

NiO ELECTRO-DEPOSITION TECHNIQUE OF γ -Al₂O₃ WASHCOAT ON FeCrAl SUBSTRATE BY USING SULPHAMATE TYPE SOLUTION

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Abstract

Electro-deposition process to develop surface layer on the substrate material in catalytic converter (CATCO) become interesting area due to that process was purposed to improve the physical properties of substrate material. Currently, precious metals such as Platinum (Pt), Palladium (Pd), and Rhodium (Rd) were used due to excellent oxidation resistant but it limited and easily oxidized. Therefore, Nickel Oxide (NiO) catalyst used as electro-deposition material. NiO electro-deposition technique that called by EL was conducted by using NiO as cathode and FeCrAl as substrate and γ -Al₂O₃ as washcoat material. This technique was performed by varying times of 15, 30, 45, 60 and 75 minutes and current density of 8 A/dm². The results shows that coating layer of NiO and γ -Al₂O₃ has been developed on surface of FeCrAl substrate. The coating layer increased the surface roughness which showed by surface morphology data that coated FeCrAl substrate has uneven surface and some particles has been embedded on that surface. The composition of raw material consisted of Fe for 74.13wt%, Cr of 20.25 wt% and Al of 5.62 wt%. Meanwhile, for composition of EL samples was 52.56-63.54wt% for Fe element, Al for 3.56-11.89 wt%, Cr for 14.97-18.56 wt%, O for 2.47-11.78 wt%, C for 8.33-11.85 wt%, Na for 0.11-0.48 wt% and Ni for 0.17- 1.58 wt%. Higher elements of the EL samples potential to improve the thermal stability at high temperature due to CATCO operate at high temperature of 600-850^oC and in extreme condition.

Keywords: Electro-Deposition, Catalytic Converter, Electrolyte, Thermal Stability

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1. Introduction

A washcoat is a catalyst carrier with high surface area. It acts as an intermediate layer in order to oxidation and reduction processes occur[2]. 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 μ m of thickness with a high surface area on the top of substrate.

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[3-7]. The existing of excellent oxidation catalyst materials was usually based on the precious metal Platinum (Pt), Palladium (Pd), and Rhodium (Rd). However,

those materials are expensive, limited supply[8], high specific activity, easily oxidized and easily broken at temperature of 500-900^oC[9]. The cheaper ranges of oxides (CuO, V₂O₅, NiO, MoO₃, and Cr₂O₃) compared to precious metals are being investigated as an alternative catalyst[9].

2. Review of Electroplating Method

Coating technology is used to protect a surface from environmental attack. Coating is applied to the organic, inorganic or metallic in order to extend the product life and to reduce surface change.

Some coating technology for coating the substrate has been investigated by previous researchers such as dip coating[11], co-precipitation, spray-pyrolysis and sol-gel methods[12], electrophoretic deposition[13], aluminizing technique[14] and solution combustion synthesis (SCS)[15]. Those techniques are successfully conducted, however there are some limitations in applying the catalyst which is in the form of powder.

Currently, electroplating technique is one of the most promising techniques in coating.

There are some electroplating materials which are used in previous research such as Cu, Zn, Cr, Sn and Ni[16]. However, coatings material by Cu, Zn, or Zn-Sn will not be able to withstand friction and wear in automobile exhaust system applications. That materials are useful only as an intermediate plating layer. Directly plating Ni into the alloys is desirable. Current methods are limited to electroless plating using nickel citrate, nickel carbonate, or nickel sulphate[2,16]. Ni compound have been used in wide application both of research and industrial application in many different forms. Ni is broadly used in industrial because its unique temperature resistance and superior corrosion resistant properties to produce stainless steel, non-ferrous alloy, electroplating, Ni based super-alloy, batteries and catalyst. Ni is an element located in tenth group and its atomic number is 28 and atomic mass is 58.71 g/mol[17]. Ni electroplating is the current method to achieve high corrosion resistant and higher hardness[10]. In addition, nickel electroplating is special surface finishing technology which commonly used in electronic and automobile industries[17]. According to Kanani[10] Nickel has Vickers micro-hardness (HV) number for metallurgical of 150 HV and for electrodeposition of 500 HV. Its parameter can be applied in various types of coating formed by metal finishing such as coating of metallic, multi-layered, alloy coating, composite coating and electroforming.

2.1 The Principles of Electroplating

One of the materials which is used as a buffer layer for surface coating to improve the conductivity

and corrosion resistant is nickel. Nickel plating requires the passage of direct current between electrodes that immersed in a conductive and aqueous solution of nickel salt[13]. The flow of direct current causes the anodes to dissolve and the cathode to become covered with Ni. The Ni in electrolyte solution is present in the form of divalent positively charged ions (Ni²⁺). The positive ion reacts with 2 electrons (2e) when the current flow and it converted to metallic Ni at the cathode surface[17]. Meanwhile, Ni in anode is dissolved to form divalent positive ions charged which enter the electrolyte solution. The discharge of Ni ions is not only occur in the cathode when the current flow but it also consumed in discharge of hydrogen ions. The nature of electrolyte is influence to the reduces of cathode efficiency from 100% to 92-97% of Ni deposition[18].

2.2 Solutions for Nickel Electroplating

Bright nickel solution is one of the technologies of decorative nickel plating. Other functions of decorative nickel solution are to develop multilayer nickel coating and as micro-discontinuous chromium. It influences to the corrosion resistance of the FeCrAl material without increment of deposit thickness[2]. Fundamental of bright nickel-plating solution are watt formula as listed in Table 1. It consists of organic and other additive in order to produce full bright without mechanical finishing. Coating thickness was influenced by portion of addition agent molecules which effect to the hardness and fine-grain coating. Typical properties of the bright nickel deposition such as Elongation of 2 to 5 (%), Vickers hardness of 100 g load of 600 to 800 HV; internal stress of 12 to 25 (MPa)[2].

Table 1. Fundamental of bright nickel plating solution[2]

Chemical solution and parameter	Electrolyte Composition (g/L)		
	Watts Nickel	Nickel Sulphamate	Typical Semi-Bright Bath
Nickel Sulfate, NiSO ₄ .6H ₂ O	225 to 400		300
Nickel Sulfamate Ni(SO ₃ NH ₂) ₂		300 to 450	
Nickel Chloride NiCl ₂ . 6H ₂ O	30 to 60	0 to 30	35
Boric Acid H ₃ BO ₃	30 to 45	30 to 45	45
Operation Conditions			
Temperature (°C)	44 to 66	32 to 60	54
Agitation	Air or mechanical	Air or mechanical	Air or mechanical
Cathode Current Density (A/dm ²)	3 to 11	0.5 to 30	3 to 10
Anodes	Nickel	Nickel	Nickel
pH	2 to 4.5	3.5 to 5	3.5 to 4.5
Mechanical Properties			
Tensile strength (MPa)	345 to 485	415 to 610	-
Elongation (%)	10 to 30	5 to 30	8 to 20
Vickers Hardness (100) g load	130 to 200	170 to 230	300 to 400
Internal stress (MPa)	125 to 185 (tensile)	0 to 55 (tensile)	35 to 150 (tensile)

2.3 Thermal Analysis of Nickel Electroplating Product

Electrodeposition process of hybrid coating was investigated by Wua et al.,[19]. Electrodeposition material of Co–Ni–Al₂O₃ using sulphamate type of electrolyte is applied into the electroplating process where the effect of ratio on the composition, pH, morphology and magnetic properties of Co–Ni–Al₂O₃ thin film from a glycine bath is investigated. The results shows that the appropriate condition to obtain a good coating by maximizing alumina content. Therefore, Al₂O₃ potential as electrodeposition material on FeCrAl substrate[20,21]. Moreover, γ -Al₂O₃ powder is more challenged to explore as coating material as investigated by Kim et al.[22]. The electroplating wastes is containing some heavy metals such as nickel, copper, zinc, chromium, iron, aluminium which usually generated as a by-product[17].

2.4 Materials and Methods

The raw material of this study are FeCrAl foils as substrate, γ -Al₂O₃ as a washcoat material, Nickel oxide (NiO) plate and electrolyte solution.

2.5 NiO electroplating on FeCrAl substrate

Electroplating process is conducted through some components such as electrolyte, anti-pitting agent, anode and cathode. Sulphamate type which consists of nickel (ii) sulphate 6-hydrate (NiSO₄.6H₂O), nickel (ii) chloride (NiCl₂.6H₂O), boric acid (H₃BO₃), and sodium dodecyl sulfate (C₁₂H₂₅OSO₃Na) with the composition is shown in Table 2 was used as the electrolyte medium. The electrolyte prepared with distilled water, at a constant temperature of 40±5°C, and pH value of solution adjusted to 5 using HCl and NaOH reagent. The electrolyte agitated using a magnetic stirrer. A nickel (Ni) 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 was adjusted 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. Drying process was performed after electroplating process at temperature of 60°C for 12 hours.

Table 2: Chemical composition of electrolyte

Electrolyte solution	Composition (%)
NiSO ₄ .6H ₂ O	51.25
NiCl 6H ₂ O	0.85
H ₂ BO ₃	5.12
C ₁₂ H ₂₅ SO ₄ Na	42.73

The calculation of the current of DC power supply in order to obtain the various current densities of 8 A/dm² is calculated using Eq. 3.1:

$$J = \frac{I}{A} \quad (3.1)$$

where

J: current density

I: current

A: total surface area

The calculation of the current of DC power supply is:

$$I = J \cdot A = 8 \text{ A/dm}^2 \cdot 0.16 \text{ dm}^2 = 1.28 \text{ A}$$

The characterization of this research was conducted by scanning electron microscopy (SEM) that coupled by energy dispersive spectroscopy (EDS) in order to investigate the surface morphology, composition and cross section of coated FeCrAl substrate. In cross section analysis was started by few stages which are cutting, mounting, grinding and polishing process. Mounting process was conducted using cold mounting mixed between resin and hardener ratio of 4:1. The grinding process was conducted using Silicon Carbide (SiC) paper in hand grinder machine from 320, 400, 600 and 2000 grit. The polishing was carry out by using polishing machine with addition of diamond paste (6 μ m, 3 μ m and 1 μ m). Moreover, the sample was coated by Platinum (Pt) coating using JOEL JFC-1600 Auto fine coater that conducted by holding time of 60 second with the vacuum mode in Pressure of below than 5 Pa. Cross section analysis were conducted by using magnification of 700 times with 15 kV and probe current of 50 as well as working distance of 15 mm.

3. Results and Discussion

3.1 Surface Morphology of Raw Material

The morphology study on FeCrAl foil, γ -Al₂O₃ powders and Ni plate are shown in Fig. 1 to 3, respectively. That materials are used as raw materials in this study and as main material for CATCO fabrication. Microstructure of FeCrAl foils as shown in Fig. 1 which the uneven surface was observed. There are small percentage of O in FeCrAl of 1.88

wt% that potential to develop Cr_2O_3 oxide scale which promote high thermal stability of materials. A high protective oxide scale formed by inside diffusion of chromium (Cr^{2+}) and it will improve the

mechanical properties in high temperature as well as will reduce the possibility of phase transformation from ductile to brittle[23].

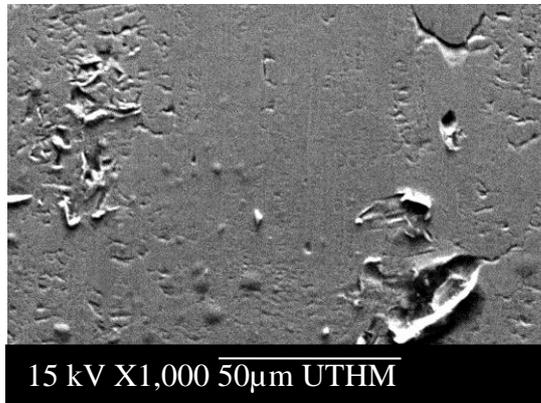


Fig. 1. Microstructure analysis of FeCrAl foil

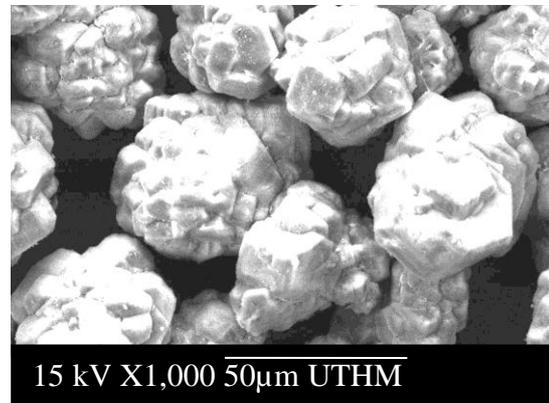


Fig. 2. Microstructure analysis of $\gamma\text{-Al}_2\text{O}_3$ powders

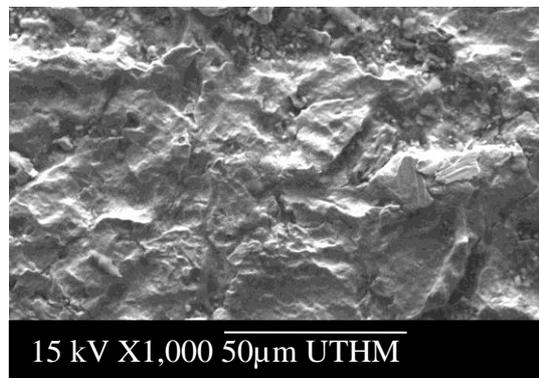


Fig. 3. Microstructure and composition analysis of NiO plate

Fig. 2 shows the morphology and composition analysis of $\gamma\text{-Al}_2\text{O}_3$ powders which conducted by 1,000 times. $\gamma\text{-Al}_2\text{O}_3$ powders shows irregular shape with fully solid particle. However, each particle consists of many rectangle shapes which bonding each other that cause some cavities in boundaries. $\gamma\text{-Al}_2\text{O}_3$ powders have higher brittleness compared with Al_2O_3 powder due to $\gamma\text{-Al}_2\text{O}_3$ powders is formed in high temperature at the range of 900 to 1394°C. Brittleness is needed because $\gamma\text{-Al}_2\text{O}_3$ is used as washcoat material which work in high temperature and function as protective oxide layer of FeCrAl substrate that developed by coating activity. Fig. 3 shows the NiO plate in uneven surface as a catalyst.

NiO plate was consists of Ni and O as main elements that contribute to the high thermal stability of FeCrAl as a substrate material through coating process due to high protective oxide layer on FeCrAl substrate. According to Redjidal et al.[23] that Ni give a major influence on the improving thermal stability by combination with oxygen and Cr_2O_3 oxide scale.

3.2 Surface Morphology of EL Sample

The EL samples that coated by $\gamma\text{-Al}_2\text{O}_3$ as washcoat material, NiO as anode material and FeCrAl as a substrate with various EL times of 15, 30, 45, 60 and 75 min is shown in Fig. 4 (a-e).

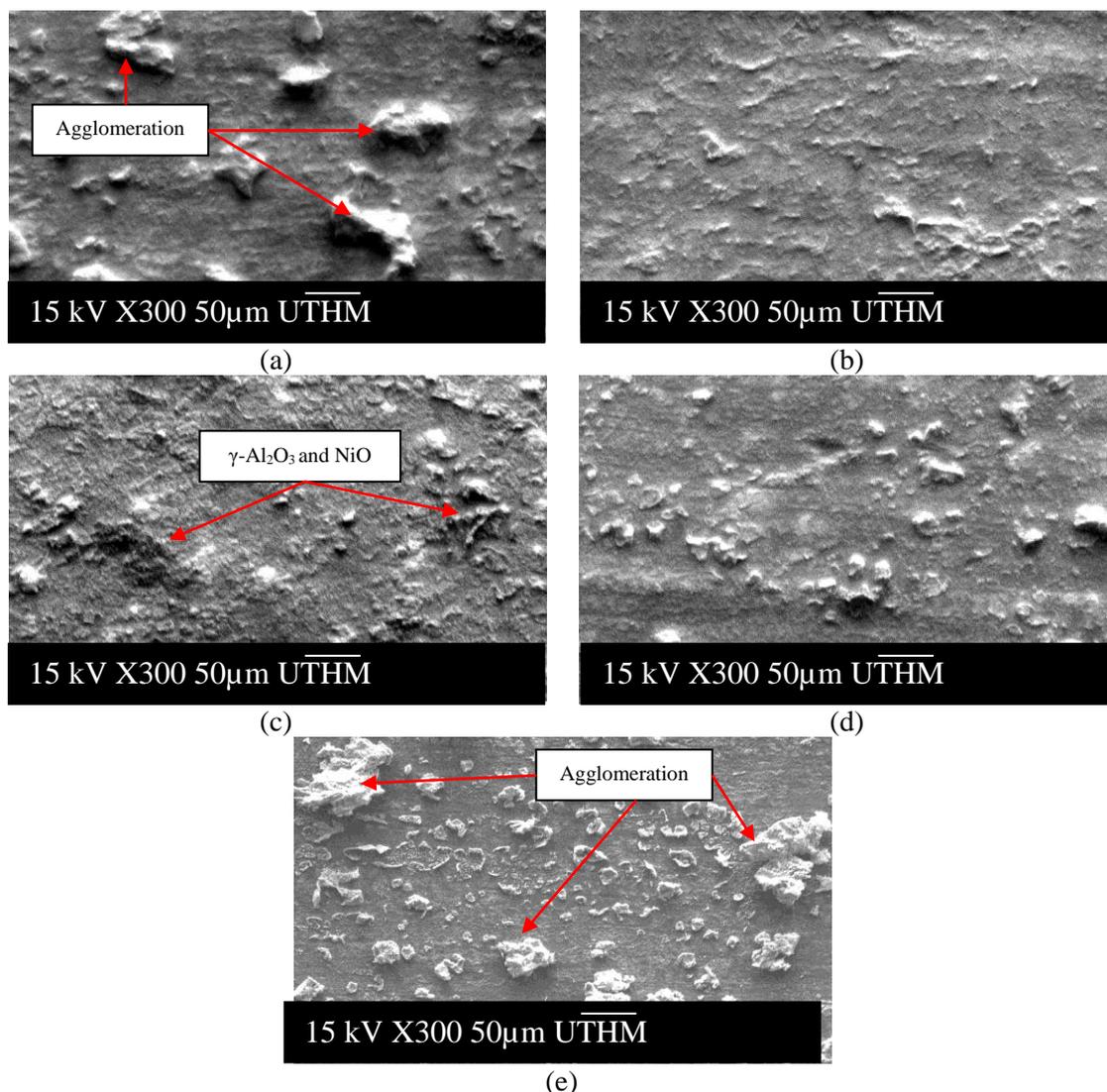


Fig. 4. Morphology of (a) EL 15 min; (b) EL 30 min; (c) EL 45 min; (d) EL 60 min and (e) EL 75 min

The surface morphology of the EL samples shows that there are γ - Al_2O_3 , NiO that embedded on FeCrAl surface that shown by many particles embedded on the surface. The bonding activity of coating material and catalyst has been occurred that promote high coating thickness of EL samples up to $11.3\mu\text{m}$ that located at EL 75min. This samples consists of uneven surface due to coating layer of γ - Al_2O_3 , NiO and other chemical which come from sulphamate type electrolyte solution.

Fine surface morphology after EL technique has been observed on the surface of FeCrAl substrate even there are some agglomerations of γ - Al_2O_3 and NiO that showed by large particle on the surface morphology was inevitable. Large particle promotes the sample has relatively high O content due to there are some oxygen cavities on the coating material and catalyst that supported by EDS data that the highest O content was 11.78 wt%.

3.3 Composition Analysis of Raw Material and EL samples

Composition analysis of FeCrAl substrate as raw material and coated FeCrAl by γ - Al_2O_3 powder using ultrasonic technique as listed in Table 3. Raw material mainly consists of 3 major elements of Fe, Cr and Al with Fe element was dominated for 74.13wt%. This material has high temperature range operation up to 1400°C because from that composition potential to develop protective oxide compound such as Cr_2O_3 , Al_2O_3 and FeO.

This technique was conducted by sulphamate type solution, NiO as anode and FeCrAl as cathode as well as γ - Al_2O_3 as coating material. The composition of EL samples mainly consists of 7 elements such as Fe, Cr, Al, O, C, Ni and Na elements. The range of composition for each elements was 52.56- 63.54wt% for Fe element, Al for 3.56-11.89 wt%, Cr for 14.97-18.56 wt%, O for 2.47-11.78 wt%, C for 8.33-11.85 wt%, Na for 0.11-

0.48 wt% and Ni for 0.17- 1.58 wt%. The element was dominated by Fe and Cr elements with high weight percentage in each sample. In addition, Na and Ni also present in EL samples which play the important role when the coated material is performed in high temperature of 1000°C[24]. Those elements develop some compounds such as FeCrAl, FeO, γ -

Al_2O_3 , FeCr_2O_3 , NiO, NiAlO_4 , NiCr_2O_4 and Na_2O . Interaction between γ - Al_2O_3 and FeCrAl substrate is related to the diffusion activity where the alumina scale grows exclusively by oxygen diffusion along grain boundaries. Higher Al content and O content will promote higher diffusion phenomena of the samples[25,26].

Table 3. Composition analysis of EL samples

Sample name	Elements (Wt%)						
	O	C	Al	Cr	Fe	Na	Ni
Raw material			5.62%	20.25%	74.13%		
EL 15 min	2.47%	11.85%	3.56%	18.43%	63.04%	0.48%	0.17%
EL 30 min	2.96%	8.33%	4.92%	18.56%	63.54%	0.11%	1.58%
EL 45 min	5.2%	10.75%	4.13%	16.47%	62.66%	0.28%	0.52%
EL 60 min	11.78%	10.65%	7.74%	16.09%	53.83%	0.42%	0.51%
EL 75 min	10.46%	9.23%	11.89%	14.97%	52.56%	0.14%	1.03%

Coating activity and coating layer believe has high diffusion of γ - Al_2O_3 to the FeCrAl substrate since there is cation and anion transport which led to higher diffusion coefficient[27]. The compounds that have been observed shown promising improvement on substrate properties in high thermal stability at high temperature application up to 1000°C.

4. Conclusions

The surface morphology, composition and cross section analysis were successfully investigated. In surface morphology analysis achieve that NiO and γ - Al_2O_3 has been deposited on to FeCrAl substrate but there are some agglomerations was occurred. The composition of raw material consists of Fe, Cr and Al while EL samples consists of Fe, Cr, Al, C, O, Ni and Na elements. Higher elements of EL samples caused by deposition process of NiO as Cathode, γ - Al_2O_3 as washcoat material, sulphamate solution as electrolyte. Deposition process was occurred on FeCrAl substrate that showed by cross section analysis. Coating layer of NiO and γ - Al_2O_3 has been observed with non-uniformity thickness layer. For further study should be investigate the compound analysis of the coated FeCrAl substrate by NiO and γ - Al_2O_3 in order to obtain the protective oxide layer that may developed during NiO-deposition process

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