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# Microstructural Evaluation of Salt Nitrocarburizing H13 Steel as a Function of Time

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**Abstract.** A set of H13 steels were nitrocarburized with the aim of improving their performance as matrices and pins. The nitrocarburizing process was performed in an ETSA liquid salt bath furnace at 570°C for 45, 60, 90, 120, 150 and 180 minutes in order to evaluate the effect of time during the nitrocarburizing treatment on the quality of the nitrocarburizing layer. Microstructural evolution of the compound and diffusion layers were studied by scanning electron microscopy and by X-ray diffraction analysis. From microstructural characterization, it was observed mainly a continuous nitrocarburizing  $\epsilon$ -Fe<sub>2-3</sub>(C,N) layer with nitrocarburizing times between 45 to 60 minutes showing better wear performance as compared with specimen with nitrocarburizing times between 90 to 180 minutes.

## 1. Introduction

Nitrocarburizing is a surface heat treatment employed to improve hardness and resistance to wear and fatigue [1] in which the elements nitrogen and carbon are diffused into the surface of steels, commonly in the temperature range of 520 to 580°C [2]. The nitrocarburized surface of steels present two different structures known as the compound layer and diffusion zone from surface to core respectively. The compound layer consists of  $\epsilon$ -Fe<sub>2-3</sub>(C,N) and  $\gamma'$ -Fe<sub>4</sub>(C,N). The wear and corrosion resistance is attributed to the presence of the  $\epsilon$ -Fe<sub>2-3</sub>(C,N) carbonitride [3], while the diffusion zone improves fatigue strength when compared to an untreated material. In this zone, N and C atoms are dissolved interstitially in the ferritic lattice and form nitride precipitates [4]. Regarding the tribological performance, the optimum compound layer will be that which consists of  $\epsilon$ -Fe<sub>2-3</sub>(C,N), although other phases can be present, mainly  $\gamma'$ -Fe<sub>4</sub>(C,N); from the nitrocarburizing methods [5], liquid nitrocarburizing is regarded as effective and low cost method with many advantages such as low treatment temperature, short treatment time, high degree of shape, dimensional stability and reproducibility.

This work present results of microstructural characterization, microhardness and wear resistance of liquid nitrocarburized H13 tool steel as a function of treatment time.

## 2. Experimental procedure

The chemical composition of the steel H13 under study is presented in Table 1. Previous to the nitrocarburizing treatment, the tool steel was austenized at 1015 °C for 180 minutes, quenched in high vacuum with nitrogen followed by two tempering temperatures at 540°C and 560°C for 180 minutes each one. The liquid nitrocarburizing process was performed in an ETSA liquid salt bath furnace (with 3% cyanide, 36.7% cyanates and 15.3% carbonates) at start temperature of 350°C, when the salt bath temperature reached 580°C, specimens were withdraw at 45, 60, 90, 120, 150 and 180 minutes of treatment time. Hardness Vickers measurements were performed in a microhardness tester Shimadzu



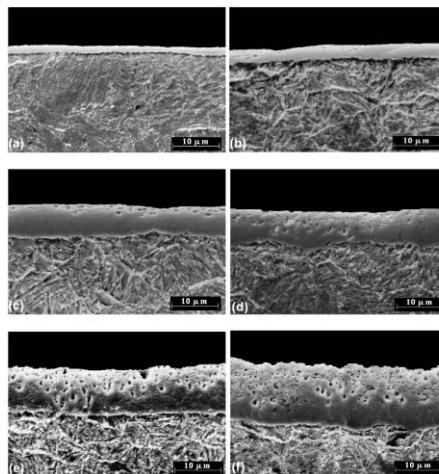
at a load of 0.1 mN for 15 sec. After grinding and polishing, the specimens were etched with 5% nital. Microstructural characterization was performed by using a Leica 440 scanning electron microscope operated a 20 kV and 3 A. The phase composition of layer was determined using a Siemens 5000 diffractometer at a scanning rate of 1 deg/min in characteristic  $K\alpha$  Co-radiation and the wear test was performed on pin-on-disk tribometer according with ASTM standard G-99 under a normal load of 2 N of load with 0.1 m/s of speed and  $1 \times 10^4$  cycles with track radius of 10 and 8 mm. The pin used was of Cr6-steel ball with diameter of  $\frac{1}{4}$  in, in dry condition at atmospherically pressure and room temperature.

**Table 1. Chemical composition of steel H13 (in wt. %).**

C	Si	Mn	V	Cr	Mo	Fe
0.40	0.97	0.41	0.98	5.60	1.38	Bal.

### 3. Results and Discussion

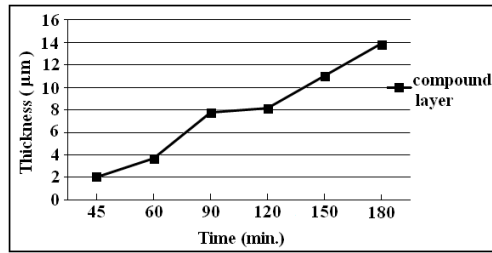
Figure 1 shows the evolution of the compound layer after liquid nitrocarburizing of tool steel H13 as a function of time, for instance, it is clearly seen that there is an increase in thickness as the nitrocarburizing treatment time increased. This compound layer shows mainly the presence of the  $\epsilon$ - $\text{Fe}_{2-3}(\text{C},\text{N})$  at 45 and 60 minutes of treatment time, and as the time increase, it was observed the presence of  $\gamma'$ - $\text{Fe}_4(\text{C},\text{N})$  associated with porosity, as is observed in figures 1e and 1f.



**Figure 1.** SEM micrographs of the compound layer after nitrocarburizing treatment: (a) 45, (b) 60, (c) 90, (d) 120, (e) 150 and (f) 180 minutes of treatment time.

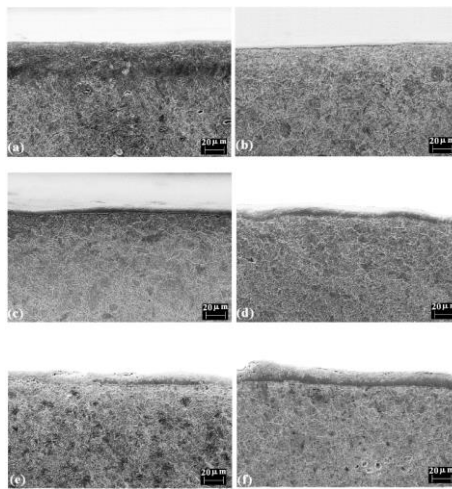
As reported in [6], the  $\epsilon$ - $\text{Fe}_{2-3}(\text{C},\text{N})$  carbonitride shows very good wear and corrosion resistance, compared to an untreated steel, therefore it is expected a better wear performance for nitrocarburizing specimen when the amount of  $\gamma'$ - $\text{Fe}_4(\text{C},\text{N})$  is lower and there is no presence of porosity as is observed mainly in specimens with treatment time with 150 and 180 minutes.

Regarding thickness increment of the compound layer as a function of nitrocarburizing time has been attributed to the diffusion mechanism that controls growth rate of this layer [7, 8], quantifying  $\sim 2 \mu\text{m}$  in thickness for the lowest nitrocarburizing time and  $\sim 14 \mu\text{m}$  for the longest as is shown in figure 2.

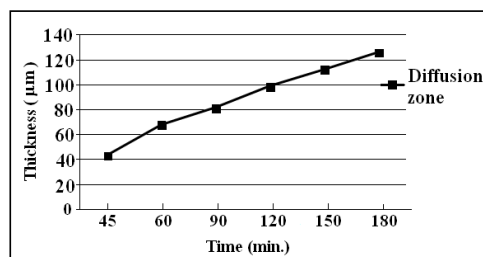


**Figure 2.** Thickness of compound layer as a function of time.

The microstructure of the diffusion zone is shown in figure 3 and its thickness is plot in figure 4, where a thickness of  $\sim 43 \mu\text{m}$  was measured for the shortest nitrocarburizing time and  $\sim 126 \mu\text{m}$  for the longest. As is shown in figure 4, the diffusion zone depth followed a lineal path.

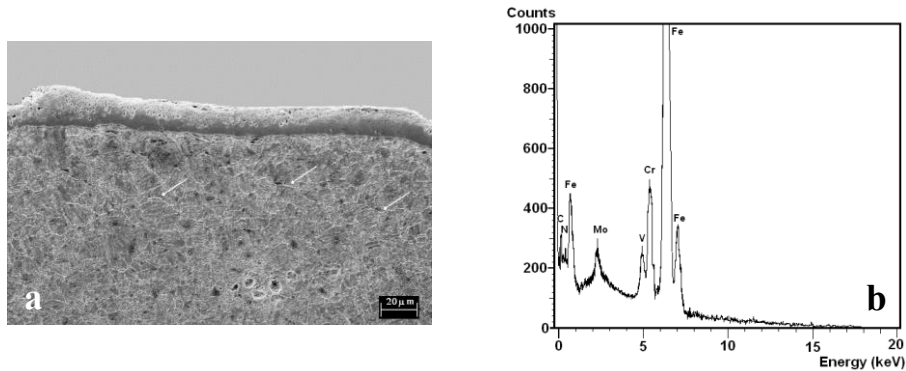


**Figure 3.** SEM micrographs of the compound layer and diffusion zone after nitrocarburizing: (a) 45, (b) 60, (c) 90, (d) 120, (e) 150 and (f) 180 minutes.



**Figure 4.** Thickness of diffusion zone as a function of time.

Examination of the microstructure of the diffusion zone reveals in the uppermost region, beneath the compound layer, the presence of precipitates at grain boundaries which were identified by SEM-microanalysis (see figure 5) as (Cr,V,Mo) (C, N), being chromium the main alloying element in these precipitates, because chromium has relatively a strong interaction with nitrogen during the nitrocarburization process.



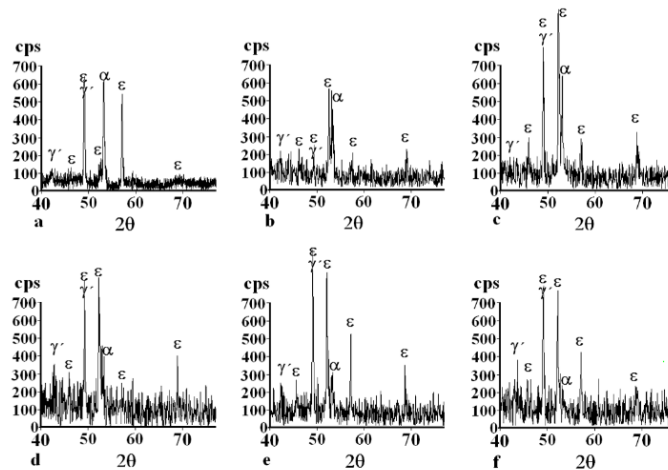
**Figure 5.** a) SEM micrograph showing by arrows the presence of precipitates at grain boundaries, b) SEM microanalysis of precipitates.

During the nitrocarburizing process, more nitrogen atoms than carbon atoms are introduced in the steel and the phase compositions found are more similar to the ones found in the Fe-N system than in the Fe-C system [9]. After ferritic nitrocarburizing the microstructure consists of three zones, the compound layer at the surface, the diffusion zone just below it and further the unaffected base material. The compound layer is typically 2–30 μm thick and consists of iron carbonitrides  $\epsilon$ -Fe<sub>2-3</sub>(C,N) and  $\gamma'$ -Fe<sub>4</sub>(C,N). The  $\epsilon$ -phase has a hexagonal crystal structure whereas  $\gamma'$ -phase is cubic [10]. The diffusion zone can reach 100 to 500 μm in thickness and consists of carbon and nitrogen in interstitial solid solution in the base material combined with small carbide and nitride precipitates.

The growth rate of the compound layer is governed by which phases it contains. In accordance with a isothermal section of the Fe-C-N phase diagram at 580 °C, the  $\gamma'$ -phase is a stoichiometric compound with a narrow solubility range for carbon and nitrogen. This makes the growth rate of  $\gamma'$ -phase relatively slow while the  $\epsilon$ -phase has a higher solubility range for both nitrogen and carbon and a high nitrogen mobility, which lets it grow quicker. For specimens treated for 90 to 180 minutes, it was observed that the outer part of the compound layer contains pores, that means that long process time increases the amount of porosity, this phenomenon occurs particularly at grain boundaries and other discontinuities [11].

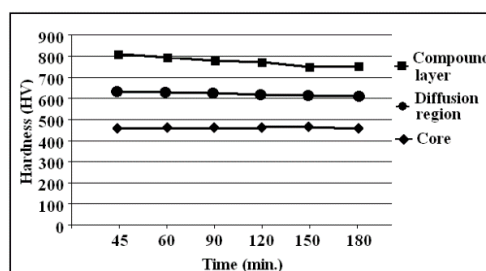
The results of phase analysis by X-ray diffraction for the H13 tool steel nitrocarburized from 45 to 180 minutes are shown in figure 6 where an important feature observed is the difference in the quantity of  $\epsilon$ -Fe<sub>2-3</sub>(C,N) and  $\gamma'$ -Fe<sub>4</sub>(C,N) phases. For instance when the steel is nitrocarburized with a holding time between 45 to 60 minutes, the main detected phase corresponded to the  $\epsilon$ -Fe<sub>2-3</sub>(C,N) and as the holding time increases, it is observed that the diffracted peaks for the  $\gamma'$ -Fe<sub>4</sub>(C,N) phase increased.

It has been reported in references [12, 13] that  $\epsilon$ -Fe<sub>2-3</sub>(C,N) phase is hard and brittle, while the  $\gamma'$ -Fe<sub>4</sub>(C,N) phase is hard and tough and that the usefulness of the formed compound layer depends on mainly two factors: 1) the homogeneity which can produce higher ductility of the structure of the compound layer and 2) its thickness. Therefore, the optimum properties will be obtained when compound layer is mono phased ( $\epsilon$ -Fe<sub>2-3</sub>(C,N) and  $\gamma'$ -Fe<sub>4</sub>(C,N) ) and has an optimum thickness, which is necessary to meet specific property requirements such as wear resistance.



**Figure 6.** X-ray diffraction patterns. (a) 45, (b) 60, (c) 90, (d) 120, (e) 150 and (f) 190 minutes.

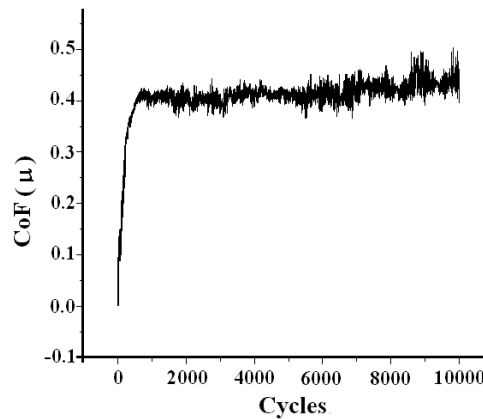
Microhardness distribution of compound layer, diffusion zone and core are shown in figure 7. As can be observed, the highest microhardness value of compound layer corresponded to specimens kept for 45 to 60 minutes in the nitrocarburized liquid bath. Diminution of microhardness as the nitrocarburized treatment time increased is attributed to the presence of both the  $\gamma'$ -Fe<sub>4</sub>(C,N) phase and porosity. The microhardness values of the compound layer for each nitrocarburized time has almost a constant hardness value throughout most of its cross section, however in the outer region of specimens kept for 90 to 180 minutes microhardness values lowered presumably by the presence porosity.



**Figure 7.** Hardness HV of compound layer and diffusion zone as a function of time.

These microhardness values can be related directly to the steel grade and its microhardness increment as the alloy content increases, for instance, the compound layer has been reported [14] to have a microhardness value of around 700 HV in low alloy steels and about 800 HV for steels with 1.5 wt% chromium content. Regarding the microhardness values of the diffusion zone follow the concentration gradients of nitrogen and carbon.

Disks of H13 tool steel with 60 minutes of nitrocarburizing time were tested in dry, sliding wear against a stationary ruby ball (pin) up to  $1 \times 10^4$  cycles as is shown in figure 8 where is shown the development of friction throughout the course of  $\sim 250$  m wear tests. An almost constant friction behavior was observed up to 5000 cycles afterwards is seen an increasing or decreasing friction behavior between the ruby pin and the nitrocarburized sample.



**Figure 8.** Friction coefficient of H13 tool steel nitrocarburized for 60 minutes.

Wear resistance with sample subjected to nitrocarburizing has been associated with the presence of the compound layer in the structure, although its thinner thickness, with the predominant  $\epsilon$ -Fe<sub>2-3</sub>(C,N) phase with high nitrogen content, preferably affecting the tribological properties [9] and other steel properties [10, 11].

#### 4. Conclusions

Liquid nitrocarburizing H13 tool steel from 45 to 60 minutes showed to be more effective than applying longer times, in terms of forming an almost uniform 2 to 4  $\mu\text{m}$  compound layer formed mainly of the  $\epsilon$ -Fe<sub>2-3</sub>(C,N) phase, which resulted in microhardness values close to 810 HV and a coefficient friction between 0.4 to 0.5. The increment on treatment time provokes an increment of the  $\gamma$ -Fe<sub>4</sub>(C,N) phase which is associated with the presence of porosity.

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