# Electrochemical Porosity Measurement of Electroless Nickel Coatings on Ferrous Substrates

Information resulting from AESF Research Project By L. Das, D-T. Chin, G.L. Evarts & R.L. Zeller III

A non-destructive electrochemical method is used to measure the porosity of electroless nickel (EN) coatings on steel and gray cast iron. The method is based on determining the mixed potential of a duplex metal surface consisting of an EN coating and the ferrous substrate exposed by the pores in a corrosive electrolyte; the measured porosity is quantitatively expressed in terms of the fraction of pore area on the coating surface. The accuracy of this method is compared to the conventional ferroxyl and salt fog spray tests. The electrochemical technique is found to be more sensitive than the ferroxyl test, and provides the porosity information comparable to that of the salt fog test in a much shorter time without destroying the appearance of test samples. The electrochemical porosity measurement is subsequently used to examine the effect of substrate preparation, including surface polishing and an electrolytic nickel strike, on the porosity of EN coating on carbon steel and gray cast iron substrates. It is found that the porosity of EN coatings decrease with decreasing roughness of the substrate surface. Mechanically polishing the substrates to 1- $\mu$ m smoothness prior to EN plating eliminates nodular EN deposits and reduces the porosity of the EN coating. Electropolishing of the substrate surface also results in EN deposits with low porosity. An electrolytic nickel strike on carbon steel and gray cast iron prior to EN plating decreases the porosity of the EN coating by covering exposed graphite and other noncatalytic inclusions on the substrate surface with a catalytic nickel layer. The porosity of an EN coating decreases with increasing thickness of the nickel strike. A nickel strike of 1 µm in thickness virtually eliminates nodular EN deposits and reduces the coating porosity by a factor of 100 to an area fraction of pores less than 10<sup>-5</sup>.

Electroless nickel (EN) coatings are used in engineering applications to provide protection to ferrous alloys exposed to corrosive environments.<sup>1</sup> Electroless nickel coating on a ferrous substrate is an example of a more noble coating on a less noble substrate. If the less noble substrate is exposed to a corrosive environment through pores and discontinuities in the coating, corrosion of the substrate leading to failure of the coating would take place. For EN coatings to function satisfactorily as a corrosion barrier, it is necessary to be free of pores.

The porosity of EN coatings is usually measured using the ferroxyl test or the salt fog test.<sup>2</sup> Although these methods are useful for detecting pores in the deposit, their test results are not always reproducible. The salt fog test is destructive to the coating and requires 24 to 48 hr to complete. The purpose of this study is to develop a fast and non-destructive method to measure the porosity of EN coatings on ferrous substrates. The method is based on an electrochemical principle of measuring the mixed potential of a duplex metal surface consisting of the EN coating and the ferrous substrate ex-

posed by the pores to a corrosive electrolyte. The method needs only a few minutes to complete a test, and the measured porosity may be quantitatively expressed in terms of the fraction of pore area on the coating surface.

In the current study, the electrochemical porosity measurement is further used to examine the effect of substrate preparation, including the surface polishing and electrolytic nickel strike, on the porosity of EN coatings on ferrous alloys. The morphology of the substrate prior to plating is a key factor controlling the porosity of EN coatings. The studies by Coussement *et al.*<sup>3</sup> and Deng and Møller<sup>4</sup> indicated that surface roughness of steel substrate had important consequences on the EN deposit. They found that the corrosion resistance of EN deposits decreased with increase in surface roughness of the substrate prior to plating. A study by Kudrak *et al.*<sup>5</sup> also indicated that the porosity of palladium and gold coatings increased with increasing roughness of the substrate surface.

The corrosion resistance of EN is also affected by the formation of nodular deposits. A study by Jones<sup>6</sup> has shown that the corrosion resistance of nodular hard chromium deposits may be improved by decreasing nodule size or eliminating the nodules. Substrate defects, such as inclusions and debris on the surface, are the main cause of the formation of nodules and pores in EN deposits. A previous publication by the authors<sup>7</sup> revealed that non-catalytic graphite present in the microstructure of ferrous alloys was responsible for the formation of pores in EN deposits. Electroless nickel deposits on nickel substrates, on the other hand, are nodule-free. Nickel is catalytic to EN deposition and the EN coating initiates and grows uniformly over the entire substrate surface. Accordingly, a thin nickel strike to cover graphite and other passive inclusions in ferrous substrates prior to EN plating may potentially reduce the probability of pore formation on EN coatings. The current study was undertaken to examine the effect of substrate surface polishing and electrolytic nickel strike on the porosity of EN coatings using the electrochemical porosity measurement.

#### Experimental Procedure

Substrates and Surface Preparation Prior to EN Plating

Three different ferrous substrates were used: (1) AISI 1010 carbon steel containing 0.1 percent carbon; (2) AISI 1075 carbon steel containing 0.75 percent carbon; and (3) a gray cast iron containing 3 percent carbon and 2.7 percent silicon.

To examine the effect of substrate polishing on the porosity of EN deposits, five different types of surface finishes were prepared for the AISI 1010 carbon steel and gray cast iron coupons. They were: (1) as received from a commercial metal sample supplier; (2) polished with 120-grit silicon carbide paper; (3) polished with 600-grit silicon carbide paper; (4) polished with 1- $\mu$ m alumina paste; and (5) electropolished. Electropolishing of the carbon steel was carried out in an electrolyte composed of 75 percent by volume of phosphoric acid (85 wt percent in concentration) and 25 percent by volume of lactic acid (85 wt percent in concentration) at 25 A/dm<sup>2</sup> and 60 °C for 30 min.<sup>8</sup> Electropolishing of the gray cast iron was performed in a solution containing 94 percent by volume of glacial acetic acid and 6 percent by volume of perchloric acid (60 wt percent in concentration) at 0 °C and a cell voltage of 40 V for a period of 5 min.<sup>8</sup>

A nickel strike on some carbon steel and cast iron coupons prior to EN plating was carried out with a Wood's bath composed of 240 g/L of nickel chloride and 384 g/L of hydrochloric acid. The substrates were mechanically polished to a 600-grit surface finish prior to the strike. Platinum was used as the anode and a cathodic current density of 6 A/ dm<sup>2</sup> was maintained during the strike. The strike was carried out for 2, 5 and 10 min to yield nickel coatings of different thickness.

### EN Coatings for Porosity Measurement

Two types of EN coatings were used. The first EN coating (89.5% Ni, 10.5% P) was prepared using a commercial bath. To prepare the samples for porosity measurements, AISI 1010 carbon steel coupons were soaked in a commercial cleaning solution, electrocleaned with periodic reverse current, activated by acid dip in 37-percent HCl, and immersed in the commercial EN bath. The substrates were then plated for various lengths of time to yield EN coating thicknesses of 0.4 to 64  $\mu$ m. These samples were tested for porosity using the electrochemical technique as well as with conventional ferroxyl and salt fog tests.

The second type of EN coating (90.3% Ni, 9.7% P) was prepared by dipping the ferrous substrates in a generic EN bath containing 44 g/L nickel sulfate hexahydrate, 10 g/L sodium hypophosphite hydrate, 42 g/L aminoacetic acid and 10 g/L acetic acid at 88 °C and pH 4.5. The ferrous coupons were polished to various degrees of roughness. They were anodically electrocleaned in 1 M NaOH at 5 A/dm<sup>2</sup> for 2-3 min and were activated by dipping in 37-percent HCl for 1 min. The EN plating was carried out in the generic bath for different time intervals to produce coating thicknesses ranging from 2 to 35  $\mu$ m. The morphology of the EN-coated samples was observed under a scanning electron microscope (SEM), and the coating porosity was measured using the electrochemical technique.

# Electrochemical Porosity Measurement

The principle of the electrochemical porosity measurements is based on the fact that a duplex metal surface (part coating and part substrate exposed through pores in the coating) exhibits a corrosion potential on exposure to a corroding electrolyte. The value of the corrosion potential depends on the area fraction of pores and the polarization of the anode (base metal) and cathode (coating). An equation by Mansfeld<sup>9</sup> for the corrosion potential  $E_{corr}$  of a galvanic couple where the anodic area is much smaller than the cathodic area may be given as:

$$E_{corr} = k - [b_a b_c / (b_a + b_c)] \log (\theta_A)$$

where  $\theta_A$  is the area fraction of the anode (or porosity);  $b_a$  is the Tafel slope for the anodic reaction;  $b_c$  is the Tafel slope for the cathodic reaction; and k is a constant. This equation indicates that a plot of the corrosion potential vs.  $\log \theta_A$  would yield a straight line. Electroless nickel on ferrous substrate is an example of a noble coating on an active metal with low porosity. In a corrosive electrolyte, the anodic area is the ferrous substrate exposed through the pores and the cathodic area is the EN coating. Accordingly, the above principle for porosity measurement is applicable. If a calibration plot in the form of area fraction of exposed iron vs. corrosion potential is available, the porosity of an EN coating on a ferrous substrate may be determined from the calibration plot by measuring the corrosion potential of the EN-coated sample.

In this study, a galvanically connected EN-iron couple was used to calibrate the change in corrosion potential as a function of the area fraction of iron. Because the galvanic corrosion potential depends on the type and composition of the EN deposit, two different EN sheets were used for the calibration. The first was prepared by depositing 67.5 µm of high-phosphorus EN (89.5 wt % Ni, 10.5 wt % P) on a large nickel sheet, 64 cm<sup>2</sup> in surface area, from the commercial EN bath, while the second was prepared by depositing 30 µm of EN (90.3 wt % Ni, 9.7 wt % P) on another nickel sheet of the same surface area from the generic EN bath. An iron wire (0.25 mm in dia.), insulated with wax except at the tip, was galvanically connected to the EN-plated nickel sheet. The area ratio of the iron wire to EN coating was varied by changing the length of the exposed iron tip. The corrosion potential of this EN-iron couple was measured in a solution containing 3 wt percent NaCl and 1.5 wt percent H<sub>2</sub>O<sub>2</sub> at 23 °C, as described by Shome and Evans.10 A saturated calomel electrode (SCE) was used as the reference, and the corrosion potential of the EN-iron couple was measured with a high input impedance electrometer.<sup>a</sup> The results of the calibration are shown in Fig. 1. It was noted that the calibration lines for the commercial and generic EN have a different slope and intercept. Because the phosphorus content in the two coatings was nearly the same, this suggests that additives used in the commercial bath significantly modified the electrochemical properties of the EN deposit.

To measure the porosity of a ferrous substrate coated with EN, the sample was immersed in the same electrolyte at 23 °C and its corrosion potential measured with reference to the SCE. The area fraction of pores corresponding to the measured corrosion potential was then determined from the calibration plot.

#### Ferroxyl Test

The porosity measurement using the ferroxyl test was carried out in accordance with ASTM B733 (1994). The test solution



Fig. 1—Calibration plot for area fraction of pores on EN coating vs. corrosion potential of EN-coated ferrous substrate.

## Table 1 Comparison of Electrochemical Porosity Measurement With Results of Ferroxyl & Salt Fog Tests

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EN Deposit Thickness :m	Ferroxyl Test (no. of pores on 8 cm <sup>2</sup> sample area)	Salt Fog Test (no. of pores on 8 cm <sup>2</sup> sample area)	Electrochemical Test (area fraction of pores)	Estimated Pore Size* (µm)
0.42	-	-	2.0 x 10 <sup>-3</sup>	-
1.05	-	-	1.9 x 10 <sup>-3</sup>	-
2.1	11	156	1.4 x 10 <sup>-3</sup>	96
6.4	0	61	1.3 x 10 <sup>-3</sup>	147
26	0	3	0.93 x 10 <sup>-3</sup>	560
38	0	5	0.97 x 10 <sup>-3</sup>	440
51	0	2	0.95 x 10 <sup>-3</sup>	700
64	0	1	0.94 x 10 <sup>-3</sup>	980

\* Estimated from the results in Columns 3 and 4, using the formula:

Average pore diameter=  $\sqrt{\frac{4}{\pi}} \frac{(area fraction of pores) (sample area)}{number of pores}$ 

was prepared by dissolving 25 g potassium ferricyanide and 15 g sodium chloride in 1 L of distilled water. The test samples were cleaned and immersed in the test solution for 30 sec at 25 °C. The samples were then rinsed in water, dried in air, and examined for blue ferrous ferricyanide formed at the pore sites.

# Salt Fog Test

The salt fog test was carried out in accordance with ASTM B117 (1994). The EN-coated specimens were cleaned and exposed in a salt spray chamber for 24 hr. The salt solution was 5-percent sodium chloride in distilled water. The temperature in the salt spray chamber was maintained at 35 °C. After exposure, the test samples were rinsed in water to remove salt deposits, dried and examined for rust spots on the surface.

Results and Discussion

Comparison of Ferroxyl, Salt Fog, &

Electrochemical Porosity Measurements

Table 1 summarizes the results of the ferroxyl, salt fog, and electrochemical porosity measurements for various thicknesses of the commercial EN coating. With ferroxyl tests, blue spots appeared at the location of pores. The ferroxyl test showed positive identification of pores only on the substrate

with 2.1 µm of EN deposited; it was unable to detect pores in thicker coatings. The salt fog tests, on the other hand, were successful in detecting pores on all the samples, shown by the presence of rust spots on the sample surface. The number of pores decreased with increasing in coating thickness, as shown in Table 1. The electrochemical tests were capable of measuring the area fraction of pores on EN coatings.

The porosity of the commercial EN coating was plotted against deposit thickness in Fig. 2. The results obtained with salt fog tests agreed with the results of electrochemical porosity measurements. The porosity of EN on steel decreased with increase in deposit thickness in the range of 0-30 µm, beyond which the porosity remained approximately constant. The electrochemical technique gives results comparable to those of the salt fog tests and requires a much shorter time without destroying the sample.

The salt fog test and electrochemical porosity measurement data can be combined to estimate the average pore size present on the EN coating. The estimated results are listed in the last column of Table 1. It is noted that the average pore size

increases in a linear fashion with an increase in coating thickness. This suggests that during the EN deposition process, small pores were being filled with EN, with only large pores expected after a thick EN coating was deposited.

The porosity of EN coatings on AISI 1010 and 1075 carbon steel, prepared from the generic EN bath, is shown in Fig. 3. The porosity of EN coatings on both substrates decreased with increasing deposit thickness. The porosity was slightly lower on AISI 1010 steel than on AISI 1075 steel when coating thickness was less than 10 µm. This may be a result of the fact that AISI 1075 steel contains more carbon in its microstructure than AISI 1010 steel.

# Substrate Surface Roughness & Porosity of EN Coating

The effect of substrate roughness on EN porosity was tested with AISI 1010 carbon steel and gray cast iron coupons. They were polished to various roughnesses and coated with EN in the generic bath to a common thickness of 12  $\pm$ 0.7  $\mu$ m. Coating porosity was then measured with the electrochemical technique. Table 2 lists the porosity of generic EN coatings on the carbon steel and gray cast iron with different surface roughness. For AISI 1010 carbon steel, the porosity of EN coatings on the as-received substrate surface and on the



Fig. 2— Porosity of commercial EN coating on AISI 1010 carbon steel as function of coating thickness: (a) results of electrochemical measurement; (b) results of salt fog test.

120-grit finish were higher than that on the 600-grit surface finish. The porosity of EN coating on the substrates polished with 1- $\mu$ m alumina paste and electropolishing was the lowest. For the gray cast iron substrates, the porosity was reduced by half when the surface roughness decreased from 120-grit to a 600-grit finish prior to EN plating. Mechanical polishing to 1  $\mu$ m in smoothness and electropolishing further reduced the porosity.

Figure 4 is a SEM photomicrograph showing the morphology of EN deposit on the as-received surface of an AISI 1010 carbon steel coupon. Figure 5 is a SEM photomicrograph of EN deposit on a carbon steel coupon polished to 1  $\mu$ m smoothness prior to EN plating. The inset in each photomicrograph shows the substrate surface prior to EN deposition. The EN on the as-received steel coupon was nodular in structure. The size of nodules was found to decrease with decreasing surface roughness and, when the substrate surface was polished to 1  $\mu$ m in smoothness, the EN deposit became free of nodules, as shown in Fig. 5. Small nodules were observed on the EN deposit on the electropolished steel surface. The electropolished steel surface was passive to EN deposition and had to be activated by etching in 37-percent HCl for a longer period of time. The etching in HCl may have destroyed the surface smoothness and caused the formation of small nodules and a slight increase in the porosity of the EN



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Table 2 Effect of Substrate Roughness on Porosity of Generic EN Deposit						
Type of Substrate Porosity (Area Fraction of Pores) Surface Finish						
	AISI 1010 Steel	Gray Cast Iron				
Blast with glass beads (as received)	3.5 x 10 <sup>-3</sup>	-				
120-grit	3.2 x 10 <sup>-3</sup>	4.0 x 10 <sup>-3</sup>				
400-grit	-	2.3 x 10 <sup>-3</sup>				
600-grit	1.2 x 10 <sup>-3</sup>	1.4 x 10 <sup>-3</sup>				
1-μm alumina paste	0.67 x 10 <sup>-3</sup>	1.3 x 10 <sup>-3</sup>				
Electrochemical polish	0.75 x 10 <sup>-3</sup>	0.9 x 10 <sup>-3</sup>				



Fig. 3—Porosity of generic EN coating on AISI 1010 and 1075 carbon steel as function of coating thickness by electrochemical technique.

coating on electropolished AISI 1010 carbon steel, as shown in Table 2.

The SEM examination of EN coatings on gray cast iron coupons with different surface finishes (120-grit, 400-grit, 600-grit, 1 $\mu$ m, and electropolish) revealed the presence of nodular deposits on all the samples. The nodules were observed along the grinding marks on the coupons with 120-grit, 400-grit and 600-grit finish. When the gray cast iron was polished to 1  $\mu$ m in smoothness, the graphite flakes present in the microstructure showed up on the surface as shown in Fig. 6a. Figure 6b shows that the nodular EN deposit was formed on the graphite inclusion, whereas the EN deposit on the surrounding iron portion of the substrate was free of nodules.

Effect of Nickel Strike on the Porosity of EN Deposits

The results in the previous section show that surface rough-

Table 3 Effect of Electrolytic Nickel Strike Prior to Plating on Porosity of Generic EN Deposit On Carbon Steel & Gray Cast Iron

Substrate	Nickel Strike Time (min)	Nickel Strike Thickness (µm)	<b>Porosity</b> (Area Fraction of Pores)
ISI 1010 Carbon Steel	0	0	1.2x10 <sup>-3</sup>
	2	0.25	0.47x10 <sup>-3</sup>
	5	0.46	< 10 <sup>-5</sup>
Gray Cast Iror	n 0	0	1.4x10 <sup>-3</sup>
	2	0.22	1.7x10 <sup>-3</sup>
	5	0.45	0.9x10 <sup>-3</sup>
	10	0.97	< 10 <sup>-5</sup>



Fig. 4—SEM photomicrograph of generic EN deposit (12  $\mu$ m thick) on the as-received surface (without any polishing) of an AISI 1010 carbon steel coupon. The inset shows the surface prior to EN plating.

ness of the substrate affects the porosity of the EN deposit. The presence of graphite flakes on gray cast iron surfaces also results in nodular EN deposits with an increased porosity. One way to reduce the porosity of EN coating on ferrous substrates is to cover exposed graphite and other non-catalytic surface inclusions with a thin layer of electrolytic nickel. Experiments were carried out to determine the effectiveness of electrolytic nickel strikes prior to EN plating in reducing the porosity of EN deposit on AISI 1010 carbon steel and gray cast iron coupons having a 600-grit surface finish. Figures 7a and 8a show the surface of gray cast iron coupons after 2 min (0.2  $\mu$ m coating thickness) and 10 min (1  $\mu$ m coating thickness) nickel strike, respectively. Figures 7b and 8b are the SEM photomicrographs of the same coupons coated with 12 ±0.7  $\mu$ m of EN. Two min of nickel strike was not long enough



Fig. 5—SEM photomicrograph of generic EN deposit (12  $\mu$ m thick) on an AISI 1010 carbon steel coupon; surface mechanically polished to 1  $\mu$ m smoothness (inset) prior to EN plating.



Fig. 6—SEM photomicrograph of gray cast iron coupon polished to 1  $\mu$ m smoothness: (a) before EN plating; (b) after coating with 12  $\mu$ m generic EN.



Fig. 7—SEM photomicrograph of gray cast iron coupon pre-coated with 0.2  $\mu$ m of an electrolytic nickel strike: (a) before EN plating; (b) after coating with 12  $\mu$ m generic EN.

to cover all the graphite flakes present on the surface of gray cast iron. The EN deposit still exhibited a nodular structure, as shown in Fig. 7b. Two min of nickel strike eliminated nodular deposits on AISI 1010 carbon steel, however. When the nickel strike time was increased to 10 min, all the graphite flakes on gray cast iron were covered by the electrolytic nickel coating, and the subsequent EN coating became free of nodules, as seen in Fig. 8b.



Fig. 8—SEM photomicrograph of gray cast iron coupon pre-coated with 1  $\mu m$  of an electrolytic nickel strike: (a) before EN plating; (b) after coating with 12 µm generic EN.

The EN-coated carbon steel and gray cast iron coupons, pre-coated with an electrolytic nickel strike, were tested for porosity using the electrochemical porosity measurement. Table 3 lists the porosity of EN deposits on the substrates with different nickel-strike thickness. The porosity of EN on both gray cast iron and steel substrates decreased with an increase in the thickness of the nickel strike. Without a nickel strike, the area fractions of pores of EN was on the order of  $10^{-3}$ . When the thickness of the nickel strike was 1  $\mu$ m, the porosity of the EN coating on both AISI 1010 carbon steel and gray cast iron was reduced to less than 10<sup>-5</sup> (area fraction of pores).

#### Summary

- 1. The results of porosity measurements of EN coatings on steel substrate using the electrochemical, conventional ferroxyl and salt fog spray techniques indicate that the electrochemical technique is a quick and quantitative method for measuring EN porosity in the form of area fractions of pores on the coating surface. The electrochemical method is more sensitive than the ferroxyl test, and provides porosity information comparable to that obtained with the salt fog spray test in a much shorter time without destroying the appearance of test samples.
- The porosity of EN coatings on carbon steel and gray cast iron substrates decreases with decreasing roughness of the substrate surface. Mechanically polishing the substrates to 1 µm smoothness prior to EN plating eliminates nodular EN deposits and reduces the porosity of the EN coating. Electropolishing of the ferrous substrates also results in EN deposits with a low porosity.
- An electrolytic nickel strike on carbon steel and gray cast iron prior to EN plating decreases the porosity of EN coating by covering exposed graphite and other noncatalytic inclusions on the substrate surface with a catalytic nickel layer. The porosity of EN coatings decreases with increasing thickness of a nickel strike. One µm of nickel strike virtually eliminates nodular EN deposits and reduces the coating porosity to less than 10<sup>-5</sup> (area fraction of pores).
- <sup>a</sup> Model 610C, Keithley Instruments, Cleveland, OH

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