

Coating

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COATING THICKNESS ANALYSIS OF DEPOSITED FeCrAl SUBSTRATE BY γ -AL₂O₃ THROUGH NiO-ELECTROPLATING

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Abstract – Nickel Oxide (NiO) have widely applied in industrial and automotive to forming oxide layer on the substrate due to it have high thermal stability and corrosion resistant. However, NiO was not directly diffused to the substrate via co-precipitation, spray-pyrolysis and sol-gel methods. Therefore, NiO-electroplating was performed in coating FeCrAl substrate by γ -Al₂O₃ powder. This method was conducted using various time of 15, 30, 45, 60 and 75 minutes, a current of DC power supply is 1.28A and sulphamate type solution. From the data shows that smallest surface roughness (Ra) of NiO-electroplating (EL) samples located at EL 30 min sample for 0.69 μ m. Ra of raw material was lower than coated samples due to it performed by coating activity on the substrate. It supported by coating thickness analysis that treated samples has a higher coating thickness up to 11.3 μ m. EDS analysis observed some elements on coated and uncoated samples such as Carbon (C) of 4.62-20.67%, Oxygen (O) of 6.16-19.62%, Nickel (Ni) of 6.76-25.14%, Iron (Fe) of 23.96-48.53%, Sodium (Na) of 2.21-9.95%, Chromium (Cr) of 7.97-15.59% and Aluminum (Al) of 2.17-3.12%. Those elements promote to develop a protective oxide layer on FeCrAl at a high temperature of 1000 °C.

Keywords: Electroplating; Coating thickness; NiO; FeCrAl

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INTRODUCTION

Nickel oxide compound have several forms which applied in commercial and industrial application. There are several purposes of NiO such as for production of non-ferrous alloys, nickel based super-alloys, batteries, electroplating, catalyzing and stainless steel because it's had many superiorities in high thermal and corrosion resistant properties (Eyupoglu & Recep, 2015). The substrate material should meet a requirement of high thermal stability at high temperature operation. Therefore, the coating process in order to make a protective oxide layer need to be conducted.

There are several famous coating materials such as Al₂O₃ (Ruigang, 2013), α -Al₂O₃ (Casella et al., 1998), CeO₂ (Ruigang, 2013), CeO₂-Mn (Liwei, Shen & Wang, 2007), SiO, SiO-Al₂O₃ (Kalam et al., 2009) and γ -Al₂O₃ (Putrasari et al., 2010). However, it has several limitations such as trapping sulfur, segregation of alumina layer at high temperature, low heat transfer, low deposition activity of γ -Al₂O₃. In particular, the properties of the washcoat material is well controlled by using γ -Al₂O₃ suspension as compared to other coating materials. According

to Santos and Costa (2008) mention that the washcoat material which commonly used was γ -Al₂O₃ powders with high surface area of 100 m²/g. According to Ersson and Jaras (2006) the γ -Al₂O₃ is not change while extended temperature operation of the catalyst in Honeycomb Monolith substrate at the range between 1000 and 1400°C. Therefore, nowadays γ -Al₂O₃ become interesting material because it has higher surface area as compared to other coating material, high temperature stability up to 1400 °C.

In coating activity, there are several common methods such as dip coating, co-precipitation, sol-gel, spray-pyrolysis, solution combustion synthesis (SCS), aluminizing and hydrothermal methods (Leman, Feriyanto & Sebayang, 2016). However, that methods rather complicated to applying the catalyst which is in powder form. Therefore, one of the promising techniques i.e. NiO-electroplating technique. NiO-plating have unique pattern in surface finishing technology and its widely used in automobile, ship, air space, machinery, jewelry, toy, defense and electronic industries (Nirvana, Shayma & Thaer, 2014). Basic principle of electroplating is

an electro-deposition for producing uniform, adherent coating and dense upon the metal or alloy surface by using electrical current (Nirvana, Shayma & Thaer, 2014). Most important part of electroplating process is located at electrolytic cell where the current is passed through bath containing electrolyte, cathode and anode. Main purposed of coating technique of FeCrAl alloys are to decorate surface substrate material and to protect the substrate in high temperature by protecting coating layer. FeCrAl substrate is selected because it has several advantages such as high corrosion resistant, thermal stability and high oxidation resistant. This material is used to replace the precious metal like Pt, Pd and Rd because limited supply, expensive, easily oxidized and easily broken at low temperature of 500-900°C (Bagus, Purwanto & Hadiyanto, 2015).

This study proposes two deposition material which are γ -Al₂O₃ coating material and

NiO plate in electroplating process. It purposed to develop the substrate that has high thermal stability by improving the coating thickness on

Effect of the current density on the deposit thickness is shown in Table 1. That calculated based on an assumption that nickel efficiency is 95% because most of the cases of Ni-electroplating used 95.5% as nickel efficiency (Sebayang et al., 2012). From the tables can be described that when the researchers willing to obtain certain wall thickness, the Ni-electroplating should be conducted using certain current density and certain holding time. However, wall thickness is highly depending on the uniformity distribution of current density. Moreover, wall thickness able calculated using coulometric method by referring ISO Standard 2177 and ASTM B 504.

Table 1. Nickel electrodeposition data - Based on 95.5% cathode efficiency (Sebayang et al., 2012)

Deposit Thickness µm	Time (min) to obtain deposit at various current density (A/dm ²)									
	0.5	1	1.5	2	3	4	5	6	8	10
2	20	10	6.8	5.1	3.4	2.6	2	1.7	1.3	1
4	41	20	14	10	6.8	5.1	4.1	3.4	2.6	2
6	61	31	20	15	10	7.7	6.1	5.1	3.8	3.1
8	82	41	27	20	13	10	8.2	6.8	5.1	4.1
10	100	51	34	26	17	13	10	8.5	6.4	5.1
12	120	61	41	31	20	15	12	10	7.7	6.1
14	140	71	48	36	24	18	14	12	8.9	7.1
16	160	82	54	41	27	20	16	14	10	8.2
18	180	92	61	46	31	23	18	15	11	9.2
20	200	100	68	51	34	26	20	17	13	10
40	410	200	140	100	68	51	41	34	26	20

METHOD

The electroplating activity of spontaneous reaction, the electron flow from the anode (oxidation) to the cathode (reduction). But in a non-spontaneous reaction occurred by energy supply from external sources. Electrons are forced from the external circuit at cathode to produce the reduction. The cathode is the negative electrode and is the black connection on the power supply. Electrons are drawn into the external circuit at the anode to produce the oxidation. The anode is the positive electrode and is the red connection on the power supply.

Any electrochemical cell can be galvanic or electrolytic depending on the direction in which the reaction is proceeding.

Electrolyte preparation

According to Ade (2012) they have been used sulphamate types as electrolyte which

consists of consisted of nickel sulphamate (Ni(SO₃NH₂)₂·4H₂O), nickel chloride (NiCl₂·6H₂O), boric acid (H₃BO₃) and sodium lauryl sulphate (C₁₂H₂₅SO₄Na) as electrolyte and it shown believing result in improve optimization of Ni-electroplating process.

Therefore, this research is conducted with sulphamate types with little difference composition of the chemical agent which purposed to more accelerate the deposition activity. This solution is consist of nickel (II) sulphate 6-hydrate (NiSO₄·6H₂O), nickel (ii) chloride (NiCl₂·6H₂O), boric acid (H₃BO₃), and sodium dodecyl sulfate (C₁₂H₂₅OSO₃·Na) with the composition is shown in Table 2. The electrolyte prepared using distilled water at a constant temperature of 60 °C, and pH of 2.5-5. The electrolyte agitated using a magnetic stirrer.

Table 2. Chemical composition of electrolyte (Rose, & Whittington, 2002)

Electrolyte solution	Composition (wt%)
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

NiO-electroplating set-up

A NiO plate was positioned as anode with the fixed size of 50 mm x 10 mm and FeCrAl was

positioned as cathode with fixed size of 40 mm x 20 mm. Fixed distance between anode and cathode also adjusted by 25 mm. The γ -Al₂O₃ mass of 5 g for each parameter where Ni-electroplating is conducted by 5 variation in holding time which are 15, 30, 45, 60 and 75 minutes, fixed current density of 8 A/dm². The schematic diagram of Ni-electroplating is shown in Fig. 1.

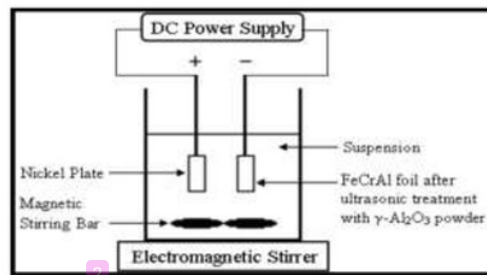


Figure 1. Schematic diagram of electroplating process

Current of DC power supply calculation

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. 1:

$$J = \frac{I}{A} \quad (1)$$

Where, J : The current density
I : The current
A : Total surface area

The calculation of the current of DC power supply is:

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

Coating thickness measurement

The thickness of the graphite paste electrode samples were measured using Alpha-Step IQ Surface Profiler (KLA Tencor Corporation) as shown in Fig. 2 with scan length of 5000 μ m and scan speed of 200 μ m/s.



Figure 2. Alpha-Step IQ Surface Profiler

RESULT AND DISCUSSION

Surface roughness analysis of raw material and NiO-electroplating samples in various holding time is shown in Fig. 3. Surface

roughness of raw material is even for 3 times measurement. surface roughness specifically is divided into 2 which are surface roughness (Ra) and surface roughness mean square (Rq). Ra of

raw material show in constant value for 0.02 μm while for coated material show in fluctuation value. The lowest and highest number of Ra value EL sample was 0.69 and 1.11 μm at EL 30 min and EL 15 min, respectively. That phenomena caused by $\gamma\text{-Al}_2\text{O}_3$ 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. The optimum time of EL technique in EL 30 min. In this parameter, the particle size has decreased due to high speed bubbles which generated shock waves through a mass of particles, high speed inter-particle collisions are occurred with the right angle of the collision.

Low current density and low voltage that performed in electroplating technique my cause

insignificant effect to the Ra of coated material. In addition, in surface roughness machine also shows Rq value of coated and uncoated material which is shown Fig. 4. 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 exhaust toxic emission flow through the CATCO that the surface roughness influence 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 CO_2 and H_2O forms (Specchia et al., 2004).

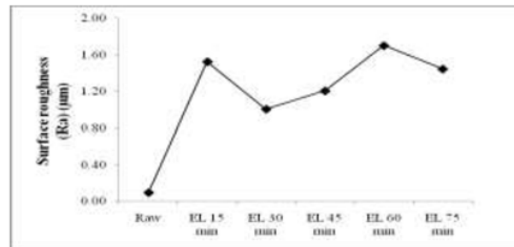


Figure 3. Ra value of raw material and EL samples

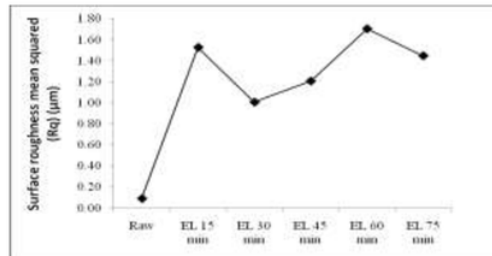


Figure 4. Rq value of raw material and EL samples

Fluctuation Rq is observed for coating material. It caused by not uniformity of wall thickness of FeCrAl surface. It supported by microstructure and cross section data that the surface of the coated FeCrAl substrate was not uniform.

In addition, influence to the non-uniformity temperature distribution and protection when it applied in high temperature of 1000-1400 $^{\circ}\text{C}$ also give an impact to the fluctuation of Rq.

That phenomenon effect to the thermal stability and conversion efficiency of FeCrAl substrate in converting pollutant which is HC, NO_x and CO to the H_2O and CO_2 .

Morphology and composition analysis

FeCrAl shown in Fig. 5 where uneven surface has been observed. It may be caused by oxygen trapping during fabrication process which led to little explosion when it heated. However, high percentage of Oxygen may promote Cr_2O_3 compound development by inside diffusion of chromium (Cr^{2-}) which become protective scale in producing high corrosion and oxidation resistant of FeCrAl substrate. EDS analysis of FeCrAl is consist of O:26.77, Al:5.29, Cr:14.83 and Fe:53.11 % mass. Strongest peaks are showed by Fe that the material have high ductility and relatively low Coefficient Thermal Expansion as properties of Fe (Redjidal et al., 2013).

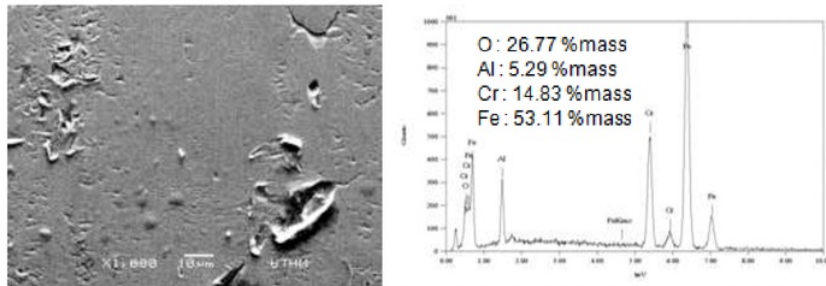


Figure 5. Morphology and composition analysis of FeCrAl foil Fig. 6 (a-e) show the EL samples that coated by γ - Al_2O_3 as washcoat material, NiO as anode material and FeCrAl as a substrate with various EL times of 15, 30, 45, 60 and 75 min.

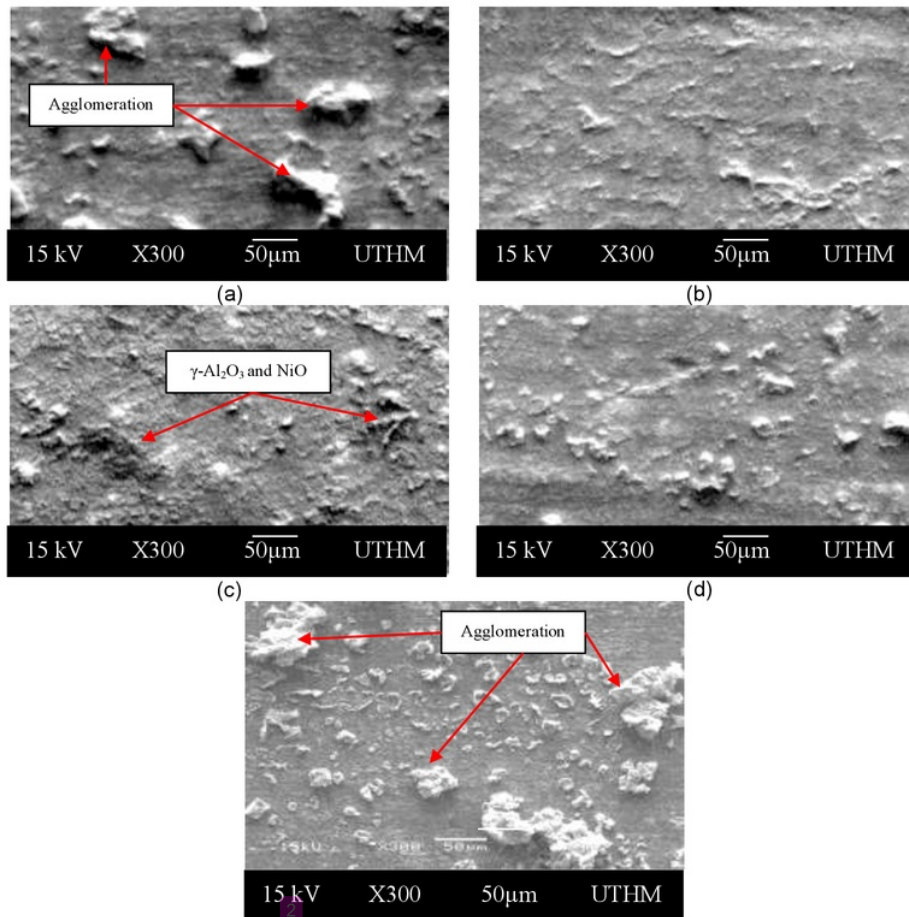


Figure 6. Morphology of (a) EL 15 min; (b) EL 30 min; (c) EL 45 min; (d) EL 60 min and (e) EL 75 min

The surface morphology of the EL samples shows that there are $\gamma\text{-Al}_2\text{O}_3$, NiO that embedded on FeCrAl surface that shown by many particles observed 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 μm that located at EL 75min. This samples consists of uneven surface due to coating layer of $\gamma\text{-Al}_2\text{O}_3$, 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 agglomeration of $\gamma\text{-Al}_2\text{O}_3$ 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. It supported by EDS data that the highest O content was 11.78 wt%.

Composition analysis of EL samples with various time of 15, 30, 45, 60 and 75 min as listed in Table 3. This technique was conducted by sulphamate type solution, NiO as anode and

FeCrAl as cathode as well as $\gamma\text{-Al}_2\text{O}_3$ 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 element 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 (Xiang *et al.*, 2004).

It approved by compound analysis that those elements develop some compounds such as FeCrAl, FeO, $\gamma\text{-Al}_2\text{O}_3$, FeCr_2O_3 , NiO, NiAlO₄, NiCr₂O₄ and NaO₂. Interaction between $\gamma\text{-Al}_2\text{O}_3$ 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 (Heesemann *et al.*, 1999).

Table 3. Composition analysis of EL samples

Sample name	Elements (Wt%)						
	O	C	Al	Cr	Fe	Na	Ni
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 $\gamma\text{-Al}_2\text{O}_3$ to the FeCrAl substrate since there is cation and anion transport which led to higher diffusion coefficient (Heesemann *et al.*, 1999). The compounds that has been observed shown promising improvement on substrate properties in high thermal stability at high temperature application up to 1000°C. It supported by thermal stability

analysis that EL samples have lower mass change of 3.99 mg as compared to raw material, UB and UBdEL samples. It promoted by protective oxide layer on FeCrAL that supported by cross section data that $\gamma\text{-Al}_2\text{O}_3$ and NiO was develop coating layer on FeCrAl substrate for coating thickness of 11.3 μm .

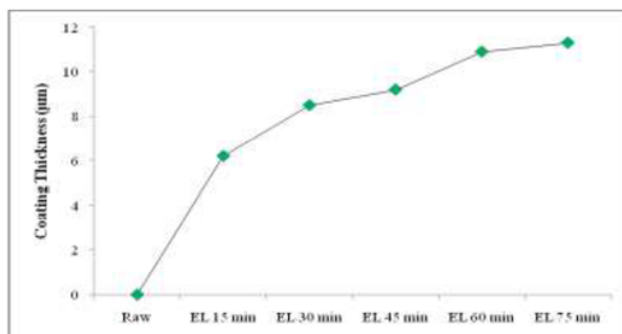


Figure 7. Coating thickness of coated and uncoated FeCrAl substrate

Fig. 7 shows the differences of coating thickness may be caused by non-uniformity distribution of current density performed. Raw material has 0 μm coating thickness due to it not performed by any coating methods. Higher coating time will increase $\gamma\text{-Al}_2\text{O}_3$ powder deposition. Low current density of 1.28 A/dm^2 and 12 V is performed in electroplating technique. It may be influenced to the $\gamma\text{-Al}_2\text{O}_3$ and Ni deposition but holding time shows high impact in increasing coating thickness. The lowest and highest coating thickness is shown by EL 15 min and EL 75 min for 6.2 μm and 11.3 μm , respectively. According to Graham and Hussey (2002), at temperature of 850 to 1100 $^\circ\text{C}$, scale formed in FeCrAl substrate is $\alpha\text{-Al}_2\text{O}_3$ phase and dominated by $\gamma\text{-Al}_2\text{O}_3$ phase with highly developed surface. Therefore, the highest coating thickness may have higher oxidation resistant and higher thermal stability at high temperature condition as compared to lower coating thickness.

CONCLUSION

Coating process by NiO-Electroplating were successfully conducted with various times of 15, 30, 45, 60 and 75 minutes and it also successfully characterize on surface roughness (R_a and R_q), morphology and composition. R_a and R_q of treated sample higher than raw material which caused by uneven coating layer of NiO and $\gamma\text{-Al}_2\text{O}_3$. However, EL 30 min has smallest R_a and R_q of 0.69 and 1.01 μm respectively which led to more retention time when exhaust gaseous flow through the FeCrAl substrate. However, in terms of coating thickness that EL 75 min has a highest coating thickness of 11.3 μm which may have more protective oxide layer in high temperature operation and extreme condition. From SEM and EDS analysis shows that uneven morphology caused by non-uniformity current density which cause non-uniform NiO and $\gamma\text{-Al}_2\text{O}_3$ layer on FeCrAl. Several chemicals observed which potential to develop protective scale like FeCrAl, FeO, $\gamma\text{-Al}_2\text{O}_3$, FeCr₂O₃, NiO, NiAlO₄, NiCr₂O₄ and NaO₂ which believe can improve the thermal stability.

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