

Available online at www.sciencedirect.com

Physics Procedia 2 (2009) 1399–1403

**Physics
Procedia**www.elsevier.com/locate/procedia

Proceedings of the JMSM 2008 Conference

Growth kinetics of the compound layers: Effect of the nitriding potential

M. Keddam*, B. Bouarour, R. Kouba, R. Chegroune

Département S.D.M, Faculté de Génie Mécanique et Génie des Procédés, USTHB, B.P.32 El-Alia, Bab-Ezzouar 16111 Algiers, Algeria

Received 1 January 2009; received in revised form 31 July 2009; accepted 31 August 2009

Abstract

The aim of this work is to study the effect of the nitriding potential on development of the compound layers during the gas nitriding of Armco Fe and XC38 carbon steel. The gas nitriding experiments were realized in an atmosphere of partially dissociated gas ammonia (NH₃) at 520 °C under a variable nitriding potential (from 0.25 to 3.5 atm^{0.5}) for 2 h.

Using XRD analysis and SEM observations of the cross-sections of the treated samples, it was shown that the compound layer was composed of γ' iron nitride after exceeding a critical value of the nitriding potential that depends on the substrate's nature.

A linear semi-logarithmic relationship relating the nitriding potential to the experimentally measured layer thickness for the γ' phase was used to evaluate the critical nitriding potential giving rise to its formation on the material substrate. It was found that the required value of the critical nitriding potential for XC steel is greater than that of Armco iron.

© 2009 Elsevier B.V. Open access under [CC BY-NC-ND license](https://creativecommons.org/licenses/by-nc-nd/4.0/).

Keywords: Nitriding; Growth kinetics; Iron nitride; Compound layer; Nitriding potential; XRD analysis.

1. Introduction

Gas nitriding is a thermochemical process used to improve surface properties such as wear, fatigue and corrosion resistance, promoting the increase of the useful life time of the treated workpieces [1]. It involves the diffusion of atomic nitrogen into the substrate's surface to form the iron nitrides (ϵ and/ or γ') in the compound layer, followed by a relatively thick diffusion zone. The microstructural nature of the compound layer depends upon the nitriding potential to generate either a mono (γ' phase) or biphasic configuration ($\epsilon + \gamma'$) at a given temperature [2, 3]. This nitriding potential, which is a fundamental parameter of this treatment, is given by the relationship between ammonia and hydrogen partial pressures. It reflects the ability of gas mixtures and temperatures of introducing nitrogen into the sample [4].

In this work, a series of samples from Armco iron and XC38 carbon steel were gas nitrided at 520°C during 2 h under a variable nitriding potential ranging from 0.25 to 3.5 atm^{0.5} in order to evaluate, from the experimental data, the critical value of the nitriding potential which allows the formation of γ' phase.

* M. Keddam. Tel.: +213 21 24 79 19; fax: +213 21 24 79 19
E-mail address: keddam@yahoo.fr.

2. Experimental details

Armco iron and XC38 carbon steel of chemical composition given in table 1 were used for the gas nitriding experiments. Prior to the gas nitriding treatment, the Armco iron samples were annealed at 900°C for 1 h followed by a slow cooling in the furnace. The samples from XC38 carbon steel were austenitized at 850°C during 1 h, water-quenched and tempered at 600°C for a holding time of 1.5 h. The used nitriding apparatus is a laboratory vertical quartz tube furnace which allows a change of the nitriding potential in a retort as well as precise setting of its value. The gas flow was controlled with Bronkhorst mass flow controllers and the linear flow rate of the gas mixture through the quartz retort was 1.4 cm s⁻¹. The gas nitriding treatment was then carried out in an atmosphere of partially dissociated gas ammonia (NH₃) at 520 °C (controlled by a thermocouple within ± 2°C) during 2 h under different nitriding potentials: (0.25, 0.8, 1.5, 2 and 3.5 atm^{-0.5}). The cross-sections of the samples were mechanically polished, followed by fine polishing with alumina slurry, and etched with 3% Nital. The nature of the compound layers was identified by XRD analysis (XPert Philips X-ray Diffractometer using Co K_α, λ=0.178897 nm). The cross-sections of the samples were observed by Scanning Electron Microscopy (SEM-JOEL 5600 LV).

Table 1 Chemical composition of the investigated materials by GDOS analysis.

Wt. %	C	Si	Cr	Mn	Mo	Ni	P	S
Armco iron	0.003	0.003	0.001	0.01	0.02	0.01	0.014	0.003
XC38	0.38	0.27	0.25	0.66	0.02	0.02	0.02	0.01

3. Characterization of the compound layers

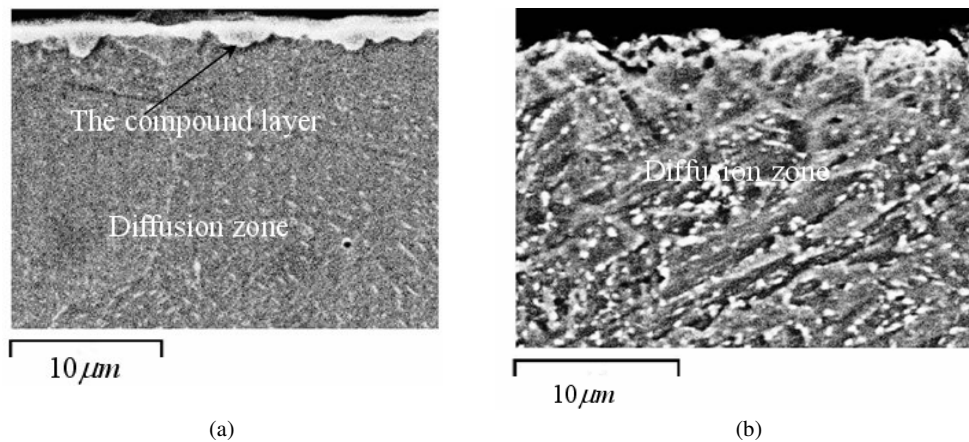


Fig. 1 SEM photomicrographs of the cross-sections of the samples nitrided at 520°C during 2 h under a nitriding potential of $K_N = 0.8 \text{ atm}^{-0.5}$. (a) Armco Fe, (b) XC38 steel.

Fig.1 shows SEM photomicrographs of the cross-sections of the samples treated at 520°C under a nitriding potential equals to $K_N = 0.8 \text{ atm}^{-0.5}$. The average thickness of the compound layer is 0.8 μm for the Armco iron sample. The compound layer is absent for the sample from XC38 steel.

Fig.2 presents the cross-sectional photomicrographs obtained by SEM of the samples nitrided at 520°C for 2 h with $K_N = 2 \text{ atm}^{-0.5}$. For Armco iron sample, the compound layer which has a thickness of 2 μm is formed on top of the diffusion zone where dense and fine precipitates of γ iron nitrides are present inside the ferrite grains. The compound layer reaches a value of 2.48 μm for XC38 steel.

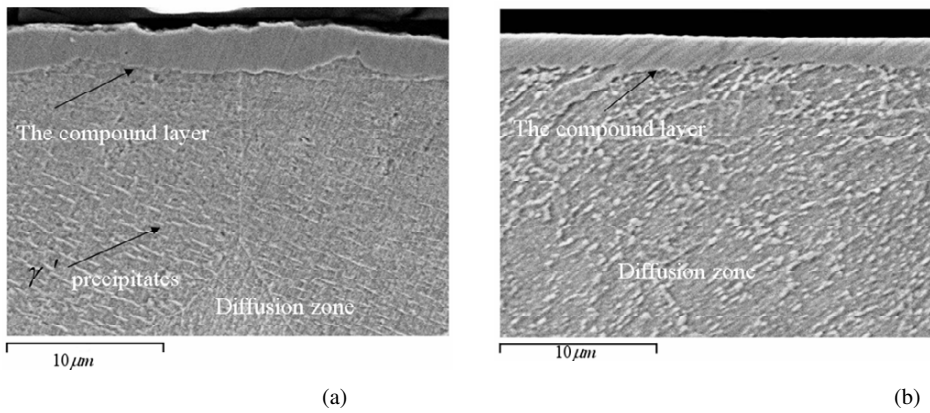


Fig. 2 SEM photomicrographs of the cross-sections of the samples nitrided at 520°C during 2 h under a nitriding potential of $K_N = 2 \text{ atm}^{-0.5}$: (a) Armco Fe, (b) XC38 carbon steel.

Table 2 provides the values of the layer thickness of γ' phase as a function of the nitriding potential [5, 6]. The obtained values were measured from SEM observations of the cross-sections of the treated samples. Each reported value is a mean of at least six measurements. From this Table, it can be seen that the γ' layer thickness grows as the nitriding potential increases.

Table 2: Change in the γ' layer thickness as a function of the imposed nitriding potential

$K_N (\text{atm}^{-0.5})$	0.25	0.8	1.5	2	3.5
	Thickness of γ' layer (μm)				
Material					
Armco Fe	0	0.8	1.64	2.07	2.70
XC38	0	0	1.10	2.48	2.52

Fig.3 gives the XRD patterns of the treated samples at 520°C for 2 h under various nitriding potentials. In Fig.3a, it is observed that no iron nitride is formed for $K_N = 0.25 \text{ atm}^{-0.5}$, whereas the two phases α and γ' are present for $K_N \geq 0.8 \text{ atm}^{-0.5}$ for Armco iron. For XC38 steel (Fig. 3b), the γ' iron nitride appears for a nitriding potential of $K_N \geq 1.5 \text{ atm}^{-0.5}$.

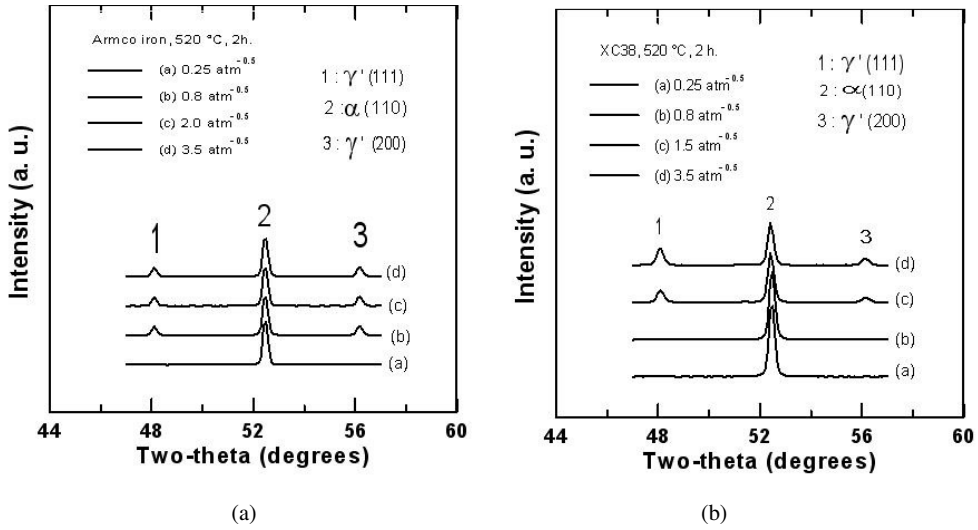


Fig.3 XRD patterns of Armco Fe and XC38 steel gas nitrided at 520 °C for 2 h at different nitriding potentials.

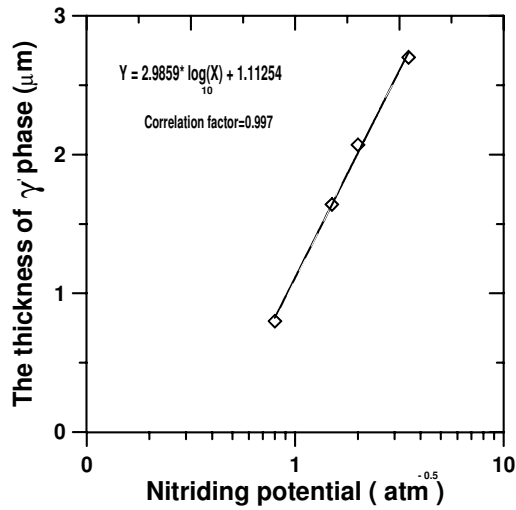


Fig. 4 Variation of the γ' layer thickness versus nitriding potential.

The experimentally determined values of the γ' layer thicknesses for Armco iron are plotted versus nitriding potential in Fig.4. The experimental points were well fitted by a semi-logarithmic curve and a good agreement was obtained. Eq.1 describes the variation of the γ' layer thickness as a function of nitriding potential and it is given by:

$$x_{\gamma'} = 1.2968 \ln(K_N) + 1.11254 \tag{1}$$

For Armco iron, the critical value of the nitriding potential corresponding to the formation of γ' iron nitride can be estimated from Eq.1 as $0.424 \text{ atm}^{-0.5}$. For XC38 steel, it can also be possible to estimate the critical nitriding potential for generating the γ' phase using the same mathematical form as Eq.1:

$$x_{\gamma'} = \alpha \ln(K) + \beta \quad (2)$$

where α and β are the constants to be determined from the experimental data of Table 2, and corresponding to the values of nitriding potential (1.5 and $2 \text{ atm}^{-0.5}$) for which the γ' layer thicknesses are, 1.10 and $2.48 \mu\text{m}$, respectively. Eq.2 is then rewritten as:

$$x_{\gamma'} = 1.1926 \ln(K_N) - 0.845 \quad (3)$$

From Eq. 3, the critical value of nitriding potential is found to be equal to $1.192 \text{ atm}^{-0.5}$ for XC38 steel. It can be noted that this value is greater than that of Armco iron. This difference can be attributed to the presence of carbon element in this steel, which retards the appearance of γ' phase in comparison to Armco iron.

4. Conclusions

In this present work, the effect of the nitriding potential on development of the compound layers during the gas nitriding of Armco Fe and XC38 carbon steel was taken into consideration and the following concluding points are drawn as follows:

1. From XRD analysis, the compound layer is composed of a single phase (γ' iron nitride) and its formation depends on the imposed nitriding potential.
2. From SEM observations, the thickness of the compound layer increases when varying the nitriding potential. The maximal value of the γ' layer thickness is obtained for Armco iron and under a nitriding potential of $3.5 \text{ atm}^{-0.5}$.
3. A linear semi-logarithmic relationship relating the nitriding potential to the γ' layer thickness was used to predict the threshold value which allows its formation. The critical values of nitriding potential were estimated as $1.192 \text{ atm}^{-0.5}$ for XC38 steel and $0.424 \text{ atm}^{-0.5}$ for Armco iron.

Acknowledgements

This work was carried out in the framework of CNEPRU project under code number J0300220070045 of the Algerian ministry of high education and scientific research.

References

- [1] Source Book on nitriding, ASM, Metals Park, OH, 1977.
- [2] M. Keddam, Defect Diff. Forum, 258-260 (2006) 172.
- [3] M. Keddam, M. E. Djeghlal, L. Barrallier, Applied Surf. Sci. 242 (2005) 371.
- [4] E.J. Mittemeijer, M.A.J. Somers, Surf. Eng. 13 (1997) 483.
- [5] M. Keddam, B. Bouarour, R. Kouba and R. Chegroune, Defect Diff. Forum, 283-286 (2009) 133.
- [6] B. Bouarour, Mémoire de Magister, USTHB, 2008 (In French)