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Electrochemical and Microstructural Characterization of Cr-Coated NdFeB in Neutral Wet Environment

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Abstract

Naturally, NdFeB–based permanent magnets exhibit high chemical instability, especially, in wet environments. This makes them highly susceptible to corrosion, which leads to severe structural collapse. Applying thin surface coatings can improve the resistance to wet corrosion. In this work, 0.5 µm of chromium (Cr) film was sputtered on a sintered NdFeB–based magnet and the resistance to corrosion and microstructural damage was investigated in 3.5% NaCl solution with respect to an uncoated magnet. This was achieved by complementing potentiodynamic polarization and electrochemical impedance spectroscopy with scanning electron microscopy (SEM). The Cr coating caused a positive displacement of corrosion potential and lowered the rate of anodic dissolution. It also favored higher charge transfer resistance at the magnet–solution interface, and delivers a protection efficiency ~60 %. Furthermore, grain boundary attack and deterioration were also significantly reduced by the Cr coating.

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1. Introduction

Permanent magnets are important components of electronic devices used in automobiles, aerospace, medicals and even communications [1-3]. In recent times, permanent magnets composed of a rare earth metal (RE) and a

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transition metal (TM), also known as RE-TM magnets, are gaining significant relevance because of greater mass–to–performance ratio and easier fabrication [4-6]. Microstructurally, NdFeB magnets consist of a Fe($Nd_2Fe_{14}B$)-rich ferromagnetic phase, a Nd–rich Nd_4Fe phase and a B–rich $Nd_{1+\varepsilon}Fe_4B_4$ phase. Due to these multiphase microstructure and intergranular phases, NdFeB magnets exhibit poor chemical stability and corrosion resistance which causes them to lose magnetic grain cohesion and commercial value [7–12]. Protecting these magnets from corrosion is imperative, provided it does not interfere with their magnetic properties.

Alloy doping and surface modification are the most adopted approaches for protecting magnets against corrosion [13-18]. Alloy doping increases corrosion resistance by reducing the potential difference between the different phases. Nevertheless, the approach may significantly damage the magnetic property. Consequently, surface modification by applying thin films of single or multi-element metallic coatings has become the most adopted method for protecting NdFeB magnets against corrosion. Some reported multi-element metallic coatings include Al–Mn [19], Ni–TiO_2 composite [20], Al/Al_2O_3 multilayers [21], Ti/Al multilayers [22], Ni–Co alloy [23]. On the other hand, single metal coatings are cheaper and easier to deposit on the magnet surface. Al thin film is the most visible single metal coating reported for enhancing the resistance of NdFeB magnets to corrosion [24, 25]. The Al coating provided long-term corrosion protection courtesy of protective Al_2O_3 film formation on the magnet surface.

Like the Al film, applying a thin film of chromium (Cr) on the magnet surface could also increase its corrosion resistance through the formation of the highly protective Cr_2O_3 layer, as have been shown for Cr films on some metallic materials [26]. While electrodeposition is a well-known technique to fabricate Cr films on metal substrates, the technique can slightly distort the magnetic properties of the substrate. Thus, the use of sputtering techniques is a preferred alternative and has been well demonstrated for the Al films. So far, there is no visible work in the literature where Cr coating has been investigated for improving NdFeB magnet corrosion resistance. This is the motivation for this present investigation. Consequently, we apply magnetron sputtering to deposit a thin film of 0.5 μm–thick Cr on NdFeB magnet and, thereafter, investigate its corrosion performance compared with a commercial sintered NdFeB magnet, in 3.5% NaCl solution. We employ electrochemical techniques like electrochemical impedance spectroscopy and potentiodynamic polarization to understand the Cr effect on kinetics and mechanism of corrosion. Scanning electron microscopy (SEM) enabled the understanding of the Cr effect on microstructural degradation of the magnet. Findings from this work will further advance the industrial application and commercialization of NdFeB magnets.

2. Materials and Methods

Commercial sintered NdFeB magnet coupons with dimensions $10 \times 10 \times 1$ mm^3 were prepared by abrading with 800 grit size SiC paper and then cleaned in acetone. Thin films of Cr (averaging 0.5 μm in thickness) were deposited on the primary surfaces of the NdFeB magnet samples with the aid of a double glow sputtering machine. The target cathode used in the chamber for sputtering was a Cr plate (99%) while the magnet samples were positioned as sample cathode. The anode was the grounded chamber wall. The sputtering process involved glow discharges encircling both cathodes such that one glow bombarded the Cr source cathode and the magnet samples were heated by the other. The sputtered Cr was deposited onto all the surfaces of the magnets by subsequently repeating the process. Table 1 shows details of the sputtering parameters. The corrosion characteristics of the Cr-coated magnet was analyzed, relative to the bare magnet, using a PARSTAT 273A Potentiostat/Galvanostat (Princeton Applied Research) using electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP) techniques at the end of 1 h immersion of the samples in naturally–aerated and unstirred 3.5% NaCl solution at room temperature. The samples were cold mounted and a surface area of 100 mm^2 was exposed for the testing. Pure copper wires were employed to make electrical contacts. The electrochemical cell was a three–electrode system comprising the samples (as working electrodes), a saturated calomel electrode connected through a Luggin capillary (as reference electrode) and a platinum sheet (as counter electrode). During the potentiodynamic polarization, the potential of the samples was scanned between ±250 mV versus the open circuit potential at a scan rate of 0.166 mV/s. EIS was measured with a signal recovery model 5210 lock–in amplifier attached to the potentiostat, over a frequency range of 10^5 Hz to 10^{-1} Hz by applying a perturbation amplitude of 10 mV. The electrochemical analyses were complemented with scanning electron microscopy (SEM) to view the surface features of the samples after polarization, with the aid of FEI-Inspect/OXFORDINSTRUMENTS–X–Max spectrophotometer.

Table 1. Sputtering parameters for the deposition of Cr on NdFeB magnet

Source cathode	Voltage (-900 V) Duty cycle (50 %)
Target Cathode	Voltage (–200 V) Duty cycle (10 %)
Parallel distance between the source cathode and the sample	15 mm
Base pressure	$1 \text{ X } 10^{-1}$ Pa
Working pressure	60 Pa (Ar gas)
Deposition time	2 h
Temperature of the sample	400 °C

3. Results

NdFeB magnet corrodes in the 3.5% NaCl solution via an electrochemical process. The results from the potentiodynamic polarization analysis can elucidate how the thin Cr film modifies the anodic and cathodic half–processes which occur during the exposure of the NdFeB magnet in the 3.5% NaCl solution. The potentiodynamic polarization curves obtained for the NdFeB magnet without and with Cr film are provided in Figure 1. The values of polarization parameters such as corrosion potential (E_{corr}) and corrosion current density (j_{corr}) were obtained within the range of ±10 mV from the measured cathodic–anodic transition potential, and are provided in Table 2. From the results, the E_{corr} value for the magnet shifted more positively from –746 mV (without Cr coating) to –734 mV (with Cr coating) while the j_{corr} value was lowered from 8.284×10^{-5} Acm^{-2} to 2.877×10^{-5} Acm^{-2} due to the Cr coating. This amounted to a protection efficiency (%IE) of 60.00 %, based on Equation (1). The shift in j_{corr} is more impactful on the anodic arm of the polarization plot, which indicates that the Cr coating diminishes the rate of Nd and Fe oxidation during the corrosion.

$$IE_{PDP} = 1 - \frac{j_{corr(\text{with Cr})}}{j_{corr(\text{Without Cr})}} \times 100\%. \qquad (1)$$

Table 2. Polarization parameters for bare and Cr-coated NdFeB magnet in 3.5% NaCl solution

Substrate	E_{corr} (mV)	j_{corr} (A/cm^2)	β_a (mV/dec)	β_c (mV/dec)
Magnet	-746	$8.284 \text{ X } 10^{-5}$	25	-188
Magnet + Cr	-734	$3.313 \text{ X } 10^{-5}$	73	-196

The results from EIS analysis can describe how the Cr film influences the interfacial and dielectric properties at the magnet–3.5% NaCl solution interface. The results obtained from the EIS analysis are presented as Nyquist and Phase angle plots in Figure 2. From the Nyquist plots in Figure 2(a), the magnet without and with Cr film, both, displayed a capacitive loop during the high frequency perturbation and an incomplete capacitive loop at low frequency. With Cr film, the magnet displayed a greater capacitive loop size both at high and low frequencies, which is indicative of greater corrosion resistance. The phase angle plots in Figure 2(b) reveal large maxima spanning a wide frequency range, and smaller phase angle peaks at low frequency for both the uncoated and Cr–coated magnet. These relate to

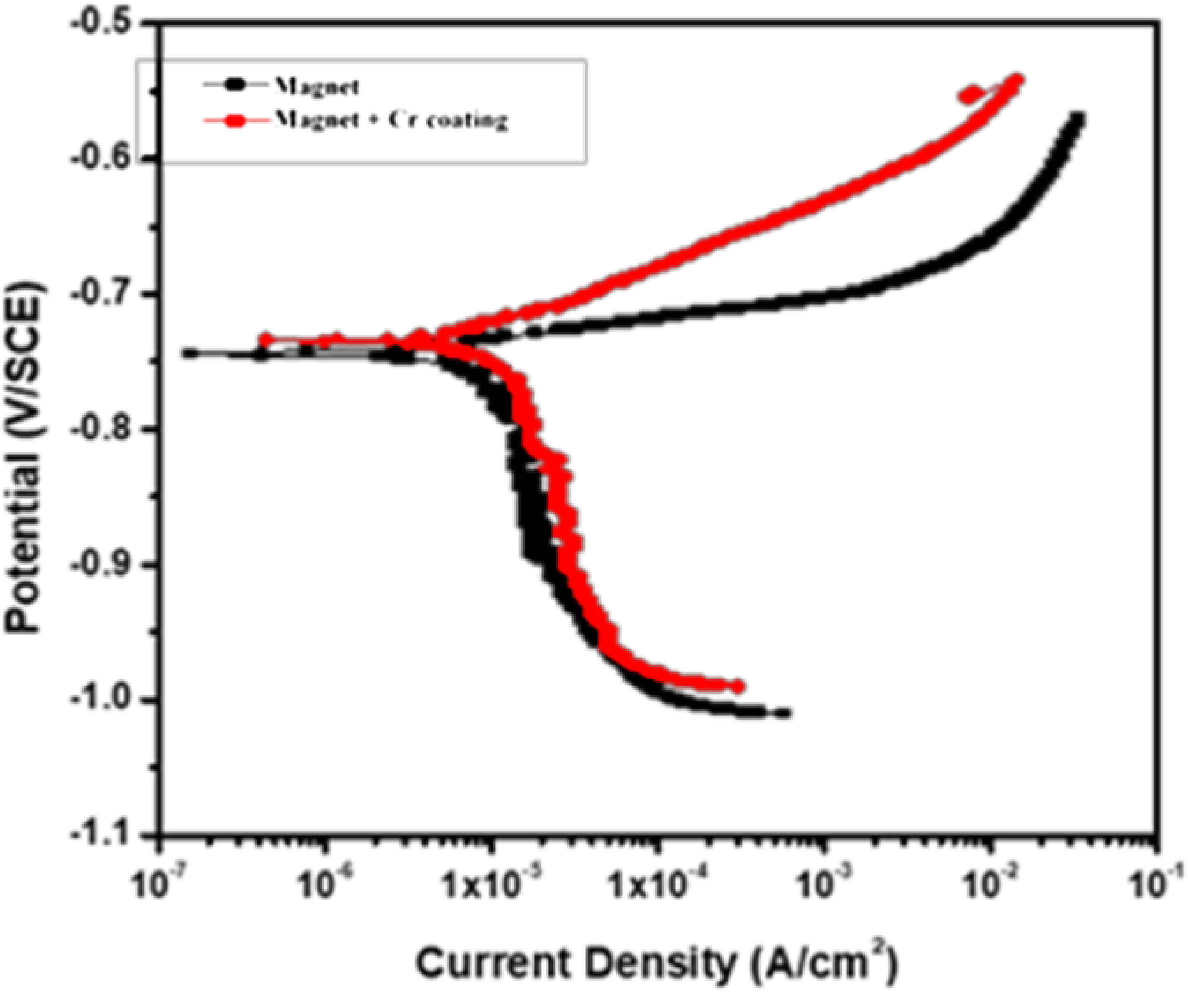

Figure 1. Potentiodynamic polarization curves for bare and Cr-coated NdFeB magnet in 3.5% NaCl solution.

two time constants controlling the corrosion of the magnet in the solution. Such time constants are defined by the dielectric phenomena at a corrosion product layer–solution interface (represented by the high frequency maxima) and the processes taking place in the interface beneath the layer i.e. in the substrate–solution interface (represented by the low frequency maxima). In order to correlate the impedance behavior of the samples with electrical elements, the equivalent model in Figure 2(c) was adapted after fitting with Zsimpwin software. Similar models have been employed to define corrosion mechanisms with two time constants [27-31]. The values of the electrical parameters are provided in Table 3. In the model, constant phase elements (CPE) were employed in place of capacitors in order to account for the inhomogeneity of the sample surfaces and their corrosion product layers. The capacitance of the CPE is deduced from $Z_{CPE} = Q^{-1}(j\omega)^{-n}$; so that Q and n stand for the CPE constant and exponent, respectively, whereas the imaginary number is $j = (-1)^{1/2}$, the angular frequency is $\omega = 2\pi f$, and the frequency (f) is in Hz. The derived impedance parameters are detailed in Table 3, where R_s is the solution resistance, R_f and CPE_f, respectively, describe the corrosion product layer porosity and its dielectric properties at the corrosion product layer–NaCl solution interface. On the other hand, R_{ct} and CPE_{dl} describe the Faradaic properties of the double layer formed beneath the corrosion products.

After being polarized in 3.5% NaCl solution, surfaces of the bare and Cr-coated NdFeB magnet were visualized in order to analyze how the Cr coating affects the magnet microstructures during corrosion. Low magnification surface

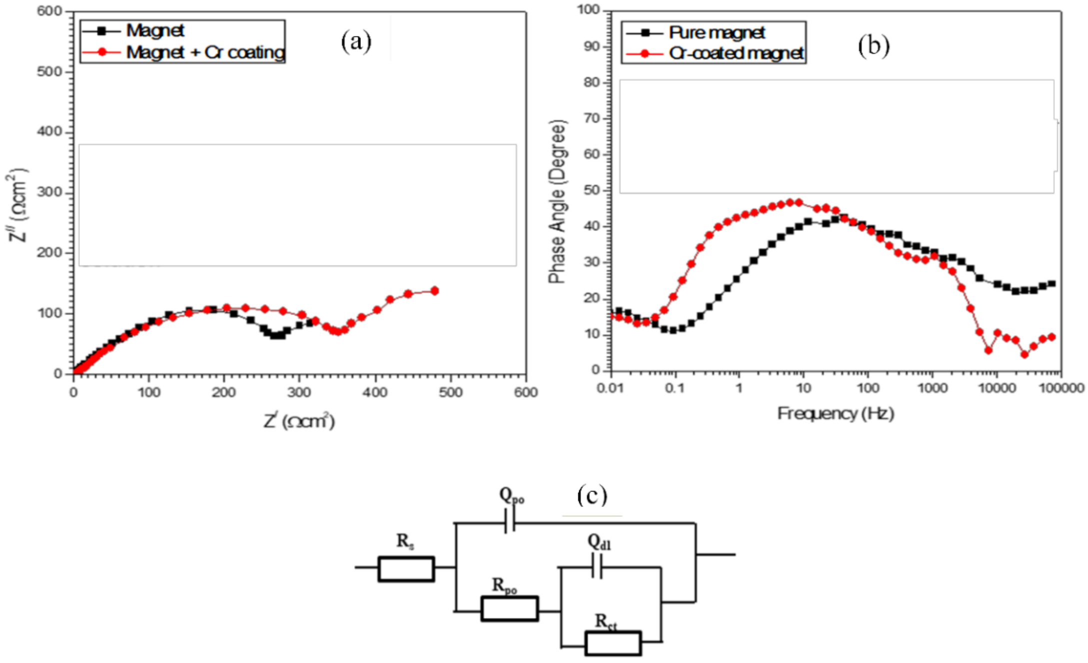

Figure 2. Impedance curves for uncoated and Cr–coated NdFeB magnet in 3.5% NaCl solution (a) Nyquist format (b) Phase angle format (c) Equivalent circuit model.

Table 3. EIS parameters for uncoated and Cr–coated NdFeB magnet in 3.5% NaCl solution

Substrate		CPE$_{dl}$				CPE$_f$			
	R_s	Y_{dl}	n_1	R_{ct}	C_{dl}	Y_f	n_2	R_f	$(R_p = R_f + R_{ct})$
	(Ωcm^2)	$(\mu F cm^{-2} s^{n-1})$		(Ωcm^2)	$(\mu F cm^{-2})$	$(\mu F cm^{-2} s^{n-1})$		(Ωcm^2)	(Ωcm^2)
Magnet	1.062	205	0.9	265	80.30	51	0.7	43	308
Magnet + Cr	3.125	70	0.9	490	27.43	12	0.8	244	734

image of the uncoated magnet, Figure 3(a), reveals several pieces of grains of the magnet; making up a seriously rough surface. A larger magnification image of the polarized bare magnet clearly shows that the grains of the magnet were seriously shattered, especially along the grain boundaries as Figure 3(b) shows. Conversely, Figure 3(c) shows that the surface of the magnet with Cr film was less destroyed during the polarization. Indeed, the coated magnet exhibited a smoother surface while several smaller pits with sparsely distributed larger pits occurred on the surface. Such pitting distribution could be attributed to the areas on the magnet surface depleted of the protective Cr–oxide. Larger magnification of one of the pits, Figure 3(d), reveals that the pitting attack on the grains of the magnet substrate was also greatly reduced for the Cr–coated magnet.

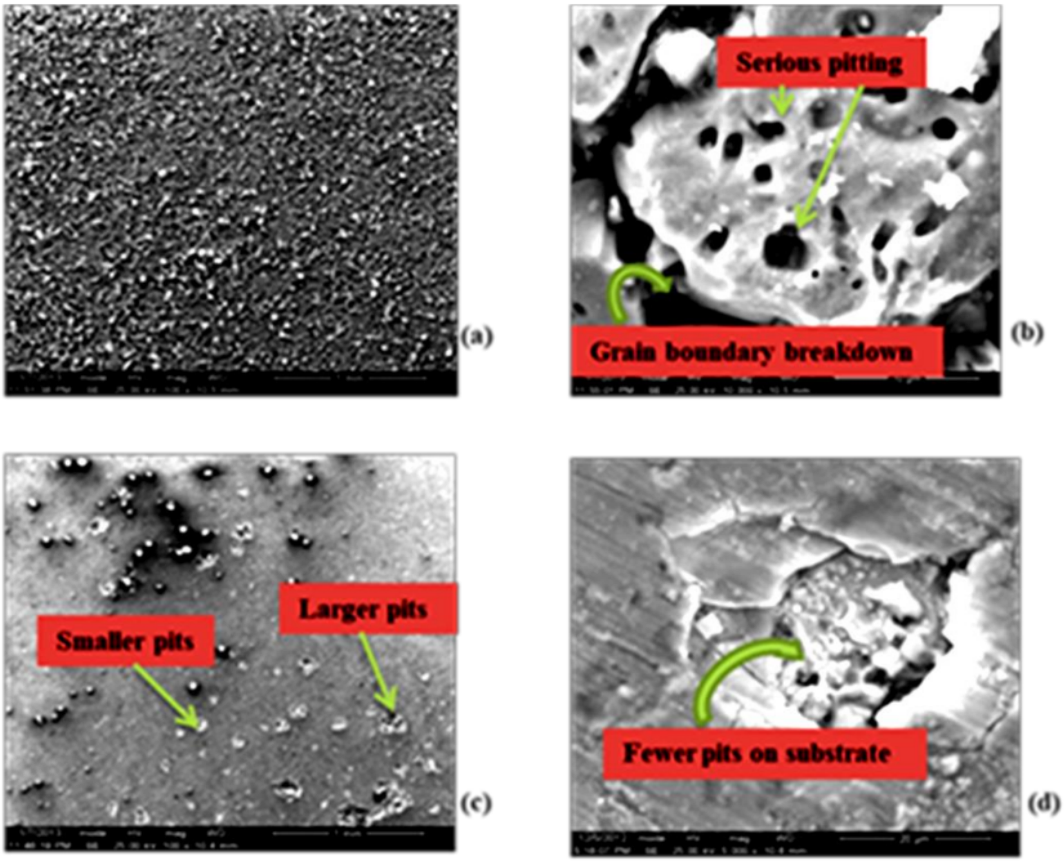

Figure 3. SEM images for bare Nd–Fe–B magnet (a & b) and Cr–coated NdFeB magnet (c & d) after polarization in 3.5% NaCl solution.

4. Discussion

The E_{corr} value for the magnet is highly related to its susceptibility to corrosion in the solution, whereas the j_{corr} value discloses its corrosion rate. More positive E_{corr} value exhibited by the magnet with Cr film magnet signifies that the Cr film lowers the propensity of the magnet to become chemically unstable, and the lower j_{corr} value attests to the protection ability of the Cr film. It is reported that out of the three phases making up the NdFeB magnet structure, the Nd-rich phase is the most electrochemically-active because of the high rare earth metal (Nd) content [32,33]. Thus, the corrosion of NdFeB magnet under wet conditions is triggered in the Nd–rich phase. Since oxygen reduction into hydroxide ions (Equation (2)) and Nd oxidation into Nd^{3+} (Equation (3)) should constitute, respectively, the major cathodic and anodic half–reactions during the corrosion of the magnet in such neutral solution, the overall electrochemistry can enrich the magnet corrosion product layer with a $Nd(OH)_3/Nd_2O_3$ corrosion product layer system; the Nd_2O_3 being formed via dehydration of the $Nd(OH)_3$ (Equation (4)).

$$H_2O + O_2 + 4e^- \rightarrow 4OH^-, \tag{2}$$

$$Nd \rightarrow Nd^{3+} + 3e^-, \tag{3}$$

$$2Nd^{3+} + 6OH^- \rightarrow 2Nd(OH)_3 \rightarrow Nd_2O_3 + 3H_2O. \tag{4}$$

With the Cr film preferentially deposited within defects such as the boundaries of the magnet grains, an abundance of adsorption sites for the water molecule occurs which increases the concentration of hydroxide ions on the magnet surface. This must be the reason for the slightly higher cathodic current displayed by the NdFeB magnet with Cr film during polarization. Nevertheless, it must be reasoned that the well-reduced anodic current density observed for the magnet with Cr film should be attributed to the enrichment of the $Nd(OH)_3/Nd_2O_3$ layer with a highly resistant product, namely Cr_2O_3. This is definitely obtainable, given that the electrochemical reaction during the corrosion is usually triggered at defect sites like grain boundaries wherein the Cr particles are concentrated. The formation of the Cr_2O_3 could occur via the dehydration of $Cr(OH)_3$, Equation (5), which is highly compact and can promote the diffusion of oxygen (rather than water molecules) to react with Cr^{3+} ions formed in the interior, Equation (6).

$$2Cr^{3+} + 6OH^{-} \rightarrow 2Cr(OH)_3 \rightarrow Cr_2O_3 + 3H_2O, \tag{5}$$

$$2Cr^{3+} + 3[O] \rightarrow Cr_2O_3. \tag{6}$$

From the shapes of the Nyquist plots, it can be inferred that the Cr film of about 0.5 µm does not actually change the magnet corrosion mechanism. This suggests that the Cr coating will also have minimal influence on the magnetic properties. Rather, the Cr coating greatly improves the corrosion performance of the magnet in the 3.5% NaCl solution. The R_{ct} depicts the corrosion resistance of each sample in the solution. The data in Table 3 confirms that the Cr coating promotes the formation of a less porous and highly resistant corrosion product layer. This must be because this corrosion layer has been enriched with Cr_2O_3. It is well known that the boundaries of the different phases in the NdFeB magnet are majorly concentrated within the highly reactive rare earth metal, Nd, which pins down the domain walls of the magnet. It appears that during the consumption of the Nd for the formation of the Nd corrosion products, the grain boundaries of the magnet break down and the magnet becomes less coercive. Similar observation was reported by [33,34] that the Nd-rich phase dissolution occurs at more negative potentials than the corrosion potential of NdFeB magnet leading to pulverization of the magnet. Furthermore, the hydrogen gas, produced during the reaction of Nd with water, can diffuse throughout the bulk of the magnet and lead to inter–granular hydrogen embrittlement which facilitates the corrosion of the magnet in the 3.5% NaCl solution. In addition, the Figure 3(b) reveals several large pits forming on the magnet grains. Such pitting phenomenon is characteristic of metal corrosion in solutions containing chloride ions. It, therefore, concludes that the magnet corrosion product layer is highly un–protective. This affirms that the thin Cr coating on the magnet could form a more resistant product layer which blocks off aggressive ions and increases the corrosion resistance of the NdFeB magnet in the 3.5% NaCl solution, as observed from the electrochemical analyses.

5. Conclusions

The impact of Cr coating on the electrochemical corrosion performance of NdFeB magnet has been analyzed in 3.5% NaCl solution, compared with a conventional sintered NdFeB magnet. The Cr coating has insignificant effect on the corrosion mechanism of the magnet, but causes an anodic shift of the corrosion potential. The coating also lowers the corrosion rate of the magnet in the wet environment and significantly mitigates localized grain boundary deterioration and pitting corrosion on the magnet. This was attributed to the enrichment of the magnet corrosion product layer with the protective Cr–oxide. Giving the important findings from this work, some limitations can still be identified. Although this work provides electrochemical elucidation of the Cr coating effect, there is still a lack of adequate scientific information regarding the true mechanism and chemistry of the corrosion product formation by the Cr-coated magnet (both instantaneously and over time). It is also not clear, at the moment, the effects of Cr coating and corrosion products on the magnetic properties of the magnet, as well as the threshold thickness of Cr coating for optimum corrosion protection of the magnet. These limitations can serve as the focuse for future research works on improving the corrosion and magnetic properties of NdFeB magnet.

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