CHAPTER 2

SYSTEM DESCRIPTIONS AND INTRUMENTATION

2.1 Introduction

Planar coil inductively coupled plasma has the ability to produce plasma density in the order to 10^{11} to 10^{12} cm^{-3}. This type of plasma source is electrodeless and has a relatively simple setup. The system consists of a vacuum chamber, RF generator, matching network, vacuum system and gas supply system for processing.

2.2 Vacuum Chamber

The chamber is constructed from 3 mm thick stainless steel with 29 cm internal diameter and 30 cm in height. 12 circular ports, each having 7 cm in diameter is located around the chamber. These ports are used for vacuum pumping, gas inlets, pressure gauges, substrate holders and glass window for observation and plasma diagnostics. The chamber is separated from the planar coil by a 1 cm thick quartz plate placed at the bottom of the chamber opening and sealed with O ring. The chamber can be accessed by removing the top plate of the chamber so that changing of substrate and cleaning can be performed easily.

2.3 Vacuum/Pumping System

The chamber is evacuated by a 250 l/s Edwards Turbomolecular Pump backed by a two stage Edward RV18 rotary pump. The ultimate pressure achieved after pumping for 12 hours is in the range of 10^{-4} mbar. The vacuum level is monitored by a Pirani gauge for rough vacuum, and ion gauge for high vacuum.

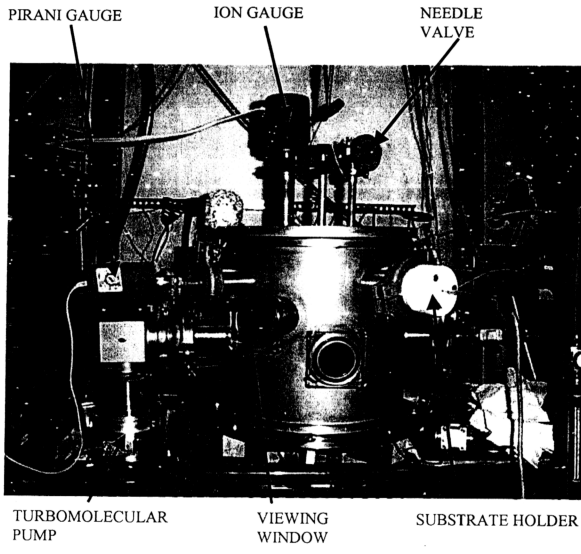


Figure 2.1: The RF ICP system for nitriding of materials.

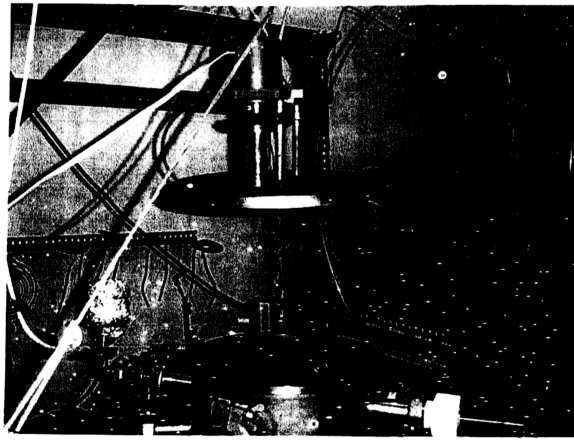


Figure 2.2: A photograph showing chamber access by removing the top cover.

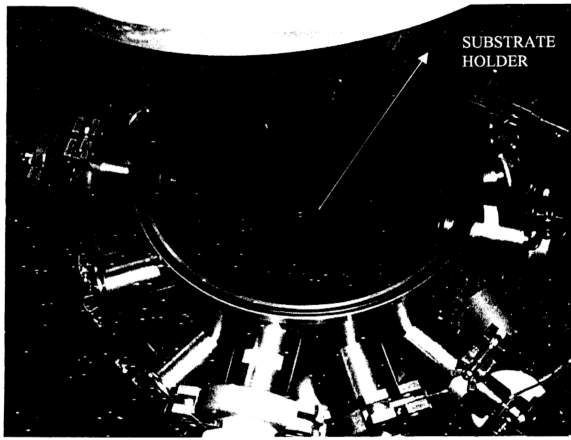


Figure 2.3: A photograph showing the interior of the nitriding chamber.

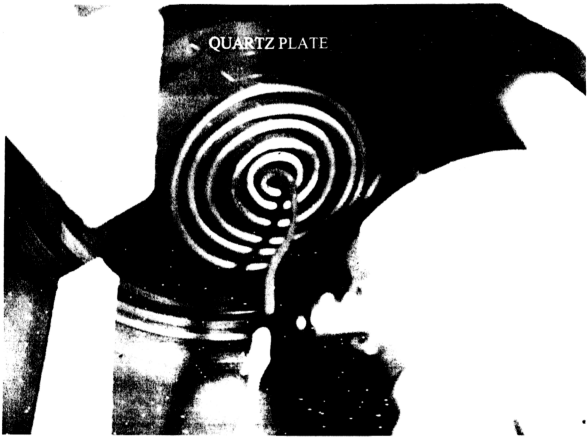


Figure 2.4: A photograph showing the planar coil placed at the bottom of the chamber.

2.4 Process gas Delivery

Gas filling into the chamber is controlled by using two needle valves, one for argon and one for nitrogen. Teflon tubes having diameter of ¼ inch, are used to transfer argon and nitrogen gas from the gas container into the chamber.

2.5 Substrate Holder

The substrate holder consists of a stainless steel rod mounted through one of the side port. The substrate is placed inside the chamber by hanging it with a 5 cm long stainless steel wire. The substrate holder is also connected to a 2 KV DC power supply. This is for biasing the substrate during the nitriding process.

2.6 RF Generator and impedance matching

The RF generator used is ENI model ACG-5 XL with a maximum power output of 600 W, operating at 13.56 MHz and its output impedance is rated at 50 Ω. Readings of the forward and reflected power can be read directly from the built-in power meter on the generator front panel. The plasma is generated by the RF driven planar coil. The coil is made from 1/8 inches hollow copper tube having maximum diameter of 8.5 cm. Since the quartz plate is heated up when the RF power is transferred to the coil, air cooling is necessary. A fan is used to achieve this purpose. This will also help to cool the quartz plate.

The generator used in this work is designed to have an output impedance of 50 Ω, so it must be matched a load having 50 Ω impedance. The RF power is transferred to the planar coil through a matching network. The impedance must match in order for the generator to maximize power dissipation (forward power) and to minimize the reflected power. Impedance matching is achieved by using a LC resonant circuit, shown in Figure 2.5. The circuit consists of a variable vacuum capacitor connected in

series to a planar coil, which acts as the inductor. The tuning capacitor range is from 56 pF to 720 pF.

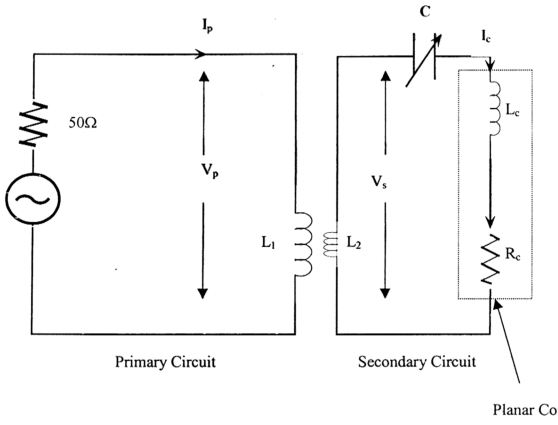


Figure 2.5: The equivalent circuit of the impedance matching network of ICP.

The variables in Figure 2.5 are:

V_p = Voltage across primary coil

V_s = voltage across secondary coil

I_p = Primary coil current

I_c = Secondary coil current

L_1 = inductance of transformer's primary coil

L_2 = inductance of transformer's secondary coil

L_c = inductance of the planar coil

R_c = resistance of the planar coil

The matching network consists of a step down transformer with a transformer ratio of 7:1. The number of turns in the primary coil is 14, while the number of turns in the secondary coil is 2. This is shown in Figure 2.6.

Let N_p = the number of turns in the primary

N_s = the number of turns in secondary

Therefore,

$$\frac{V_p}{V_s} = \frac{N_p}{N_s} = a,$$

and $$\frac{I_p}{I_s} = \frac{N_s}{N_p} = \frac{1}{a}$$

Where a = transformer ratio

$$\frac{\frac{V_p}{V_s}}{\frac{I_p}{I_s}} = \frac{\frac{N_p}{N_s}}{\frac{N_s}{N_p}} = \frac{a}{\frac{1}{a}}$$

So that

$$\frac{V_p}{I_p} = a^2 \frac{V_s}{I_s}$$

Taking,

$$\frac{V_p}{I_p} = Z_p \quad \text{and} \quad \frac{V_s}{I_s} = Z_s$$

So, $Z_p = a^2 Z_s$

Where Z_s is the impedance across secondary circuit or the matching load impedance

Take $Z_p = 50\ \Omega$ and $a = 7$

The matching load impedance is $Z_s = \frac{Z_p}{a^2} = 1.02\,\Omega$.

Therefore, the transformer is able to give an effective impedance of $\approx 1\ \Omega$ so that the resonant condition can be achieved. In this case the source is said to match the load if the following condition is satisfied:

$$\left|\omega L - \frac{1}{\omega C}\right| = 1$$

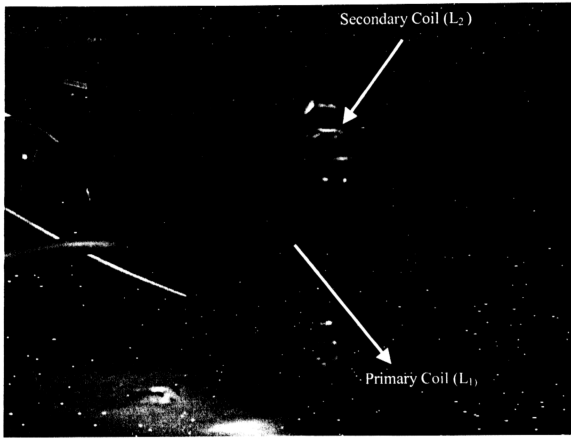


Figure 2.6: A picture showing a primary and secondary induction coil setup.

2.7 **Diagnostics system**

The diagnostic of the system consists of a Rogowski coil for measuring coil current and a photodiode array (PDA) system for optical emission spectroscopy.

2.7.1 **Rogowski coil**

The coil current is measured by using a Rogowski coil. The Rogowski coil is put inside a brass enclosure for RF shielding. The Rogowski coil used is made of 80 turns of copper wire. The coil is shown in Figure 2.7.

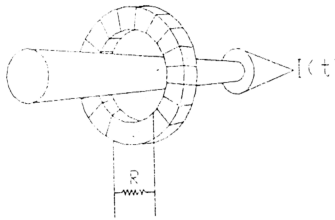


Figure 2.7: Schematic diagram of the Rogowski coil.

For coil current measurement, the Rogowski coil is set up as shown in Figure 2.8. The Rogowski coil is calibrated against a standard Pearson probe (with a factor 2 A/V). The value obtained for the Rogowski coil is 1.5 A/V. Details of the experiment had been described in an earlier thesis [45].

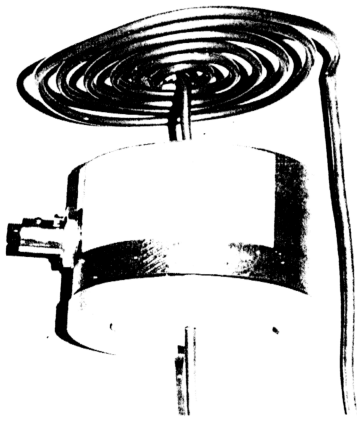


Figure 2.8: The Rogowski coil setup for measuring the coil current.

2.7.2 Photodiode array (PDA)

The system used for optical emission spectroscopy is a photodiode array with 1024 pixels of reversed biased silicon photodiodes covered with a quartz faceplate. When the light strikes a photodiode, the capacitance of each of the pixels is discharged; and the amount of the discharge depends on the intensity of the light. The detector operates at temperature of -20^0 C. This temperature is achieved by using a Peltier cooler. A two stage rotary pump is used to evacuate the detector housing, for removal of atmospheric moisture and to prevent condensation of water on the detector. The

detector is connected to the computer via GPIB connection to control the PDA and for data acquisition.

2.8 Material characterization instrumentation

2.8.1 X-ray diffraction (XRD)

X-ray diffraction is a versatile, non-destructive analytical technique for identification and quantitative determination of the various crystalline compounds, known as 'phases', present in solid materials and powders. X–ray diffraction analysis is based on the phenomenon in which the atoms of a crystal, and because of their uniform spacing, cause an interference pattern of the waves in an incident beam of X-rays. The crystal's atomic plane acts on the X-rays in the same way a uniformly ruled grating acts on a beam of light. The interference pattern is specific to each substance and gives information on the structure of the atoms or molecules in the crystal. Basic concept of XRD analysis is shown in Figure 2.9.

A crystal lattice is a regular three-dimensional distribution (cubic, rhombic, etc.) of atoms in space. These are arranged so that they form a series of parallel planes separated from one another by a distance d, which varies according to the nature of the material. For any crystal, planes exist in a number of different orientations - each with its own specific d-spacing. This phenomenon is governed by Bragg's Law. Bragg's Law is given by the following formula:

$n\lambda = 2d \sin\theta$

When a monochromatic x-ray beam with wavelength lambda (λ) is projected onto a crystalline material at an angle theta (θ), diffraction occurs only when the distance

traveled by the rays reflected from successive planes differs by a complete number of wavelengths.

By varying the angle θ, the Bragg's Law conditions are satisfied by different d-spacings in polycrystalline materials. Plotting the angular positions and intensities of the resultant diffracted peaks of radiation produces a pattern, which is characteristic of the sample. Where a mixture of different phases is present, the resultant diffractogram is formed by addition of the individual patterns.Identification is achieved by comparing the x-ray diffraction pattern - or 'diffractogram' - obtained from an unknown sample with an internationally recognized database containing reference patterns for more than 70,000 phases. The equipment used for analysis in this project is Bruker AXS Model . This system is capable of performing Grazing Incident Angle (GIA), especially important for thin film identification.

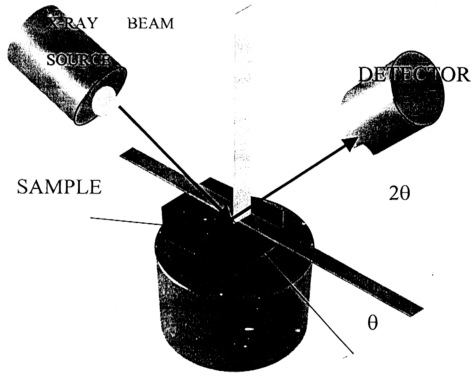


Figure 2.9: Basic concept of XRD analysis.

2.8.2 Scanning electron microscope (SEM)

The scanning electron microscope is used for analyzing surfaces of materials. In principal, it is similar to normal light microscope. However, it differs from the light microscope in the sense that SEM uses electrons for image formation, while the light microscope uses the light reflected from the sample surface. SEM is also capable of generating images at a very high magnification. It can perform an analysis at magnification higher than 70000 times. Another advantage of SEM is its capability to maintain the focus across the field of view, regardless of the roughness of the sample.

SEM operates under vacuum ($< 10^{-4}$ torr). This is essential to prevent scattering of electrons beam by collision with air molecules and to protect the electron gun from oxidation. The instrument consists of an electron gun, a scanning coil, an electron collector and a display panel. Figure 2.10 shows the schematic diagram of SEM. The electron gun consists of a filament, an aperture shield and an anode. Electrons are produced by passing a current through the filament and heating it to a point where the voltage gradient between the filament and the anode produces electrons. The electrons are accelerated by potential difference between the anode and the filament; and this is known as the accelerating voltage. Electrons emitted are then passed through a series of focusing lenses, and scanned across the sample surface through a scanning coil. When the electrons hit the sample, secondary electrons, backscattered electrons and photons (X-ray) are emitted from the sample. The secondary electrons are detected by an Evernet-Thorney detector, which translates into image on the display panel.

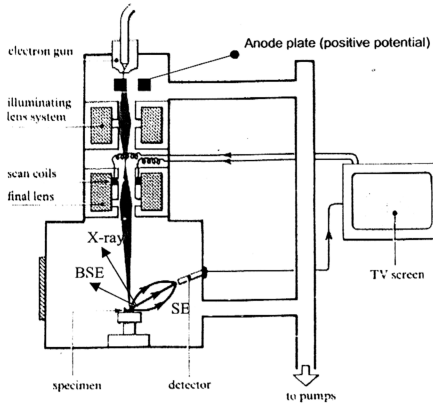


Figure 2.10: Schematic diagram of scanning electron microscope.

2.8.3 Hardness test

2.8.3.1 Micro Vickers Hardness Test

This is the standard method for measuring the hardness of metals, particularly those with extremely hard surfaces. The surface is subjected to a standard pressure for a standard length of time by means of a pyramid-shaped diamond. The diagonal of the resulting indention is measured under a microscope and the Vickers Hardness value read from a conversion table. Devised in the 1920s by engineers at Vickers Ltd., in the United Kingdom, the diamond pyramid hardness test, as it also became known, permitted the establishment of a continuous scale of comparable numbers that accurately reflected the wide range of hardness variation found in steels.

Vickers hardness is a measure of the hardness of a material, calculated from the size of an impression produced under load by a pyramid-shaped diamond indenter whose opposite sides meet at the apex at an angle of 136°. The diamond is pressed into the surface of the material at loads ranging from 5 to 500 gram force, and the size of the impression is measured with the aid of a calibrated microscope. The Vickers number (HV) is calculated using the following formula:

$HV = 1.854(F/D2)$,

with F being the applied load (measured in kilogram-force) and D2 the area of the indentation (measured in square millimeters). The applied load is usually specified when HV is cited.

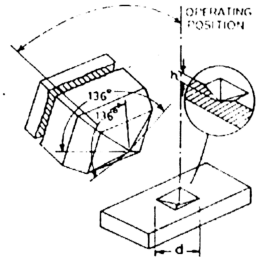


Figure 2.11: Schematic diagram of Vickers measurement [46].

2.8.3.2 Knoop Hardness

The relative microhardness of a material is determined by the Knoop indentation test. In this test, a pyramid-shaped diamond indenter with apical angles of 130° and 172°30". (called a Knoop indenter) is pressed against a material, making a thombohedral impression with one diagonal seven times longer than the other. The hardness of the material is determined by the depth to which the Knoop indenter penetrates.

This test method was devised in 1939 by F. Knoop and colleagues at the National Bureau of Standards in the United States. By using lower indentation pressures than the Vickers hardness test, which had been designed for measuring metals, the Knoop test allowed the hardness testing of brittle materials such as glass and ceramics.

The diamond indenter employed in the Knoop test is in the shape of an elongated four-sided pyramid, with the angle between two of the opposite faces being approximately 170° and the angle between the other two being 130°. The area of the impression under load can be calculated after measuring only the length of the longest side with the aid of a calibrated microscope. The final Knoop hardness (HK) is derived from the following formula:

$HK = 14.229(F/D2)$,

with F being the applied load (measured in kilograms-force) and D2 the area of the indentation (measured in square millimeters). Knoop hardness numbers are often cited in conjunction with specific load values.

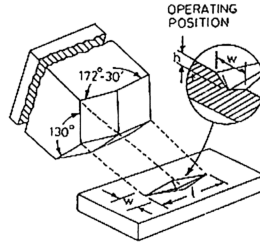


Figure 2.12: Schematic diagram of knoop indenter [46].

2.8.4 Wear test

The equipment used for wear analysis is CSEM Tribometer. In this test, a sample is rotated at certain speed, and a ball with a certain load is made to contact with the

rotating sample, as shown in Figure 2.13. After a specified duration or distance travelled, the sample is taken out and the wear track width or depth is measured. A bigger width or depth of the wear track indicates a higher wear rate.

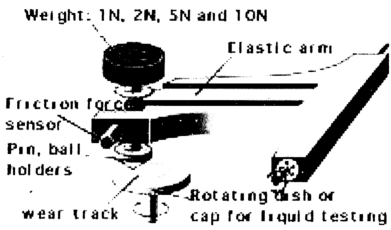


Figure 2.13: Schematic diagram of ball on disc measurement for measuring wear [47].

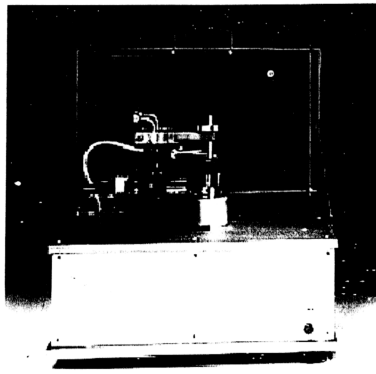
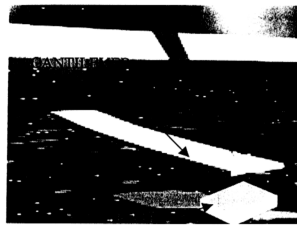


Figure 2.14: A picture of the CSEM tribometer used for wear test.

2.8.5 Atomic force microscope (AFM)

Atomic Force Microscope (AFM) is used for producing three dimensional image of resolution down to nanometer and angstrom range. AFM operates by measuring the attractive or repulsive force between a tip and a sample, as shown in Figure 2.15. In this work, measurements were done in repulsive forces or contact mode. In this mode, a sharp tip, mounted at one end of a cantilever, lightly touches the sample surface. As a raster-scan drags the tip over the surface, the cantilever moves up and down, conforming to the surface topography being measured. The motion of the cantilever is detected by the optical lever. The optical lever operates by reflecting a laser beam off the cantilever. The reflected laser beam strikes a position sensitive photodetector consisting of two side by side photodiodes. The difference between the two

photodiode signals indicates the position of the laser spot and thus, the deflection of the cantilever. By measuring the deflection of the cantilever, the local height of the sample can be measured. Three dimensional topographic map of the surface is then constructed by plotting the local height against horizontal probe tip position.



(a)

(b)

Figure 2.15: Concept of AFM (a) a cantilever touching a sample (b) laser beam for measuring cantilever deflection.