RES MILITARIS
REVUE EUROPEENNE D ETUDES EUROPEAN JOURNAL OF MILITARY STUDIES
Social Science Journal

Coating Thickness Analysis Of Electroplated Fecral Material For Catalytic Converter Application

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Abstract

One of the most technological to develop and adhere to the catalysts on the FeCrAl substrate are based on electrophoretic deposition. The objectives of this research is to investigate the coating thickness on FeCrAl metallic material for catalytic converter. The electrolyte prepared with distilled water, at a constant temperature of 40 ± 5^0C, and pH value is 5 using HCl and NaOH reagent. A nickel (Ni) plate substrate acted as anode with dimension of 50 mm x 10 mm, whereas a FeCrAl acted as cathode with dimension of 40 mm x 20 mm. The electroplating was conducted for several times of 15, 30, 45, 60 and 75 minutes, current density of 8 A/dm^2 and 3 g γ-Al_2O_3 inserted into the beaker for each sample. Coating thickness analysis on cross section of the coated samples were carried out using Scanning Electron Microscope (SEM)-Energy Dispersive Spectroscopy (EDS). The result shows that the UBdEL samples has lowest coating thickness of 5µm, the coating thickness of Electroplated FeCrAl increased for 11.3µm and the highest coating thickness signed by UB+EL samples for 12µm. Higher coating thickness potential to increase the thermal stability due to protective oxide layer on FeCrAl substrate material.

Published/ publié in ***Res Militaris*** (resmilitaris.net), **vol.12, n°4, December Issue 2022**

Introduction

One of the material which 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 (Corni *et al.,* 2008). 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 (Ni+). The positive ion react with 2 electrons (2e) when the current flow and it converted to metallic Ni at the cathode surface (Eyupoglu and Kumbasar, 2015). 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 (Bai *et al.*, 2011).

Ceramic coating is mainly aimed to protect the material from the external chemical reaction and improve the oxidation resistance. There are several coating material used for coating FeCrAl alloy such as SiO, Al_2O_3, γ-.δ-, α- and θ-Al_2O_3. FeCrAl have high heat resistant in high temperature promoted by formation of aluminium oxide scale on FeCrAl surface. The compact structure with FeCrAl alloy for coating is Al_2O_3 however the thermodynamically stable is γ-Al_2O_3. According to Graham and Hussey (2002) that scale formed on FeCrAl alloy at temperature of 850 to 1100^0C which consist of α-Al_2O_3 phase and predominated by γ-Al_2O_3 phase which highly developed surface.

Thickness coating is very high contributed to the properties. When the thickness is too thin to be used as a catalyst carrier, when it applied in long time will lead to prospective properties become insufficient due to increasingly more aluminium take apart in scale formation and the FeCrAl is depleted by aluminium and Graham and Hussey, 2002). If scale chip is applied, the regeneration is more difficult. However, FeCrAl alloy scale forming is basically able to form permanently bonding ceramic coating material. Electrodeposition process of hybrid coating was investigated by Wua *et al.*, (2004). Electrodeposition material of Co–Ni–Al_2O_3 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–Al_2O_3 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, Al_2O_3 potential as electrodeposition material on FeCrAl substrate (Domínguez *et al.,* 2014 and Masakuni and Kenichi, 2015). Moreover, γ-Al_2O_3 powder is more challenged to explore as coating material as investigated by (Wu *et al,*. 2004 and Kim *et al.,* 2012). The main objective is to investigate the coating thickness and its homogeneity on FeCrAl metallic material for catalytic converter.

Methods

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 ($NiSO_4.6H_2O$), nickel (ii) chloride ($NiCl_2{\cdot}6H_2O$), boric acid (H_3BO_3), and sodium dodecyl sulfate ($C_{12}H_{25}OSO_3.Na$). The electrolyte prepared with distilled water, at a constant temperature of 40 ± 5^0C, 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/dm^2, 3 g γ-Al_2O_3 inserted into the beaker for each sample and total surface area of 1600 mm^2 in two sides. Drying process was performed after electroplating process at temperature of 60^{0}C for 12 hours. The schematic diagram of electroplating process were shown in Figure 1.

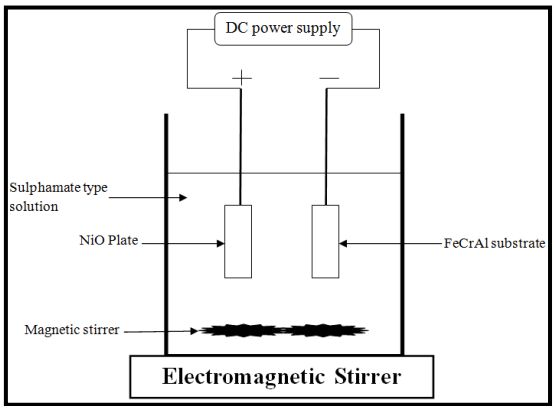

Figure 1 *Schematic diagram of electroplating process*

Cross section analysis

Coating thickness analysis on cross section of the coated samples were carried out using Scanning Electron Microscope (SEM)-Energy Dispersive Spectroscopy (EDS). The samples are cut, mounted, grinded and polished. Mounting process is 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 µm, 3 µm and 1 µm). The hand grinder and polishing machine is shown in Figure 2. The Back Scattered Mode (BSE) was used to obtain the high quality observation of coated coated FeCrAl since BSE provides more information on compositional contrast.

Figure 2 *Hand grinder machine and polishing machine*

Coating process is conducted by Platinum (Pt) coating using JOEL JFC-1600 Auto fine coater. This process is 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.

Results and Discussions

Cross section of raw material

Cross section analysis of the raw material shows that coating layer is not observed in this material. The composition of cross sectional view is mainly consists of Fe for 74.13 wt%, Cr for 20.25 wt% and Al for 5.62 wt%. Raw material was uncompleted by protective oxide layer due to no coating activity that performed in this sample which lead to low thermal stability at high temperature of 1000^0C. The data was supported by thermal stability analysis that raw material has highest mass change of 23.39 mg as compared with coated samples.

Cross section of UB samples

Cross section analysis of coated FeCrAl substrate by Ultrasonic bath technique is shown in Figure 3. This treatment is conducted in various ultrasonic time of 1, 1.5, 2, 2.5 and 3 h holding time. All UB samples is mainly consists of Fe, Cr, Al, O and C. Coating material which is γ-Al_2O_3 not fully embedded in FeCrAl substrate due to low frequency of ultrasonic bath and also uneven surface of FeCrAl as shown in microstructure analysis that led to defect to embedding mechanism. Figure 4.8 shows that ultrasonic time increase cause coating thickness increase as well.

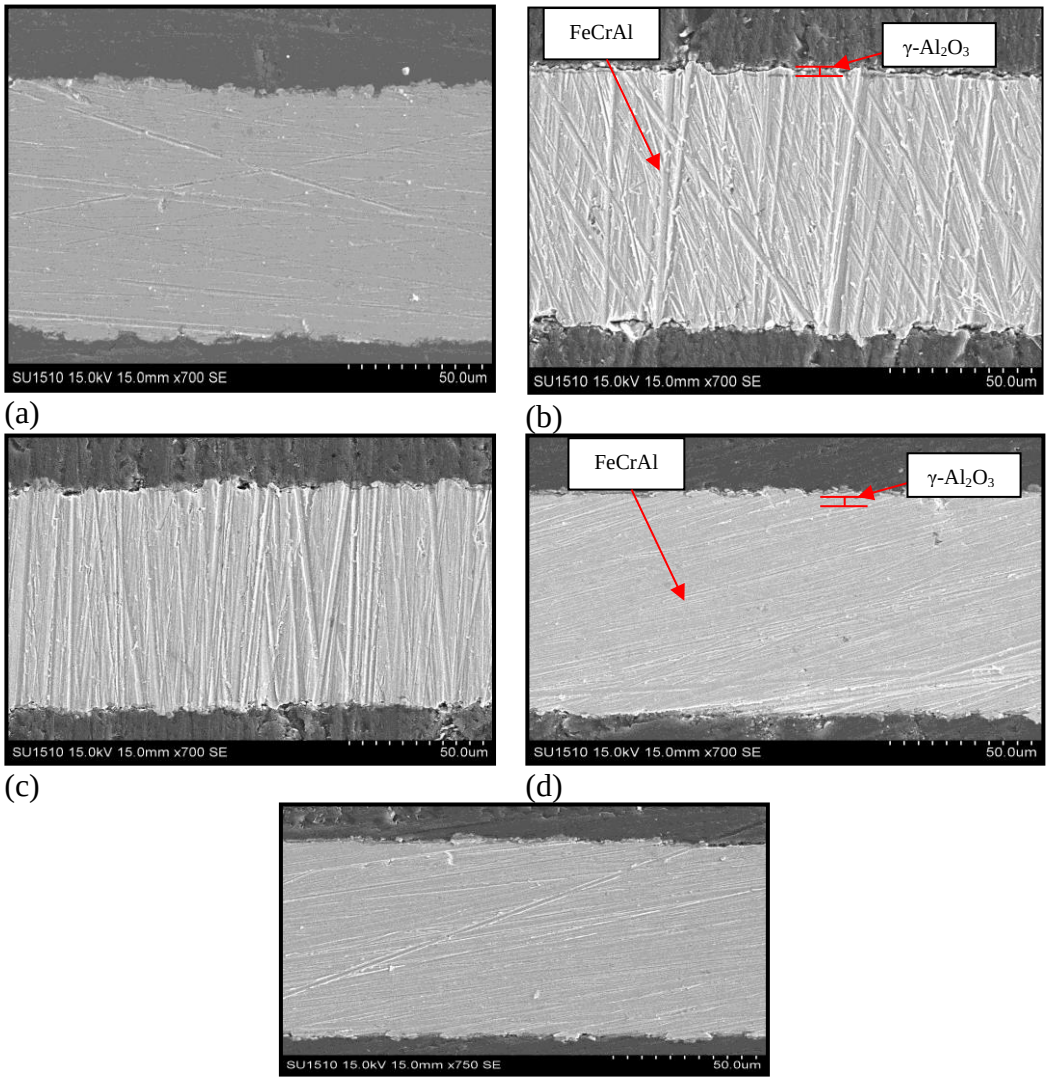

Figure 3. *Cross section analysis of (a) UB 1 h; (b) UB 1.5 h; (c) UB 2 h; (d) UB 2.5 h and (e) UB 3 h*

The correlation of cross sectional analysis and coating thickness is inline that UB 3 h is the highest coating thickness for 2.8 µm as compared with other UB samples. However, in thermal analysis shown the inversely proportional that UB 1.5 h is the highest thermal stability with lowest mass change of 18.75 mg that caused by an agglomerate coating material because the material led to deformation plastic which will effect to dense material. The agglomeration occur when micro bubbles or cavities which then collapse during compression that provide smaller γ-Al_2O_3. However, in certain time the agglomeration occurred on coating material which produce the larger particle that embed on substrate material (Kim, *et al.*,2012). Dense of coating material and fine material promote better properties regarding to high thermal stability. However, thin coating thickness of the UB samples cause prospective properties become insufficient when it applied at high temperature due to Al will more take a part in scale formation and FeCrAl is depleted by Al (Jung and Bae, 2015 and Dafit Feriyanto *et al.*, 2021).

Cross section of UBdEL samples

UBdEL samples means that the FeCrAl substrate is coated by using ultrasonic bath during electroplating technique and the cross section analysis is shown in Figure 4. This Figure consists coating layer of γ-Al_2O_3 and small diffusion of NiO plate which used as anode material. Therefore, the UBdEL samples is mainly consists of Ni, Fe, Cr, Al, Na, O and C. The highest Ni content of the sample is in UBdEL 75 minutes and the other sample in range of 0.16 to 0.82 wt%. In cross section analysis shows that there are some agglomeration of coated material and NiO catalyst on FeCrAl substrate as shown in UBdEL 45 min which led to material has relatively high surface roughness for 1.84 µm.

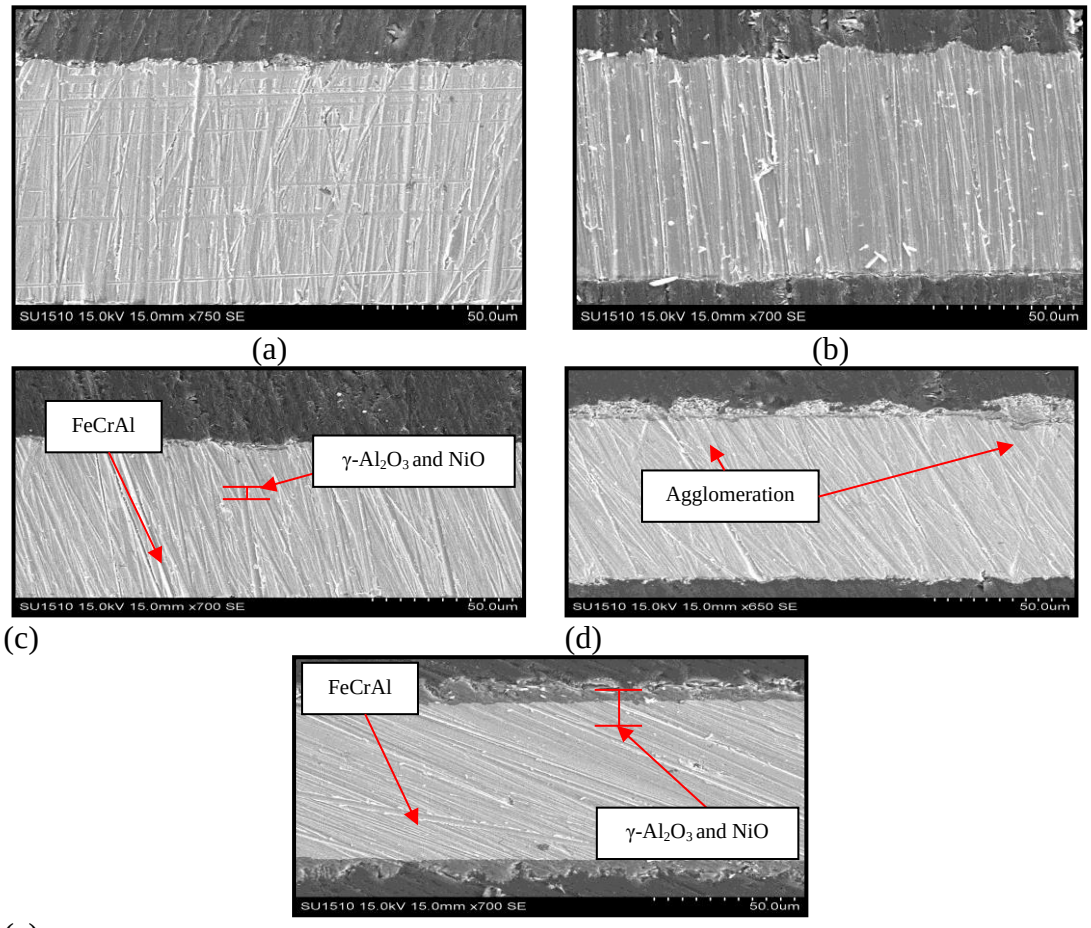

Figure 4. *Cross section analysis of (a) UBdEL 15 min; (b) UBdEL 30 min; (c) UBdEL 45 min; (d) UBdEL 60 min and (e) UBdEL 75 min*

From the cross section image shows that UBdEL time increase and coating thickness increase that supported by coating thickness data that the highest coating thickness of UBdEL samples is located at UBdEL 75 min for 5 µm. However, in UBdEL 60 and 75 min consists of high O and C content which indicated that in coating layer consists of higher oxygen cavities and containing an agglomerates γ-Al_2O_3 powder that decrease the protective at high temperature (Panta and Subedi, 2012). That data approved by thermal stability of UBdEL 60 and 75 min is tend to be lower than UBdEL 45 min. The coating thickness phenomena is close related to protective oxide layer development where dense and higher coating thickness increase physical properties of the material especially in extreme condition (Dafit Feriyanto *et al.*, 2020).

Cross section of UB+EL samples

Cross section analysis combination of ultrasonic bath and electroplating in coating FeCrAl by γ-Al_2O_3 powder and NiO catalyst which called as UB+EL with various time of 15, 30, 45, 60 and 75 minutes are shown in Figure 5. Prior to electroplating process, the sample is ultrasonically for preliminary coating or first deposition process. That technique potential to improve the coating thickness and physical properties of the FeCrAl substrate as compared with Ultrasonic bath and electroplating technique separately that provided by higher coating thickness and dense coating layer. Cross section analysis of the sample shows that UB+EL coating time increased as coating thickness increased as well.

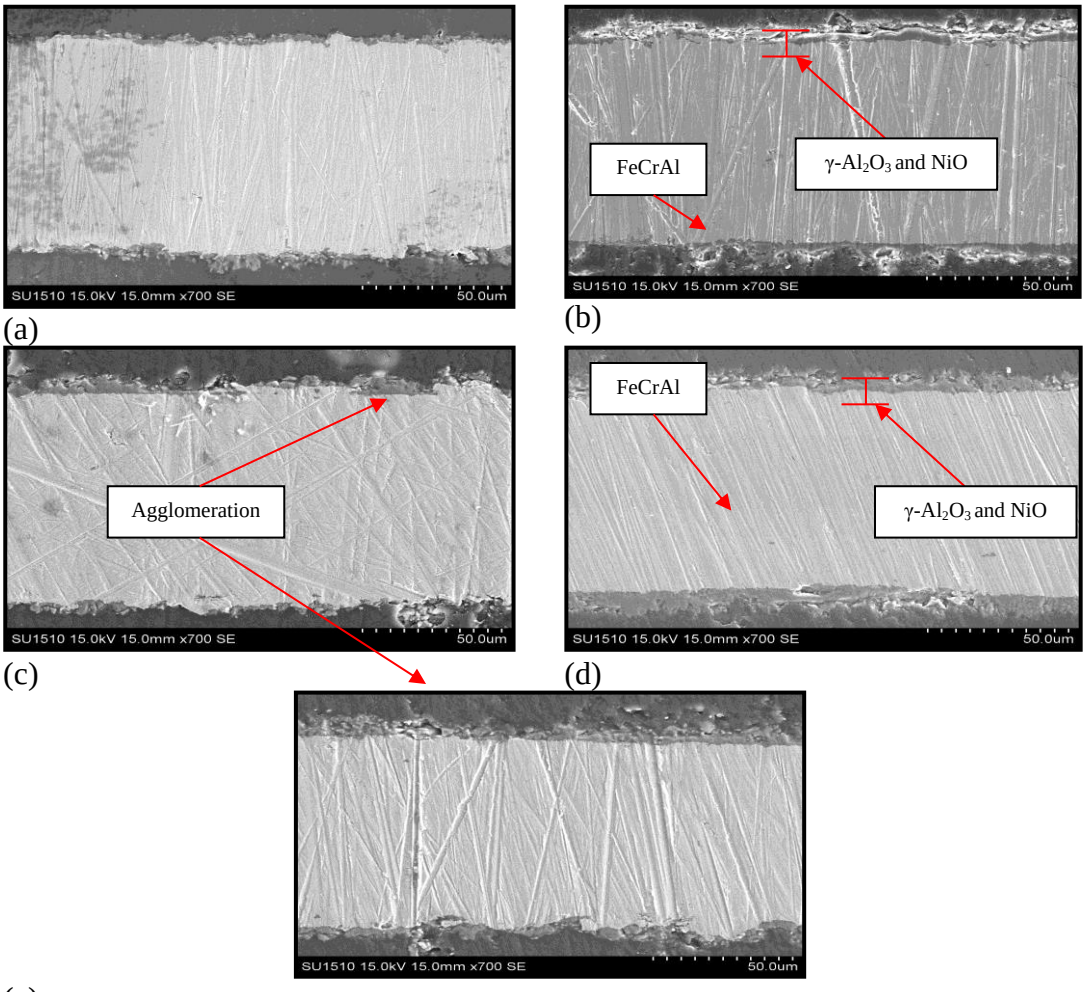

Figure 5. *Cross section analysis of (a) UB+EL 15 min; (b) UB+EL 30 min; (c) UB+EL 45 min; (d) UB+EL 60 min and (e) UB+EL 75 min*

Higher coating thickness in UB+EL samples provided by 2 deposition process Therefore, coating thickness of the UB+EL sample is generally higher as compared with UB, UBdEL and EL samples. That statement was supported by coating thickness data that coating thickness of UB+EL samples are 9.1, 10.3, 10.6, 11.7 and 12 µm for UB+EL 15, 30, 45, 60 and 75 minutes, respectively. Where the coating thickness of UB sample in between 2-2.8 µm, UBdEL samples in between 4.1-5 µm and EL samples in between 6.2-11.3 µm. Uneven coating layer has been observed in this analysis due to uneven surface of FeCrAL substrate as shown in Surface structure of raw material. However, FeCrAl alloy scale forming is able to form permanently bonding γ-Al_2O_3 as ceramic coating material (Checmanowski and Szczygiel, 2008).

Cross section of EL samples

Electroplating was performed to FeCrAl substrate where NiO as anode and FeCrAl as cathode and γ-Al_2O_3 powder as coating material. This method was conducted by various time of 15, 30, 45, 60 and 75 minutes and the cross section analysis is shown in Figure 6. Cross section image shows that electroplating time increase and coating thickness increase. However, coating layer shows in uneven surface which indicated that the sample has high surface roughness due to large particle that embedded on FeCrAl substrate that correlated with surface roughness analysis that longer electroplating time as higher surface roughness as well. Sulphamate type of electrolyte is used to effective coating process. Therefore, one of the electrolyte contents has been observed in cross sectional composition analysis in small value. Nickel electroplating were performed to develop NiO protective layer in FeCrAl substrate and react to O and γ-Al_2O_3 coating material to develop NiO, $NiAl_2O_4$ and $NiCr_2O_4$ compounds (Dafit Feriyanto and Supaat Zakaria, 2020).

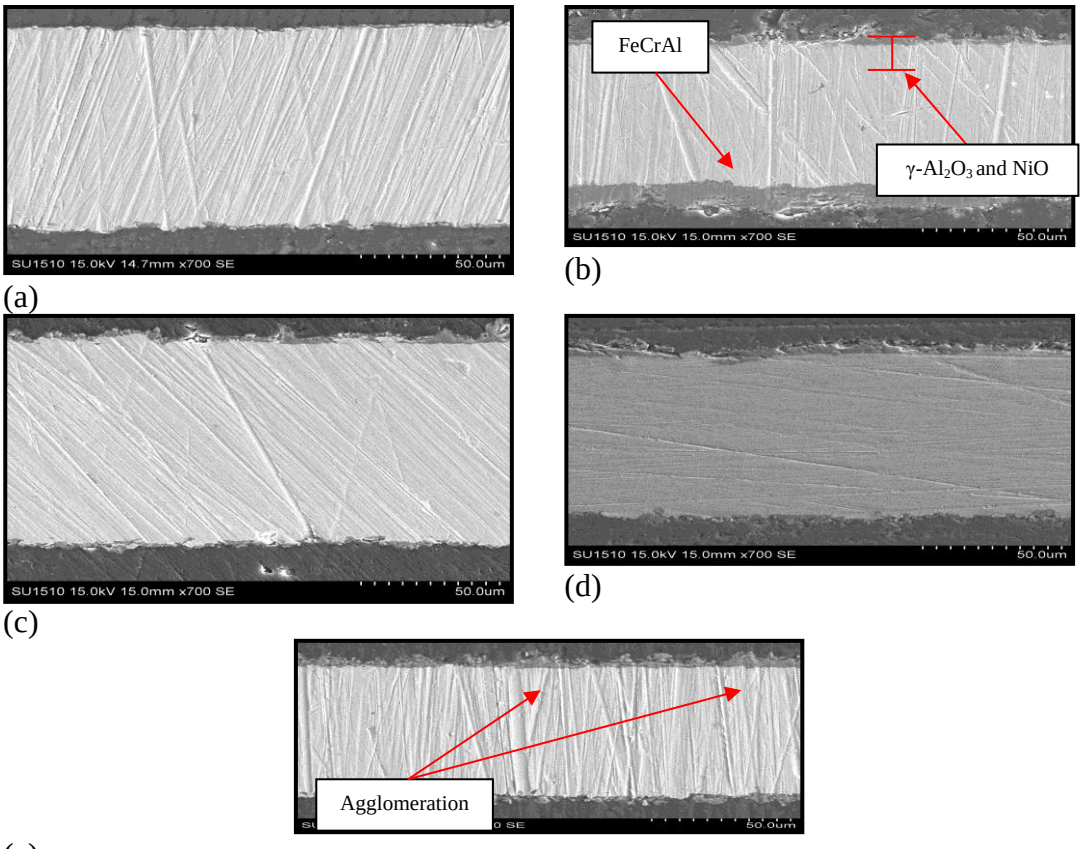

Figure 6. *Cross section analysis of (a) EL 15 min; (b) EL 30 min; (c) EL 45 min; (d) EL 60 min and (e) EL 75 min*

In NiO electroplating, NiO is positioned as anode material in order to able for transform to the NiO and $NiCr_2O_4$ form after oxidation process under normal pressure condition (Birks *et al.*, 2006). NiO is significantly developed at temperature of 900^0C and above. Regarding to Birks *et al.*, (2006) that the oxidation at temperature of 700-1300^0C will improve parabolic rate constant (k_p) which promoted by NiO form. In addition, upon heating process, Cr subsequently form another oxide layer which is Cr_2O_3 as protective oxide layer at high temperature (Tad *et al.*, 2005). Moreover, Cr very potential to improve the physical properties of coated FeCrAl substrate when Cr_2O_3 combined with Ni to be $NiCr_2O_4$ compound (Bastidas, 2006).

Conclusions

Developing $Fe_{80}Cr_{20}$ interconnect material was achieved by high energy ball milling and ultrasonic technique and the results shows that the ball milling was very effective to decrease the particle size from 38.67 to 6.27 µm and improve the specific surface from 865.22cm^2/g to 1980.92 cm^2/g. This research achieves the finer surface structure and even particle size through ultrasonic treatment. In interconnect application, this result gives a significant recommendation regarding to treatment that need to proposed in interconnect material which is combination of high energy ball milling and ultrasonic treatment. Where the optimum parameter was achieved in Milled 60 h and UT 4.5 h.

Acknowledgments

The authors acknowledge Universitas Mercu Buana for funding support and lab facility. Special thanks to those who contributed to this project directly or indirectly.

References

Bai, C. Y., Leec, J. L., Wend, T. M., Houe, K. H., Wue, M. S. & Ger, M. D. (2011). The Characteristics of Chromized 1020 Steel with Electrical Discharge Machining and Ni Electroplating Pre-Treatments. Applied Surface Science, 257, pp. 3529–3537.

Bastidas, D. M. (2006). High temperature corrosion of metallic interconnects in solid oxide fuel cells. Revista De Metalurgia, 42 (6), pp. 425-443, 2006; ISSN: 0034-8570.

Birks, N., Meier, G. H. & Pettit, F. S. (2006). Introduction to The High Temperature Oxidation of Metals, 2nded, Cambridge University Press, ISBN 978-0-511-16089-9, New York, USA.

Checmanowski, J. G. & Szczygiel, B. (2008). High Temperature Oxidation Resistance of FeCrAl Alloys Covered with Ceramic SiO_2–Al_2O_3 Coatings Deposited by Sol–gel Method. Corrosion Science, Vol. 50, No. 12, pp. 3581-3589.

Corni, I., Ryan, M. P. & Boccaccini, A. R. (2008). Electrophoretic Deposition: From Traditional Ceramics to Nanotechnology. Journal of the European Ceramic Society, Vol. 28, No. 7, pp. 1353–1367.

Dafit Feriyanto and Supaat Zakaria. (2020). New Method of Fabrication of Fe80Cr20 Alloy: Effect of its Technique on Crystallite Size and Thermal Stability. International Journal of Advanced Technology in Mechanical, Mechatronics and Materials. Vol 1, No.1, pp. 26-31.

Dafit Feriyanto, H Pranoto, H Carles and AM Leman. (2020). Comparison of metallic (FeCrAl) and Ceramic Catalytic Converter (CATCO) in reducing exhaust gas emission of gasoline engine fuelled by RON 95 to develop health environment. IOP Conference Series: Earth and Environmental Science. Vol. 485, No.1, p. 012004.

Dafit Feriyanto and Samir Sani Abdul Malik, Muhamad Fitri, Imam Hidayat, Hadi Pranoto, Supaat Zakaria. (2021). Effect of Material Composition on Thermal Stability Analysis of Coated and Uncoated FeCrAl CATCO by γ-Al2O3 Ultrasonic-Electroplating Technique. Journal of Sustainable Materials Processing and Management. Vol 1, No.1, pp. 1-7.

Domínguez M. I., A. Pérez, M. A. Centeno, J. A. Odriozola. (2014). Metallic Structured Catalysts: Influence of The Substrate on The Catalytic Activity. Applied Catalysis A: General, 478, pp. 45–57.

Eyupoglu, V. & Kumbasar, R. A. (2015). Extraction of Ni(II) from Spent Cr–Ni Electroplating Bath Solutions Using LIX 63 and 2BDA as Carriers by Emulsion Liquid Membrane Technique. Journal of Industrial and Engineering Chemistry, 21, pp. 303–310.

Graham, M. J. & Hussey, R. J. (2002). Characterization and Growth of Oxide Films. Corrosion. Sci., 44, pp. 319–330.

Jung, Y. and Bae, C. (2015). Immaturity of soot particles in exhaust gas for low temperature diesel combustion in a direct injection compression ignition engine. Fuel, Vol. 161, pp. 312–322.

Kim, D. H., Byung Yong Yu, PilRyung Cha, Woo Young Yoon, Ji Young Byun & Sang Hoon Kim. (2012). A Study on FeCrAl Foam as Effective Catalyst Support Under Thermal and Mechanical Stresses. Surface & Coatings Technology, 209, pp. 169–176.

Masakuni, O., & Kenichi, A. (2015). Effect of La Modification on The Stability of Coating Alumina Layer on FeCrAl Alloy Substrate. Surface & Coatings Technology, 271, pp. 80–86.

Panta, G. P. and Subedi D. P. (2012). Electrical Characterization of Aluminum (Al) Thin Films Measured by Using Four- Point Probe Method. Kathmandu University Journal Of Science, Engineering and Technology, Vol. 8, No. II, pp 31-36

Tad, J. A., and Michael, A. H. (2005).Evaluation of metallic interconnects for use in intermediate temperature SOFC. University of Utah, Salt Lake City, USA.

Wua, G., Lia, N., Zhoua, D. & Mitsuo, K. (2004). Electrodeposited Co–Ni–Al_2O_3 Composite Coatings. Surface and Coatings Technology, Vol. 176, No. 1, pp. 157–164.