

ABSTRACT

The work presented in this thesis deals with the Cold Plasma, generated by a cost effective 50 Hz ac and 100 Hz pulsed dc electric power source. Characterization of nitrogen and argon-nitrogen plasma is carried out using different plasma diagnostics (Langmuir probe and optical emission spectroscopy) as well as various parameters, for the nitridation of materials such as AISI-4140 steel and aluminium alloy (*Al-Mg₂-Si*). Surface changes induced in materials by nitrogen and argon-nitrogen plasma are described using different material's surface diagnostic techniques.

In the first experiment, Langmuir probe measurements are carried out to characterize 50 Hz ac generated nitrogen plasma as a function of filling pressure (2–5 mbar) under constant power level. Plasma nitriding of AISI-4140 steel is executed for different time durations (2, 6, 10 and 14 h) at filling pressures of 2 and 3 mbar, keeping the input power at 100 W and substrate temperature at 300 °C. The plasma nitrided samples are characterized for their surface properties by using X-ray diffractometer (XRD) and Vickers microhardness tester. Results demonstrate the formation of nitrogen infused ferrite phase ($\alpha_N - Fe$) owing to the inclusion of nitrogen in the original iron lattice as interstitial solid solution. Significant increase in the surface hardness is observed when samples are treated for 14 h at a filling pressure of 2 mbar. The rapid decrease in the hardness value with increase in imposed load suggests the formation of the skinny modified layer.

In the second experiment, 50 Hz ac abnormal glow discharge is characterized as a function of filling pressure (1–4 mbar) using a Langmuir probe. It is found that electron temperature, electron density and electron energy distribution function decrease with the gas filling pressure. Nitriding of aluminum alloy (*Al-Mg₂-Si*) is performed for various time durations (3, 6, 9 and 12 h) and pressures (1 and 2 mbar) by keeping the input power (100 W) and substrate temperature (250 °C) constant. To explore the surface properties induced by plasma, X-ray diffractometer, optical micrography, atomic force microscopy and Vickers micro-hardness tester diagnostic techniques are used. Results exhibit the formation of cubic aluminum nitride (*AlN*). A significant increase in surface

hardness is observed when samples are exposed at 1 *mbar* pressure for 12 *h* of processing time. A rapid decrease in surface hardness values with the imposed load suggests the formation of a thin modified surface layer.

Langmuir probe diagnostic is used to determine the plasma parameters for argon mixing in nitrogen using ac generated plasma source. Results reveal that argon mixing in nitrogen plasma is an effective mode to increase electron density. Nitriding of aluminum alloy is performed in nitrogen-argon mixture (1:1) plasmas, to investigate the effect of argon mixing on surface properties. Samples are exposed for 3, 6, 9 and 12 *h* at a filling pressure of 2 *mbar* and an electrical input power of 100 *W*. The analysis is carried out by using x-ray diffractometer, scanning electron microscopy and Vickers micro hardness tester to investigate surface changes induced by plasma. X-ray diffractometer results exhibit the formation of cubic aluminum nitride (*AlN*) thin modified surface layer by reactive ac sputtering with argon as a sputtering gas and nitrogen as reactive gas. Moreover, significant increase in surface hardness is found for 12 *h* nitrided samples in nitrogen-argon mixture plasma.

In this experiment, nitriding behavior of stainless steel is investigated using pulsed dc (100 *Hz*) plasma. Active species of nitrogen in plasma play key role in nitriding the substrate. Therefore, optical emission spectroscopy and Langmuir probe diagnostics are used to determine the plasma parameters. Measurements are carried out for different pressure (1, 1.5, 2 and 2.5 *mbar*) and power (25, 50, 75 and 100 *W*) to optimize the plasma nitriding process. Electron temperature and electron density are evaluated by using intensity ratio of two atomic nitrogen lines (746.8 *nm* and 869.1 *nm*) and full width at half maximum (FWHM) of a Stark broadened line (746.8 *nm*) respectively. Langmuir probe is also used for determination of electron temperature, density, flux and velocity in plasma. These parameters are found to be strongly affected by the pressure and power. However, more energetic electrons are produced at 1 *mbar* pressure and 100 *W* power, which are responsible for enhanced plasma-reaction with stainless steel. Nitriding is performed under this optimum condition for 4, 8 and 12 *h* of treatment time. Nitrided samples are characterized using x-ray diffraction (XRD), fourier transformation infrared (FT-IR) spectroscopy, scanning electron microscopy (SEM) and atomic force microscopy (AFM) in order to study the growth and structural properties of nitrided layer. XRD

results exhibit the formation of dominant $\varepsilon - Fe_3N$ phase which is also observed by FT-IR spectrum. SEM and AFM micrographs showed the modified surfaces of exposing samples.