



# Specimen Test Analysis of Different Forms in Process Blackening for Layer Thickness of Surface Coatings and Glossiness Level

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**Abstract.** There are many options for metal plating processes to improve the quality of metal and non-metallic materials. Metal coating without the use of electrical energy is known as conversion coating. One form of conversion coating that is widely used in the industrial world is blackening or black oxide coating. One of the determining factors in the blackening process is the cleanliness of the workpiece to be processed, the immersion time, and the heating temperature. So far, heating in the blackening process has only been carried out using an estimate that the heated solution is ready when it boils. Just looking at the boiling solution is not enough to ensure that the heating temperature is in accordance with the provisions. So to get perfect quality results, it is necessary how much temperature will be used for the blackening process. The parameters of this research are to improve the quality of blackening results which are influenced by the length of heating time, heating temperature, workpiece thickness, and workpiece surface gloss. AISI 1015 steel is low-carbon steel. The purpose of this study was to examine the effect of heating time and temperature on the blackening process on the thickness and gloss of the surface layer. The test material in this study was AISI 1015 steel in the form of plates and cylinders in time variations of 50 min, 75 min, and 100 min and with temperature variations of 100 °C, 150 °C, and 200 °C. The results showed that the highest value of the surface layer thickness occurred in the coating with a time of 100 min and in the coating with a temperature of 200 °C with a thickness of 20.27  $\mu\text{m}$  on the plate workpiece and 20.03  $\mu\text{m}$  on the cylindrical workpiece. Meanwhile, the highest gloss value was obtained for coating with a time of 50 min and a temperature of 200 °C with a gloss rate of 68.14 GU for plate workpieces and 67.86 GU for cylindrical workpieces.

**Keywords:** Blackening process · Surface thickness · Surface gloss

## 1 Introduction

The coating is the process of depositing a substance or material onto the surface of the material to be coated to protect the material from being in direct contact with the environment, beautifying the surface and giving certain properties to a workpiece surface, where it is hoped that the object will experience improvements both in terms of

microstructure and microstructure. Durability, and does not rule out the possibility of improvement in its physical properties. The coating is the final part of the production process of a product. The process is carried out after the workpiece reaches the final shape or after the machining process and smoothing of the workpiece surface is carried out.

Thus, the coating process is included in the category of finishing work or often also called the completion stage of a workpiece production. Chemical conversion coatings are also known as chromatin, chromate conversion, and allowing. Unlike anodizing, chemical conversion coatings do not require electrical energy in the manufacturing process, so they are more cost-effective to manufacture. Chemical conversion coatings can be colored or colorless, depending on preference. Chemical conversion coating produces a thin and thorough crystalline layer on the surface of the object that has not been treated or prepared. If an object to be coated is properly prepared, this coating will produce an excellent base coat for painting and adhesives. A simple mechanism for the formation of a black oxide layer was proposed by Samartsev, who proposed that in the presence of oxidants, cast iron would react with an alkaline solution to produce dissolved sodium ferrite by the following chemical reaction [1].

One form of conversion coating is blackening or black oxide coating. Blackening or black oxide coating is a chemical conversion process formed from the reaction between iron in ferrous metal with oxidizing salts to form magnetite ( $\text{Fe}_3\text{O}_4$ ) [2]. Based on the research of [3] with the title "Influence of the Temperature of Film Formation on the Electronic Structure of Oxide Films Formed on 304 Stainless Steel", showed that the thickness of the oxide layer on AISI 304 steel increased from 8  $\mu\text{m}$  at a temperature of 150 °C to approx. 30  $\mu\text{m}$  at 450 °C.

A similar study by [4] entitled "Optimization of Black Oxide Coating Thickness As An Adhesion Promoter for Copper Substrate In Plastic Integrated-Circuit Packages" showed that the surface roughness increased from 180 nm to around 290  $\mu\text{m}$  during the 60 s heating process, and the thickness of the oxide layer continued to increase from the first 150 s and became stable after passing through 180s. The results of [1] research entitled "A Study of Coating Process of Cast Iron Blackening" showed that the thickness of the oxide layer on cast iron increased from 0.4 m to 1.2 m in a heating time of 30 min and a temperature of 145 °C.

## 2 Research Methods

This study uses AISI 1015 steel workpieces which have several advantages in the machining process of machine component objects. The characteristics of the chemical composition of the workpiece can be seen in Table 1..

Dimensions specimens plate has a length of 200 mm, width 20 mm, and 3 mm thick. And cylinder with size 200 mm, diameter 15 mm.

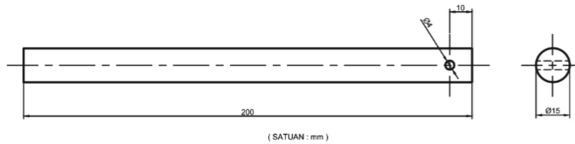
In this study, a 3 x 3 x 1 factorial experimental design was used. The response variables used are the surface thickness and surface gloss of the magnetite ( $\text{Fe}_3\text{O}_4$ ) workpiece. For the dependent variable the length of heating time (50 min, 75 min, and 100 min) and heating temperature (100 °C, 150 °C, and 200 °C). Thus obtained 18 observations to experiment. The procedure begins with the preparation of a research

**Table 1.** Characteristics of the chemical composition of AISI 1015 steel

Number	Element	Amount (%)
1	Carbon	0.15
2	Manganese	0.082
3	Silicone	0.166
4	Phosphor	0.054
5	Sulfur	0.15
6	Iron	99.398
Total		100



**Fig. 1.** Specimen Shape Plat.



**Fig. 2.** Specimen Shape cylinder.

workpiece in size 200 mm x 20 mm x 3 mm for a plate and cylinder with a size of 200 mm, and a diameter of 15 mm. 18 workpieces were coated with no electricity using variations in the dependent variable, namely temperature of 100 °C, 150 °C and 200 °C with a variation of 50 min, 75 min and 100 min. 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 (Figs. 1 and 2).

### 3 Result and Discussion

The coating thickness measurement test specimen prior to the coating process is 0 μm, and the average glosness is 163.73 GU. After blackening coating process, the thickness and gloss coating results are as follows the Table 2. and Table 3.. Coating Time and Temperature to Thickness variations in the length of time and temperature of the coating greatly affect the metal plating process. This is influenced by the surface area of the

**Table 2.** After the coating layer thickness

No Specimen Shape Plat	Temperatur (°C)	Time (min)	Average Thickness (μm)	No Specimen Cylinder	Temperatur (°C)	Time (min)	Average Thickness (μm)
1	100	50	14,46	1	100	50	14,13
2		75	15,34	2		75	15,20
3		100	16,29	3		100	16,18
4	150	50	16,48	4	150	50	16,34
5		75	17,36	5		75	17,22
6		100	18,09	6		100	18,02
7	200	50	18,12	7	200	50	18,06
8		75	19,10	8		75	19,02
9		100	20,27	9		100	20,03

**Table 3.** After a gloss layer coating

No Specimen Shape Plat	Temperatur (°C)	Time (min)	Average Thickness (μm)	No Specimen Cylinder	Temperatur (°C)	Time (min)	Average Thickness (μm)
1	100	50	68,14	1	100	50	67,86
2		75	66,42	2		75	66,08
3		100	65,18	3		100	65,04
4	150	50	65,06	4	150	50	64,76
5		75	64,34	5		75	63,68
6		100	63,18	6		100	62,44
7	200	50	62,66	7	200	50	62,26
8		75	61,24	8		75	61,02
9		100	60,56	9		100	60,28

workpiece and the shape of the surface area of the workpiece carried out by the metal plating process. The surface area of the plate-shaped workpiece will make the coating process even faster and the thickness of the magnetite (Fe<sub>3</sub>O<sub>4</sub>) surface layer on the AISI 1015 steel substrate compared to the cylindrical workpiece. Below is a diagram of the average layer thickness of plate and cylindrical specimens against variations in coating time and temperature.

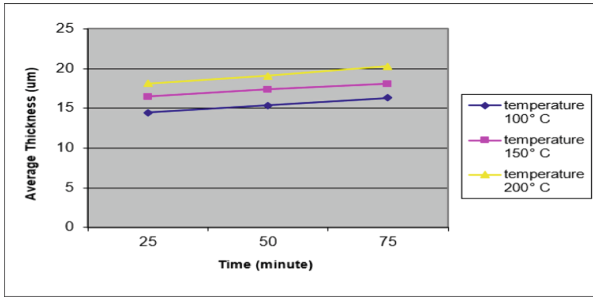


Fig. 3. Diagram of the average thickness of the plate-shaped specimen layer

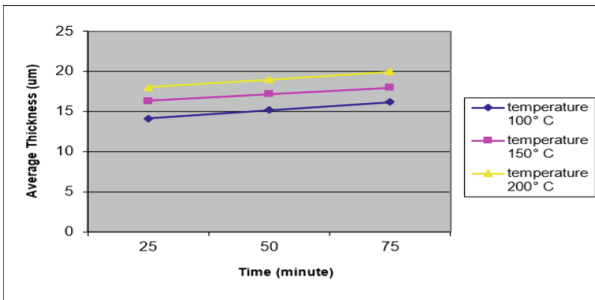


Fig. 4. Diagram of the Average Layer Thickness of Cylindrical Specimen

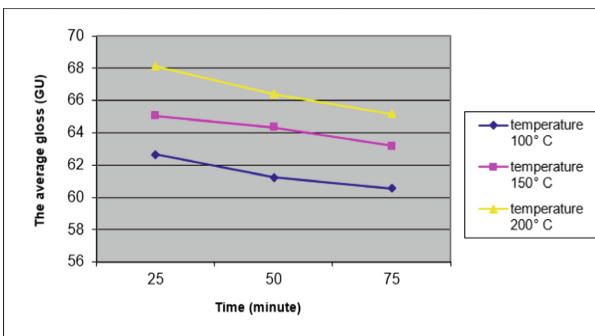
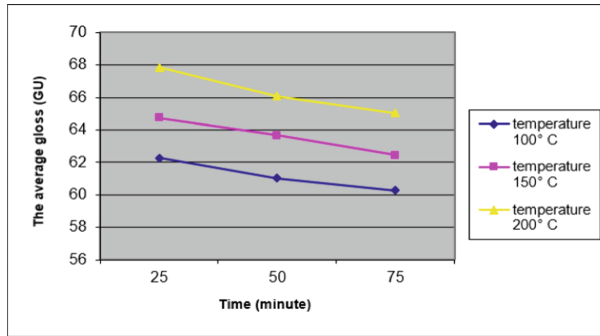


Fig. 5. Diagram of the Average Glossiness of Plate Shape Specimen Layers

Based on the diagrams in Figs. 3 and 4, it shows that the thickness of the magnetite ( $Fe_3O_4$ ) layer formed will be thicker, which goes with the length of time and the higher the coating temperature [6–8]. The highest thickness value was obtained on the workpiece with a coating time of 100 min and a coating temperature of 200 °C with a value of 20.27  $\mu m$  for plate-shaped specimens and 20.03  $\mu m$  for cylindrical workpieces.



**Fig. 6.** Diagram of the Average Glosiness of Cylindrical Specimen Layers

**Coating Time and Temperature to Gloss** The length of time and temperature of the coating will affect the number of gloss layers formed on the AISI 1015 steel substrate, both on plate-shaped workpieces and on cylindrical workpieces [9, 10]. The following is a diagram of the average gloss number of plate and cylindrical workpiece layers against variations in coating time and temperature.

Based on the diagrams in Figs. 5 and 6, it is shown that the gloss number of the magnetite ( $\text{Fe}_3\text{O}_4$ ) layer decreases with the length of time and the higher the coating temperature. The highest gloss value was obtained on the workpiece with a coating time of 50 min and a coating temperature of 100 °C with a value of 68.14 GU for plate-shaped workpieces and 67.86 GU for cylindrical workpieces [5].

## 4 Conclusion

In this study resulted in several conclusions are the longer the time of coating and the higher the temperature the more thick layer of magnetite ( $\text{Fe}_3\text{O}_4$ ) which is formed on a metal substrate, but decreases the level of gloss coating and the shape of the test specimens affect the thickness and gloss surface layer.

**Authors' Contributions.** Dyah Riandadari and Arya Sakti contributed writing, Firman Utama and Ferly Abdi contributed correcting, editing, read and approved the final manuscript.

## References

1. Arab, N. and Soltani. 2009. A Study of Coating Process of Cast Iron 'sa. Iran: Journal of Applied Chemical Research, 13–23.
2. Schwartz, Mel. 2002. Encyclopedia and Handbook of Materials, Parts and Finishes Second Edition. Florida: CRC Press LLC.
3. Ferreira, Essential, et al. 2001. Influence of the Temperature of Film Formation on the Electronic Structure of Oxide Films Formed on Stainless steel 304, *Eletrochimica Acta* 46, 3767–3776.
4. Lebbai, Kim, et al. 2003. Optimization of Black Oxide Coating Thickness as an Adhesion Promoter for Copper Substrate-Integrated Circuit in Plastic Packages. Hongkong: Journal of Electronic Materials, Vol. 32, No. 6.

5. UNESA. 2000. Guidelines for Writing Journal Articles, Surabaya: Research Institute of the State University of Surabaya.
6. Afzal, S., Khan, R., Zeb, T., Ali, S., Khan, G., & Hussain, A. (2018). Structural, optical, dielectric and magnetic properties of PVP coated magnetite (Fe<sub>3</sub>O<sub>4</sub>) nanoparticles. *Journal of Materials Science: Materials in Electronics*, 29(23), 20040–20050.
7. Antarnusa, G., & Suharyadi, E. (2020). A synthesis of polyethylene glycol (PEG)-coated magnetite Fe<sub>3</sub>O<sub>4</sub> nanoparticles and their characteristics for enhancement of biosensor. *Materials Research Express*, 7(5), 056103.
8. Torres-Gómez, N., Nava, O., Argueta-Figueroa, L., García-Contreras, R., Baeza-Barrera, A., & Vilchis-Nestor, A. R. (2019). Shape tuning of magnetite nanoparticles obtained by hydrothermal synthesis: effect of temperature. *Journal of Nanomaterials*, 2019.
9. Barrena-Rodríguez, M. D. J., Acosta-González, F. A., & Téllez-Rosas, M. M. (2021). A review of the boiling curve with reference to steel quenching. *Metals*, 11(6), 974.
10. He, Z. W., Wang, M. H., Zhang, L., & Peng, W. (2015). Fabrication of Convex Plate-shaped Micro Tool Electrode and its Application in Micro hole Machining by ECM. In *Key Engineering Materials* (Vol. 645, pp. 1043–1048). Trans Tech Publications Ltd.

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