

THE INFLUENCE OF PLASMA NITRIDING ON THE MICROSTRUCTURE OF X153CrMoV12 AND X165CrV12 STEELS

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This study presents the results of research into the influence of the time of the plasma nitriding process on the microstructure of the coatings obtained on cold-work tool steels X153CrMoV12 and X165CrV12. The processes were carried out under industrial conditions using an Ionit system (Oerlikon Metaplas) with variable process times of 2, 4 and 6 hours. Nitriding mixture consisting of 5 % nitrogen and 95 % hydrogen was chosen, which allowed the expected diffusion layer to be obtained without a white layer (composed of iron nitrides). Analysis of relative elemental concentrations indicates that the presence and content of nitride-forming elements influences the formation of alloy additive nitrides in the microstructure of the diffusion layer. Nitrides of alloying additives, present in the diffusion layer, indicating that investigated steels are the most suitable for plasma nitriding.

Keywords: X153CrMoV12, X165CrV12, tool steel, plasma nitriding, X-ray research

INTRODUCTION

Thermochemical treatment processes are performed to improve tribological and mechanical properties and increase corrosion resistance. They are implemented by combining thermal treatments with a targeted change in the chemical composition of the surface layer and might be used in different applications [1-3]. At a specific temperature and time, the near-surface zone is diffusely saturated with an element or elements in solid, liquid, gaseous and plasma media [4,5]. One such process is plasma nitriding, which aims to improve the anticorrosive properties, fatigue resistance and abrasive wear of steels [6,7]. The main advantage of plasma nitriding is that it is possible to form a layer of compounds with only the ϵ or γ' phase, or to produce a nitrided layer consisting only of the diffusion zone. Plasma nitriding allows diffusion of nitrogen across grain boundaries and lattice diffusion [8]. The advantage of this process is a surface with much less roughness than gas nitriding and minimal distortion of the feedstock. Therefore, products after the process do not require additional machining [8, 9].

In order to make the best use of the nitriding potential, special steels with a specific chemical composition have been developed containing, among others, alumin-

ium, chromium, molybdenum and vanadium, which have a high affinity for nitrogen [8, 9].

The aim of this study was to determine the influence of the plasma nitriding process parameters and the chemical composition of the substrate material on the microstructure of the nitride layers formed, on the substrates of selected tool steels, and to determine the growth kinetics of the diffusion layer.

EXPERIMENTAL PROCEDURE

The samples made from X153CrMoV12, X165CrV12 cold work steels, measuring $f30$ mm x 30 mm, were supplied in tempered condition. The surfaces to be tested were sandblasted with electrocorundum and then ground on an ATM GmbH Saphir 330 grinder with SiC grit sandpaper with a gradation of 80 to 500, 90 specimens were prepared - 2 specimens for each steel grade at three time variants: 2 h, 4 h and 6 h, Immediately before the processes, the samples were cleaned in an ultrasonic cleaner. Plasma nitriding was carried out in an Oerlikon-Metco IONIT unit. The invariant process parameters are shown in Table 1.

After the plasma nitriding processes, metallographic samples were taken. The microstructure of the nitrided layers was studied on a Phenom XL Scanning Electron microscopy (SEM) from PIK Instruments, using a back-scattered electron detector - BSE. The chemical composition was analysed using a secondary X-ray energy dispersive spectrometer (EDS) integrated with the Phenom XL microscope. Maps showing the relative content of chemical elements from the analysed areas were obtained. All samples were etched with nital 4 % to measure the depth of the layers formed.

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Table 1 Basic parameters of plasma nitriding process

Composition of nitrided mixture / l/h	5N ₂ +95H ₂
Temperature / °C	550
Frequency / kHz	10
Voltage / V	400
Intensity / A	15
Pressure / mbar	3,6
Pulse	20/80
Process time / h	2,4,6

RESULTS AND DISCUSSION

Results of X153CrMoV12 plasma nitriding

The microstructure of X153CrMoV12 steel after plasma nitriding at 2, 4 and 6 h (Figure 1 a-c) consists of a light matrix and two types of dark precipitates. Literature data [8] suggest that one type of precipitates is primary and secondary carbides. The lighter-coloured precipitates are probably chromium and vanadium carbides and it was confirmed by X-ray diffraction (XRD) phase analysis (Figure 2.). Areas with increased chromium content are iron-poor. The large, longitudinally

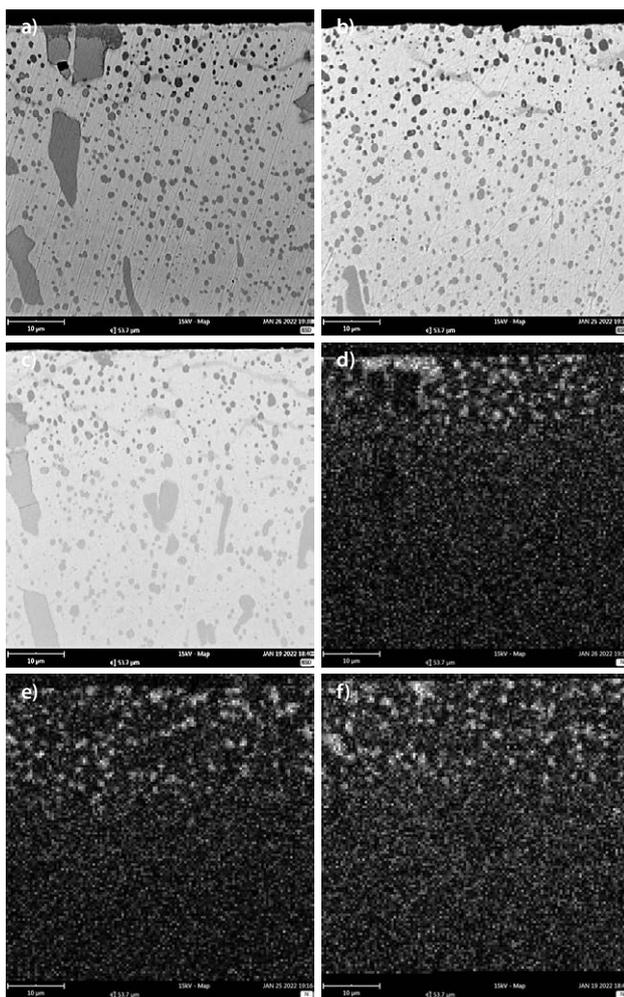


Figure 1 Microstructure of the layer after 2 (a), 4 and 6 h of the nitriding process carried out on the substrate of tool steel grade X153CrMoV12 and results of the analysis of the relative concentrations of nitrogen

shaped precipitates are arranged in bands perpendicular to the surface of the sample, and the fine precipitates are evenly distributed in the microstructure. The darker separations are chromium and vanadium nitrides, as evidenced by the chromium content of the steel (11,0-13,0 wt.%). Their presence was detected by XRD phase analysis (Figure 2). The near-surface layers of the samples show areas of increased nitrogen concentration, the amount of which decreases with increasing distance from the surface of the samples (Figure 1d-f). This demonstrates the formation of the diffusion layer itself on the substrate of the plasma nitrided X153CrMoV12 steel at 2, 4 and 6 h. The average depth of the diffusion layer is 7,32 μm for the treatment after 2 h. After 4 h, a layer with an average depth of 33,86 μm was obtained while after 6 h: 39,04 μm (Figure 3).

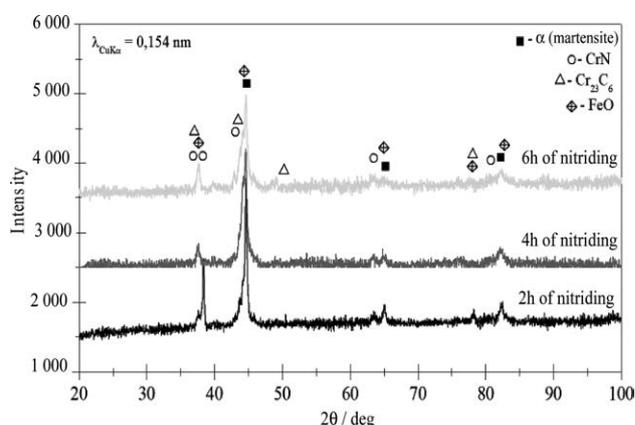


Figure 2 An XRD pattern of the layer after 2, 4 and 6 h of the nitriding process carried out on the substrate of X153CrMoV12 steel

Results of X165CrV12 plasma nitriding

The microstructure of X165CrV12 steel, after plasma nitriding for 2, 4 and 6 h (Figure 5a-c), consists of a bright matrix and two types of precipitates. The chromium high carbon content of the steel (1,65 wt.%) indicates that the lighter-coloured precipitates are chromium carbides confirmed by XRD phase analysis (Figure 4). The large longitudinal and globular-like precipitates are arranged in bands perpendicular to the sample surface. The fine precipitates are evenly distributed in the

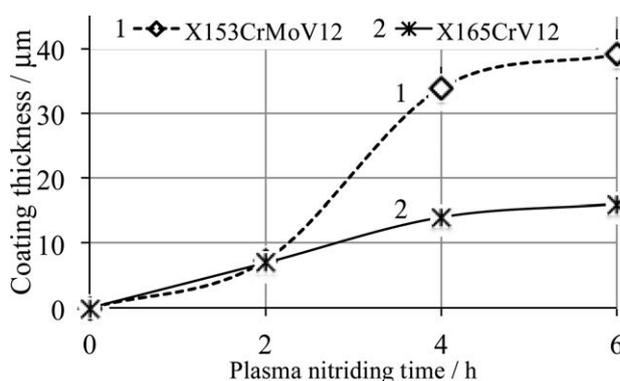


Figure 3 Average thickness of diffusion layer after 2 (a), 4 and 6 h of plasma nitriding process of investigated steels.

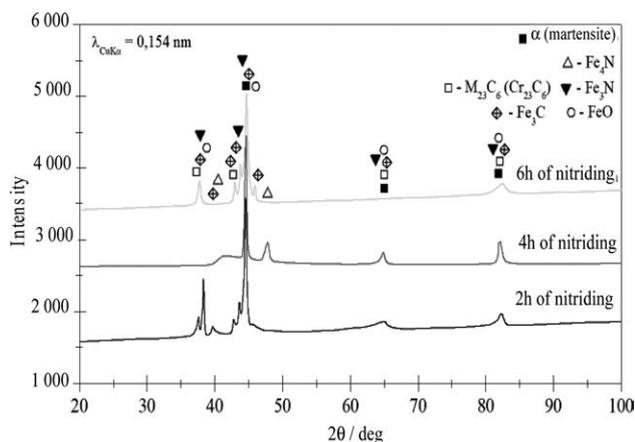


Figure 4 An XRD pattern of the layer after 2, 4 and 6 h of the nitriding process carried out on the substrate of X165Cr12 steel

microstructure. The darker globular-shaped precipitates present in the nitrided layer are chromium nitrides. (Figure 5d-f). The XRD phase analysis showed only the presence of Fe_3N and Fe_4N nitrides (Figure 4). Nitriding of X165Cr12 steel at 2, 4 and 6 h leads to the formation of diffusion layers with average depths of 6,95 μm , 14,00 μm , and 16,02 μm , as evidenced by the increased

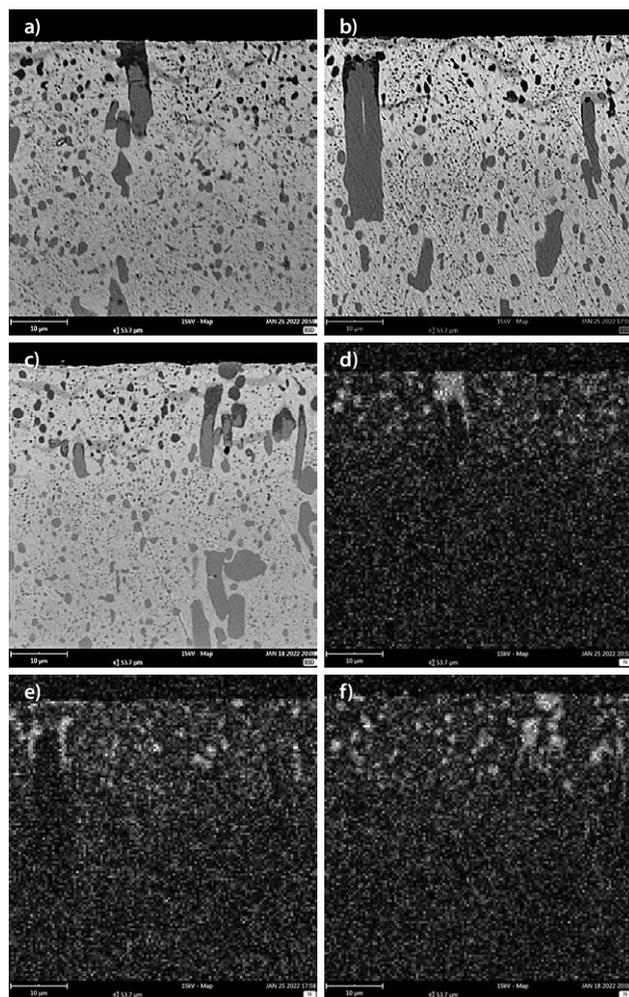


Figure 5 Microstructure of the layer after 2 (a), 4 and 6 h of the nitriding process carried out on the substrate of tool steel grade X165Cr12 steel and results of the analysis of the relative concentrations of nitrogen

nitrogen concentration in the chemical composition analysis maps and the change in microstructure near the surface of the samples (Figures 3 and 5d-f).

SUMMARY

The formation of chromium nitrides was observed in the diffusion layer of the cold-work tool steels X153CrMoV12 and X165CrV12. These steels contain at least 4,8 wt.% chromium - the element responsible for the formation of permanent, finely dispersed nitrides. Its increased content increases the maximum achievable nitrogen content in the diffusion layer. X153CrMoV12 and X165CrV12 steels are referred to as high-alloyed ledeburitic steels with high chromium content. In such steels, during nitriding, the primary carbides $(\text{Cr},\text{M})_7\text{C}_3$ are transformed into nitrides as a result of the high affinity of chromium with nitrogen. During this process, the transformation of $(\text{Cr},\text{M})_7\text{C}_3$ to $(\text{Cr},\text{M})\text{N}$ takes place [8-14]. The parabolic-like growth pattern of the nitrided layer was found to be characteristic of investigated steels. On the basis of microstructure photographs, relative elemental concentration analysis and depth measurements (Figure 3), it was shown that plasma nitriding of selected tool steels, carried out for 2, 4 and 6 h, in a nitriding mixture of 5 % N_2 and 95 % H_2 and at 550 °C, leads for most of the analysed steels to the formation of diffusion/nitrided layers on their substrate [15]. They developed plasma nitriding process might be used for non-ferrous metals manufacturing process in different applications [16,17].

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