Effects of Pretreatment Prior to Electroless Ni-P Plating on Fatigue Behavior of SAE 1045 Steel

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Abstract

Electroless Ni-P (EN) plating, as an important group of metallic coatings, is employed in a wide range of industrial applications. The current work aims to investigate the effects of pretreatment process before EN plating on fatigue behavior of SAE 1045 steel. The specimens of rotating bending fatigue test were prepared from the steel in two series. A group of samples was used in as-polished condition as the base metal and the next group of samples was degreased using Aston and then NaOH 150 gr/lit at the room temperature. Subsequently, pickling carried out in an aqueous solution of HCl 30 Vol.% for 3 min. Finally, the etching of samples was done for 30 seconds in an aqueous solution of HCl 20 Vol.% at 50 °C. In the following, rotating bending fatigue test results showed an increase in the fatigue strength of pretreated samples compared to that of the base metal. Reducing the roughness of the surface caused by the pretreatment operation was responsible for this behavior. According to SEM observations, the pretreatment process influenced the crack initiation stage, which eventually resulted in a better performance in the fatigue strength.

Keywords: Pretreatment; Electroless; Fatigue; SAE 1045.

1. Introduction

Electroless Ni-P (EN) plating, as an important group of metallic coatings, is employed in a wide range of industrial applications. These coatings provide higher hardness, remarkable wear resistance and better corrosion resistance properties ¹⁻⁴. However, protection against corrosion and wear can result in a variation of other properties such as fatigue strength.

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According to some literature 5-6), EN plating may lead to the deterioration of fatigue life of the steel. It has been also reported 7) that the fatigue strength of the steels can be deteriorated if the P content of the coating would be less than 10 wt.%. Lower P contents tend to induce residual tensile stresses of the deposits. Zhang et al.⁸⁾ have reported that the fatigue limit of 30CrMo steel decreased by about 39 % after EN plating. Also Graces et al.⁹⁾ have reported that plating of AISI 4340 steel with EN leads to a significant reduction in the fatigue limit of the material up to 92 %. Fig. 1 shows the effects of electroless Ni-Cu-P plating on rotating bending fatigue behavior of the SAE 1045 steel as S-N_c (Maximum Stress as a function of Number of Cycles prior to Fracture) curves. Fatigue limit of quenched and tempered SAE 1045 steel as the base metal decreased up to 18 % after plating with electroless Ni-Cu-P as deposited. It is noteworthy that some of this reduction is compensated

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by shot peening before the EN plating ¹⁰). On the other hand, Rahmet et al. have reported that higher P contents in EN plating induce compressive residual stresses ¹¹). In another research by Lonyuk et al.; it was showed that the fatigue strength increased in the steels subjected to EN plating of 12 wt.% P ¹²).



Fig. 1. S-N_f comparative curves of the Base, As Deposited and Shot Peened samples $^{10)}$.

The ratio of substrate strength to coating strength, coating thickness, coating internal residual stresses and the heat treatments applied for hydrogen affect the fatigue properties of Ni-P coated steel substrates ⁷⁻¹². The effects of residual stresses, plating-substrate interface and micro cracks are well known for many different materials; while there are no reports on the effect of pretreatment process on the fatigue behavior in the literature.

The applications areas of SAE 1045 steel include the production of Bolts, Studs, Gears, Axles, Shafts and Machine parts ¹³⁾. The issue that is significant about these parts is the importance of fatigue and wear resistance in these applications. Also according to the previous study ¹⁰, SAE 1045 steel has shown to be of good quality in Ni-P plating. The aim of this work is to study the effect of pretreatment process prior to EN plating on the fatigue behavior of SAE 1045 steel. The variables used in this paper, including the type of steel, the chemical composition, pretreatment parameters prior to electroless Ni-P plating, fatigue tests, etc., are identical to the work of T. Saeid et al. 10). By comparing the results of this study and the results of work has been done by Saeid et al., the exact reduction in the fatigue strength due to the electroless Ni-P plating will be achieved.

2. Experimental Procedure

According to the work by T. Saeid et al. ¹⁰, material used in this investigation was SAE 1045 steel. The chemical composition of the steel has shown in Table 1. Samples for tensile and fatigue tests were machined off from bars of 14 mm diameter in the as-rolled condition according to ASTM E 08 ¹⁴ and DIN 50 113 ¹⁵ standard test methods, respectively. The dimensions of tensile and fatigue test samples have been shown in Figs. 2 and 3, respectively.

The machined samples were austenitized at 850 °C for 15 min. and then hardened by quenching in agitated oil at 60 °C. The samples then were stress relieved at 550 °C for 60 min to minimize the possible residual stresses caused by quenching. Fatigue samples were ground and polished longitudinally to give them a high degree of surface finish and to remove the decarburized layer. A group of samples used in as-polished condition as the base metal

Table 1. The chemical composition of the SAE 1045 steel.



Fig. 2. Dimensions of the tensile samples.



and the next group of samples degreased by Aston and then NaOH 150 gr/lit at room temperature. Subsequently, pickling carried out in an aqueous solution of HCl 30 Vol.% for 3 min. Finally, the etching of samples done for 30 seconds in an aqueous solution of HCl 20 Vol.% at 50 °C. In order to compare the achieved fatigue test results, the pretreatment process used in this work, was quite similar to T. Saeid et al. work ¹⁰.

For each group of samples, three tensile tests were conducted to obtain mechanical properties. A computer controlled electro-hydraulic test system was used to perform tensile tests. All tensile tests were done at room temperature with a cross head speed of 0.1 mm.min⁻¹ according to ASTM standard test method E 08⁻¹⁴.

The effect of pretreatment process on the fatigue resistance of the material was assessed using a Roell Amsler UBM 200^{TM} rotating bending fatigue test machine under cyclic loading conditions. Test samples were subjected to constant amplitude loading conditions, R= -1, with maximum stress values ranging from approximately 450 up to 650 MPa. All fatigue tests were conducted at room temperature in a frequency of 3500 rpm according to DIN 50 113 standard test method ¹⁵). In all cases, the fatigue limit was assumed to be the stress at which the steel would not break by completion of 10^6 cycles. Finally, fracture surface examinations were carried out on a CamScan MV-2300TM scanning electron microscope (SEM).

3. Results and Discussion

The average mechanical properties obtained from the tensile test have been shown in Table 2 for the base metal and pretreated samples. The results revealed that the tensile properties have not shown significant variation because of pretreatment process. It could be said that the pretreatment has no effect on these properties.

In addition, the issue that needs to be considered is the effect of released hydrogen during pretreatment process. Preparation of samples prior to EN plating, involves hydrogen release during pickling and etching processes. The hydrogen evolved in the process can diffuse into the surface layer of steel and be trapped by lattice defects. It is expected that the trapped hydrogen would result in the worsening of mechanical properties i.e. reduction in area and acceleration of the fatigue crack growth in the matrix. However, the results presented in Table 2 do not indicate such a deterioration in reduction in area properties, which it means that hydrogen did not have the ability or enough time to penetrate into the samples.

Fig. 4 shows the results of rotating bending fatigue test in the well-known Wohler diagram for two groups of samples at the room temperature. In this figure, the number of cycles prior to fracture as a function of the applied alternating stress plotted for two groups of samples.

Table 2. SAE 1045 average mechanical properties obtained from the tensile test.

Mechanical Properties	Base Metal	Base Metal + Pretreated
Ultimate tensile strength (MPa)	960	980
Yield strength (MPa)	860	860
Reduction in area (%)	41.5	41.8
Total elongation (%)	12	12.8



Fig. 4. The number of cycles prior to fracture as a function of the alternating stress for two groups of samples.

Fig. 4 clearly indicates that fatigue strength increased about 5.5 % at 106 cycles because of pretreatment process. Fatigue strengths at 10^6 cycles are 450 and 475 MPa for the base metal and pretreated samples, respectively. It can be concluded that hydrogen embrittlement did not happened in the pretreatment process. By comparing the results of the work of T. Saeid et al. ¹⁰ and the results of this study, the precise reduction of fatigue strength due to the EN plating is 23.4 %. In fact, this is a total of 18 % from Saeid's work and 5.4 % from this study.

Penetration of hydrogen into the samples during the preparation could have a negative effect on the fatigue behavior, but in practice the pretreatment process has improved the fatigue behavior of the steel. It is noteworthy that the base metal and pretreated samples indicated similar mechanical properties. The surface roughness measurements indicated that R_a (average surface roughness) decreased from 0.35 to 0.15µm with employing pretreatment. It shows that the surface pretreatment has improved the surface quality by reducing the differences between the height of peaks and depth of valleys that exist even after surface polishing. Deoxidizing and etching of the samples would result in such a decrease in roughness.

Fatigue failures include a four-step process of crack initiation, stage I crack growth, stage II crack growth and finally ultimate fracture. The crack initiation stage begins with the nucleation of cracks within the material that normally occurs at stress concentration points. Then they propagate slowly during this stage along the crystallographic planes, with the highest shear stresses. Once they reach a critical size, propagate quickly during stage II crack growth in a direction perpendicular to the applied force. These cracks can eventually lead to the ultimate failure of the material, often in a brittle mode ¹⁶. Several researchers have investigated the effect of surface roughness on fatigue limit. Ryu et. al. ¹⁷) reported that the fatigue life of CrMoV steel with a rough

surface is approximately half of that of steel with a smooth surface at a test temperature of 550 °C. Sigmund Kyrre et. al. ¹⁸⁾ have developed models to predict fatigue life based on surface roughness characterization. Mohamed et. al. ¹⁹⁾ showed that the surface finish parameters had a significant correlation with the fatigue initiation life, final separation life and the fatigue endurance limit.

It can be anticipated that, fatigue cracks initiations have not occurred easily or that propagation rate of cracks has been retarded by pretreatments process. Based on the results of previous studies on the effect of surface finish on the fatigue life, it can be concluded that the reduction in surface roughness of samples as a result of pretreatment process has led to the improvement of fatigue behavior of studied steel.

The fatigue fracture surfaces of samples examined to describe the fracture morphology. From each series, typical samples were selected for SEM analysis. The microscopic fracture surfaces of the base metal and pretreated samples are shown in Figs. 5 (a, b) and 6 (a, b), respectively.

The regions of crack initiation, crack propagation and the final fracture zone are obvious on fatigue fracture



Fig. 5. Typical SEM micrographs of the base metal, tested at 475 MPa, a) Fatigue fracture surface and b) Final fracture region.



Fig. 6. Typical SEM micrographs of the base metal + pretreatment, tested at 475 MPa, a) Fatigue fracture surface and b) Final fracture region.

surfaces in figures 5 (a) and 6 (a). It is also evident that the fracture surfaces are almost identical in two different samples. All the fractures resulted from crack initiation at the surface of samples. No evidence of sub-surface fatigue crack initiation revealed from the fracture surfaces. As seen in the figures 5 (b) and 6 (b), final fracture zone in both series of samples are dominated by ductile dimples. In addition, shear lips appeared at the final fracture zone. It is worthwhile to mention that fracture shear lips of different planes do not seem smooth.

The crack initiation and propagation regions are responsible for more than 70 % of the fatigue life of the components ¹⁵). In order to determine the effect of pretreatment process on the relative area of the regions, the fracture surfaces evaluated quantitatively. Trapping of hydrogen in the defects can result in the faster fatigue crack propagation that consequently influences the surface area of crack propagation region. The variations of surface area ratio (SAR) of crack propagation region to the total fracture region for two different series of samples plotted as a function of applied stress in Fig. 7. The similarity of SAR values attribute to the fact that the crack propagation stage has not been affected by pretreatment. Thus, the improvement of fatigue strength of the samples is due to the effect of the pretreatment on fatigue crack initiation. In addition, fatigue crack propagation has not had a positive or negative contribution in fatigue behavior.



Fig. 7. Variations of surface area ratio (SAR) of crack propagation region to total fracture region as a function of applied stress.

4. Conclusions

 Pretreatment process prior to Electroless Nickel (EN) plating has not affected the mechanical properties of the steel obtained from tensile tests. By applying pretreatment process, the fatigue strength of the SAE 1045 steel has improved about 5.5 % at 10^6 cycles. Surface roughness reduction is responsible for this improvement.

- By comparing the results of the work of T. Saeid et al. ¹⁰⁾ and the results of this study, the precise reduction in fatigue strength due to the EN plating is 23.4 %. In fact, this is a total of 18 % from Saeid's work and 5.4 % from this study.
- Observation of the microscopic fracture surface showed that fatigue fractures were caused by crack initiation at the surface of samples. Only the initiation of fatigue crack has been retarded by pretreatment, and propagation of fatigue crack has not been affected by pretreatment. In addition, the final fracture mode exhibited predominant dimples for two groups of samples.

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