

# Correlating the Layer Properties of Ni-alumina Composite Coatings and the Mechanism of Codeposition

Amir Sadeghi<sup>1</sup>, Maximilian Sieber<sup>1</sup>, Hosein Hasannejad<sup>2</sup>, Ingolf Scharf<sup>1</sup>, Thomas Lampke<sup>1</sup>

<sup>1</sup>Chemnitz University of Technology, Institute of Materials Science, Chemnitz, Germany

<sup>2</sup>Arak University, Department of Materials Engineering, Iran

Correspondence: Amir Sadeghi, Chemnitz University of Technology, Institute of Materials Science and Engineering, Chemnitz, Germany. email: amir.sadeghi@mb.tu-chemnitz.de Tel: +4917657802623.

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## Abstract

Ni-Al<sub>2</sub>O<sub>3</sub> composite coatings electrodeposited from Watt's electrolyte include Al<sub>2</sub>O<sub>3</sub> nano- and submicron particles. The effect of particle size and concentration of the particles in the electrolyte were investigated on the morphology and incorporation value of particles into the deposits. The influence of alumina particles on the electrodeposition behavior of Ni was also studied by means of electrochemical impedance spectroscopy. The results achieved from the impedance measurements and the correlation with the layer characterization could help to better understand the codeposition mechanism derived from the impact of different particle characteristics including size and concentration on the nature of the double-layer. The increase in the concentration of particles from 1 to 20 g/l resulted in an increase of the double-layer capacity and decrease of the charge transfer resistance, while the addition of submicron particles had a higher influence on the characteristics of the double-layer compared to the nano particles. Although the alumina particles with submicron size could stimulate the incorporation of particles faster than those with nano size, the strengthening performance of the layers not only depended on the incorporation value of the particles, but also on the microstructure of the deposits.

**Keywords:** Ni-Al<sub>2</sub>O<sub>3</sub> electrodeposited coating, particle size, codeposition mechanism, electrochemical impedance spectroscopy, layer properties

## 1. Introduction

Composite coating has been well developed for the last decades. The simultaneous deposition of the non-metal inclusions and metal phases to form composite layers entails a significant improvement on several physical and mechanical properties of the coating. Among the non-metal inclusions, ceramic particles like Al<sub>2</sub>O<sub>3</sub>, SiC, TiO<sub>2</sub>, WC, etc. have been frequently used in the literature for synthesizing composite coatings particularly with better mechanical properties (Guglielmi, 1972; Viswanathan, 1979; Sun, 1996; Ghouse, 1984; Periene, 1993; Zahavi 1982; Stankovic, 1996; Bakhit, 2013).

The final performance in the composite coatings is highly dependent on the characteristics of the particles used in the electrodeposition process including type, size and concentration (Zahavi 1982; Stankovic, 1996; Bakhit, 2013; Ehrhardt, 1985; Bazzard, 1972; Verelest, 1991; Chen, 1971; Roos, 1978; Stojak, 1999; Stojack, 2001). The characteristics of Al<sub>2</sub>O<sub>3</sub> as a typical reinforcement used in the electrodeposition process have already been investigated by others (Stojak, 1999; Stojack, 2001; Osborne, 2006; Lee, 1988; Shao, 2002). It is stated that the size of alumina particles can strongly influence the codeposition value of particles in the deposited metal. As it was reported by Shao and his coworkers (Shao, 2002), the volume percentage of Al<sub>2</sub>O<sub>3</sub> 300 nm is much higher than Al<sub>2</sub>O<sub>3</sub> 50 nm, although the number of codeposited nano particles is higher compared to submicron particles. Meanwhile, except for the effect of the particle size, the influence of the particle type cannot be neglected. It seems that there are some characteristics between alumina alpha and gamma like different zeta-potential or magnetic properties, which can eventually change their codeposition behavior in different solutions (Chen, 1971; Roos, 1978; Stojak, 1999; Stojack, 2001).

Up to now, different models have been reported for the codeposition mechanism of particles in the electrolyte under various working conditions by researchers like Guglielmi (Guglielmi, 1972), Celis (Roos, 1978), and other researchers. Generally, the early suggested models for the codeposition mechanism of solid particles in the metallic matrix initiated from three main phenomena: electrophoresis, mechanical entrapment and adsorption. More details about each of these phenomena are broadly explained in ref (Low, 2006). These theoretical models can provide us with a good understanding of the codeposition phenomena. The validity of the various theoretical models underlying particle

incorporation in the metal matrix, still requires more empirical laboratory trials and attention to the interactive variables such as the particle characteristics like particle size and particle concentration.

In recent years, it has been established that electrochemical impedance spectroscopy (EIS) can be a reliable tool in order to better understand the effect of particles in the codeposition process (Benea, 2001; Hu, 2005; Hu, 2007; Sohrabi, 2012). For instance, Benea et al. (Benea, 2001) studied the influence of silicon carbide nano particles on nickel electroplating. They stated that particles affected the nickel reduction, which became obvious by the lower charge-transfer resistance. They also stated that the surface morphology of nanostructured composite layers is different compared to a pure nickel coating. The purpose of the SiC particles was to increase the number of nucleation sites with a reduction in crystal growth resulting in a smaller grain size of the nickel matrix.

The aim of this work is to investigate the influence of nano- and submicron  $\text{Al}_2\text{O}_3$  particles with different concentrations in the electrolyte on the codeposition behavior of particles in a Ni matrix by the aid of impedance spectroscopy and also by relying on the effect of particle size and concentration on the layer properties including morphology, microstructure and hardness.

## 2. Experimental Setup

In this study, a typical Watt's electrolyte including 250 g/l  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$  (Sigma Aldrich), 30 g/l  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (Merck), 30 g/l boric acid (Merck) and 0.3 g/l sodium dodecyl sulfate (Merck) was used for the fabrication of Ni and Ni-alumina composite coatings. One liter of each electrolyte was prepared with distilled water in a 2000 cc glass container. The pH and temperature were held at 3.5 and 55 °C, respectively. Suspensions were prepared by separately adding alumina (Alfa) 13 nm and (Gamma) 150-200 nm to the solution. The mix of suspension and solution stirred for at least 6 h before the deposition process. The coatings electrodeposited from electrolyte with three different particle concentration of 0, 5, 20 g/l. It should be pointed that higher concentration of particles, due to the agglomeration challenge and decrease in incorporation value of particles into the deposit was not investigated. An IM6-Zahner working station in a galvanostatic mode was simultaneously used for the production of the deposits and also for the impedance measurements in a three-electrode cell as it can be seen in figure 1. Ultrasound vibration (25 Hz and power of 55 W) was applied during the deposition while the electrolyte was agitating with a rate of 400 rpm. Carbon steel with the polishing surface area of 20  $\text{cm}^2$  and pure nickel with 45  $\text{cm}^2$  were used as working electrode and counter electrode, respectively. A distance of 5 cm was maintained between working electrode and counter electrode, and the Ag/AgCl reference electrode was placed in the middle. The applied current was -800 mA, which is equal to 4  $\text{A}/\text{dm}^2$ , according to the active area of the working electrode. The frequency range for impedance measurements was between 100 KHz and 10 Hz with a 10 mV amplitude. Before performing the first impedance test, in order to reach a constant and stable condition, each sample was deposited in the electrolyte for 10 minutes and after that the impedance measurements were recorded in three more cycles each three minutes with adjusting in Polysequencer configuration available on the Zahner-software. Each impedance measurement took 81 seconds. In order to ensure the reproducibility of the results, except performing four cycle tests on each sample, some of the tests were repeated randomly with new samples, and the average values were finally reported for the impedance measurements.

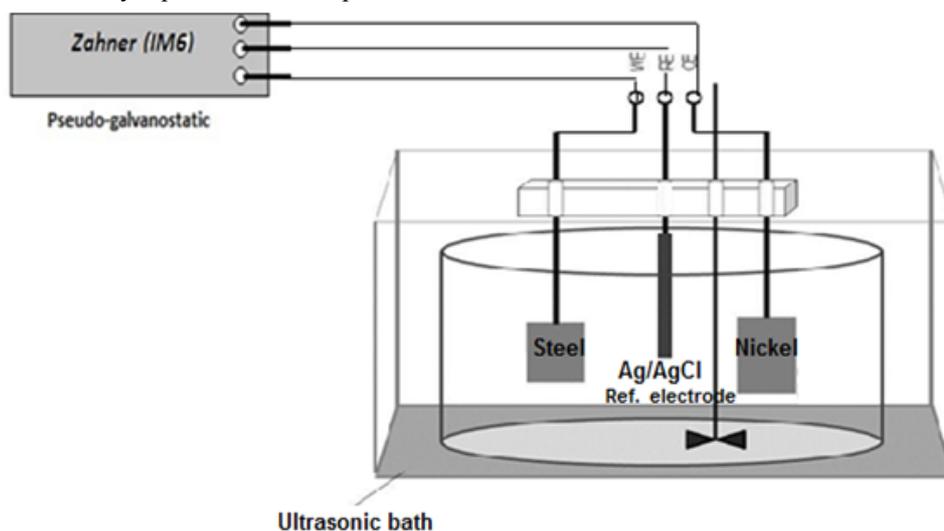


Figure 1. Experimental setup

The thickness of coatings was measured at 10 different points by means of optical microscopy (Olympus GX 51). The morphology of the deposits and also the value of the incorporation of the particles were examined by the help of SEM (NEON 40EsB, Zeiss) and EDX (Genesis). The EDX measurements were performed around the cross-section. Image analyzer software (a4i-Analysis) was used for the estimation of the volume percentage of the particles in a certain surface area. The nickel grain size was estimated by the X-ray diffraction (XRD) method of Scherrer's equation (Patterson, 1939) using the Co-K $\alpha$  line (D8 Discover, Bruker AXS).

It should be mentioned that the obtained values for the grain size represent the average size in the analyzed volume and not allow for a statement about the distribution of the grain size. Moreover, the evaluation of the grain size achieved by this method is only true for the grain size under about few hundreds of microns; over almost 300 nm the real average of grain size can be even larger. For detecting the Martens hardness value, instrumented indentation (HM2000XYm, HELMUT FISCHER GmbH) was applied on the cross-sections of the deposits by applying a 25 mN force for 30 s loading time, 30 s holding time and another 30 s unloading time on a Vickers indenter. The final value quoted for a deposit represents the average of 5 measurements.

### 3. Results and Discussion

#### 3.1 Effect of Alumina Particles on the Morphology, Microstructure and Mechanical Properties

As illustrated in figure 2, the morphology of Ni-Al<sub>2</sub>O<sub>3</sub> composite coatings is different compared to a pure nickel coating. With the addition of Al<sub>2</sub>O<sub>3</sub> particles in the electrolytic, the borders of the Ni grains become fuzzy and more compact in comparison to pure Ni with no particles. In the case of adding nano-Al<sub>2</sub>O<sub>3</sub>, the morphology seems to be even more compact and homogenous. Moreover, for the coating electrodeposited with submicron particles, it is obvious that the particles were mainly indulged with a certain gap from the Ni matrix and growth of nickel around the particle becoming rough. This behavior can be related to the non-inductive nature of alumina particles as it was also mentioned by (Erler, 2002). Furthermore, according to the cross-section SEM images in figure 3, it is observed that the dark spherical Al<sub>2</sub>O<sub>3</sub> particles are uniformly distributed in the white nickel matrix, thanks to the use of ultrasound during the deposition process. Nevertheless, due to the higher surface energy of nano particles, agglomeration at higher particle concentrations is still unavoidable despite of the application of ultrasound.

The EDS analysis in figure 4 displays the atomic percentage of Al for Ni-Al<sub>2</sub>O<sub>3</sub> composite coatings electrodeposited from Watt's bath including nano- and submicron Al<sub>2</sub>O<sub>3</sub> particles with different particle concentrations in the electrolyte. As can be seen, under the same conditions, the atomic percentage of Al for submicron particles is higher than the coatings prepared with nanoparticles. In addition, in both cases, the incorporation value of particles increases with increasing the loading of particles in the electrolyte.

However, as plotted in figure 5, the measurement of the volume percentage of particles in a certain surface by means of the image analyzer shows that, under the same working conditions, the number of incorporated particles, in the case of adding nanoparticles, is slightly higher compared to the addition of submicron particles, namely 5.1 to 4.6 vol%. The same behavior for Al<sub>2</sub>O<sub>3</sub> 50 nm and 300 nm was also detected by Vereecken et al. (Shao, 2002). In fact, by the reduction of the particle size, the number of the particles codeposited into the matrix is higher; but, due to the smaller size, the atomic percentage would be lower.

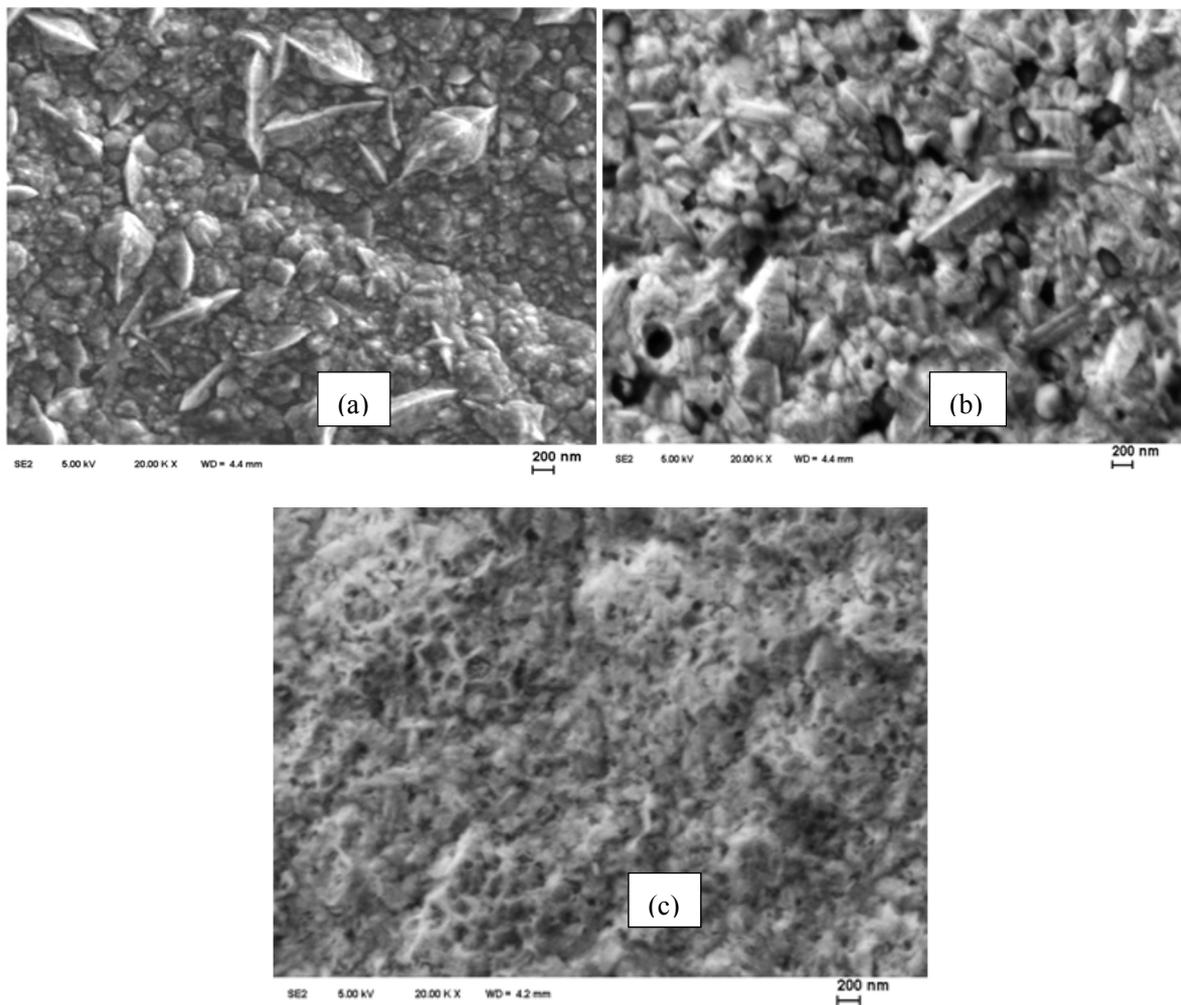


Figure 2. SEM images of the surface morphology:

(a) Ni, (b) Ni-submicron  $\text{Al}_2\text{O}_3$  20 g/l (c) Ni-nano  $\text{Al}_2\text{O}_3$  20 g/l electrodeposited at  $i=4\text{A}/\text{dm}^2$  for 40 min

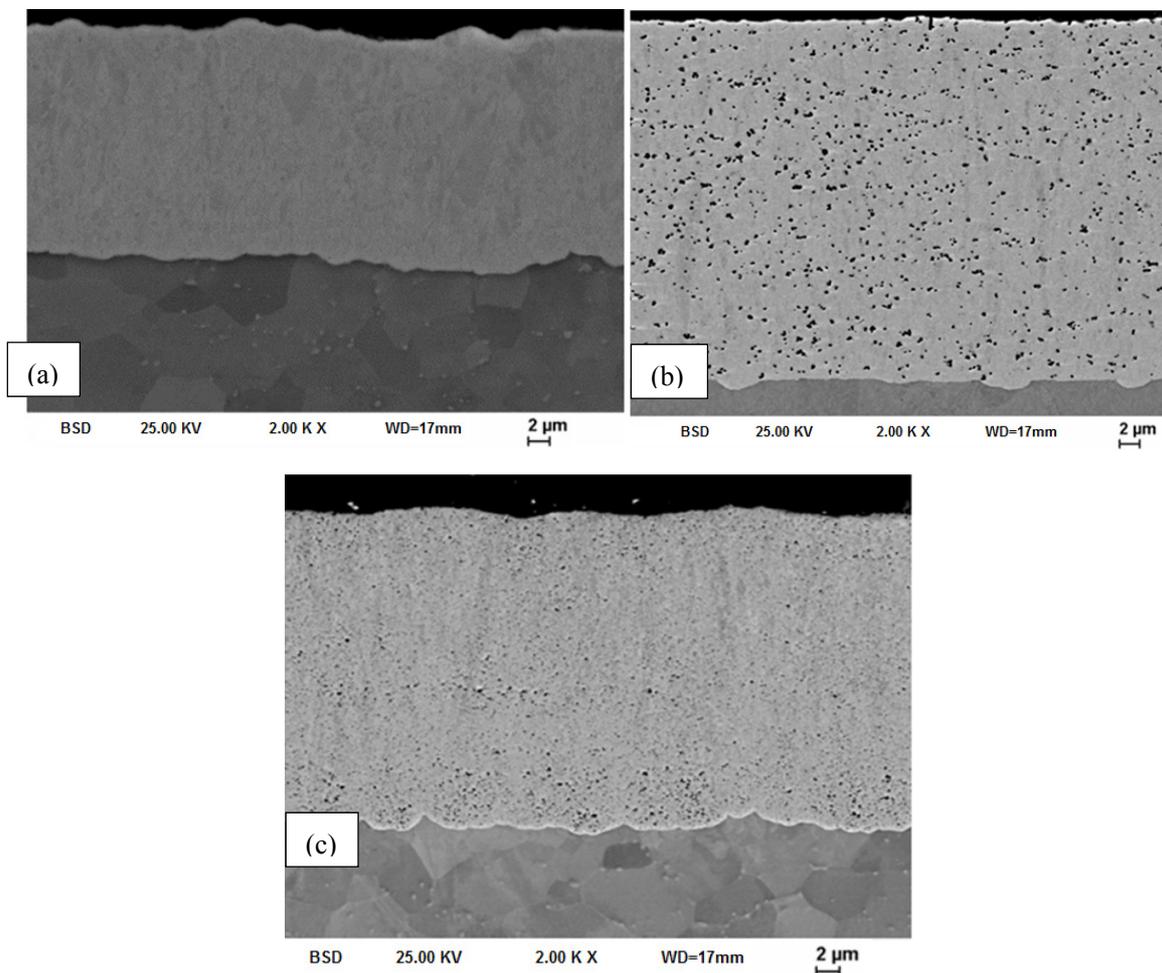


Figure 3. SEM images of cross-section:

(a) Ni, (b) Ni-submicron Al<sub>2</sub>O<sub>3</sub> 20 g/l (c) Ni-nano Al<sub>2</sub>O<sub>3</sub> 20 g/l electrodeposited at  $i=4A/dm^2$  for 40 min

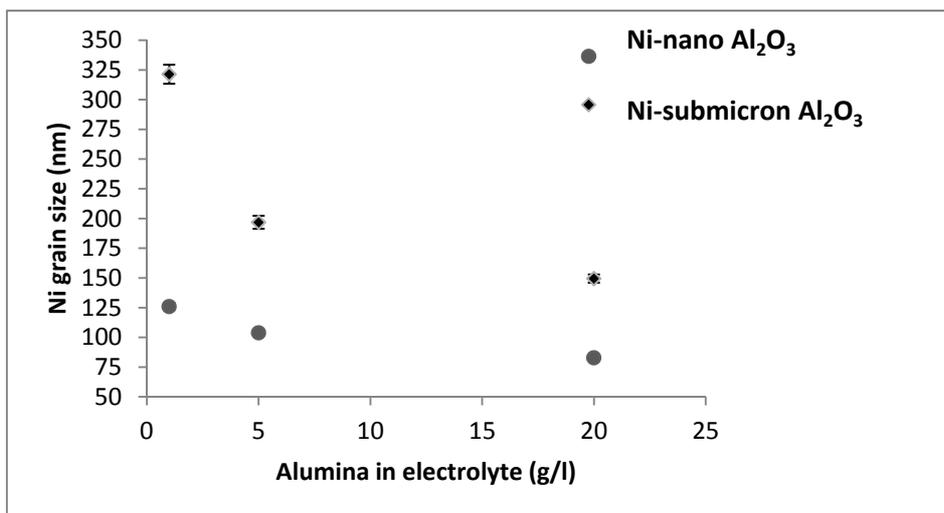


Figure 4. Effect of particle size and concentration on the incorporation value of particles achieved from EDX mapping analysis from the thickness of the deposits

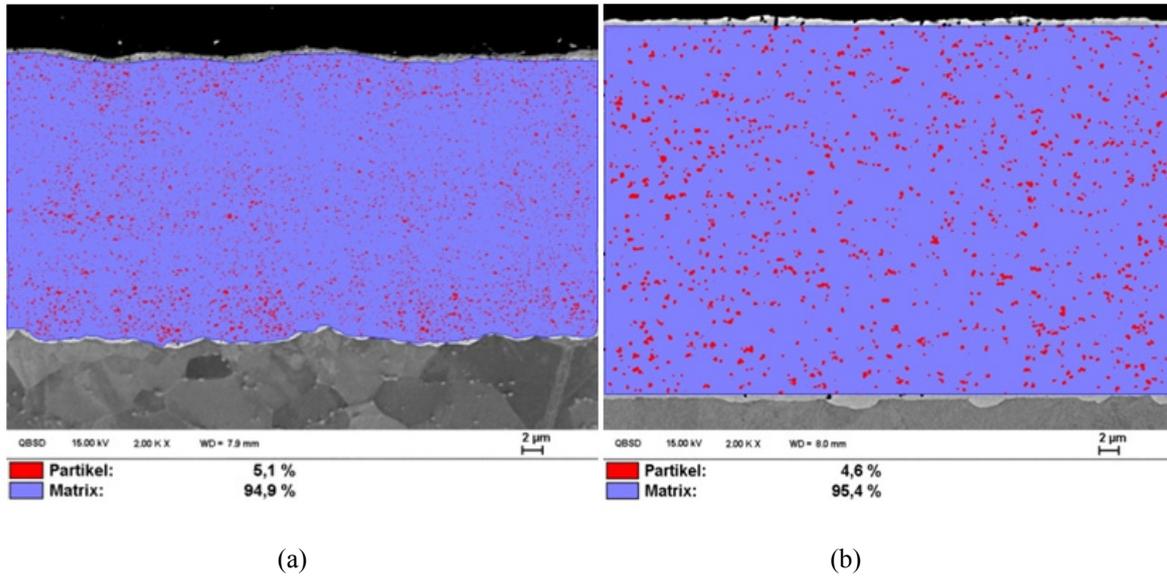
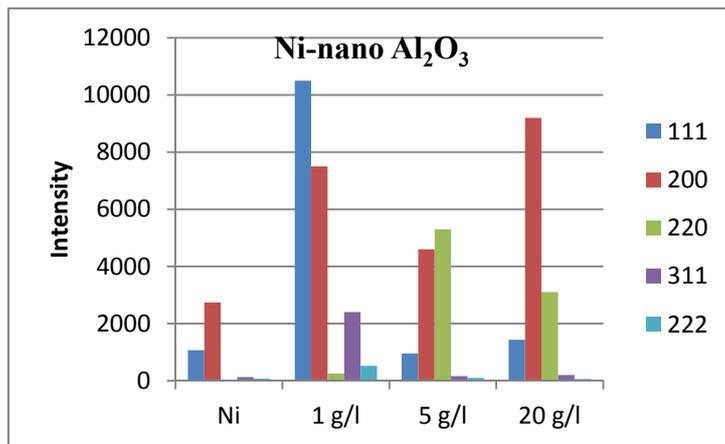
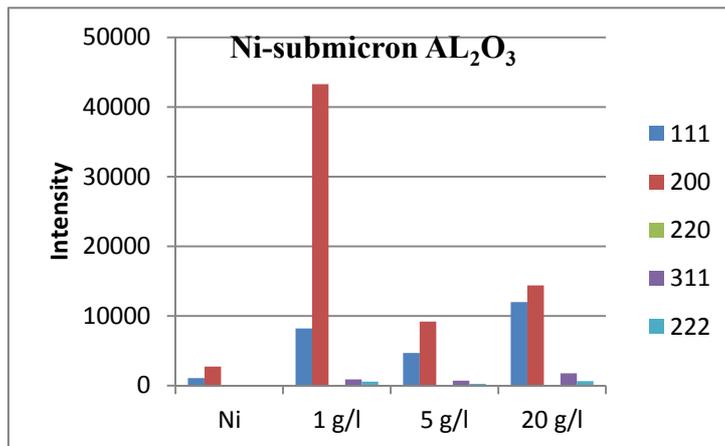


Figure 5. Volume percentages (vol%) of alumina particles estimated by image analyzer software:  
 (a) Ni-nano  $Al_2O_3$  20 g/l (b) Ni-submicron  $Al_2O_3$  20 g/l electrodeposited under the same working conditions



(a)



(b)

Figure 6. Texture values in (a) Ni-nano  $Al_2O_3$  (b) Ni-submicron  $Al_2O_3$  electrodeposited at current density of  $4A/dm^2$  with 20 g/l particle concentration into Watts's electrolyte

According to the crystal mapping which has been done specifically for the coatings with nano and submicron particle for the concentration of 20 g/l, as shown in Figure 6, the co-deposition of  $\text{Al}_2\text{O}_3$  obviously affects the relative intensity corresponding to different crystal faces. The relative intensity corresponding to crystal face (1 1 1) increases, but the relative intensity of crystal face (2 0 0) decreases. For composite coatings with nanoparticles, the peak of crystal face (2 2 0) is more obvious. It seems that also the peak of (311) becomes stronger with increasing of  $\text{Al}_2\text{O}_3$  content. Erler et al. (Erler, 2002) demonstrated that the XRD patterns of nickel nano-composite coatings indicate changes in texture of such coatings which are dependent on the particle content in electrolyte. Chang et al. (Chang, 2003) was also presented similar results.

Figure 7 exhibits the grain size of the Ni matrix for the samples estimated from Scherrer's equation using the XRD peak width. In fact, the reduction in the grain size of the Ni crystal by adding particles approves the compactness of the coatings as also confirmed by the results of the morphology. It can be seen that, by increasing the concentration of particles in the electrolyte, on the one hand, the incorporation value for the case submicron particles will be higher than nano particles; but, on the other hand, the grain size value shows a higher reduction compared to when nano particles are added. The reason here is that smaller particles can offer more sites for nucleation, which is detrimental for the growth of Ni crystal while on larger particles; Ni crystal finds more opportunity to grow (Zhang, 2014).

One possible mechanism to explain the codeposition behavior of particles here is the phenomenon proposed by Guglielmi (Guglielmi, 1972). In the first step, the particles in the electrolyte clouded by nickel ions and due to the agitation in the electrolyte, clouded particles approach the cathodic surface and there, a certain amount of particles can be trapped and buried by reducing new ions over them. According to the thickness measurements in figure 8, it is understandable that in lower particle concentrations till 5 g/l, the possibility of transferring Ni ions through the particles is high such as the value of the thickness at the given concentration is large, namely around 65  $\mu\text{m}$ . At the higher concentration of 20 g/l, however, the thickness values for both particle types showed a declining trend. It is possible that the addition of more particles into the solution could block the cathodic surface and therefore, a more active area for the reduction of Ni ions.

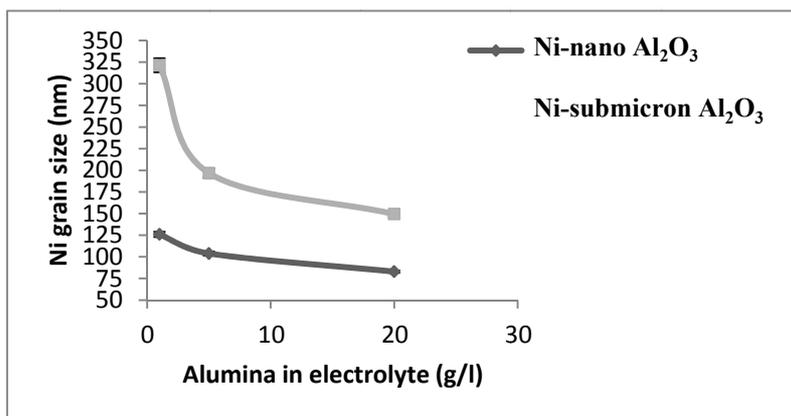


Figure 7. Effect of particle size and concentration on the Ni grain size

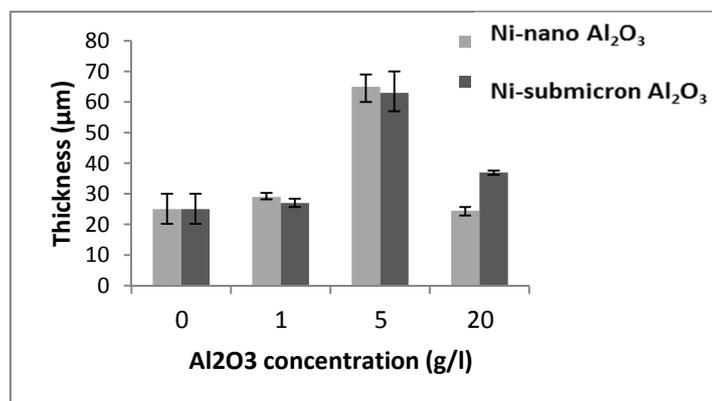


Figure 8. Effect of particle size and concentration on the thickness value of the deposits electrodeposited under the same conditions

Nevertheless, at the higher concentration of 20 g/l, the thickness of the layer deposited with submicron particles is higher than the layer deposited with nano particles,  $37\pm 0.7$  and  $25\pm 1.2$   $\mu\text{m}$  respectively. The reason here, as explained above, can be the better opportunity of Ni crystals to grow on larger particles rather than on smaller ones. Since the particle hinders the current flow, the local overvoltage should rise respectively, thus the number of crystallization sites would be increased.

The Martens hardness of Ni- $\text{Al}_2\text{O}_3$  composite coatings was also carried out on the cross section of the layers by means of the Martens test as it was already explained in the experimental section (Chang, 2003)

According to the results of the Martens hardness presented in figure 9, it can be discerned that the Martens hardness of the Ni- $\text{Al}_2\text{O}_3$  composite coatings electrodeposited with both nano- and submicron particles follow an increasing trend by increasing the particle concentration in the electrolyte. It is obvious that the addition of nano particles leads to a higher Martens hardness at each single concentration in contrast to the addition of submicron particles. According to the results of the Martens hardness and the suggested codeposition mechanism for alumina particles with nano and submicron size, it can be concluded that, even though the codeposition can arise easier for the larger particles, which can be related to easier transfer of the larger particles from the double layer to the surface, this does not mean that in the case of larger particles, the nucleation rate of nickel and subsequently, the hardness value should also necessarily be higher.

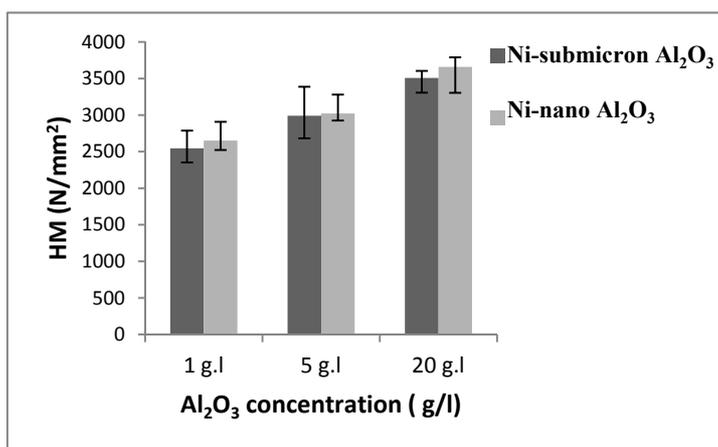


Figure 9. concentrations including the effect of grain size and particle content

The Martens hardness can be controlled by dispersion hardening or the amount of dispersed particles embedded in the layer based on the Orowan mechanism (Epelboin, 1971). As it could be seen, by increasing the concentration of particles in the electrolyte, the incorporation value for submicron particles will be higher than that of nano particles but on the other hand the number of incorporated particles in the case of applying nano particles is higher. This means that the possibility of hindering the dislocations in the coating deposited by nano particles is higher. Moreover, as it was already seen, the grain size of the nickel matrix is smaller in the case of nano particles. This means that the grain size of the matrix, based on the Hall-Petch effect (Orowan, 1984), plays an important role in the hardening mechanism of coatings.

In order to shed more light on our assumptions about the codeposition behavior of alumina particles with different sizes and concentrations, impedance spectroscopy was used to explain the effect of particle features on the behavior of the charge transfer and double layer capacity for the Ni electrodeposition.

### 3.2 In-situ EIS Study of Ni- $\text{Al}_2\text{O}_3$ Electrodeposition

Figures 10 (a-c) show the Nyquist plots from electrochemical impedance spectroscopy results during the electrodeposition process for Ni and Ni- $\text{Al}_2\text{O}_3$  with particle nano- and submicron particles and different concentrations of 1, 5 and 20 g/l under the same deposition conditions. The impedance data comprises of a single capacitance loop in high and medium frequencies and an inductive loop in lower frequencies.

According to the mechanism of Ni electrodeposition proposed by Epelboin and Wiart (Wiart, 1990; Epelboin, 1971), the simple circuit model excluding inductive in very low frequencies was suggested in this study. Therefore, the impedance measurements could be achieved much faster within a shorter range of frequency and provide us with information about charge transfer and double layer capacity, which can be directly related to the Ni reduction. Nevertheless, the trace of inductive loops is indeed visible in rather lower frequencies. For simplicity we have just considered the effect of particles on higher frequencies which can achieve faster without variations in working parameters during

electrodeposition process such as pH. In this model,  $C_{dl}$  is the double layer capacitance of the interface of the electrode/electrolyte, and  $R_{dl}$  is a resistance against the charge transfer through the double layer at the electrode/electrolyte interface. As it was seen in figure 10, the loop size for both particle sizes was found to decrease with increasing particle concentration till 20 g/l. Moreover, as it is seen in the figure 10c, at the concentration of 20 g/l, the usage of submicron particles caused higher shrinkage in the semicircle of Nyquist spectrums compared to the addition of nanoparticles to the electrolyte.

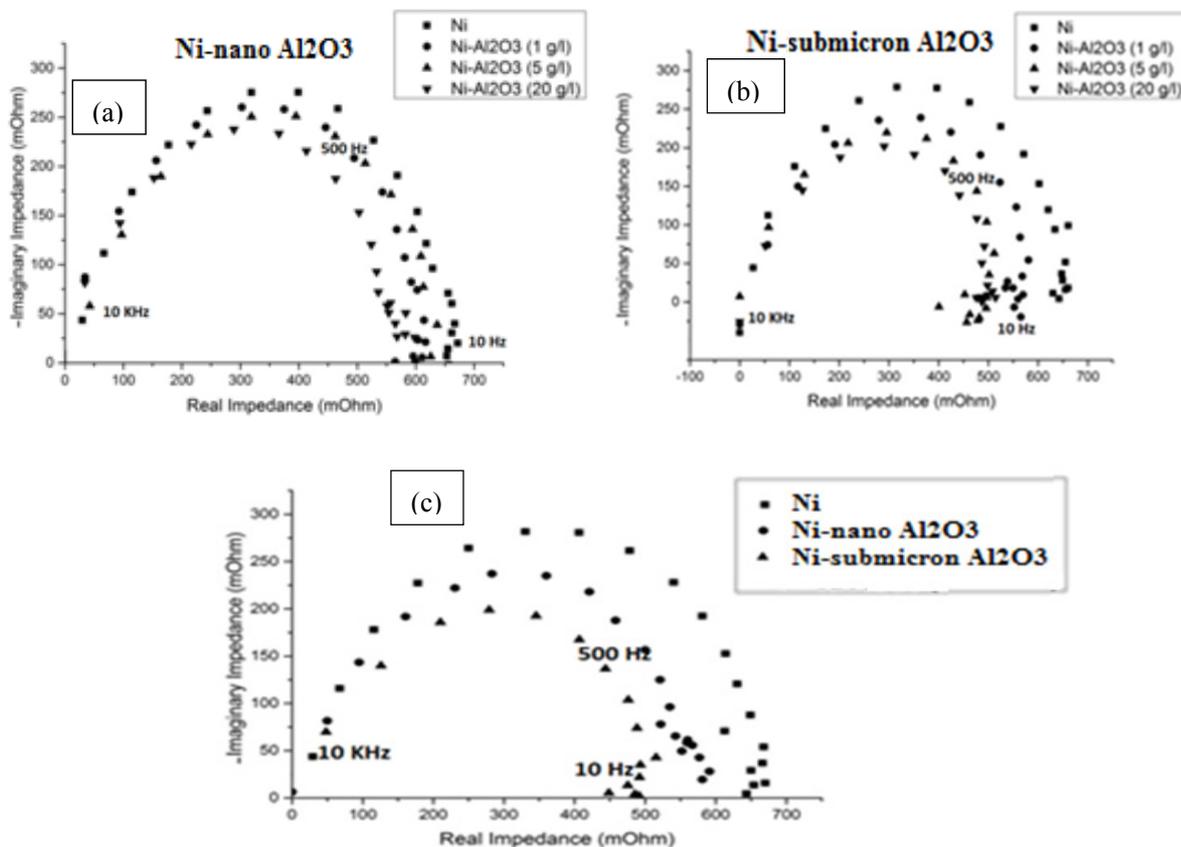


Figure 10. Effect of particle size and concentration on the Nyquist spectrums:

(a) Ni-nano  $\text{Al}_2\text{O}_3$  (b) Ni-submicron  $\text{Al}_2\text{O}_3$  (c) comparison at the concentration of 20 g/l

Increasing the concentration of the particles resulted in the decrease of the charge transfer resistance or the increase of the double layer capacity as it was also shown by Benea or Dolati for SiC particles (Sohrabi, 2012; Benea, 2001). As it can be deduced from the impedance results in figure 10, a descending trend in the impedance of the charge transfer as well as an ascending trend in the doubled-layer capacity was observed by increasing the  $\text{Al}_2\text{O}_3$  particle concentration till 20 g/l. According to the impedance results, by the increase in the concentration of the particles in the electrolyte, the possibility that nickel ions are transferred by means of the particles towards the cathodic surface is higher. In the other words, the declines in the thickness of the double layer can finally influence the nickel reduction. Moreover, according to the results of figure 12, the value of the charge transfer resistance for submicron particles is lower than that of the nano particles, and the double layer capacity for larger particles is moderately higher compared to smaller particle sizes.

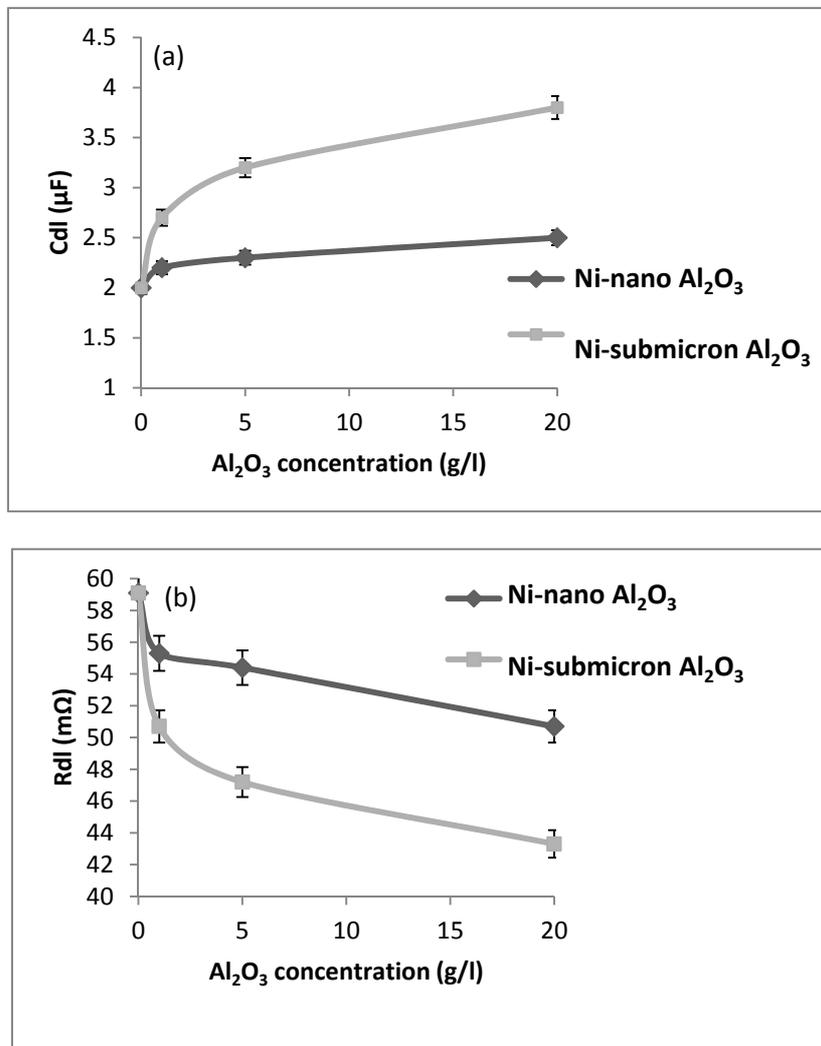


Figure 12. Results of  $C_{dl}$  (a) and  $R_{dl}$  (b) calculated by fitting the impedance results with the suggested circuit model by means of Matlab

This means that the thickness of the double layer on the cathodic surface is less than that of nano particles in the case of adding submicron particles. Thus, as it is illustrated in the schematic of figure 13, submicron particles can be easier incorporated into the deposit than nanoparticles due to having a larger active surface for free ions and a higher mass transfer. Nevertheless the deposition of nickel at the higher concentration of 20 g/l arises faster due to the higher charge transfer and double layer capacity. The reason for the decline in the thickness of coatings or for the reduction of the thickness of deposits at the higher particle concentration of 20 g/l, as it is seen in figure 13b, can be partly related to the blocking of the active surface of the cathode due to the accumulation of particles on the surface.

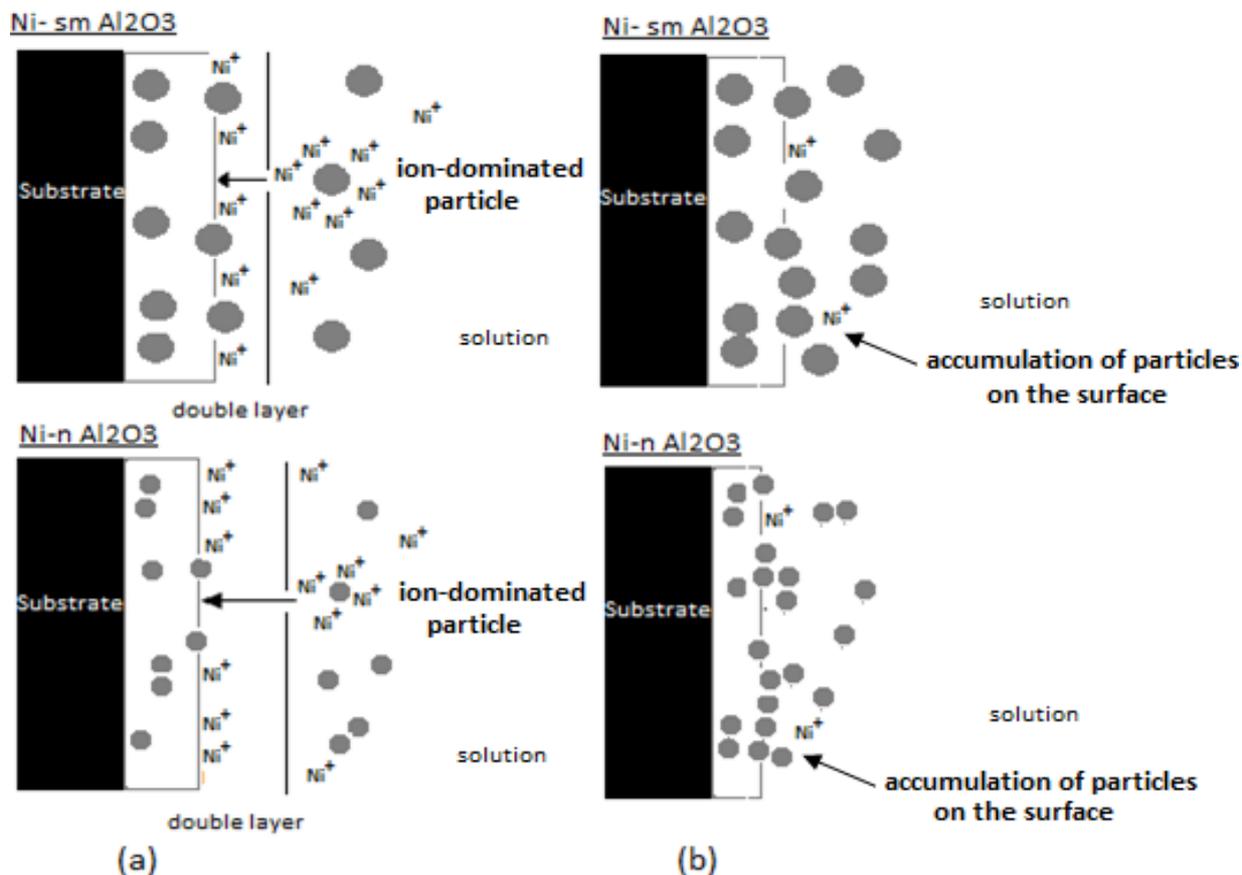


Figure 12. Schematic of the particle incorporation mechanism based on particle size and concentration:

(a) at low concentration of particles (b) at high concentration of particles

#### 4. Conclusion

A descending trend in the impedance of the charge transfer as well as an ascending trend in the doubled-layer capacity was observed by increasing the  $\text{Al}_2\text{O}_3$  particle concentration till 20 g/l. The value of the charge transfer resistance for submicron particles is also lower than that of nano particles, and the double layer capacity for larger particles is moderately higher compared to smaller particle sizes. The results of the impedance measurements considering the layer characteristics could correspond well with the mechanism proposed by Guglielmi. The Martens results showed that for both nano- and submicron alumina particles, increasing the particle concentration to 20 g/l leads to highest hardness values. Based on the results of the Ni grain size, despite of the lower incorporation value of nano particles compared to submicron particles, the hardness ability of coating deposited with nano particles is rather higher than of the coatings prepared with submicron particles even with higher incorporation value. For both coatings produced with submicron and nano particles, at the middle concentration of 5 g/l, the current efficiency is much higher and both of the coatings show an almost similar strengthening performance, which relates to the simultaneous effect of Hall-Petch and the Orowan mechanisms.

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