

TiN Coating and Fe₂B Layer Obtained by PVD and Powder-pack Boriding Treatments Formed on ASTM A1011 Steel

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Various hard coatings, like borides, nitrides, diamond and cBN, are currently used in the automotive industry, and are considered key to efficient automotive manufacturing processes. Several families of such coatings have been developed during the past 30 years, ranging from different metal nitrides, like TiN, TiAlN, or CrN, to various carbon-based films. Conventional nitride coatings have been prepared using physical and chemical vapor deposition. Superhard cBN will soon be available for industrial applications; this coating bridges the gap between nitrides and diamond, and has superior temperature stability to that of diamond. Due to their hardness and low friction coefficient, diamond-like carbon films will be indispensable for engine and power train components. However, their relevance to the automotive and aerospace industries is still limited, though it is likely to grow substantially in the future, pushed by the need to reduce fuel consumption and the desire for components with longer lifetimes [1]. A proposed solution consist of developing a hard, dense, wear- and corrosion-resistant coating formed on a surface with certain affinity in fracture toughness, as well as the creation of a dislocation interface that prevents the propagation of the corrosive medium to the substrate and the propagation of micro-cracks [2-9]. In particular, boriding is a recognized surface treatment for achieving high surface hardness (up to 2000 HV) and low coefficient of friction while improving the corrosion and erosion resistance and is being increasingly used in many industrial applications. Likewise, the hard TiN (Titanium Nitride) coatings are also used for coating machine tools such as drills, lathe tool inserts, stamps and punches, and expensive forming tools such as injection molds for plastics. In the present study, the microstructure of two coating configurations (TiN and Fe₂B) formed on an ASTM A1011 steel surface have been investigated at different temperatures by dehydrated paste-pack boriding and reactive PVD treatments. Moreover, the addition of a TiN coating on the boride layer is seen as a possibility of reduction in the rate of crack propagation. The material involved in the research, is the ASTM A1011 steel produced by powder metallurgy. The boriding and PVD treatments were carried out in two stages: boriding and then PVD. The samples were embedded in a closed in a closed cylindrical case (AISI 316L stainless steel) having a dehydrated paste of boron powder mixture inside with an average particle size of 10 μm (see Figure 1). Boriding mixture contains of B₄C (active source of boron), Na₃AlF₆ (activator), SiC (inert filler), and SiC₈H₂₀O₄ which is used to protect surfaces. The boriding process was carried out in a conventional furnace under a pure argon atmosphere at 1123 and 1273 K for 8 h of exposure for each temperature. Once the boriding treatment was finished the container was removed from the furnace and slowly cooled to room temperature. In the second step, the pre-boriding 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 2).

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 2 μm . The coatings were deposited at 450°C with an Nitrogen/Argon (99.97% pure) atmosphere (Ar:N₂ Ratio = 24:5) and total pressure of 350 mPa and 120 min of exposure time. The hard samples were grinded with SiC abrasive paper up to grit 2500. Afterwards, the samples were polished using a diamond suspension with particle size of 6 μm , finishing with particle size of 3 μm . The depth of the surface coatings and morphology were analysed by SEM and EDS (JEOL JSM-6360 LV at 20 kV). Figure 3 shows the cross-sections and the EDS analysis obtained by SEM at the TiN/Fe₂B interphases for the ASTM A1011 steel (see Figure 4). These results demonstrate that the formed the TiN-Fe₂B top layer produced a hardness in the expected range, around 2500 HV, and no incompatibility with the supporting boride layer was observed. The duplex treatment proved to be effective in the production of high hardness and wear resistant layers. Boriding provides a uniform hardness layer from the surface on to the entire depth of the diffused layer. The hardness achieved is many times higher than any other surface hardening process. The combination of high hardness and low coefficient of friction enhance wear, abrasion and surface fatigue properties. Other benefits associated with boriding are retention of hardness at elevated temperature, corrosion resistance in acidic environment, reduction in use of lubricants and a reduced tendency to cold weld, but the TiN layer improved its properties even further.

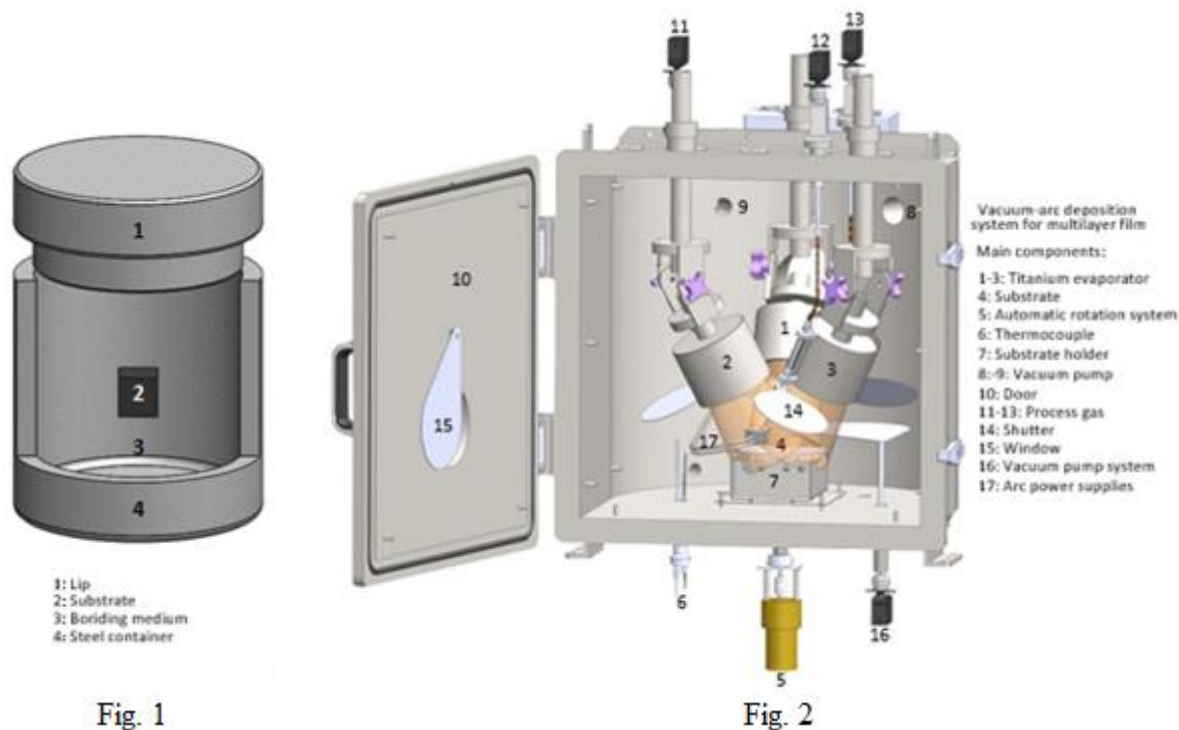


Figure 1. Schematic view of the stainless steel AISI 316L container for the powder-pack boriding treatment and the schematic representation of the deposition reactor (**Figure 2**).

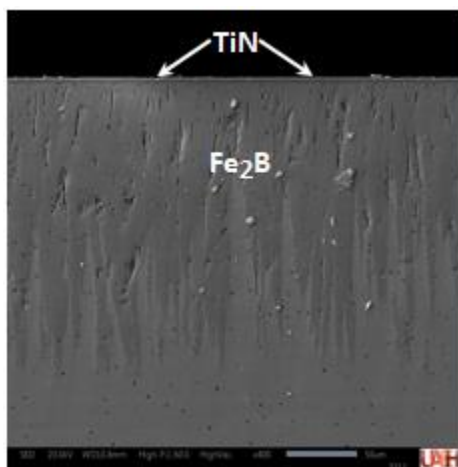


Fig. 3

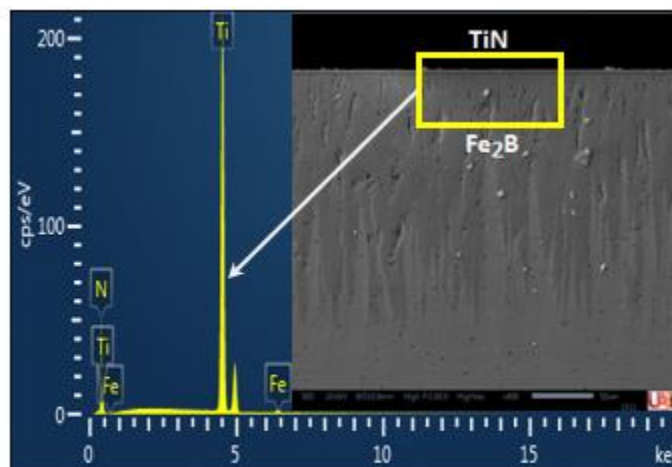


Fig. 4

Figure 3. Images (SEM) of cross-sections of borided: ASTM A1011 steel during 8 hours of treatment at 1273 K and EDS (**Figure 4**) spectrum of the TiN/Fe₂B interphases for the ASTM A1011 steel.

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