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Surface hardening by plasma nitriding on high chromium alloy steel

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One of the principal methods of improving workpiece performance in the metal-mechanical industry is to achieve an increase in wear resistance. To achieve this, a change of bulk material is not always the best solution for reasons of costs or R&D efforts in new material development. Surface engineering is an optional process to increase the surface wear resistance of low cost conventional materials.

Nitriding is a thermochemical surface treatment used in steels and alloys to improve wear and friction properties by surface microstructure modification, while maintaining adequate substrate properties [1]. Not only wear resistance is improved, but also corrosion and fatigue resistance may be increased by the same treatment. Plasma nitriding is the most versatile nitriding process and has many advantages over conventional salt-bath and gas nitriding. The close control of the metallurgical properties of the nitrided surface is the most important advantage of the plasma nitriding process [2]. Nowadays duplex and hybrid layers are growing in importance [3], requiring prior plasma nitriding of the substrate to avoid compound layer formation.

Response to nitriding is not only derived from process parameters, such as time, temperature and nitrogen potential, but is highly dependent upon the substrate composition. Alloy elements in the steel composition affect the nitriding response, mainly when nitride-forming elements are present. Alloy elements such as Al, Ti, Cr, V and Mo interact differently with nitrogen and directly affect the nitriding response, mainly regarding surface hardening properties [4, 5].

The aim of the present investigation is to study the nitriding characteristics of high-alloy chromium steel. As pointed out by Jack and Lightfoot [4, 5], elements such as Al and Ti interact strongly with nitrogen, while the interaction of Cr is dependent on the alloy content of the steel. It is considered that a chromium content below 5.6 wt. % has an intermediate interaction behavior, and for higher contents this element possesses strong interaction characteristics. The hardening effect changes due to the interaction between the alloy element and nitrogen. Where there is an intermediate or low interaction the hardness profile is diffuse, but where there is a strong interaction a flat type profile is expected to be found.

To investigate the interaction characteristics between chromium and nitrogen during plasma nitriding, a high chromium alloy martensitic stainless steel was used, with a Cr content higher than 5.6 wt. %. This steel is used in a variety of applications such as in structural parts, molds for plastic injection, cutlery, and surgical instruments. In all of these applications, wear resistance is a key property of performance.

Application of plasma nitriding on stainless steels is of particular importance, because of the need to remove the passive film from the surface before nitriding. When using conventional liquid and gas processes, a mechanical or chemical process must be used to remove the passive layer before the nitriding treatment. These procedures not only increase costs, but may also impair the surface quality of the workpiece. During plasma nitriding, the passive film is removed by high intensity hydrogen sputtering before the nitriding step; both process occur during the same treatment cycle.

The wrought martensitic stainless steel type 420 used in this investigation was received in round bar form in the as-annealed condition with a base chemical composition Fe-0.40%C-13.5%Cr. Before nitriding, all the samples were quenched and tempered to obtain 350–370 HV substrate hardness. During the thermochemical treatment, but before the nitriding step, the samples were sputtered in high-density pure hydrogen plasma during heating and held there at 450 °C for 45 min, to remove the passive film from the surface. The nitriding temperatures ranged from 480 to 560 °C, using a fixed time of 4 h and a gas mixture ratio of $N_2 : H_2 = 3 : 1$. For nitriding, the voltage used was 470 V at a pressure of 250 Pa.

The nitrided surface microstructure was observed using optical and scanning electron microscopy, after Nital 4% etching. The nitriding depth was measured on the optical microscope using an image analysis system. Microhardness measurements were performed on a Vickers machine using a 25 g load. Additionally X-ray analysis and EDS microprobe analysis were used for the characterization of the nitrided surface.

Under the process conditions, the presence of the compound layer was verified for all nitriding temperatures. Upon increasing the nitriding temperature there was a thickening of the compound layer, up to 8.8 μm at 560 °C. The diffusion zone depth increased by a parabolic growth rate up to 115 μm maximum at 560 °C. Regarding the diffusion zone morphology, Fig. 1 shows that a well-defined planar interface

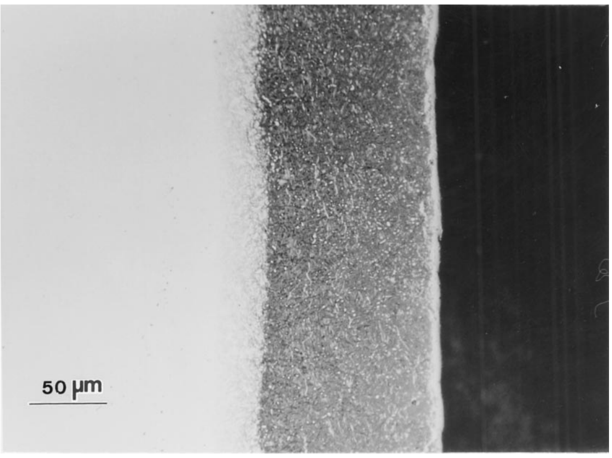

Figure 1 Surface microstructure after plasma nitriding at 560 °C.

between the diffusion zone and the substrate was developed; this behavior was verified for all nitriding temperatures. With increasing temperature, grain boundaries nitrides precipitated on the diffusion zone, however, the grain boundaries nitrides did not form a network.

The hardness profiles, Fig. 2, with the formation of a maximum hardness plateau, show a strong hardening effect on the nitriding, increasing the substrate hardness up to 4 times. This hardening effect is due to a fine and homogeneous coherent chromium nitride precipitation; type CrN [6], confirmed by X-ray analysis. The formation of a maximum hardness plateau, which increases in depth with increasing nitriding temperature and exhibits a sharp decrease after the maximum hardness, is a very important characteristic of the hardness profiles of high chromium steel. This plateau is associated with

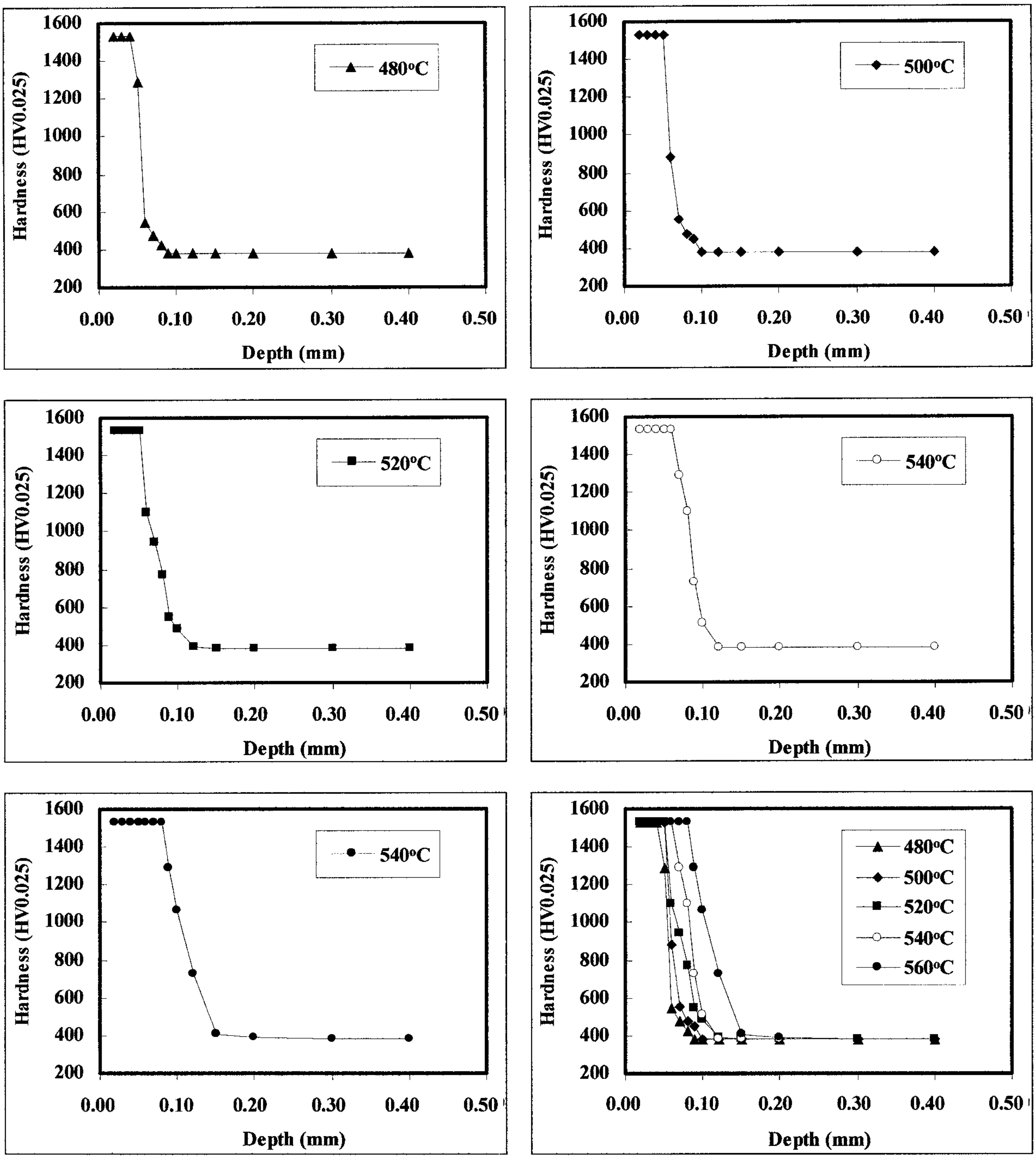

Figure 2 Individual microhardness profiles and the set of curves for the nitrided surfaces at each tested temperature. A constant nitriding time of 4 h and a gas mixture of 75%N_2 : 25%H_2 were utilized.

the nitrogen compositional profile through the nitrided surface, developed as a consequence of the nitriding mechanism [7]. EDS microprobe results showed a constant nitrogen concentration in the diffusion zone and a sharp decrease in the element content at the nitriding interface.

The morphology of the nitrided surface and the hardness profiles obtained may be explained as a result of the interaction behavior between chromium and nitrogen. As pointed out in literature [4, 5], alloys with a high chromium content tend to exhibit a strong interaction between the Cr and N. Assuming that complex nitride precipitation reactions take place at the nitriding interface, the displacement of such an interface is only possible after all the solid state reactions have taken place, considering that only nitrogen diffusion is possible.

Therefore, surface hardening characteristics with a maximum hardness plateau, was a consequence of the growth mechanism for such nitriding interfaces, confirming a strong interaction effect between chromium and nitrogen on the 13.5%Cr steel. The resulting effect is planar nitriding interface morphology, rather than the diffuse type found for lower chromium content steels.

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