The Effect of Heating Time and Heating Temperatures to Layer Thickness and The Glossiness Level of A Surface in Blackening Coating Process

Arya Mahendra Sakti, Aisyah Endah Palupi, Dyah Riandadari

Abstract: Metal coating without electrical energy using usually could be the conversion coating technique. One method of the conversion coatings that are widely used in the industrial field is blackening or black oxide coating. Blackening or black oxide coating technique is a chemical conversion process that is formed from the chemical reaction between the iron in ferrous metals by an oxidizing salt to form a layer of magnetite (Fe_3O_4) thin metal substrates. The metal coating process has a lot of choices to improve the quality of materials from metallic and nonmetallic. This study aims to investigate the effect of the time and the temperature of the heating process of the steel AISI 1015 on the thickness and the value of the glossiness level by the blackening processing technique. The research variables are used various heating process time: 30; 60; and 90 minutes, temperature: 100 °C; 150 °C and 200 °C, and other variables are controlled constantly. The workpieces made from a steel metal plate AISI 1015 has a dimension size = $200 \times 30 \times 3 \text{ mm}^3$ in 27 pieces. The experimental method was designed by formation 3x 3×3 . It could be revealed the longer the healing process time could increase the thickness of the layer of the workpiece surface. A higher heating temperature could decrease the value of the glossiness level of a metal surface. The surface layer thickness level is found the best in the plating process time about 90 minutes and a temperature of 200°C with a coating thickness values of 20,27µm. The highest value of the glossiness level is obtained at the highest coating time of 30 minutes at a temperature of 100°C with the value of the shiny level of about 68.50 GU.

Keywords: blackening technique, layer thickness, and glossiness level.

I. INTRODUCTION

A metal coating is a metal layer deposition process on the electrode aimed at forming a surface with properties or dimensions different from the original metals [1]. A metal coating is one way to tackle the damage caused by corrosion on the metal and also serves as a resistance to the original material. Besides, plating also provides an aesthetic value of

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the metal to be coated, for example, color and texture, as well as to reduce the resistance of contacts and increase surface conductivity or reflectivity. Metal coating without the use of electrical energy usually called the conversion coating. Conversion coating is a coating method that utilizes a chemical reaction to form a protective layer on the metal surface. Conversion coatings can be divided into two groups: natural and chemical conversion coating. Natural conversion coating commonly called a natural oxide layer formed on a metal substrate, where as the chemical conversion coating is an artificial oxide layer formed on the metal substrate of the results of immersion in liquid chemicals [2,8,9]. In the industrial world, the metal is the dominant material used for components and machine tool supporting the engine. Among these materials, is a material widely used in engine components, which in practice is only done using a lubricating oil to inhibit corrosion rate due to the nature of the components that have high dimensional tolerances and require coatings that do not add significantly dimensional objects. One form of conversion coating is blackening or black oxide coating. Blackening or black oxide coating is a chemical conversion process that is formed from the reaction between the iron in ferrous metals by oxidizing salt. Blackening/black oxide coating used increasingly popular for durability against corrosion and aesthetic results without changing the dimensions of an object. Blackening coating occurs when the Fe atoms that exist on the steel surface reacts to form magnetite (Fe₃O₄). Surfaces cleaned product in alkaline soak and then rinse before soaking in a solution of blackening. After the second rinse, the final result will be covered by rust prevention, which can produce the final shape that varies from slightly oil until firm and dry [3,7,10]. The material used in a study is also affected by the heating temperature. The material will show that the thickness of the oxide layer on the steel AISI 304 increased from 8 nm at a temperature of 150°C to about 30 nm at a temperature of 450°C [4]. The black oxide coating process is determined by the optimum operating conditions and proper, for example, by determining the length of time of 150 seconds immersion and become stable after passing 180 seconds, the heating temperature 85°C. Existing operating conditions will affect surface roughness

numbers increased from 180

nm to about 290 nm, and also

The Effect of Heating Time and Heating Temperatures to Layer Thickness and The Glossiness Level of A Surface in Blackening Coating Process

on the thickness of the copper material surfaces [5]. The heating time in the process of cast iron blackening effect on the thickness of the oxide layer, the oxide layer thickness on cast iron increased from 0.4 μ m to 1.2 μ m in 30 minutes heating time and temperature 145°C [2].

This study aims to determine the thickness of the surface layer and the gloss layer of the workpiece surface black oxide coating results.

II. MATERIAL AND METHODS

A. Material

In this research, we used metal-coated AISI 1015 steel. The chemical composition of AISI 1015 steel (5% wt): 0.15C, 0.082Mn, 0166 Si, 0.054P, 0.15S, and Bal. Fe [11,12]. In this experiment, the workpiece in the form of steel plates AISI 1015 form. Dimensions specimens plate has a length of 200 mm, width 20 mm, and 3 mm thick.

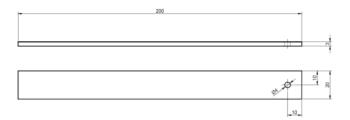


Fig. 1. Specimen Shape Plat

B. Methods

The experimental design used was a factorial design 3x3x3 fixed-effect model. The response variable in this study is the layer thickness of the workpiece and the workpiece gloss layer of black oxide coating results [14]. As the independent variable is the time of heating and the heating temperature. There are 3 levels of heating time, namely: 30; 60; and 90 minutes. The heating temperature also has three levels, namely 100 °C; 150°C and 200°C, other variables are held constant. The control variable is the type of workpiece, a saline solution used was a mixture of 30% sodium hydroxide (NaOH), 10% sodium nitrate (NaNO₃), 10% Sodium Nitrite (NaNO₂), 50% distilled (H₂O) [6,13]. The randomized experiment with three replications. Thus obtained 27 observation to experiment. The procedure begins with the preparation of a research workpiece by size 200 mm x 20 mm x 3 mm, Puncture specimens with a diameter of 4 mm, and clean the workpiece by means of polished. Clean the specimen before the blackening process by dipping the specimen into the ethanol solution. Preparation of salt solution, by mixing 30% sodium hydroxide (NaOH), 10% sodium nitrate (NaNO₃), and 10% Sodium Nitrite (NaNO₂) into 50% distilled (H₂O). Once well mixed, heated saline solution using the stove and keep the temperature steady 100°C, 150°C, and 200°C. Coating specimens by preparing a specimen that has been cleaned and is associated with a holder with wire so that the position of the specimen hangs in the plating bath. Specimen numbered 27 pieces that had been prepared coating process is then performed at a temperature of 100°C, 150°C, and 200°C with a variation of 30 minutes, 60 minutes and 90 minutes. After the coating process is completed, the specimens are dried and then soaked in the oil. After the specimen is completed the coating process, then proceed with the entire specimen coating thickness measurement using an ultrasonic thickness gauge. Followed by entire specimen surface gloss measurements using a gloss meter.

III. RESULT AND DISCUSSION

A. Layer Thickness Testing

Coating thickness measurement test specimens after blackening coating process, the coating thickness results are as follows Table 1 below. After the layer thickness test data is obtained, the results obtained are made in graphical form as can be seen in Figures 2 to 4.

TABLE 1. THE TEST DATA LAYER THICKNESS OF THE WORKPIECE

WORKPIECE							
No.	Time	Temperatures	Test Thickness (GU)			Average	
	(minute)	(°C)	I	II	III	(GU)	
1	30	100	16.52	16.50	16.48	16.50	
2	30	100	16.48	16.46	16.42	16.45	
3	30	100	16.46	16.44	16.45	16.45	
4	30	150	17.48	17.42	17.46	17.45	
5	30	150	17.46	17.48	17.44	17.46	
6	30	150	17.47	17.46	17.42	17.45	
7	30	200	18.12	18,14	18.12	18.13	
8	30	200	18.10	18.16	18.16	18,14	
9	30	200	18.16	18.20	18.18	18.18	
10	60	100	17.44	17.40	17.42	17.42	
11	60	100	17.50	17.44	17.46	17.47	
12	60	100	17.48	17.46	17.45	17.46	
13	60	150	18.36	18,32	18.34	18.34	
14	60	150	18.40	18.38	18.38	18.39	
15	60	150	18.38	18.36	18.36	18.37	
16	60	200	19.10	19.12	19.16	19.13	
17	60	200	19.14	19.16	19.12	19.14	
18	60	200	19.08	19.18	19.18	19.15	
19	90	100	18.06	18.10	18.16	18.11	
20	90	100	18.12	18,14	18,14	18.13	
21	90	100	18.18	18.16	18.15	18.16	
22	90	150	19.24	19,20	19.22	19.22	
23	90	150	19.18	19.22	19.24	19.21	
24	90	150	19,20	19.26	19.26	19.24	
25	90	200	20.27	20.28	20.26	20.27	
26	90	200	20.20	20.26	20.23	20.23	
27	90	200	20.22	20.25	20.27	20.25	

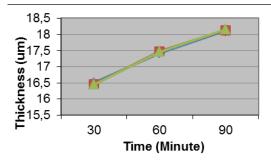


Fig. 2. Graph thickness of the metal coating results with a temperature of 100°C



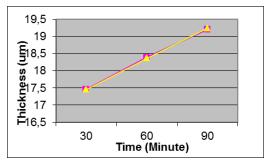


Fig. 3. Graph thickness of the metal coating results with a temperature of $150^{\circ}\mathrm{C}$

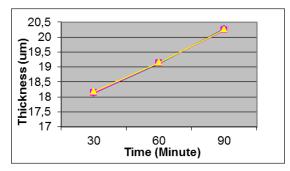


Fig. 4. Graph thickness of the metal coating results with a temperature of $200^{\rm o}{\rm C}$

The electroplating process using blackening was affected at surface thickness value as the surface plating production. In Fig. 2 to Fig. 4 were seen the holding time that was observed at 30, 60, 90 min created the plating thickness at 16,45 mm to 20,27 as the highest thickness. The heating time of the blackening process affected most significant to the workpiece, that caused by the magnetic layer on the metallic surface until magnetite (Fe₃O₄)) formed perfectly and the color of the metallic surface was becoming darker (atomic Fe at the surface was reacted with the carbon into magnetite (Fe₃O₄)). At those processes, the ion OH shifted to the surface and created diffusion-reaction on the plating surface then created the new atomic chemical bond with negative ions of Fe at that time become uniform thickness at the workpiece surface. That diffusion process was created and pilled up on the workpiece surface along the heating time [15,16,19].

A well as the heating temperature in the blackening process that affected the metallic surface thickness, because of the high heating temperature created the diffusion-reaction of the ion OH increased significantly and move to the metal surface. Sufficiency of ion OH at workpiece surface that made the surface been a more alkaline condition. Then, the chemical bond reaction has been increased significantly to formed the magnetite (Fe₃O₄) layer on the metal surface.

TABLE 2. DATA FROM THE RESULTS OF GLOSS TESTING OF THE WORKPIECE LAYER

THE WORLD BUILDIN						
No.	Time	Temperatures	Test Gloss (GU)			Average
	(Minute)	(°C)	I	II	III	(GU)
1			68.5	68,5	68.4	68,50
	30	100	2	0	8	08,30
2			68.4	68.4	68.4	68.46
	30	100	8	6	4	08.40
3			68.4	68.4	68.4	68.44
	30	100	4	2	5	06.44
4	30	150	67,4	67.2	67.3	67.33

			0	8	2	
5			67.3	67.3	67.2	
	30	150	6	4	0	67.30
6	50	150	67.3	67,5	67.1	
U	30	150	0	07,3	0	67.30
7	30	150	66.1	66.4	66.1	
,	30	200		0	00.1	66.20
8	30	200	0	66.3	66.4	
8	20	200	66.2			66.33
- 0	30	200	4	4	0	
9			66.2	66.4	66.3	66.31
	30	200	0	2	2	
10			67.4	67.4	67.4	67.46
	60	100	4	6	8	071.0
11			67.5	67.4	67.4	67.47
	60	100	2	4	6	07.47
12			67.4	67.4	67,5	67.46
	60	100	6	2	0	07.40
13			65.2	65.2	65.1	CF 10
	60	150	5	0	2	65.19
14			65.1	65.4	65.2	
	60	150	5	0	3	65.26
15			65.1	65.1	65.1	
10	60	150	0	0	0	65.10
16	- 00	100	64.4	64.4	64.1	
10	60	200	0	6	0	64,32
17	00	200	64.3	64.3	64,2	
1 /	60	200	4	6	4	64.31
18	00	200				
18	60	200	64.2	64.4	64.3	64.36
10	60	200	6	5	6	
19	0.0	100	66.1	66.1	66.1	66.15
	90	100	6	0	8	
20			66.1	66.1	66.1	66.14
	90	100	2	4	5	
21			66.1	66.1	66.1	66.17
	90	100	8	6	7	00.17
22			64.4	64.3	64,2	64,32
	90	150	2	0	4	07,34
23			64.4	64.4	64,3	64.42
	90	150	8	2	5	04.42
24			64.4	64.4	64.4	CA 45
	90	150	6	6	2	64.45
25			62.6	62.6	62.7	
	90	200	4	0	2	62.65
26			62.7	62.5	62.6	
20	90	200	4	6	8	62.66
27	70	200	62.6	62.6	62.6	
21	90	200		2	5	62.64
L	30	200	6		J	

B. Gloss Testing

Measurement gloss layer test specimens after blackening coating process, coating gloss results are as follows in Table 2. After taking the gloss test data, the results obtained are made in graphical form as can be seen in figure 5 to 7 below.

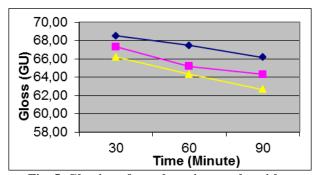


Fig. 5. Glossing of metal coating results with a temperature of 100°C



The Effect of Heating Time and Heating Temperatures to Layer Thickness and The Glossiness Level of A Surface in Blackening Coating Process

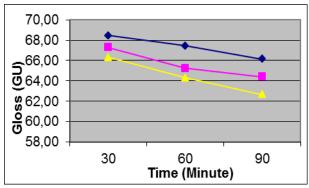


Fig. 6. Glossing of metal coating results with a temperature of $150^{\circ}C$

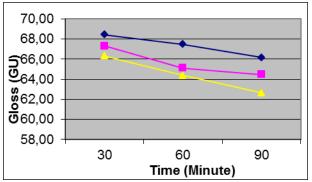


Fig. 5. Glossing of metal coating results with a temperature of 200°C

The blackening affected to the shiny grade of the metal surface as the plating metal production. Figures 5 to 7 were shown the holding time that investigated at 100, 150, 200 min created the shiny grade level was 68,50 GU with the lowest grade at 62,64 GU. The heating time of the blackening affected the layer thickness on the metal surface significantly. Because the heating time created the plating layer of the metal increased in the thickness obviously. Then the surface colors been darker and reduced the shiny grade level of the workpiece surface [15,17,18].

The heating temperature importantly influenced increased the thickness of the metal surface. By the investigation described the higher heating temperature created an increased in the thickness of magnetite (Fe_3O_4) on metal surface nevertheless reduced the shiny grade level.

IV. CONCLUSION

Based on the results of research and discussion of the data that has been done, it can take several conclusions, among others:

- The longer time the thicker layer of coating the magnetite (Fe₃O₄) which is formed on the metal substrate and smallest number of gloss.
- The higher the temperature, the more thick layer of magnetite (Fe₃O₄) which is formed on the metal substrate and the smallest number of gloss.

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