

## Shot Peening as an Alternative to Fatigue Life Improvement of CK45 Steel Coated with an Electroless Ni-Cu-P

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### Abstract

The effect of an electroless Ni-Cu-P coating on the fatigue behavior of quenched and tempered CK45 steel has been investigated. The fatigue tests under rotating bending conditions have been conducted in three different conditions of uncoated, as-deposited and shot peened prior to the coating deposition. The results indicate that plating of the base steel leads to a fatigue life reduction. The decrease in the fatigue strength was observed to be less marked for the shot peened specimens. The microscopic observations indicate that in the as-deposited condition, the reduction in fatigue life is associated with the nucleation of fatigue cracks on the lateral side of coating. On the contrary, when the samples were shot peened prior to the coating deposition, the crack initiation sites were displaced to the coating-substrate interface which led to an improvement of fatigue behaviour.

*Keywords:* Electroless Ni-Cu-P deposit, Fatigue, Shot peening, CK45 steel, Coating.

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### Introduction

Electroless Ni-P (EN) deposits represent an important group of metallic coatings that are employed on a wide range of substrates to provide protection against corrosion and abrasion wear. Such platings have found different applications in a variety of industries like electrical and electronics, oil and gas, machinery, aerospace, motor, chemical, foodstuff, textile and printing industries. Currently, their use continues to increase due to their better corrosion and chemical resistance and the ability to achieve extremely uniform thickness, even on parts with complex configurations<sup>1,2)</sup>.

On the other hand, it is well known that Ni-Cu alloys with the copper content of about 30 wt. % are highly corrosion resistant in many aggressive environments such as seawater. For these attractive features, many attempts were made to plate Ni-Cu alloys on metallic bases, and the introduction of copper in to electroless Ni-P deposits has been conducted in this regard<sup>3-5)</sup>. Recent studies<sup>6-10)</sup> have indicated that the characteristics of electroless Ni-Cu-P deposits, such as thermal stability, corrosion resistance, solderability, magnetic and electrical properties are superior to those of the electroless Ni-P deposits, and the electroless Ni-Cu-P deposits are regarded as a potential substitute for the Ni-P deposits<sup>11)</sup>.

However, protection against corrosion and wear can be gained at the expense of a decrease in other important properties such as fatigue life and fatigue limit, which could be of greatest importance in some of the applications pointed out above.

Regarding previous studies carried out on the effect of EN deposits on the fatigue properties of high strength steels (ultimate tensile strength of the order of 1200 MPa), Wu and coworkers<sup>12)</sup> conducted an investigation on the fatigue resistance of 30CrMo steel oil-quenched from 870°C and tempered at 620°C for 3h. In this investigation, a reduction in the fatigue limit of approximately 39% for the plated substrate in comparison with a reduction of 20% when the substrate was previously shot peened before plating has been reported.

The low fatigue strength of the coatings was found to be responsible for the decrease in the fatigue limit of the plated steel. Also Garces et al.<sup>13)</sup> reported that plating a quenched and tempered AISI 4340 steel with an EN deposit lead to a significant reduction in the fatigue life of the materials that can be reached up to 92%. The microscopic observations of the fracture surfaces of the samples conducted in this work indicated that the fatigue process was initiated at the surface of the deposit and subsequently, transferred to the substrate, with assistance of the metallic bonding established at the substrate-deposit interface. They also reported that in their study, the bonding between the deposit and the base steel was observed to be rather poor, a fact that was supported not only by the presence of extensive secondary cracking along the coating-substrate interface after fatigue testing, but also by the observation of the complete

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separation of the deposit from the substrate during tensile testing.

More recent studies conducted by Diaz et al.<sup>14)</sup> regarding the fatigue properties of quenched and tempered CrNiMo steel similar to that employed by Garces et al. coated with an EN deposit with P content of approximately 10 wt.% and thickness of 18µm, indicated that in the as-deposited condition, the reduction in fatigue life could reach up to 94% in comparison with the substrate, and the fatigue limit can also be reduced to 28-51%. Also it is concluded that the EN coating acts as a stress concentrator on the surface of the base steel which leads to the decrease in the time required for the nucleation of the fatigue cracks.

In the current work, an investigation has been conducted in order to accomplish two different objectives: (a) to study the effect of the electroless Ni-Cu-P deposit on the fatigue behavior of the CK45 steel and (b) to determine the effect of shot peening on fatigue life of base steel coated with this new kind of coating.

### Experimental procedure

The present investigation was carried out on samples of quenched and tempered CK45 steel with chemical composition given in Table 1.

Table 1. Chemical analysis of the samples (wt. %).

Element	C	Mn	P	S	Si	Fe
CK45 steel	0.43	0.35	0.02	0.02	0.15	balance

The material was provided as bars of approximately 13 mm diameter and 6 m length. Such bars were cut to pieces of different lengths: 9 pieces of approximately 80 mm for machining tensile specimens, 54 pieces of approximately 60 mm for machining the fatigue samples and some complementary samples of approximately 10 mm for characterizing the deposit, and to conduct hardness tests.

The tensile specimens had a gage diameter of 6.25 mm, a shoulder diameter of 10 mm, a gage length of 25 mm and a fillet radius of 5 mm according to the ASTM standard A-370. The fatigue specimens had a gage diameter of 5 mm, shoulder diameter of 8 mm and machined following a continuous radius of 35 mm according to the DIN 50 113 standard. All the samples were heat treated at 850°C for 15 min and oil-quenched from that temperature and then tempered at 550°C for 1 h in an electrical salt bath furnace.

In order to determine the effect of shot peening prior to coating deposition on the fatigue properties of coated substrate, at least 18 fatigue specimens and 3 tensile specimens were shot peened for 5 min in an air pressure type of equipment using

S460 type cast-steel shots according to ASTM standard B-851. All specimens were rotated at 20rpm during peening operation and the peening intensity at air pressure of 30 psi was 0.3 mm (Almen A). Subsequently, the samples were cleaned with acetone for 5 min, degreased in a 150 g/l NaOH solution at 70°C for 15 min, rinsed in distilled water, deoxidized with a solution of HCl 30% for 3 min and again rinsed in distilled water. The surface of the samples was finally activated with a solution of HCl 20% at 50°C for 30 s, rinsed in distilled water and submerged in the deposition solution. The chemical composition of the bath consisted of 27.5 g/l nickel sulphate, 1.2 g/l copper sulphate, 30 g/l sodium hypophosphite, 20 g/l sodium acetate and 55g/l Sodium citrate. The deposition was conducted at a pH of 9 at 90°C. The bath was continuously agitated and deposition time of 1 h was employed.

Microhardness measurements were carried out on the cross section of the samples employed for this purpose on each specimen according to the ASTM standard E-384. Such measurements were done employing Knoop indenter with a load of 50 g applied during 10 s.

A computer-controlled servohydraulic machine (Adamel Dy-26) was used to evaluate the static mechanical properties of the uncoated and coated samples, employing a constant cross head speed of 0.1 mm/s. Such a characterization required the testing of at least three samples for each condition.

Rotating bending fatigue tests were conducted using a sinusoidal load at a frequency of 55 Hz and load ratio of R= -1, employing a Roell Amseler (UBM 2000) equipment, at room temperature.

Three groups of fatigue tests were prepared to obtain S-N curves for rotating bending fatigue tests as follows:

Smooth specimens of base metal;

Specimens of base metal coated with an electroless Ni-Cu-P deposit;

Specimens of shot peened base metal coated with an electroless Ni-Cu-P plating.

A total of 18 samples were employed in each case, which satisfied the number of specimens required in S-N testing for reliability data according to ASTM standard 739 (12-24 samples). SEM techniques (Camscan MV-2300) with EDS facilities were employed both for determining the chemical analysis of the plating and for examining the fracture surfaces of the samples.

### Results and discussion

#### Characteristics of the deposit

Figure 1 illustrates the typical microstructure of the substrate evaluated with the scanning electron microscope. The presence of a large number of martensite plates can be observed. Such a structure had a mean hardness of 33.5 HRC. Figure 2 shows a

view of the interface between the electroless coating and substrate after fatigue failure at 400 MPa.

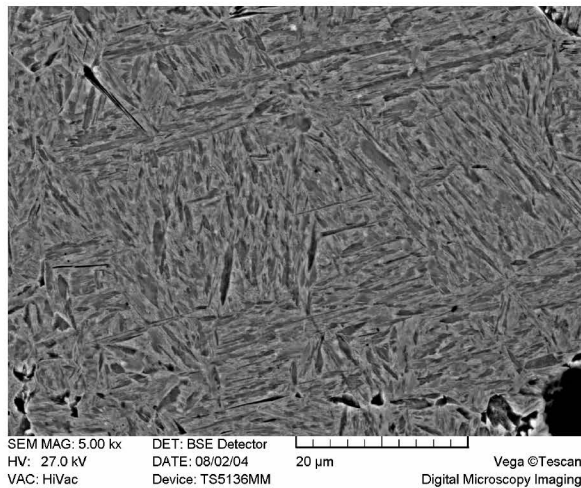


Fig. 1. Typical microstructure of the substrate (Quenched and tempered CK45 steel at low magnification).

In general, it would be observed that the adhesion of the deposit to the substrate was quite good, a fact that was confirmed with the fatigue experiments. Even after application of a very large number of cycles under complete reverse tensile-compressive condition, the deposit remained well adhered to the substrate. It can also be appreciated that an important feature commonly observed, namely the presence of an ‘Orange-peel’ effect on the lateral surface of the specimen, is believed to be associated with trapped hydrogen in the deposit.

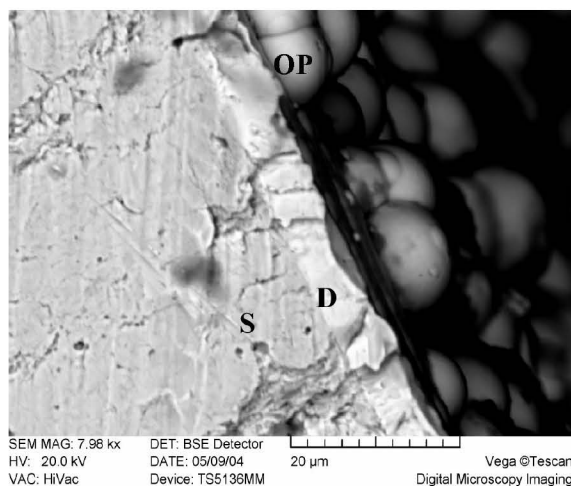


Fig. 2. SEM view of the interface between the electroless Ni-Cu-P coating (D) and substrate (S) after fatigue failure at 400 MPa. The deposit seems to be uniform and has apparently satisfactory adhesion characteristics due to the absence of visible cracks along the interface. ‘Orange-peeling’ type effects (OP) are also observed at the lateral surface of the sample.

It is a well-known fact that during EN plating, a certain amount of hydrogen is produced by reduction and evolves at the surface of the part being plated<sup>15</sup>.

For all samples the thickness of the deposit was determined to be about 7µm with homogeneous thickness throughout the entire section as shown in Figure 2. The EDS analyses conducted on the deposit allowed determining the Cu and P contents of 6.4 wt.% and 13.7 wt. %, respectively. As-deposited samples displayed a mean Knoop hardness of approximately 480 KHN.

#### Evaluation of mechanical properties

The monotonic tensile tests conducted on the samples including the base metal, the coated specimens and the specimens of shot peened base metal in the as-deposited condition revealed that the main mechanical strength parameters did not show a significant variation from one condition to another (Table 2). Thus, it can be stated that the deposits plated onto the substrate employed in the present study did not give rise to any significant change either in yield stress or UTS of the base metal, which is not surprising since, in the present case, the thickness of such deposits is too small to have a considerable effect.

Table 2. Mean tensile properties of the material in all conditions of present work.

Condition	$\sigma_y$ (MPa)	UTS (MPa)
substrate	912	1125
as-deposited	908	1083
shot peened substrate in the as-deposited condition	920	1132

As far as fatigue testing is concerned, the evaluation of the monotonic mechanical properties of the material allowed determining a stress amplitude range of 480-680 MPa for the uncoated specimens, that is to say, approximately  $0.5-0.75\sigma_y$ . The coated samples in the as-deposited condition were tested at stresses in the range of 360-560 MPa ( $0.4-0.6\sigma_y$ ), whereas the shot peened and coated specimens were tested at stresses in the range of 440-640 MPa ( $0.5-0.7\sigma_y$ ).

Tables 3-5 summarize the number of cycles prior to fracture ( $N_f$ ) as a function of the alternating stress applied to the material (S) for all conditions investigated.

Figure 3 illustrates the obtained results in the well-known S-N diagram. It clearly indicates that the plating of electroless deposit on the CK45 steel gives rise to significant reduction in fatigue life. This result agrees with previous work conducted with high strength steel substrates coated with different types of EN deposits<sup>15</sup>.

Table 3. Mean number of cycles to failure ( $N_f$ ) vs. stress amplitude ( $S$ ) for the uncoated specimens.

S (MPa)	480*	520	560	600	640	680
$N_f$	No fracture	167600	89300	43350	32890	26220
	No fracture	251200	118700	40610	44388	17248
	No fracture	196700	95400	63320	42506	17394

\* Non-fractured samples tolerated  $10^7$  cycles.

Table 4. Mean number of cycles to failure ( $N_f$ ) vs. stress amplitude ( $S$ ) for the coated specimens in the as-deposited condition.

S (MPa)	360*	400	440	480	520	560
$N_f$	No fracture	119800	67372	62300	47979	35952
	No fracture	151500	91790	59900	75989	33667
	No fracture	132400	75810	64200	42480	27950

\* Non-fractured samples tolerated  $10^7$  cycles.

Table 5. Mean number of cycles to failure ( $N_f$ ) vs. stress amplitude ( $S$ ) for the shot peened substrates in the as-deposited condition.

S (MPa)	440*	480	520	560	600	640
$N_f$	No fracture	186750	119913	71650	55299	35119
	No fracture	304980	74549	74549	50821	32703
	No fracture	261300	88478	71589	49140	33283

\* Non-fractured samples tolerated  $10^7$  cycles.

Figure 3 also shows that a shot peened CK45 steel as substrate, which is coated with an electroless Ni-Cu-P deposit, has much better fatigue behavior than that of samples without shot peening prior to coating. Thus, shot peening can be considered as an alternative to fatigue life improvement of CK45 steel coated with such a deposit.

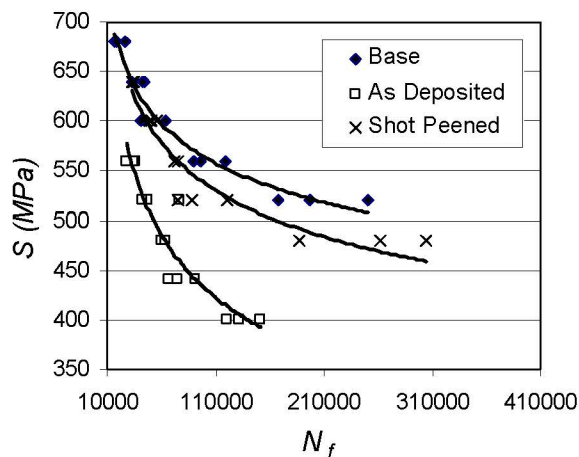


Fig. 3.  $S$ - $N$  comparative curves of all cases analyzed in this study.

Another important aspect that can be pointed out from Figure 3 is that at low stresses the reduction in fatigue life for both conditions is much more significant with the trend of decreasing as the applied stress increases. This may be attributed to the effect of the coated layer on the fatigue crack nucleation since, as it will be discussed, the coated layer acts as a stress concentrator with respect to the base steel, especially at low stresses.

#### Evaluation of the fracture surface of the samples

Several specimens tested at different alternating stresses were examined after failure by SEM in order to study more closely the sites of crack initiation and to analyze the microstructural changes that take place both in the coating and substrate during the subsequent propagation of such cracks. For example, the photomicrograph of Figure 4(a) illustrates the general fracture surface of a coated sample tested at 400 MPa.

On the other hand, Figure 4(b) illustrates a closer view of crack initiation site where it can be observed that the fatigue crack initiated at the deposit and propagated toward the substrate, and in general, after fracture the coating remains well adhered to the substrate. However, secondary cracks (SC) which

develop along the coating-substrate interface (Figure 5) can be rarely observed.

In relation to the samples which were shot peened before plating, Figure 6(a) represents a typical fracture surface of the specimen tested at 480 MPa, and Figure 6(b) shows that the metallic bonding between deposit and substrate is poor because complete separation of deposit and substrate has occurred.

Finally, Figure 7 shows fatigue crack nucleation sites in two different samples, which were shot peened prior to the coating deposition, and tested at 520 and 600 MPa. These figures clearly show that the crack initiation sites were displaced to the coating-substrate interface, and there is no fatigue failure feature on the deposit.

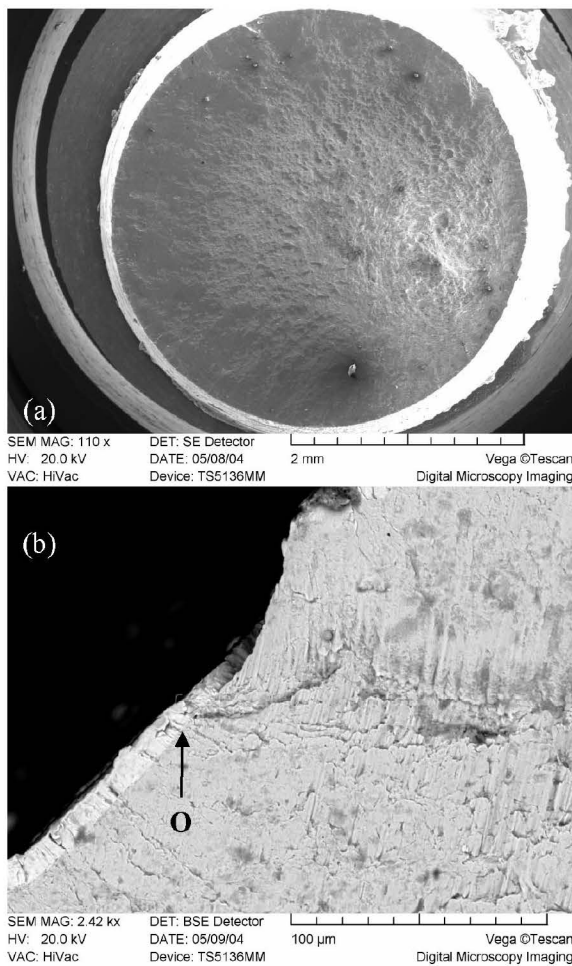


Fig. 4. (a) General fracture surface of a coated sample tested at 400 MPa. The transition between the areas where the crack propagated by fatigue (smooth) and the area where the specimen failed by overloading (rough) is visible. (b) Detailed view of the initiation site of the crack, showing the origin (O) of the main crack.

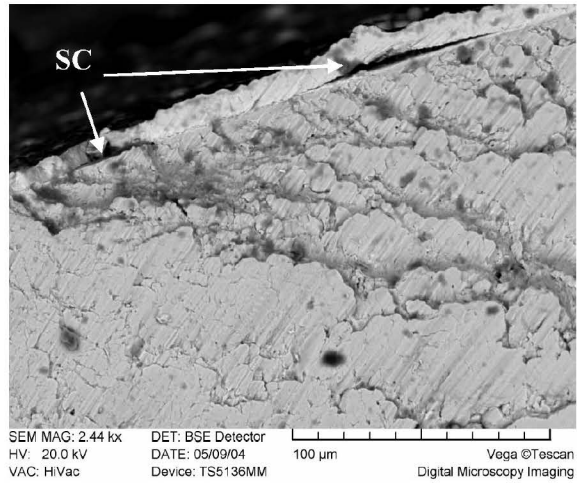


Fig. 5. Secondary cracks (SC) are observed to run parallel to the substrate-deposit interface.

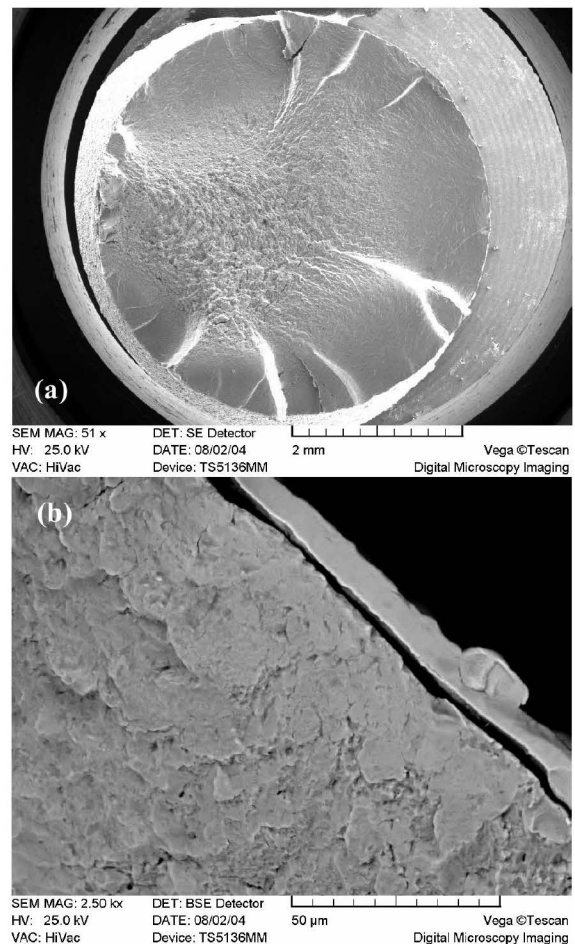


Fig. 6. (a) General fracture surface of a specimen in the shot peened condition, tested at 480 MPa. (b) Detailed description of the interface which indicates complete separation of the deposit from substrate and poor adhesion characteristics.



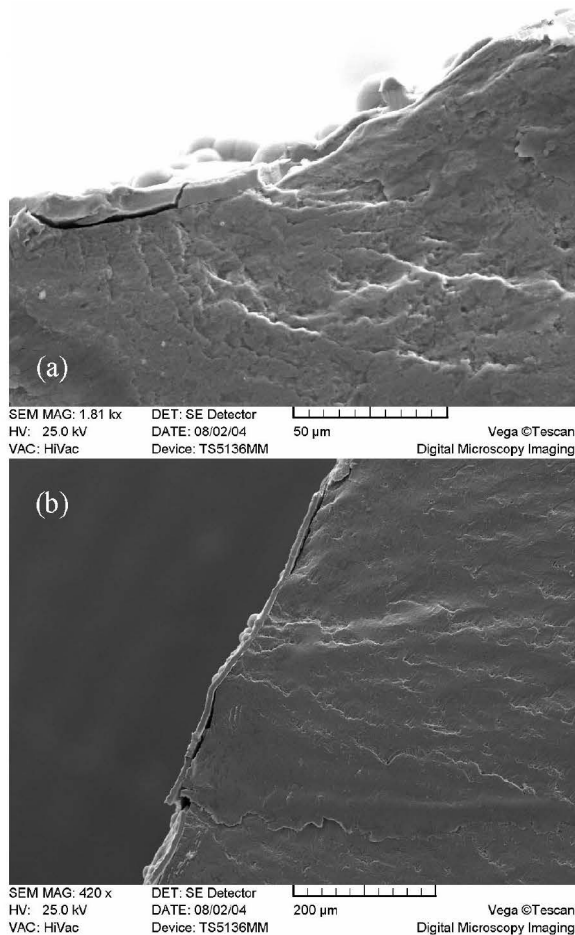


Fig. 7. Fatigue crack nucleation sites in two different samples, tested at (a) 520 and (b) 600 MPa.

### Conclusions

- 1- The fatigue strength of quenched and tempered CK45 steel is severely decreased by plating it with an electroless Ni-Cu-P deposit, even of 7µm thickness.
- 2- It is believed that the decrease in the fatigue strength is due to fatigue cracks initiation within the deposit and their propagation towards the substrate.
- 3- The reduction in the fatigue strength was found to be less marked for shot peened samples in the as-deposited condition. It seems that such

behavior is a result of fatigue crack nucleation on the interface.

- 4- Shot peening can be considered as an alternative to fatigue life improvement in CK45 steel coated with an electroless Ni-Cu-P.

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