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Microwave ECR plasma nitriding of AISI 4140 steel

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A microwave electron cyclotron resonance plasma source (ECR) was employed to modify the mechanical surface characteristics of pieces of AISI 4140 steel. Experiments were carried out in a pressure range between 4 x 10^{-4} and 7 x 10^{-4} Torr, using a 60/40 hydrogen/nitrogen gas mixture and an incident microwave power of 400 W. Prior to the samples treatment the plasma was studied using a Langmuir probe to determine the electron temperature and the plasma density, the excited plasma species were determined by means of optical emission spectroscopy. All samples were treated for 50 min. in a low temperature regime (~ 250 ^{o}C) and the surface hardness increased up to 100 % of the initial value, with a nitrogen depth penetration of about 7.5 microns. The highest hardness and depth penetrations were obtained when the highest plasma densities were used to carry out the treatments.

Keywords: Microwave plasma; Nitriding.

Introduction

In the metal mechanical industry there is a constant need to improve the performance of some tools, for this, some thermal treatments are applied to metals. Nevertheless these treatments are not enough to improve the tools performance. It is well known that some thermo-chemical surface treatments improve the fatigue performance of machine parts. These processes cause increase in surface hardness and strength of material, which results in the formation of high compressive residual stresses on or near the surface of tools, as well as low tensile residual stress in the core. The residual stresses affect the net distribution of stresses under cyclic loading resulting consequently in a decrease in the tensile stress, which is effective in the surface. Nitriding is one of the most widely used thermo-chemical methods, which produces strong and shallow case with high compressive residual stress on the surface of steel components such as gears, crankshafts, dies, etc [1]. Plasma or ion nitriding process is being preferred recently in most surface hardening applications, to the conventional techniques such as gas or liquid nitriding, since the process has the characteristics of faster nitrogen penetration, simplicity in application cleanliness and economical aspects, as well as an easier control of compound and diffusion layer formation. The requirement of lower process temperatures, shorter process periods and suppressed compound layer formation are said to be the other advantages of ion nitriding [2]. Nevertheless, a better performance of the plasma nitriding process is required, and research in this area is focused on the plasma parameters study in order to enhance the whole process. In the present paper the plasma parameters of an Electron Cyclotron Resonance (ECR) microwave plasma source are studied and used to nitride AISI 4140 steel.

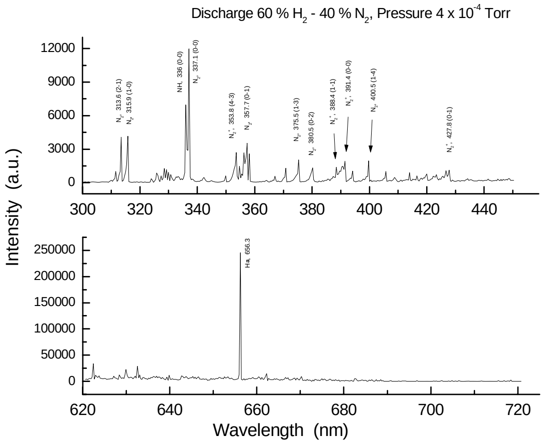

Figure 1. Optical emission spectra for a discharge at 4 x 10^{-4} Torr

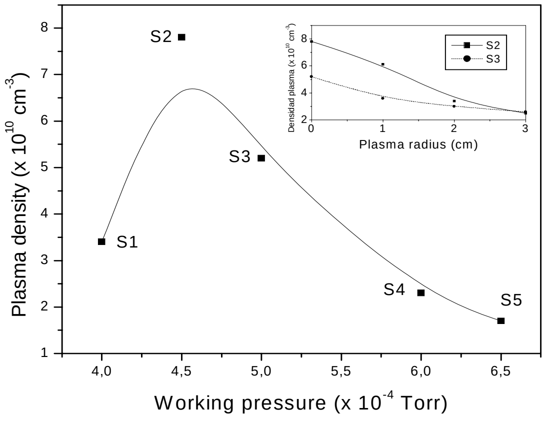

Figure 2. Plasma density as a function of the working pressure. The inset shows the variation of plasma density with plasma radius.

Experimental

Details of the microwave plasma source were given elsewhere [3]. The plasma was created by microwave excitation (f = 2.45 GHz) in an external magnetic field in order to establish the electron cyclotron resonance (875 Gauss). The power used in all the experiments was 400 W. The pressure inside the working chamber was varied from 4×10^{-4} up to 7×10^{-4} Torr with a gas mixture H_2/N_2 of 60/40. The heat transferred by the plasma is not enough to heat samples, so the samples were additionally heated with the sample holder. The temperature value was calculated after obtaining the relationship between the temperatures measured by a chrome-alumel thermocouple connected to the back of the sample and that produced on the front face of the sample. The temperature during treatments was kept constant at a value of 250 ^{o}C on the front face of samples.

The plasma specifications were determined by means of a single Langmuir probe, 5 mm length and 0.4 mm diameter. With this probe the values of plasma density, electron temperature and plasma potential were determined. Interpretation of data was done using the program SONDA [4]. Optical emission spectroscopy was used to determine the excited chemical species present in the discharge. For this purpose the light emitted by the plasma was focused by a set of lenses on a quartz optical fibre bundle and directed to a 0.5 m spectrograph. At the output of the spectrograph a CCD camera collected and analysed the light emitted by the plasma.

In the present experiments AISI 4140 samples 1 cm diameter and 0.5 cm thickness were exposed to the plasma for 50 min. Samples were previously tempered to a hardness of 520 Vickers Hardness. The composition of samples was (%wt): C (0.38 – 0.43), Si (0.15 – 0.35), Mn (0.75 – 1), P (0.035), S (0.04), Cr (0.8 – 1.1), Mo (0.15 – 0.25). The samples were electrically isolated from the chamber, in this way attaining the plasma floating potential or a bias dc voltage.

In order to determine the optimal experimental conditions to carry out the nitriding process, the surface hardness was measured. Surface hardness was measured by Vickers microhardness and depth-sensitive nanoindentation as well. The nitrogen content in samples after nitriding was determined by means of Energy Dispersive Spectroscopy (EDS). The microstructural details of the nitride layers were studied by X-ray diffraction using Cu $K\alpha$ radiation in order to assure the presence of the nitrided compounds.

Results and discussion

Nitriding in the microwave discharge was carried out using a 60/40 H_2/N_2 gas mixture as working gas. The majority of the species are vibrationally excited at a fundamental level. These molecules are adsorbed onto the iron surface and subsequently dissociate and dissolve into the steel matrix. Due to the low pressure of the microwave discharge there is little probability for the formation of atomic nitrogen, as this process requires a high excitation of the gas and a three-body recombination process. This is accompanied by the formation of molecular nitrogen with a photon emission corresponding to the first positive system of N_2. Optical emission shows that no emission in that range was observed. On the other hand, as fig.1 shows the most intense peaks come from the second positive system, corresponding to the excitation of the nitrogen molecule by electronic collisions. Together with these peaks, an intense peak corresponding to the emission of the NH molecule was present. From the spectroscopic point of view the excitation ion mechanism in the microwave discharge is due to electronic collisions, contrary to the case of discharges working at higher pressure and lower frequencies.

Langmuir probe measurements showed that the plasma density attains its maximum value close to 8×10^{10} cm^{-3} at 4.5×10^{-4} Torr in the central part of the plasma tube. The electron temperature remains practically constant in the range of pressures used and is equal to 6 eV. Radial measurements of plasma density showed a high variation of this parameter; and only between 2 cm from central part, the density is almost constant, at four centimeters from the central part the density decays. These results are shown in fig.2. In this figure with numbers S1, S2, etc., are shown the experimental conditions under which samples were treated. From here it is seen that a slight increase of the working pressure yield a considerable reduction in plasma density. Samples treated under S4 and S5 conditions showed no increment of hardness, in the 50 min that all samples were exposed to the plasma.

Figure 3 shows a comparison of the microstructure of an untreated sample and two samples treated under different conditions (mainly S2 and S3 conditions). From this plot it is seen that the peaks corresponding to nitrides begin appearing and become very clearly seen as the plasma density used in the nitriding process is increased. There were no detected peaks associated to the presence of the compound layer, which usually appear in $2\Theta = 42$ (ε-$F_{2-3}N$), and 40.9 (γ-F_4N). The absence of white layer is due to the fact, that with this type of plasma, it is possible to carry out the process at low temperature and low nitrogen concentration in the working gas.

Hardness measurements of samples treated with different plasma density were carried out at different loads and results are shown in figure 4. As it can be seen samples treated at the highest plasma densities show the highest hardness values and the thicker diffusion layer, it is worth mentioning that the treatment time is the same for all samples and equal to 50 min. Hardness as high as 950 Hv were obtained even with 50-gf loads. Using a depth sensitive indentation is useful for estimation of the diffusion layer thickness. With this method a maximum load of 30-gf was used and an indenter penetration depth of 2.5 microns was obtained in the case of S2 (maximum plasma density) experimental conditions. In this case the influence of the substrate is negligible, so that it can be considered that the thickness is equal to 7.5 microns (3 times the penetration depth at least). Then the thinner layer

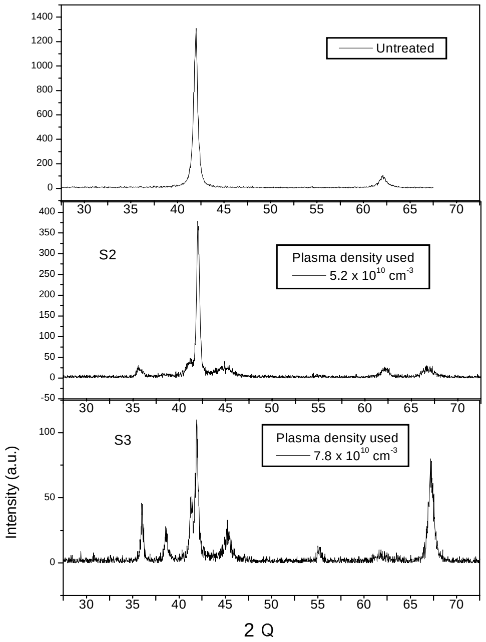

Figure 3. X-Ray diffraction patterns for samples nitrided at different plasma densities.

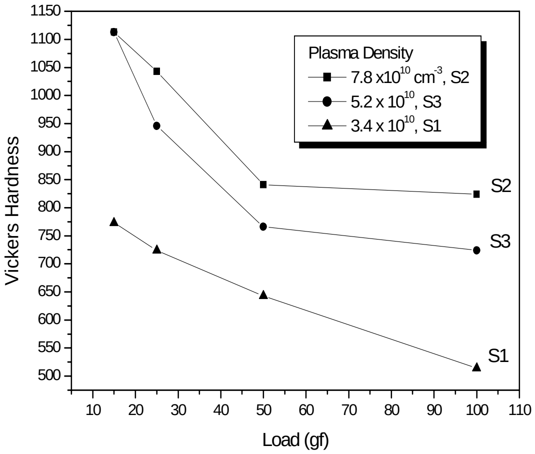

Figure 4. Measurements of Vickers Hardness at different loads for samples treated under different experimental conditions.

is obtained when the lowest plasma density is used (S1 case).

Conclusions

Nitriding of AISI 4140 samples was carried out in a microwave discharge. The highest values of hardness are obtained at the highest values of plasma density; nitriding was performed at low temperatures. With these plasma parameters it is possible to obtain nitrogen diffusion layers of up to 7.5 microns in 50 min treatment, and no white layer is formed.

The next step in this work will be to carry out the nitriding of tools in order to measure their performance in real work.

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