

## A STUDY OF PLASMA NITRIDED LAYER THICKNESS TIME RELATIONSHIP

M.M. TOŠIĆ, R. GLIGORIJEVIĆ\*, I. TERZIĆ, Z. RAKOČEVIĆ

*The Boris Kidrič Institute of Nuclear Sciences - Vinča  
The Institute for Atomic Physics, P.O.Box 522, Yu-11001 Belgrade  
\*Motor Industry, Rakovica, Yu-11000 Belgrade*

### 1. Introduction

Plasma processing involves the extended exposure of a metal surface to any ionized and dissociated gas in a glow discharge at a temperature sufficiently high to permit the active plasma species to react with the surface of the treated specimen which serves as a cathode of a glow discharge diode. The altered surface properties such as increased hardness and wear resistance are a direct result of the chemical changes introduced in the surface. In plasma nitriding the atomic nitrogen introduces into the ferrite phase at the temperatures below the eutectic temperature in the binary Fe-N system. The single most important feature of plasma nitriding lies in its capability to control precisely the microstructure of treated components producing the desired physical properties to meet the demand of services<sup>1</sup>. The aim of work is to study the time dependences of diffusion layer and compound zone thicknesses of plasma nitrided surface layers.

### 2. Experimental conditions

Schematic diagram of the experimental setup used in this work is shown in Fig.1. It comprises<sup>1</sup> essentially of vacuum chamber, the mechanical vacuum pump, DC power supply unit, the unit to adjust and control glow discharge parameters and gas supply and control unit. Treated samples are connected cathodically and positively charged ions bombard their surfaces supplying them with energy. The temperature of workpieces is measured by an insulated chromel-alumel thermocouple.

Samples of four different iron based materials have been treated: (i) JUS C.4732 steel (chemical composition: 1,1%Cr; 0,25%Mo; 0,9%Mn; 0,35%Si; 0,4%C) with the tensile strength  $R_m = 1$  GPa (the hardness of 310 HB); (ii) JUS C.5420 steel (chemical composition: 1,5%Cr; 1,4%Ni; 0,71%Mn; 0,35%Si; 0,18%C) with the hardness of about 200 HB; (iii) pearlitic-sorbic nodular cast iron JUS NL.80 (chemical composition: 3,06%C; 2,63%Si; 0,42%Mn; 0,53%Cu) with the tensile strength of 860 MPa (the hardness of 290 HB); and (iv) powder metal sintered steel (chemical composition: 7,35%Cr; 4,32%Mo; 1,27%Cu; 1,37%C) with the hardness of about 300 HB. The effects of plasma nitriding temperature, type of gas mixture and glow discharge parameters have been previously investigated to get optimum results. Before plasma nitriding the samples were hydrogen sputter etched for 1/2 h. The nitriding temperature was 520 C, the process was performed for 3 h, 5 h, 7,25 h, 9,25 h, 16 h, 25 h, 50 h and 75 h in different gas mixtures of  $N_2$ ,  $H_2$  and  $CH_4$  at the pressure in the mbar range to

produce monophase compound layers of  $\gamma'$ -Fe<sub>4</sub>N and  $\epsilon$ -Fe<sub>2-3</sub>N. Evaluation of nitrided surfaces was conducted by optical microscopy, X-ray diffractometry, and microhardness measurements.

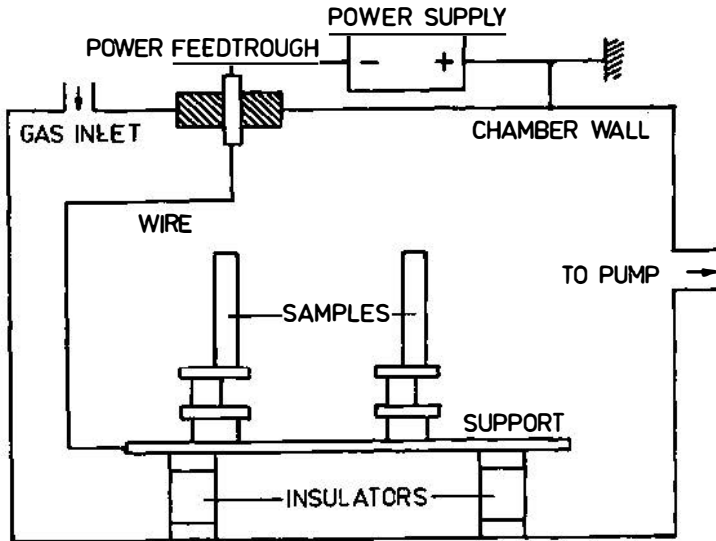


Fig.1. Schematic diagram of the experimental setup (see text).

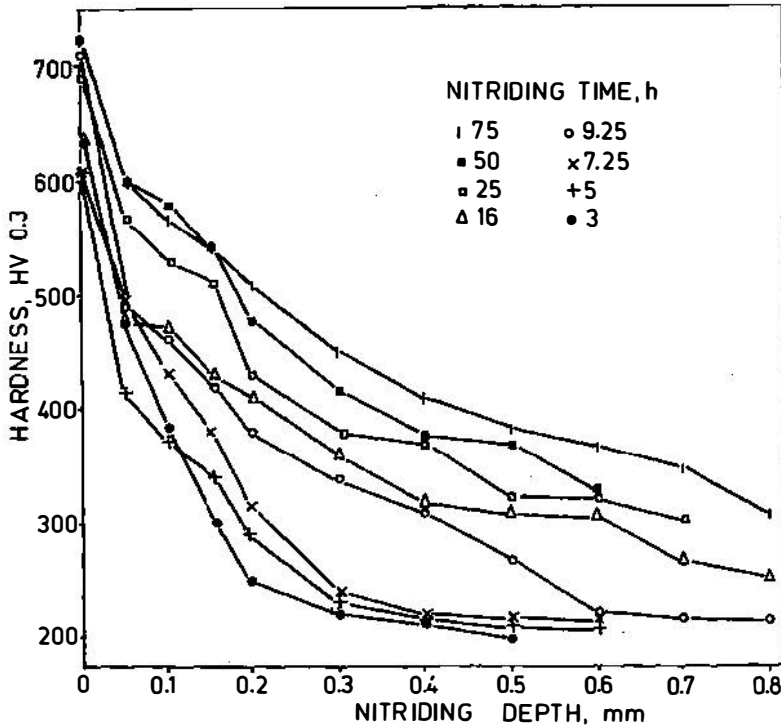


Figure 2. Microhardness depth profiles of plasma nitrided samples of JUS C.4732 steel (corresponds to DIN 42CrMo4) as a function of nitriding time: working gas is 80%N<sub>2</sub>+20%H<sub>2</sub> at the pressure of 2,66 mbar, nitriding temperature is 520 C.

### 3. Results

Nitrided layer thickness was measured by microhardness depth profiling at the cross-sections along the normal to the surfaces of the samples. The compound layer thickness was measured by optical microscopy; their phase compositions have been identified by X-ray diffractometry methods. Both monophasic compound layer types have been produced on the samples of all iron materials. This fact is of great value because these layers are responsible for very good wear resistance properties<sup>2,3</sup>.

Microhardness depth profile of plasma nitrided samples of the steel C.4732 for different nitriding times is shown in the Fig.2. It can be seen that nitrided layer thickness grows with time. In order to evaluate the functional relation, in Fig.3. this dependence is plotted by taking  $(\text{time})^{1/2}$  as the argument. It can be seen that the straight lines are good fits. This means that the plasma nitrided layer thickness grows according to the diffusion law, as it is well known for the case of the gas nitriding process. In the same figure it is shown that the same conclusion is valid for other types of iron materials treated in this work. This conclusion is of great value because it has been shown that diffusion layer is very important for the improvements of fatigue properties achieved with plasma nitrided crankshafts<sup>4</sup>.

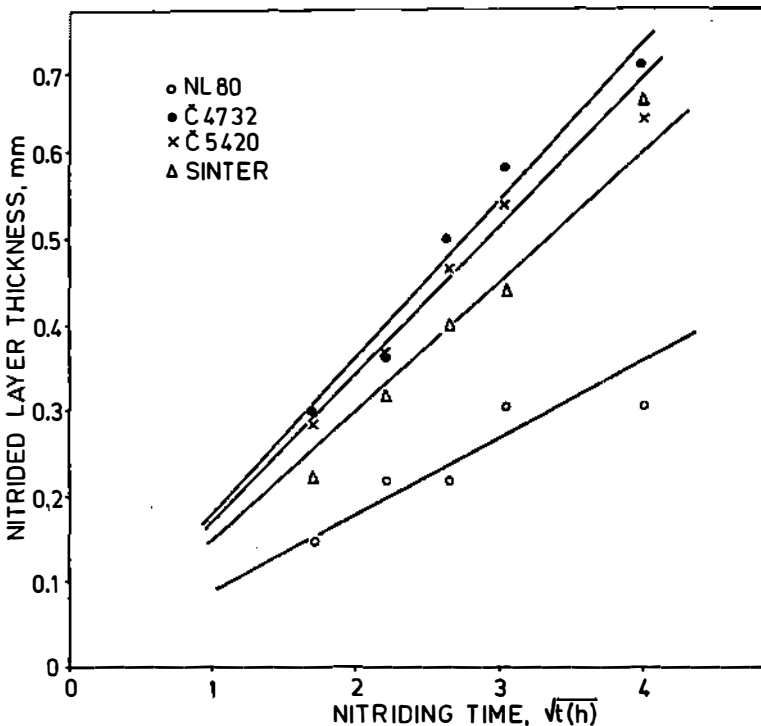


Figure 3. Nitrided layer thickness of JUS C.4732 and JUS C.5420 steels, nodular cast iron NL.80 and powder metal sintered steel as a function of time; nitriding parameters as in Fig.2.

Compound zone thicknesses of nitrided layers as functions of time are shown in the left part of Fig.4. It can be seen that the character of their growth is different than the previous one. In order to study it in more details, the surface microhardnesses as functions of time are shown in the right part of Fig.4. It can be seen that the time dependences of the compound layer thickness and of surface microhardness are very similar, indicating that after some time the equilibrium between the quantity of nitrogen introduced in the very surface of workpiece and those diffused into the bulk and sputtered back from the very surface into the vacuum is reached. This behaviour is valid only for plasma nitrided compound layers. It is known that in gas nitriding the compound layer grows steadily with time, the consequence of that being that in order to produce thick diffusion layer it has to produce thick compound layer, too. This is unacceptable because there is no control of the layer type being the mixture of two conflicting phases with bad mechanical properties, what means that it has to be removed.

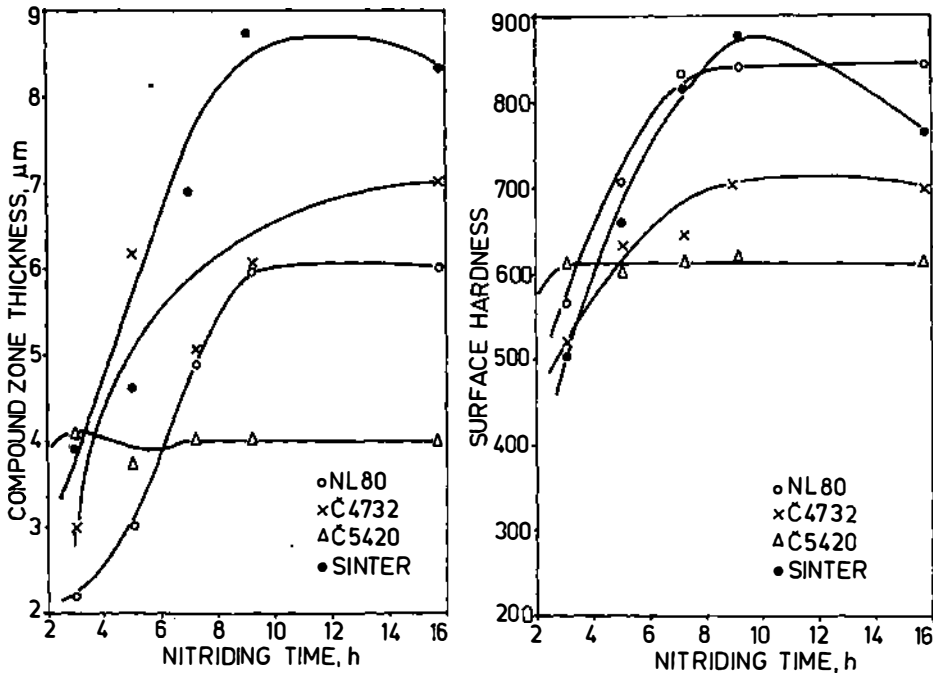
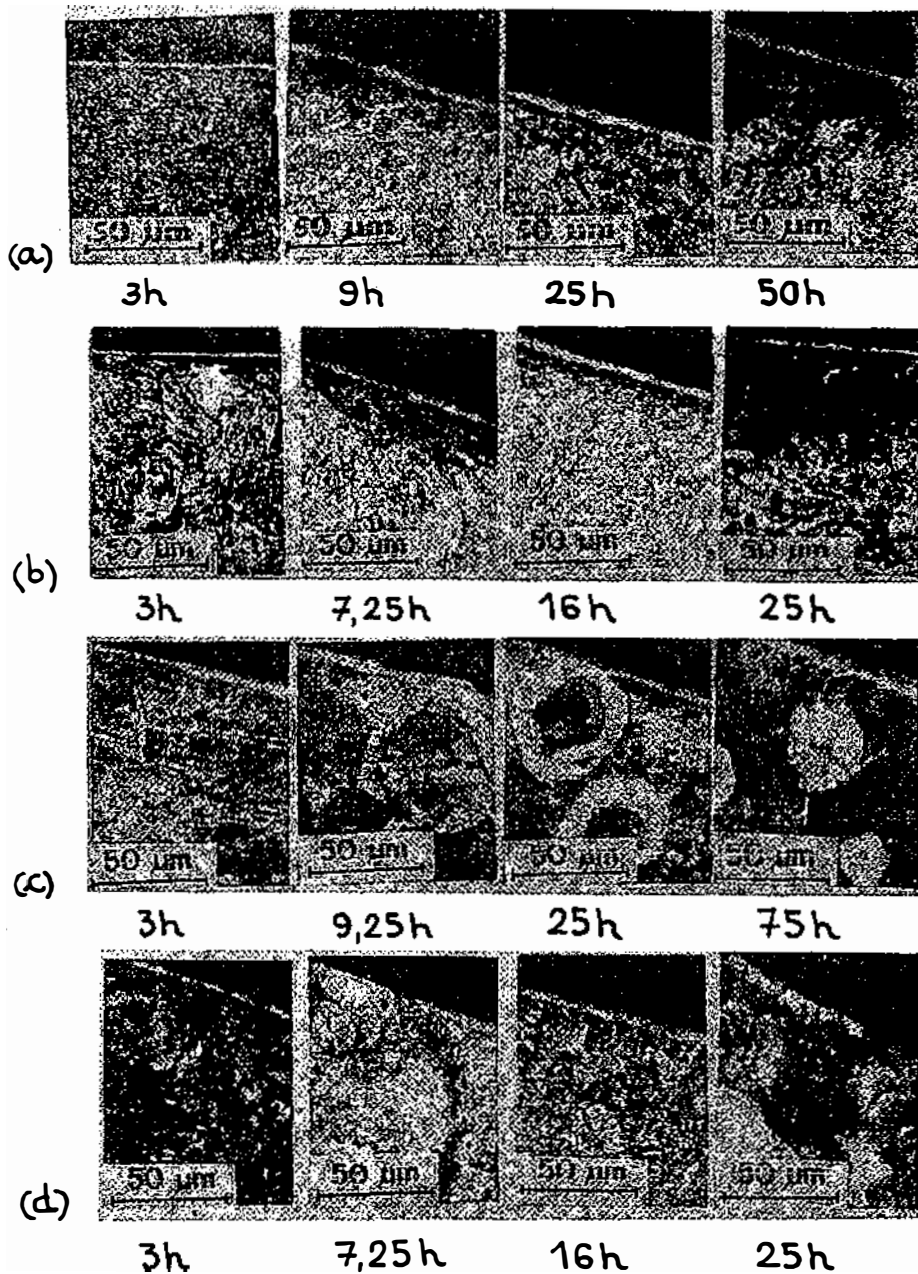


Figure 4. Compound zone thickness (left) and surface hardness (right) of nitrided layers of JUS C.4732 and JUS C.5420 steels, nodular cast iron NL.80 and powder metal sintered steel as a function of time; nitriding parameters as in Fig.2.

Optical photomicrographs of the cross-sections of plasma nitrided layers of the steels C.4732, C.5420, nodular cast iron NL.80 and powder metal sintered steel are shown in Fig.5. These are the raw data used to plot the curves shown in Fig.4.

The parameters to be considered during the evaluation of the results are<sup>5</sup>: the neutrals impingement rate about  $10^{20} \text{ cm}^{-2} \text{ s}^{-1}$ ,



**Figure 5. Optical photomicrographs of the cross-sections of the plasma nitrided layers of JUS C.4732 steel (a), JUS C.5420 steel (b), nodular cast iron NL.80 (c) and powder metal sintered steel (d) for different plasma nitriding times (time grows from the left to the right); other nitriding parameters as in Fig.2.**

viscous flow, mean free path 0,01 cm, monolayer time  $10^{-5}$  s, the bombarding ions rate about  $10^{16}$   $\text{cm}^{-2}\text{s}^{-1}$  and energetic particles impingement rate about  $10^{17-18}$   $\text{cm}^{-2}\text{s}^{-1}$ . The data show that the most numerous encounters with surface atoms are those of low energy neutral gas species, being (2-4) orders of magnitude greater than those originating from glow discharge plasma. The relationship shown in Fig.4. means that the mechanism of compound layer growth is dependent mainly on the concentration of energetic particles that are taking part in the collisions with the very surface. This conclusion could have serious influence on the formulation of quantitative description of plasma nitriding process.

#### 4. Conclusions

From the data presented in this work the following conclusions have been obtained:

(i) The plasma nitrided diffusion layer grows following the diffusion law, as in the gas nitriding process. As diffusion layer is much thicker than compound layer, the same conclusion is valid for nitrided layer as a whole.

(ii) The compound layer thickness shows the saturation reached after short nitriding time, indicating that there is an equilibrium between the quantities of nitrogen introduced in it and leaving it by sputtering and diffusion processes. This fact is of great technological value, enabling the control of the nitrided layer properties being mainly dependent on compound layer properties.

(iii) Based on the data on the glow discharge plasma basic parameters it has been found that the previous conclusion is due to the presence of energetic particles that interact with the very surface of plasma nitrided samples. This could help in the formulation of quantitative description of the process.

#### References

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