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Formation of a nanostructured CrN layer on nitrided tool steel by low-temperature chromizing

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A duplex chromizing process involving the salt bath chromizing of a plasma-nitrided tool steel was carried out at 773 K for 3–24 h. A single-phase CrN layer with a grain size ranging from 20 to 50 nm and an underlying layer consisting of α-Fe and nano-sized CrN grains with a grain size ranging from 40 to 80 nm were formed by a 24 h chromizing at 773 K. The duplex chromizing mechanism was discussed.
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Surface nanocrystallization can be realized in three ways [1]. The first method is based on various surface coating and deposition processes [2,3]. The second method transforms the surface layers of materials into a nanocrystalline structure while keeping the overall composition unchanged [4,5]. The third method is a duplex process in which a nanostructured transformable layer is developed and joined with chemical, thermal or metallurgical processes to produce nanostructured layers with different chemical compositions or different phases [6].

The purpose of this discussion is to report a novel process with which a nanostructured CrN layer can be formed directly on a nitrided steel surface by a low-temperature chromizing process. By this method, a tool steel (AISI H13) was surface nanocrystallized with CrN grains.

The substrate material was a commercial AISI H13 steel containing 0.38C, 5.41Cr, 1.18Mo, 0.94Si, 0.93V, 0.23Mn, and balance Fe (in wt.%). The specimens were vacuum austenized at 1303 K for 1.5 h, oil quenched and tempered at 823 K for 3 h. The heat-treated specimens were then polished with up to 900 grade emery paper, degreased, cleaned and plasma nitrided at 813–833 K for 5.5 h. The plasma-nitrided specimens were finally salt bath chromized at 773 K for 3, 6 and 24 h, then water quenched. All the specimens concerned were plasma nitrided together in a single batch to ensure an even nitrided layer throughout them. X-ray diffraction of the specimens were conducted using a PHILPS X'Pert MPO Pro X-ray diffractometer with Cu K_α radiation. Cross-sectional specimens were etched with 3% Nital and characterized using a LEO1530VP scanning electron microscope (SEM) with a quadrature back-scattering detector. The Cr $K_{\alpha1}$ and Fe $K_{\alpha1}$ profiles across the coatings were determined by X-ray energy-dispersive spectroscopy (EDS) and that of N K_α was determined by electron probe microanalysis (EPMA). Transmission electron microscopy (TEM) of the cross-sectional foils was carried out with a JEM 2010 transmission electron microscope working at 200 kV.

The back-scattered electron SEM microstructure of the as-plasma-nitrided specimen, shown in Figure 1a, indicates that on the outer surface of the specimen was a compound layer (layer C) consisting mainly of ε iron nitride phase (confirmed by the XRD spectrum shown in Fig. 2). A "cover layer", marked as layer C_0 in Figure 1a, was formed on the outer surface. This cover layer, which was reported [7] to be harmful to the finished coatings, was mechanically removed before the specimens were salt bath chromized. In addition, as clearly revealed by optical microscopy, the nitriding formed a diffusional zone of about 150 μm beneath the compound layer in which the nitrides of the alloying elements were precipitated.

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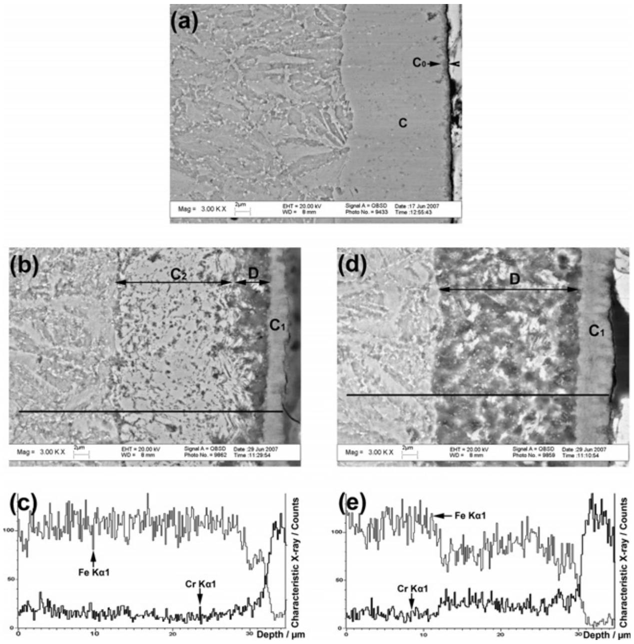

Figure 1. Cross-sectional microstructures of the coatings on H13 specimens subjected to various treatments. (a) Specimen plasma nitrided for 5.5 h at 813–833 K, SEM back-scattered electron image; (b) specimen first plasma nitrided and then chromized for 6 h at 773 K, SEM back-scattered electron image; (c) EDS profiles of Cr $K_{\alpha 1}$ and Fe $K_{\alpha 1}$ along the line marked in (b); (d) specimen first nitrided and then chromized for 24 h at 773 K, SEM back-scattered electron image; (e) EDS profiles of Cr $K_{\alpha 1}$ and Fe $K_{\alpha 1}$ along the line marked in (d).

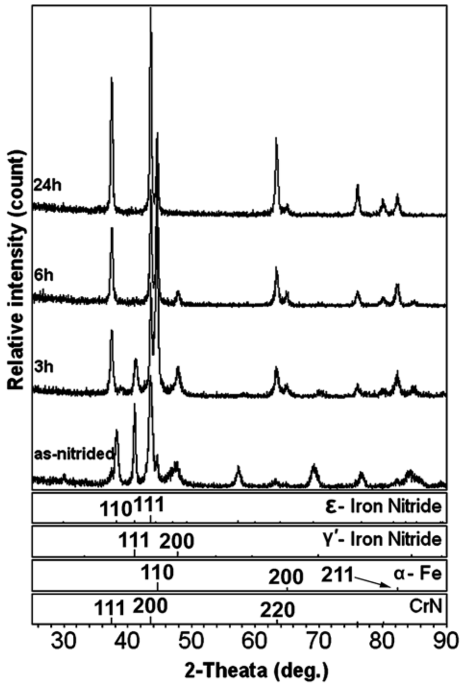

Figure 2. XRD patterns showing the phase structure evolutions in the near-surface layer during the duplex chromizing process for the as-nitrided specimen, and the specimens first nitrided and then chromized for 3, 6 and 24 h respectively.

As shown in Figure 1b and d, a complete chromized layer, consisting of three sublayers, C_1, D and C_2, was formed within, and at the expense of, the former compound layer formed by nitriding, marked as layer C in Figure 1a. Here, sublayer C_1 is a chromized compound layer consisting of single-phase CrN, sublayer D is a mixture of CrN and α-Fe, and sublayer C_2 consists mainly of γ'-Fe_4N. Sublayer ($D + C_2$), also named the diffusional layer of chromizing, became a single layer D with the layer C_2 having disappeared as the chromizing time was increased from 6 h (Fig. 1b) to 24 h (Fig. 1d). It was found that the chromized layer as a whole became thicker as the chromizing time was prolonged, thus leading to a chromized layer thickness larger than that of the original nitrided compound layer, whereas the chromized compound layer C_1, having been formed quickly in the early stages of chromizing, remained nearly unchanged in thickness with increasing chromizing time. For example, the original nitrided compound layer C in Figure 1a, while splitting into the three sublayers C_1, D and C_2 during chromizing, was thickened from about 12 to 23 μm during a 24 h chromizing at 773 K, with the chromized compound layer C_1 having a constant thickness of 3–4 μm.

The XRD patterns in Figure 2 show that the intensity of the nitriding-formed ε-Fe_2N and γ'-Fe_4N phases is decreased, while that of the CrN phase is increased, with prolonged chromizing duration, and that the ε-Fe_2N in the original nitrided compound was nearly completely consumed during a 6 h chromizing, with only γ'-Fe_4N remaining, whereas both ε-Fe_2N and γ'-Fe_4N were present in the as-nitrided compound layer. A comparison of the light contrast of layer C_2 with the darker contrast of layer D in the back-scattered electron SEM image shown in Figure 1b suggests that layer C_2 contained none of the α-Fe that was detected to be present in layer D and assumed to be produced via reaction (6), and that the release of nitrogen atoms [N] from the ε-Fe_2N during chromizing would follow the following reaction in which no α-Fe was produced:

$$2\varepsilon\text{-}Fe_2N \rightarrow \gamma'\text{-}Fe_4N + [N] \tag{1}$$

The nitrogen atoms [N] so released from ε-Fe_2N could be consumed during chromizing in three possible ways: (i) they diffuse outward to the surface and react with the incoming Cr atoms to form chromium nitrides; (ii) they recombine with each other to form N_2; and (iii) they diffuse inward to transform the adjacent substrate (previous supersaturated with nitrogen during plasma nitriding) into iron nitrides. With [N] atoms diffusing outward, a dense layer consisting of single-phase CrN (marked as layer C_1 in Fig. 1b) was formed on the surface layer of the specimen via the reaction

$$[N] + [Cr] \rightarrow CrN, \tag{2}$$

where [Cr] represents the activated chromium atoms provided by the chromizing salt bath. The [N] and [Cr] atoms diffused in opposite directions in the ε-Fe_2N layer and combined to form CrN.

The diffusion process may, however, be slowed down somewhere in the interior of the ε-Fe_2N layer close to the surface, since the path taken by the [N] atoms diffusing to the surface and reacting there with the incoming [Cr] atoms would be too long to sustain reaction (2).

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