by the varying stand of distance (SOD), ignition point system, and relative quantity of the flash powder to obtain maximum hardness. The coatings are characterized in terms of thickness, crystalline phase, microstructure, roughness, hardness, and adhesion by using an optical microscope, X-ray diffractometer (XRD), Field emission scanning electron microscope (FESEM), stylus profilometer, Vickers microhardness tester, and scratch tester respectively. The coating obtained with 4 gm flash powder (2 gm each of aluminum (Al) and barium nitrate $Ba(NO_3)_2$), double point ignition system, and SOD of 80 mm indicated the highest hardness around 950 HV_{0.05} and coating thickness of 85 μ m. XRD results of the coating obtained from optimized process parameters indicate the presence of barium (Ba), Al, and barium aluminate (BaAl₂O₄) phases. Non-uniform coating with unmelted Al particles is observed through FESEM and EDS (Electron Dispersive Spectroscopy) analysis. Heat treatment is also carried out on the coated samples obtained from varying aluminium compositions to analyze the hardness. In the second section, a cBN monolayer grinding wheel has been successfully fabricated by spraying flash powder along with cBN grits.

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ACRONYMS

CVD: Chemical Vapor deposition

FESEM: Field-Emission Scanning Electron Microscope

HVOF: High velocity oxy-fuel

PVD: Physical vapor deposition

XRD: X-ray Diffraction

Ra: Average surface roughness

BaAl₂O₄ : Barium Aluminate

cBN: Cubic Boron Nitrate

AISI: American Iron and Steel Institute

SOD: Stand-off distance

Chapter 1

Introduction

1.1 Surface Engineering

Surface engineering is the science that deals with improving the properties of the substrates so that apart from enhancing the life of the component, other properties for a specific requirement can be imparted. Improving the surface properties becomes the primary solution when mostly the failure begins from the surface because of poor mechanical properties, irregularities, and defects.

Surface engineering by application of various types of coatings is described as the process of establishing a surface that has features that differ from the bulk material in terms of improving the engineering product's life and functionality. The desired properties or characteristics of surfaceengineered components include:

Abrasion wear resistance

Improving aesthetic look

Improving mechanical, electrical, and optical properties

Increasing the surface finish [1].

1.2 Thermal Spray Coating

It is a versatile technique where coating material is heated to a molten state and subsequently coated or sprayed over the substrate. It is applied for polymer, high melting ceramics having several advantages. The phrase "thermal spray" refers to a variety of coating methods for applying metallic or non-metallic coatings. Electric arc spray, flame spray, and plasma arc spray are the three most prevalent types of these techniques. The coating material (in powder, wire, or rod form) is melted or semi-molten using these energy sources propelled onto substrates as micrometer-sized particles.. Atomization jets or process gases accelerate and propel the resulting hot particles toward a prepared surface. Following impact, a bond forms with the surface, producing more thickness and the formation of a lamellar structure. The thin "splats" are subjected to extremely high cooling speeds, often exceeding 100°F for metals is 106 K/s. Thermal spray methods have a significant benefit as follows:

- They can generate coatings from a wide range of materials. Almost any substance can be utilized that melts without disintegrating.
- Most of the thermal spray processes can apply coatings to substrates without significant heat input. Thus, materials with very high melting points, such as tungsten, can be applied to finely machined, fully heattreated parts without changing the properties of the part and without excessive thermal distortion of the part.
- A third advantage is the ability to recoat worn or damaged coatings without affecting part qualities or dimensions in most circumstances [2].

The line-of-sight character of these deposition techniques is a drawback. They can only "see" what the torch or rifle can coat. Coating small, deep cavities with a torch or cannon is impossible. When compared to other coating methods such as electroplating, CVD, and PVD, thermal spraying can give thick coatings over a large area at a rapid deposition rate. Metals, alloys, ceramics, polymers, and composites are among the coating materials used in thermal spraying. The most common source of energy for thermal spraying is combustion or electrical arc discharge. Resulting coatings are made by the accumulation of numerous splats. The surface may not heat up significantly, allowing the coating of flammable substances [2].



Figure 1.1 Basics mechanism of thermally sprayed coating [2]

1.3 Explosive material

Explosive material is a kind of material that produces energy by the explosion. They are chemical compounds or a mixture of such chemical compounds which decompose upon ignition. Explosive materials generate severe heat, noise, and highly pressurized gases when they are given some kind of ignition stimuli like heat, impact, friction, shock, or its combination. Classification of the explosive can be done in the following ways [3].



Figure 1.3: Types of Explosives

1.3.1 Classification of Explosives

1.3.1.1 According to the Velocity

(a) Low Explosives:

Low explosives are chemicals whose rate of decomposition is slower than the speed of sound through the medium. The decomposition is propagated via a flame front (deflagration), which goes much more slowly through the explosive material than the shock wave of a high explosive. Low explosives detonate with speeds ranging from a few cm/s to about 0.4 km/s under typical conditions. They have the potential to deflagrate swiftly, producing a detonation-like effect [3]. Examples: flash powder, gun powder, etc.

Flash powder: Flash powder is low explosive. If confined, the flash powder is a combustible compound made up of an oxidizer and a metallic fuel that burns quickly and generates a loud noise, pressure, and heat. It can have barium nitrate, potassium chlorate, and potassium perchlorate-based oxidizing agents, including Al and Mg as the metallic fuel and adding S as

an igniter. It is commonly employed in theatrical, pyrotechnics, and fireworks.

Gunpowder: Gunpowder is a low-explosive. It is a pyrotechnic mixture that includes potassium nitrate (KNO₃), charcoal (C), and sulfur (S), releasing KJ/mol of energy when ignited. The ideal gunpowder consists of a particular composition of potassium nitrate, sulfur, and carbon. The energy released will be affected if these optimal proportions are changed. Stoichiometric composition in gun powder includes KNO₃ (75%), S (5%), and C (10%) [3].

(b) High Explosives:

High explosives (HE) are explosive materials that burst at supersonic speeds, causing the explosive shock wave to pass through the substance. High explosives have a detonation velocity of 3–9 km/s. Upon detonation they can generate energy up to MJ/mol. Examples: TNT, TNP, TNX, RDX, PETN [3].

1.3.1.2 According to Sensitivity(a) Primary explosives:

Primary explosives are those explosives that are very sensitive to any the stimuli like impact, friction, heat, and electricity. Few of the primary explosives are also called contact explosives. It requires only a small amount of energy for ignition. In general, these are more sensitive than PETN. Its ignition is possible with a blow of the hammer. Examples: acetone peroxide, nitroglycerine, halogen azides, ammonium chloride, etc. [3].

(b) Secondary explosives:

A secondary explosive has inferior sensitivity and requires substantially higher energy to ignite than a primary explosive. For detonation, these explosives require larger shocks. As they are less sensitive, they are easier to handle and maintain. Examples: nitroglycerine, PETN, nitrocellulose [3].

(c) Tertiary explosives:

Tertiary explosives, often known as blasting agents, are so shockinsensitive that they require an intermediate secondary explosive booster to ignite effectively. They are so shock-insensitive that they cannot be properly detonated with ordinary amounts of primary explosive, necessitating the use of a secondary explosive intermediate booster. Because of their safety and lower material and production costs, they are frequently used. The largest consumers are large-scale mining and construction companies.

In tertiaries, a fuel and an oxidant are normally included. ANFO (Ammonium Nitrate Fuel Oil Explosive) can be used as a tertiary explosive if the reaction rate is slow enough [3].

1.3.2On the basis of phases

(a) Solid Explosives:

It may be used in the form of powder, stick, or granular form like TNT (Tri-Nitro toluene), black powder, etc.

(b) Liquid Explosive:

These explosives are found in liquid form like nitro glycerin.

(c) Gaseous Explosive:

It is found in the form of a gaseous substance like $O_2+C_2H_2$.

1.4 Explosive in the manufacturing processes

1.4.1Explosive welding

Explosive welding is a solid-state welding technique that can be used to join materials with significantly different physical properties that use high energy rate deformation generated by an explosive detonation [4-5]. The detonation of an explosive accelerates a flyer plate, resulting in metallurgical welds between two or more dissimilar metals [6]. Explosive welding uses a compression force created by explosions to join overlapping metal sheets. The joining elements are oriented 1–15 °C (inclined) towards each other, depending on the material and process, and prepared with an

explosive layer on top. The connecting parts are propelled against each other after ignition. Continuous connecting results from local plastic deformation of the contact area [7-8].

Explosive welding can be done in two different ways: parallel or inclined. Here, it is necessary to spread explosive on the top surface of the material that is being welded. As the explosives are ignited, a welding forms between the surface of two metals due to the impact of an explosion. Once the ignition is stimulated on a particular side, burning starts from that side, eventually reaching the other side. During explosive welding, ignition creates a high-velocity impact on the flyer plate, which leads to its welding to the parent metal. The welding of two comparable or dissimilar materials occurs in this fashion, with tremendous pressure and energy output. Optimization of different selected parameters, such as explosive amount, SOD, impact pressure, and impact velocity, are needed for strongly bonded welds. Figure 1.3 is describing the explosive welding/cladding.



Figure 1.4 Explosive Welding/Cladding [13]

1.4.2 Explosive Forming

Explosive forming is one of the methods for forming a metal plate by utilizing the tremendous shock pressure produced during an explosion. When an explosive detonates, its energy is promptly released, causing a temporary increase in pressure. The water pressure wave moves downward, colliding with the plate that has to be created.

When an under shock wave hits a metal plate, it instantly speeds to a high speed, forcing it to collide with the die. When a metal plate is subjected to such shock loading, the rate of deformation is quite high, making job hardening relatively straightforward [9]. In this manufacturing process, the explosives are utilized to give the desired shape of the base metal. The setup includes the explosives, base metal, die, and blank. The whole setup is placed under the water. As the air is evacuated between die and blank, the ignition of the explosive occurs and the waves are generated which propagates through the water medium and the deformation of the metal occurs in the form of the die. The water minimizes the sound produced during the explosion. Explosive forming can be performed in two ways:

1. Stand-off method

2.Contact Method

1.4.2.1 Stand-off method

The unit is lowered into a water-filled tank with the sheet metal workpiece blank clamped over a die. The die's air supply is drained. The explosive charge is set at a height from the work plate that is predetermined. A very high-intensity pressure pulse is produced when the explosive is detonated. In addition, a gas bubble is formed, which expands spherically before collapsing. The metal is bent into the die when the pressure pulse hits the workpiece [13]. Figure 1.5 is showing the schematic presentation of a standoff explosive forming.



Figure 1.5 Schematic diagram of a stand-off explosive forming[23]

1.4.2.2 Contact method

In this method, the explosive charge in the form of a cartridge is kept in constant contact with the workpiece before the detonation is started. The explosion generates extremely high pressures (up to 30,000 MPa) on the work piece's surface, producing metal deformation and probable fracture. Bulging tubes are frequently treated using this method [13]. Figure 1.6 describes the contact type of explosive forming.





1.5 Monolayer Grinding Wheel

Monolayer grinding wheels are comprised of a single layer of super abrasive grains bonded to a metallic wheel by either an electroplated nickel layer or a brazed filler layer. The metal wheel hub, bond layer, and super abrasive grains make up the electroplated and brazed wheels. Compared to SiC, Al₂O₃, and regular super hard abrasive wheels, monolayer super hard abrasive wheels can deliver acceptable grinding results due to their high grain protrusions, high wear resistance, and high heat conductivity of abrasive grains. Monolayer brazed super hard abrasive wheels, for instance, can provide greater grain protrusion and a stronger holding force to the abrasive grains than electroplated wheels, resulting in increased wheel life [11]. Monolayer cBN wheels have significant advantages such as:

- Over a significant duration of grinding, the shape is maintained well
- Feasibly higher material removal rate by grinding because of large grain protrusion and chip storage space.
- After the grains have worn away, the wheel hub can be reapplied (such as stripping of the abrasive layer) [22].

1.5.1 Brazing

The brazing techniques are used for the fabrication of the monolayer grinding wheel. In these techniques, the grits are brazed with the help of brazing alloy and heated up to the brazing temperature of around 1000 °C. The brazing of the grits depends on the following three factors

- i) Active braze alloy
- ii) Heating method for brazing
- iii) Heating parameters (brazing temperature and dwell time).

The braze alloy material is selected on the basis of wettability, ductility, adequate wear-resistance, good strength, and containing an active element that can make the chemical bond between the substrate and the grits. Like brazing alloys for cBN are Ag–Cu–Ti, Cu-Sn–Ti, and Cu–Sn–Ni-Ti contain

Ti as the active element. Brazing alloys for diamonds are Cr-Ni-P, and Cr-Ni-B-Si contains Cr as the active element.

Different heating methods are used for heating purposes. Like vacuum resistance furnace heating (5–20 min), which includes electron beam-activated heating and can be employed for the cBN brazing. Heating methods for the diamond grits include induction furnace, induction, electric resistance furnace, etc.

Characteristics of a brazed monolayer wheel are mentioned below:

- Longer tool life due to the strong holding of the grits
- They outperform the electroplated grinding wheels
- Has increased bonding strength between grains
- Has greater protrusion of grits
- Flexible grain placement in any desired pattern
- Large inter-grain chip-accommodation space leads to less prone wheel loading [18].

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1.5.2 Electroplating Technique

In the industry, electroplating is utilized to fabricate monolayer grinding wheels. The anode is made up of bonding material, and the cathode is made up of the wheel hub. The hub of the wheel is fully covered in cBN granules and inserted into the electrolytic bath. The electrolytic bath is a fluid solution of metallic salts that are to be deposited, such as Ag, Co, Cu, and Ni. It is set to run at room temperature with an applied external power supply. Figure. 1.7 represents the electroplating deposition of the monolayer grinding wheel in the two steps, which are takedown and electrodeposition.

The substrate is covered with the grits that will be plated. The entire arrangement, including the electrodes, is submerged in the electrolytic bath. The anode material begins to deposit on the substrate as soon as the process commences. When the grits are attached to the substrate loosely, electrical supply is interrupted. The remaining grits that do not adhere are removed mechanically, and the power supply is continued to allow further deposition of the coating until the desired thickness is achieved. For uniform grit height, reverse electroplating can be performed.



Figure 1.7. Electroplating process for fabricating monolayer grinding wheel.

Chapter 2

Literature Review

This chapter discusses prior work on thermal spray-based coatings, explosive application in manufacturing processes, research gaps found, research objectives set to address the gaps, and current research techniques.

2.1 Explosives in Manufacturing

Daisuke Inao et al. successfully welded thin Al plates onto Mg alloys and discovered characteristic wavy interfaces without intermediate layers using a pressure-transmitting medium of gelatin. Gelatin's role as a pressure-transmitting substance was shown to play a significant role in improving explosive welding [14].

D. Meuken and E. P. Carton showed the capability of explosive foil cladding as an alternate coating process with one or two foils on either side in one processing step. This technique can be used to clad any material combination, and ideal for increasing the parent material properties [15]

Zaheer-ud-din et al. studied the thermal, kinetic, and ignition behavior of three pyrotechnic mixtures consisting of Al + Ba(NO3)2, Mg + NH4ClO4, and Mg + KMnO4. The findings revealed that all of these compositions are thermally stable. Mg & NH4ClO4 is the most reactive, followed by Al & Ba(NO3)2 [16].

K. Raghukandan created explosive cladding of copper plate with low carbon steel plate by adjusting the stand-off distance, loading ratio, and flyer thickness, and the effect of varying these parameters was investigated using microstructure analysis and shear strength measurements. The thickness of the flyer, the loading ratio, and the angle of inclination all play a role in the morphology and mechanical behavior of the weld. At the same time, the stand of distance has a minor impact [17].

2.2 Monolayer Grinding Wheel

Chattopadhyay et al. found that brazed bonded equivalents show strong grit-bond adhesion when compared to galvanically bonded single layer cBN tools. By putting the grains in a consistent pattern, the performance of a brazed cBN wheel might be greatly enhanced [19].

Bhaskar Pal et al. showed that increased grit density reduces chip load per grit, increasing the overall service life of the brazed wheel in a tough environment by lowering attrition wear, fracturing, and grit pull-out [18].

Malkin et al. investigated the wear processes of electroplated CBN wheels in high-speed silicon carbide grinding. They found that the major process of wheel wears to be attrition wear, followed by minor grain fracture and grain withdrawal from the bond [24].

Pal et al. showed that when compared to the galvanically attached wheel, the brazed-type wheel outperformed in bearing steel grinding. Over a wide range of grinding parameters, the brazed-type wheel could grind the bearing steel more effectively and economically with lower grinding forces and without wheel loading. Higher bond strength, bigger grit protrusion, and more control over grit spacing uniformity on the wheel surface are the major characteristics of the brazed-type wheel that contribute to its superior performance. As the MRR is increased, the relative benefits of using a brazed-type wheel over a galvanically bonded wheel increased [19].

2.3 Identified Research Gap

Based on the review of the past work done research gap is identified. To the best of our knowledge, no literature has been found on the usage of explosives for coating applications.

2.4 Objectives and Research Methodology

- To deposit Al-BaAl₂O₄ composite coating on the flat steel substrate by varying the feedstock compositions.
- Characterization of the deposited coatings in order to obtain optimized Al-BaAl₂O₄ composite coating with superior mechanical properties
- To find the effect of heat treatment on the properties of Al-BaAl₂O₄ coating
- An attempt to produce a monolayer grinding wheel by using optimized composite coating along with cBN grits

2.5 Research Methodology

- Review of past work on the usage of explosives in manufacturing processes, monolayer grinding wheel, and coating process
- Identifying the research gap and research objectives
- Coating deposition on the flat steel substrate with the help of the novel coating setup
- Mechanical and physical characterization of the deposited coating
- The coating on the round steel substrate with the optimized parameter

Chapter 3

Experimental Procedure

3.1 Material Selection

3.1.1 Steel substrate

AISI 1020 steel is selected as the substrate material in both flat rectangular $(15 \times 15 \times 10 \text{ mm}^3)$ and the round (ϕ =15 mm, h=33 mm) shape. Table 3.1 shows the elemental composition of AISI 1020 steel.

Table 3.1 Elemental composition of AISI 1020 steel [3]

Elements	Fe	Mn	С	S	Р
Contents	99.08-99.53	0.30-0.60	0.170-0.23	< 0.05	< 0.04
(%)					

3.1.2 Explosive material

Explosive materials are quickly decomposed to produce a large amount of energy, usually followed by the generation of pressure, heat, and sound. A varying constituent of explosive powder (Al+Ba(NO₃)₂)is employed in the fabrication of the coating. Table 3.2 represents the composition of the explosive powders.

Table 3.2 explosive powder composition [3]

Elements	Content (weight %)
Al	33.3
Ba(NO ₃) ₂	66.6

Flash powder

Flash powder, a low-explosive pyrotechnic compound, is made up of an oxidant and a metal fuel. When ignited, it burns rapidly and generates energy, heat, pressure, and loud noise. It is commonly employed in theatrical, pyro, and fireworks. The XRD diffraction pattern, and FESEM

micrograph with EDS plot of Al powder are shown in Figures 3.1 and 3.2, respectively. The XRD pattern and FESEM micrograph with EDS plot of $Ba(NO_3)_2$ powder are represented in Figures 3.3 and 3.4, respectively. The following is a simplified equation for flash powder combustion:

$$10Al + 3Ba(NO_3)_2 \rightarrow 5Al_2O_3 + 3BaO + 3N_2 + Heat$$
 at 601°C [25]



Figure 3.1 XRD pattern of Al powder



Figure 3.2 a) SEM micrograph ,b) EDS plot of Ba(NO)3 powder



Figure 3.3 XRD plot of Ba(NO3)2 powder



Figure 3.4 a) SEM micrograph, b) EDS plot of Ba(NO₃)₂ powder

3.1.3 Abrasives grains

cBN grits are used for the monolayer grinding wheel fabrication. The grit sizes range from 150 to 180 μ m. These grits are golden-colour, blocky crystal shape having the high strength and high thermal stability which are used for the electroplating tool and the metal and the vitrified bond system.

3.2 Experimental Setup

The setup used for obtaining the coating on the substrate is shown in Figure 3.5. This indigenously developed experimental setup has the following components: substrate holder, gun barrel, switch, spring, ignition coil, and base. Heat-treated grit blasted rectangular AISI 1020 steel substrate is placed on the substrate holder. The preheated mixture component of flash powder is poured into the gun barrel from the pouring holes. When ignition of the flash powder inside the confined barrel takes place, exploded material comes out of the gun barrel at high velocity and makes an impact against the substrate. As a result, the coating is obtained. The distance between the final layer of flash powder and substrate is maintained at 80 mm as SOD. Deposition of coating is achieved with a two-point ignition system and utilizing a mixed form of constituent of the flash powders.



Figure 3.4 Experimental setup [2]

3.3 Substrate Preparation

The surface preparation technique involves surface cleaning, roughening, and sometimes preheating.

3.3.1 Surface Cleaning:

It includes cleaning and making surfaces free from contaminants such as dirt, grease, and oil. A good degree of cleanliness guarantees the good adhesion of the coating on the substrate. Cleaning of the substrate is accomplished by chemical or manual means.

Substrate cleaning has the following significance:

- 1. To clean the substrate surface by removing oil and grease.
- 2. Good coating and substrate adhesion.

3.3.1.1 Ultrasonic Cleaning

Ultrasonic cleaning utilizes high-frequency sound waves to remove loose particles from a surface while immersed in inorganic solvents such as isopropyl alcohol or acetone. These inaudible sound waves are produced in the fluid medium and eliminate impurities from all surfaces when the fluid makes contact with the surface. After the grit blasting, ultrasonic cleaning in an ethyl alcohol bath is performed to scour any grits or attached impurities from the hidden parts.

3.3.2 Grit Blasting

Grit blasting is employed for providing roughness on the substrate surface. Grit blasting provides the uniform desired 5 μ m to 7 μ m roughness to the surface for better adhesion of the coating.

Grit blasting conditions are tabulated below.

Substrate	AISI 1020
Grit type	Alumina (24 mesh)
Blasting pressure (kgf/cm ²)	5
Blasting Time (minutes)	1
Stand-off distance (mm)	100

Table 3.3 Grit blasting conditions

3.3.3 Preheating the Substrate and Powders:

The constituents of flash powder are preheated separately at 100 °C for 10 minutes. Substrate preheating is performed in the muffle furnace for 15 minutes at 350 °C to remove residual thermal stresses and assure a good mechanical anchorage.

Preheating of the substrate has the following major objectives

- 1. Removing the residual thermal stresses.
- 2. To drive off the moisture

 Table 3.4
 Substrate preheating parameters

Time (minutes)	15
Preheating Temp. (°C)	350

Table 3.5 Preheating parameters of constituents of flash Powder

Time (minutes)	10
Preheating Temp. (°C)	100

3.4 Selection of Process Parameters

Process parameters directly influence the mechanical and tribological properties of the coated surface. Experiments are performed by utilizing optimized parameters like stand-off distance, distance between powders, type of ignition, number of ignition points, surface roughness of the substrate, and relative quantity of precursor powders. Such process parameters are shown in a sectional view of the gun-barrel in Figure 3.6

Substrate



Figure 3.5 Process parameter for spray coating [2]

Table 3.6 Process p	arameters used in	our experimental	work
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Stand-off distance	80 mm
Ignition Force	Multi point ignition system
Preheat temperature (°C) for powders	100 °C
Substrate preheating Temp (°C)	350 °C

3.5 Performing Coating Deposition on Flat Substrate

The coating is deposited over the AISI 1020 low carbon steel substrate by using the explosive coating setup. The quantity of the Al is taken as 0.5, 1, 1.5, 2, and 2.5 gm in the precursor powder keeping constant quantity of $Ba(NO_3)_2at 2$ gm. Variation of composition of precursor powders in the explosive coating are shown below in Table 3.7.

Sl. No.	Samples code	Al/pyro quantity(gm)	Barium nitrate quantity(gm)
1.	Al(0.5) Ba(NO ₃) ₂ (2)	0.5	2
2.	$Al(1) Ba(NO_{3})_{2} (2)$	1	2
3.	Al(1.5) Ba(NO ₃) ₂ (2)	1.5	2
4.	$Al(2) Ba(NO_{3})_{2} (2)$	2	2
5.	Al(2.5) Ba(NO ₃) ₂ (2)	2.5	2

Table 3.7 Varying composition of the flash powder and sample codes

3.6 Heat-treatment of the Coated Samples

Heat-treatment is carried out to investigate its hardening effect on the coating. The heat treatment is performed in the muffle furnace at 900 °C for 2 hours at heating rate of 20 °C/min followed by furnace cooling. Parameters of heat treatment of as-deposited are listed on Table 3.8.

Table 3.8 Parameters used in post-heat treatment Process

Maximum Temperature	900°C
Heating rate	20 °C/min
Holding Time	2 hours

3.7 Deposition of the Coating on Wheel Hub.

Following are the steps for applying the coating to the grinding wheel hub:

The grits are attached to the wheel hub using adhesive, and the coating is performed. Grits are placed on the wheel hub in the following ways.

- The random orientation of the grits.
- The patterned attachment of the grits.
- Placed the grits along with the explosive material.

The coating is deposited three times on the one wheel-hub by dividing the wheel hub surface into three parts with an area of exposure of 120 °.

The coating on the wheel hub is deposited by taking 2 gm of each precursor powders Al and $Ba(NO_3)_2$ employing the optimized parameters such as 80 mm SOD, a two-point ignition system, by placing the Ni-Al in between flash powder. Then the same process is repeated for the remaining two-third peripheral part of the wheel hub.

3.8 Characterization of deposited coating:

3.8.1 X-ray Diffraction

The coating phases are analyzed by using XRD patterns obtained with an X-ray diffractometer (Rigaku Smart Lab 3 kW having Cu-K_{α} radiation of

 λ =0.15406 nm with Ni-filter). The sample diffraction range is selected from 10° to 80° with a step size of 0.01°. The phases of the compositions are calculated with the help of "X'Pert HighScore" software and plotted with the help of Origin Pro software.

3.8.2 Microstructure Study

The coated samples are sectioned by using a high-speed cutter and followed by mounting with the help of a hot mounting press. The cross-section samples are polished on the polishing machine using the SiC papers of the mesh size ranging from 220 to 2500, followed by diamond polishing through diamond paste consisting of grits ranging from 3 to 0.25 μ m. Top and cross-section of coatings microstructure, morphological aspects, and elementary quantitative analyses are evaluated in Field Emission Scanning Electron Microscope (FESEM) coupled with Energy Dispersed Spectroscopy (EDS), Zeiss Supra-55. The high-resolution magnified images of a cross-section of the coating and the top surface are captured.

3.8.3 Thickness analysis

The thickness of the coating is measured from the cross-sectioned coatings using an optical microscope. After acquiring the images, the coating thickness is measured with the help of image analysis software "Leica LAS EZ" by evaluating the distance between two points. For thickness measurement, average values of the different readings are taken

3.8.4 Roughness analysis

With the help of a stylus profilometer, surface roughness and irregularities of the coatings are measured. The stylus has been employed to move on the coating surface and record the profile roughness parameters (Ra, Rq, Rp, Rv, and Rz). The tip of the stylus makes contact with the surface asperities and moves along the traverse axis. The stylus sensor measures the different surface roughness parameters. The transducer connected opposite to the stylus is responsible for converting vertical movement into an electrical signal. Surface roughness is measured with the following parameters; evaluation length 4 mm using a Gaussian filter.

3.8.5 Microhardness Analysis

The hardness, which is the characteristic of the material, is defined as the resistance against deformation. It is measured by making a permanent indentation on the material and measuring its dimensions. In this work, highly polished mounted samples are used so that the impressions can be identified clearly to measure their dimensions. A pyramid-shaped diamond having a square base is used for testing, keeping the dwell time 12 sec and the normal load of 50 gf. Indents are measured on the as-deposited as well as heat treated coating surface, near to interface on the substrate and the coatings. The values of diagonals d1 and d2 are measured, and the microhardness values are calculated using the standard below-mentioned formula.

Vickers Hardness = $0.1891F/d^2$

3.8.6 Porosity Analysis

Voids in the microstructure of the coating, also called porosity, influences the mechanical properties such as micro-hardness, and strength. With the help of "ImageJ" software, the percentage of the porosity present in the coating is calculated. The area of the pores present in a particular location in the coating is measured, and then it is divided by the whole area. A number of SEM images have been utilized to calculate the average porosity of each composite coating.

3.8.7 Scratch test Analysis

A Scratch tester is used for performing the scratch test on the samples. The test samples are polished ($R_a <= 1 \ \mu m$) [21]. A progressive load scratch test from a normal load of 1 N to 20 N is executed for a distance of 10 mm with 0.01 mm/sec scratch speed. Data is analyzed with the help of the Origin Pro

software. The graphs are plotted between i) tractional force and the sliding length and ii) COF and sliding length.

3.8.8 Linear Reciprocating Wear Study

The dry sliding wear test is done by a "Ducom CM-9104" linear reciprocating type tribometer against a WC (ball $\phi=6$ mm) counter body. The wear is performed on the coating by applying a 12.5 N normal load at 20 Hz frequency and a 3 mm stroke for 15 minutes. The depth and width of the wear tracks are measured with the help of the stylus profilometer to calculate the volume loss.

The formula for calculating the specific wear rate is as follows:

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

Here, the specific wear rate of the samples is denoted by ω_d having unit mm³/(N·m), and V in mm³ is the volume of *the* wear track, sliding distance is the d in m, L denotes the normal load[20].

The following formula is considered for calculating the wear loss volume.

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

Here *l* is sliding distance in meter, and r and b are the ball radius in mm and track width in mm, respectively. The wear resistance is calculated by performing a wear test on individual samples [20].

Chapter 4

Results and discussion

4.1 XRD Analysis

The XRD patterns of coated samples with varying aluminium composition are shown in Figure 4.1. Results reveal the presence of the different phases such as Ba, Al, and BaAl₂O₄ with various intensities depending upon the composition of the coating. In the case of coating containing 1gm of Al, peaks of Ba with higher intensity can be observed. It indicates an incomplete reaction of the precursor powders. With an increasing quantity of the Al in the precursor powder, the new peaks of the Al and BaAl₂O₄ are observed. The coating containing 1.5 gm of Al has the peaks of either Al or BaAl₂O₄. When the Al amount is increased to 2 gm, the XRD pattern shows the highest intensity of BaAl₂O₄, which signifies the complete reaction of the precursor powders. As the content of Al increases to 2.5 gm, new emerging peaks of the Al can be observed. These peaks indicate the presence of excess Al in the coating.



Figure 4.1 XRD pattern of the coated sample

4.2 Microstructural and EDS Analysis

Figure 4.2-4.5 represents FESEM micrographs of the cross-sections along with their respective plots of X-ray counts against energy (in keV) as obtained from the EDS analyses for coatings containing aluminium content 1, 1.5, 2, 2.5 gm, respectively. The results of EDS analyses for all four coatings confirm the presence of Al, Ba, and O. Point EDS of globules present in a cross-section of all coatings confirm unmelted Al particles. Features like cracks, voids , compressed zone, wavi interface can be observed from the cross sectional image of the SEM.



Figure 4.2 (a) FESEM micrograph and (b) EDS profile of cross-section of explosive coating containing aluminum 1 gm



Figure 4.3 (a) FESEM micrograph and (b) EDS profile of cross section of explosive coating containing aluminium 1.5 gm



Figure 4.5 (a) FESEM micrograph and (b) EDS profile of cross-section of explosive coating containing aluminum 2 gm



Figure 4.4 (a) FESEM micrograph and (b) EDS profile of cross-section of explosive coating containing aluminum 2.5 gm

4.3 Thickness Analysis

The thickness of the coatings obtained by varying Al composition from 1 to 2.5 gm is shown in Figure 4.6. Samples are prepared by the standard metallographic technique to measure coating thickness by observing its cross-section under an optical microscope. With an increasing amount of Al powder in the feedstock, coating thickness is found to be increased. This is an expected result because the higher volume of Al powder in the feedstock is added, which leads to increased coatings thickness.



Figure 4.6 Thickness variation with increasing Al content in the feedstock

4.4 Roughness Analysis

Surface roughness and irregularities of the grid blasted substrates and coatings are analyzed by employing a contact-type stylus profilometer. For comparing different specimens, the average roughness of the surface (Ra) is determined. The roughness values of coated samples with varying aluminium content are shown in Figures 4.7 b), 4.8 b), 4.9 b),4.10 b

The roughness profiles of grit blasted and coated samples obtained by variation of aluminium content from 1 to 2.5 gm are depicted in Figures 4.7-4.10. The grit-blasted substrates have a lower roughness than the as deposited coatings containing 1 gm and 2.5 gm of Al powder, as shown in Figures 4.7 a), 4.8 a), 4.9 a),4.10 a). Increased roughness value of coating is due to the incomplete reactions or presence of un-melted Al particles.

Minimum surface roughness is observed for coating containing 2 gm of Al powder because of the occurrence of complete reaction. The roughness trend of the coatings obtained is represented by Figure 4.11.



Figure 4.7 Surface profile of a)grit blasted substrate substrat(Ra= 5 μ m) b) Coating with 1 gm of Al (Ra= 4.39 μ m)



Figure 4.8 Surface profile of a)grit blasted substrate substrat(Ra= 5 μ m) b) Coating with 1.5 gm of Al (Ra= 4.39 μ m)



Figure 4.9 Surface profile of a)grit blasted substrate substrat(Ra= 5.85 μ m) b) Coating with 2 gm of Al (Ra= 4.7 μ m)



Figure 4.10 Surface profile of a)grit blasted substrate substrat(Ra= 5.54 μ m) b) Coating with 2.5 gm of Al (Ra= 3.59μ m)



Figure 4.11 Coating roughness variation with increasing Al content in the feedstock

4.5 Vickers Microhardness Analysis

Vickers microhardness tests are performed on the diamond polished, mounted samples. The indents are made on the cross-section of coating against a load of 50 gm and 12 seconds of dwell time. The hardness value of the bare substrate, coatings, and heat-treated coated samples are compared.

4.5.1 Vickers Microhardness of As-deposited Coating

The hardness of the coating is found to be higher than the substrate hardness. Microhardness obtained with the coatings containing 1, 1.5, 2, 2.5 gm of Al content is 644, 601, 977, 566 HV_{0.05}, respectively. Among different compositions of the explosive coatings, the coating containing 2 gm of Al powder has the highest measured hardness. This behavior is because of the complete combustion of precursor powders resulting formation of a high-intensity BaAl₂O₄ phase.



Figure 4.12 Microhardness of as-deposited coatings obtained by varying aluminium content

4.5.2 Vickers Microhardness of the Heat-treated Coating

The coatings are heat-treated inside a muffle furnace at 900 °C for 2 hours. An increase in the hardness of the coatings is observed irrespective of their composition after the heat treatment, as represented in Figure 4.13. The Al containg 1, 1.5,2, 2.5 gm shows the hardness 674,758,1313,728 HV_{0.05} respectively. The change of metastable phases of aluminium oxides and barium aluminates to their respective stable phases can be responsible for this increased hardness value. Another possible reason for the improvement in hardness can be a reduction of porosity caused by remelting of the coating material during the heat-treatment process.



Aluminium content in the feedstock (gm)

Figure 4.13 Microhardness of post heat-treatment of coatings obtained by varying aluminium content

4.5.3 Vickers Microhardness Analysis of the Substrate at Different Depth of Coated samples

Vickers hardness is calculated at various distances from the coating substrate interface on the substrate for all compositions. Variation of the substrate's hardness at different depth away from the interface is calculated starting from 20 μ m from coating's substrate interface. Indents are made at a distance of two times the value of diagonal of the indents to eliminate the influence of work hardening due to indentation. The effect of the strain hardening during the explosive coating is studied. It is found that the microhardness value at a depth of 20 μ m is influenced by the composition of the coating, which controls the condition of complete reaction between the Al and the Ba(NO₂)₃. Comparing the hardness at 20 μ m from the interface for different composition of the coatings, coatings with 2 gm Al precursor powder has the highest hardness value of 273 HV_{0.05}. This is due to the highest strain hardening of the substrates because of the execution of

the complete reaction. The depth of strain hardening is also influenced by the composition of the coatings, as shown in Figures 4.14 and 4.15.



Figure 4.14 Variation of microhardness of the substrate from 20 μ m distance from the interface of the coating obtained by varying Al content.



Figure 4.15 Plot of variation of depth of hardened substrate and maximum hardness at 20 µm depth with varying Al content

4.6 Porosity Analysis

The porosity affects the mechanical properties of materials such as hardness, strength, etc. In explosive coating, porosity is created by the escape route of the gases and unmelted particles produced during the explosion. The porosity of the coating is found to be least when the quantity of the Al is 2 gm. However, coating porosity is observed to be maximum in the case of 1.5 gm of Al. The Figure. 4.16 shows the porosity percentage of coatings with different compositions of aluminium.



Figure 4.16 Porosity variation of coatings with increasing Al content in the feedstock

4.7 Coating Adhesion Analysis

The scratch test is performed at a progressive normal load from 0-20 N to calculate the scratch adhesion strength of the coatings. Under an optical microscope, scratch tracks are observed to identify and measure the critical length for calculating critical load. Among different compositions, delamination is observed for the coating containing 1 gm of Al. FESEM micrograph and EDS of the scratch tracks are shown in the Figure 4.17 (a) for coating containing 1 gm of aluminium. EDS analysis indicates the critical length of the coating after which the substrate has been exposed. Point EDS is carried out after critical length confirms the presence of only Fe composition, which further ensures the detachment. The graphs of tractional force and normal load against the scratch length of coating containing 1 gm Al are depicted in Figure 4.18, which further help in

determining the critical length and critical load to obtain coating adhesion. Detachment of the coating has occurred at a distance of 8.77 mm from the start of the scratch. So, the critical length (Lc) of coating is 8.77 mm. The normal load at Lc, also called the critical load of coating, is 18.77 N. So, the coating adheres to the substrate with a strength of 18.77 N. None of the other Al-based coatings fails under this tested load condition.



Figure 4.17 (a) FESEM micrograph, (b) Point EDS at delamination area of the scratch track for coating containing aluminium 1 gm.



Figure 4.18 Variation of normal load and tractional force with respect to scratch length for coating obtained from 1 gm of aluminium

4.8 Linear Reciprocating Wear Analysis

A Linear reciprocating wear test is performed against a WC ball (ϕ =6 mm) with a normal load of 12.75 N for an amplitude of 3 mm at 20 Hz frequency for 15 minutes to identify different tribological aspects of the fabricated explosive coatings. The frictional behavior of the deposited coating is analyzed from the plot of friction coefficient against sliding distance. Variation of COF w.r.t sliding distance for coatings containing various content of aluminium is shown in Figure 4.19.



Figure 4.19 Friction coefficient of the for as deposited explosive coatings against sliding distance representing the dependence of frictional properties on Al content

At the initial stage of sliding, unusual behavior of the COF can be observed. This is primarily due to the running-in period where contact between the asperity junctions is not fully developed, and it causes unpredictable frictional behavior. After a few repeated cycles of sliding, the friction coefficient becomes relatively steady, which is related to the actual frictional behavior of materials under investigation. Coatings containing 2.5 gm of Al powder possess higher frictional resistance due to their larger friction coefficient of approximately 0.8 compared to other coatings. This nature of the coatings is also reflected in Figure 4.19, where the lowest wear resistance corresponding to the highest wear rate is observed. The wear rate of coatings obtained from the variation of aluminiun content is shown in Figure 4.20. Coatings containing 1, 1.5, and 2 gm of Al powder have an average friction coefficient of roughly 0.5 to 0.6. They also have comparable wear resistance.



Figure 4.20 Influence of Al content on the wear rate after sliding wear on the top surface of explosive coating.

4.9 Monolayer Grinding Wheel Fabrication

Wheel hub made of AISI 1020 steel is employed as a substrate to manufacture a monolayer grinding wheel. The geometry of the wheel hub is designed considering the ease of its utilization. Figures 4.21 and 4.22 represents the 2D drawing and lathe fabricated wheel hub, respectively. The explosive coating on the wheel hub is performed in three subsequent steps, and each one covers exposure of 120°.



Figure 4.21 Schematic engineering drawing of the wheel hub



Figure 4.22 Grinding wheel hub fabricated from different lathe operation

Several attempts have been made during experiments to coat flash powder on the cBN grit attached wheel hub by using the explosive coating setup. As the substrates are required to go through a preheating process at 350 °C before the explosive coating, applied adhesives are required to be capable of sustaining that temperature.

Deposition of cBN grits in the wheel hub through explosive coating has been achieved by the following steps:

Grits are randomly preplaced on the surface of the grinding wheel hub with adequate adhesive. Next, the explosive coating of Al, $Ba(NO_3)_2$ (2gm each) is performed on 1/3 (120°) of its exposed surface. In the subsequent two steps, the whole surface area of the cylindrical wheel hub is covered, and at the same time, some portion of the previous coating has been loosely removed. Overlapping of layers of coating at the boundary of two successive coatings is observed. This eventually results in disruption of the monolayer configuration of the cBN grits in the wheel hub.

The next attempt is made by organizing the grits at particular locations, maintaining a constant distance between the attached grits, which is shown in Figure 4.23. After completion of the explosive coating, a small number of grits are detaching from the wheel hub. This phenomenon may be caused by shock wave of the explosion and higher velocity explosive material.

At the initial stage of experiments, problems were faced with the adhesion of the cBN grits on the surface of the wheel hub. Afterward, the deposition of the coating is achieved successfully when explosive material is placed along with the cBN grits inside the gun barrel. By spraying cBN grits along the explosive materials, a monolayer grinding wheel is fabricated. This technique can be further explored for more complex manufacturing processes. The image of the successful deposition is shown in Figure 4.24.



Figure 4.23 cBN (150-180um) Grit attached wheel hub



Figure 4.24 Coating Deposition successfully

4.10 Conclusions

- With an increase in Al quantity, different types of composite coatings are formed because of the incomplete reaction of Al & Ba(NO₃)₂.
- All four coatings show the features like crack, pore, melted zone, unmelted particle, etc., as shown in FESEM images. The EDS results indicate that unmelted particles belong to aluminium.
- Coating thickness is increased from 65 to 82 μm with an increase in the quantity of aluminium from 1 to 2.5 gm.
- When the Al content is increased 1 to 2 gm, coating roughness is found to be decreased. Minimum roughness is obtained for 2 gm of Al because, in this case, less number of unmelted particles are observed, which leads to good particles spreading. With a further increment of Al content to 2.5, roughness is increased.
- The top surface and cross-section of all coatings reveal unmelted Al particles, which in turn increase the roughness and porosity of the coating. However, the minimum roughness and porosity are obtained at a 2 gm Al quantity.
- Maximum coating hardness is obtained at 2 gm of Al because, at this condition, most of the peaks belong to the hard phase of BaAl₂O₄, as clear

from the XRD pattern. After heat treatment, the hardness of the coatings can be improved significantly for all varying Al content samples.

- For all the coatings, the substrate hardness is increased below the interface due to the strain hardening effect caused by the shock wave of the explosion, which also helps to improve coating adhesion strength.
- With increasing Al content, the depth of hardened substrate is showing a reducing trend. However, maximum hardness near the substrate depends upon the complete reaction of Ba(NO₃)₂, which is maximum in the case of 2 grams of Al due to the complete reaction of such composition.
- The coating obtained with a low aluminium content of 1 gm shows maximum wear resistance because of a low COF value of around 0.5.
- A successful monolayer grinding wheel is produced by spraying a pyrotechnic mixture along with cBN grains in the presence of Ni-Al.

4.11 Future scope

- The performance of fabricated monolayer grinding wheels can be studied in terms of material removal rate, produced chip morphology, grit breakage, grit pullout, etc.
- A proper mechanism can be implemented in the setup to make the process continuous, which can help to achieve a higher coating thickness along with uniformity in the deposition of coating material

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