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Study on the Effect of EN Process Parameters on Ni-YSZ Co-Deposition Coating Thickness

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ABSTRACT

Background: The paper discusses the effect of electroless nickel (EN) process parameters on the coating thickness of Ni-YSZ co-deposition. The Ni-YSZ co-deposition was produced by EN process and 8mol% yttria stabilized zirconia (YSZ) powder on alumina substrate. EN process parameters investigated in the study were bath loading ranges between 225-150 ml, bath temperature ranges 84-94^{o}C, stirring speed between 250-500 rpm and pore former namely graphite, activated carbon and starch. Coating quantity is measured in the form of its thickness and quality in term of no cracking and delamination. It was found that bath loading give positive effect whereas stirring speed give negative effect on coating thickness. The effects of bath temperature on coating thickness are varied depending on types of pore former. The best thickness obtained is 0.97 mm for EN process parameter of 225 ml bath loading, 84^{o}C bath temperature, 375 rpm bath stirring speed and starch pore former. Coating surface was analysed using SEM coupled with EDX. The microstructure showed the presence of superficial microcracks without delamination.

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INTRODUCTION

Nickel-ceramic composite have numerous applications in the industries especially in engineering applications. Its outstanding thermal (Gengler *et al*., 2010), wear and corrosion (Kundu *et al*., 2014). The performance and lifespan of certain object or devices could be affected by the fracture and failure of the coating thickness (Li *et al*., 2014). Nickel ceramic coating can be produced by many ways such as thermal and chemical deposition. One of the chemical deposition methods is electroless coating.

Electroless coating of various metals such as copper, gold, nickel, cobalt, palladium, iron, silver and their alloys, was found in many micro system applications in the last 30 years (Diamand *et al*., 2014). Hence electroless nickel (EN) coating is one of the most important and are widely used in the industries for having some of good properties such as uniform coating thickness, electrical conductivity and also high corrosion resistance (Farzaneh *et al*., 2013) while porous nickel- yttria-stabilized zirconia (Ni-YSZ) showed reasonable electrical conducitivity at 800^{o}C, reasonable ionic conductivity and high electrocatalytic for hydrogen oxidation (Baba and Davidson 2011, Davarpanah *et al*., 2014) .

Electroless coating is a catalytic reduction of a metallic ion from an aqueous solution where the metal is deposited without the use of electrical energy (Balaraju and Seshadri, 1999). Thus, EN coating is a chemical reaction which reduces nickel ions to nickel metal (Panja and Sahoo, 2014). The reducing agent in EN solution is oxidized and Ni^{2+} ions are deposited on to the substrate surface as Ni metal and the first layer of deposited Ni acts as a catalyst for the continuous process (Khosroshahi *et al*. 2014). The autocatalytic metal electroless plating process demand an extremely slow process and therefore plating rate enhancement is required (Agarwal *et al*., 2013). In order to achieve optimum Ni metal deposition quantity and quality, varying EN coating parameters such as bath temperature, bath loading, bath pH, agitation methods and others should be investigated.

Surface coating thickness is one of the most important characteristics. EN deposit is very well known for its ability to produce uniform thickness on complex geometries and shapes parts. Due to the presence of phosphorous as an alloying element, the density of EN deposits becomes lower than pure nickel (Kundu *et al*., 2014). The coating thickness form will be altered by varying EN process parameters during electroless coating.

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The thickness play important role in addition to void fracture of the composite. Thickness of the coating, hardness, structure and morphology of deposit is found to be significantly affected by heat treatment (Sorkhabi and Rafizadeh, 2003). Li *et al*, 2014 reported low coating thickness caused multiple transverse cracks whereas thickness greater than 255µm caused delamination.

Experimental:

Electroless nickel co-deposition is an autocatalytic process. The addition of ceramic YSZ particles in the bath will cause a co-deposition of metallic nickel and ceramic YSZ onto the substrate. In order to enhance the amount of Ni-YSZ composite coating, several EN process parameters were chosen as listed in Table 1. Previous studies found that particle sizes of 2 micron and mechanical stirring agitation were the dominant parameters affected the amount of ceramic YSZ particle co-deposited into the coating (Baba, Davidson & Muneer, 2011). Thus in this study, EN process parameters such as bath loading, bath temperature, stirring speed and incorporation of pore former were investigated for high quantity and quality surface coating.

Table 1: Experimental EN process parameters.

Parameters	Range		
Bath Loading / ml	150	180	225
Bath Temperature/ ^{o}C	84	89	94
Stirring Speed/ rpm	250	375	500
Pore Former	Graphite	Activated Carbon	Starch

EN experimental procedure was started by pre-treatment of alumina substrate followed by EN composite coating as described in details in the previous study (Baba 2012). The thickness was measured by digital micrometer Mitutoyo 293-340.

RESULTS & DISCUSSION

Several sets of experiments were conducted to study the effect of EN process parameters on the coating thickness of Ni-YSZ co-deposition. EN process parameters involve are bath loading ranges between 150-225 ml, bath temperature ranges between 84 - 89^{o}C, bath agitation stirring speed ranges between 250-500 rpm and pore former namely graphite, activated carbon and starch. Pore former parameter was included as to investigate whether or not their presence affects the quantity and quality of coating.

The source of nickel in the form of nickel ions (Ni^{2+}) is presence in the EN bath solution. Hence varying amount of EN bath solution so called bath loading will affect the coating thickness. The experimental result of the coating thickness against variation of bath loading for different pore former is given in Figure 1. Generally, the trend showed that as the bath loading increase from 180 to 225 ml, the coating thickness also increase. The highest coating thickness obtained is 0.95 mm at 225 ml bath loading for starch pore former. Positive increment was seen for both starch and graphite pore formers whereas activated carbon showed inconsistency. Starch pore former resulted in good coating thickness as it also functions as adhesive (Jarowenko, 1977). However, high thickness of starch contained coating causes high tensile residual stress state, which leading to poor quality performance while the thin thickness may cause quick worn out (Martin *et al*., 2012).

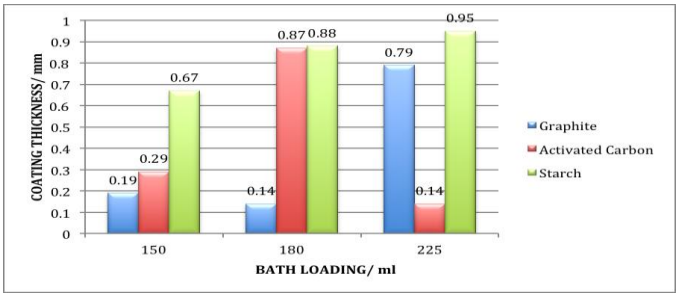

Fig. 1: Coating Thickness against bath loading for different types of pore former.

Bath temperature plays an important parameter to ensure good quantity and quality of coating. Coating quantity is measured in the form of its thickness and quality in term of no cracking and delamination. In the experiment, the bath temperature was varied at 84°C, 89°C and 94°C for three types of pore former. The EN Slotonip suggest 89^{o}C as the optimum bath temperature but this is for optimum Ni deposition alone. In this experiment, Ni will be co-deposited with YSZ ceramic particle as well as the addition of pore former. Figure 2 illustrate the variation of coating thickness with bath temperature for different types of pore former. It was found that the performance of coating thickness trend depends on the types of pore former. Starch showed optimum coating thickness at 94^{o}C while activated

carbon was found almost consistence at all temperature but most optimum at 89°C as suggest by the EN datasheet. On the other hand, graphite pore former is directly proportional to the bath temperature. The previous study reported that graphite was low conductive fillers, hence as the bath temperature increases, the coating thickness increases (Tongxiang *et al*., 2007).

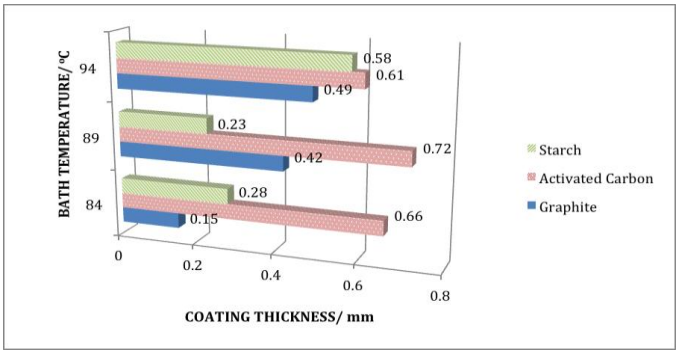

Fig. 2: Coating thickness against bath temperature for different types of pore former.

Another set of experiment was carried out to study the effect of bath agitation stirring speed of 250rpm, 375 rpm and 500 rpm on the coating thickness. The variation was conducted in 3 groups of different types of pore former. The highest thickness coating obtained is 0.97mm as showed in Figure 3 at 250 rpm for starch pore former. General trend showed in Figure 3 is the coating thickness of Ni-YSZ co-deposition decreases as the stirring speed of bath agitation increases. This finding was supported by Beygi *et al*, 2012 whom reported that higher speeds lead to the less plating rates and finer coated layers. Besides, activated carbon and graphite pore former batch did not have great influence by the change in bath stirring speed as shown by the starch pore former batch.

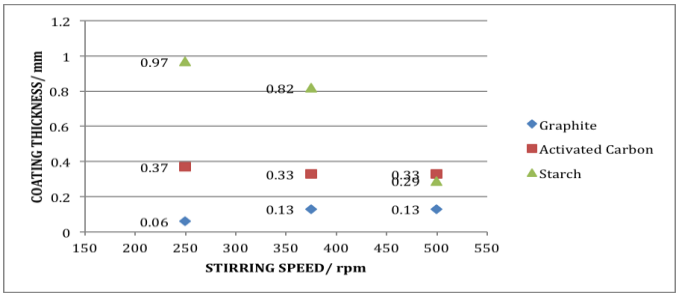

Fig. 3: Coating thickness against stirring speed for various bath loading.

SEM-EDX:

The surface of Ni-YSZ co-deposition coating was analysed using scanning electron microscope (SEM) couple with energy dispersive X-ray (EDX) analysis. The purpose of the SEM-EDX analysis is to determine the coating quality by analysing the micrograph as well as the elemental analysis obtained.

A sample of Ni-YSZ co-deposition coating at 225 ml bath loading, 84°C bath temperature, 375 rpm bath stirring speed and starch pore former where the coated thickness measured is 0.97mm which is the highest was chosen. Figure 4 present the microstructure of the chosen Ni-YSZ co-deposition coating at various magnification. At lower magnification shown in Figure 4(a), few microcracks are visible. But this microcrack is very superficial as shown in Figure 4(b) at 10 time higher magnification. The microstructure of the coating is seemed jelly like polymer-based materials due to starch that exist on the surface. Upon heating the sample at 700°C in the oven for one hour, the microstructure has changed as shown in figure 4(c) at 70 times higher magnification. Upon heating, the starch is burn out leaving pores or cavity on the coating surface. The white particles are the YSZ ceramics, the grey background is the Ni metallic matrix and the black area are the pores in the coating. However, these are very small pores and may not have penetrated to the substrate surface. At higher magnifiaction, the microcrack was not visible indicating that the microcrack is only superficial. Hence, it was clearly shown there only microcrack without delamination present in the Ni-YSZ co-deposition coating.

A comparison of microstructure at 10 kX magnification for different types of pore former is given in Figure 5. These micrographs of Ni-YSZ co-

deposition coating was captured after the removal of pore former by heating to 700°C for one hour. Comparing these 3 microstructures, there are no doubt the porosity are presence in the coating. The shape and type of porosity formed are differ with the types of pore former used. Graphite and activated carbon pore formers in Figure 5(a) and 5(b) showed similarity where the porosity seemed to have interaction underneath the upper surface. The microstructure is overlapped and staggered show the possibility for more porosity underneath the upper surface. Comparing these two to the starch pore former in Figure 5(c), the porosity seemed to be more superficial. It looks like the microstructure is clog underneath the first surface. Visually, the microstrucutre of graphite and activated carbon are less dense compared to the starch pore former.

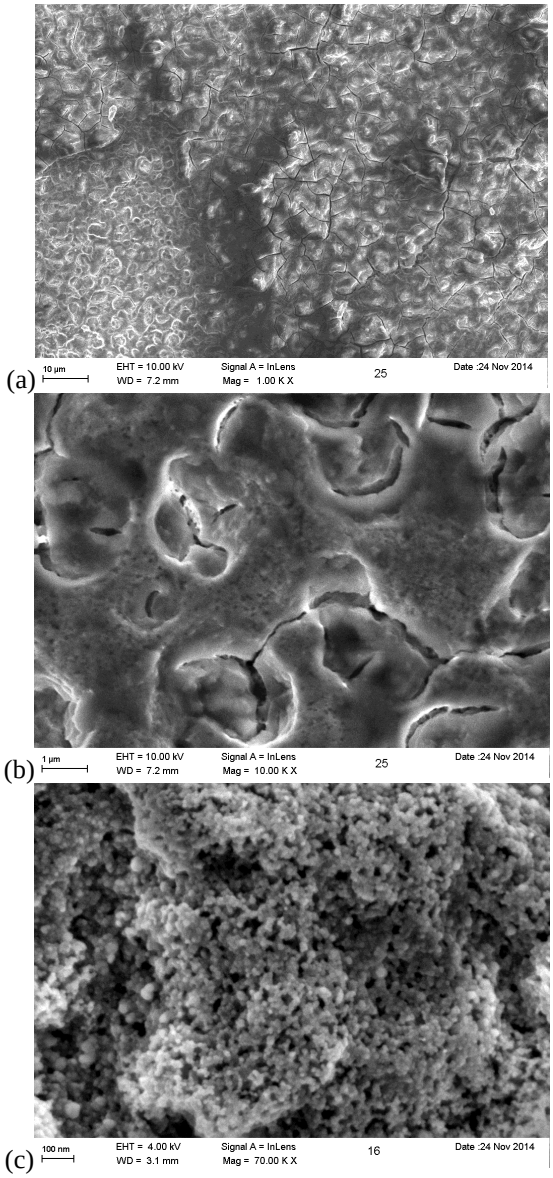

Fig. 4: SEM micrograph of Ni-YSZ co-deposition coating for 225 ml, 84°C, 375 rpm and starch pore former at magnification of (a) 1 kX (b) 10 kX (c) 70kX

Figure 6 below is the EDX spectrum of the Ni-YSZ co-deposition coating using SEM coupled with energy dispersive X-ray (EDX) analyzer. The spectrum showed two peaks of nickel (Ni) that represent the K-α and K-β. Besides Ni as the major element, phosphorous (P) element also exists as where the electroless Ni-P deposits is usually form during the deposition. Other than these two elements, there are also sodium (Na), oxygen (O) and carbon (C) as sub element in the coating.

Conclusions:

The effect of EN process parameters on the Ni-YSZ co-deposition coating thickness was investigated. There are four EN process parameters chosen namely bath loading, bath temperature,

stirring speed and types of pore former. The effect of bath loading on coating thickness regardless of types of poreformer is generally directly proportional as the bath loading represent the source of Ni ions. The effects of bath temperature on coating thickness are varied depending on types of pore former. The best thickness obtained is 0.97 mm for EN process parameter of 225 ml bath loading, 84^{o}C bath temperature, 375 rpm bath stirring speed and starch pore former. The microstucture of the sample showed the presence of microcracks that is proven only superficial as at higher magnification there is no sign of crack or delamination. The addition of poreformer showed the presence of high porosity in all types of pore former. The micrographs compared the microstructure where the graphite and activated carbon pore former give better type of porosity compared to the starch pore former.

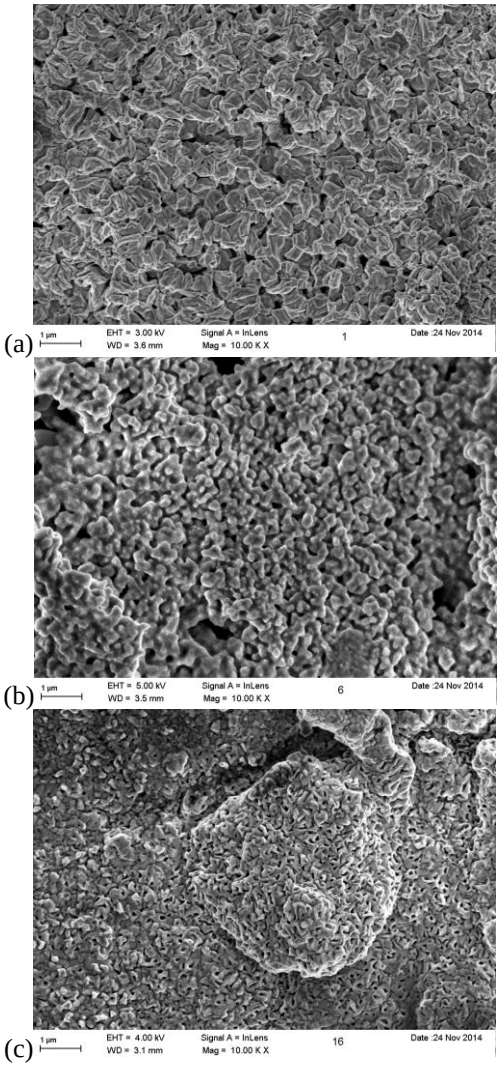

Fig. 5: SEM micrograph of Ni-YSZ co-deposition coating after removal of pore former (a) graphite, (b) activated carbon and (c) starch.

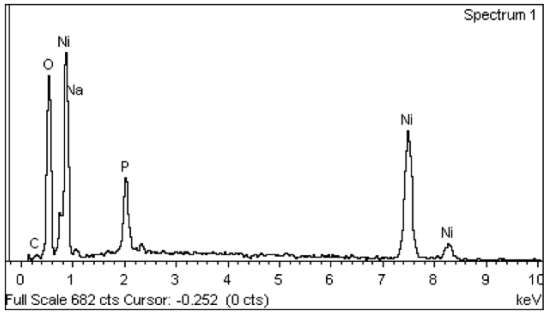

Fig. 6: EDX spectrum of Ni-YSZ co-deposition coating.

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