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Characterization of PVD Coating and Carbo-boro-nitride Layer on ARMCO® Pure Iron

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In recent years, the production of hardened surfaces on components to obtain improved wear-resistance has been a continuing technological challenge in surface engineering, which has led to the development of layers with high hardness. There are several types of processes that can be used to increase surface wear resistance and performance, foremost among them: CVD (Chemical Vapor Deposition), PVD (Physical Vapor Deposition), carburizing, nitriding, and boriding. Boriding stands out due to its simplicity and low cost when compared to PVD and CVD processes, and the surface hardness is far superior to those obtained by nitriding and carburizing [1-6]. A thick hard coating can help a soft substrate in carrying the loads and thus reduce the contact area and friction. In particular, boriding is typically carried out at high temperatures, above A_{C3} for an iron-based material, and the hardening mechanism occurs through boron-forming borides with iron and also with alloying elements such as nickel, cobalt, molybdenum, tungsten, and titanium. This creates a compound layer of iron borides of high hardness which, combined with their structure, results in extraordinary wear resistance. Likewise, titanium nitride (TiN) has been used in the coating of tool steels since the mid-sixties. The reasons to coat cutting tools in a production situation are to increase tool life, to improve the surface quality of the product, and to increase the production rate. The advantages of TiN coating include high hardness and adhesion, good ductility, excellent lubricity, high chemical stability and tough resistance to wear, corrosion and temperature [7]. In the present study, the microstructure of two coating configurations (TiN coating and carbo-boro-nitride layer) formed on an ARMCO® pure iron surface has been investigated at different temperatures by powder-pack mixture ($CaCN_2$ + CaSi + B_4C + SiC + KBF_4) (see Figure 1) and reactive PVD treatments. 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 to solely analyze the characteristic of mixture layers and some of their mechanical effects. Furthermore, the addition of a TiN coating on the carbo-boro-nitride layer is seen as a possibility of a reduction in the rate of crack propagation. The material involved in the research is the ARMCO® pure iron produced by powder metallurgy. The carbo-boro-nitriding and PVD treatments were carried out in two stages: carbo-boro-nitriding and then PVD. The samples were embedded in a closed in a closed cylindrical case (AISI 316L stainless steel) (see Figure 2) having the powder mixture having the composition of: $CaCN_2$ (calcium cyanamide) + CaSi (calcium silicate) + B_4C (active source of boron), Na_3AlF_6 (activator), SiC (inert filler), and $SiC_8H_{20}O_4$ which is used to protect surfaces with an average particle size of 10 µm (see Figure 1). The powder-pack carbo-boro-nitriding 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. Once the carbo-boro-nitriding treatment was finished the container was removed from the furnace and slowly cooled to room temperature. In the second step, the pre- carbo-boro-nitriding iron samples were cleaned using sputtering etching with 650 V, 240 kHz, 1600 ns for 15 min. The TiN coatings were obtained by using a target with high power impulse magnetron sputtering (HIPIMS) with

2000 W, 500 Hz, 200 ns and three targets with direct current magnetron sputtering (DCMS) with 2500 W on each (see Figure 3). The sputtering targets were Ti (> 99.8%). Interlayers of pure Ti were deposited with 400 V bias-voltage on all substrates. TiN layers were deposited by using bias-voltages of 75 V and 150 V. The TiN layers had thicknesses of approximately 1 µm and 5 µm. The coatings were deposited at 450°C with a Nitrogen/Argon (99.97% pure) atmosphere (Ar: N_2 Ratio = 24:5) and total pressure of 350 mPa and 120 min of exposure time. The hard samples were ground with SiC abrasive paper up to grit 2500. Afterward, the samples were polished using a diamond suspension with a particle size of 6 µm, finishing with a particle size of 3 µm [9]. The depth of the surface coatings and morphology were analyzed by SEM and EDS (JEOL JSM-6360 LV at 20 kV). Figure 4 shows the cross-sections and the EDS analysis obtained by SEM at ARMCO® pure iron and Figure 5 shows the cross-sections and the EDS analysis obtained by SEM at the TiN/γ'- Fe_4N_{1-x} + ε - Fe_2N + Fe_2B + CaC_2 interphases for the carbo-boro-nitrided ARMCO® pure iron with a thickness of 500 µm approximately. These results showed that the TiN top layer produced a hardness in the expected range, around 2500 HV, and no incompatibility with the supporting γ'- Fe_4N_{1-x} + ε - Fe_2N + Fe_2B + CaC_2 layer was observed. The duplex treatment proved to be effective in the production of high hardness and wear-resistant layers. The carbo-boro-nitriding treatment is recognized to produce high-performance coatings, but the TiN layer improved its properties even further. The existence of the TiN and γ'- Fe_4N_{1-x} + ε - Fe_2N + Fe_2B + CaC_2 were verified by X-ray diffraction (with 2θ varying 20° to 90°, using CuK_α radiation and λ = 1.54 Å). These results showed that the TiN and γ'- Fe_4N_{1-x} + ε - Fe_2N + Fe_2B + CaC_2 layers formed on the surface of carbo-boro-nitrided ARMCO® pure iron provide high resistance to abrasive wear and corrosion as well as high surface hardness. The combination of a hard surface and softer interior, made possible, for example, by case hardening method, is of inestimable value in modern engineering practice. Finally, the CaC_2 (Calcium Carbide) layer formed on the surface provides deoxidation (see Fig. 5).

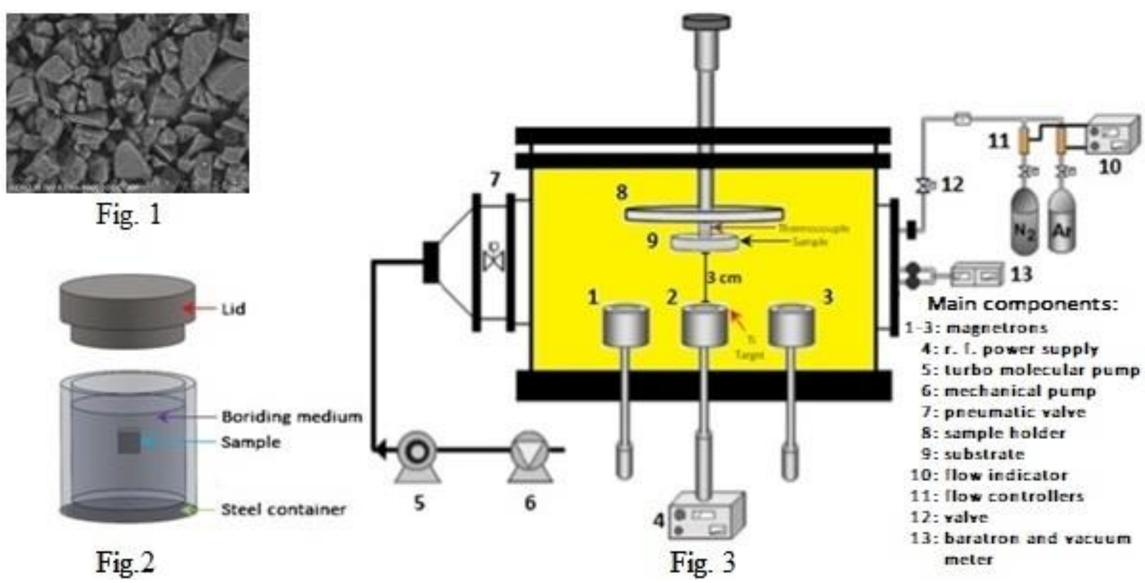

Figure 1. (Figure 1) Powder mixture, schematic view of the stainless steel AISI 316L container for the powder-pack treatment (Figure 2) and the schematic representation of the deposition reactor (Figure 3).

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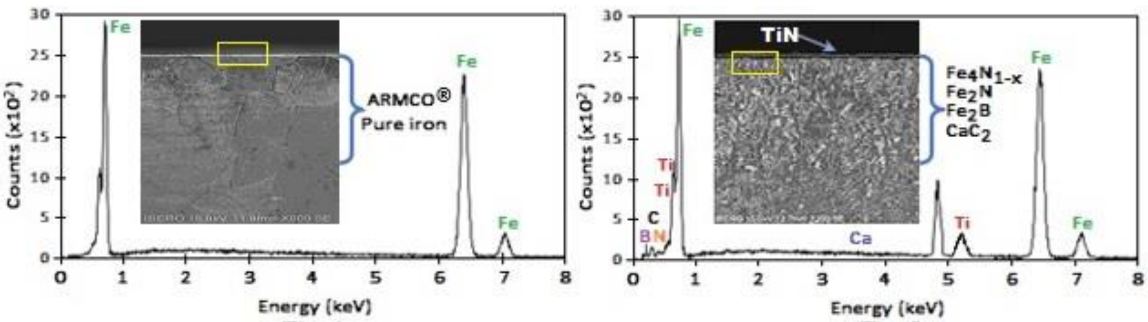

Figure 2. (Figure 4) SEM cross-sectional micrograph and EDS analysis of ARMCO® pure iron and SEM (Figure 5) cross-sectional micrograph and EDS spectrum at surface of the combination of: TiN and γ´-Fe4N1-x + ε - Fe2N + Fe2B + CaC2 layers developed on the surface of TiN and carbo-boro-nitrided ARMCO® pure iron with 1273 K for 8 h.

References

[1] J. R. Davis. "Surface Hardening of Steels: Understanding the Basics", 1st ed. ASM, Ohio, p. 213.
[2] M. Ortiz-Domínguez, I. Morgado-González, A. Cruz-Avilés, A. Soto-García, R. Trujillo-Sánchez, M. L. Moreno-González, G. Moreno-González, O. A. Gómez-Vagas, J. Zuno-Silva, Microsc. Microanal. 25 (Suppl 2) 2019, p. 2400.
[3] M. Ortiz-Domínguez, O. A. Gómez-Vargas, I. Simón-Marmolejo, M. A. Flores-Rentería, L. E. Martínez-Martínez, A. Cruz-Avilés, M. A. Paredes-Rueda, Microsc. Microanal. 24 (Suppl 1) 2018, p. 1076.
[4] K. Y. Li and Z. D. Xiang, Surf. Coat. Technol. 204 (2010), p. 2268.
[5] H. Du and J. Agren, Z. Metallkd. 86 (1995), p. 522.
[6] K. H. Habig, Mater. Eng. 2 (1980) p. 83. [7] M. Flores, S. Muhl, L. Huerta, Surf. Coat. Technol. 200 (2005) p.1315-1319. [8] J. L. Bernal, E. Vera, A. I. Martínez, A. García, Microsc. Microanal. 24 (Suppl 1) 2018, p. 1102.
[7] O. A. Gómez-Vagas, M. Ortiz-Domínguez, A. Cruz-Avilés, I. Morgado-González, J. Solis-Romero, V. A. Castellanos-Escamilla, E. Coronel-Guerra, E. Cardoso-Legorreta, Microsc. Microanal. 25 (Suppl 2) 2019, p. 770.

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