Effects of Process Parameters on the Properties of $BaAl_2O_4$ based Coating Deposited on Steels by Novel Explosive Spray Technique and Prediction of Coating Hardness by using Machine Learning Technique

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Effects of Process Parameters on the Properties of $BaAl_2O_4$ based Coating Deposited on Steels by Novel Explosive Spray Technique and Prediction of Coating Hardness by using Machine Learning Technique

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INDIAN INSTITUTE OF TECHNOLOGY INDORE

CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled **Effects of Process Parameters on the Properties of $BaAl_2O_4$ based Coating Deposited on Steels by Novel Explosive Spray Technique and Prediction of Coating Hardness by using Machine Learning Technique** 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 2021 to June 2023 under the supervision of Dr. Kazi Sabiruddin, Associate Professor, Department of Mechanical Engineering, and Dr. Chandresh Kumar Maurya Associate Professor, Department of computer science Engineering, 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.

Signature of the student with date
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This is to certify that the above statement made by the candidate is correct to the best of our knowledge.

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With Regards

Shubham Birle

ABSTRACT

In the present work, the mixture of barium nitrate ($Ba(NO_3)_2$) and aluminum (Al) powders are burned to deposit barium aluminate ($BaAl_2O_4$) composite coatings on mild steel substrate by a novel explosive spray coating setup. Various aluminium amounts (1, 1.5, 2, and 2.5 gm) and different stand-off distances (60, 80, and 100 mm) are employed during the deposition process. The X-ray diffractometer (XRD) is used to analyze the phases present in the different coatings. The significant peaks of barium aluminate phase along with some secondary phases, are observed with varying Al contents and changing stand-off distances (SOD). The hardness of the coating and substrate is estimated by using Vickers microhardness tester. The maximum hardness value (1313$HV_{0.05}$) is noticed for the coating fabricated by employing 2 gm of Al at 80 mm SOD. The hardness of the substrate at 20 μm, below the interface of coating-substrate is found to be maximum for coating obtained through 1 gm of Al at 100 mm SOD. The depth of the hardened zone of coating is observed to be decreased with an increase in Al amount from 1 to 2.5 gm at all three different SODs. A stylus profilometer is engaged to measure the average surface roughness of the coatings and grit-blasted substrate. The coating fabricated by using 2 gm of Al at 80 mm SOD shows the minimum roughness value of 3.5 μm. The thickness of the coating is measured through an inverted optical microscope. The maximum coating thickness is achieved by employing 2.5 gm of Al at 60 mm of SOD among all different combinations of Al content and SOD. Using a machine learning model to predict the hardness of the coating. Random Forest model shows an accuracy of 99.22% prediction of hardness coating and its RMSE value is also 19.95, which is less than other machine learning models (Decision Tree and Linear Regression).

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CHAPTER 1

INTRODUCTION

1.1 Surface Engineering

Surface engineering is the science that deals with improving the properties of the material surface 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 surface-engineered components include:

Abrasion wear resistance, improving aesthetic look, improving mechanical, electrical, and optical properties, and 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 component and it has several advantages. This technique can be applied to polymer and high-melting ceramics. 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 thermal (and/or kinetic) energy obtained from combustion of gases 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 (a single impacted droplet/particle)" are subjected to extremely high cooling speeds, often exceeding 100°F for metals is 106 K/s. Thermal spray methods have significant benefits 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 heat-treated 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].

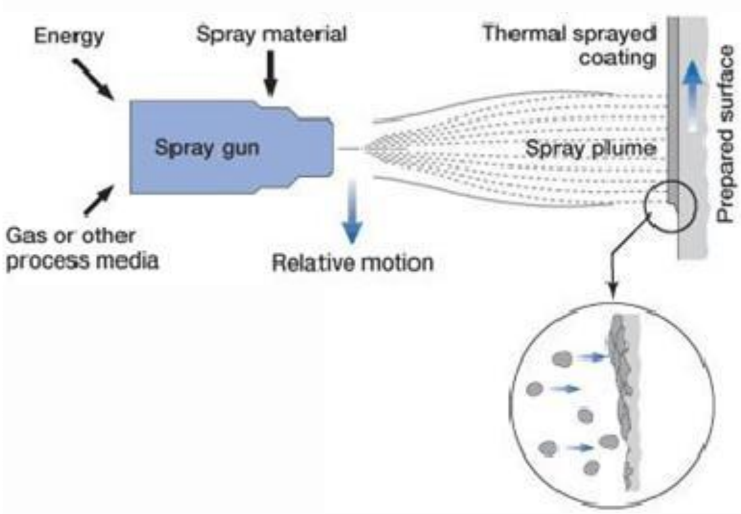

Figure 1 Basics mechanism of thermally sprayed coating [2]

1.3 Explosive Material

Explosive material is a kind of material that produces energy by an 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].

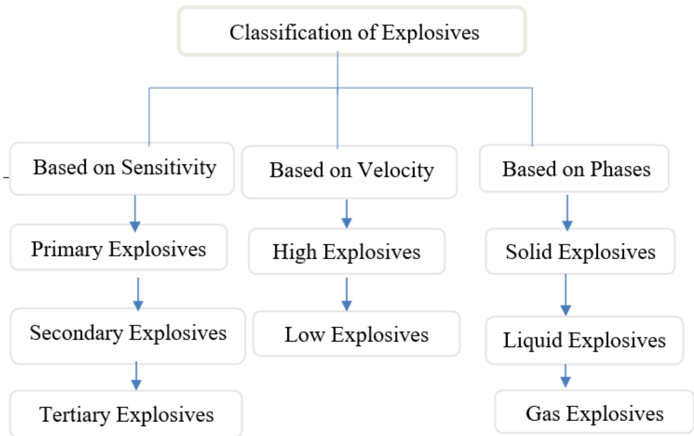

Figure 2 Types of Explosives

1.3.1 Classification of Explosives

1.3.1.1 According to the Velocity

(a) Low Explosive

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 (discussed in a later section). Low explosives detonate with speeds ranging from a few cm/s to about 0.4 km/s

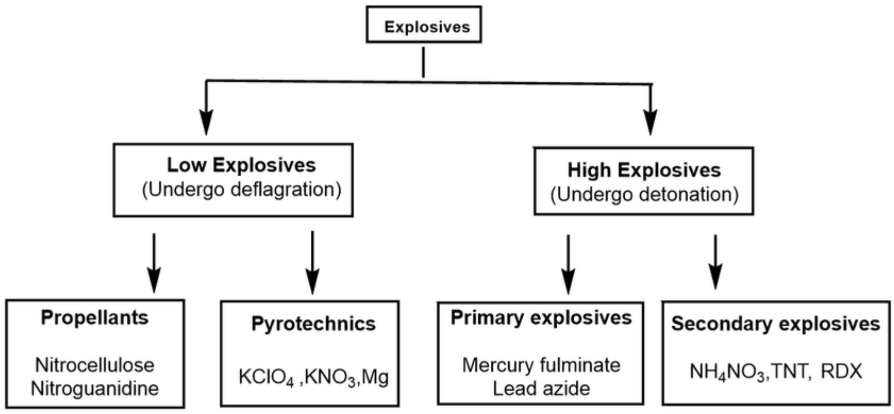

Figure 3 classification of explosives

under typical conditions. They have the potential to deflagrate swiftly, producing a detonation-like effect [3]. Examples: flash powder, gunpowder, 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 gunpowder includes KNO3 (75%), S (5%), and rest C (20%) [3].

(b) High Explosive: 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(Trinitrotoluene), TNP (trinitrophenol), TNX (TNT is a mixture of 40 percent trinitro-xylene (TNX) and 60 percent TNT.), RDX (Royal Demolition explosive), PETN (Pentaerythritol tetranitrate) [3].

1.3.1.2 According to sensitivity

(a)Primary Explosive

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 Explosive

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: PETN, and nitrocellulose [3].

(c)Ternary Explosives

Ternary explosives, often known as blasting agents, are so shock-insensitive 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].

1.3.1.3 On 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 explosives

These explosives are found in liquid form, like nitroglycerin.

(c) Gasioues explosive

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

1.4 Explosives in the Manufacturing Processes

1.4.1 Explosive 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 (this is another welding plate that is going to be welded on base 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 º (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, 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 4 describes the explosive welding/cladding.

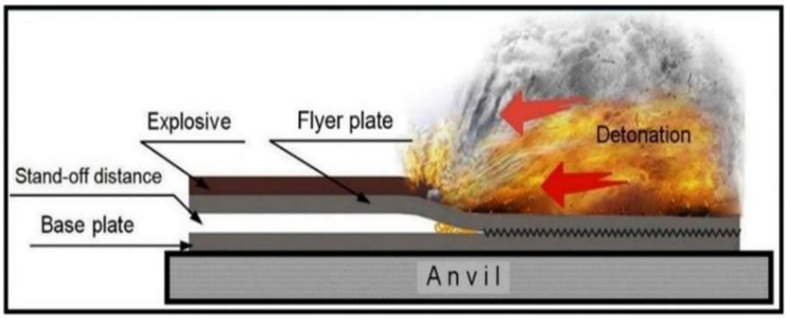

Figure 4 Explosive Welding/Cladding [13]

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 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:

(a) Stand-off method

(b) 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 throw the evacuation outlet . 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 5 is showing the schematic presentation of a stand-off explosive forming.

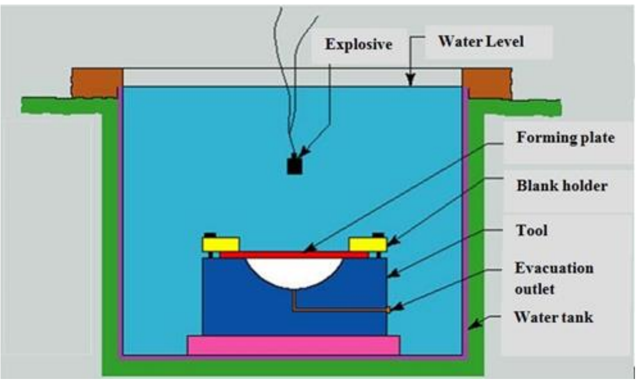

Figure 5 Schematic diagram of a stand-off explosive forming [23]

1.4.2.2 Contact method

The explosive charge in the form of a cartridge is held in direct contact with the workpiece while the detonation is initiated. The detonation builds up extremely high pressures (upto 30,000MPa) on the surface of the workpiece, resulting in metal deformation, and possible fracture. The process is used for bulging tubes locally.

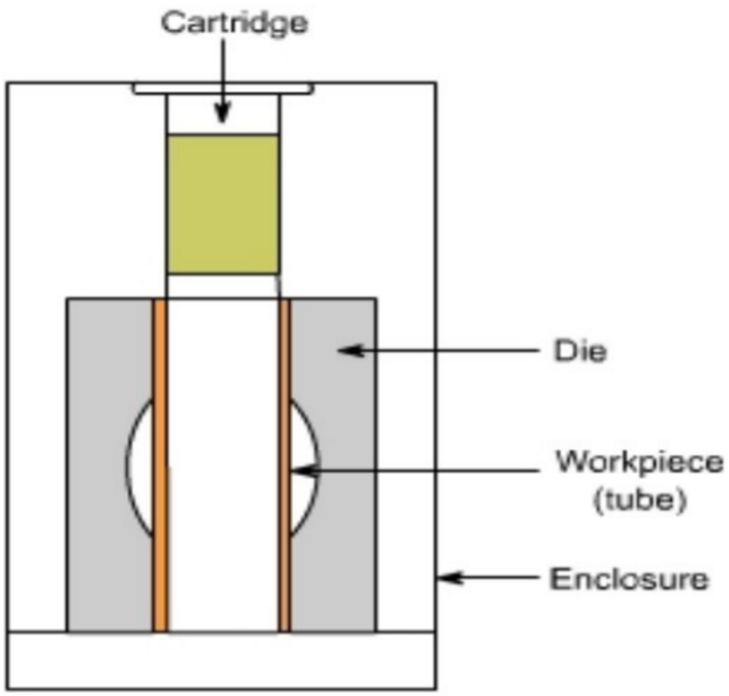

Figure 6 Schematic diagram of a contact explosive forming [12]

Chapter 2

Literature Review

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

2.1 Explosives in Manufacturing

Hussain et al.$BaAl_2O_4$ based coating is fabricated successfully.$BaAl_2O_4$-Based Coating contains amorphous phases in it. Addition of Ni-Al helps to improve the crystallinity of the coating. With adding Ni in with Al, Due to interdiffusion of materials at the interface, the adhesion strength of the coatings are significantly high**.**

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 + $Ba(NO_3)_2$, Mg + NH_4ClO_4, and Mg + $KMnO_4$. The findings revealed that all of these compositions are thermally stable. Mg & NH_4ClO_4 is the most reactive, followed by Al &$Ba(NO_3)_2$,Mg & $KMnO_4$[16].

K. Raghukandancreated 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 Thermal Spray-based coatings

Bing-yuan Han Performance analysis of plasma spray Ni60CuMo coatings on a ZL109 via a back propagation neural network model,a Back Propagation (BP) Artificial Neural Network model to predict the performance of Ni-based coatings.it was found that the increase and decrease trends of the two were similar and that the fitting degree was high, which indicates that the network has a high prediction accuracy.

Zaheer-ud-din,Abdul Qadeer MALIK Thermal Decomposition, Ignition and Kinetic Evaluation of Magnesium and Aluminium Fuelled Pyrotechnic Compositions.Thermally stable - Mg + NH4ClO4 < Mg + KMnO4 < Al +Ba(NO3)2

2.2 Identified Research Gap

Based on the review of the past work done research gap is identified. To the best of our knowledge, few literature has been found on the usage,but they are not varying contents of Al, $Ba(NO_3)_2$ and SOD. fabricated using novel explosive setup. Based on the data collected from the experiments no one predicticting the hardness of coating using machine learning model,

2.3 Objectives and Research Methodology

- To deposit Al-$BaAl_2O_4$ composite coating on the flat steel substrate by varying the feedstock compositions and Stand of distance(SOD).
- Characterization of the deposited coatings to obtain optimized Al-$BaAl_2O_4$ composite coating with superior mechanical properties.
- After collecting data from the experiment prediction of hardness using machine learning modeling random forest regression, decision tree, linear regression.

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 (15×15×10 mm^3).

Table 1 Elemental composition of AISI 1020 steel [3]

Elements	Fe	Mn	C	S	P
Contents (%)	99.08-99.53	0.30-0.60	0.17-0.23	<0.05	<0.04

3.1.2 Explosive material

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.

A varying constituent of explosive powder ($Al+Ba(NO_3)2$)is employed in the fabrication of the coating at different SOD(60 mm, 80 mm, 100 mm).All the coating made at different SOD by keeping constant $Ba(NO_3)_2$ gm.

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

Table 2 Explosive powder combinations

Sample Names.	Al(1)	Al(1.5)	Al(2)	Al(2.5)
Al/pyroquantity(gm)	1	1.5	2	2.5
$Ba(NO_3)_2$(gm)	2	2	2	2

3.2 Substrate Preparation

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

3.2.1 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.

3.2.2 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.

3.2.3 Grit Blasting

Grit blasting is employed for providing roughness on the substrate surface. Grit blasting provides the uniform desired 5 μm to 7 μm roughness to the surface for better adhesion of the coating.Grit blasting conditions are tabulated below.

Table 3 Grit blasting conditions

Substrate	AISI 1020
Grit type (mesh)	Alumina (24 mesh)
Blasting pressure (kgf/cm^2)	5
Blasting Time (minutes)	1

Stand-off distance (mm)	100

3.2.4 Preheating the Substrate and Powders:

The constituents of flash powder are preheated separately at 100 ºC for 10 minutes. Substrate preheating is performed in the muffle furnace for 15 minutes at 350 ºC 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 4 Substrate preheating parameters

Time(minutes)	15
PreheatingTemperature(ºC)	350

Table 5 Preheating parameters of constituents of flash Powder

Time(minutes)	10
Preheating Temperature(ºC)	100

3.3 Experiment Setup

The setup used for obtaining the coating on the substrate is shown in Figure11. This developed experimental setup [2] has the following components: substrate holder, gun barrel, switch, spring, ignition coil, and base.The long steel gun barrel 145 mm shown in Figure 11 is the major component of the developed setup. And substrate holder is hold on vertical base plate by adjusting it we can vary the SOD. Preheat-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 the substrate is maintained at varying SOD, such as 60 mm, 80 mm, and 100mm. Deposition of coating is achieved with a two-point ignition system and utilizing a mixed form of the constituent of the flash powders.

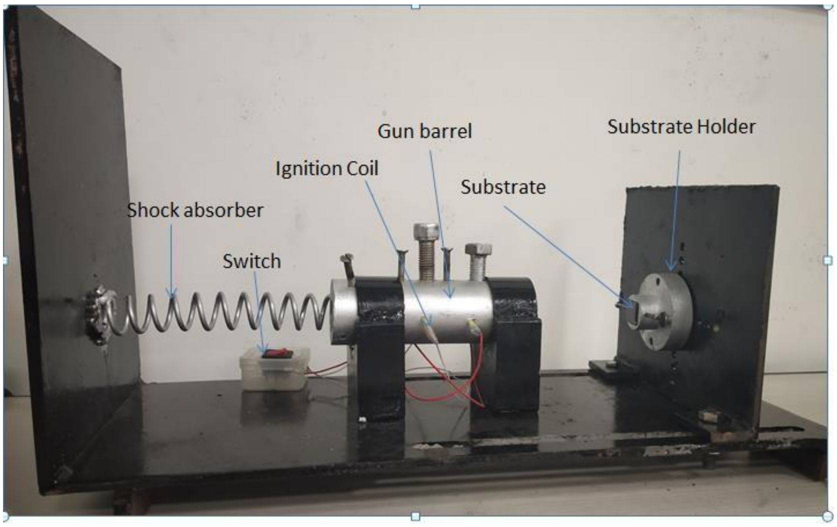

Figure 7Experimental setup [2]

3.4 Selection of 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. Two ignition coil system ,distance between the powder, stand of distance, substrate are the process parameters are shown in a sectional view of the gun-barrel in Figure 8.

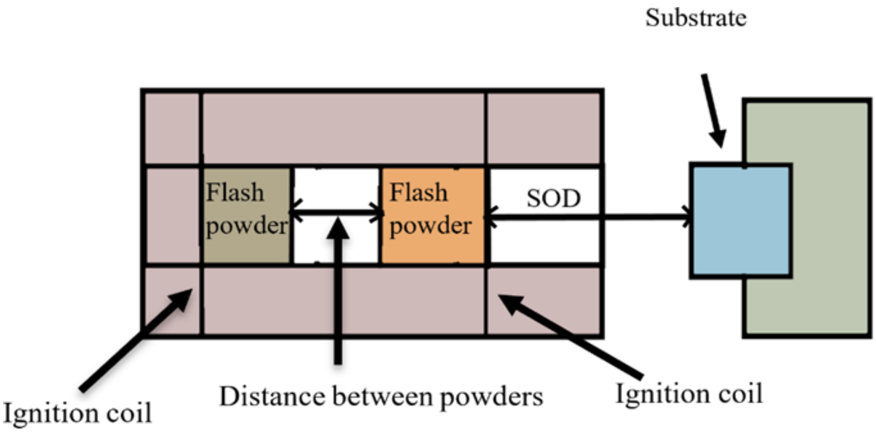

Figure 8 Process parameter for spray coating [2]

Stand-off distance	60 mm,80 mm,100 mm
Ignition type	Multi point ignition system
Preheat temperature for powders	100 °C
Substrate preheating Temperature	350 °C

Table 6 Process parameters used in our experimental work

3.7 Characterization of deposited coating:

3.7.1 X-ray Diffraction

The coating phases are analysed by using XRD patterns obtained with an X-ray diffractometer (Rigaku Smart Lab 3 kW having Cu-Kα 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'PertHighScore" software and plotted with the help of Origin Pro software.

3.7.2 Microstructure analysis of coating

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 mesh size ranging from 220 to 2500, followed by diamond polishing through diamond paste consisting of grits ranging from 3 to 0.25 µm. 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.

3.7.3 Thickness Analysis

The thickness of the coating is measured by observing the cross-section of coatings through an inverted 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 considered.

3.7.4 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; cutoff length .8 mm,evaluation length 4 mm using a Gaussian filter.

3.7.5 Microhardness Analysis

The hardness, which is the characteristic of the material, is defined as the resistance against indentation. It is measured by making a permanent indentation on the material and measuring its diagonals of the indent d1,d2. In this work, highly polished mounted samples are used so that the impressions can be identified clearly to measure their diagonals. 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-depositedand 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 = 0.1891*F/d*^2[27]

3.7.6 prediction of hardness of coating using machine learning models

To apply a machine learning model on surface coating data, we have to follow these general steps:

1. Data Collection: Collect the relevant surface coating data, including input features like SOD, compositions andand mechanical properties from the characterization of coating corresponding target values (Hardness of coating).

2. Data Pre-processing: Clean the data by handling missing values, outliers, and noise. Perform necessary transformations such as scaling or normalization to ensure consistency in the data.

1. Feature Selection/Engineering: Select the relevant features that have a significant impact on the surface coating process. Alsoengineer new features based on domain knowledge to enhance the model's performance.

2. Split the Data: Divide the data into training, validation, and testing sets. The training set is used to train the model, the validation set helps tune hyperparameters and evaluate model performance, and the testing set is used to assess the final model's generalization.

3. Model Selection: Choose an appropriate machine learning model that suits the nature of the surface coating problem. This can include algorithms such as linear regression, decision trees, random forests, support vector machines (SVM), or neural networks.

4. Model Training: Train the selected model using the training data. The model learns to identify patterns and relationships between the input features and target values.
5. Hyperparameter Tuning: Optimize the model's hyperparameters using techniques like grid search, random search, or Bayesian optimization. This helps improve the model's performance by finding the best combination of hyperparameters.
6. Model Evaluation: Evaluate the trained model using the validation set. Common evaluation metrics for regression problems include mean squared error (MSE), root mean squared error (RMSE), and coefficient of determination (R^2). Select the metrics that best measure the performance based on your specific requirements.
7. Model Testing: Assess the final model's performance on the unseen testing set to measure its ability to generalize to new data. Use the same evaluation metrics as in the validation step.
8. Model Deployment: Once satisfied with the model's performance, deploy it to make predictions on new and unseen surface coating data. Ensure that the model is integrated into a production system or pipeline for real-time or batch predictions.
9. Monitor and Update: Continuously monitor the model's performance in production and update it as needed. Collect feedback and new data to retrain or fine-tune the model periodically to maintain its accuracy and relevance.

Chapter 4

Result and discussion

4.1 XRD Analysis of Coating

Figure 9 shows the XRD patterns of coating fabricated by burning the pyrotechnic mixture with the varying aluminium amount from 1 to 2.5 gm at fixed SOD of 60 mm. The coating obtained from 1 gm Al shows the presence of $BaCO_3$and $BaAl_2O_4$, as shown infigure 9(a). When Al amount is increased to 1.5 gm, an additional Al phase is observed apart from $BaCO_3$ and $BaAl_2O_4$, as depicted infigure 9(b). The phases such as $BaAl_2O_4$and Al are formed in the coating fabricated from 2 gm Al as shown in figure 9(c). In this case, the intensity of most of the $BaAl_2O_4$ phases is increased as compared to the previous two cases. With the further increment in Al amount to 2.5 gm leads to the formation of the same phases $BaAl_2O_4$ and Al in the resultant coating, as depicted in figure 9(d). However, most of the peaks in the pattern belong to Al phase, and few peaks belong to $BaAl_2O_4$. Also, the intensity of these Al is very high. The large number of Al peaks may be due to the presence of unreacted Al powder during the explosive reaction.

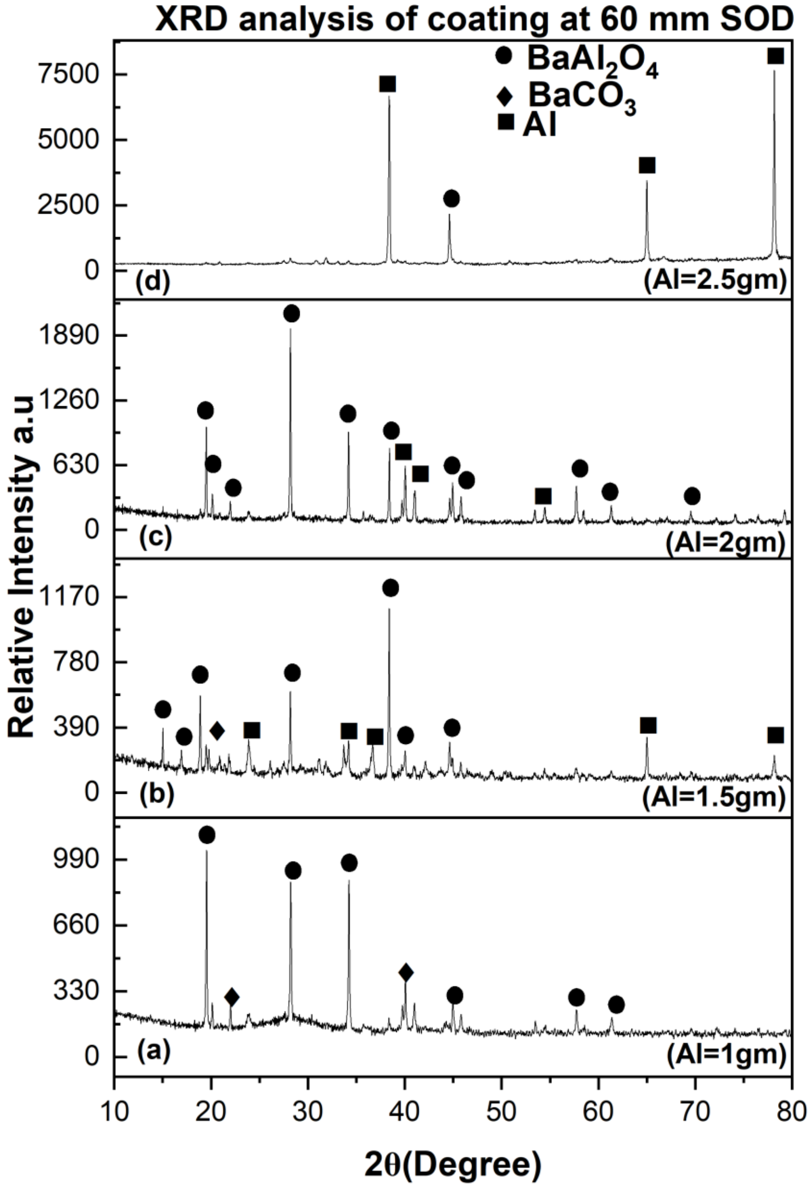

Figure 9 XRD pattern of coatingfabricated at SOD mixture with varying Al content

Figure 10 shows the XRD patterns of coating fabricated by burning the pyrotechnic mixture with the varying aluminium amount from 1 to 2.5 gm at fixed SOD of 80 mm. The coating obtained from 1 gm Al shows the presence BaOand $BaAl_2O_4$, as shown infigure 10(a). When Al amount is increased to 1.5 gm, an additional Al phase is observed apart from $BaAl_2O_4$ as depicted infigure 10(b). The phase such as $BaAl_2O_4$ are formed in the coating fabricated from 2 gm Al as shown in figure 10(c). In this case, the intensity of most of the $BaAl_2O_4$ phases is increased as compared to the previous two cases. With the further increment in Al amount to 2.5 gm leads to the formation of the samephases $BaAl_2O_4$ and Al in the resultant coating, as depicted in figure 10(d). However, most of the peaks in the pattern belong to Al phase, and few peaks belong to $BaAl_2O_4$. Also, the intensity of these Al is very high. The large number of Al peaks may be due to the presence of unreacted Al powder during the explosive reaction.

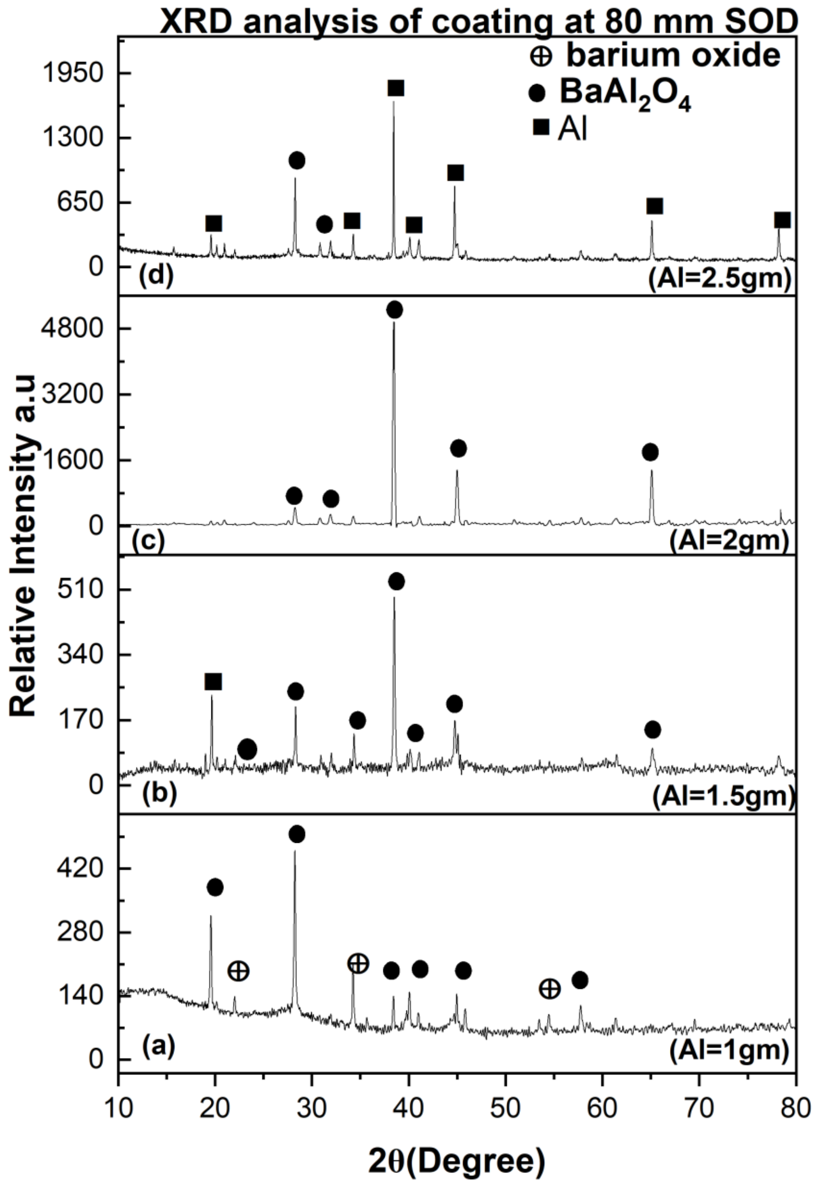

Figure 10 XRD pattern of coatingfabricated at 80 mm SOD mixture with varying Al content

Figure 11 shows the XRD patterns of coating fabricated by burning the pyrotechnic mixture with the varying aluminium amount from 1 to 2.5 gm at fixed SOD of 80 mm. The coating obtained from 1 gm Al shows the presence martensite and $BaAl_2O_4$, as shown infigure 11(a). When Al amount is increased to 1.5 gm, an additional Al phase is observed apart from$BaAl_2O_4$ as depicted infigure 11(b). The phase such as $BaAl_2O_4$ are formed in the coating fabricated from 2 gm Al as shown in figure 11(c). In this case, the intensity of most of the $BaAl_2O_4$ phases is increased as compared to the previous two cases. With the further increment in Al amount to 2.5 gm leads to the formation of the samephases $BaAl_2O_4$ and Al in the resultant coating, as depicted in figure 11(d). However, most of the peaks in the pattern belong to Al phase, and few peaks belong to $BaAl_2O_4$. Also, the intensity of these Al is very high. The large number of Al peaks may be due to the presence of unreacted Al powder during the explosive reaction.

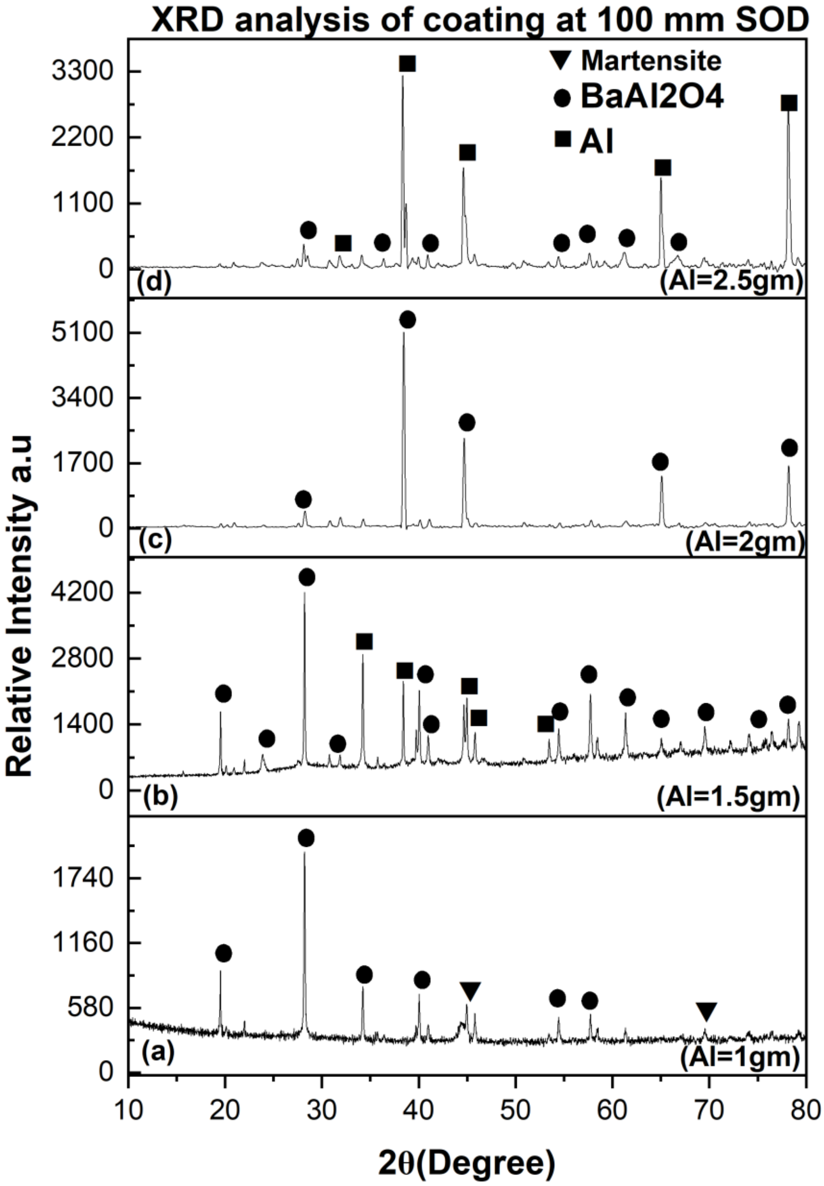

Figure 11XRD pattern of coating fabricated at 100 mm SOD mixture with varying Al content

4.2 ThicknessAnalysis

Coating thickness on steel substrates plays a critical role in various industries and applications, including manufacturing, construction, automotive, and corrosion protection. The coating serves as a barrier between the steel substrate and the external environment, providing protection against corrosion, wear, and other forms of damage. The thickness of the coating is a crucial factor that determines its effectiveness and longevity.

Coating thickness is typically measured in micrometers (μm).The desired thickness can vary depending on the specific application and the requirements of the industry. In some cases, thin coatings are sufficient for aesthetic purposes or minor protection, while thicker coatings are necessary for heavy-duty applications.

The thickness of the coating fabricated by burning varying Al amount and fixed 2 gm of $Ba(NO_3)_2$ at 60 mm SOD is shown in figure 12. The coating thickness is found to be increased with an increase in Al amount in flash powder. The thickness of the coating is noticed to be 69.81μm for 1 gm of Al. The maximum coating thickness is estimated as 215.38μm for 2.5 gm of Al. The thickess is increases because the aluminiumn does not get sufficient time to melt beause of very less SOD.

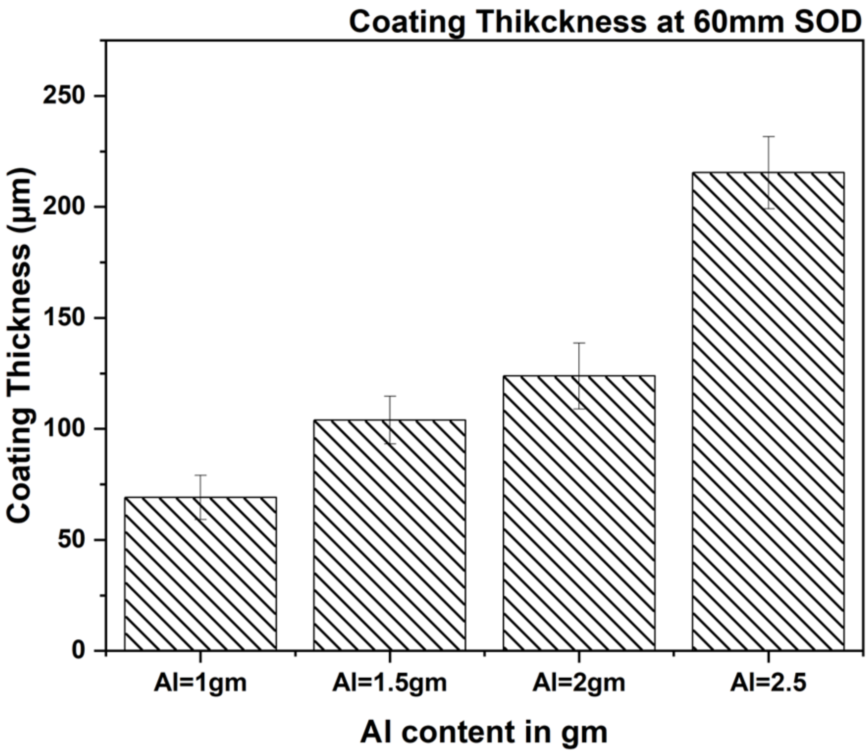

Figure 12Thickness of coating at varying Al content at 60mm SOD

The thickness of the coating fabricated by burning varying Al amount and fixed 2 gm of $Ba(NO_3)_2$ at 80 mm SOD is shown in figure 13. The coating thickness is found to be increased with an increase in Al amount in flash powder. The thickness of the coating is noticed to be 65μm for 1 gm of Al. The maximum coating thickness is estimated as 83µm for 2.5 gm of Al.

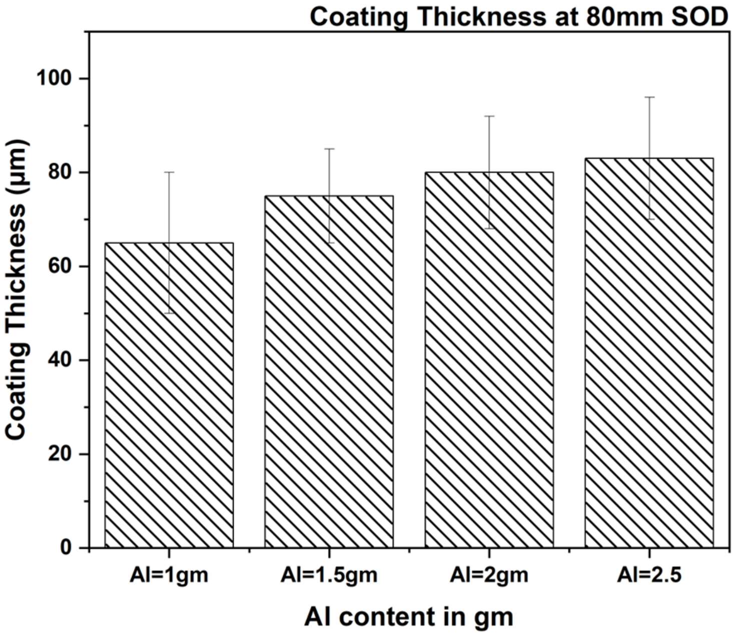

Figure 13Thickness of coating at varying Al content at 80mm SOD

The thickness of the coating fabricated by burning varying Al amount and fixed 2 gm of $Ba(NO_3)_2$ at 100 mm SOD is shown in figure 14. The coating thickness is found to be decresases with an increase in Al amount in flash powder till 1.5gm. The thickness of the coating is noticed to be 57.4838µm for 1.5 gm of Al. The maximum coating thickness is estimated as 166.7492µm for 2.5 gm of Al.

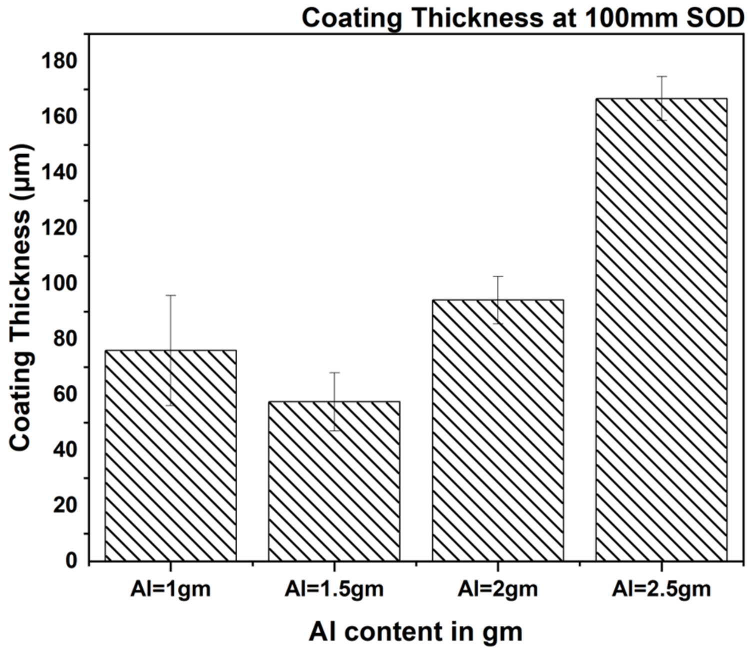

Figure 14Thickness of coating at varying Al content at 100mm SOD

From the above graphs the study found that increasing the coating thickness, coatings on steel substrates led to improved corrosion resistance. This is attributed to the formation of a thicker layer, which provides greater protection to the steel substrate from environmental exposure.

From the thickness graph we have conclude that at 60mm SOD the thickness of coating is maximum but hardness of coating is that much good and also roughness is more.

4.3 Surface Roughness

Surface roughness is a critical parameter that significantly influences the performance and functionality of coatings. Coating surfaces can exhibit varying degrees of roughness, which can impact various aspects such as adhesion,durability, and functionality.

The roughness of the coating fabricated by burning varying Al amount and fixed 2 gm of $Ba(NO_3)_2$ at 60 mm SOD is shown in figure 15.The coatings as-sprayed surfaces are seen to have a rough and irregular texture. This is primarily caused by the existence of iron globules.with increase in Al content in the mixture the roughness will increase. The coating produced by explosion of a pure pyrotechnic mixture has an minimum average surface roughness (Ra) of about 5.75 µm at Al of 1gm.

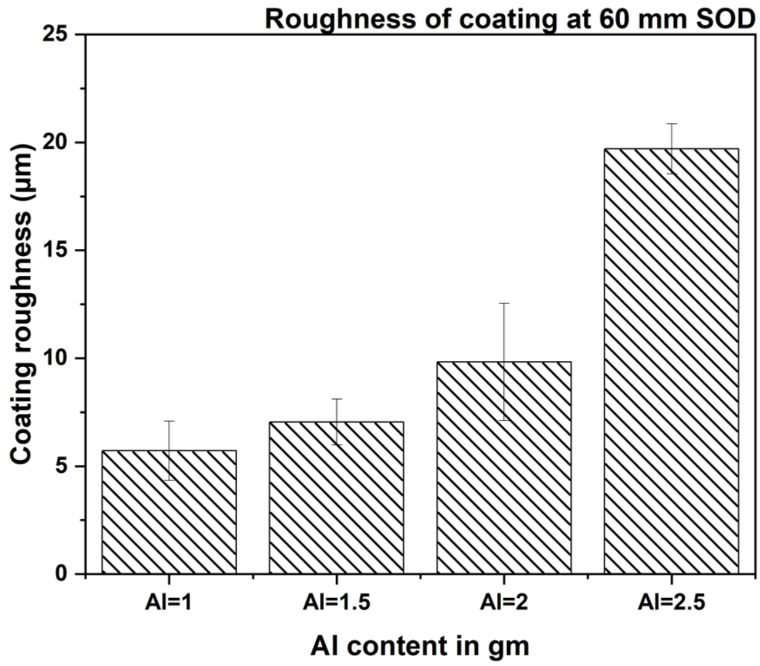

Figure 15 Surface roughness of coating at varying Al content at 60mm SOD

The roughness of the coating fabricated by burning varying Al amount and fixed 2 gm of $Ba(NO_3)_2$ at 80 mm SOD is shown in figure 15. with increase in Al content in the mixture the roughness will decreases till 2gm. The coating produced by explosion of a pure pyrotechnic mixture has an minimum average surface roughness (Ra) of about 3.5 µm at Al of 2gm.

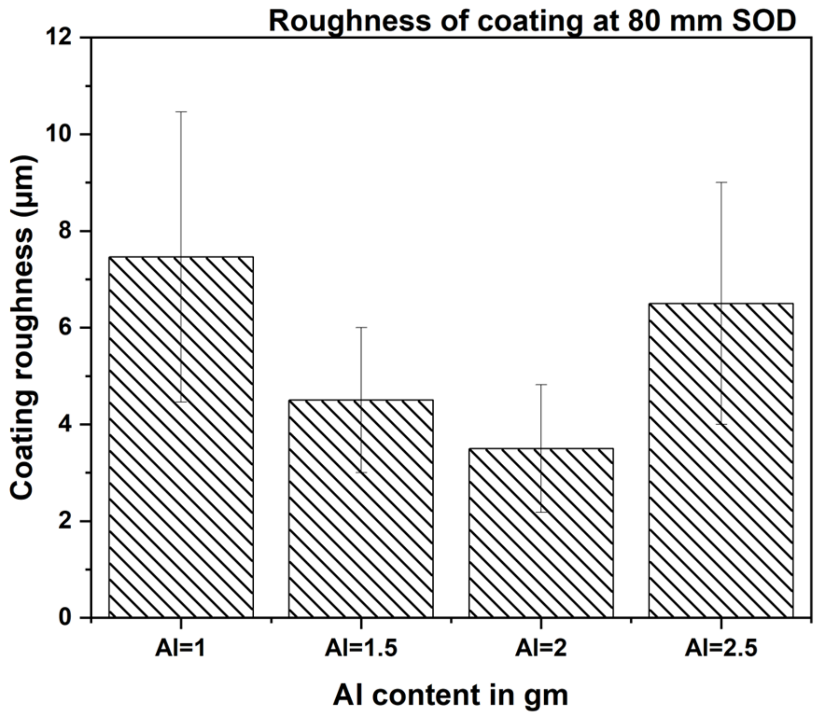

Figure 16Surface roughness of coating at varying Al content at 80mm SOD

The roughness of the coating fabricated by burning varying Al amount and fixed 2 gm of $Ba(NO_3)_2$ at 100 mm SOD is shown in figure 17,with increase in Al content in the mixture the roughness will decreases till 1.5 gm. The coating produced by explosion of a pure pyrotechnic mixture has an minimum average surface roughness (Ra) of about 4.85 µm at Al 1.5 gm.

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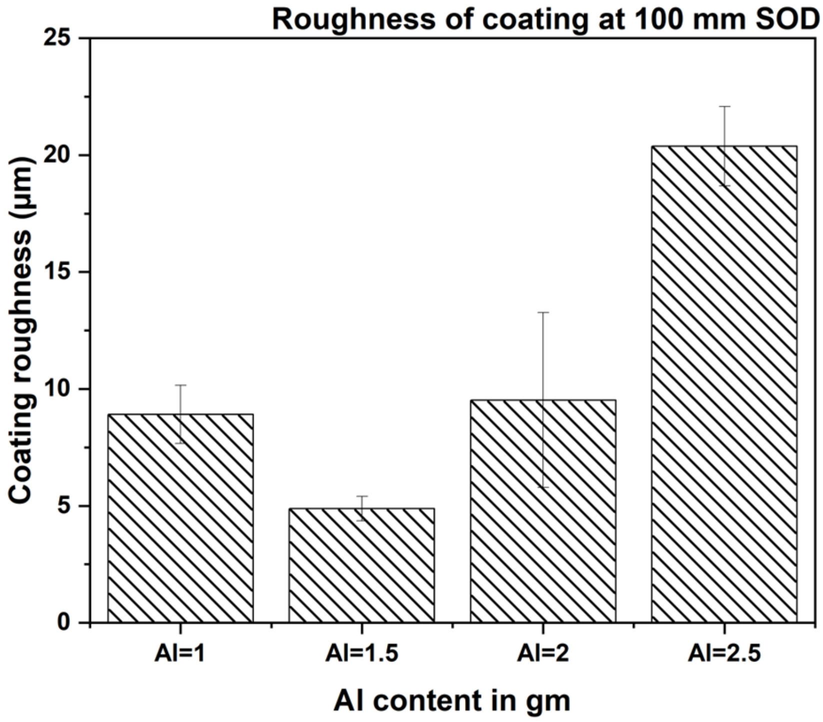

Figure 17Surface roughness of coating at varying Al content at 100mm SOD

4.4 Vickers microhardness analysis

Vickers microhardness tests are performed on the diamond polished, mounted samples. The indents are made on the cross-section of the coating against a load of 50 gm and 12 seconds of dwell time. The hardness value of the bare substrate andas-deposited coatings is analyzed.It involves applying a controlled load to the coating surface using a Vickers diamond indenter and measuring the size of the resulting indentation.

4.4.1 Vickers microhardness analysis of as-deposited coating

The Vickers microhardness value of coating fabricated by burning varying Al amount in flash powder at 60 mm of SOD is shown in Figure 18. When the Al amount is increased from 1 to 2 g, the hardness value is found to be increased gradually. Further increase in Al amount leads to the reduction in hardness value. Because of unreacted Al is present in the coating.The maximum hardness value is achieved for coating obtained from 2 gm of Al, because more amount of $BaAl_2O_4$ phase is present in the coating which is confirmed by the XRD pattern.

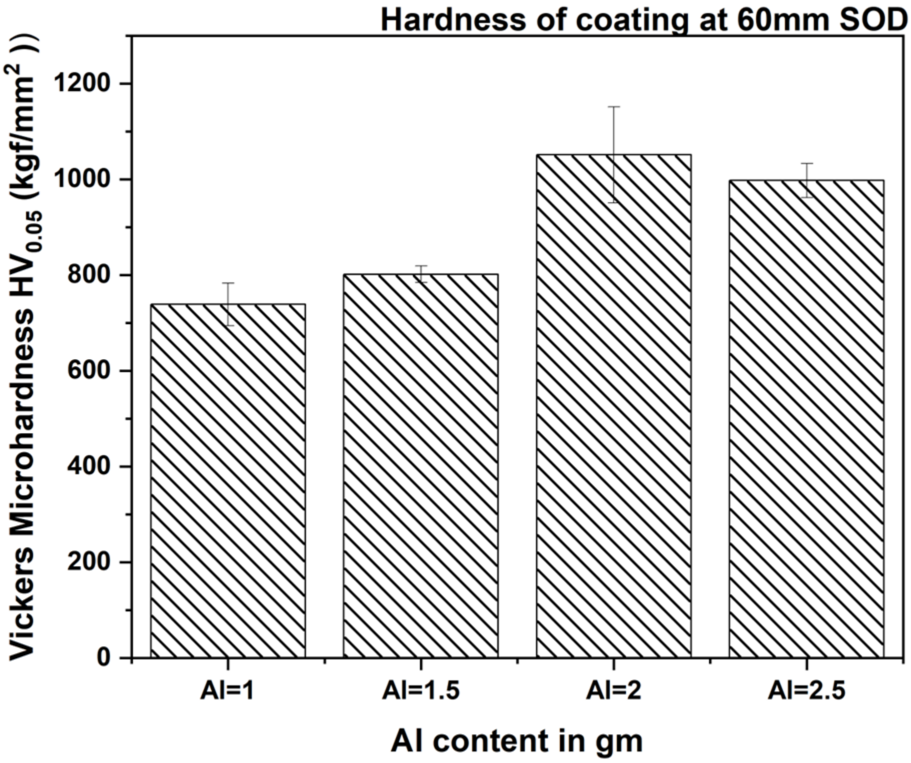

Figure 18Average vickers microhardness of deposited coating obtained from the from spraying Ba(NO$_3$)$_2$ and Al powders at SOD of 60mm with varying composition

The Vickers microhardness value of coating fabricated by burning varying Al amount in flash powder is shown in Figure 19. When the Al amount is increased from 1 to 1.5 gm, the hardness value is found to be increased gradually,with further increase in Al to 2 gm the hardness of the coating is drastically inceases .After 2gm of Al amount leads to the reduction in hardness value because of unreacted Al is present in the coating.The maximum hardness value is achieved for coating obtained from 2 gm of Al, because of more amount of phases $BaAl_2O_4$ is present it the coating which is confermed from the XRD pattern.

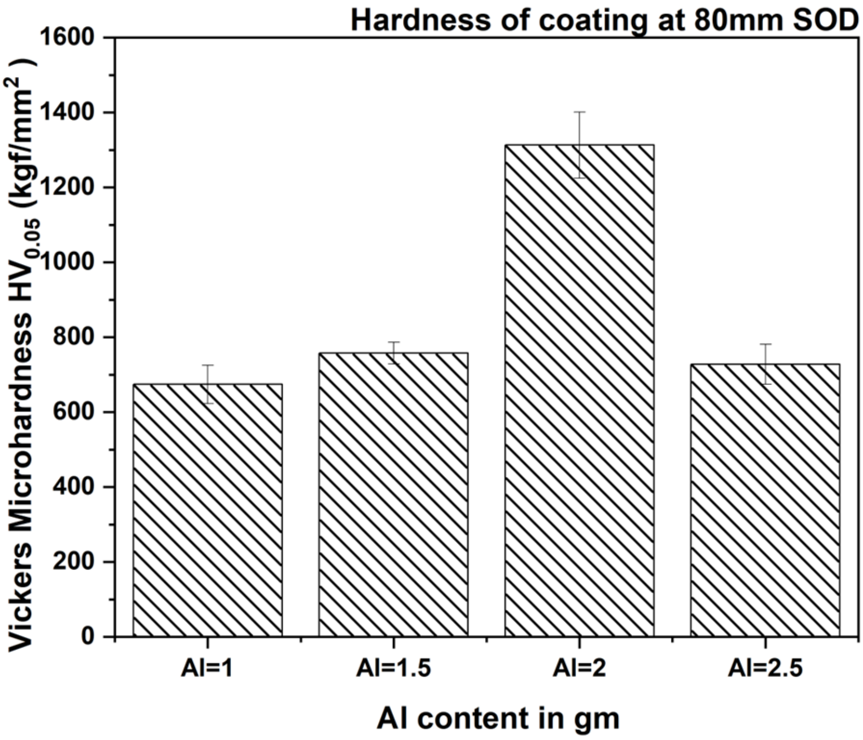

Figure 19Average vickers microhardness of deposited coating obtained from the from spraying $Ba(NO_3)_2$ and Al powders at SOD of 80mm with varying composition

The Vickers microhardness value of coating fabricated by burning varying Al amount in flash powder is shown in Figure 20. When the Al amount is increased from 1 to 2.5 gm, the hardness value is found to be increased gradually.the hardness of the coating is increase because of the the formation of $BaAl_2O_4$ phase increase.

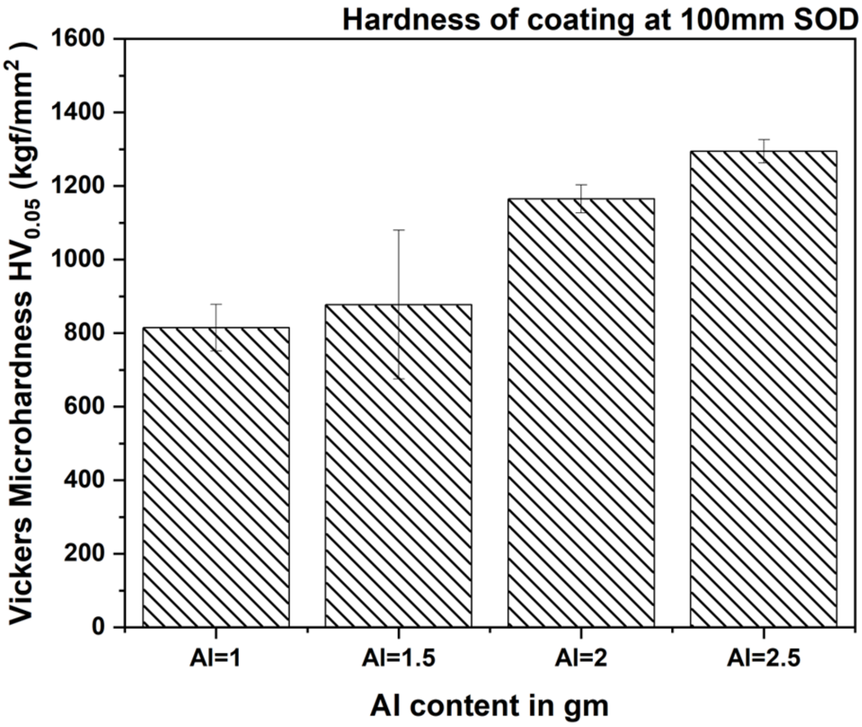

Figure 20Average vickers microhardness of deposited coating obtained from the from spraying $Ba(NO_3)_2$ and Al powders at SOD of 100mm with varying composition

It can be concluded from above three hardness plots that the maximum hardness value is estimated for 2 gm of Al and maintaining 80 mm of SOD during the explosive reaction among the different SOD employed.The minimum hardness of coating found to be at 1 gm of Al in each case.

4.4.2 Hardness of the hardened zone of the substrate

Explosions can have a significant impact on the structural integrity of steel surfaces, including their hardness (i.e., resistance to indentation).The effects of an explosion on steel surfaces will depend on various factors, such as the type of explosion, the distance from the blast, the amount of explosive used, etc.

In general, explosions create high-pressure shock waves that can cause deformation, displacement, or even fracture of the steel substrate. The shock wave can create a rapid compression and expansion of the steel, which can result in microstructural changes that affect the hardness ofthe steel material. For example, the surface can become harder due to strain hardening or softer due to thermal softening or microstructural changes.

The hardness of the subsurface regions of the substrates is observed to be increased significantly. This is attributed to the hardening effect of the steel substrate caused by the explosion impact, which has led to the formation of an equiaxed nano-crystalline austenite structure in the impact surface layer.

Figure 29 representsVickers microhardness value of the bulk steel substrate and hardened substrate (below the interface zone) obtained from the explosive coating fabricated with varying Al content in flash powderand maintaining a fixed 60mm of SOD.Below 20μm from the interface,with an increase in Al the hardness of substrate increase till 2gm of Al, because of high velocity forming,beyond 2 gm the there is not very much significant change hardness.The depth of hardened zoneis noticed to be decreased because of high energy at higher amount of Al is release, only the grains at the top surface gets affected.

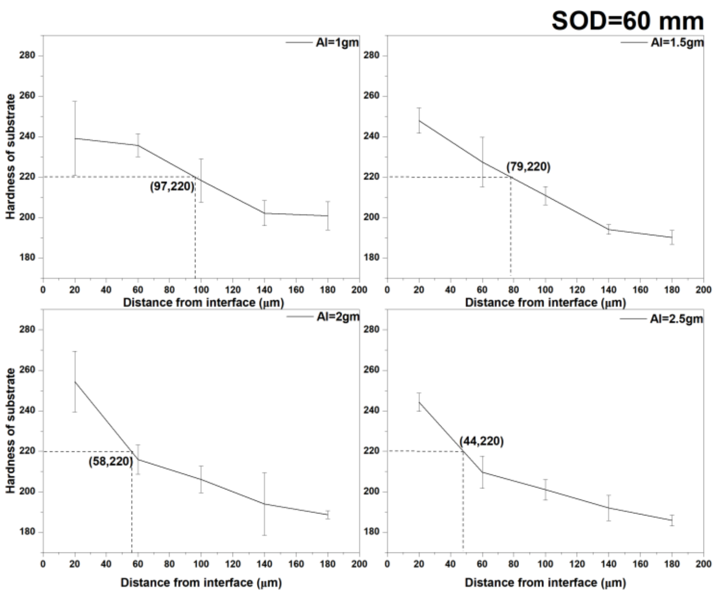

Figure 21 Hardness of hardened zone of substrate at 60mm SOD

Figure 22 representsVickers microhardness value of the bulk steel substrate and hardened substrate (below the interface zone) obtained from the explosive coating fabricated with varying Al content in flash powderand maintaining a fixed 80mm of SOD.

Below 20μm from the interface, with an increase in Al the hardness of substrate goes on increase till 1.5 gm of Al, because of high velocity forming,beyond1.5 gm the there is not slightly decrease in hardness.The depth of hardened zone is noticed to be decreased because of high energy at higher amount of Al is release, only the grains at the top surface gets affected.

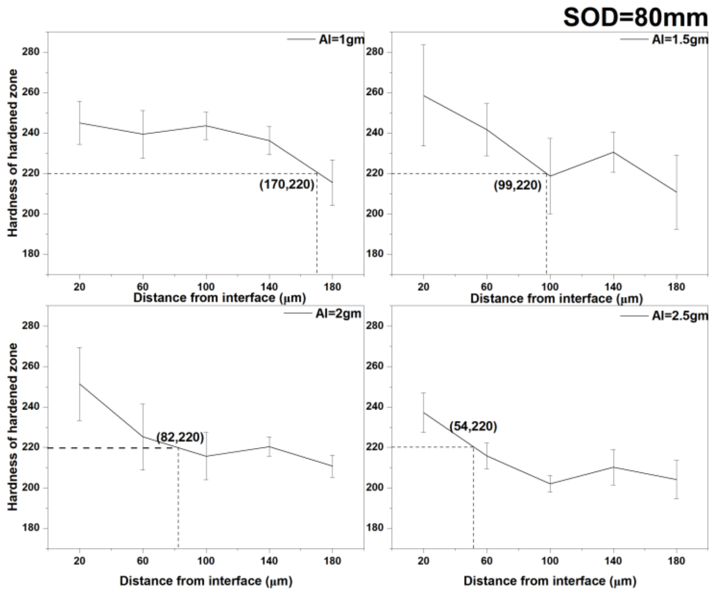

Figure 22 Hardness of hardened zone of substrate at 80mm SOD

Figure 23 representsVickers microhardness value of the bulk steel substrate and hardened substrate (below the interface zone) obtained from the explosive coating fabricated with varying Al content in flash powderand maintaining a fixed 100mm of SOD.

Below 20μm from the interface, with an increase in Al the hardness of substrate goes on.The depth of hardened zone is noticed to be decreased because of high energy at higher amount of Al is release, only the grains at the top surface gets affected.

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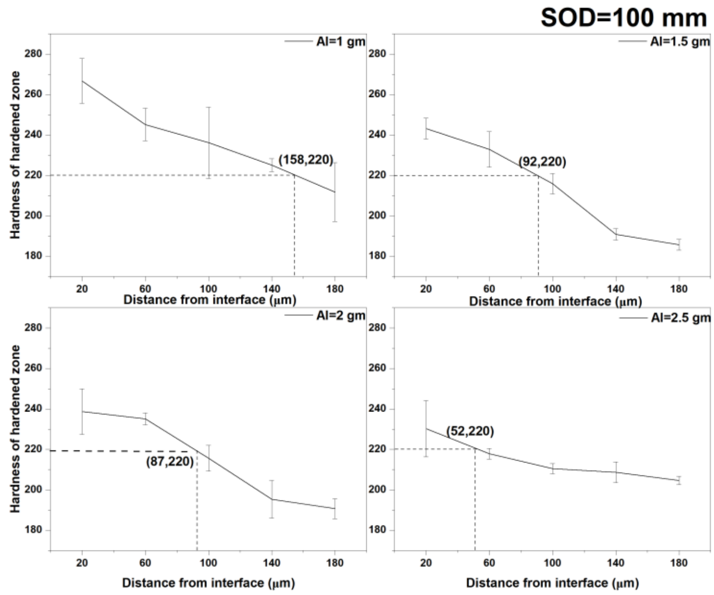

Figure 23 Hardness of hardened zone of substrate at 100mm SOD

The value of hardness of substrate is more then the hardness of base material at 20 μm below the interface, because of explosion grain at the top surface gets affected.

4.5 Prediction of hardness using Machine learning model

Decision Tree:

A decision tree regressor is a simple and intuitive machine learning algorithm used for predicting continuous numeric values. It resembles a flowchart-like structure where each internal node represents a feature or attribute, and each branch represents a decision based on that attribute. The algorithm learns from the data by recursively splitting the feature space to minimize the differences between predicted and actual values. At the leaf nodes of the tree, the regressor provides the predicted numeric values. Decision tree regressors are effective in capturing complex relationships between input features and the target variable. They are particularly useful when the data exhibits nonlinear patterns or interactions. Decision tree regressors are easy to interpret, making it straightforward to understand the decision-making process and explain the results. They also handle missing data and outliers gracefully. Overall, decision tree regressors offer a powerful and versatile approach for conducting regression analysis in various fields, enabling researchers to make accurate predictions and gain valuable insights from their data.

Random Forest:

Random forest regression is a popular machine learning algorithm that combines the power of multiple decision trees to make accurate predictions for regression tasks. It works by creating an ensemble of decision trees, where each tree independently predicts the target variable. The random forest algorithm introduces randomness during the tree-building process by selecting a subset of features and a random subset of the training data for each tree. This randomness helps reduce overfitting and improve the model's generalization ability. The final prediction is obtained by averaging the predictions of all the individual trees in the forest. Random forest regression is known for its robustness, as it can handle noisy data, outliers, and missing values effectively. It can capture nonlinear relationships, interactions, and complex patterns present in the data. The algorithm is highly scalable, making it suitable for large-scale regression problems. Random forest regression is widely used in various domains due to its accuracy, robustness, and interpretability, allowing researchers to make reliable predictions and gain insights from their regression analysis.

Linear Regression:

Multivariate linear regression is a statistical modelling technique used to understand the relationship between multiple input variables and a continuous target variable. It extends the concept of simple linear regression, which analyzes the relationship between two variables, to handle scenarios where there are multiple predictors. In multivariate linear regression, the goal is to fit a linear equation that best represents the relationship between the input variables and the target variable. The model estimates the coefficients or weights for each input variable, indicating their contribution to the predicted outcome. By considering multiple predictors simultaneously, multivariate linear regression enables us to explore the joint effects of these variables on the target variable. It also allows us to assess the statistical significance and interpret the impact of each predictor while controlling for other variables. Multivariate linear regression is commonly used in various research fields to analyze complex relationships and make predictions based on multiple input features.

Table 7 Accuracy of Machine learning models

Model	**R^2**	**RMSE**
Decision Tree	0.9908	21.69
Random Forest	0.9922	19.95
Linear Regression	0.6880	126.32

Random Forest shows accuracy of 99.22% prediction accuracy of hardness and its RMSE value is also 19.95 which is less than other machine learning model. Decision Tree is second best model with accuracy of 99.08% and RMSE value 21.69. Both this machine learning model can handle the complex non-linear relationship of independent variables with dependent variable. This theory is further supported by prediction from the Linear regression model where its prediction accuracy is only 68.80 % and RMSE value is also quite high 126.32.

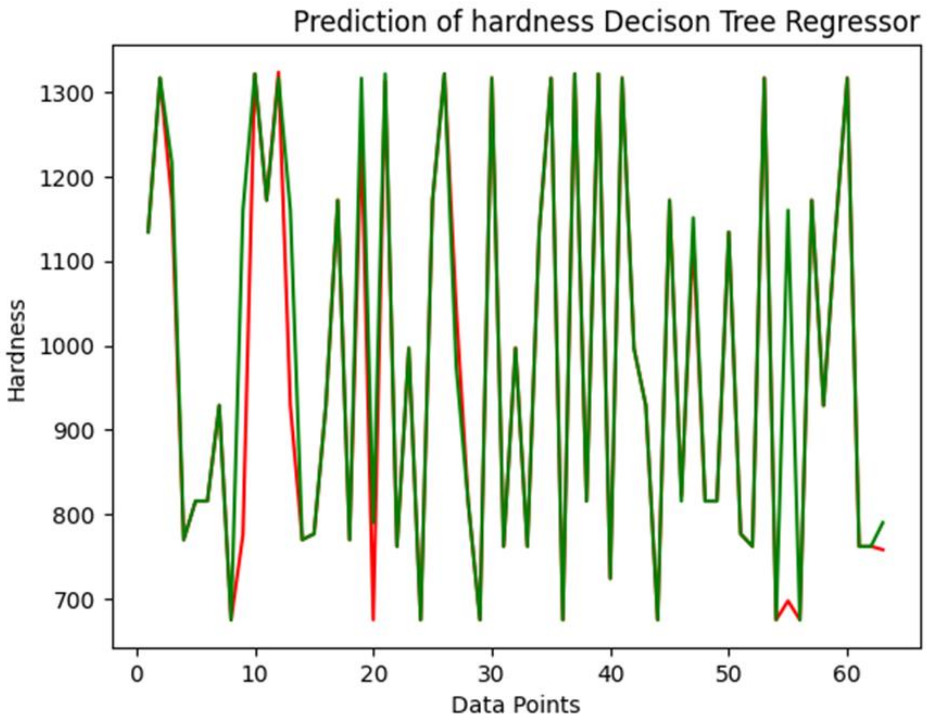

Figure 24predctions using Decison Tree Regressor

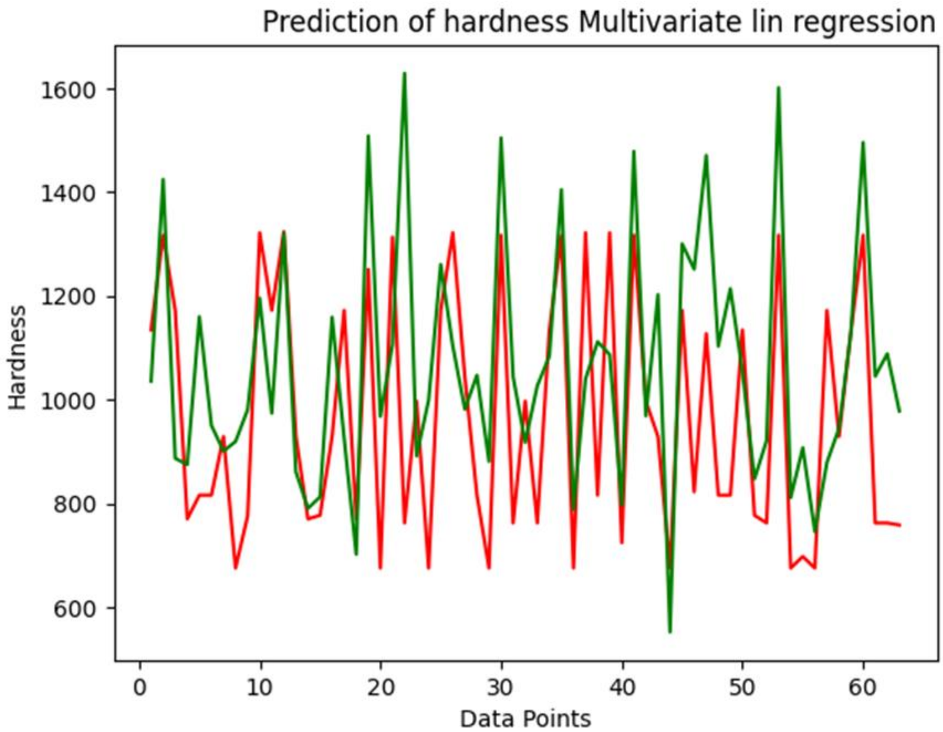

Figure 25 predictions using Multivariate linear regression

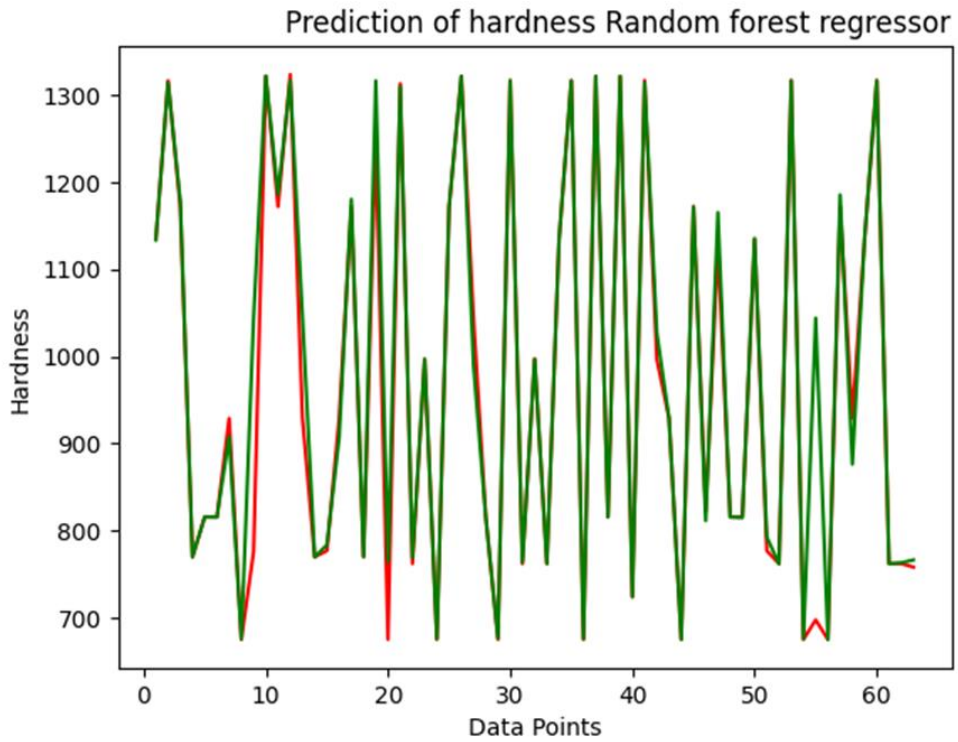

Figure 26 prediction using random forest regressor

4.6 Conclusions

In this study, pyrotechnic mixtures made of $Ba(NO_3)_2$ and Al powders are burned using a novel explosive spray coating method to effectively deposit $BaAl_2O_4$-based composite coatings on low-carbon steel with varying Al content and SOD. The following pointwise conclusions can be drawn from this study.

- The hardness of the fabricated coating is found to be increased with an increase in Al amount up to 2 gm at all three different SODs.
- The explosive coating obtained from 2 gm of Al and at 80 mm SOD shows maximum coating hardness due to the higher presence of hard phase such as $BaAl_2O_4$ confirmed by the XRD analysis among all different fabricated coatings.
- For all three different SODs, the depth of the hardened substrate zone of coating is observed to be decreased with an increment in Al content from 1 to 2.5 gm.

- The substrate hardness right below 20 μm from the coating-substrate interface is observed to be increased compared to the bare mild steel substrate for all coatings fabricated with varying Al content and different SODs.

- With enhancement of Al amount in flash powder, the coating thickness is noticed to be increased at all different SODs except 100 mm. The coating thickness is first decreased from Al content 1 to 1.5 gm and further increment in Al content leads to enhancement in coating thickness at 100 mm SOD.

- The coating fabricated by burning 2 gm of Al at 80 mm of SOD shows the minimum average surface roughness due to the proper melting and well spreading of the feedstock powder.

- Random forest model predicts better than all other ML models.

4.7 Future Scope

A proper mechanism can be implemented in the developed coating setup to make the process continuous, which can help to achieve a higher coating thickness along with improved physical and mechanical properties of the deposited coating.

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