

## Microstructural Characterization of Nitro-Boriding Coating on ARMCO® Pure Iron

M. A. Flores-Rentería<sup>1</sup>, M. Ortiz-Domínguez<sup>1</sup>, I. Simón-Marmolejo<sup>1</sup>, L. E. Martínez-Martínez<sup>1</sup> and J. Zuno-Silva<sup>1</sup>

<sup>1</sup> Department of Mechanical Engineering, Universidad Autónoma del Estado de Hidalgo-Campus Sahagún, Hidalgo, México.

Corrosion, wear and fatigue involve chemical and/or mechanical interaction of the material component considered with loads imposed by the environment. Hence, materials performance and service life rely in many cases to a high degree on the properties of a material component in its surface region [1-2]. Nitriding on the surface of ferrous alloys results in the formation of a compound layer of  $\gamma'$ -Fe<sub>4</sub>N<sub>1-x</sub> and  $\epsilon$ -Fe<sub>3</sub>N nitrides or a mixture of  $\gamma'$  and  $\epsilon$  with a nitrogen diffusion zone beneath the nitride layer [3-5]. Likewise, the boriding is a thermochemical treatment in which boron atoms are diffused into the surface of a workpiece and form borides with the base metal, which is performed to increase the hardness, wear and corrosion resistance of these materials in engineering components for industrial applications that require those properties. In case of ferrous alloys, the boride layer may be either a single phase layer (Fe<sub>2</sub>B) or a double phase layer (FeB and Fe<sub>2</sub>B) depending on the boron activity in the boriding agent and also on the boriding parameters (time and temperature) depending on the boriding conditions [1,5]. In this study, the microstructure of a mixture of  $\gamma'$ -Fe<sub>4</sub>N<sub>1-x</sub>,  $\epsilon$ -Fe<sub>3</sub>N and Fe<sub>2</sub>B layers formed on an ARMCO pure iron surface have been investigated at different temperatures by the combination of two powder-pack processes.

Cubic commercial samples were cut from an ARMCO iron bar with composition: Mn, 800 ppm; C and P, 200 ppm; and S, 150 ppm. The substrate pure iron used in this work was selected to curb the effect of alloying elements in order to solely analyse the characteristic boride and nitride layers and some of their mechanical effects. The nitro-boriding treatment was carried out in two stages: nitriding and then boriding. Powder-pack nitriding and boriding procedures were preferred in this study for its cost-effectiveness, and simplicity of the required equipment. The samples were embedded in a closed cylindrical case (AISI 304L stainless steel) having a nitrogen powder mixture inside with an average particle size of 30  $\mu$ m. The nitriding agent contained an active source of calcium cyanamide (CaCN<sub>2</sub>, ~24% of N) and calcium silicate (CaSi, ~35 wt.% of the mixture) as an activator. The powder-pack nitriding process was carried out in a conventional furnace under a pure argon atmosphere at 773 and 823 K for 2 and 8 h of exposure for each temperature. Once the nitriding treatment was finished the container was removed from the furnace and slowly cooled to room temperature. In the second step, the pre-nitriding iron samples were borided by the pack method in the boriding agent contained an active source of boron (20% of B<sub>4</sub>C), an inert filler (70% of SiC), and an activator (10% of KBF<sub>4</sub>). The powder-pack boriding process was carried out in a conventional furnace under a pure argon atmosphere at 1223 and 1273 K for 2 and 8 h of exposure for each temperature using the same furnace and conditions. The depth of the surface coatings and morphology were analysed by SEM and EDS (JEOL JSM-6360 LV at 20 kV). X-Ray Diffraction (XRD) analyses of the layers were carried out with  $2\theta$  varying 20° to 90°, using CuK $\alpha$  radiation and  $\lambda = 1.54 \text{ \AA}$ . Figure 1 shows the cross-sections and the EDS analysis obtained by SEM at the  $\epsilon$ -Fe<sub>3</sub>N/Fe<sub>4</sub>N interphase for the nitrided ARMCO pure iron and Figure 2 shows the cross-sections and the EDS analysis obtained by SEM at surface of the mixture of  $\gamma'$ -Fe<sub>4</sub>N<sub>1-x</sub>,  $\epsilon$ -Fe<sub>3</sub>N and Fe<sub>2</sub>B layers for the nitrided ARMCO pure iron. The existence of the  $\gamma'$ -Fe<sub>4</sub>N<sub>1-x</sub>,  $\epsilon$ -Fe<sub>3</sub>N

and  $\text{Fe}_2\text{B}$  were verified by X-ray diffraction (Fig. 3). These results demonstrate that the mixture of  $\gamma'$ - $\text{Fe}_4\text{N}_{1-x}$ ,  $\epsilon$ - $\text{Fe}_3\text{N}$  and  $\text{Fe}_2\text{B}$  layers formed on the surface of nitrated ARMCO pure iron provide high resistance to abrasive wear and corrosion as well as high surface hardness 2000 HV.

#### References:

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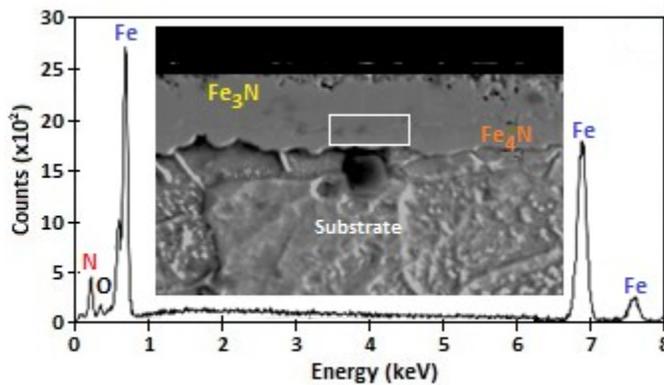


Fig. 1

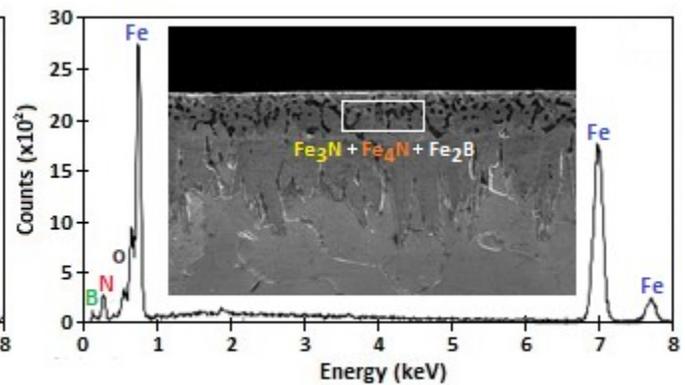
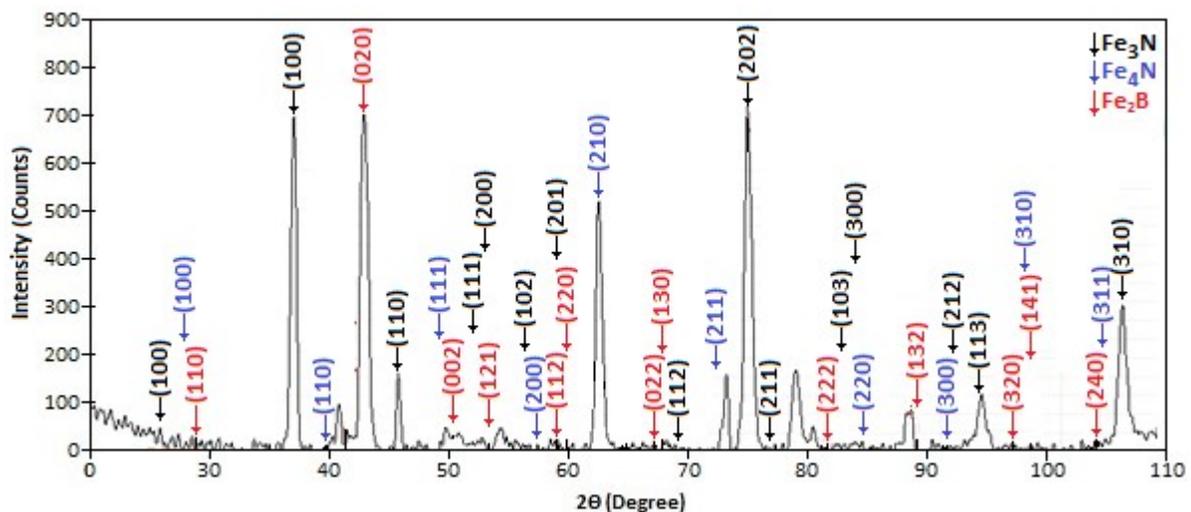


Fig. 2

**Figure 1.** SEM cross-sectional micrograph and EDS analysis of nitrated ARMCO pure iron with 823 K for 8 h and SEM.

**Figure 2.** cross-sectional micrograph and EDS analysis at surface of  $\gamma'$ - $\text{Fe}_4\text{N}_{1-x}$ ,  $\epsilon$ - $\text{Fe}_3\text{N}$  and  $\text{Fe}_2\text{B}$  layers developed on the surface of nitrated ARMCO pure iron with 1273 K for 8 h.



**Figure 3.** X-ray diffraction patterns obtained at the surface of nitro-borided pure iron exposed to the nitrating conditions of 823 K with 8; and boriding conditions of 1273 K with 8 h.