Influence of Electroless Nickel-Phosphorus Deposits on the Corrosion-Fatigue Life of Notched and Unnotched Samples of an AISI 1045 Steel

J.A. Chitty, A. Pertuz, H. Hintermann, and E.S. Puchi

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Electroless nickel-phosphorus deposits of approximately 10% phosphorus and about 20 μm thickness are shown either to have no effect or sometimes to increase the corrosion-fatigue properties of a quenched and tempered AISI 1045 steel in the stress amplitude range of 481 to 687 MPa, in the presence of an aqueous solution of 3% sodium chloride. Such an increase is produced when the stress amplitude is below 516 MPa. For the notched specimens, no substantial differences are found between the fatigue life of the coated and uncoated specimens.

Keywords corrosion, electroless nickel, fatigue, type 1045 steel

1. Introduction

In the last 40 years, many reports have been published about electroless nickel-phosphorus and nickel-boron deposits. However, few of these have dealt with the deposit-substrate fatigue and corrosion-fatigue properties. Additionally, the information is not entirely reliable, and sometimes contradictory results have been presented. Some studies (Ref 1, 2) have reported a decrease between 10 and 46% in the fatigue strength for high-strength steels as a result of the deposition. In notched specimens, the loss of fatigue strength can reach up to 15% (Ref 1).

Turns and Browning (Ref 3), working with low-strength steels, found a decrease between 10 and 15% in the fatigue strength when the tests were conducted in the as-deposited condition and a decrease between 20 and 25% in deposits heat treated at 110 and 500 °C. On the other hand, according to Safranek (Ref 4), high-strength steels with tensile strength of about 1400 MPa can be electroless nickel-phosphorus deposited without loss in fatigue strength. Also, a few reports suggest an increase in the fatigue strength after the application of the nickel-phosphorus electroless coatings (Ref 1). For example, Broszeit et al. (Ref 5) studied the fatigue-corrosion response of a 52 type steel coated with a nickel-boron alloy, which has similar properties to nickel-phosphorus alloys; the corrosive environment was a 3% sodium-chloride solution and the coated samples were heat treated at different temperatures. The results of this study showed that the fatigue-corrosion strength increased after the deposition process but that there was a decrease in this property after the application of post-heat treatments at 400 °C for 1 h and for 12 h. It is important to note that with the 12 h post-heat treatment, the fatigue life was found to be longer than the life of the samples with a post-heat treatment of 1 h.

The general belief concerning this topic is that due to their tendency to crack under cyclic loads, electroless nickel-phosphorus deposits can cause a significant reduction of the fatigue properties of steel substrates, although the magnitude of such reduction would depend upon composition, heat treatment, and thickness of the coating. For example, according to the early work conducted by a number of researchers (Ref 4-9), depending on the type of electroless nickel-phosphorus bath, phosphorus content, heat treatment temperature, and the presence of macroscopic notches, the reduction in the fatigue strength and endurance limit could range from 10 to 50%. Such deleterious effects have been particularly marked in the hypophosphite-reduced deposits with less than 10% P, as well as with nickel-boron alloys which are believed to be under high levels of tensile internal residual stresses (Ref 10).

More recently, Wu et al. (Ref 6) studied the influence of an electroless nickel-phosphorus deposit on the fatigue limit of a 30CrMo quenched (870 °C) and tempered (620 °C, 3 h) steel. The bath used nickel sulfate as a source of nickel ions and sodium hypophosphite as a reducing agent in an acid medium. The deposit was post-heat treated at 200 °C for 1.5 h in order to decrease the hydrogen content. These researchers found a decrease of about 39% in the fatigue limit of the steel and a reduction of about 20% when the substrate was subjected to a shot peening process before coating. Another investigation, conducted by Zhang et al. (Ref 7), was devoted to the influence of a nickel-phosphorus electroless coating with 9% P and 43 μm thickness on the fatigue life of the same steel previously mentioned. In this case, a shot peening process was also previously applied to some of the samples. The results of this investigation were very similar to those of the previous study, but in addition, some fractographic evidence of the presence of fatigue marking in the coating was shown. Therefore, it was concluded that the overall failure mechanism was the fatigue failure of the coating and the subsequent stress concentration on the substrate surface. Zhang et al. also measured the residual stresses

J.A. Chitty, Department of Applied Mathematics, Faculty of Engineering, Central University of Venezuela, Apartado Postal 47885, Los Chaguaramos, Caracas 1045, Venezuela; **A. Pertuz,** School of Mechanical Engineering, Faculty of Engineering, Central University of Venezuela, Apartado Postal 47885, Los Chaguaramos, Caracas 1045, Venezuela; **H. Hintermann,** Department of Mechanical Engineering, University of Neuchatel, Switzerland; and **E. S. Puchi,** School of Metallurgical Engineering and Materials Sciences, Faculty of Engineering, Central University of Venezuela, Apartado Postal 47885, Los Chaguaramos, Caracas 1045, Venezuela.

in the coating and determined that these were compressive stresses in all the conditions analyzed.

In previous studies, Puchi et al. (Ref 8) and Chitty et al. (Ref 9) have analyzed the fatigue and fatigue-corrosion behavior of two low carbon steels, namely, AISI 1010 and 1045 respectively, previously annealed. The corrosive agent used in the fatigue-corrosion tests was a sodium-chloride 3% solution. The bath employed for the deposition contained nickel chloride as a source of nickel ions and sodium hypophosphite as a reducing agent in an alkaline medium. A 20 μm thick coating was obtained after 3 h of deposition and the phosphorus content was approximately 10%. It was found that the electroless nickel-phosphorus coating increased the fatigue life of both the 1010 and 1045 steels. In the case of the 1045 steel, the fatigue experiments were conducted within a stress range of 220 to 331 MPa. As expected, it was observed that the improvement in the fatigue life of the samples was much higher when the stress amplitude decreased. In the case of the fatigue-corrosion tests also an increase in the fatigue life as high as 154% was observed for the samples tested at 220 MPa. This stress amplitude corresponded to about 60% of the yield stress of the material. At a stress amplitude of 331 MPa the improvement in the fatigue life was about 70%. Both research works also investigated the effect of the application of a post-heat treatment to the coating, which was conducted in an argon atmosphere at 543 K for 4 h. It was found that this treatment increased significantly the fatigue life of the material in comparison with the as-deposited condition but decreased the fatigue-corrosion properties.

Therefore, the present investigation has been conducted to study further the corrosion-fatigue behavior of a plain carbon steel (AISI 1045) with an initial mechanical strength that has been increased in relation to the annealed condition by quenching and subsequent tempering, and which has been coated with a commercial electroless nickel-phosphorus deposit. Also, the effect of such a coating on the corrosion-fatigue behavior of notched samples of the same material has been investigated and compared with the results obtained using unnotched samples.

2. Experimental Techniques

The present investigation has been conducted with notched and unnotched samples of AISI 1045 plain carbon steel with 0.43% C, 0.65% Mn, 0.30% Si, 0.013% S, and < 0.02% P. The material was initially provided as bars of approximately 25.4 mm diameter, which were subsequently cut to pieces of about 105 mm length. Cylindrical fatigue specimens were prepared with a total length of 100 mm and central diameter of 6.35 mm. All the samples were ground with emery papers (ranging from grit 220 to 600) applied consecutively. A set of samples was notched by means of a Rockwell hardness indentation produced with a conic diamond indentor. The load applied was 60 kg, which corresponds to a Rockwell A hardness scale. This step was conducted before the heat treatment of the material in order to avoid any plastic deformation in the areas near the notch. The depth of the notch was estimated by means of the Rockwell scale, which indicated a penetration of approximately 2 μm by each hardness unit. After the notching process, all the samples were oil quenched from a temperature of 1123 K and subsequently tempered at 773 K for 1 h. The final surface finish of the samples was obtained by a fine polish process with emery papers (grit 600 and 1200) applied consecutively after the heat treatment. Half of the specimens were coated with a commercial electroless nickel-phosphorus bath using the following composition per liter: 30 g nickel sulfate and 30 g sodium hypophosphite. The bath temperature was kept at approximately 85 °C and a pH of 5. The energy dispersive x-ray (EDX) evaluation of the coating showed a phosphorus content between 10 and 11%.

Fatigue-corrosion tests were conducted under fully reversed conditions on a rotating beam fatigue testing machine (Fatigue Dynamics Inc.) at a frequency of 50 Hz. The corrosive medium was a 3% sodium-chloride solution at room temperature. Four stress level amplitudes were chosen for the fatigue tests: 481, 516, 550, and 618 MPa which corresponded approximately to 60, 65, 70, and 80% of the yield stress of the material. A different set of uncoated samples was tested in air at stress levels of 481, 618, and 687 MPa in order to compare the behavior displayed with that of the samples tested under corrosive conditions. Also, in order to determine the fracture surface characteristics and the role of the coating in the failure mode, some fracture surfaces were observed in the scanning electron microscope (SEM).

3. Experimental Results and Discussion

3.1 Monotonic Mechanical Properties and Fatigue Tests

The results of the tensile tests in samples of the substrate material after the heat treatment was applied indicated a yield stress of approximately 787 MPa and a tensile strength of about 938 MPa. The reduction in area achieved was approximately 48% and the hardness of the material attained a value of about 23 HRC. The application of the coating did not affect any of the mechanical properties determined in tension. The number of

Table 1 Average value of the number of cycles to failure (N_f) versus the stress amplitude (S) for the fatigue and corrosion-fatigue tests conducted on both notched and unnotched specimens.

Stress amplitude, MPa	Substrate	Substrate notched specimens	Substrate specimens in corrosive medium	Substrate notched specimens in corrosive medium	Notched and coated specimens in corrosive medium	Unnotched and coated specimens in corrosive medium
481	…	…	213,220	123,940	116,560	261,133
516	…	…	130,240	…	…	131,266
550	344,780	89,210	96,717	65,180	63,740	92,133
618	144,180	45,900	44,980	45,520	42,080	47,280
687	65,980	28,930	…	…	…	…

cycles to failure for both the fatigue and the corrosion-fatigue tests conducted on the substrate material and the substrate coated samples, both notched and unnotched, are given in Table 1. Each value represents the average of at least five tests.

3.2 Corrosion-Fatigue Tests

Figure 1 illustrates the results presented in Table 1 plotted in a double logarithmic scale as a number of *S-N* curves. The results for each condition can be described by means of straight lines, which suggest a possible relationship between the stress amplitude (*S*) and the number of cycles to failure (*N*) of the form:

$$S = AN^{-m}$$

Here, *A* and *m* represent material parameters that are determined from the experimental data. Table 2 summarizes the results obtained by applying a linear least square analysis to such data. In general, it can be observed that the correlation coefficients for the samples tested in air are much better than those determined from the corrosion tests. As expected, there was a significant reduction in the fatigue life as a result of the corrosive environment, as shown in Fig. 2, in comparison with the fatigue life of the specimens tested in air. This reduction is more pronounced for low stress amplitudes because, under these conditions, there is more time for the occurrence of the corrosive damage mechanisms.

Table 2 Least square analysis of the *S* versus N_f data determined experimentally

Material condition	Resulting equation, MPa
Unnotched samples of substrate material tested in air	$S = 3055\ N_f^{-0.135}$
Unnotched samples of substrate material tested in air	$S = 5113\ N_f^{-0.196}$
Notched samples of substrate material tested in NaCl	$S = 5113\ N_f^{-0.163}$
Notched samples of substrate material tested in NaCl	$S = 8488\ N_f^{-0.245}$
Coated unnotched samples tested in NaCl	$S = 3001\ N_f^{-0.148}$
Coated notched samples tested in NaCl	$S = 8279\ N_f^{-0.244}$

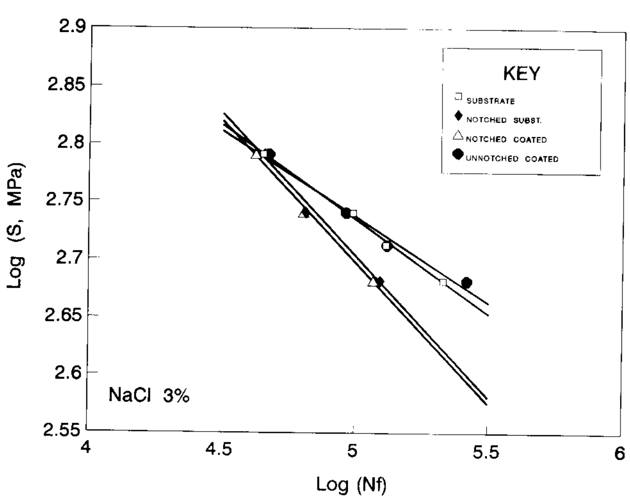

Fig. 1 Number of cycles to failure as a function of the stress amplitude under corrosion-fatigue testing. Substrate samples and substrate and coated samples both in notched and unnotched condition

In most corrosion controlled processes, the corrosion pits are formed before or concurrently with the nucleation of fatigue cracks. These pits act as stress raisers and decrease the fatigue life. This behavior is evidenced by multiple steps observed in the fracture surface of the samples tested in the corrosive medium as a result of the crack growth from different corrosion pits. It was expected that this behavior would be strongly dependent on the test frequency. According to the results obtained by Barsom (Ref 10), it was believed that at this test frequency there would be little influence of the corrosive environment in the crack growth rate during stage two of propagation, and therefore, the principal contribution of the sodium-chloride solution would be the crack nucleation and acceleration of stage one crack growth. Thus, the differences obtained in the fatigue life between the notched specimens tested in air and in sodium-chloride could be the result of the fatigue nucleation process at the notch and the subsequent growth of multiple cracks due to the corrosive environment, a fact shown in the photomicrograph corresponding to Fig. 3. At higher stress amplitudes this difference vanishes.

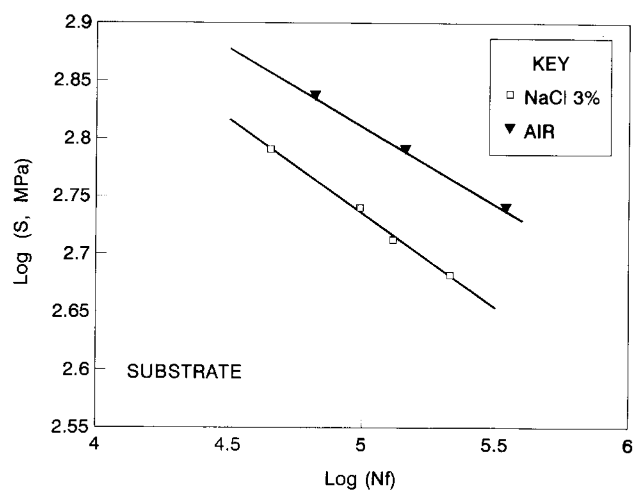

Fig. 2 Number of cycles to failure as a function of the stress amplitude both under corrosion-fatigue and air testing

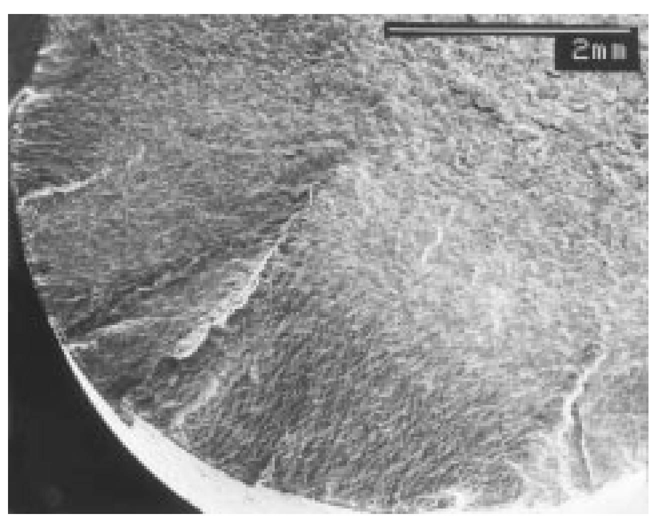

Fig. 3 SEM of a fracture surface showing multiple steps

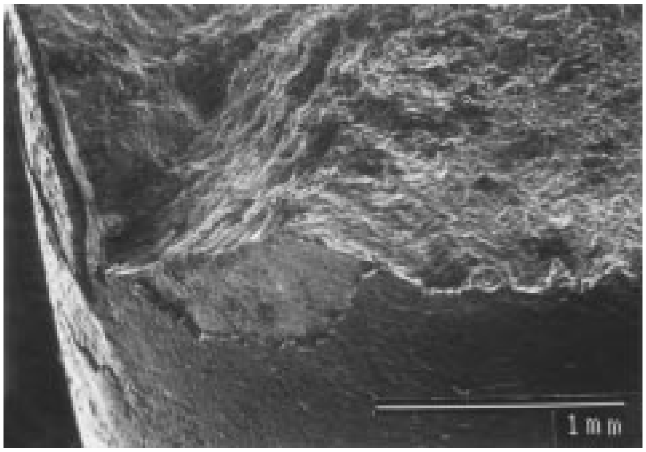

Fig. 4 SEM of a coated sample tested under corrosion-fatigue conditions at 481 MPa illustrating the detachment of the coating

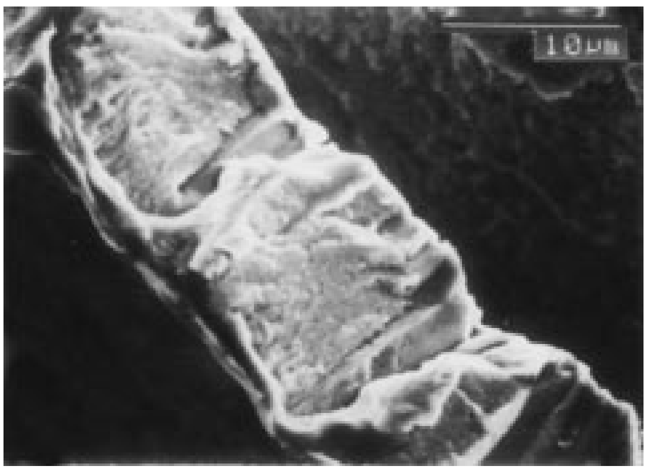

Fig. 5 SEM showing a brittle fracture of the coating of a sample tested under corrosion-fatigue conditions at 481 MPa

The effect of the electroless nickel-phosphorus coating on the corrosion-fatigue properties of the AISI 1045 steel is also presented in Fig. 1, which includes the fatigue behavior of the substrate and the unnotched specimens. A small increment in the fatigue life is observed after the application of the coating for samples tested at 516 MPa and below this value the difference becomes higher until it reaches an increment of approximately 20% at 481 MPa. For stress amplitudes higher than 516 MPa, there are no appreciable differences between curves. The fracture surface analysis of the coated specimens shows a significant detachment of the deposit for all the stress levels employed during testing as shown in the photomicrograph of Fig. 4. In some cases, these features also could be seen with the naked eye. It would seem that the early fracture of the coating would be produced by the cyclic straining of the material rather than by the initial deformation. In order to corroborate this hypothesis, a coated specimen was bent to an angle of 90° and the subsequent observation in the microscope did not reveal cracks or any detachment of the deposit. Figure 1 also illustrates the results obtained with the coated and notched specimens as compared with the uncoated notched substrate, where a small decrease of approximately 6% in the fatigue life of the coated specimens indicates only a slight influence of the coating on the notched samples.

No microscopic feature regarding the occurrence of intergranular fracture in the early stages of the crack growth was observed, as reported by Chitty et al. (Ref 9), after testing this same steel but in the annealed condition. Neither were any microscopic signs present indicating the possible occurrence of hydrogen embrittlement. Finally, the photomicrograph corresponding to Fig. 5 shows the brittle fracture of the deposit and the defective adhesion of the coating to the substrate.

4. Conclusions

The corrosive environment generated by a 3% sodium chloride solution produces a significant reduction in the fatigue life of the quenched and tempered AISI 1045 steel. This reduction could reach up to approximately 73% for an amplitude stress level of 550 MPa. Also, a higher reduction of the fatigue life could occur for lower stress amplitude levels. The fracture surface of the samples tested under a corrosive environment revealed the presence of multiple steps as a result of the growth of numerous cracks that nucleated at the corrosion pits. The electroless nickel-phosphorus coating has a slight influence on the corrosion-fatigue behavior of the AISI 1045 steel when tests are conducted in a 3% sodium chloride solution. In this case, an increment of about 20% in the fatigue strength was found at a stress amplitude of 481 MPa which corresponded to approximately 60% of yield stress of the material. For higher stress levels no appreciable differences in the fatigue life of the coated and uncoated specimens were observed. The corrosion-fatigue life of notched specimens was not affected by the deposition, at least from 481 to 618 MPa, stress levels studied in this investigation.

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