International Journal of Minerals*, *Metallurgy and Materials
Volume 26*, *Number 5*, *May 2019*, *Page 649
https://doi.org/10.1007/s12613-019-1773-1

Nanoscale electropolishing of high-purity nickel with an ionic liquid

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(Received: 11 August 2018; revised: 14 January 2019; accepted: 15 January 2019)

Abstract: High purity (>99.9% composition) nickel metal specimens were used in electropolishing treatments with an acid-free ionic liquid electrolyte prepared from quaternary ammonium salts as a green polishing solution. Voltammetry and chronoamperometry tests were conducted to determine the optimum conditions for electrochemical polishing. Atomic force microscopy (AFM) revealed nanoscale effectiveness of each polishing treatment. Atomic force microscopy provided an overall observation of the material interface between the treated and unpolished regions. Surface morphology comparisons summarized electrochemical polishing efficiency by providing root-mean-square roughness averages before and after electrochemical polishing to reveal a mirror finish six times smoother than the same nickel metal surface prior to electropolishing. This transition manifested in a marked change in root-mean-squared roughness from 112.58 nm to 18.64 nm and producing a smooth mirror finish. Finally, the mechanism of the ionic liquid during electropolishing revealed decomposition of choline in the form of a transient choline radical by acceptance of an electron from the nickel-working electrode to decompose to trimethylamine and ethanol.

Keywords: electrochemical polishing; ionic liquid; Ni; 2EG:1VB$_4$

1. Introduction

The field of electrochemical polishing continues to expand as literature suggests that commercial methods of acid-based electropolishing can be replaced with non-hazardous alternatives based on ionic liquid mediums [1]. Samples of high grade metals of nickel (99.95%) are frequently utilized components in electrochemical treatment studies [2]. This investigation utilized an environmentally-friendly, conducive ionic liquid treatment for five high-purity nickel metal samples at 70°C for 900-s electropolishing treatments.

An ionic liquid is a liquid that consists only of ions [3]. Ionic liquids serve as a safe alternative to the use of phosphoric and sulfuric acid mixtures, thus encouraging their use in large-scale applications such as metal deposition and electropolishing [4–5]. A prominent ionic liquid composed of ethylene glycol ($HOCH_2CH_2OH$) and choline chloride ($HOC_2H_4N(CH_3)_3{}^+Cl^-$) was used in this paper. Often abbreviated as 2EG:1VB$_4$, this solution has been used to successfully electropolish various metal alloys, but has only been used in a limited capacity to polish pure rare earth metals, such as nickel, the focus of this paper [6–7]. Electropolishing is often shown to perform a smoother mirror finish than magnetoelectropolishing plasma electrolytic polishing, pulse/pulse reverse electropolishing, and electrochemical jet machining, when compared with alternative methods of micro-to-nanoscale surface refinishing [3].

Nickel is useful for a variety of practical material applications, and is directly utilized in the construction of mobile phones, medical equipment, transportation infrastructure, buildings, and power generators [8]. Nickel offers superior corrosion resistance, better toughness, and sufficient strength at variably high and low temperatures relative to its somewhat low material cost [9–10]. There are also several special magnetic and electronic properties nickel possesses that make it a somewhat utilitarian metal compared with other materials. Nickel metal is a key part of several rechargeable battery systems including the commercially popular nickel metal hydride

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cells used in electronics, transportation, and power tools [11].

While the focus of this research is on pure nickel metal, it is important to note that alloys of iron, nickel, and chromium are heavily used in electronics. Nickel is also incorporated into various forms of stainless steel in engineering applications, which typically contain 8%–12% nickel content, and copper-nickel alloys are used for coinage and marine applications [7]. Furthermore, nickel metal can be engineered to form effective protective coatings like ‘nickel-plating,’ ‘electroless nickel coating,’ or ‘electroformed nickel surfaces’ [8–10].

It is the interest of this study to survey pure nickel metal samples and compare their results to those of other pure metals of interest that are commonly electropolished for scientific, commercial, or biomedical applications [6,7,12–15]. A pure metal is any metal that is deemed to possess greater than 99.5% of its composition in one type of metal. Some slight impurities exist in most metals, making a 100% pure sample of any of the metal types used in this section unlikely or impossible to achieve [16]. In this study, pure metals of nickel, not uncommon in the field of electropolishing, were studied under the effects of an ionic liquid medium.

The process of using ionic liquids to electropolish conductive surfaces has been gaining traction recently, as it results in a chemically cleaner treatment without the need for hydrogen degassing, typically required after electropolishing treatments with strong acids like HNO_3, or HF [17]. Additional benefits to polishing nickel and other pure metals with ionic liquid solutions, such as the one used in this study, include decreased cost of materials over electropolishing treatments with acid-based electrolytes, as the cost to acquire and dispose of these industrial acidic solutions is higher, and the acids are inherently more caustic to handle [18]. Also, with the growing applicability of nickel plating in material goods to provide hard-wearing decorative and engineering coatings referred to in recent literature as ‘nickel-plating’ [8], ‘electroless nickel coating’ [9], or ‘electroforming’ [10], the ability to effectively reclaim the dissolved pure nickel metal after electropolishing from an ionic liquid solution by reversing the cathode and anode to electroplate it to another conductive surface is an option afforded by this method [19–20]. In the interest of generating a smooth surface for electrochemical polishing, pure metal specimens of nickel (99.95%) were electropolished to produce a cleaner and smoother finish, then measured to characterize their nanoscale roughness.

2. Experimental

2.1. Materials and apparatus

In each experiment, an ionic liquid was prepared, consisting of two ingredients: choline chloride (Acros Organics 99%), and ethylene glycol (Sigma-Aldrich 99.8%), both chemicals being used as-received. The ionic liquid was created by stirring the two chemicals together at a 2:1 ratio of ethylene glycol to vitamin-B_4 (2EG:1VB_4), respectively, at 70°C until a homogeneous colorless liquid remained [17]. This ionic liquid’s effects on electropolishing the pure metal surfaces of interest were analyzed using the necessary machines. Voltammetry and amperometry were carried out using a Gamry PCI4-G750 potentiostat and controlled using the accompanying ‘framework’ and ‘e-chem Analyst’ software.

The electropolishing procedure made use of a platinum plate electrode with a silver wire as a reference electrode for the experimental setup. Both electrodes were degreased using deionized water and acetone to preserve the purity of the nickel specimens during testing. The working electrode was abraded with 150-grit glass sand paper, and was subsequently rinsed with deionized water and dried prior to each recorded measurement to ensure reproducible voltammetric effects. The nickel metal electropolishing sample region had a surface area of 2 cm^2, distributed between two 1 cm^2 treatment areas on each sample, bounded by polyimide film tape to control the electropolishing region. To clarify, one 1 cm^2 is present on the exposed front face, while the other 1 cm^2 treatment region is on the back face of the thin nickel metal totaling 2 cm^2 for the electropolishing area used when computing current density among nickel samples.

Once prepared, a series of experiments involving chronoamperometry and linear sweep voltammograms were used to determine the optimal relationship between voltage and current for nickel (Fig. 1). Usually, excessive electric current will cause grain boundary attack and pitting on the anode, however too much voltage can result in the formation of an oxide layer. Electrochemical measurements were performed at 70°C with a constant scan rate of 20 $mV\cdot s^{-1}$ used in voltammetric experiments.

Setting diffusion at the anode as the rate limitin28g step, chronoamperometric and voltammetric analysis revealed advantageous current potentials for successful electropolishing of nickel metals at optimum settings during 900-s experimental durations at 70°C. Upon completion of electropolishing procedures, metal surfaces were thoroughly cleansed with deionized water, dried, and subsequently surveyed for root-mean-square roughness average calculations via atomic force microscopy (AFM). Coupled with the additional insight afforded by enhanced digital microscopy, information regarding surface morphology and relative conformity of surface smoothness was easily compared to benchmark each metal’s industry-produced counterpart.

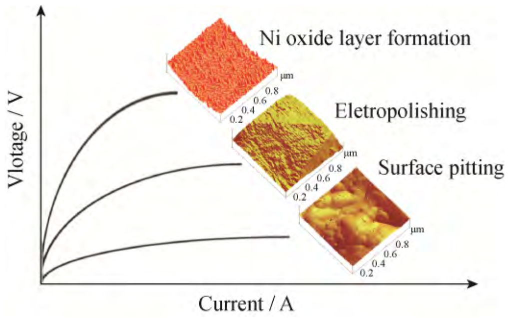


Fig. 1. Schematic diagram describing the optimal relationship between voltage and current for nickel electropolishing and examples of resulting 10 μm × 10 μm surface characteristics via AFM micrographs.

2.2. Surface characterization

AFM images were obtained using a Dimension 3100 Digital Instruments-manufactured Nanoscope IV Scanning Probe Microscope with accompanying software in tapping mode with a frequency of 300 kHz, a drive amplitude of 310 mV, and a 1-μm maximum range above the surface of the sample.

The electrochemical polishing assay was developed to improve the degree of smoothness that may be afforded to conductive surfaces over mechanical polishing methods. In this application, significant parameters were measured in terms of relative roughness of the samples and subsections of the samples (referenced as box statistics in the figure) as functional height parameters. Among these parameters, two are frequently utilized to characterize the relative smoothness of a surface, those being roughness average (R_a), and root-mean-squared roughness (R_q). R_a is most widely used, due to the relative ease of calculation for surface profiling equipment, and was calculated via AFM with the following numerical formulation:

$$R_a = \frac{1}{L}\int_0^L |Z(x)| \mathrm{d}x \tag{1}$$

where $Z(x)$ is a function of the assessed surface profile in terms of height (Z) and position (x) of the sample over the evaluation length (L), such that R_a is the arithmetic mean of the absolute values of the height of the surface profile $Z(x)$ [21].

Apparent advantages to comparing samples using R_a include the potential repetition of measuring similar heights among other samples of the same height parameter. This indicates statistical robustness, which explains its use as a recommended smoothness parameter in the characterization of random surfaces, as it is usually used in industrial applications to describe machined surfaces [22].Since the average roughness explicitly represents the mean absolute profile, without regard for distinguishing between individual peaks and valleys in sample surfaces, it can be non-advantageous to characterize nickel surfaces using R_a if these data are relevant. Thus, more sophisticated parameters such as R_q may be used to supplement R_a to fully characterize a surface, such as when distinguishing between peaks and valleys is needed for more significant roughness information. R_q is a function that takes the square of the measures, distinguishing itself from the R_a by being the mean-squared absolute values of the surface roughness profile [23]:

$$R_q = \sqrt{\frac{1}{L}\int_0^L |Z^2(x)| \mathrm{d}x} \tag{2}$$

As a result of the squaring of the amplitude in its calculation, the R_q is more sensitive to peaks and valleys than the R_a, and depends on the scan size/surveyed area of the sample [24].

3. Results and discussion

3.1. Electrical potential

Linear sweep voltammograms for nickel revealed a local minimum (0.004 A/cm^2, 1 V) and maximum (0.008 A/cm^2, 0.5 V) to be present early in the curve for nickel metals treated with the ionic liquid eutectic mixture (Fig. 2). The increase in current density continued to rise until 4 V was reached, before the effective electropolishing range was diminished. Beyond 4 V, electropolishing conditions became irreproducible via linear sweep voltammetry calculations.

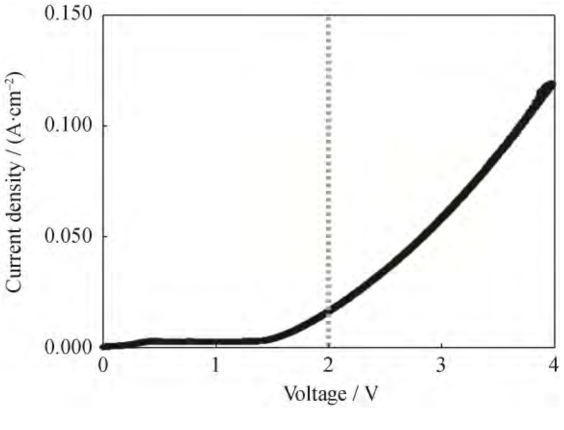


Fig. 2. Nickel sample displayed running linear sweep voltammetry for a scan rate of 20 mV·s^{-1} in step from 0 to 4 V. The electrochemical setup idled at 70°C while the current ranged from 0 to 0.127 A/cm^2 revealing a local minimum (0.004 A/cm^2, 1 V) and maximum (0.008 A/cm^2, 0.5 V). The dashed line indicates the ideal voltage utilized for chronoamperometry in Fig. 3.

Nickel electropolishing was conducted at 2 V, since there was a miniscule difference in current between the local minimum (0.004 A/cm^2, 1 V) and maximum (0.008 A/cm^2, 0.5 V) prior to the nearly exponential increase in current density in the linear sweep experiment. Given the conceptual relationship in Fig. 1, a voltage of 2 V was selected to provide suitable conditions for (a) a smooth polish for nickel without overly high voltage and low current, which would invite Ni oxide build up, and, (b) to likewise avoid the low voltage and high current conditions that can promote nanowave or nanocavities or potenially cause surface pitting.

The 2 V point was chosen as ideal, related to the local minimum reported from the linear sweep voltammogram for nickel from 0–4 V (Fig. 2). The experiments began with an observed immediate burst of slow bubbling at the commencement of electric current through the ionic liquid electrolyte solution. The irregular bubbling reaction decreased significantly after the first interval of measurements, peaking at a current density of 0.021 A/cm^2. It became apparent that any film barrier on the metal surface of nickel was removed in the first 15 s of chronoamperometry when fixed at 2 V (Fig. 3). Electrical current dwindled to 0.013 A/cm^2 from 480 s to the end of electropolishing at 900 s, operating at a critical low for current density at 2 V. These slow low-voltage polishing conditions produced favorable surface roughness results.

A prior average mass balance across all five samples was recorded as 6.766 g to compare with a post-electropolishing weight of 6.705 g, to yield a calculated mean difference of (0.060 ± 0.005) g, for an overall electropolishing rate of 66.8 μg/s (Table 1). Thus, a minimal amount of surface material was removed from the high-purity nickel metal in the electropolishing procedure. Observed roughnesses obtained using an AFM revealed a calculated R_q equivalent of (138.41 ± 5.48) nm, and an R_a of (112.58 ± 4.45) nm prior to treatment of nickel (Fig. 4). Post-treatment results revealed a R_q of (28.41 ± 1.10) nm and an R_a of (18.64 ± 0.72) nm (Fig. 5). This resulted in calculated roughness reductions of (110.00 ± 4.38) nm and (93.94 ± 3.73) nm, for R_q and R_a, respectively (Table 2).

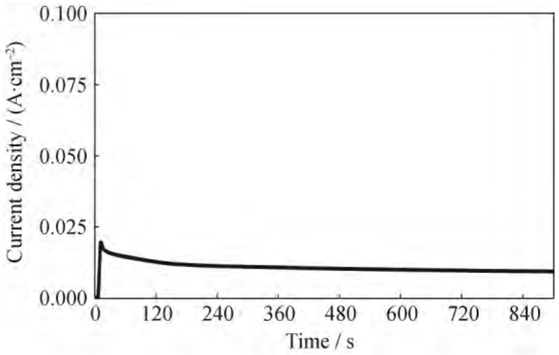


Fig. 3. Nickel sample shown running chronoamperometry for a 900-s electropolishing procedure with an ionic liquid. Voltage fixed at 2 V with a temperature of 70°C and current ranging from 0 to 0.0196 A/cm^2.

Table 1. Average and standard deviations before and after assessments of mass and electropolishing rate (μg/s) calculations for each metal sample over a 900 s treatment period

Ni sample No.	Mass before electropolishing / g	Mass after electropolishing / g	Mass differential / g	Surface degradation rate / (μg·s^{-1})
#1	6.769	6.713	0.056	62.2
#2	6.761	6.701	0.060	66.6
#3	6.775	6.713	0.062	68.8
#4	6.759	6.703	0.056	62.2
#5	6.764	6.697	0.067	74.4
Average	6.766	6.705	0.060	66.8
Std. deviation	0.006	0.007	0.005	5.10

3.2.Mass reduction and elecropolishing efficiency

Nickel metal is noted as having a mass loss of 0.060 g from pre-polishing to post-polishing procedures, significant to $p < 0.001$ (8.149×10^{-6}) via a t-test comparing sample weights before and after electropolishing treatments (Table 1). The metal displays an R_q roughness decrease of 110.00 nm and an R_a decrease of 93.94 nm, as reported in Table 2. Nickel metals later electropolished conveniently for a roughness of 28.41 nm (Fig. 5), when compared to an original sample R_q root-mean-square roughness average of 138.41 nm (Fig. 4), resulted in a smoothing efficiency of 79.308% ± 3.17% (Table 2).

A reasonably high relative smoothness for nickel metals may be achieved using the electropolishing method described in this study. Relative to other pure rare earth metals, electropolishing of nickel results in a higher roughness average than for silver, but a lower average than for copper or aluminum in a survey of past electrochemical studies [19]. Overall, this accounted for a mean nickel surface degradation rate of 67.667 μg/s (Table 1), resulting in a slightly slower rate than those reported for electrochemical polishing using 2EG:1VB4 for pure silver metals of 72.333 μg/s [13,25].

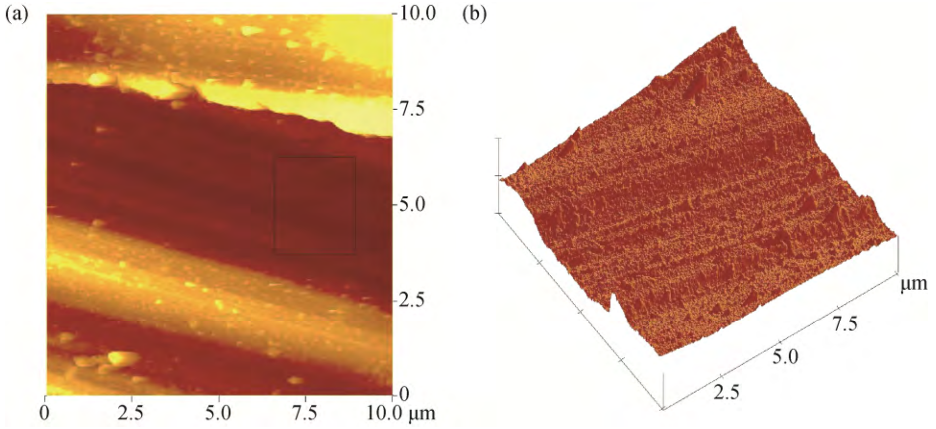


Fig. 4. AFM roughness analysis of a nickel sample in 2D (a), and 3D (b), prior to electropolishing treatments — recording an R_q of 138.41 nm utilizing the root-mean-square method for calculation, and an R_a of 112.58 nm. A 10 μm × 10 μm recording region was utilized.

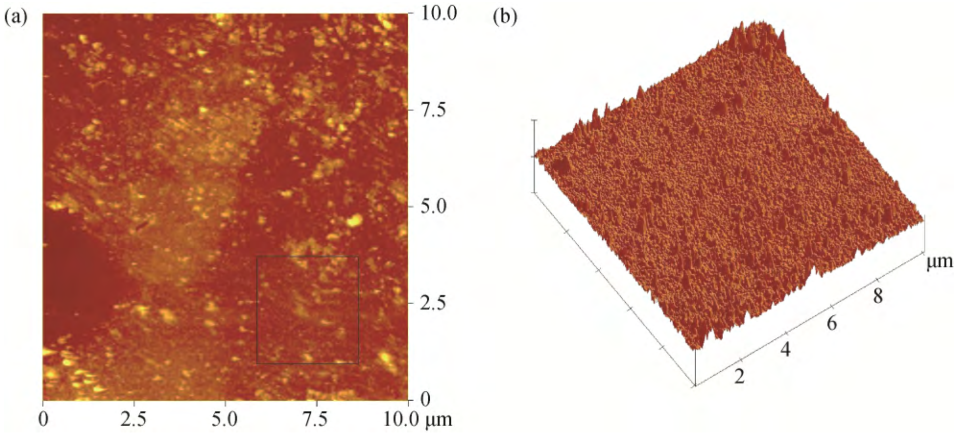


Fig. 5. AFM roughness analysis of a nickel sample in 2D (a), and 3D (b), after electropolishing treatments — recording an R_q of 28.41 nm utilizing the root-mean-square method for calculation, and an R_a of 18.64 nm. A 10 μm × 10 μm recording region was utilized.

Table 2. Averages and differences for R_q and R_a (nm) for five nickel metal samples prior to and post-electropolishing (EP) treatments with ionic liquid solution at 70°C for 900 s. Calculated differences determined smoothing efficiency (SE) for each sample

Surface roughness of Ni sample	Average before EP / nm	Average after EP / nm	Difference / nm	SE / %
R_q	138.41	28.41	110.00	79.308
R_a	112.58	18.64	93.94	

A measured difference of 181.450 nm for silver with a reported 82.906% electropolishing efficiency ultimately reveals that silver yielded a slightly higher surface degradation rate than the nickel tested in this study during polishing [25]. However, nickel metal treatments provided for an R_q of 28.41 nm (Fig. 5), while the silver samples yielded less smooth surfaces, on average, with a post-treatment R_q roughness of 31.017 nm [25]. Fig. 6 depicts a schematic comparison before and after electropolishing of the nickel, to illustrate that the rate of dissolution was greatest at exposed peaks in the metal surface prior to electropolishing due to the increased surface area exposed to the ionic liquid solution. Thus, the metal oxidation rate was greatest at these hills, relative to the neighboring troughs or valleys in the exposed metal surface, polishing away as much as 734.28 nm in vertical height (Fig. 5) over the 900-s treatment period (Table 2).

This information only further supports the point that faster electropolishing rate may not necessarily indicate a more efficient polish in terms of sample smoothness. The conditions for electropolishing seem to indicate quite the opposite conditions, whereas it may be beneficial to facilitate a stable low current density condition for electropolishing to provide for the most efficient settings with each unique metal for polishing [26]. This is achieved via targeting the local minimum setting directly after the first peak in linear sweep voltammograms [27–28]. It has been noted in some literature sources that while a higher current density can provide for a faster electropolishing rate, the current density becomes more variable, as erratic fluctuations in the current can cause pitting at the metal surface as a result of the vigorous redox reaction at work [29–31]. Finally, the degree to which one is able to reclaim the dissolved nickel metal from the ionic liquid using electroplating procedures could be affected by the relative rate of electropolishing if the reaction is not stable [32].

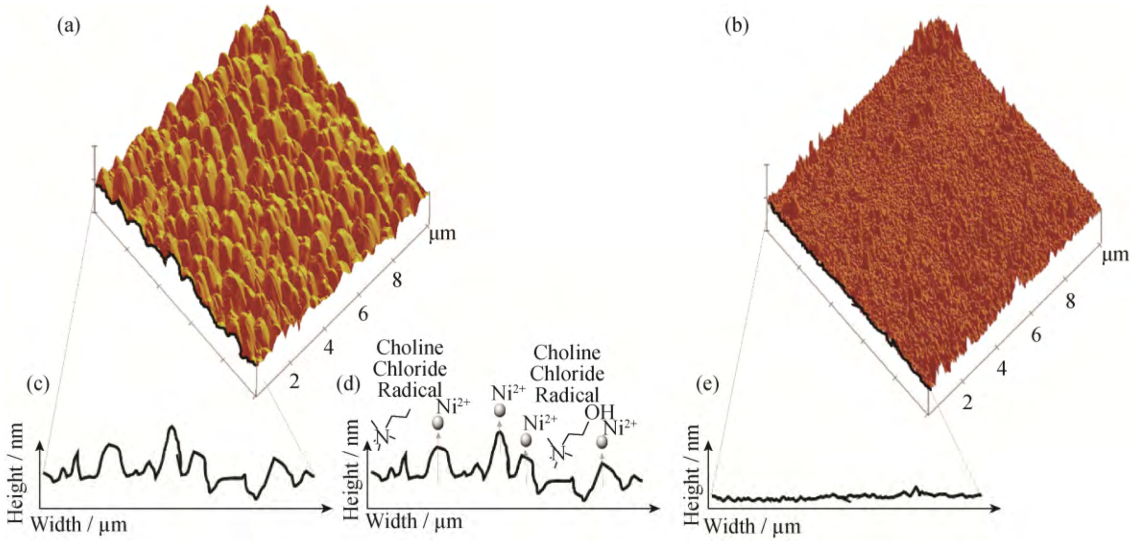


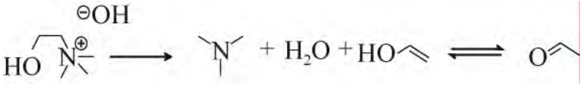
Fig. 6. Schematic explanation of anodic leveling of nickel via AFM before electropolishing (a), and after electropolishing treatments (b), with representative 2D surface profiles depicted before the experiment (c), during the experiment (d), and after treatments (e). Vertical height scales are the same across all micrographs and profiles.

3.3. Observations of the mechanism of electrolysis

For the 900-s experimental duration, the ionic liquid's hue became slightly blue, as did the nickel sample surface due to the oxidation of Ni and formation of $NiCl_2$ (Eq. (3)):

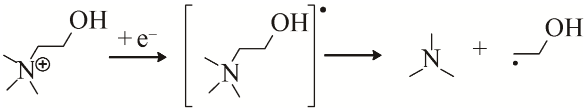
$$\mathrm{Ni} \rightarrow 2\mathrm{e}^- + \mathrm{Ni}^{2+} \xrightarrow{2\mathrm{Cl}^-} \mathrm{NiCl}_2 \qquad (3)$$

Haerens *et al.* [33] observed the presence of trimethylamine, ethenol, ethylene glycol, and other products, with the incidence of trimethylamine being accounted for by Hoffman elimination of the choline base (= choline hydroxide, Eq. (4)):



(4)

The reaction at the cathode involves the decomposition of choline by formation of a choline radical via acceptance of an electron:



(5)

Thus, the transient choline radical, shown in parentheses in Eq. (5), resulting from the addition of an electron from the anode at the cathode, quickly decomposes to trimethylamine and an ethanol radical. The relative instability and decomposition of the choline base is well-documented [33].

4. Conclusions

In this study, the rate of smoothing pure metal specimens of nickel was found to be significantly linked to electropolishing efficiency. Nickel metals were smoothed to form a mirror finish via electrochemically polishing from an R_q of 138.41 nm to an R_q of 18.64 nm, becoming measurably smoother by an order of six and resulting in a smoothing efficiency of 79.308%. Thus, the following relevant conclusions can be made:

(1) Nickel metal has been shown to display a mass loss of 0.060 g from pre-polishing to post-polishing procedures, significant to $p<0.001$ (8.149×10^{-6}) via a t-test comparing sample weights before and after electropolishing treatments.

(2) The insurmountable correlation of electropolishing rate seems to hold true for nickel pure metals, and reveals that the faster the electropolishing rate, the more likely the reduction reaction occurring at the electrode will form a vigorous bubbling reaction that is detrimental to the overall roughness average of the sample.

(3) It can further be supported that the greater the current

density present across an exposed metal surface during electropolishing, the more likely the surface is to experience pitting conditions associated with low current density fluctuations.

(4) The mechanism of 2EG:1VB_4 observed at the nickel-working electrode revealed decomposition of choline by formation of a choline radical via accepting an electron at the cathode to decompose to trimethylamine and an ethanol radical.

From this study, it has been determined that pure nickel metals can be successfully electropolished using an ionic liquid consisting of 2EG:1VB_4, and that the rate of electropolishing does, indeed, result in a statistically significant improvement in surface roughness.

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