Abstract
This paper presents a study of microstructure and mechanical properties of low alloyed steel 32CDV13 treated by plasma nitriding. This nuance is used in manufacturing mechanical pieces that are greatly solicited in fatigue as the transmission gearings on the helicopters' rotors and the rolling used in aeronautic; an ion nitriding treatment was carried out. The increase of the nitrogen percentage, at a middle temperature and at determined treatment time, conducts to the formation of a compound layer, increases significantly the diffusion layer thickness and improves the mechanical properties. With the traditional techniques of analysis as the optic microscopy, the scanning electron microscopy and microhardness tests we can determine the microstructural and the mechanical properties of nitried layers.

Keywords: Ion nitriding; Steel 32CDV13; Microstructure; Microhardness.

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]. It can improve surface hardness, fatigue strength, wear and corrosion resistance. 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 [2–13].
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 [7].
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 work is to study the effect of gas mixture (N2-H2) on the microstructure of 32CDV13 low alloyed steel treated by ion nitriding process.

2. EXPERIMENTAL
A series of experiments were carried out to investigate the plasma nitriding of low alloyed steel 32CDV13. The chemical composition of low alloyed steel 32CDV13 is shown on (Tab. 1).
pressure ($10^{-3}$ mbar) to minimize the oxygen contamination. The temperature is measured using thermocouple. The nitriding parameters were fixed similar to previous works [2,7].

The samples morphology surfaces were observed by optical microscopy and scanning electron microscope (SEM). The composition of the nitried layers was verified by Energy Dispersive Spectroscopy (EDS). The specimens for optical microstructure observation were prepared by chemical etching using 5% hydrofluoric acid solution. The nitried layers were revealed, at room temperature, by etching the samples with Nital 2% (2% v/v nitric acid in absolute ethanol). X-ray diffraction analyses were obtained by using Co Kα tube in Bragg–Brentano geometry in the range from 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 Subchapter title

The compound layer thickness and diffusion zone of the plasma nitried 32CDV13 low-alloy steel depending on the N$_2$ in the gas mixture are shown in (Fig. 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 773 K and 4 h treatment time.

![Fig. 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$_2$](image)

The micrographic SEM of sample nitried during 4h in gas mixture (20% H$_2$ - 80% N$_2$) at 773 K (Fig. 2) shows the formation of compound layer (white layer) which increases during the process to achieve a thickness around 5 μm. 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.
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 (Fig. 3).

### 3.2 X-ray diffraction

Treatment of nitriding by plasma at 773 K and 4 h 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$_4$N phase. XRD analysis was performed on treated samples (Fig. 4). To demonstrate the effect of the treatment, the diffraction pattern obtained from the untreated material is displayed in the same graph.

When the XRD patterns were examined, it has been seen that both γ'-Fe$_4$N 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$_4$N and ε-Fe$_{2-3}$N. However, the relative peak intensities of the two phases are different in samples with different...
conditions [13]. It is possible to infer that the presence of ε and γ' iron nitrides is determinant to produce the higher hardness.

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

### 3.3 Micro-hardness measurements

(Fig. 5) shows micro-hardness profiles of samples treated at 773 K for 4 h of treatment time at 20/80, 50/50, 60/40, 80/20 and 100/0 of N₂-H₂ gas mixture. Micro-hardness 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 micro-hardness values that drop decreasingly at the case/core interface to substrate micro-hardness values. As seen in the (Fig. 4), higher surface hardness values and big depth are obtained at 80% N₂ + 20% H₂ gas mixture. We can see a higher hardness to 100% N₂ in near of the sample surface, but this value decreases to a depth of about 50 μm which explains the role of H₂ 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.

**Fig.5.** Micro-hardness profiles of samples treated at 773 K for 4 h of treatment at different nitrogen percentage
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 show that after 4 hours of treatment and in gas mixture (80% N₂, 20% H₂) at 773K a compound layer and diffusion zone was formed. The compound layer corresponds mainly to Fe₂₃N and Fe₄N 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.

LITERATURE


