The Development of Fine Surface Roughness of FeCrAl Substrate by Gamma Alumina Coating Material Through Nickel Oxide Catalyst

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Abstract. The most commonly used method for protecting atmospherically exposed steel against corrosion, is the application of protective organic coating systems. It is widely recognized that the stability of the coating-substrate interface is related to the interfacial adhesion forces and electrochemical properties of this region. This study aims to develop fine surface roughness by ultrasonic and electroplating coating methods that applied for FeCrAl catalytic converter. This method consists of two methods which are ultrasonic bath that carried out by frequency of 35 kHz and various ultrasonic times of 1, 1.5, 2, 2.5 and 3 hours is imposed, and the electroplating was conducted for several variation times of 15, 30, 45, 60 and 75 minutes, current density of 8 A/dm². The result shows that the surface roughness of UB samples in between 0.11 to 0.21 μm, UBdEL samples of 0.81 to 2.17 μm, UB+EL samples of 0.64 to 1.63 μm and EL samples of 0.69 to 1.11 μm. The finest surface of each technique are located at UB 1.5 h, UBdEL 45 minutes, UB 1.5 h+EL 30 minutes and UB 30 minutes. That data is supported by coating thickness of coated FeCrAl substrate where UB samples in between 2 -2.8 μm, UBdEL samples of 4.1 to 5 μm, UB+EL samples of 9.1 to 12 μm and EL samples of 6.2 to 11.3 μm.

Keywords: surface roughness; electroplating; ultrasonic; catalytic converter

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1. Introduction

A washcoat is a catalysts carrier with high surface area. This material is usually an oxide layer such as Al₂O₃, SiO₂, TiO₂, or SiO₂-Al₂O₃ [1]. In this research, washcoat material is a thin layer of Gamma-Alumina (γ-Al₂O₃) coating, typically 20-150 μm of thickness with a high surface area on the top of substrate. It also called as a catalyst carrier and acts as an intermediate layer in order to oxidation and reduction processes occur [2].

Currently, the metallic CATCO was interesting component to explore as compared to ceramic material. Therefore, this study has chosen FeCrAl metallic substrate as a main CATCO component. The FeCrAl is generally considered as metallic substrates due to their advantage in the high thermal stability, high corrosion resistance, including the strong adherence of oxide film on the surface of substrate when applied the appropriate surface treatment [3].

The conventional technique for adhering NiO catalyst on substrate is by wash coating techniques that generally comprise of preparing a coating formed from a high surface area oxide blended with one or more catalysts and dipping the monolith structure into that coating blend [4-6]. One of the most common methods to form a thin layer of oxide coatings on the metallic substrate is dip coatings,
which combined with some pre-treatments, such as growing a number of textured alumina whiskers on the surface of the metal support and shortened the diffusion path before depositing the washcoat [7]. There are some problems that observed which are Rough surface roughness of FeCrAl catalytic converter has been observed as a main problem that need to solve due to it effect on exhaust gas conversion that performed by Catalytic converter. The exploration of FeCrAl metallic material and Gamma alumina as coating material not fully explored in catalytic converter Application [8-9]. Therefor the main objective of this research was to investigate an appropriate parameter of coating process in order to achieve fine surface roughness of FeCrAl substrate. To Analyze the effect of surface roughness in exhaust gas conversion on gasoline engine.

2. Research Methodology

Ultrasonic bath is conducted to FeCrAl foil which cut in size of 40 mm x 20 mm and γ-Al₂O₃ powder. The samples are cleaned in water and then proceed in ethanol for 5 minutes. During ultrasonic process, the frequency of 35 kHz and various ultrasonic times of 1, 1.5, 2, 2.5 and 3 hours is imposed. The samples is immersed into the beaker with γ-Al₂O₃ powder for 3 g in each samples and ethanol with the concentration of 20 g/l. The other methods will be conducted i.e. ultrasonic bath during electroplating. It principle of the process is similar with electroplating process with sulphamate type as electrolyte, but the differences is the stirrer is replaced by ultrasonic sources. It conducted in order to achieve homogenizing of coating process or levelling the spread coating material. Those process is conducted by referring Sebayang et al., (2012) [2]. Figure 1 shows the mechanism of ultrasonic bath and ultrasonic bath during electroplating process.

![Figure 1](image1.png)

**Figure 1.** Schematic diagram of a) ultrasonic cleaning bath and b) ultrasonic bath during electroplating:(1) Ethanol; (2) Specimen; (3) Beaker; (4) Water; (5) Bath; (6) Ultrasonic source; (7) Plating tank and (8) Electrolyte solution

According to Sebayang et al., (2012) [2] that the electroplating process was conducted using some equipments such as laboratory power supply, retorch clamp, hot plate, magnetic steering, glass beaker, petri disc, and, etc. Main purpose of the nickel electroplating process is for depositing nickel onto FeCrAl and embed the γ-Al₂O₃ into FeCrAl substrate. Electroplating process is conducted through some components such as electrolyte, anti-pitting agent, anode and cathode. Sulphamate type is used as the electrolyte medium. It based on Ade (2012) which has been used the sulphamate types as electrolyte which consists of nickel sulphamate (Ni(SO₃NH₂)₂·4H₂O), nickel chloride (NiCl·6H₂O), boric acid (H₃BO₃) and sodium lauryal sulphate (C₁₂H₂₅SO₄Na) and it resulted that electrolyte can be optimize the electroplating process. Therefore, in this research will be generated using sulphamate types which consists of nickel (II) sulphate 6-hydrate (NiSO₄·6H₂O), nickel (ii) chloride (NiCl₂·6H₂O), boric acid (H₃BO₃), and sodium dodecyl sulfate (C₁₂H₂₅SO₄Na) with the composition is shown in Table 1. The electrolyte prepared with distilled water, at a constant temperature of 40-60 ºC, and pH value of solution adjusted to 2.5-5 using HCl and NaOH reagent. The electrolyte agitated using a magnetic stirrer. A nickel plate substrate acted as anode with the size of 50 mm x 10 mm, whereas a FeCrAl acted as cathode with the size of 40 mm x 20 mm. The distance between anode and cathode is set at 25 mm. The electroplating was conducted for several variation times of 15, 30, 45, 60 and 75 minutes, current density of 8 A/dm², 3 g γ-Al₂O₃ inserted into the beaker for each sample and total surface area of 1600 mm² in two sides. The schematic diagram of electroplating process is shown in Figure 2.
Table 1. Chemical composition of electrolyte

| Electrolyte solution | Composition (%) |
|----------------------|-----------------|
| NiSO₄·6H₂O           | 51.25           |
| NiCl₆H₂O             | 0.85            |
| H₂BO₃                | 5.12            |
| C₁₂H₂₅SO₄Na          | 42.73           |

Figure 2. Schematic diagram of electroplating process

There is a correlation of the surface roughness in CATCO regarding to the toxic emission flow through the CATCO. Surface roughness is can effect to the chemical reaction (Zhao et al., 2001) which automatically effect to the result of exhaust emission of the CATCO. In this research will investigate which effective between low or high surface roughness in field of reducing emission. Surface roughness machine (Mitutoyu SJ-400) is arranged by $\lambda_c$ of 0.8 mm, $\lambda_c$ of 2.5 µm, eva-L of 4 mm and Roughness range of 800.

3. Results and Discussions

3.1 Surface Roughness Analysis

Surface roughness analysis were performed to coated and uncoated FeCrAl substrate by $\gamma$-Al₂O₃. From the surface roughness machine can be observed surface roughness (Ra) of material and surface roughness mean squared (Rq). Ra of raw material and coated material (UB, UBdEL, UB+EL and EL samples) are listed in Table 2. Surface roughness analysis is measure for 3 times in order to investigate real surface roughness of coated and uncoated material. Ra of raw material show in constant value for 0.02 µm while for coated material show in fluctuation value. The lowest number of Ra value for UB, UBdEL, UB+EL and EL samples are 0.11 µm, 0.81 µm, 0.64 µm and 0.69 µm which located at UB 1.5 h, UBdEL 45 min, UB+EL 30 min and EL 30 min, respectively. Highest Ra value is located at UB 3 h, UBdEL 75 min, UB+EL 75 min and EL 15 min for 0.21 µm, 2.17 µm, 1.63 µm and 1.11 µm, respectively. Different treatment has shown he different optimum treatment time such as for UB is 1.5 h, UBdEL is 45 min, UB+EL is 30 min and EL is 30 min. It may caused by $\gamma$-Al₂O₃ powder as coating material show decreasing particle size up to optimum time for each treatment and continued by small agglomeration phenomena which lead to Ra increment. It supported by previous researcher [10] that higher ultrasonic process will produce smaller surface roughness of material. However, there is certain limit ultrasonic time which caused the agglomeration of washcoat material. Therefore, in ultrasonic time of 45 minutes and above shown the rougher surface roughness. It also may caused by the washcoat material leading to reach the deformation plastic area (Feriyanto et al., 2017a). Level of surface roughness of coated and uncoated material from fine to rough surface is raw material, UB samples, UB+EL samples, EL samples and UBdEL samples. In UB samples, $\gamma$-Al₂O₃ powder is not fully embedded by high cavitations bubbles which led to small amount of washcoat material into FeCrAl substrate. In UB+EL samples shown the potential coating technique to produce fine surface roughness in FeCrAl substrate through combination of ultrasonic and electroplating methods due to beside washcoating, ultrasonic also purposed to cleaning FeCrAl substrate from other chemical [11]. Therefore, the washcoat material more intents to embedded into FeCrAl substrate.
Table 2. Surface roughness of coated and uncoated FeCrAl substrate

| Sample designation | Ra Test 1 (µm) | Ra Test 2 (µm) | Ra Test 3 (µm) | Average (µm) |
|--------------------|---------------|---------------|---------------|-------------|
| Raw                | 0.02          | 0.02          | 0.02          | 0.02        |
| UB 1 h             | 0.14          | 0.13          | 0.15          | 0.14        |
| UB 1.5 h           | 0.1           | 0.12          | 0.1           | 0.11        |
| UB 2 h             | 0.13          | 0.13          | 0.12          | 0.13        |
| UB 2.5 h           | 0.13          | 0.12          | 0.14          | 0.13        |
| UB 3 h             | 0.27          | 0.17          | 0.19          | 0.21        |
| UBdEL 15 min       | 1.41          | 2.35          | 1.08          | 1.61        |
| UBdEL 30 min       | 1.07          | 1.22          | 1.56          | 1.28        |
| UBdEL 45 min       | 1.05          | 1.04          | 0.34          | 0.81        |
| UBdEL 60 min       | 1.73          | 1.59          | 2.21          | 1.84        |
| UBdEL 75 min       | 1.65          | 3.01          | 1.85          | 2.17        |
| UB 1h+EL 15 min    | 0.87          | 1.13          | 1.18          | 1.06        |
| UB 1.5h+EL 30 min  | 0.97          | 0.6           | 0.35          | 0.64        |
| UB 2h+EL 45 min    | 0.97          | 0.88          | 0.74          | 0.86        |
| UB 2.5h+EL 60 min  | 1.56          | 1.73          | 0.86          | 1.38        |
| UB 3h+EL 75 min    | 2.7           | 1.56          | 0.62          | 1.63        |
| EL 15 min          | 1.27          | 1.04          | 1.03          | 1.11        |
| EL 30 min          | 0.8           | 0.7           | 0.56          | 0.69        |
| EL 45 min          | 0.85          | 0.92          | 1             | 0.92        |
| EL 60 min          | 1.25          | 1.05          | 0.9           | 1.07        |
| EL 75 min          | 0.79          | 1.06          | 1.27          | 1.04        |

Ra increment of treated samples shown a significant difference between lowest Ra and highest Ra. For UB shows the increment of 47.62%, UBdEL of 62.67%, UB+EL of 60.74% and EL of 37.84%. From these results shows that ultrasonic technique give a significant effect to the coating layer and it caused by agglomeration and decrement γ-\(\text{Al}_2\text{O}_3\) powder as compared to electroplating technique. Low current density and low voltage that performed in electroplating technique may cause insignificant effect to the Ra of coated material.

3.2 Surface Roughness Mean Square Analysis

In surface roughness machine also shows Rq value of coated and uncoated material which is shown in Table 3. Rq value is reflected from the Ra value. Therefore, when Ra is increased, Rq is increased as well. Ra and Rq is quite related to the CATCO product and its effectiveness on the exhaust emission reaction. That correlation regarding to the toxic emission flow through the CATCO where it can affect to the chemical reaction of exhaust emission of the CATCO. When surface roughness is low, it will extend the reaction time of exhaust emission which led to preferable on conversion process to the green emission of \(\text{CO}_2\) and \(\text{H}_2\text{O}\) forms [12].

The lowest Rq for each treatment where the lowest Rq is shown by UB 1.5 h followed by EL 30 min, UB+EL 30 min and UBdEL 45 min with the value of 0.49 µm, 3.02 µm, 3.7 µm and 4.9 µm, respectively. Fluctuation Rq is observed for coating material. It may caused by not uniformity of wall thickness of FeCrAl surface. It may influence to the non-uniformity temperature distribution and protection when it applied in high temperature of 1000-1400°C. That phenomenon may affect to the thermal stability and conversion efficiency of FeCrAl substrate in converting pollutant which is HC, NO\(_x\) and CO to the \(\text{H}_2\text{O}\) and \(\text{CO}_2\) [13,14].

Table 3. Surface roughness mean squared of coated and uncoated FeCrAl substrate

| Sample name | Rq Test 1 (µm) | Rq Test 2 (µm) | Rq Test 3 (µm) | Average (µm) |
|-------------|---------------|---------------|---------------|-------------|
| Raw         | 0.03          | 0.03          | 0.03          | 0.03        |
| UB 1 h      | 0.24          | 0.1           | 0.23          | 0.57        |
| UB 1.5 h    | 0.14          | 0.16          | 0.19          | 0.49        |
| UB 2 h      | 0.24          | 0.16          | 0.22          | 0.62        |
| UB 2.5 h    | 0.27          | 0.14          | 0.22          | 0.63        |
4. Conclusions
Surface roughness and coating thickness were investigated by Surface roughness machine (Mitutoyu SJ-400) and Alpha-Step IQ Surface Profiler. Surface roughness of UB samples in between 0.11 to 0.21 µm, UBdEL samples of 0.81 to 2.17 µm, UB+EL samples of 0.64 to 1.63 µm and EL samples of 0.69 to 1.11 µm. The finest surface of each technique are located at UB 1.5 h, UBdEL 45 minutes, UB 1.5 h+EL 30 minutes and UB 30 minutes. That data is supported by coating thickness of coated FeCrAl substrate where UB samples in between 2-2.8 µm, UBdEL samples of 4.1 to 5 µm, UB+EL samples of 9.1 to 12 µm and EL samples of 6.2 to 11.3 µm. To fulfill the requirement of industry that the product need to be fine surface and appropriate coating thickness, the appropriate techniques is shown by UB+EL technique especially for UB+EL 30 minutes.

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