Analysis of Nitride Layers on ARMCO Pure Iron: The Powder-Pack Nitriding Process

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The nitriding of iron and steel is of considerable technological importance, because it can make a pronounced improvement in the fatigue, the wear, and the corrosion resistance of these materials. Nitriding on the surface of ferrous alloys results in the formation of a compound layer of γ' - Fe_4N_{1-x} and ε - Fe_3N nitrides or a mixture of γ' and ε with a nitrogen diffusion zone beneath the nitride layer. The broad range of nitride layer properties needed for different applications requires good control of nitriding process [1-3]. In the powder-pack nitriding process, which is similar to the powder-pack carburizing process, samples are placed in an annealing box with a powder mixture that consists of nitrogen-rich material and an activator. The nitriding temperatures are between 723 K and 823 K, a range in which the nitriding potential is a function of the mount of activator used in the powder mixture. The powder-pack method is a low-cost process particularly suited for the formation of uniform nitride layers on structural alloy components with complex shapes and of various sizes [4-5]. In this study, the microstructure of the γ' - Fe_4N_{1-x} and ε - Fe_3N layers formed on an ARMCO pure iron surface have been investigated at different temperatures by the powder-pack process.

The powder-pack nitriding process (Pulnieren©, H. E. F. Durferrit method) was conducted on square commercial samples of ARMCO pure iron with a thickness of 5 mm. Prior to the nitriding process, the samples were ground, polished cleaned at room temperature in H_2SO_4 for 10 s, and dried and stored in clean-room conditions. The samples were embedded in a closed in a closed cylindrical case (AISI 304L stainless steel) having a boron powder mixture inside with an average particle size of 30 µm. The pack method in the powder mixture consisting of calcium cyanamide ($CaCN_2$, ~24% of N) and calcium silicate (CaSi, ~35 wt.% of the mixture) as an activator. The samples were directly immersed in the powder mixture in another stainless steel cylindrical case. The nitriding temperatures were 723, 773 and 823 K for 2 - 12 h of exposure for each temperature. Nitriding followed after placing the case in a resistance furnace without the use of inert gases. Once treatment was complete, the container was removed from the furnace and slowly cooled to room temperature. The samples were cross-sectioned and resin-embedded for traditional metallographic preparation; the polished samples were etched in a 2% nital solution to observe the nitride layer depths and morphology by SEM and EDS (JEOL JSM-6360 LV at 20 kV). X-Ray Diffraction (XRD) analyses of the layers were carried out with 2θ varying 20° to 90°, using CuK_α radiation and λ = 1.54 Å. Finally, to determine the nitrogen depth profiles, Glow-Discharge Optical Emission Spectroscopy (GDOES) technique utilising a Horiba Jobin Yvon RF GD-Profiller2 operating with an RF source at 20 W and an argon working pressure of 650 Pa, the tests were carried out in the nitrided samples developed at 823 K with 12 h of exposure. Figure 1 shows the cross-sections and the EDS analysis obtained by SEM at the ε - Fe_3N/ γ' - Fe_4N_{1-x} interphase for the nitrided ARMCO pure iron. The corresponding profile composition revealed maximum nitrogen content on the surface of 25 at% N (Fig. 2). The existence of the ε - Fe_3N and γ' - Fe_4N_{1-x} were verified by X-ray diffraction (Fig. 3). The results demonstrate that the morphology of nitrided irons were homogeneous

and flat. Figure 2 shows the profile composition with maximum nitrogen content on the surface of 25 at% N, indicating the development of two nitride layers (ε - Fe_3N and γ'- Fe_4N_{1-x}), as confirmed by XRD shown in Figure 3.

References:

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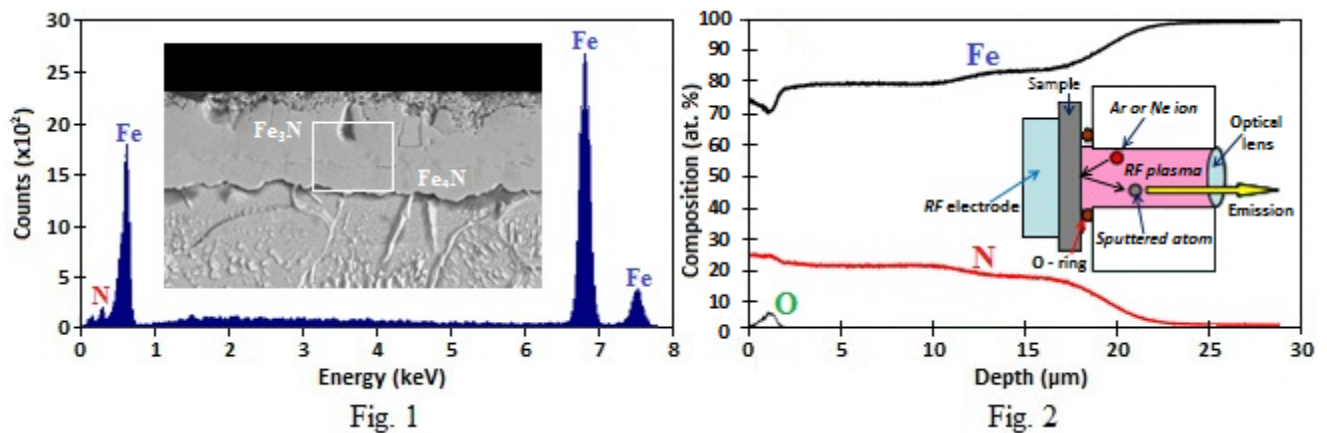
Figure 1. Image (SEM) cross-sectional of boro-nitrided ARMCO pure iron showing the produced layers and GDOES (**Figure 2**) spectrum of the elements distribution from the surface to the interior of the nitride γ'- Fe_4N and ε- Fe_3N layers at 823 K with 12 h.
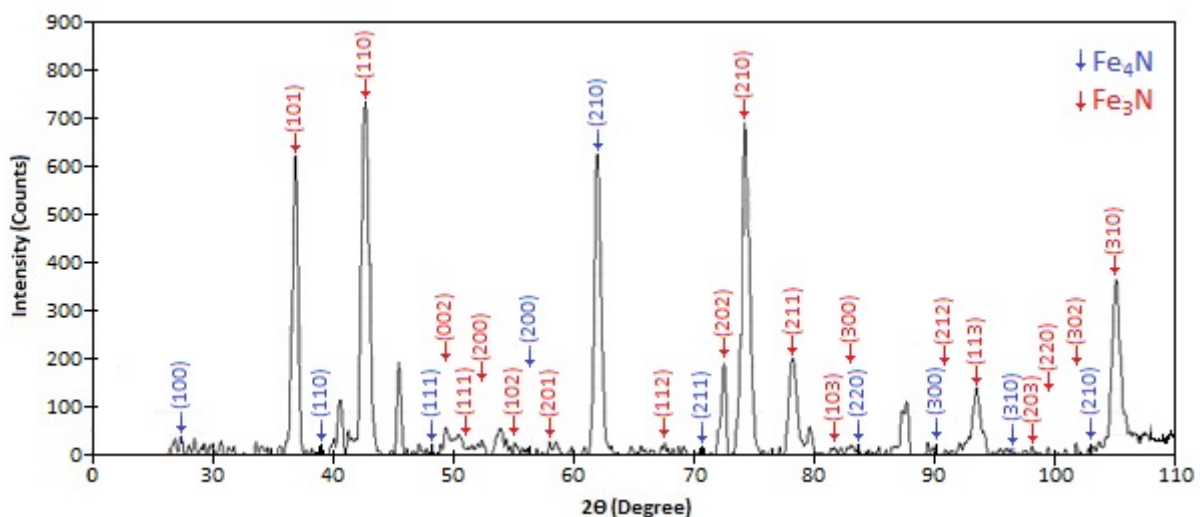

Figure 3. XRD from the cross-section of nitride coating, with 823 K for 12 h of exposure time.