Ion nitriding of supermartensitic stainless steel HP13Cr by the glow discharge and cathodic cage techniques

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Abstract

The equipments used for the marine petroleum extraction ("subsea trees") must be prepared for extreme conditions such high temperature, extreme pressure, corrosion and erosion. A surface protection means is necessary for valves, connection points and peripheral devices installed on it. Among the existing methods, the plasma-based ones are widely used in the surface modification of materials, since they afford beneficial effects for the work conditions, such as improvement in the tribo-mechanical properties of metallic alloys. Surface coatings produced by plasma methods can be well integrated with substrate due to the concentration gradient of chemical species, added during the coating process, which has its maximum at the workpiece surface. In this study, samples of commercial stainless steel HP13Cr, obtained from a cross-sectioned petroleum duct ceded by the Brazilian oil company Petrobras, were nitrided by two DC plasma methods: glow discharge (GD) and cathodic cage (CC). The samples were analyzed by X-ray diffraction (XRD), atomic force microscopy, scanning electron microscopy, energy dispersive X-ray spectroscopy, instrumented indentation and nanoscratch. Prior to the surface treatment, XRD analysis showed that the HP13Cr supermartensitic stainless steel also presented the austenite phase (gamma), possibly originated from the duct working conditions. In order to obtain exclusively the martensite phase (alfa), samples were submitted to solution heat treatment at temperatures among 1248-1373 K during 0.5 h with subsequent quenching in water or oil. The better results were obtained for 1373 K in oil quenching, for which the austenitic phase was completely converted to martensite, with residual densities of M23C6, M7C3, M3C carbides (where M can be any metal atom of the material). Steel samples were then mechanically polished to a mirror finishing. Plasma treatments were performed in a two-step process: (i) H2 sputtering at 473 K during 1 h; (ii) nitriding with the temperature-time parameters 623K-6h, 673K-6h, 723K-6h. In CC, a smooth film with thickness up to 800 nm, comprising crystalline phases Fe(2+x)N and Fe4N, was produced in all the studied conditions. However, the critical load for detaching the films from substrate was as low as 11 mN. In GD, the nitrogen solid solution in martensite prevailed, coexisting with small amounts of nitride phases. The modified surface regions were larger than 10 micra, which hardness profiles increased from 3.9 to 14 GPa. Moreover, the scratch resistance was increased twice the value of the substrate in the GD process.

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