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Surface Roughness And Taguchi Analysis Of Electrodeposited Cutio$_2$ Coating On Mild Steel

[1]Sushma A Patil, [2]Thippeswamy Ekbote, [3]K Sadashivappa

[1,2,3]Assistant Professor, [2]Professor, [3]Professor

[1]Mechanical Department,

[1,2,3]Bapuji Institute of Engineering and Technology, Davangere, India

Abstract: This study has been undertaken to investigate this study Cu-TiO$_2$ nano coating were coated through the electroplating technique onto a mild steel substrate utilizing an acid copper plating bath infused with dispersed nanoscale TiO$_2$. Three distinct sets of electroplating parameters—voltage, speed, and time—were employed during the process. The composition of both uncoated and coated specimens was determined using the Thermo Scientific Niton XL12 analyzer machine.

Voltage, Speed, and Time were considered as opposing factors for determining the optimal combination of control parameters using the Taguchi Technique and ANOVA analysis. Different tests were performed to assess the mechanical properties of coated specimens under the optimal combination of control parameters. The results of the tensile test reveal a decrease in stress values for electroplated specimens compared to those of normal mild steel specimens. The average surface roughness values of coated specimens were determined using the Mitatatyo. It is observed that the surface roughness increased on electroplated shot-peened specimens, while it was minimized on electroplated specimens.

Index Terms –Electroplating, Taguchi, Anova.

I. Introduction

Composite coating is an advanced scientific method that generates novel materials by combining different methods to produce metallic matrix composites. These coatings serve the purpose of imparting various functional properties, such as wear resistance, corrosion resistance, or oxidation resistance, to the plated surface.

The elaboration of these coating involves numerous and varied techniques which make it possible to obtain the wide range of coatings, each having very specific qualities and characteristics. Electroplating is one of the most technologically realizable and economically superior techniques for the production of metal matrix composites.

1.1 *Electroplating*

Electroplating is one of the strong coating technologies used to deposit the thin layerof coating metal over the base metal with the help of electric current. Corrosion resistance,coating adhesion inner tension, hardness, and abrasion resistance are the main properties ofelectro deposition process. Among these other properties can be varied by considering the process. parameters like voltage, temperature, time, agitation speed, components concentration, electrolyte pH value, electrolyte additives and current density. Compared to the all other techniques in the coating technology the Electroplating technology or Electroplating deposition could be versatile vasoconstrictor method. In Electroplating technology the deposition of the coating material in an exceedingly single step while not going any secondary treatment.

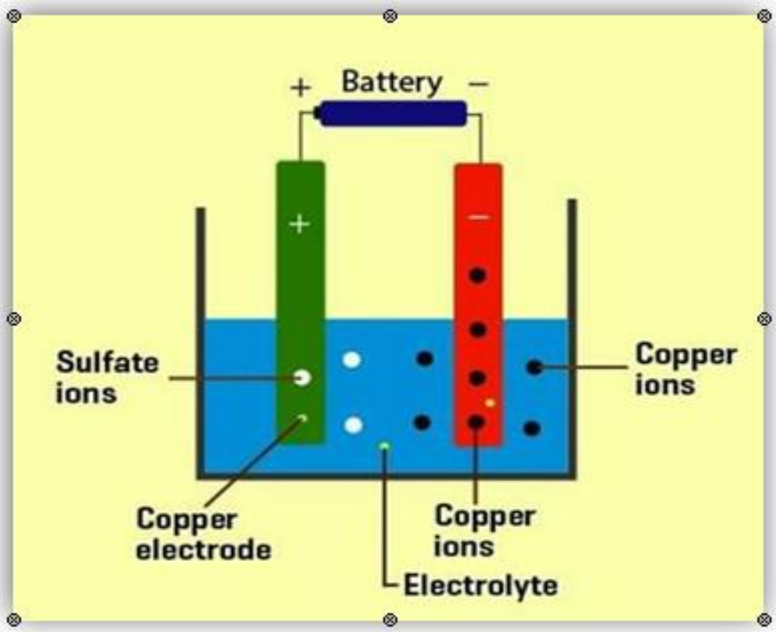
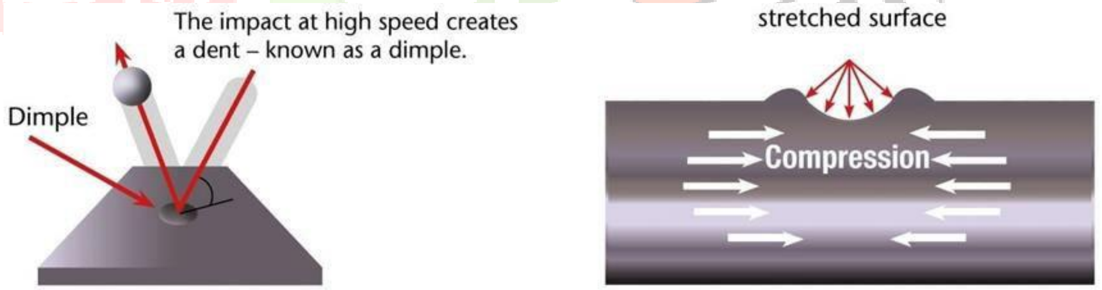


Fig.1 Electro Plating Process

The required electrolyte solution is filled in the container and the electrodes are immersed in the electrolyte solution. Before immersion of the electrodes the object is cleaned if the object is not cleaned, polished and degreased the deposition term may not be well adherent to the base metal. The both anode and cathode electrodes are connected to the DC power supply and the circuit is completed. Required amount of direct current (DC) is supplied to the electro deposition setup through the completed circuit, which causes the metal ions dissolved from the anode and entering in to electrolyte solution. Metal ions already present in the solution are provided by the dissolved salt, migrate towards the cathode(work piece) the dissolved electrons deposited on the base metal as pure metal again.

1.2 Shot Peening

Shot peening is a cold working process employed to induce a compressive residual stress layer and alter the mechanical properties of metals and composites. This process involves impacting a surface with shot, which are typically round metallic, glass, or ceramic particles, with enough force to cause plastic deformation.



Shotpeening Process

1.3 Optimization Techniques

The adoption of scientific methodologies for enhancing quality was becoming increasingly prevalent in industrial practice. The utilization of statistical techniques, particularly experimental design, has exerted significant influence.

1.3.1 Taguchi Method

Taguchi's experimental designs, known as orthogonal arrays, essentially represent incomplete factorials. Determining the number of parameters and levels, these elegant symmetrical designs are systematically applied throughout various works. In our study, we have focused on three plating parameters with three levels each. Based on these parameters and levels, we employed a L27 symmetrical array from the Taguchi approach for experimentation. The primary objective of this study is to enhance the microhardness of metallic component thin film coatings using the "larger is better" module.

1.3.2 ANOVA Method

ANOVA will be employed to assess the impact of the process parameters on the performance characteristic. The Taguchi method aims to reduce process variability through robust experimental design. Its overarching goal is to deliver high-quality products at low cost to manufacturers. Developed by Genichi Taguchi, this method involves designing experiments to investigate how various parameters influence the mean and variation of a process performance characteristic, which indicates the process's effectiveness.

The experimental design prescribed by Taguchi utilizes symmetrical arrays to organize the parameters affecting the process and the levels at which they should vary. Unlike testing every possible combination as in factorial designs, the Taguchi method tests sets of combinations. This enables the collection of essential data to determine which factors most significantly impact product quality with minimal experimentation, thereby saving time and resources. The Taguchi method is most effective when there is a moderate number of variables ranging from 3 to 50, few interactions among factors, and when only a minority of factors substantially contribute.

Nano particle composite coating on aluminium substrate by Electroplating process. The aluminium surface requires a specific pre-treatment for better adherence of coating. In light of this a thin zinc layer is coated on the aluminium substrate by electroless process [1]. The good quality on painted body is important to reduce repair cost and achieve customer satisfaction. In order to achieve the good quality, it is important to reduce the defect at the first process in painting process which is Electroplating process. [2]. The microscopic analysis of pulse electrodeposited Ni- AlN nanocomposite coatings using SEM and AFM techniques and their performance evaluation (mechanical and electrochemical) by employing nanoindentation and electrochemical methods [3]. Pure zinc coatings have been found ineffective., Zn/nano- TiO2 composite coatings with various contents of TiO2 nanoparticles (diameter size of 10 nm) were prepared on low-carbon steel by Electroplating technique. The deposition was carried out at different cathodic potentials ranging from −1600 mV to −2100 mV for different deposition times between 5-15 min [4]. Nano sized TiO2 particles were prepared by sol-gel method. TiO_2 nano particles were dispersed in zinc-nickel sulphate electrolyte and thin film of Zn-Ni-TiO_2 composite was generated by Electroplating on mild steel plates. [5]. Influence of the current regime on the Electroplating of zinc–ceria composite coatings obtained from a chloride based electrolyte, by using both direct and pulse current Electroplating techniques. [6]. the adsorption of surfactant CTAB on the SiC particles increases with increasing concentration of CTAB. The enhancement of adhesion between particles and the cathode increases the possibility of embedding larger particles. The effect of surfactant on the codeposition behaviors of SiC particles was explained through the concept of effective particle. [7]. Nanocrystalline materials exhibit very high strengths compared to conventional materials, but their thermal stability may be poor. Electroplating is one of the promising methods for obtaining dense nanomaterial's.[8]. Study Electroplating of $BaCO_3$ from aminecarboxylate stabilized- Ba $(HCO_3)_2$ baths, results in oriented crystallization when the bath conditions promote the decomposition of the Ba complex. The crystallites exhibit three-fold twinning (trilling) consequent to the evolution of the planes as planes of reflection. Pairs of trilling's are seen to grow about a four-sided polygon formed by the crystal faces whose centre is a point of inversion. [9]. nickel boron composite coatings can be obtained by electroplating nickel from a bath containing dispersed boron particles. This deposit heated to 300°C forms Ni-Ni3B composite. Further heating to 400°C converts it into Ni-Ni2B composite. Mechanical properties, corrosion resistance and wear resistance Ni-Ni2B composite are better than nickel or the electroless Ni-B composite in the as-deposited condition. The properties of heat-treated electroplated Ni-B composite are similar to that of heat-treated electroless Ni-B composites. [10].

II Objectives

Mild steel (as substrate) specimens coated with Cu-TiO2 Nano particles by Electroplating process and followed by Shot Peening and study the mechanical properties of coating specimens such as: Tensile property, Surface roughness and To study the influence of process parameters on Surface roughness of coated specimens by Taguchi method of orthogonal array and analysis of variance technique (ANOVA).

III Materials and methods

The Mild Steel [Low Carbon Steel] is the most common materials used in many industrial and commercial applications, advantages of MS price are low, availability of the material as well as good mechanical properties compared to the other materials.

The Mild Steel plate is used, it is composition of iron with some minimum percentage of Carbon, and the mild steel plate is initially tempered. This plate is also known as Plain Carbon Steel or low carbon steel.

Copper is a chemical element with the symbol Cu (from Latin: cuprum) and atomic number 29. It is a soft, malleable, and ductile metal with very high thermal and electrical conductivity. A freshly exposed surface of pure copper has a pinkish-orange colour. Copperis used as a conductor of heat and electricity, as a building material, and as a constituent of various metal alloys, such as sterling silver used in jewelry, cupronickel used to make marine hardware and coins, and constantan used in strain gauges and thermocouples for temperature measurement. Titanium dioxide, also known as titanium di oxide or titania, is the naturally occurring oxide of titanium, chemical formula TiO2. When used as a pigment, it is called titanium white, Pigment White 6 (PW6), or CI 77891. Titanium dioxide is one of the extensively studied transition metal dioxides with innumerable applications which includes the development of biosensor, electronic devices, and batteries. Titanium dioxide has been used for a century in a range of industrial and consumer products, including paints, coatings, adhesives, paper, plastics and rubber, printing inks, coated fabrics and textiles, as well as ceramics, floor coverings, roofing materials, cosmetics, toothpaste, soap, water treatment agents. Copper sulphate, , are the inorganic compounds with the chemical formula $CuSO_4 (H_2O)$ x, where x can range from 0 to 5. The pentahydrate (x = 5) is the most common form. Older names for this compound include blue vitriol, bluestone, vitriol of copper and Roman vitriol. Copper sulphate is blue in the colour. Composition of the copper sulphate is commonly about 98% of pure copper sulphate, 39.81% of copper and 60.19% of sulphate by mass. Ethylenediaminetetraacetic acid (EDTA) is an aminopolycarboxylic acid with the formula $[CH_2N(CH_2CO_2H)_2]_2$. This white, water-soluble solid is widely used to bind to iron and calcium ions. It binds these ions as a hexadentate ("six-toothed") chelating agent. EDTAis produced as several salts, notably disodium EDTA, sodium calcium edetate, and tetrasodium EDTA.

IV Experimentation

4.1Electroplating

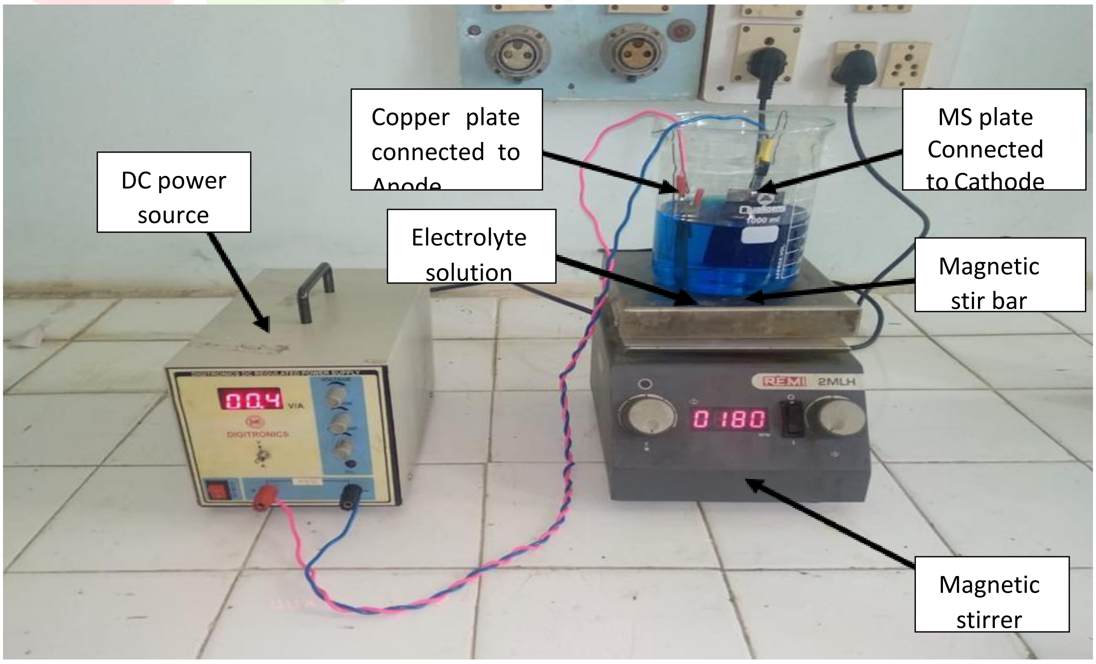


Fig 1. Electroplating Setup

Place electrolyte beaker on magnetic stirrer and put magnetic stir bar in the solution beaker which helps solution to rotate on setting the speed of magnetic stirrer at required rpm. Anode is connected to copper plate and Cathode is connected to steel plate of DC power source through wire and electrical clips to hold specimen in electrolyte solution. Bywearing the supply of current and speed of magnetic stirrer note the reading of speed and current supply on various gap of time process in a tabular column (Table 1). After each reading take out the steel plate carefully without touching the coated surface and let it dry. After completing the experiment specimens are taken to testing where these specimens aretested and results were found.

Table 1 List of parameters maintained in the Electroplating

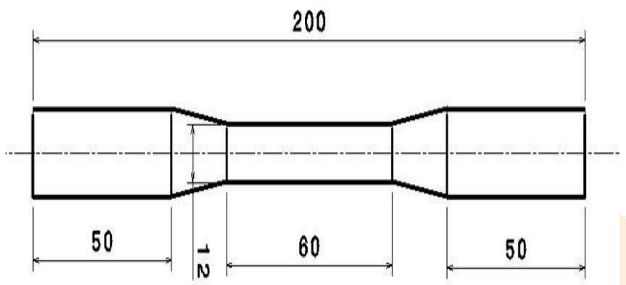
Experimental parameters for Electroplating specimens			
Trial No.	**Voltage (V)**	**Speed (rpm)**	**Time (Min)**
1	1	250	5
2	1	250	8
3	1	250	12
4	1	350	5
5	1	350	8
6	1	350	12
7	1	450	5
8	1	450	8
9	1	450	12
10	2	250	5
11	2	250	8
12	2	250	12
13	2	350	5
14	2	350	8
15	2	350	12
16	2	450	5
17	2	450	8
18	2	450	12
19	3	250	5
20	3	250	8
21	3	250	12
22	3	350	5
23	3	350	8
24	3	350	12
25	3	450	5
26	3	450	8
27	3	450	12
Experimental parameters for Electroplated-shot peened specimens			
Trial No.	**Voltage (V)**	**Speed (rpm)**	**Time (Sec)**
1	1	450	12
2	2	450	12
3	3	450	12

4.1.1Preparation of specimens

The two types of specimens used to conduct the mechanical test, mild steel round rod for conducting fatigue test and tensile test and mild steel plate is used to conduct micro hardness test, surface roughness tests and scanning electron microscope analysis. Initially cut the sheet metal and round specimen for the required shape and size. The round rod is machined for the required shape and size in the machine shop in the lathe machine.

The size of the specimen for Tensile specimen ASTM standard ASTM E 646-98 (Fig 2)

Clean the prepared specimens with the help of emery paper of small grain size for finishing purpose. After the finishing of the specimen wash the specimen with tap water and de-ionized water. Clean the moisture content with the help of clean cotton cloth.



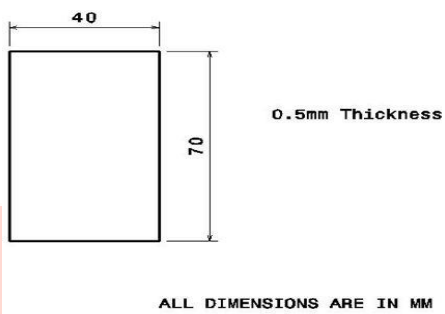


Fig. 2(a) Tensile specimen Fig. 2(b) Surface Roughness specimen

4.1.2 Preparation of Electrolyte Solution

In a glass beaker take 500 ml of distilled water. Take 2.5 grams of copper sulphate and 7 grams of EDTA (Ethylenediaminetetraacetic acid) and titanium oxide a side. Slowly by continuous stirring of distilled water mix the weighed copper sulphate, EDTA and titanium oxide one by one. Stir the solution till it completely dissolves. After complete desolation of crystals in distilled water check pH value of the solution. If the solution is not reached the required pH value add buffer solution drop by drop by stirring the solution till it reaches 6-7 pH value. After setting pH value correctly the solution will be ready for conducting experiment.

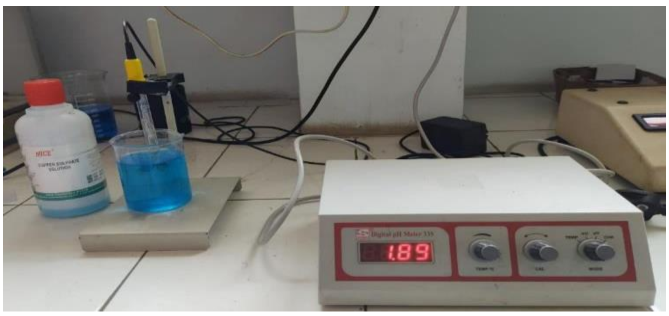


Fig 3(a) PH meter Fig. 3(b) Electrolyte solution

V RESULTS AND DISCUSSIONS

This experimental work was conducted to describe and compare the mechanical properties values such as tensile test values, and surface Roughness values of normal Mild Steel specimen, electroplated shot Peened specimen, and only shot. Peened specimen.

5.1 Composition Analysis by Analyser

With the help of Thermo Scientific NitonXL12 analyser machine the composition of all four types of specimens. This composition analysis is done before and after electroplating process and as well as before and after shot peening process.

Table 2. Composition of Coated and Uncoated specimen

Composition of all the metals								
Specimen type	**Trial no**	**Cu %**	**Fe %**	**Mn %**	**Ti %**	**S%**	**Si %**	**Co%**
Normal mildsteel plates specimen	-	-	98.40	0.305	-	-	0.384	-
Electroplated shot peened MS plate specimen	2	4.91	94.54	0.311	0.115	0.066	0.093	-
	14	1.99	96.68	0.310	0.102	0.040	0.145	-
	16	2.41	96.70	0.331	0.109	0.032	0.208	-
	24	11.66	87.36	0.273	0.122	0.028	0.293	0.167
Normal mild steel round bar specimen	-	0.083	98.08	0.396		0.045	0.515	-
Electroplated shot peened MS round bar specimen	31	23.17	75.81	0.334	0.165	0.046	0.402	-
	32	18.07	80.86	0.354	0.151	0.042	0.295	-
	33	25.36	73.65	0.367	0.158	0.039	0.386	0.125

5.2 Tensile Test Results

In this project work 18mm round rod is used to conduct to the tensile test to determine the yield stress, ultimate tensile stress, breaking stress, young's modulus and strain with help of the universal testing machine. Initial observation and formula are listed below

Initial dia = 12 mm

Initial area (A_o) = 113.1 mm^2 Initial gauge length = 50 mm Yield stress (σy) = F_y/A_o N/mm^2

Ultimate stress (σu) = F_{max}/A_o N/mm^2

Breaking stress (σb) = F_b/A_o N/mm^2

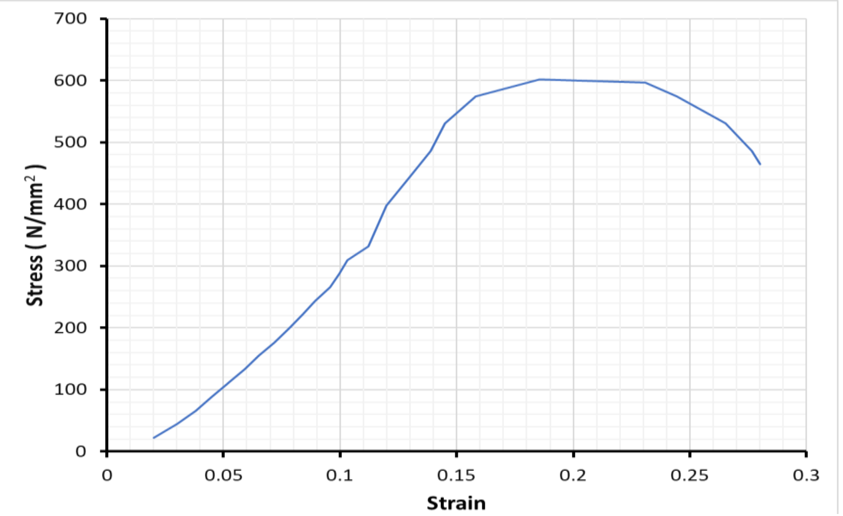


Fig. 4 Stress-Strain graph for Uncoated specimen

5.2.1 Uncoated Mild Steel specimen tensile test results

Yield point = 35000 N, yield stress (σy) = 35000/113.1 = 309.47 N/mm^2

Ultimate point = 68000 N, ultimate tensile stress (σu) = 68000/113.1 = 601.25 N/mm^2

Breaking point = 52500 N, breaking stress (σb) = 52500/113.1 = 464.20 N/mm^2

5.2.2 Electroplated specimen tensile test results

A) Voltage = 1, rpm = 250, time = 5 min

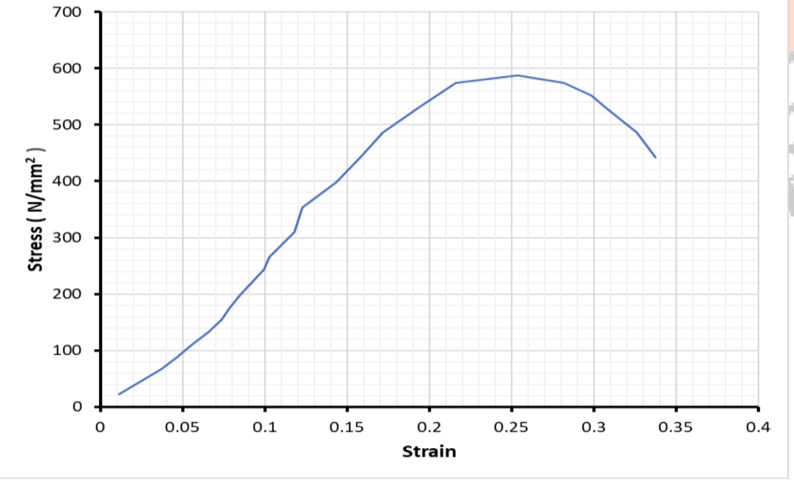


Fig. 5 Stress-Strain graph for Electroplated specimen 1

Yield point = 30000 N, yield stress (σy) = 30000/113.1 = 265.25 N/mm^2

Ultimate point = 66500 N, ultimate tensile stress (σu) = 66500/113.1 = 587.98 N/mm^2

Breaking point = 50000 N, breaking stress (σb) = 50000/113.1 = 442.09 N/mm^2

B) Voltage = 2, rpm = 3500, time = 8 min

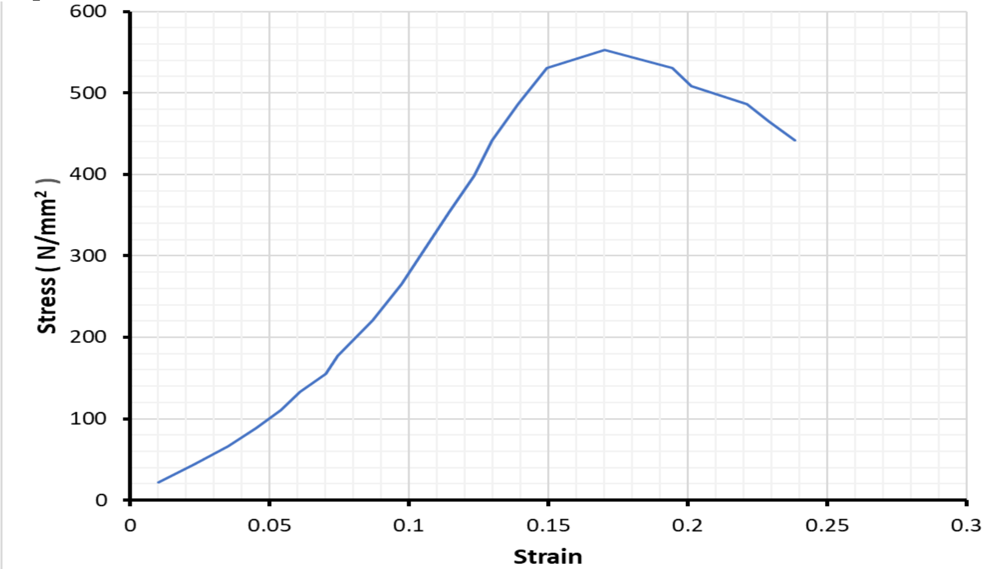


Fig. 6 Stress-Strain graph for Electroplated specimen 2

Yield point = 25000 N, yield stress (σy) = 25000/113.1 = 221.04N/mm^2

Ultimate point = 62500 N, ultimate tensile stress (σu) = 62500/113.1 = 552.61 N/mm^2

Breaking point = 50000 N, breaking stress (σb) = 50000/113.1 = 442.09 N/mm^2

***C)** Voltage = 3, rpm = 450, time = 12 min*

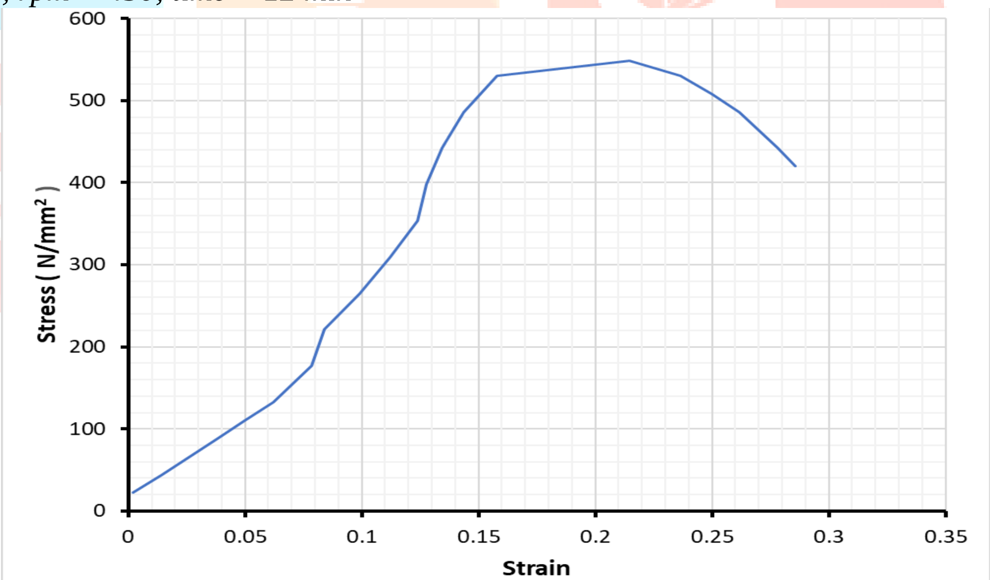


Fig. 7 Stress-Strain graph for Electroplated specimen 3

Yield point = 25000 N, yield stress (σy) = 25000/113.1 = 221.04 N/mm^2

Ultimate point = 62000 N, ultimate tensile stress (σu) = 62000/113.1 = 548.19 N/mm^2

Breaking point = 47500 N, breaking stress (σb) = 47500/113.1 = 419.98 N/mm2

5.2.3 Electroplated – Shot peened specimen tensile test results

Voltage = 1, rpm = 250, time = 5 min

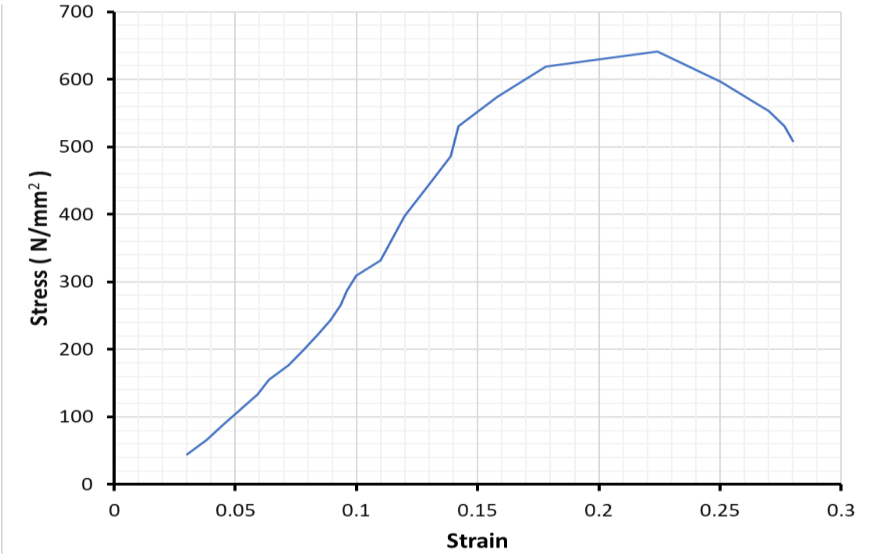


Fig. 8 Stress-Strain graph for Electroplated– Shot peened specimen

5.3 Surface Roughness Test Results

Table 3 Surface Roughness results

Surface roughness results							
Specimen type	Trial no.	Control Parameters			Tested surface roughness value in µm		
		VOLLTAGE (V)	SPEED (rpm)	TIME (min)	Average roughness	Maximum height	
					Ra	Ry	Rz
Electroplated specimens	1	1	250	5	1.16	7.43	7.43
	2	1	250	8	1.19	7.02	7.02
	3	1	250	12	1.40	9.10	9.10
	4	1	350	5	0.71	5.12	5.12
	5	1	350	8	0.96	8.29	8.29
	6	1	350	12	1.71	10.75	10.75
	7	1	450	5	1.31	9.92	9.92
	8	1	450	8	0.73	13.62	13.62
	9	1	450	12	0.75	5.90	5.90
	10	2	250	5	0.97	6.04	6.04
	11	2	250	8	0.67	8.14	8.14
	12	2	250	12	1.86	16.05	16.05
	13	2	350	5	1.04	5.62	5.62
	14	2	350	8	1.51	10.06	10.06
	15	2	350	12	1.37	8.71	8.71
	16	2	450	5	1.74	15.14	15.14
	17	2	450	8	0.91	6.60	6.60
	18	2	450	12	0.89	6.22	6.22
	19	3	250	5	0.91	5.99	5.99
	20	3	250	8	1.12	7.70	7.70
	21	3	250	12	2.04	15.11	15.11
	22	3	350	5	1.44	8.92	8.92
	23	3	350	8	067	5.76	5.76
	24	3	350	12	1.61	8.76	8.76
	25	3	450	5	1.01	6.01	6.01

	26	3	450	8	0.80	8.05	8.05
	27	3	450	12	1.74	8.92	8.92
Normal mild steel specimen	-	-	-	-	1.6	12.3	12.3
Electro plated – Shot peened	31	1	250	5	5.4	17.5	17.5
	32	2	350	8	6.3	18.3	18.3
	33	3	450	12	8.1	18.9	18.9

This test is conducted to known the surface characteristics of different cases specimens with different parameters such as voltage, agitation speed and electroplating time. This test is carried out on the Tally surf gauge, the Ra, Ry and Rz values are noted down.

On seeing the values of surface roughness and maximum height values (Ry and Rz) we found out that surface roughness varies in specimens. By seeing the above table, we conclude that shot peened electroplated specimens and specimen 6,8,12,14,16 and 21 having higher surface roughness value and deposition of copper is also high respectively compare to the other specimen. The reaming all specimen having normal surface roughnessvalue and deposition of copper.

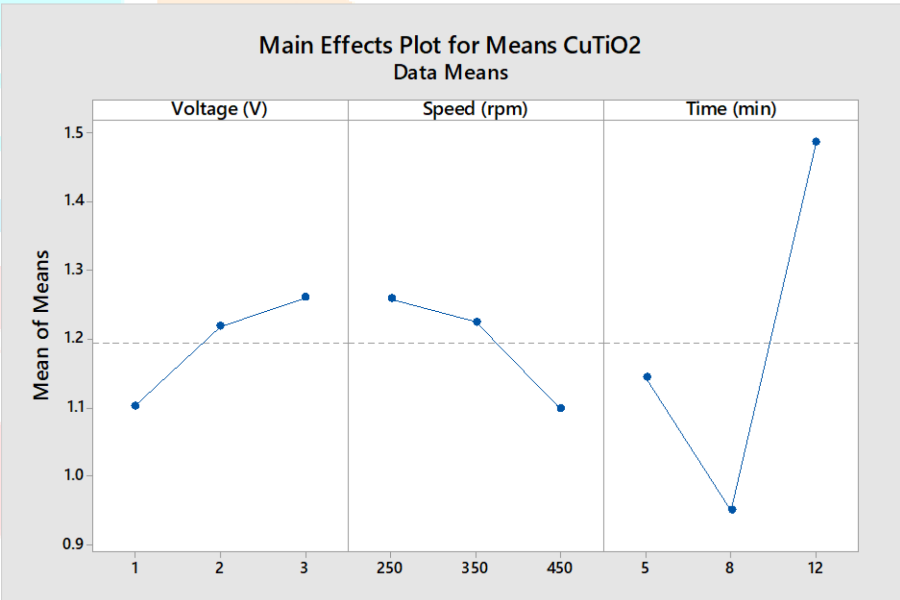


Fig 9. Main effects plots for Means

An interaction plot in the context of $CuTiO_2$ coating on given parameters for surface roughness (as indicated by Fig. 9) It visualizes the interaction effects between two or more parameters on the surface roughness. Interaction plots help understand how changes in one parameter may influence the surface roughness differently depending on the level of another parameter.

A response table for means typically presents the average or mean values of responses (possibly surface roughness, coating thickness, or other relevant metrics) obtained from experiments or observations involving $CuTiO_2$ coating which are shown in Table 5.6. As per the observations it can be found that the time impacts more.

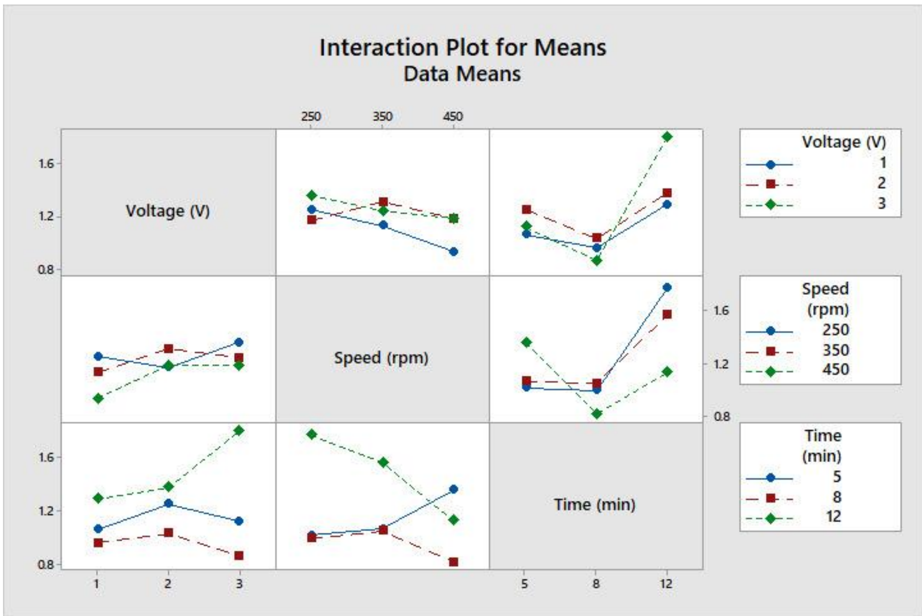


Fig 10 Interaction plot for the $CuTiO_2$ coating on given parameters for surface roughness

Table 4. Mean effects of parameters on Surface Roughness

Level	Voltage (V)	Speed (rpm)	Time (min)
1	1.1022	1.2578	1.1433
2	1.2178	1.2244	0.9511
3	1.2600	1.0978	1.4856
Delta	0.1578	0.1600	0.5344
Rank	3	2	1

A residual plot, shown in Fig. 11, on given parameters for surface roughness is a graphical representation that shows the differences between the observed values (actual experimental results) and the predicted or expected values obtained from a statistical model or analysis. A random and even distribution of residuals around zero implies a good model fit, reflecting an accurate representation of the relationship between parameters and surface roughness.

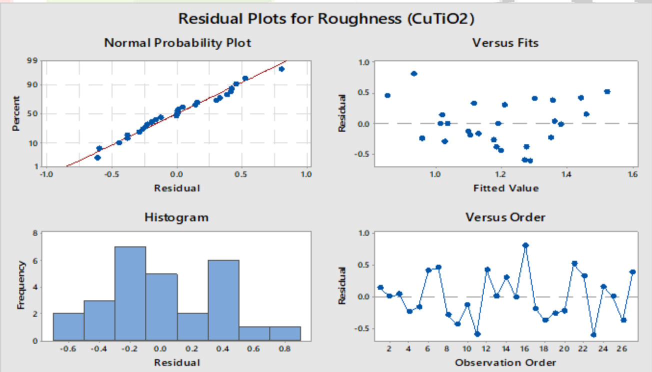


Fig. 11: Residual plot for the $CuTiO_2$ coating on given parameters for surface roughness

5.4 Energy Dispersive X-Ray Analysis (EDX) Results

5.4.1 Normal mild steel specimen EDX results

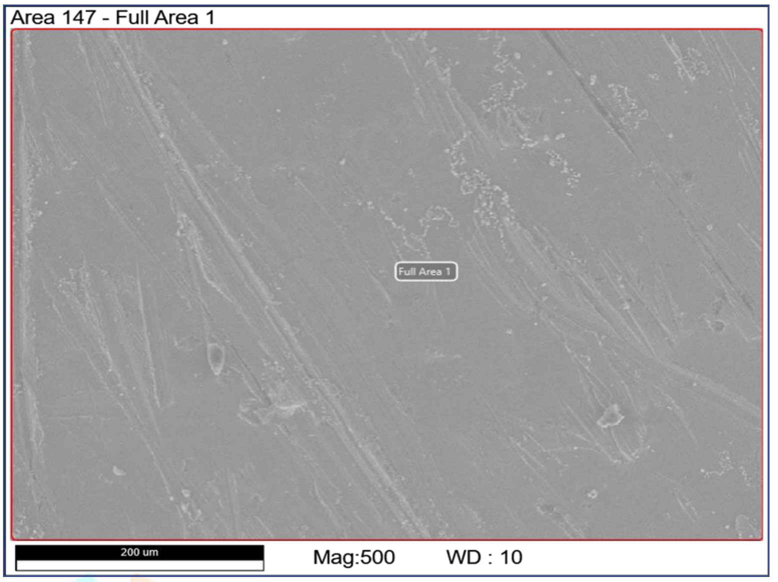


Fig 12 (a) Normal mild steel specimen EDX image

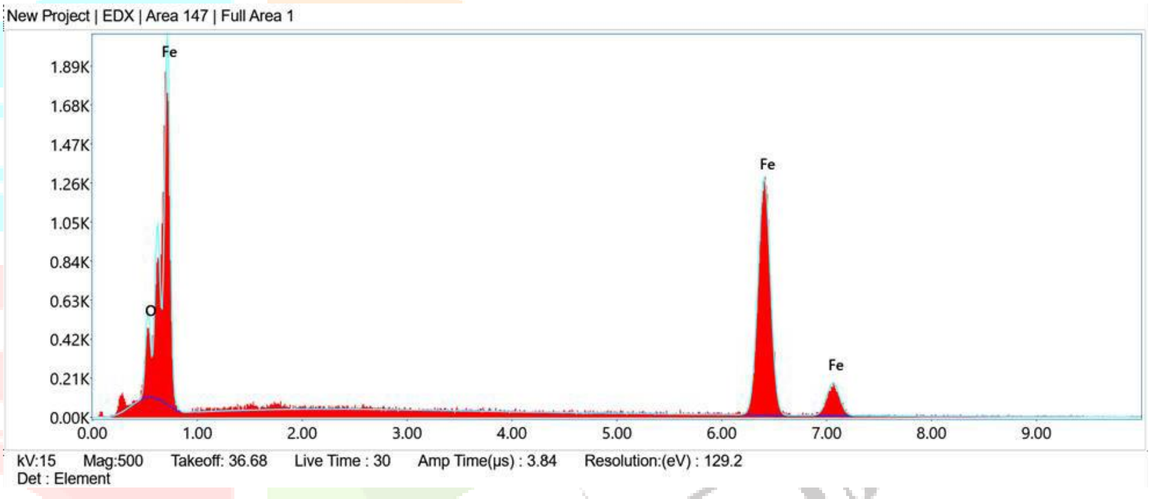


Fig 12 (b) Normal mild steel specimen EDX results

Table 5 eZAF Quant Results of Normal mild steel specimen

eZAF Quant Result - Analysis Uncertainty: 4.99 %			
Element	**Weight %**	**Atomic %**	**Error %**
O K	4.4	13.8	11.2
Fe K	95.6	86.2	3.3

5.4.2 Copper specimen EDX results

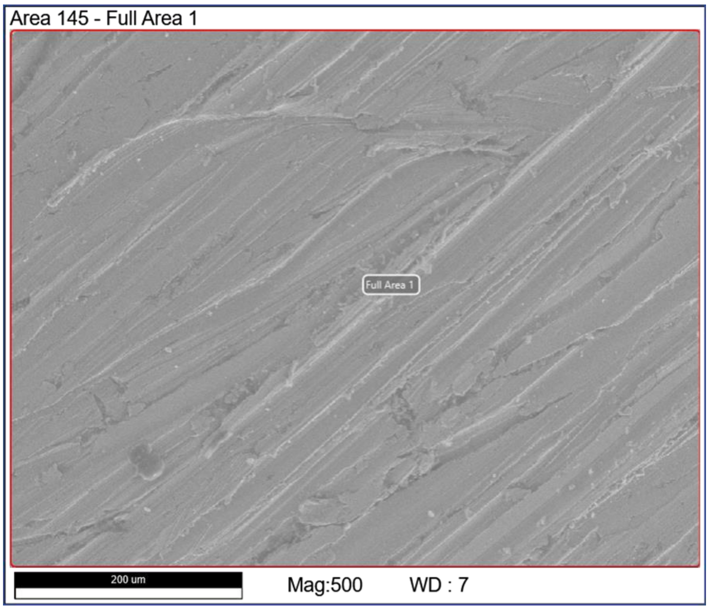


Fig 13 (a) Copper specimen EDX results Table 8.15

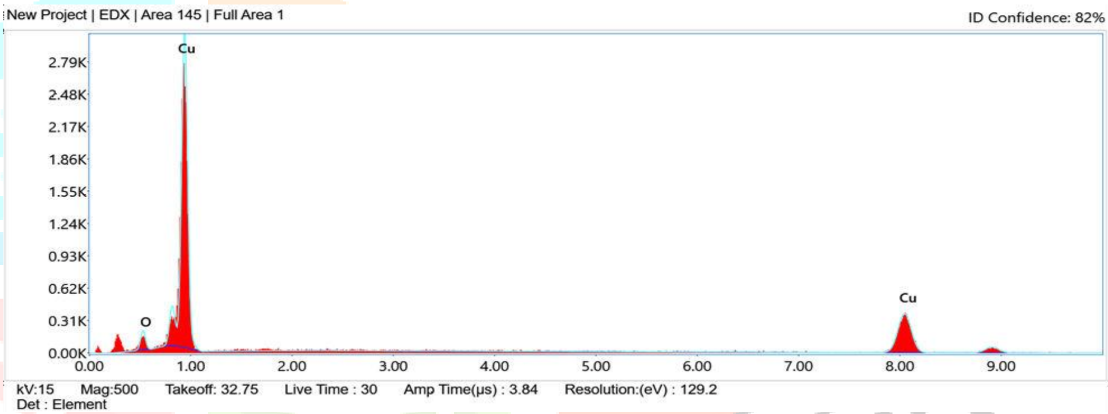


Fig. 13(b) Copper specimen EDX results

Table 6 eZAF Quant Results of copper specimen

eZAF Quant Result - Analysis Uncertainty: 6.36 %			
Element	**Weight %**	**Atomic %**	**Error %**
O K	3.7	13.3	13.4
Cu K	96.3	86.7	4.9

5.4.3 Electroplated specimen EDX results

***A)** at 20um*

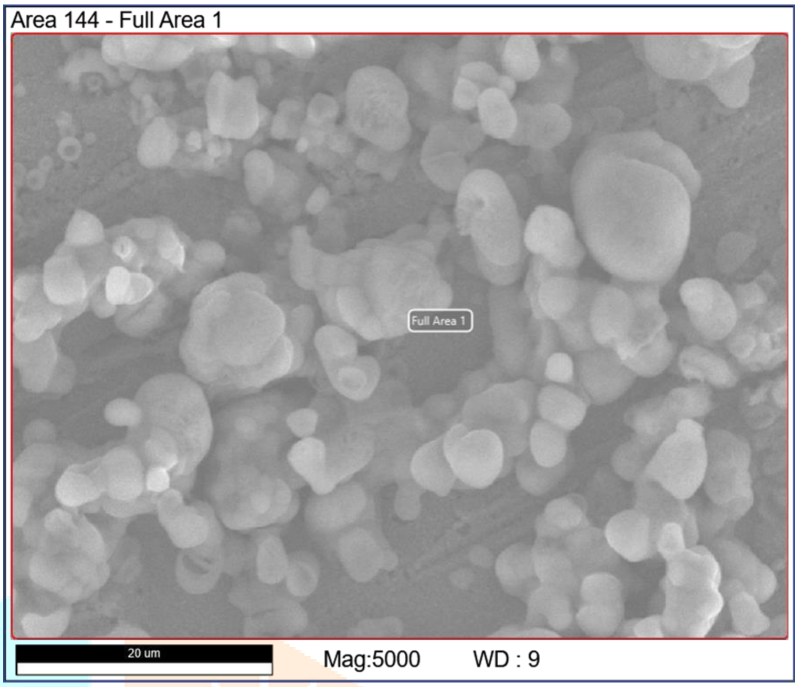


Fig. 14 (a) Electroplated specimen

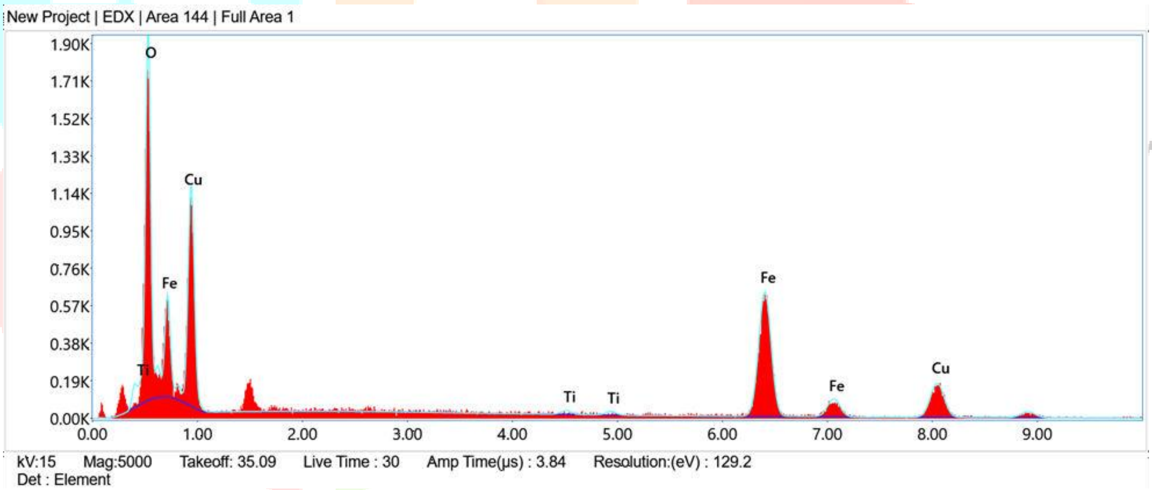


Fig. 14(b) Electroplated specimen EDX image

Table 7 eZAF Quant Results of Electroplated specimen

eZAF Quant Result - Analysis Uncertainty: 99.00 %			
Eleme nt	**Weight %**	**Atomic %**	**Error %**
O K	19.8	47.4	8.8
Ti K	0.6	0.5	31.4
Fe K	48.5	33.3	4.1
Cu K	31.1	18.8	6.8

VI CONCLUSION

The following conclusions are established from the experimental and analysis studies on Cu-TiO2 Nano composite coating on Mild Steel substrate by electroplating with and without shot peening of the specimens. Tensile test results of electroplated shot peened specimen have shown higher values of ultimate and yield point stresses compared to other types of specimens. In case of electroplated specimens (for control parameters combination of V=1, S=250, T=5), the maximum ultimate tensile stress is 587.98 N/mm^2 whereas in case of electroplated shot peened specimens, this value is 641.04 N/mm^2 for the same control parameters combination.

The average surface roughness value of electroplated Shot Peened specimens is higher compared to electroplated specimens.

II. ACKNOWLEDGMENT

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