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Effect of Nickel-Niobium Coating on Fatigue Resistance of SAE 1020 Carbon Steel

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***Abstract* - Effect of nickel-niobium coating by electrodeposition on the fatigue resistance of SAE 1020 carbon steel is studied in this work. The nickel-niobium coating was performed using a nickel Watts bath with added niobium powder. The specimens for fatigue tests were taken from SAE 1020 steel bar with 7.94mm (5/16 '') diameter. The fatigue tests were carried out by rotary bending load control with loading balance. It employed a rotation speed of approximately 1500rpm (25Hz). Vickers microhardness tests were performed on steel and deposited layer of nickel-niobium. Analysis of the electroplated layer connection interface in the steel and fatigue fractures were performed by scanning electron microscopy (SEM). The fatigue tests were performed in order to obtain S-N curves (Wöhler curves). It was observed that nickel-niobium coating gave only a tendency to increase the fatigue strength of the steel, although the coating layer has shown high hardness compared to the steel. The detachment of the deposited nickel-niobium layer was regarded as the main cause of non-contribution of the increase in fatigue limit coated steel compared to the same in its original condition.**

Keywords: Fatigue, Wöhler curve, nickel-niobium electroplating.

1. INTRODUCTION

The SAE 1020 steel is a low-cost steel that exhibits excellent plasticity and weldability, but its mechanical strength is low due to the high ductility. This steel has applications in mechanical components such as gears, shafts, guide pins, bolts, wheel discs and general parts for machines and equipment that have moderate mechanical stress. With the increase in the surface hardness of this steel, it can achieve a desirable balance between a high strength surface layer and a soft and ductile core [1,2]. To this, are usually employed nitriding and carburizing thermochemical treatments, for example. Coatings by electrodeposition with harder elements like nickel, zinc and chromium, among others, are also employed to increase the surface strength of steel, and improving its corrosion resistance properties [3]. The main mechanism of fatigue crack nucleation is associated with persistente slip bands (PSB) that occur on the surface of parts under cyclic loading [4]. Increase in surface hardness of steel parts dificult the process of PSB formation, increasing with this the fatigue limit [5].

The nickel electroplating process occurs using a soluble anode (nickel) and one cathode that is the material that desired cover. These two electrodes are immersed in a solution containing nickel salts and a flow of direct current between them causes the nickel anode dissolves, transferring its ions to the cathode, producing the nickel coating. The divalent nickel ions (Ni^{+2}) in the solution are converted to metallic nickel (Ni^0) on the surface of the cathode. Several nickel salts bath are used to electroplating this element, with most of them based on nickel chloride and / or nickel sulfates [6].

The nickel salt bath more commercially used is the Watts bath. This bath is composed of nickel sulfate, nickel chloride and boric acid. The nickel sulfate is its main ingredient, being the main source of nickel ions due to its high solubility. The presence of chloride in the salt bath aids in the anode corrosion and increases the diffusion coefficient of nickel ions. Boric acid is used as a buffering agent in Watts bath in order to control its pH. The presence of boric acid in the nickel bath solution inhibits the formation of bubbles insoluble $Ni\ (OH)_2$. These bubbles disturb homogeneous deposition of nickel on the substrate surface and affect the pH of the solution [7-8].

The nickel coatings obtained from Watts bath, present from 20% to 30% elongation, hardness between 200HV and 150HV, tensile strength 380MPa to 450MPa and yield stress 220MPa to 280MPa. Experimental data indicate that the hardness of the electroplated nickel layer increases with a higher concentration of nickel ions in the bath and current densities below $10A/dm^2$. For best performance of the electroplating process, the bath pH should be maintained at around 3.5 and temperature approximately 60°C [7].

Niobium particles can be electrodeposited together with the nickel when added to the nickel Watts bath. This technique is called coelectrodeposition (CED). In this case, the nickel bath must contain from 20 to 40g/l of niobium powder. The optimum temperature and pH for greater efficiency of coelectrodeposition nickel-niobium are considered the same for conventional nickel Watts bath, at around 60°C and 3.5, respectively. The electroplated layer of nickel-niobium has a higher corrosion resistance and hardness compared to the pure nickel layer [9].

The method of coelectrodeposition has presented more advantageous compared to other coating methods, due to greater ease in continuous processing, their use in parts with complex geometry, no application of high pressure, productivity in processing, homogeneity of layer deposited and low residue generation during processing [10-11]. Coelectrodeposition is an effective method for producing composite metal coatings containing metallic and non metallic particles to provide gains in tribological properties and corrosion resistance [12]. The quality of the coating by coelectrodeposition depends on the amount, size and distribution of particles added to the electrochemical bath [13]. The surface morphology of the coating by electro-codeposition is affected by the electroplating parameters, such as bath composition, current density, temperature, bath pH, and stirring rate of the bath [14]. The addition of very fine particles in coelectrodeposição process contributes to the formation of agglomerations in the coating, which may cause impairment in coating properties. The homogeneity of size and shape of the particles also contribute to agglomeration coating [15].

In this study, cylindrical specimens of SAE 1020 steel bars, diameter of 5/16 inch, with and without electroplated layer of nickel-niobium, were subjected to fatigue tests (load control) by rotary bending with loading balance. The aim of this study was to verify the gain in fatigue resistance in SAE 1020 steel with electroplated layer of nickel-niobium in order to verify the viability of its application in cyclic loading situations.

2. EXPERIMENTAL PROCEDURES

The SAE 1020 steel used in this study was obtained in the form of a circular cold-rolled bar, 5/16 inch diameter. The typical chemical composition of the steel is given in Table 1. In electrodeposition was used niobium metal powder having a purity of 99.8% in wt% with mean particle size around 70µm.

Table 1. Typical chemical composition of the steel studied(wt%).

C	Si	Mn	S_{max}	P_{max}
0,2	0,25	0,45	0,04	0,04

The steel tensile strength properties were obtained from the specimen according to the standard ASTM E-8 [17]. Tensile tests were performed at a rate of 5mm/min on a EMIC universal testing machine, WDW-100E model. The fatigue tests were carried out with charge control on a EDIBON fatigue rotating bending machine, EEFC model, with speed of 1500rpm (25Hz). The specimens for fatigue tests were prepared with continuous radius of 50mm, as shown in Figure 1. In all mechanical tests (tensile and fatigue), three specimens were used for each situation.

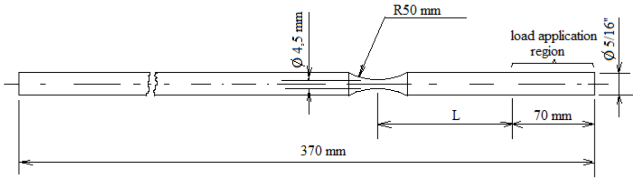

Figure 1. Specimen for fatigue test.

As shown in Figure 1, the considered fatigue stresses had their change depending on the size "L", because during the tests was used a single loading value equal to 30N. The maximum stress (σ) applied in the critical region of fatigue was determined by the Equation 1[16].

$$\sigma = \frac{P \, . \, L}{0{,}1 \, . \, d^3} \qquad (1)$$

(P - applied load; L - distance from the critical region of fatigue; d - critical diameter of fatigue)

Figure 2 shows the mounting of the specimen charging system for the production of cyclic stress rotary bending fatigue. As can be seen, the specimen was made in order to obtain the maximum stress (σ) at its point of smallest diameter (diameter d).

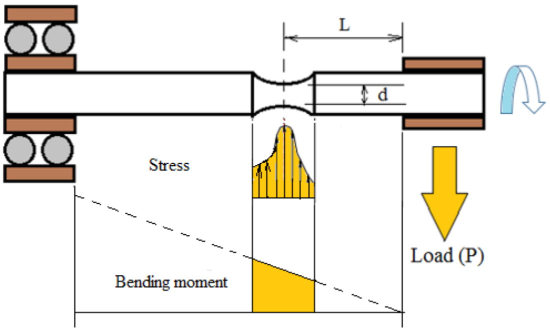

Figure 2. Assembly for fatigue test.

The nickel-niobium electrodeposition was performed using the nickel Watts bath with addition of 40g/l of niobium particles with an average size of 70µm [8]. Table 2 shows the nickel Watts bath composition. The electrodeposition was applied at 3 hours with the bath at 60°C and pH 3.5, using a INSTUTHERM DC POWER SUPPLY, FA-3030 model and a magnetic stirrer BIOMIXER, 78HW-1 model. A nickel electrode was used as anode and SAE 1020 steel substrate (specimen) was used as a cathode. Was applied current of 3.9mA/cm^2, voltage of 2.7V and stirring of the bath at 600rpm [9]. The nickel-niobium coating was performed only in the usable area of the specimen (continuous radius area). The surface of the specimen (SAE 1020 steel) was milled and washed with 0.5% solution of mild soap before the electrodeposition process [17].

Table 2. Nickel Watts bath composition [7].

Reagent	Purity (%)	Concentration (M)	Mass (g)
$NiSO_4$	98	0,22	118,02
H_2BO_3	99,5	0,13	33
$ZnSO_4$	96	0,55	330
$NiCl_2$	98	0,33	106,21
Sorbitol	70	0,39	134,0 (90ml)

The incorporation of Zn from nickel Watts bath ($ZnSO_4$) in the nickel deposit leads to a sensible grain refinement for all the deposits containing Zn up to about 7 wt.%. The addition of Zn^{2+} to the deposition bath leads to a strong decrease in the cathodic current density (indicating a remarkable inhibition of Ni reduction) and the incorporation of Zn in the face centered cubic Ni lattice. Presence of nanocrystalline Ni–Zn β-phase has been found in the alloys having Zn content higher than about 8 wt.% Zn [18].

Vickers hardness measurements were made in SAE 1020 steel with and without nickel-niobium coating. For the tests we used a microhardness Wilson Instruments, 402MVD model. For the study of the morphology of the nickel-niobium coating and fatigue fractures was used a VEGA S3 TESCAN scanning electron microscopy (SEM). The chemical composition of the coating was analyzed by dispersive X-ray spectroscopy (EDX) coupled to the SEM.

3. RESULTS AND DISCUSSION

Table 3 shows the average results obtained in the tensile tests performed on three specimens of the SAE 1020 steel.

Table 3. SAE 1020 steel tensile strength.

	σ_{UTS} (MPa)	σ_{YS} (MPa)
Average	360	241
Standard deviation	26	35

σ_{UTS}: ultimate tensile stress; σ_{YS}: yeld strength.

From five points measurement (D1 to D5) on the coating layer was obtained a thickness around 120 ± 19µm, as shown in Figure 3. It can be even observed the detachment of nickel-niobium layer in the region near the fatigue fracture.

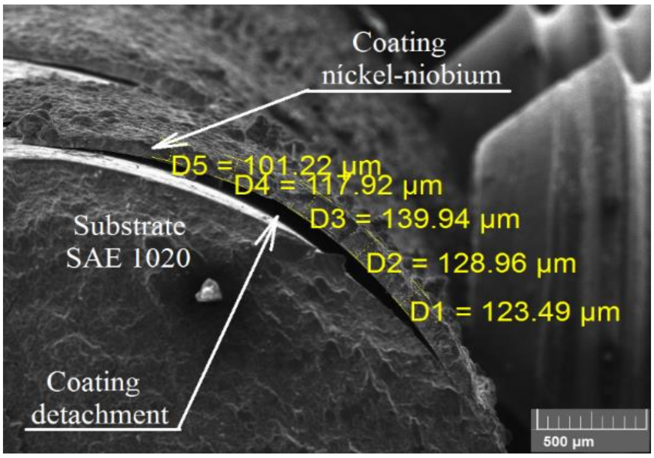

Figure 3. Detachment nickel-niobium coating on SAE 1020 steel (SEM).

The presence of nickel and niobium in the coating layer is confirmed by EDX qualitative analysis as shown in Figure 4.

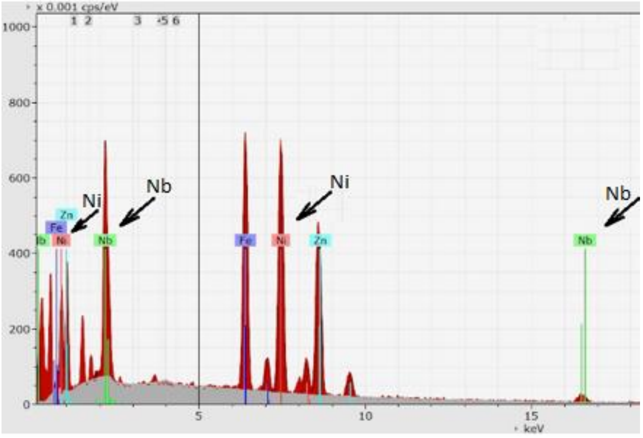

Figure 4. EDX qualitative analysis of nickel-niobium coating.

Zn present in the coating, as shown in Figure 4, comes from the Watts bath ($ZnSO_4$). Zn has the grain refining function of the coating and decreasing the current density necessary for the electrodeposition [18].

Figure 5 shows nickel-niobium coating image obtained on SEM after electroplating process and in Table 4 the chemical composition performed by EDX.

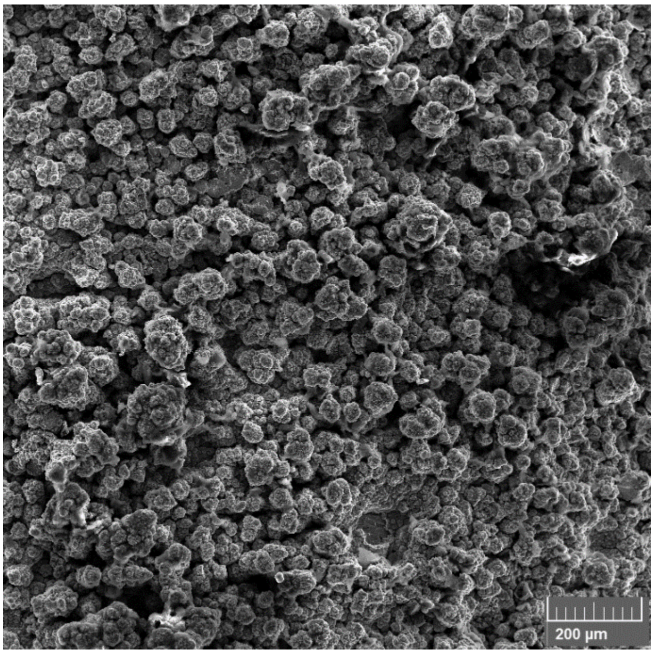

Figure 5. Morphology of nickel-niobium coating on SAE 1020 steel (SEM).

The surface morphology of the coating appears to be quite homogeneous, with the absence of differentiated agglomeration regions. According to Fratari and Robin [8], the bath stirring rate and current density are directly related to this aspect of the morphology of the coating. Also according to Fratari and Robin [8], nickel-niobium coatings obtained with current below 20mA/cm^2 and agitation of the bath above 550rpm show a finer and smoother surface morphology. As explained, the reason of having obtained a morphology considered without agglomerations on the surface of nickel-niobium coating, Figure 5, may be related to the application of low current (3.9mA/cm^2) and high agitation bath (600rpm).

The surface of the nickel-niobium coating could have been more refined and more homogeneous if the particle size of niobium were added to the bath between 20µm and 50µm [8]. In this work, the particles of niobium added to the bath have an average size about 70µm.

As shown in Figure 5, the coating surface had a rough morphology comprised of nodular clusters caused by the presence of the microparticles of niobium present in the coating. Coating compound only nickel tend to be less rough. The characteristics observed in nickel-niobium coatings in this work, are very similar morphologically to the results presented by Robin and Fratari [19]. They confirm that the increase in surface roughness and any formation of clusters on the surface of the coatings is due to the presence of niobium particles in the electrolytic bath.

The nickel-niobium electroplated layer, before carrying out the fatigue tests, showed, by analogy, good adhesion to the substrate. Tape test was performed on specimen of the same substrate (steel SAE 1020) with a flat surface submitted to similar electrodeposition process. However, as can be seen in fatigue fracture shown in Figure 6 (dashed circles), there is a marked detachment of the coating in the region where the fracture began.

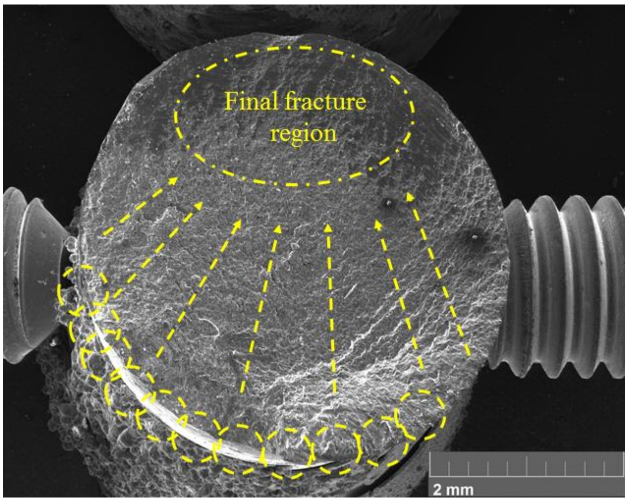

Figure 6. Fatigue microphotography of the SAE 1020 steel coated with nickel-niobium (SEM).

The high mechanical strength difference between the substrate (SAE 1020 steel) and nickel-niobium covering layer, associated to severe cyclic fatigue loading application, can be considered as a major cause of the coating layer detachment [20-23]. In this case, the great ductility of the substrate, compared to the coating, tends to induce a high load transfer to the coating layer causing its detachment. The high hardness and low deformation capacity of the covering layer induce their buckling due to compression and tensile alternative loading. The induction of this cyclical buckling produces a high detachment stress of covering layer of the substrate.

As can be seen in Figure 6, the path of propagation of the fatigue fracture (indicated by arrows) had its origin in the region of the specimen surface where there was a detachment of nickel-niobium coating layer. Probably, with the detachment of the covering layer (hardest) has facilitated the occurrence of persistent slip bands (PSB) on the exposed surface of SAE 1020 steel (softer) and thus the nucleation and propagation of fatigue fracture [21-23].

Four measurements Vickers microhardness were performed on cross section of substrate (SAE 1020 steel) and nickel-niobium electroplated layer having been obtained respectively the following results: 194 ± 6HV and 499 ± 33HV. The microhardness tests were carried out with application of 0.5kgf, 10s. The significant increase in the surface hardness of the steel coated with nickel-niobium is in agreement with the literature [8].

Figure 7 shows the results obtained in fatigue tests of the SAE 1020 steel with and without nickel-niobium electroplated coating.

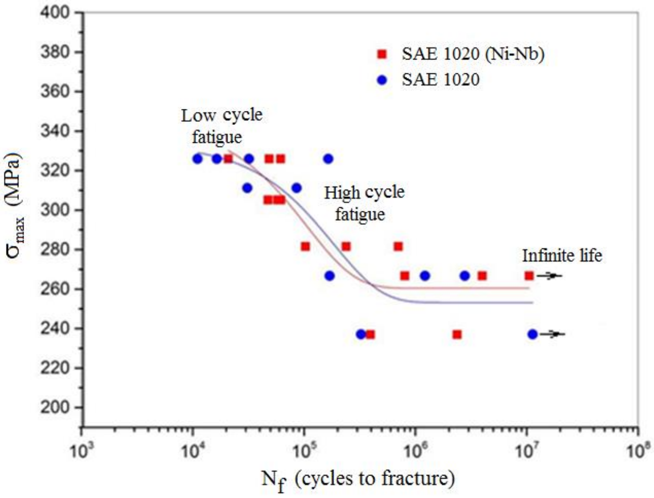

Figure 7. SAE 1020 steel fatigue curves with and without nickel-niobium coating.

Fatigue tests were conducted on specimens subjected to rotating bending with load control in balance, as shown in Figure 2. The curves with exponential decay, own of steel fatigue, reveal different regions behavior in fatigue of the material: low fatigue cycle (fatigue fracture with high stress), high fatigue cycle (fatigue fracture with average levels of stress) and "infinite life" (stress level in which no fracture 10^7 cycles). Although the coating of SAE 1020 steel with nickel-niobium has significantly increased the surface hardness of the material, as already mentioned, there was no improvement in fatigue resistance compared to steel without the coating. This may be attributed to the detachment of the covering layer, as discussed earlier and shown in Figure 6. Thus, it can be said that the adhesion of the electroplated layer, in addition to its hardness, it is also critical to improving the fatigue strength.

3. CONCLUSIONS

- The nickel-niobium coating showed considerable increase in hardness in surface of the SAE 1020 steel used as substrate;
- there was no improvement in fatigue resistance of the steel SAE 1020 with nickel-niobium coating compared to steel without the coating due to detachment of the coating layer;
- the detachment of the covering layer (hardest) has facilitated the occurrence of PSB on the exposed surface of SAE 1020 steel (softer) and thus the nucleation and propagation of fatigue fracture;
- the development of electroplating techniques that lead to greater adherence of the deposited layer will certainly contribute to increased fatigue resistance of the SAE 1020 steel.

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