A Study of Plasma Nitriding Process on the AISI 4140 Steel

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Abstract

In the present investigation, pulsed dc glow discharge plasma nitriding process on the AISI 4140 steel has been carried out at three different temperatures i.e., at 450℃, 475℃ and 500℃ for 10 hrs duration in order to achieve good nitride results. The microstructure of the base material and the nitrided layer has examined by X-ray diffraction. Hardness property, has investigated for both un-nitrided and plasma-nitrided materials. It is observed that the microstructure of the core material remains unaltered and Iron Nitride has formed in the hardened surface layer after plasma nitriding at all the three temperatures employed. Surface hardness increases substantially after plasma nitriding. Surface hardness increases with the increase in plasma nitriding temperature due to greater diffusivity at higher temperatures.

Introduction

The bond structure in transition metal nitrides consists of a mixture of covalent, metallic and ionic components, and is responsible for high hardness, excellent wear resistance, chemical inertness, good electrical conductivity and superconducting properties. The interest in thin films of metal nitrides is growing rapidly for such diverse applications as wear resistant coatings on cutting tools, and as diffusion barriers in integrated circuits. The importance of this process has grown from the fact that, through nitriding, very hard surface layers may be obtained without substantial modification of the bulk material properties. A number of processes are available by which a wide variety of alloys can be nitride. Plasma Nitriding process has become industrially important [l-3]. Number of reports on this process has been published [4-9].

In this paper, we have taken low alloy steel AISI 4140. These steels are designed to provide better mechanical properties than conventional carbon steels. The steels have low carbon contents (0.50 to ~0.25 weight percent C) in order to produce adequate formability and weldability, and they have manganese contents up to 2.0 weight percent. These steels are also called high strength low alloy (HSLA) steels.

In this study, plasma nitriding parameters were examined to reveal optimal nitriding conditions originating from gas mixture, temperature and process time.

Experimental

Materials and sample preparation

Low alloy steel AISI 4140 was selected for the present study and its chemical composition are shown in the table 1.

Table 1: Chemical composition of the alloy AISI 4140(wt. %)

Elements	wt.%
C	0.38-0.43
Mn	0.75-1.00
P	0.035 (max)
S	0.04 (max)
Si	0.15-0.30
Cr	0.80-1.10
Mo	0.15-0.25
Fe	Balance

The samples were in cylindrical shaped of height 5 mm (approx.). Samples were initially polished to give a mirror finish appearance. Before plasma nitriding, the prepared samples ultrasonically cleaned with acetone and dried. All the samples were plasma nitrided in three batches using three different temperatures with the same gas composition.

Plasma nitriding process

This process was carried out in a 500 mm diameter and 500 mm height bell shaped stainless steel vacuum chamber. Mechanically prepared four different samples of different kind of steels were grouped and mounted on the sample holder of the vacuum chamber. Initially, the vacuum chamber was evacuated to a base pressure of 0.05 mbar by a rotary pump. The samples were first sputter cleaned using N_2–H_2 gas mixture in 25%:75% ratio. The gas flow rate was controlled by the butterfly valves mount on the vacuum chamber to maintain a pressure of 1 mbar. Plasma was generated using a D.C. pulsed power supply.

Sputter cleaning process was performed for 1 h at 250℃ to remove the native oxide layer and contamination so as to expose a fresh surface of the samples for plasma nitriding. The temperature of the samples was monitored with a Fe-Constantan-Fe thermocouple (J-type), which is attached to the sample holder

through the tubular support. There is an error of ±5°C in the temperature readings. After completing the sputter cleaning process, the mixture of nitrogen and hydrogen gas was introduced in the reactor for plasma nitriding. Plasma nitriding was carried out using three different temperatures 450°C, 475°C and 500°C at same gas mixtures (25% N_2 and 75% H_2) under a pressure of 4.5 mbar for 10 hrs. After 10 hrs these nitrided samples were cooled in vacuum chamber under the flow of the gas mixture till the temperature decreased to 150°C. This is mostly done to eliminate formation of oxide layer on the surface of the samples.
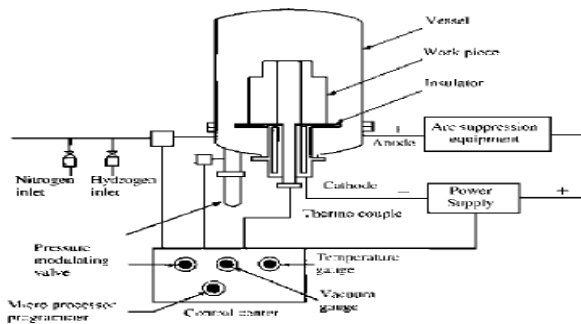

Fig. 1. Schematic of the ion nitriding set up.

Figure 1: Schematic of the ion nitriding setup

After plasma nitriding process, test parts were cut cross-sectional and examined using Vicker's Hardness method throughout the cutting surface. Surface then was polished with 3 µm diamond paste and depth of nitrided layer was measured using optical microscope equipped with image analysis system (Leica DMRX).

Microhardness of samples before and after plasma nitriding process and case depth of the plasma nitrided samples have beenmeasured. Hardness measurements were performed on nitrided surfaces with a Leitz Vickers Hardness tester using a load of 100g. The microhardness depth profile was taken by taking the hardness measurements from the edge towards the core of the cross-sectioned samples.

X-Ray diffraction of the samples was performed on SEIFERT XRD 3000PTS X-ray diffractometer manufactured by Rich Seifert & Company, Germany, using a Cu–Kα (λ=1.5418 A˚) source in the Bragg–Brentano mode. A graphite crystal monochromator was used for the glancing incidence (GI) mode.

Results and Discussion

The results obtained from hardness and nitrided layer measurements are exhibited in Table 2. At same nitriding time and three different temperatures have resulted in change of hardness and case depth of nitrided layer as well. The change in thickness of nitrided layer will probably provide superior service life.

Table 2: Test results of hardness and layer thickness measurements

Temperature (°C)	Plasma Nitriding Duration (hrs)	Gas Mixture (%, $H_2:N_2$)	Hardness (HV)
Untreated (T_0)	Untreated	75:25	256
450 (T_1)	10	75:25	345
475 (T_2)	10	75:25	481
500 (T_3)	10	75:25	506

This study has shown that the characteristics of nitrided layer could be modified in a wide range. These results have shown that at same Plasma Nitriding Time and Gas Mixture hardness increases as we increase the temperature. Although the hardest nitraded layer was obtained at 500 °C, 75% H_2+25% N_2 gas mixture and 10 hours duration, for AISI 4140 steel. These results are exhibiting properties that can be applied to different service conditions providing production economy due to the amount of H_2 in gas mixture where depth of nitrided layer is more desirable compared to higher hardness.

The hardness profiles of nitrided cases are given in above Fig 2. The surface hardness of specimens is increases as temperature increases, while the core hardness remains unchanged. The combination of high kinetic energy of ions with the thermal activation results deeper penetration in plasma nitriding. The effect of surface cleaning of steels by glow discharge of H_2 is another important feature of plasma treatment [12, 14]. As a result of both effects, it was concluded that the activation energy of ion nitriding was lower than conventional techniques, allowing higher penetration rates [16].
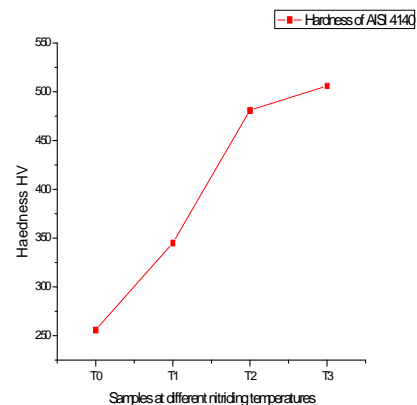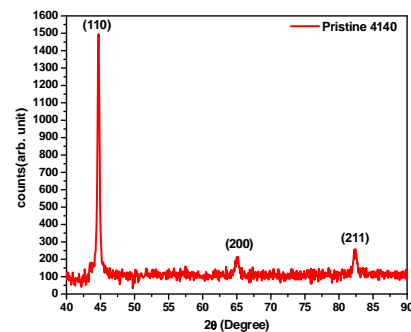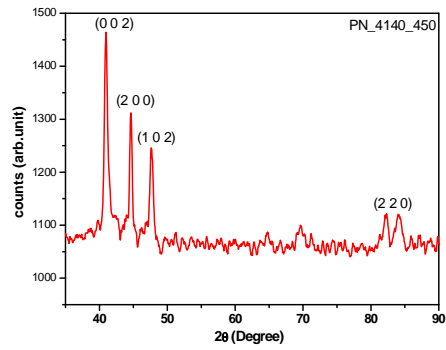

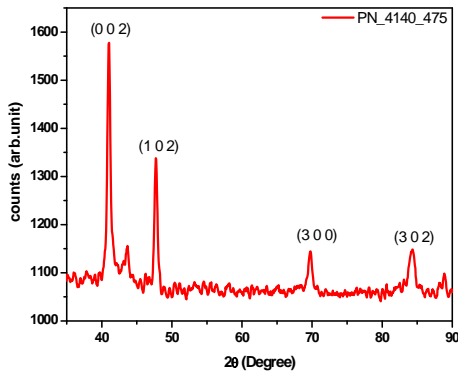**Figure 2:** Hardness curve for AISI 1045

The X-ray diffraction patterns measured at the surface of the specimens are given in Fig. 3. In Fig. 3 we show the glancing incidence XRD patterns of the untreated AISI 4140 compared with the modified surface taken at 2° incidence angle. While the pattern for the untreated sample (Fig. 3a) shows the α-Fe peak, the treated surface shows peaks in addition to that of α-Fe, which are assigned to hexagonal ε-Fe$_3$N (Fig. 3b, 3c, 3d). The broad peak at 44.2° could be due to phase of ε-Fe$_3$N which we confirm with higher resolution XRD studies. Note also the asymmetry in the α-Fe peak, which is due to hexagonal ε-Fe$_3$N. This is clearly seen when we plot the difference of the two patterns as shown in Fig. 3. We find no evidence for any other phase within the sensitivity of the technique, which probes <1 micron of the surface in GIXRD.
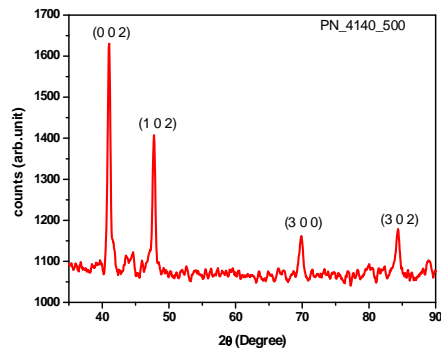

(a)

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(b)

(c)

(d)

Figure 3: Glancing-incidenceX-ray diffraction patterns of (a) untreated and plasma-nitrided AISI 1045 (b) at 450 °C, (c) at 475 °C, (c) at 500 °C.

Conclusions

In conclusion we have obtained plasma nitrided AISI 4140 with predominantly epsilon-Fe_3N phase on the surface. The broad peak of $Fe_{2-3}N$ confirms nitride phase formation. The Hardness profiles indicate a direct relation of the increased surface micro hardness in plasma nitrided AISI 4140 to the nitride phase formation in the nitride layer.

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