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Plasma nitriding and post-oxidation mechanisms in ferrous alloys

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Abstract – We report a comprehensive study about the plasma nitriding and post-oxidation mechanisms that take place in ferrous alloys. The crystalline and morphological structures were characterized by XRD and SEM, respectively. The mechanical properties were analyzed by nanoindentation experiments. A narrow window of process was determined in order to reach the magnetite phase (Fe_3O_4). A low temperature post-oxidation process is mandatory for the stabilization of the kinetic product (Fe_3O_4) in the place of the thermodynamic product (α-Fe_2O_3).

Plasma nitriding is an established technique for surface modification of ferrous alloys. The nitrogen incorporation produces physical and chemical changes on the base material mechanical properties where hardness and wear resistance can be improved. However, and although friction and corrosion resistance are affected due to nitriding, these properties should be enhanced even more. Plasma oxidation is a complementary process after plasma nitriding where iron nitrides are oxidized[1]. The objective of our work is to study the plasma nitriding and oxidation mechanisms that take place in ferrous alloys focusing on the formation of ε-$Fe_{2\text{-}3}N$ and Fe_3O_4 (magnetite) phases.

In this work, we use AISI 1045 and M2 as ferrous alloys. The plasma treatments are detailed as follows: 1. pulsed plasma nitriding in a gas atmosphere of 20 % H_2 – 80 % N_2 during 5 hr at 480^{o}C and 1 mbar; 2. pulsed plasma oxidation in a gas atmosphere of 67 % N_2 – 33 % O_2 at variable treatment temperature and process time. After synthesis, the samples were characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM), and nanoindentation experiments.

When an iron nitride is oxidized, two main iron oxides may be formed[2]. Thermodynamic (α-Fe_2O_3 - hematite) and kinetic (Fe_3O_4 - magnetite) products are able to be achieved. However, the magnetite phase shows the best mechanical, morphological and chemical properties. There is a windows process in order to reach the magnetite phase where low temperature is mandatory. Figure 1 shows a XRD pattern of a sample treated at 480^{o}C (oxidation temperature) during 2 hr. One can be seen that only the Fe_3O_4, γ-Fe_4N, and ε-$Fe_{2\text{-}3}N$ phases were formed up to 6 μm. Figure 2 shows a SEM image where an oxide layer of approximately 3 μm grows on the top of the compound layer (γ-Fe_4N, and ε-$Fe_{2\text{-}3}N$). We believe that a low process temperature diminishes the iron mobility and oxidation from Fe^{2+} to Fe^{3+}, leading to the formation of a spinel phase (Fe_3O_4). Finally, the nanoindentation experiments show a smooth hardness profile.

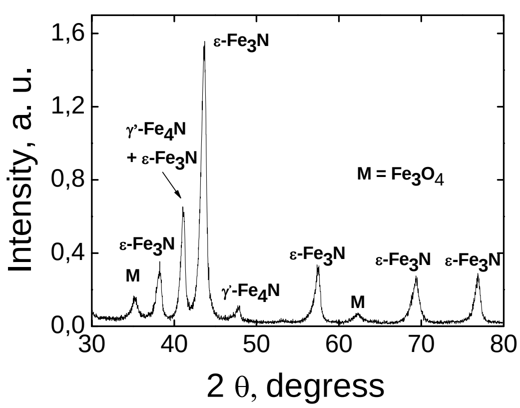

Figure 1: XRD pattern of AISI 1045 after plasma nitriding and post-oxidation at 480^{o}C.

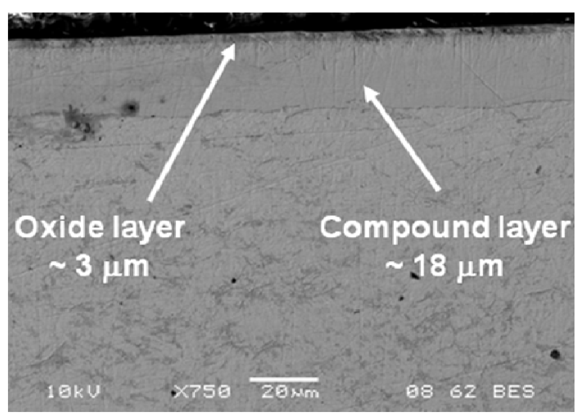

Figure 2: SEM image of AISI 1045 after plasma nitriding and post-oxidation treatment at 480^{o}C.

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