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Effect of Gas Mixture H_2-N_2 on Microstructure and Microhardness of Steel 32CDV13 Nitrided by Plasma

O. Belahssen [a]*, A. Chala [a], S. Benramache [a], B. Djamel[b], Ch. Foued[b]

[a] *Materials Science Department, Faculty of Sciences, University of Biskra, Algeria*
[b] *Mechanic Department, Faculty of Sciences and Technology, University of Biskra, Algeria*

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ABSTRACT

Nitriding treatments of low alloyed steels can be performed only at relatively low temperatures in order to avoid a decrease in corrosion resistance due to nitride layers formation. These conditions promote the formation of compound layer and diffusion zone, which shows high hardness and good corrosion resistance. In the present paper, the influence of the gas mixture N_2-H_2 in plasma nitriding process on the microstructural and mechanical characteristics of 32CDV13 steel samples was evaluated. This nuance is used in manufacturing mechanical partsthat are greatly solicited in fatigue as the transmission gearings on the helicopters' rotors and the rolling used in aeronautics. Plasma nitriding treatments were performed at temperatures in the range 773K for 4h. The modified surface layer of the nitrided samples consists mainly of the γ' and ε phases, according to metallographic technique analysis, it seems to be essentially a modification of the austenite matrix. High hardness values are observed in the modified layer with a steep decrease to matrix values.

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1. INTRODUCTION

Nitriding is a thermochemical process that is typically used to diffuse nitrogen into ferrous materials. This treatment plays an important role in modern manufacturing technologies [1]. Nitrogen ion processes are well known to improve mechanical, wear and corrosion resistance of steels. Several studies about these improvements in different steels can be found in the literature [2-11]. The basic mechanism of plasma nitriding treatment is a reaction between the plasma and the surface of the metal. In addition, depending on the steel compositions and process parameters, the plasma mass transfer has an effect on the formation and thickness of compound layer and diffusion zone [6]. Plasma nitriding owing to a number of advantages such as a lower process temperature, a shorter treatment time, minimal distortions and low energy use compared to conventional techniques has found wide application in industry [2, 3].

The aim of the present study is to investigate the microstructure and the microhardness of 32CDV13 low alloyed steel treated by ion nitriding process. The gas mixture H_2-N_2 injected into the reactor has an effect on microstructure and microhardness of steel.

2. EXPERIMENTAL

A series of experiments were carried out to investigate the plasma nitriding of 32CDV13 low alloyed steel. The chemical composition of 32CDV13 is shown in Table 1.

This steel, commonly used for nitriding, presents good toughness. The substrate surface was prepared and polished with 1μm diamond paste. Specimens were nitrided in a vacuum furnace pumped down to 10^{-3} mbar to minimise the oxygen contamination. The temperature of the samples wasmeasured using thermocouples. The nitriding parameters were fixed similar to previous works [2].

The morphology of the surfaces of samples were observed by Jeol 5900 Scanning Electron Microscope (SEM). Xray diffraction analyses with Co Kα radiations were performed to determine their structure.

*Corresponding Author Email: bel_okba@yahoo.fr (O. Belahssen)

TABLE 1. The chemical composition of steel 32CDV13

Elements	C	Si	Mn	Cr	Mo	Ni	V
% mass	0.32	0.31	0.5	3.25	0.44	0.11	0.1

The microhardness of samples is measured using microhardness tester. The morphology of the surfaces ofsamples were observed by optical microscopy and scanning electron microscope (SEM). The composition of the nitrided layers was verified by Energy Dispersive Spectroscopy (EDS). For observations by optical microscpy, the samples were prepared by chemical etching using 5% hydrofluoric acid solution. The nitrided layers were revealed at room temperature by etching the samples with Nital 2%. X-ray diffraction analyses were obtained using Co Kα tube in Bragg–Brentano geometry in the range 40° to 110°. Finally, microhardness profiles were measured to confirm the layer thickness and to evaluate its uniformity.

3. RESULTS AND DISCUSSION

3. 1. Microstructure The compound layer thickness and diffusion zone of the plasma nitrided 32CDV13 low-alloy steel depending on the N_2 in the gas mixture are shown in Figure 1. It can be observed that thickness of compound layer and diffusion zone increases with increase of N_2 at the gas mixture in plasma, at temperature 773K and 4h treatment time.

The micrographic SEM of sample nitrided during 4h in gas mixture (20% H_2 - 80% N_2) at 773K (Figure 2) shows the formation of compound layer (white layer) which increases during the process to achieve a thickness around 5µm. The layer thickness is the most important in this case. The addition of hydrogen in concentration range of 20–40% results in thicker layers and enhanced surface hardness compared with treatment in pure nitrogen. Excessive amounts of hydrogen retard the nitriding process [12].

EDS microanalysis showed that the nitrided layer contained a high amount of nitrogen on the surface and the nitrogen concentration decreased along with the increase of the distance from surface until the substrate value at a depth of about 100–150 µm (Figure 3).

3. 2. X-ray Diffraction Treatment of nitriding by plasma at 773K and 4h of treated time produced different nitrided layers in terms of morphology, thickness and phase structure: α phase (corresponding to the steel matrix), the ε-$Fe_{2-3}N$ phase and the γ'-Fe_4N phase. XRD analysis was performed on treated samples (Figure 4). To demonstrate the effect of the treatment, the diffraction pattern obtained from the untreated material is displayed in the same graph.

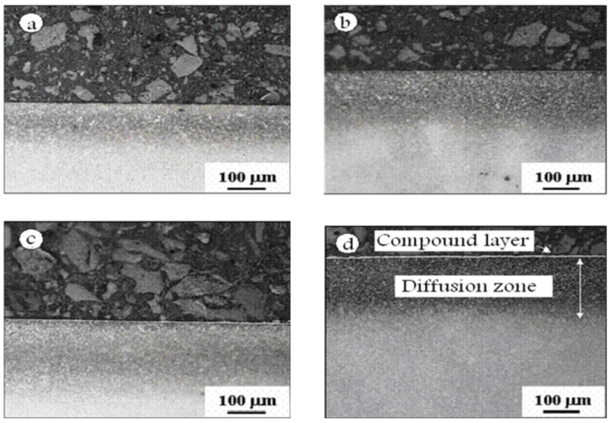

Figure 1. Optical micrographs plasma nitrided low alloyed steel 32CDV13 at 773 K and 4 h treatment time: (a) 20%N_2, (b) 60%N_2, (c) 80%N_2, (d) 100%N_2.

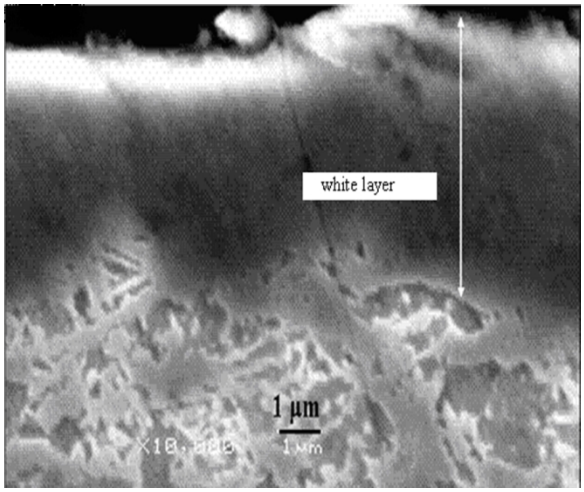

Figure 2. Micrographic SEM of sample nitrided during 4h in gas mixture (20% H_2 - 80% N_2) at 773 K.

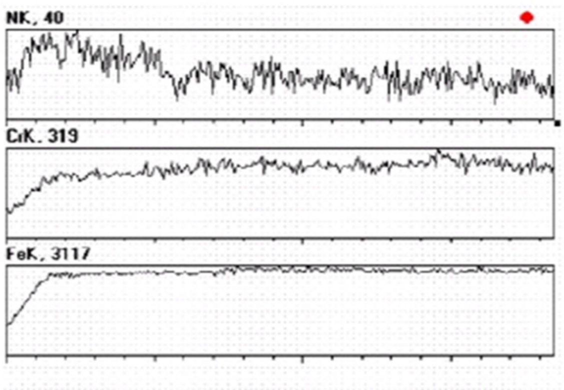

Figure 3. Concentration profile of elements N, Cr et Fe.

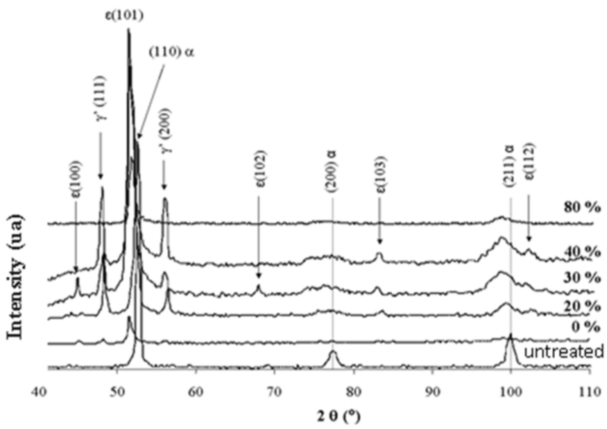

Figure 4. XRD patterns of samples treated at 773 K for 4 h of treatment at different nitrogen percentage.

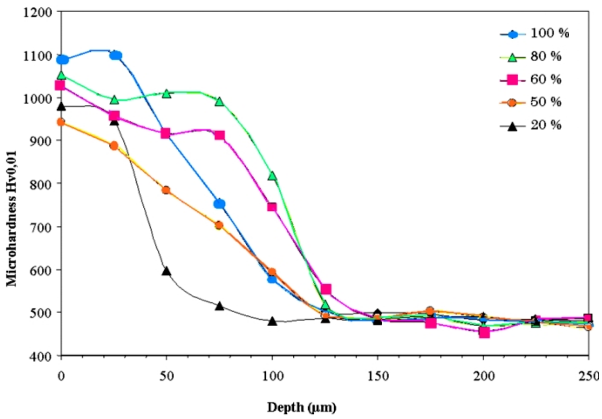

Figure 5. Micro-hardness profiles of samples treated at 773 K for 4 h of treatment at different nitrogen percentage.

When the XRD patterns were examined, it was oberved that both γ'-Fe_4N and ε-$Fe_{2-3}N$ phases have formed and the intensity of this phases in the compound layer is higher in the process, while the N_2 increases in gas mixture.

On increasing nitrogen, the α phase vanishes in thicker nitrided layers, its contribution becomes less intense to the point of disappearing.

The XRD patterns shown in Figure 4 indicate that treated samples consist of a mixed structure of γ'-Fe_4N and ε -$Fe_{2-3}N$. However, the relative peak intensities of the two phases are different in samples with different conditions. It is possible to infer that the presence of ε and γ' iron nitrides is determinant to produce the higher hardness [13].

3. 3. Microhardness Figure 5 shows microhardness profiles of samples treated at 773K for 4h of treatment time at 20/80, 50/50, 60/40, 80/20 and 100/0 of N_2-H_2 gas mixture. Microhardness profiles obtained from cross-sections of treated specimens show the presence of a slope interface between the case (nitrided layer) and the core. All samples show high surface microhardness values that drop decreasingly at the case/core interface to substrate microhardness values. As seen in the Figure 4, higher surface hardness values and big depth are obtained at 80% N_2 + 20% H_2 gas mixture. We can see a higher hardness to 100% N_2 in near of the sample surface, but this value decreases to a depth of about 50 µm which explains the role of H_2 in the diffusion of nitrogen in the substrate. These results are in good accordance with those of Krishnaraj et al. [14] who studied the mechanical properties of plasma nitrided steel. Priest and al. [15]studied the effect of hydrogen in the case of nitriding to low pressure of steels. They showed that hydrogen have an effect on the diffusion of nitrogen.

4. CONCLUSION

The microstructure and mechanical properties of low alloyed steel 32CDV13 nitrided by plasma were studied as a function of concentration of gas mixture. The results obtained after 4 hours of treatment, in gas mixture (80% N_2, 20% H_2) at 773K show that a compound layer and diffusion zone was formed. The compound layer corresponds mainly to $Fe_{2-3}N$ and Fe_4N iron nitrides and it has been observed that increasing nitrogen in plasma increases significantly the compound layer and the diffusion zone and improves the mechanical properties.

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[a] *Materials Science Department, Faculty of Sciences, University of Biskra, Algeria*
[b]*Mechanic Department, Faculty of Sciences and Technology, University of Biskra, Algeria*

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چکیده

عملیات نیتروژن‌دهی فولاد کم‌آلیاژ به علت تشکیل لایه‌های نیترید می‌تواند تنها در دمای نسبتاً پایین به منظور اجتناب از کاهش مقاومت در برابر خوردگی انجام شود. این شرایط، تشکیل لایه‌مرکب و منطقه نفوذ که سختی بالا و مقاومت خوب در برابر خوردگی دارد را مناسب می‌سازد. در این مقاله تاثیر مخلوط گاز N_2-H_2 در فرایند نیتریدی پلاسما بر ویژگی‌های ریزساختاری و مکانیکی نمونه‌ی فولاد 32CDV13 مورد بررسی قرار گرفت. این نکات جذاب در ساخت قسمت‌های مکانیکی که در معرض خستگی زیادی هستند، مانند چرخ‌دنده‌ی انتقال در روتور هلیکوپتر و غلتک مورد استفاده در هوانوردی استفاده می-شود. عملیات نیتروژن‌دهی پلاسما در دمای ۷۷۳K به مدت ٤ ساعت انجام شد. لایه‌ی اصلاح شده‌ی سطح نمونه نیتروژن‌دهی با توجه به تحلیل‌های متالوگرافی‌عمدتاً شامل فازهای γ' و ε است، که به نظر می‌رسد در اصل یک ماتریس اصلاح شده‌ی آستنیت باشد. سختی بالا در لایه‌ی اصلاح شده با کاهش شیب‌دار به سمت مقادیر ماتریس مشاهده شده است.

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