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Microhardness and Taguchi Analysis of $CuTiO_2$ Nanocoating on Mild steel

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ABSTRACT
Cu-TiO_2 nanocomposites were fabricated through electroplating onto a Mild Steel substrate using an acid copper plating solution infused with dispersed nanoscale TiO_2 particles. Three distinct sets of electroplating parameters, including voltage, speed, and time, were employed in the process.. Micro hardness testing was performed to assess the hardness of the electrodeposited samples with and without shot peening. The results of the micro hardness test served as the primary data, while electroplating control parameters such as Voltage, Speed, and Time were considered as opposing factors in determining the optimal combination of control parameters using the Taguchi Technique and ANOVA analysis.
Keywords – Electroplating, Microhardness, Taguchi ANOVA

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I. INTRODUCTION

Composite coating represents an advanced scientific method capable of generating novel materials. Various techniques exist for creating composite materials with a metallic matrix. The primary objective of composite coatings is to impart diverse functional properties, including wear resistance, corrosion resistance, or oxidation resistance, to the plated surface. Coatings are frequently defined as a process applied to the surface of a substrate or object to improve surface properties and protect the components. Metal coating involves the application of a film that alters the surface properties of the workpiece to resemble those of the metal being applied. Electroplating stands out as a robust coating technology utilized to apply a thin layer of coating metal onto a base metal using electric current. Key properties of electro-deposition include corrosion resistance, coating adhesion, internal tension, hardness, and abrasion resistance. These properties can be tailored by adjusting process parameters such as voltage, temperature, time, agitation speed, component concentration, electrolyte pH value, electrolyte additives, and current density. Electroplating technology emerges as a versatile and efficient method in the realm of coating techniques. It allows for the deposition of coating material in a single step without requiring additional secondary treatments.

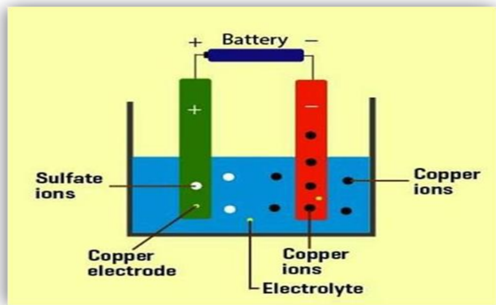


Fig 1 Electroplating process

In Electroplating technology (Fig1.1), the essential components include the positive and negative terminals, which are linked to an adjustable direct current. This serves as the power source, necessitating a low voltage direct current power supply.

The positive terminal of the power supply is attached to the anode, typically made of the deposition material. On the other hand, the negative terminal of the power supply is linked to the cathode, which is the component intended for metal deposition. The container housing the dissolved metal-bearing salts is referred to as the electrolyte solution. The container is filled with the necessary electrolyte solution, and the electrodes are submerged in it. Prior to immersing the electrodes, the object undergoes cleaning, polishing, and degreasing. Failure to clean the object adequately may result in poor adhesion of the deposited layer to the base metal. Both the anode and cathode electrodes are connected to the DC power supply, completing the circuit. A specific amount of direct current (DC) is supplied to the electro-deposition setup via the completed circuit, causing metal ions to dissolve from the anode and enter the electrolyte solution. A detailed analysis was conducted to examine the impact of twin morphology and compressive residual stress on fatigue properties. It was observed that shot peening gradually enhances the fatigue properties of specimens, particularly under low external stress and test temperatures. [1]. Enhancing fatigue life often involves employing various surface treatments and adjusting parameters that influence fatigue strength. Among the most widely utilized methods to improve fatigue life is surface treatment of the material [2]. Application of shot peening in both automotive and aircraft industries, focusing particularly on its effects on case-hardened steel. The study investigates how shot peening influences factors such as residual stress, microstructure, and surface roughness. The primary objective is to demonstrate the correlation between various measurement techniques and results for specific shot peening parameters. [3] Fatigue tests were carried out on specimens fabricated from S355 steel under bending block loading with a constant mean loading value. The experimental results revealed a notable difference in the fatigue life of the material, depending on whether the mean load value increased or decreased in the subsequent loading block sequence.[4] Copper electroplating stands as a widely recognized and extensively utilized industrial surface finishing method. The aim of the study was to assess two existing copper plating techniques and their effects on current density distribution and other pertinent plating parameters.[5] Industrial waste composed of low carbon steel, coated with electroplated copper, serves to enhance mechanical properties. The study concluded that the most favorable electrochemical performance was achieved with a current density of 7.5 A/cm^2. It was observed that the relationship between the anodic area and the cathodic area in the coating matrix increased proportionally with the utilization of current density in the electro-deposition process.[6] The Taguchi method employs a statistical approach to enhance the quality of manufacturing components. The study concluded that this method can be applied to analyze various types of problems. It provides a straightforward, systematic, and efficient methodology for optimizing process parameters based on experimental results.[7] The properties that primarily affect the electro-codeposition process include current density, concentration of ceramic particles in the plating bath, pH level, and bath temperature.[8] Shot peening, an effective mechanical surface treatment process, has long been considered to enhance residual stress by impacting shots onto the workpiece surface, thereby maximizing fatigue strength. This paper focuses on studies predicting various material responses and proposing optimization methods based on mechanics to enhance the fatigue strength of different materials. [10] This study explores various methods of surface treatment processes for metals, particularly emphasizing AWJ peening for enhancing residual stress and mechanical properties. The methodology highlights the influence of abrasive size and treatment pressure on the results. The residual stress of AISI304 ranged from 165 to over 460 MPa. [11] The higher compressive stress obtained by shot peening helps to increases the longer fatigue life.[13] A fatigue damage summation method is employed, incorporating two variables: mean stress and overloads. This method is tested on mild steel specimens subjected to complex load histories within the intermediate to long life range [14].

II MATERIALS AND METHODS

Mild Steel

Mild Steel (also known as Low Carbon Steel) is widely utilized in numerous industrial and commercial applications due to its affordability, widespread availability, and favorable mechanical properties compared to other materials. Comprising primarily of iron with a minimum percentage of carbon, Mild Steel plates are initially tempered.

They are also referred to as Plain Carbon Steel or low carbon steel.

Copper

Copper, symbolized as Cu (from Latin: cuprum) with atomic number 29, is a soft, malleable, and ductile metal renowned for its exceptionally high thermal and electrical conductivity. Pure copper exhibits a pinkish-orange hue on its freshly exposed surface. It finds extensive application as a conductor of heat and electricity, as well as a building material. Additionally, copper serves as a vital component in various metal alloys, including sterling silver for jewelry, cupronickel for marine hardware and coins, and constantan for strain gauges and thermocouples used in temperature measurement.

Titanium Dioxide

Titanium dioxide, also known as titania, is the naturally occurring oxide of titanium, with the chemical formula TiO_2. When utilized as a pigment, it is referred to as titanium white, Pigment White 6 (PW6), or CI 77891. This compound is extensively studied among transition metal dioxides and finds numerous applications, including: development of biosensor, electronic devices, and batteries. For a century, titanium dioxide has been employed across various industrial and consumer products, spanning paints, coatings, adhesives, paper, plastics, rubber, printing inks, coated fabrics, textiles, and ceramics floor coverings, roofing materials, cosmetics, toothpaste, soap, water treatment agents, etc

COPPER SULPHATE

Copper sulphate, also known as 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.

III. EXPERIMENTATION

Electroplating

Electroplating is the procedure conducted to plating the copper on the mild steel specimen [low carbon steel]. Electroplating creates a sacrificial coating on the specimen that avoided the underlying steel from rust. This is the one surface treatment procedure to improve the mechanical property.

Preparation of Electrolyte Solution

In a glass beaker, pour 500 ml of distilled water. Add 2.5 grams of copper sulfate and 7 grams of EDTA (Ethylenediaminetetraacetic acid) to the distilled water while continuously stirring. Next, introduce titanium oxide into the solution. Continue stirring until all components completely dissolve. After the crystals are fully dissolved, check the pH value of the solution. If the pH is not within the desired range, slowly add buffer solution drop by drop while stirring until the pH reaches between 6 and 7. After setting pH value correctly the solution will be ready for conducting experiment.

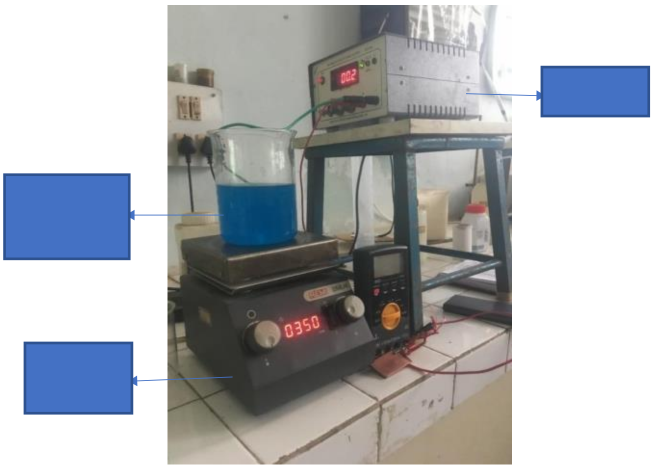


Fig.2 Experimental setup

Place the electrolyte beaker on a magnetic stirrer and insert a magnetic stir bar into the solution to facilitate rotation. Set the speed of the magnetic stirrer to the required RPM. Connect the anode to a copper plate and the cathode to a steel plate of the DC power source using wires and electrical clips to hold the specimen in the electrolyte solution. Record the readings of the speed and current supply at various intervals of time in a tabular column (Table 1) while adjusting the current supply and speed of the magnetic stirrer. After each reading, carefully remove the steel plate without touching the coated surface and allow it to dry. Once the experiment is complete, take the specimens to testing where they are analyzed, and results are obtained.

Table1. List of parameters maintained in the Electroplating

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

Micro Hardness Test

It is a mechanical test for determining material properties which are used in engineering design, analysis of structures and materials development. The principal purpose of the hardness test is to determine the suitability of a material for a given application, or the particular treatment to which the material has been subjected. The ease with which the hardness test can be made has made it the most common method of inspection for metals and alloys.

Micro hardness of the material is determined by the depth or area of the indentation. The indentation is obtained during load applied on the specimen. Elastic defamation devolved in specimen when the specimen loaded during the testing. The applied load caused the indentation and this indentation is directly proportional to the plastic deformation. The energy absorbed during the indentation is used to produce the plastic deformation and rest of energy increases the surface free energy.

Fig.3**Micro hardness machine**

IV. RESULT AND DISCUSSION

Sl. No.	V	S	T	HV
1	1	250	5	181.15
2	1	250	8	260.35
3	1	250	12	287.51
4	1	350	5	240.01
5	1	350	8	251.11
6	1	350	12	267.55
7	1	450	5	219.37
8	1	450	8	222.16
9	1	450	12	230.63
10	2	250	5	290.82
11	2	250	8	298.18
12	2	250	12	309.91
13	2	350	5	267.73
14	2	350	8	271.81
15	2	350	12	279.14
16	2	450	5	295..23
17	2	450	8	304.23
18	2	450	12	311.61
19	3	250	5	274.50
20	3	250	8	276.71
21	3	250	12	281.13
22	3	350	5	300.36
23	3	350	8	304.61
24	3	350	12	309.10
25	3	450	5	276.61
26	3	450	8	281.21
27	3	450	12	284.92

Micro Hardness Test Results

Table 2 Microhardness Test Results

Sl. No.	V	S	T	HV
31	1	450	12	301.22
32	2	450	12	292.50
33	3	450	12	230.83
Normal mild steel specimen				133.91

According to all the above results, the micro hardness value of the normal mild specimen is less compared to the all the other cases. The specimen 18 having the higher micro hardness value the electro plated shot peened case and specimen 31 in the shot peened electro plated case having the higher micro hardness value.

Analysis of Taguchi and Analysis of Variance (Anova)

Analysis of Taguchi for L27

Mean S/N ratio values for parameters on Micro Hardness

Table 3 Mean S/N ratio values for parameters on Micro Hardness

Level	V	S	T
1	47.53	48.65	48.22
2	49.30	48.82	48.73
3	49.17	48.54	49.05
Delta	1.76	0.83	0.27
Rank	1	3	2

Table 4 Mean effects of parameters on Micro Hardness

Level	V	S	T
1	240.0	273.4	260.6
2	292.1	276.8	274.5
3	287.7	269.5	284.6
Delta	52.1	7.3	24.0
Rank	1	3	2

The significance of S/N ratio and interaction mean plot of micro hardness vs voltage, speed and time is as shown in above table and graph. The above displayed results include the delta value, which is the difference between the highest S/N ratio and lowest S/N ratio values.

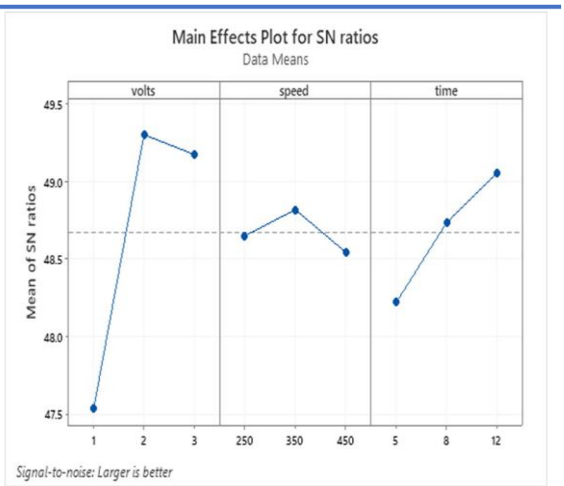


Fig 4 Main effects plot for SN ratios

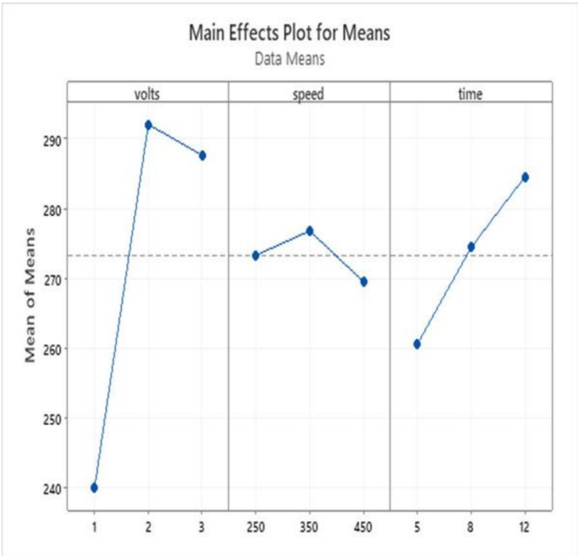


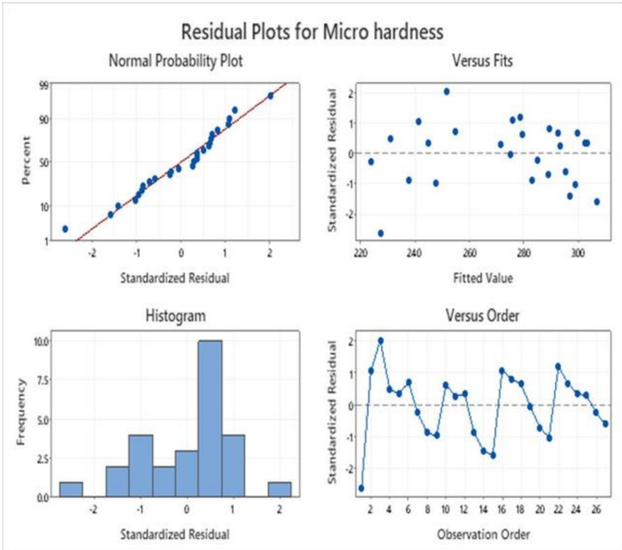
Fig. 5 Main effects plot for means

ANOVA

Table 5 General liner analysis for L27

Sl N	Df	Adj SS	Adj MS	F	P
V	2	15024.3	7512.1	17.7	0.00
S	2	238.4	119.2	0.28	0.75
T	2	2605.1	1302.6	3.07	0.06
E	20	8488.7	424.4		
T	26	26356.5			

Fig 6 **Residual plot for L27**



V. CONCLUSION

The following conclusions are established from the experimental and analysis studies on Cu-TiO_2 Nano composite coating on Mild Steel substrate by electroplating with and without shot peening of the specimens.

The results of Micro Hardness test of all specimen types, it is observed that the electroplated specimen (trial no. 18) has the highest Micro Hardness value 311.61 whereas the normal Mild steel specimen has corresponding value of 133.91.

Orthogonal arrays L27 of Taguchi approach is applied to the experimental trials of Micro Hardness test with least number of experiments to be accomplished to know the influence of process parameters such as voltage, speed and time on Electroplating process. Taguchi approach results gave good experimental response and experimental results also subjected to Analysis of Variance (ANOVA) technique.

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