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Effect of Temperature Variation Characteristics Of Crystal Structure and Morphology of Nano TiO_2 Coating On Metal Anti-Corrosion Coating by Sol-Gel Method Spin Coating

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Abstract. This research which was conducted to determine the best coatings on ferrous metals to prevent corrosion by using sol gel spin coating method. TiO_2 coating material which is used the addition of epoxy resin binder. The comparison used in these TiO_2 coatings: resin: hardener is 1: 4 : 4. The process of synthesis TiO_2 main materials used are $TiCl_3$, distilled and NH_4OH. From mixing these materials will be obtained precipitate and then the precipitate dried at a temperature of 200 °C for 6 hours to obtain titanium powder. This coating is done by varying the temperature of 100 °C, 125 °C, 150 °C for 1 hour with rotation speed of 3500 rpm. Crystal structure testing by using the XRD where known that the crystal structure formed the anatase phase. Testing the morphological structure were performed using SEM which produce images of TiO_2 cubic shape. Testing the corrosion rate showed that the coated samples is more resistant to corrosion than the uncoated samples. The rate of corrosion on metal samples without coating is 40.0320 cm/year, while the rate of corrosion on metal coated sample was 2.8420 cm/year; 3.2440 cm/year; 3.5020 cm/year. Where each temperature is 100 °C, 125 °C, 150 °C.

Keywords: *TiO2, Corrosion rate*

1. Introduction

The problem of corrosion is an event that is prevalent in the metal. Metals have the advantage of having the nature hard and strong, so that the metal is best used for the manufacture of household appliances, motor vehicles and construction materials. In addition, the metal also has the disadvantage of easily corroded [1]. The main opponent of the steel is corrosion. Corrosion is the destruction or damage to property due to reactions with its surroundings.

Corrosion on the metal can also be interpreted as a reaction to the inverse of the metal refining. This corrosion itself can lead to decreased quality of the steel, resulting in a quick steel into a weak and broken [2].Type of corrosion often encountered in everyday life is atmospheric corrosion. Atmospheric corrosion is a result of interaction with the atmosphere surrounding metal, which occurs due to moisture and oxygen in the air and was exacerbated by pollutants such as gas and salts contained in the air [3].

One of prevention and protection against corrosion is way of coating. Coating or plating is the most frequently used to treat corrosion. There are two types of coating, the liquid coating and concrete

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coating. Liquid coatings are doing the painting on steel surfaces, so that the steel can be protected by corrosion. While the concrete coating is the coating of steel by coating the steel with concrete, usually this is done in the constructions of buildings in urban areas [4]. This coating protects the metal material of the electrochemical reaction with its environment, especially for humid area that contains a lot of moisture such as in Indonesia [5].

Attached to the metal layers will separate the direct contact of metal with the environment, so that the risk of corrosion attack can be minimized [6]. In addition to one of the paint coatings that can be used for anti-corrosion protection is titanium [7]. Titanium is the ninth most abundant element in the earth's surface after aluminum, iron and magnesium. Where Ti is a transition metal that is light, strong, lustrous and corrosion resistance [6]. Hu explain that one part of the technology the nanoparticles used as an alternative coating is TiO_2 (titanium dioxide) [8].

In the coatings industry, the use of TiO_2 is quite advantageous because only a thin coating (micrometer scale) has been able to coat the entire substrate [9]. One method that is often used in the manufacture of TiO_2 layer is Sol Gel Dip Coating. Sol Gel Spin Coating method has several advantages: low cost, homogeneous composition, can be carried out at low temperatures, do not use a vacuum chamber with a high level, as well as the thickness of the layers can be controlled [10].

2. Methods

The process of research, sample preparation, synthesis of TiO_2 conducted in Chemistry Laboratory, UniversitasNegeri Medan and testing samples (SEM and XRD) was conducted at the Laboratory of Physics, UniversitasNegeri Medan.

The procedure of the first studies conducted that the preparation of the substrate, the substrate used in the form of a metal plate measuring 3cm x 2 cm placed in glass beaker containing acetone $(CH_3)_2CO$ and ethanol 98%, and then the substrate and then dried by heating at a temperature of 100C for 20 minutes to evaporate the liquid remnants that exist on the substrate. Furthermore, the Sol-Gel Preparation of Titanium Dioxide (TiO_2) with the precursor $TiCl_3$ and asopropyl alcohol in several stages.

After the successful synthesis of TiO_2 coating substrate is carried out. Coating is done using an epoxy resin as a binder. Comparison between TiO_2 powder used: resin: hardener is 1: 4: 4. Method of coating used in this research is by spin coating (rotary techniques). Samples were placed on a spin coater (1000 rpm - 10000 rpm) and etched with the coating material and then the sample is rotated. The coating was done 2 times to evenly cover the coating material, with the aim of forming a flat layer with a homogeneous thickness. The sample is rotated with rotation of 3500 rpm.

After the coating process in metals, the next step is the process of burning the sample (firing) using different temperatures. Next, conducted corrosion tests on the samples to determine the corrosive properties of the test sample. Data analysis techniques in this research is by using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), corrosion testing and corrosion rate of the sample used.

3. Result and Discussion

1. The results of X-ray Diffraction Test of Metal Samples

Structural analysis using X-ray diffraction instrument intended to identify the crystal structure as well as the size of the coating formed on the sample. This study used a sample of metal (iron) with a variation of the combustion temperature of 100 °C, 125 °C, 150 °C. The results of X-ray diffraction patterns for the three samples as follows:

a. Samples Iron 100 °C

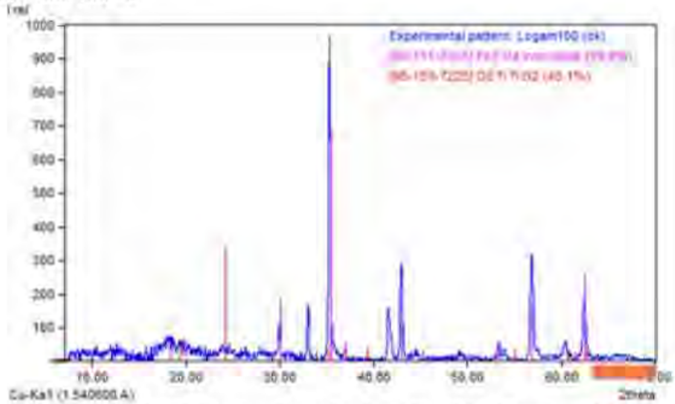

Figure 1. XRD Testing Samples Iron On With Heating 100 °C

Based on the identification result on samples of ferrous metals with a heating temperature of 100 °C is not formed, namely the desired layer of TiO_2. Phase is formed in the coating are iron oxide with a compound Fe_3O_4 which has a cubic crystal structure with lattice parameters a = b = c = 8.39850 Å and also formed the phase of TiO_2 with a compound TiO_2 has a crystalline structure trigonal (hexagonal axes) with lattice parameters a = b = 5.29100 Å c = 6.13300 Å. The particle size obtained at the heating temperature 150C coating is 111.2367 nm.

Table 1.Sample Mass fraction of iron With Combustion Temperature 100 °C.

No.	Compound Name (Name of COUMPOUND)	Phase (Phase)	Reference (Ref)	The mass fraction (Wt%)
1	Fe3O4	Iron oxide	96-151-3305	59.9
2	TiO2	TiO2	96-153-7225	40.1

b. Samples Iron 125 °C

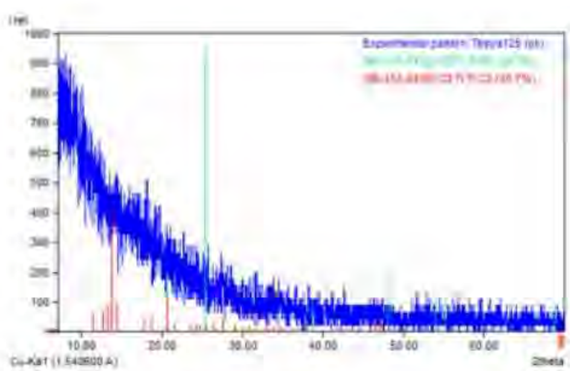

Figure 2. XRD Testing Samples Iron On With Heating 125 °C

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In the coating of metal samples with a heating temperature of 125 °C is also not form the desired coating which is formed with the compound TiO_2. TiO_2 phase but with a crystalline structure that is different tetragonal and triclinic (anorthic), lattice parameters of both compounds is a = b = 3.7800 Å c = 9.51000 Å; a = 15.62000 7.81000 Å Å b = c = 12.93200 Å. The particle size obtained at the heating temperature 125 °C coating is 185.23886 nm

Table 2. Sample Mass fraction iron With Combustion Temperature 125 °C

No.	Compound Name (Name of COUMPOUND)	Phase (Phase)	Reference (Ref)	The mass fraction (Wt%)
1	TiO_2	TiO_2	96-153-0152	59.3
2	TiO_2	TiO_2	96-412-4499	40.7

c. Samples Iron 150 °C

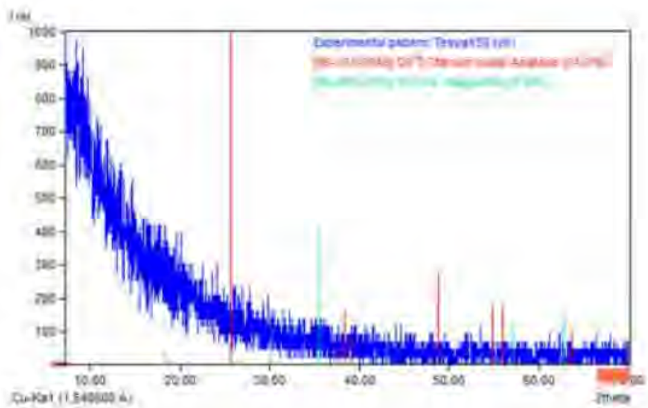

Figure 3. XRD Testing Samples Iron On With Heating 150 °C

While the coating metal samples with a heating temperature of 150 °C has been produced in two phases, namely anatase and magnetite with TiO_2 and Fe_3O_4 compound. The crystal structure formed in the TiO_2 compound is tetragonal with lattice parameters a = b = 3.7300 Å and c = 9.37000 Å in cubic Fe_3O_4 compound with lattice parameters a = b = c = 8.36850 Å. The particle size obtained at the heating temperature 150 °C coating is 182.13959 nm.

Table 3. Sample Mass fraction iron With Combustion Temperature 150 °C

No.	Compound Name (Name of COUMPOUND)	Phase (Phase)	Reference (Ref)	The mass fraction (Wt%)
1	TiO_2	Anatase titanium oxide	96-101-0943	74.2
2	Fe_3O_4	magnetite	96-900-2319	25.8

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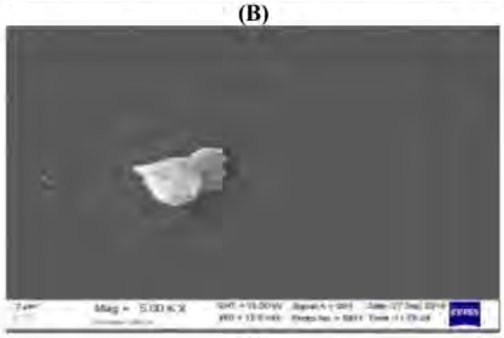
2. Test Results Scanning Electron Microscopy (SEM)

a. Samples Iron 100 ^{o}C

(A)

(B)

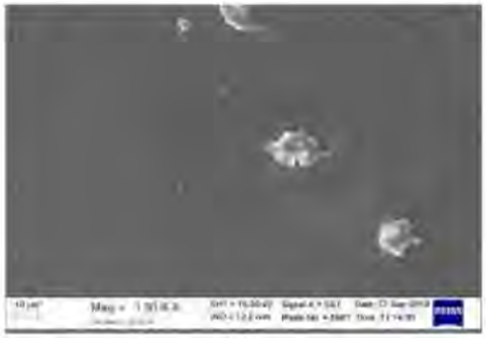


(C)

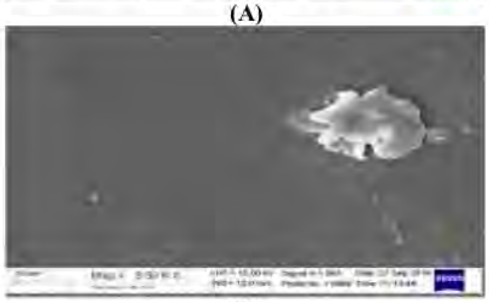
Figure 4. Results of SEM photos of ferrous metals with a metal heating temperature of 100 ^{o}C, (a) 500 times magnification, (b) 3500 times magnification, (c) 5000 times magnification

Figure 4 shows the morphology of the metal coating iron with heating temperature of 100 ^{o}C.of the image can be identified that the dark is a resin and looked white-gray is TiO2. From the image (a), (b), (c) with three types of magnification obtained the same coating morphology which contained a white blob.

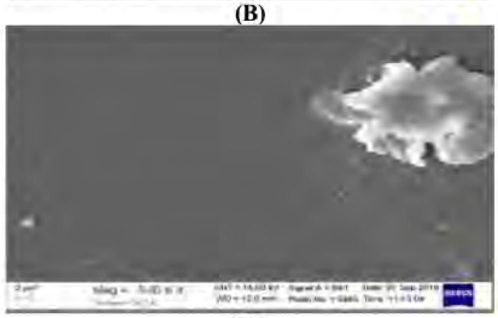
b. Samples Iron 125 °C



(A)



(B)



(C)

Figure 5. Results of SEM photos of ferrous metals with a metal heating temperature of 125 °C, (a) 1500 times magnification, (b) 3500 times magnification, (c) 5000 times magnification

Figure 5 shows the morphology of the metal coating iron with heating temperature 125 °C.of the image can be identified that the dark is a resin and looked white-gray is TiO_2. In (a) visible a few clumps of grayish-white with spots that are not too clear on the image (b) and (c) clumps of white-gray that looks only one and blotches on the samples more visible in conjunction with the zoomed in.

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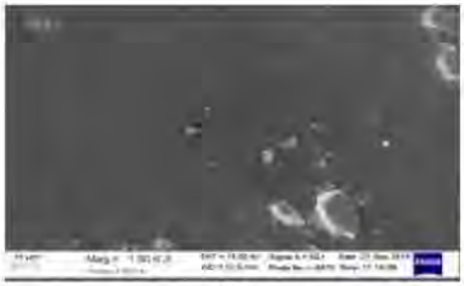
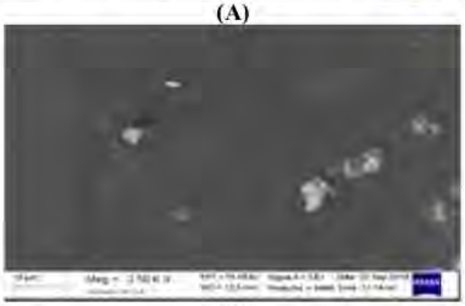
c. Samples Iron 150 °C



(A)



(B)

(C)

Figure 6. Results of SEM photos of ferrous metals with a metal heating temperature of 150 °C, (a) 1500 times magnification, (b) 3500 times magnification, (c) 5000 times magnification

Figure 6 shows the morphology of the metal coating iron with heating temperature 150 °C.of the image can be identified that the dark is a resin and looked white-gray is TiO_2. In (b) and (c) the morphology is similar to the grayish-white colored blobs scattered coating and there are also patches of smooth.

3. Testing Corrosion and Corrosion rate

Testing is done by soaking the samples in NaCl solution for 4x24 hour or equal to 96 hours. This study used a coating with TiO_2 with the combustion temperature variation 100 °C, 125 °C, 150°C. Corrosion testing results for the three samples as follows:

Table 4. Results of Corrosion Testing Metal Iron With Combustion Temperature Variations

No.	samples	Heating temperature (° C)	Massa Prior Coated (G)	Coated After the mass (g)	After mass Corrosion Test (gr)
1	A	uncoated	10.072	-	12.167
2	B	100	9.532	9.573	9.620
3	C	125	9.234	9.269	9.379
4	D	150	9,540	9.573	9.715

From the test result data is done by soaking the corrosion of ferrous metals sample that had been coated with the heating temperature variation can be seen the difference between the rate of corrosion metal samples that are not coated with the metal samples that has been coated with TiO_2. SEM image of the test also looked like a grayish-white blob of air bubbles that cause contamination of the sample with air or corrosive substances. Can be seen samples of uncoated metal corrosion rate faster than the metal samples that had been coated TiO_2, which is 0.7350 cm / year. And metal coated samples also have different corrosion rate depends on the temperature of heating. Samples with a heating temperature of 100 ^{o}C corrosion rate is slower than the sample with a heating temperature of 125 ^{o}C and 150 ^{o}C.

Where it is most likely because the results of SEM sample by heating with fewer clumps 100 ^{o}C and its coating looked uniform in comparison to other heating temperature. In the table 5 below are shown the results of calculations performed on the corrosion rate of ferrous metals samples with samples without coating and samples with temperature variations. It seems clear difference between the rate of corrosion of each sample.

Table 5. Calculation Results The rate of corrosion

No.	samples	Heating temperature (° C)	The rate of corrosion (cm / year)
1	A	uncoated	.7350
2	B	100	.1919
3	C	125	.4491
	D	150	.5798
4			

4. Conclusion

Based on the results, it can be concluded that:

a. Titanium dioxide (TiO_2) has been created with the addition of distilled water and NH_4OH into heating $TiCl_3$ with a temperature of 300 ^{o}C for 4 hours. It is not yet formed the expected size ienano. The size obtained by XRD testing on samples of coating the metal with a heating temperature of 100 ^{o}C, 125 ^{o}C and 150 ^{o}C respectively is 111.2367 nm; 185.2388; 182.1395.

b. Based on the results seen coating SEM samples with the heating temperature of 100 ^{o}C less of its contained clumps or air bubbles which tend the coating more evenly, while the heating temperature of 150 ^{o}C coating with a white blob is more dispersed throughout the sample surface.

c. From the results of corrosion testing proved that the TiO_2 coating can inhibit the corrosion rate of the sample. Samples without coating corrosion rate is 0.7350 cm / year; coating with a heating temperature of 100 ^{o}C corrosion rate is 0.1919 cm / year; coating with a heating temperature of

125 °C corrosion rate is 0.4491 cm / year; coating with a heating temperature of 150 °C corrosion rate is 0.5790 cm / year.

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