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MICROSTRUCTURE CHARACTERISATION, HARDNESS, AND CORROSION BEHAVIOUR OF NICKEL-SILICON CARBIDE ELECTRODEPOSITION COATING WITH THE INFLUENCE OF ULTRASONIC AGITATION

This study is about the electrodeposition of Ni-SiC composite coatings at various ultrasonic agitation temperatures (50, 55, and 60°C). The coatings were characterised by a scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). Vickers microhardness test and immersion test were used to determine the hardness and corrosion behaviour of the composite coating respectively. The immersion test was conducted in 0.5M sulphuric acid to analyse the coating corrosion rate. The results demonstrate that increasing the ultrasonic agitation temperature increase the corrosion resistance of the composite coating. Furthermore, the hardness of the Ni-SiC shows higher hardness values at the highest agitation temperature, 60°C. This shows that Ni-SiC composite coating sample S.60 had the best hardness and corrosion behaviour.

Keywords: Nickel electrodeposition; Corrosion; Ni-SiC; Hardness; Cermet

1. Introduction

Carbon steel is known as one of the most often popular materials in manufacturing due to its outstanding mechanical, and aesthetic capabilities. Corrosion can be define as the breakdown of a substance due to chemical assaults in its environment; corrosion itself refers to any method involving the deterioration or breakdown of metallic materials [1]. By applying coatings, the substrate surface qualities such as mechanical properties, permeability, lubrication, adhesion, corrosion resistance, wear resistance, and resistance to abrasion will improve [2].

Nickel was frequently being applied as a protective substance in numerous types of metallic composites. This is due to its beneficial properties, such as resistance to corrosion, chemical stability and high ductility [3]. Ni-based alloy coatings are preferable compares to Co-based alloy coating because of their superior anti-wear qualities and lower prices. Electrodeposition method has a capability on producing a higher quality composite coating: the nickel silicon carbide, Ni-SiC [4]. It was found that, the corrosion resistance of this type of coatings was influences by the carbide content [5], organic additive [6] and alloying element of the substrate [7].

In electrodeposition, an electrical current is passing through metals or alloys for producing a thick, homogeneous coating with great adhesion to a substrate [8]. The ultrasonic agitation has been utilized to enhance deposits, current efficiency, and material dispersion during metal electrodeposition [9]. The resultant composites' microstructure, phase structure, surface morphology, and other aspects are all influenced by the ultrasonic temperatures [10].

Thus, in this study, the coating that consists of nickel (Ni) and silicon carbide (SiC) was deposited using the electrodeposition methods for observing the effect of ultrasonic agitation on the Ni-SiC coating microstructure as well as its corrosion and hardness behaviour.

2. Experimental methods

The materials used as substrate was low carbon steel (1.2×1.2×0.8 cm), graphite rods and electrolyte that contains nickel sulphate ($NiSO_46H_20$), nickel chloride ($NiCl_26H_20$), and boric acid (H_3BO_3) and 20 g/l of silicon carbide (SiC) particles. The low-carbon steels were cut, ground, polished,

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and cold mounting before electrodeposition. The electrolyte was agitated ultrasonically at 50℃, 55℃, and 60℃ before the electrodeposition process.

2.1. Electrodeposition process

The low-carbon steel and graphite rod acted as a cathode and anode during the electrodeposition with a current density of 0.5 A/cm^2. In this experiment, the stirring speed and deposition time was fixed at 160 rpm and 90 minutes, respectively. Remove the cathode from the beaker after completing the electrodeposition process at about 90 minutes and rinse it with ionized water.

2.2. Sample morphology and microstructure analysis

SEM was used to examine the surface morphology of all coated specimens. Energy Dispersive Spectroscopy (EDS) coupled with a scanning electron microscope (SEM) was used to observed the elemental composition of the coatings.

2.3. Microhardness analysis

The Vickers microhardness measurement was used to determine the sample resistance to permanent deformation when applied force. A 100 g load was applied to the specimen's surface for 10 s by diamond indenter. Ten indentations were taken for each coating. The average of the calculated values was used as the microhardness value.

2.4. Corrosion analysis

The corrosion analysis on each deposited specimen was carried out using an immersion test. 0.5 M of the sulphuric acid solution is used to carry out the immersion test. The immersion process was done for 21 days and monitored every four days. The weight of the deposited specimen is recorded before and after the immersion test. The corrosion rate on the exposed surface is determined by using this equation:

$$Corrosion\ Rate,\ CR\ (\text{mm/year}) = 87.6\,W/DAT \qquad (1)$$

where

W – weight loss (mg),
D – density of the sample (g/cm^3),
A – area of the sample (cm^2),
T – time of exposure (hours).

3. Results and discussion

The test and analysis have been carried out on the coated surface's microstructure characteristics, corrosion behaviour, and hardness. The results were analysed and tabulated in figures, tables, and graphs for better interpretation.

3.1. Deposition potential difference analysis

The electrodeposition potential difference is a method to determine the suitable current density for the process. Fig. 1 shows the potential difference graph analysis of voltage versus current.

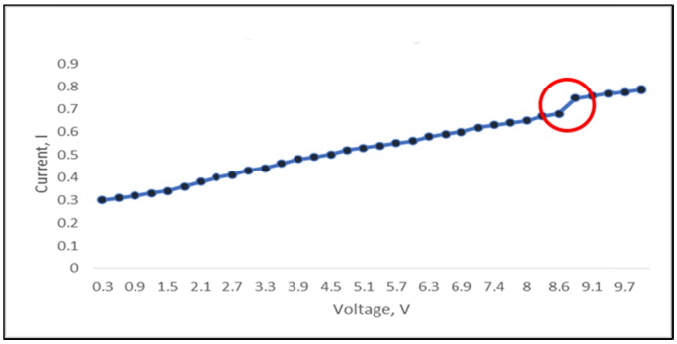

Fig. 1. Potential difference analysis graph

The increased current of 0.75 A at 8.9 V suggests that electrons are subjected to a push when the force of the chemical reaction leads to a higher potential, as seen in the sudden rise from 0.68A. This rise in current was selected from the analysis to identify the necessary current density for the electrodeposition process [11]. The current density is measured using Eq. (2):

$$Current\ Density,\ C_d = \frac{Current,\ I\ (\text{A})}{Surface\ area\ of\ the\ specimen\ (\text{cm}^2)} \qquad (2)$$

3.2. Surface morphology and microstructure analysis

Fig. 2 shows the magnified deposited surface on the specimen using SEM. It is shown that when the temperature of ultrasonic agitation increases, the deposition on the specimen also increases [12]. The application of higher temperature, as demonstrated in S60, enhances the movement of reactive components and results in improved deposition rate through improved agitation. The use of a higher temperature on the nickel silicon carbide solution before electrodeposition resulted in a noticeable increase in nodule size. The microstructure of the coating is influenced by the temperature-induced kinetic changes.

3.3. Element composition analysis

The element composition analysis uses Energy Dispersive Spectroscopy (EDS) through Scanning Electron Microscopy

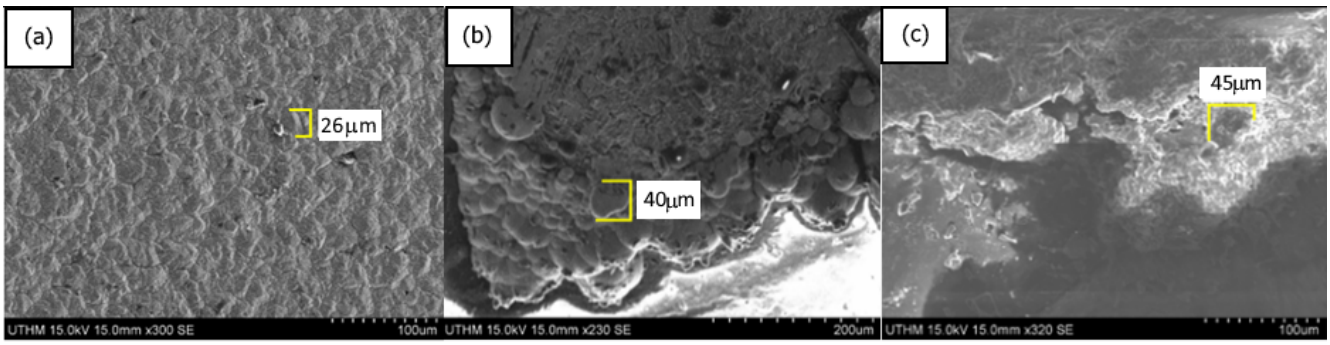

Fig. 2. SEM images of the coating at different temperatures a) 50°C, b) 55°C and c) 60°C

(SEM). The study illustrates the composition of each element in the nickel silicon carbide solution on the specimen after the deposition process. The EDS analysis results are displayed in TABLE 1. The weight percent of Si and C elements individually determines the total concentration of SiC on each deposited sample. The Ni-SiC composite coating's SiC concentration increases as the ultrasonic agitation temperature rises from 50°C to 60°C. The highest temperature of 60°C recorded the highest SiC concentration on the specimen with 11.73 wt% for Si and 44.76 wt% for C.

TABLE 1

Element Weight Percentage

Sample	Element Weight Percentage		
	Element	Weight%	Atomic%
S.50	C K	8.11	25.27
	O K	7.08	16.55
	Al K	3.56	4.94
	Si K	2.09	2.78
	Ni K	79.17	50.47
	Totals	**100.00**	
S.55	C K	19.70	41.72
	O K	17.29	27.48
	Al K	1.11	1.05
	Si K	5.80	5.25
	Fe K	9.55	4.35
	Ni K	46.55	20.16
	Totals	**100.00**	
S.60	C K	44.76	55.43
	O K	38.63	35.91
	Al K	4.07	2.24
	Si K	11.73	6.21
	Ni K	0.82	0.21
	Totals	**100.00**	

3.4. Hardness Property Analysis

The hardness analysis on the coated samples is to study the steel's ability to withstand when two hard surfaces collide while applying force [13,14]. Fig. 3 illustrates the relationship between the microhardness values and the temperature of ultrasonic agitation. As the temperature of ultrasonic agitation increases, the microhardness values also rise. The lowest microhardness value of 44.7837 HV was recorded at 50°C, while the highest value of 77.4019 HV was achieved at 60°C. The increase in hardness demonstrates that the deposition on the sample becomes harder as the temperature of ultrasonic agitation increases, as the hardness of a sample is linked to the deposited coating layers. The application of a higher temperature during ultrasonic agitation also results in an increase in coating thickness.

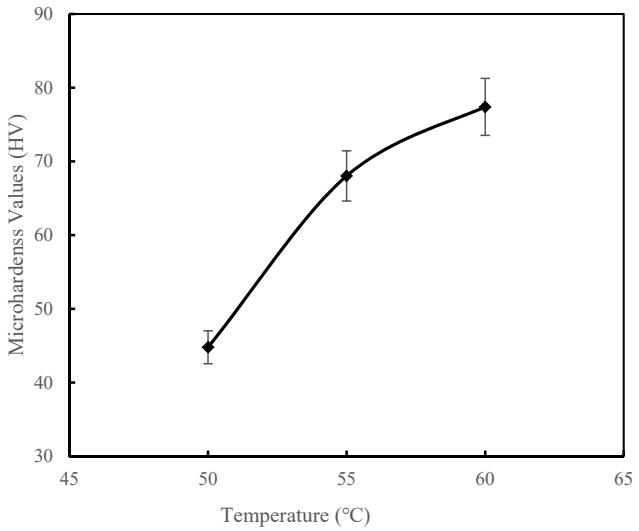

Fig. 3. Graph of Ultrasonic Agitation Temperature versus Microhardness Values

3.5. Corrosion Behaviour Analysis

The corrosion test was carried out using the sulphuric acid solution as the immersing medium [15]. The corrosion rate is only evaluated on the exposed coated surface. The weight loss of the specimen is measured before and after the immersion for three weeks.

The first eight days showed no apparent changes in the mass after the immersion. After 21 days, the deposited sample's weight reduction is determined by the difference between before and after weight as shown in TABLE 2. Later, using the weight difference, the corrosion rate was calculated [15]. The sample with a temperature of 50°C showed the largest weight difference, whereas the sample at 60°C had the smallest weight difference

TABLE 2

Weight difference before and after immersion test and its corrosion rate

Sample	Weight before (g)	Weight after (g)					Weight Difference (g)	Corrosion Rate (mm/year)
		4 days	8 days	12 days	16 days	21 days		
S.50	20.687	19.672	18.351	17.824	17.017	16.868	3.819	0.0059
S.55	21.702	21.461	20.873	20.237	19.746	19.208	2.494	0.0038
S.60	19.850	19.685	19.210	18.784	18.285	17.963	1.8872	0.0029

after 21 days. As a result, the highest corrosion rate was recorded for the sample at 50°C temperature and the lowest was at 60°C. This proves that the higher content of deposited Ni-SiC coating at a higher temperature improves the corrosion behaviour of the sample. This is due to ultrasonic agitation temperature application helping to distribute a uniform deposition of Ni-SiC on the samples. The properties of the Ni-SiC coating perform as a shield of protection on the sample for better corrosion resistance.

Fig. 4 depicts the graph of the corrosion rate at different ultrasonic agitation temperatures. The graph shows a trend of decreasing corrosion rate when the temperature of ultrasonic agitation increases.

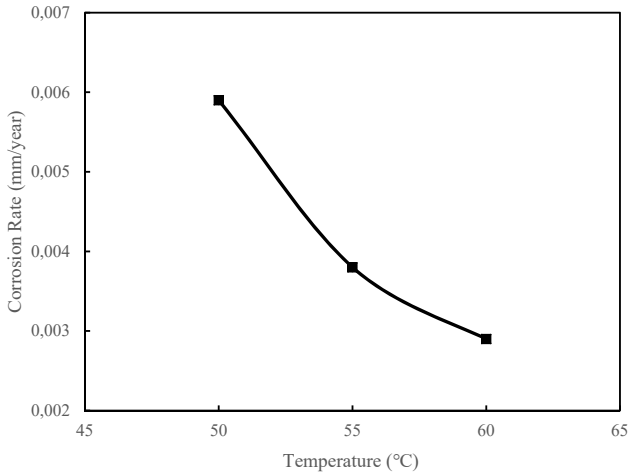

Fig. 4. Graph of Corrosion Rate against Temperature

3.6. Microstructure analysis after corrosion test

Again, Scanning Electron Microscopy (SEM) observation was conducted after the corrosion test to study the surface morphology and microstructure of the corroded specimen. The SEM images for the corroded sample were captured at ×250 magnification.

Fig. 5 presents the results of the SEM analysis after the corrosion test. The images demonstrate that the sample with a higher corrosion rate exhibits more micro-holes and cracks, indicative of corrosion. Therefore, the sample with an ultrasonic agitation temperature of 50°C demonstrated lower corrosion resistance compared to the sample at 60°C, as depicted in Fig. 4. Hence, when the temperature of ultrasonic agitation increases, the corrosion rate will decrease, increasing the corrosion holes. This is because the Ni-SiC coating improves the surface morphology of the deposited surface and limits the penetration of sulphuric acid solution during the immersion test. Thus, fewer cracks and holes were visible on the surface of the sample with higher ultrasonic agitation temperature.

4. Conclusion

In conclusion, the study investigating the electrodeposition of nickel silicon carbide on low-carbon steel was successfully carried out. The manipulated variable was the ultrasonic agitation temperature, which was set at 50°C, 55°C, and 60°C. The results of the electrodeposition process showed that the parameters had a significant impact on the final analysis. The samples with different parameters displayed significant structural changes. The EDS analysis revealed that the concentration of silicon carbide increased with temperature, and at 60°C, the highest values were recorded for silicon and carbon at 11.73 wt% and 44.76 wt% respectively.

The hardness results indicate that the coated metal becomes harder as the ultrasonic agitation temperature increases. The highest hardness value was recorded as 77.4019 HV at 60°C. The corrosion behavior analysis demonstrated that an increase in the ultrasonic agitation temperature led to a decrease in

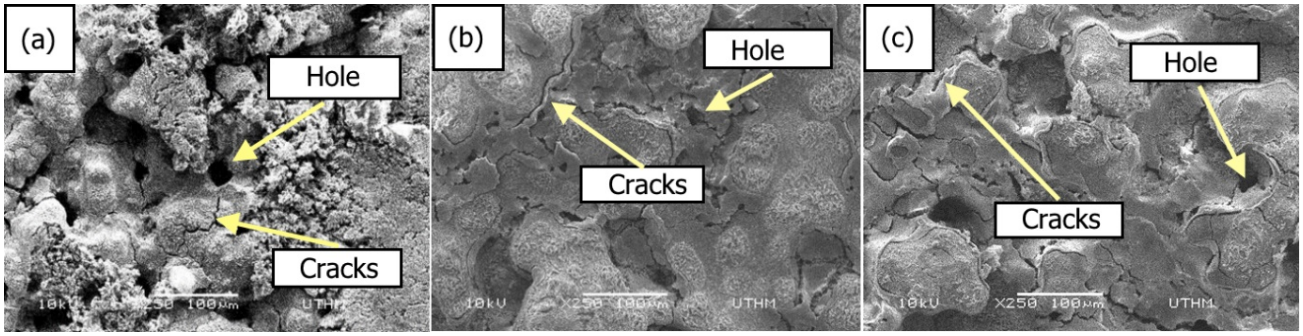

Fig. 5. SEM Image After Corrosion Test Analysis a) 50°C, b) 55°C and c) 60°C

the corrosion rate of the specimen. A specimen with a lower corrosion rate offers the best corrosion resistance. Thus, the influence of the highest temperature at 60°C provides the best corrosion resistance for the deposited metal.

Thus, the impact of ultrasonic agitation temperature was demonstrated on the specimen's hardness and corrosion behaviour. The highest hardness and the best corrosion resistance were achieved at the highest temperature of ultrasonic agitation. Thus, the contribution of the agitation temperature is crucial to enhance the properties of nickel silicon carbide coatings.

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