Fatigue properties of an AISI 1045 steel coated with an electroless Ni-P deposit

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Abstract

The fatigue behavior of samples of an AISI 1045 steel plated with electroless Ni-P (EN) deposits, both on an industrial and laboratory scale, has been evaluated under completed reverse bending or rotating bending conditions. The fatigue experiments were conducted in air at a frequency of 50 Hz, in the stress range of 292±346 MPa. On the laboratory scale, the deposition was carried out in an alkaline solution and the coatings were 17 μm thick, had a P content in the range of 8.4±9.4 wt.% and were post-heat treated in two different ways: at 473 K for 1 h and at 473 K for 1 h followed by 673 K for 1 h. On an industrial scale, the deposition was conducted in an acid bath, the deposits were 25 μm thick, had a P content in the range of 10.4–11.3 wt.% and were post-heat treated at 673 K for 1 h. Both deposits are observed to reduce the fatigue life of the coated components in relation to the uncoated substrate. The reduction in fatigue life is more severe for the samples coated at the laboratory scale, post-heat treated at 673 K. In general, the reduction in fatigue life is associated with the passage of fatigue cracks nucleated in the deposit to the substrate material rather than the nucleation of cracks at the deposit-substrate interface. The nucleation and propagation of such cracks from the surface of the EN coating is enhanced by the microstructural characteristics at the surface of the deposits, by residual stresses present in the coatings and by degradation of the fatigue properties of the coating from the interface to surface. An increase by approximately 2 wt.% in the P content of the deposit, compensates for the reduction in fatigue life induced by an increase in the coating thickness of 8 μm. The parameters involved in the relationship put forward earlier by Basquin, to describe the change in the number of cycles to failure in terms of the alternating stress, are reported for all the conditions investigated. © 1999 Published by Elsevier Science Ltd. All rights reserved.

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1. Introduction

The influence of electroless Ni-P deposits (EN) on the fatigue and corrosion-fatigue properties of plain carbon steels has been investigated in the past few years, by a number of authors [1–5]. In a preliminary study conducted by Puchi et al. [1], it was shown that by depositing these materials with EN coatings, a significant increase in the fatigue life could be achieved, both in the as-coated and coated and post-heat treated (PHT) conditions.

Although no direct measurements of residual stresses were carried out, the authors suggested that such a behavior could be associated with significant compressive residual stresses in the deposit which hindered the propagation of fatigue cracks into the substrate. The EN deposits that were investigated were observed to remain well adhered to the substrate even after the application of a large number of loading cycles. The increase in fatigue life was more noticeable at low alternating stresses since at elevated stresses both the coated material and the uncoated substrate tended to behave in a similar manner.

The mechanical strength of the substrate also had a significant influence in the fatigue results. A 0.1% C steel, both in the as-coated and coated and PHT conditions, showed a marked increase in the fatigue life than a 0.45% C steel with similar coatings. In a subsequent investigation, Chitty and co-workers [2] showed that EN deposits of approximately 20 μm thickness and 10 wt.% P increased the corrosion-fatigue properties of an AISI 1045 steel in the applied...
stress amplitude range of 221–331 MPa, in the presence of an aqueous solution of 3 wt.% NaCl.

However, in this study the increase in fatigue life was more marked when the coating was in the as-deposited condition rather than after a PHT at 473 K for 4 h, in spite of the increase in hardness observed after such a PHT. For a substrate of this mechanical strength (358 MPa yield strength and 703 MPa ultimate tensile strength), the monotonic mechanical properties remained unchanged by the presence of the coating. SEM observations of the surfaces of the coated samples after testing highlighted the importance of proper agitation of the deposition bath in order to avoid the formation of microporosities and ‘orange-peeling’ type effects.

Berrios et al. [3], in a recent investigation, carried out a number of fatigue tests under rotating bending conditions employing samples of AISI 1045 steel with and without the application of EN deposits of elevated P content and thicknesses ranging between 7 and 37 μm. In this study, the coated samples were PHT according to two different schedules involving one and two steps, respectively. Contrary to the previous findings, an increase in coating thickness gave rise to a slight reduction of both yield and ultimate tensile stress, whereas the reduction in area at fracture was severely diminished independent of the PHT cycle applied.

In this investigation, no increase in the fatigue life of the coated samples was observed. At most, it was determined that steel plus a 7 μm thickness coating behaved similarly to the substrate and that as the coating thickness increased, the fatigue life of the material was severely reduced. This decrease in fatigue properties was observed to be more marked for the two-step cycle PHT.

Chitty et al. [4], in a complementary study, determined that EN deposits of approximately 10 wt.% P and about 20 μm thickness either had no effect or sometimes increased the corrosion-fatigue properties of notched and unnotched samples of a quenched and tempered AISI 1045 steel in the stress amplitude range of 481–687 MPa, in the presence of an aqueous solution of 3 wt.% NaCl. In contrast to the results reported for the same substrate but in the annealed condition, these authors observed that the increase in the properties under evaluation was produced when the stress amplitude under rotating bending conditions was below 516 MPa.

For the notched specimens no substantial differences were observed between the fatigue life of the coated and uncoated specimens. As expected, the corrosive environment generated by a 3 wt.% NaCl solution produced a significant reduction in the fatigue life of the quenched and tempered steel, which could reach up to approximately 73% for a stress amplitude level of 550 MPa. Therefore, it was concluded that the EN deposits had only a slight influence on the corrosion-fatigue behavior of the substrate steel thus treated when tests were conducted in this corrosive medium.

Pertuz and co-workers [5], have also evaluated the influence of an EN deposit on the corrosion-fatigue properties of an annealed AISI 1045 steel employing three different corrosive media including distilled water and two NaCl solutions of different concentration (3 and 5 wt.%). Corrosion-fatigue tests were conducted under rotating bending conditions at alternating stress levels ranging between 219 and 329 MPa at a frequency of 50 Hz. In this investigation, the corrosion-fatigue properties of the coated and uncoated substrate were very similar if testing was conducted in salty water.

However, during testing in distilled water the corrosion-fatigue properties of the coated substrate diminished in relation to the uncoated material. The fractographic analysis conducted on the fracture surfaces of the samples revealed for the first time in these studies, the presence of fatigue marks within the EN deposit which indicated that the fracture mechanism of the coating was associated with the cyclic loading of the material.

The present investigation has been carried out in order to study with somewhat more detail the effect of an industrial, 25 μm thickness EN deposit with a PHT at 673 K for 1 h, on the fatigue properties (fatigue life and fatigue limit) of an AISI 1045 steel, under rotating bending conditions in air and to compare these results with those obtained from samples of the same substrate coated with an EN deposit of a smaller thickness and P content but plated at a laboratory scale where the deposition conditions can be more closely controlled.

2. Experimental techniques

The present investigation has been carried out with samples of an AISI 1045 plain carbon steel with 0.43 C, 0.65 Mn, 0.11 Si, 0.11 S and less than 0.02 P. The material was provided as bars of approximately 12.7 mm diameter and 6 m length. Therefore, it was first cut to pieces of approximately 120 mm length and subsequently machined into tensile and fatigue specimens with a gage diameter of 6.35 mm, gage length of 12.7 mm, fillet radius of 25.4 mm and shoulder diameter of 12.7 mm, according to the ASTM standard E 606.

All the samples were heat treated at 1098 K for 20 min in an argon atmosphere, and cooled to room temperature within the furnace to avoid the oxidation of the samples. The total cooling period was about 24 h. The specimens were subsequently ground with successive SiC papers grit 600–1200 and polished mechanically. Afterwards, 40 of these samples were degreased in a 5 vol.% HCl solution at 348–353 K for 7 min, rinsed again in distilled water, rinsed in a sodium bicarbonate (100 g/l) solution and rinsed in water.

The deposition was conducted industrially (the process was carried out at Tecnologías Aplicadas C.A., San Diego de los Altos, Venezuela) employing a bath composed of 30 g/l nickel sulphate, 30 g/l sodium hypophosphite, 35 g/l...
malic acid, 1.5 ppm lead sulphate, 10 g/l succinic acid and a stabilizer. During deposition the pH was maintained at approximately 4.6–4.8, at a mean temperature of 358–363 K. The deposition rate varied between 12 and 15 μm/h and the process was conducted for approximately 2 h which provided thicknesses ranging from 24–30 μm. Such thicknesses were corroborated by means of the ball craterring technique (Calotest, CSEM) and image analysis (LECO 500).

The deposition process of the samples plated on the laboratory scale (30 specimens) as well as the evaluation of the P content, thickness of the deposit and PHT applied have been described in detail elsewhere [3]. The chemical composition of the bath consisted of 21 g/l nickel chloride, 24 g/l sodium hypophosphite, 30 g/l ammonium chloride and 45 g/l sodium citrate. The deposition was conducted at a pH of 8 at 343 K, maintaining a ratio of solution volume to deposition area of approximately 60 ml/cm². The bath was continuously agitated and a deposition time of 180 min was employed to obtain a thickness of 17 μm. Both the volume and pH of the bath were maintained by adding continuously a solution of 10% KOH.

Once the EN deposition of the samples plated on an industrial scale was completed, the specimens were PHT at 673 K for 1 h in an argon atmosphere to avoid oxidation. The chemical analysis of such deposits was determined by means of scanning electron microscopy (SEM) techniques (Philips 505) with energy dispersive spectroscopy (EDS) facilities. Knoop microhardness measurements were conducted in the deposits employing a load of 100 g applied for 10 s.

Tensile tests were carried out on a computer-controlled servohydraulic machine (Instron 8502) at a cross head speed of 10 mm/min. At least three samples were employed for characterizing the monotonic mechanical properties of the coated and uncoated substrate. Fatigue tests were carried out under rotating bending conditions (Fatigue Dynamics, RBF-200) at a frequency of 50 Hz and alternating stresses of 292, 310, 328 and 346 MPa. A total of 25 samples were employed for this purpose which exceeds the minimum number of specimens required in fatigue testing for reliability data according to the ASTM standard 739 (12–24 samples).

Thus, the testing procedure followed in the present work allowed a replication of the order of 80%. The fatigue limit was determined employing ten samples, according to the staircase method. In order to allow a meaningful comparison of the fatigue life of the coated and uncoated substrate, all the samples were mechanically prepared in order to have similar polished surfaces. The fracture surfaces of the samples were analyzed by means of SEM techniques, particularly in relation to the initiation of fatigue cracks and the different stages of their propagation. Replicas of the fracture surfaces of the samples were also prepared to be subsequently analyzed by means of transmission electron microscopy (TEM) methods.

3. Results and discussion

3.1. Characteristics of the deposit

As shown in the optical photomicrograph of Fig. 1a, the metallographic analysis of the samples revealed a typical microstructure for this substrate material composed of a ferrite-perlite mixture oriented along the axis of the bar, whereas the SEM photomicrograph of Fig. 1b shows the deposition of an apparently uniform coating with satisfactory adhesion characteristics. The coating thickness had a mean value of approximately 25 μm.

The EDS analyses conducted on the deposit showed that the P content ranged between 10.4 and 11.3 wt.%. According to the investigation conducted by Parker and Shah [6] regarding the residual stresses developed by EN plating on
different substrates, for steels with EN deposits with a P content greater than approximately 10.3%, the residual stresses in the coating are expected to be of a compressive nature. However, the study carried out by Parker and Shah [6] also revealed that a PHT at 673 K, which is typically applied to achieve maximum hardness in these coatings, gives rise to an increase in the tensile residual stress to approximately 82 MPa. Knoop microhardness tests were conducted on the deposit after the PHT employing a load of 100 g. The microhardness achieved a value of 923 HKN100 which is associated with the precipitation of Ni3P particles during the PHT [3,7–9].

3.2. Evaluation of mechanical properties

Tensile tests were conducted with samples in the uncoated and coated and PHT conditions in order to evaluate if the deposit had any influence on the monotonic properties of the material. In agreement with previous findings, the deposits obtained in the present study did not give rise to any significant change in both the yield and ultimate tensile stresses (UTS). The yield stress of the material was approximately $353 \pm 14$ MPa whereas the UTS was approximately $607 \pm 10$ MPa.

During testing, the deposit remained well adhered to the substrate and free of cracks even near the fracture plane within the necked zone of the samples, which indicates that despite the relatively elevated hardness achieved after the PHT applied, the deposit is able to sustain a substantial ductility in tension. These observations are consistent with the results reported by Hammond and Williams [10] regarding the mechanical properties of low or medium strength steels plated with electrodeposited Ni. According to these authors, an adherent coating of this deposit, unlike chromium, increases the mechanical strength and tensile properties relative to those of the substrate material, in proportion to its thickness.

Thus, in relation to the fatigue tests conducted in our investigation in order to study fatigue life, the evaluation of the static properties of the material established a stress amplitude range of 274–346 MPa, corresponding to a fraction of the yield stress between 0.83 and 0.98. Table 1 presents the data corresponding to the number of cycles prior to fracture ($N_f$) in terms of the alternating stress applied to the material ($S$), including the values obtained for the uncoated substrate, for the samples plated on an industrial scale and for those coated at the laboratory level. These last samples had a coating thickness of 17 µm and were PHT according to two different schedules [3]: PHT-1 involved heating the samples at 473 K for 1 h and PHT-2 involved a treatment at 473 K for 1 h with a subsequent treatment at 673 K also for 1 h.

Fig. 2 illustrates the results obtained. At each alternating stress level at least five tests were carried out in order to fulfill the reliability conditions prescribed in the ASTM standard E 739. Several important aspects must be highlighted regarding Fig. 2. Firstly, all the plated samples displayed a decrease in fatigue life in relation to the fatigue behavior of the substrate material. At elevated alternating stress levels (346 MPa) the curves obtained for the samples plated on an industrial scale (25 µm thickness coating) and those plated at the laboratory level (17 µm thickness coating) with PHT-1 behave very similarly.

The reduction in fatigue life for both set of samples, in comparison to the uncoated substrate, is 45–51% whereas at low stresses (292 MPa) the samples with a thicker coating display a larger reduction in fatigue life (65%) than those with a thinner deposit (54%). The worst fatigue behavior is observed for the samples with a deposit of 17 µm and PHT-2 in which the maximum reduction in fatigue life is 81% at 292 MPa and 76% at 346 MPa. The interpretation of these results involves two separate issues.

Table 1

<table>
<thead>
<tr>
<th>$S$ (MPa)</th>
<th>Substrate</th>
<th>17 µm (PHT-1)</th>
<th>17 µm (PHT-2)</th>
<th>25 µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>274</td>
<td>1219780 ± 431966</td>
<td>224750 ± 25794</td>
<td>122950 ± 51073</td>
<td>165463 ± 90517</td>
</tr>
<tr>
<td>292</td>
<td>473960 ± 91956</td>
<td>115000 ± 33229</td>
<td>38380 ± 8240</td>
<td>74667 ± 18550</td>
</tr>
<tr>
<td>310</td>
<td>224560 ± 34118</td>
<td>46000 ± 7628</td>
<td>21920 ± 7933</td>
<td>38780 ± 3344</td>
</tr>
<tr>
<td>328</td>
<td>93600 ± 18828</td>
<td></td>
<td></td>
<td>25150 ± 7670</td>
</tr>
<tr>
<td>346</td>
<td></td>
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</tr>
</tbody>
</table>

![Fig. 2](image-url)
for electrodeposited Ni and confirmed by Berrios et al. [3] for EN plating, it is expected that as the coating thickness increases, the loss in fatigue strength should also increase. On the other hand, the effect of the PHT applied which, in general, are designed not only to eliminate hydrogen embrittlement of the steel substrate but also to improve the adhesion and hardness of the deposit.

Such PHT are considered to have a negligible effect on the fatigue properties of plated components employing substrates of medium and high strength steels, although Berrios et al. [3] have recently shown for the same substrate that the PHT applied to deposits of 7 and 17 \( \mu \text{m} \) can give rise to a significant reduction in the fatigue life of the coated component. Fig. 2 illustrates that the fatigue behavior of the samples with a 25 \( \mu \text{m} \) deposit, coated on the industrial scale with a PHT at 673 K for 1 h, behave similarly to the specimens with a 17 \( \mu \text{m} \) deposit with a PHT at 473 K also for 1 h.

According to Hammond and Williams [10], the fatigue behavior of the coated samples is significantly dependent on the residual stresses in the deposit, which in turn are intimately related to the P content, as shown by Parker and Shah [6]. Compressive residual stresses associated with a high P content are expected to give rise to better fatigue properties. Therefore, it could be concluded that the increase in coating thickness for the specimens plated on an industrial scale, is somehow compensated for by the type of bath employed, which induces a larger P content in the deposit (10.4–11.3 wt.\%) than the one present in the coatings deposited on the laboratory scale (8.4–9.4 wt.\%) from a different solution. In summary, these results suggest that an increase by approximately 2 wt.\% in the P content of the deposit, compensates for the reduction in fatigue life induced by an increase in the coating thickness of 8 \( \mu \text{m} \).

Fig. 2 shows a linear relationship between the alternating stress and the number of cycles to failure in a double logarithmic scale. This indicates the validity of the simple parametric expression earlier suggested by Basquin [11]

\[
S = AN_f^{-m}
\]

where \( A \) and \( m \) represent constants that depend on both the material properties and testing conditions. In particular, \( A \) is related to the fatigue strength coefficient of the material and \( m \) is sometimes termed the fatigue exponent. Table 2 summarizes the values of the parameters \( A \) and \( m \) for the four sets of data represented in Fig. 2. The fatigue limit of the samples coated on an industrial scale is of 244 ± 4 MPa.

Since EN plating is widely used for improving the appearance, wear and corrosion resistance of many industrial components and also for restoring worn or overmachined parts to plan size, the above equation together with the values calculated for the constants involved is of value to predict, as a first approximation, the expected performance of such components and parts when subjected to cyclic loading.

### 3.3. Evaluation of the fracture surfaces of the samples

Some of the fatigue specimens plated on an industrial scale and tested at 292 and 346 MPa, were analyzed after fracture by means of SEM techniques, in order to study more closely the microstructural features of the sites of crack initiation as well as the general microstructural changes that take place during the subsequent propagation of such cracks throughout the cross section of the samples.

![Fig. 3. (a) A crack initiation site in a commercially coated sample tested at 292 MPa. The upper region (S) represents the substrate material. In this particular case, the crack propagates from the deposit towards the substrate. (b) Magnified view of (a). The deposit (D) is observed to be severely damaged in a highly localized area. 'Orange-peeling' type effects (OP) are also observed at the lateral surface (LS) of the sample.](image)
The photomicrograph of Fig. 3 illustrates a typical crack initiation site in one of the samples tested at 292 MPa. The coating has been severely fractured in a highly localized area prior to the propagation of the crack towards the substrate. In particular, it is possible to observe a predominant crack nucleated at the surface of the sample which runs to an approximate angle of 45° to the normal of the cross section of the specimen. These features are consistent with the previous findings of Berríos et al. [3] which indicate that the fracture process in the coated samples with deposits of this thickness is initiated in the deposit itself rather than at the deposit-substrate interface.

The present observations are also consistent with the results reported by Hammond and Williams [10] in electrodeposited Ni which indicate that there could be a significant difference in the inherent fatigue properties of the coating from the interface to the outer surface, which coupled with the fall in the applied stress from the external surface to the interface under bending would favor the nucleation of cracks at the surface. The initiation of cracks at the surface would also be assisted by another microstructural feature visible in this photomicrograph, namely an orange peeling effect on the lateral surface of the specimen.

Such an effect has already been reported to occur during EN deposition by Chitty et al. [2] and it is widely accepted that it is associated with trapped hydrogen bubbles in the deposit which are produced by the reduction reaction that takes place during plating. These effects can be more clearly seen in the photomicrograph of Fig. 4 which illustrates the presence of a number of spheroids on the surface of the specimen and the severe fracture of the coating at the site where the main fatigue crack starts propagating towards the substrate.

Finally, in Fig. 5 it is possible to observe some secondary cracks developing along the coating–substrate interface which indicates how cyclic loading is also able to under-

mine the adhesion of this type of coating to the substrate, leading eventually to their complete separation. In summary, the microstructural evidence suggests that the decrease in fatigue performance of the specimens plated with EN deposits of the type studied in the present work is mainly due to the passage of fatigue cracks from the coating to the substrate rather than the nucleation of such cracks at the coating–substrate interface.

The propagation of the cracks from the coating is facilitated by the relatively good metallurgical bonding that is initially established between them. Both the microstructural characteristics of the coating and the decrease in fatigue properties of the coating from the interface to the surface of the sample enhance the nucleation of fatigue cracks at the surface itself. In addition, if the P content of the deposit favors the presence of tensile residual stresses, as expected for the samples plated on the laboratory scale, the propagation of the cracks already nucleated is accelerated, leading to a more marked reduction in the fatigue life of the component.

Therefore, whenever the nucleation and propagation of fatigue cracks is easier at the surface of the EN deposit plated on a structural part than at the surface of the uncoated substrate itself, such a deposit is bound to induce a reduction of the fatigue life of the coated part.

4. Conclusions

The fatigue performance of samples of a medium-strength plain carbon steel, widely employed in the manufacture of different parts and components, plated with two different electroless Ni-P deposits has been evaluated and compared with the behavior of the uncoated substrate. Both deposits reduce the fatigue life of the coated samples in relation to the uncoated steel. The reduction in fatigue life is more severe for the samples coated at the laboratory scale.
(PHT at 673 K). Such a reduction is due to the passage of fatigue cracks nucleated in the deposit, into the substrate rather than the nucleation of cracks at the deposit–substrate interface.

This fact is considered to be of utmost importance, indicating that the fatigue behavior of the coated substrate would be determined mainly by the synthesis of the electroless Ni-P deposit. The nucleation of such cracks at the surface of the electroless Ni-P coating and their subsequent propagation are enhanced by three important aspects: the microstructural characteristics at the surface of the deposits, the predominant residual stresses present in the coating and the degradation of the fatigue properties of the coating from the interface to surface. In turn, the residual stresses present in the deposit can be modified by the P content and thickness.

If the nucleation and propagation of cracks from the electroless Ni-P coating to the substrate is easier than the nucleation of such cracks at the surface of the uncoated substrate material, the coated component will always display a reduction in fatigue performance, provided that the quality of the metallurgical bonding between coating and substrate is relatively good. An increase by approximately 2 wt.% in the P content of the deposit, compensates for the reduction in fatigue life induced by an increase in the coating thickness of 8 μm. Specific equations for design applications have been proposed in order to calculate the reduction in fatigue strength of this substrate, once it has been coated with such deposits.

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References