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A PLASMA REACTOR FOR SOLID SURFACE MODIFICATION

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Abstract - A general purpose plasma reactor has been designed and built to be used in various fields such as metallurgy, and microelectronics. The important aspects of the system design are: (a) the chamber can be evacuated to a base pressure of approximately $5 \times 10^{-7}$ Torr; (b) capabilities for thermal bake out at $150^\circ$C, and nitrogen pre-burn at a pressure of about $3 \times 10^{-3}$ Torr; (c) substrate holder which is capable of heating the substrate up to $400^\circ$C, and it is able to move the target so that plasma-surface reactions can be done anywhere inside the chamber; (d) provision for inductively exciting a gas or a mixture of gases using 1.0 KW of r.f. power at a frequency of 13.56 MHZ; (e) capabilities for controlling gas flow rates, and mixing them outside the chamber, so that the deposited layer composition can be controlled by varying the ratio of the gases. Also another gas can be supplied to the chamber outside the r.f. region near the substrate. This is an important design consideration if a reactant gas is to be excited and mixed with a neutral gas outside the r.f. region. (f) A capability for axially confining the plasma using a magnetic field. In this way the plasma density at the target inside the plasma is enhanced. We have characterized nitrogen plasma in our system by measuring the plasma density and electron temperature with respect to distance from the middle of the plasma chamber towards the target in the reactor chamber. The results show electron temperature and plasma density in the range of 4-40 ev and $10^{11} - 10^{8} \text{ cm}^{-3}$ respectively.

1. Introduction

The increasing interest on promotion of chemical reactions by glow discharges for modification of surfaces and deposition of thin films coatings has led to extensive research on plasma-surface interactions and number of different applications for plasma processing [1,2]. Although plasma processes such as PECVD have been the subject of research for more than two decades, but the detailed relationships between the plasma parameters and the results of plasma processing are difficult to explain. Many experiments have been performed using a variety of plasma reactors [3,4], but they mostly suffer from several major drawbacks. (a) Plasma is a harsh environment due to energetic electrons and ions. The crystallinity of the surface immersed in plasma is a strong function
of the energy and dose of charged species [5]. Impinging ions with energies of about 10 ev could cause incorporation and trapping [6]. When energies reach to about 100ev randomization of surface orientation takes place [7], and nearly complete amorphization of the surface occurs when ion energies approach 1.0 kev. takes place[8]. Therefore, crystal damage in plasma environment is inevitable. (b) In plasma reactors with capacitive coupling, much of the reactant gases, which are usually hazardous[9], are pumped out and do not even go through the plasma zone. Thus, care has to be taken in productive use of the reactant gases. (c) Excited neutrals play an important role in thin film deposition. However, film depositions are usually performed inside the plasma region. Attempts have been made to deposit films outside the plasma region[10,11], but still the substrate is somewhere within the order of the diffusion length of plasma outside the excited region. In addition, for this case deposition rates are very low. (d) When changing experimental parameters is necessary, stationary substrates have always limited the researchers, and new design is often required.

Having all the above deficiencies in mind a plasma reactor has been designed. In this paper a detailed physical description of the reactor, operation and the characteristics of the nitrogen plasma ignited in this reactor will be given.

2. System Design

Figures (1-2) show a schematic diagram and a picture of the plasma reactor. The system consists of four major parts. These are (a) The plasma chamber where the gas discharge takes place; (b) The reactor chamber which houses the substrate holder; (c) the gas supply system; and (d) the vacuum system. Each part will be discussed in detail.

2.1. The plasma chamber. The plasma chamber is shown in Figure 3. It consists of a quartz tube 15 centimeter in length and 5 centimeter in diameter. The tube is held by two non-magnetic 316 steel feedthroughs. In each of these feedthroughs there is a circular groove which holds an o-ring that seals the tube. At one end the tube is connected to the gas supply system, and at the other end it is connected to the process chamber. The plasma chamber is electrically isolated from the other parts of the reactor via teflon couplings. A water cooled copper wire coil inductively excites gases from an r.f. source which provides 1KW power at a frequency of 13.56MHz. To axially confine the plasma, the plasma chamber is immersed inside a magnetic field.

2.2. The reactor chamber. The deposition chamber is a cylinder which is of dimension 40cm x 50 centimeters. As it is shown in Figure 3., the chamber consists of six ports. The high and low vacuum lines are connected to the side and bottom ports respectively. One of the side ports is used for various measurements while in the port infront of it a movable substrate holder is placed. The substrate holder is made of two water cooled copper feedthroughs which provide power to the susceptor at the other end inside the chamber. The substrate can be heated in the range of 100-400°C using resistive heating. A temperature regulator maintains a constant temperature throughout the experiment. The cooling rate is controlled by running dry air through the vacuum sealed susceptor. An important design feature is the gas supply system which is placed above the substrate holder and provides non-excited gas or mixture of gases near the substrate. In this way excited neutrals which are transported out of the plasma chamber to the reactor chamber can be mixed with a neutral gas or mixture of neutral non-excited gases near the substrate. This is important if only one gas constituent is to be excited. The transport of the excited neutrals outside the plasma region can be done by employing high gas flow rates. Also, to assure the confinement of the ions inside the plasma chamber a negative potential is placed between the plasma and reactor chambers. In addition, the substrate itself is biased positively to keep away any incoming ions. Since the substrate lies outside the plasma region, there is no radiation damage.

If plasma-surface reactions need to be done inside the plasma region, provision is made to move the substrate close to the insulation coupling between the plasma and reactor chambers.

2.3. The gas supply system. There are two gas feed lines to the plasma reactor apparatus. One is fed to the plasma chamber, and the other to the reactor chamber(Fig.2). Both lines are capable of mixing two different gases in a mixing chamber. Each mixing chamber has its own separate vacuum line where gas partial pressures can be measured accurately and then be released to the system through a leak valve. In this way the deposition layer composition can be controlled by varying the ratios of the gases.

2.4. The vacuum system. The vacuum system comprises a rotary pump, and a diffusion pump with liquid nitrogen cold trap. To reach high vacuum levels prior to experiment a thermal bakeout at 150°C, and nitrogen pre-burn at 3x10^{-3} Torr is performed. The highest vacuum
level obtained with this system was $5 \times 10^{-9}$Torr. During the deposition process the pressure is controlled only by the rotary pump. The working pressure is usually between 200-300mTorr. The pressure is monitored by a Pirani gauge for pressures up to one micron and for higher vacuum levels cold cathode penning is used. To minimize contamination, a filtered nitrogen gas is used to break the vacuum. Safety measures are taken by directing the exhaust of the rotary pump outside the laboratory where it is filtered by a water scrubber.

3. Operation

High vacuum grade materials are used to construct the reactor to minimize outgassing and thus, a relatively fast pumpdown is achieved. After thermal bakeout a base pressure better than $5 \times 10^{-7}$ is obtained. Prior to experiments, we ignite a nitrogen discharge to remove any residual oxygen and water vapor from the reactor walls. Gas flow rates can be altered without changing the operating pressure. This is done by using a limiting valve at the rotary pump entrance to the reactor. As a part of the design we have characterized a nitrogen plasma in our plasma reactor. The plasma is formed at a pressure of 300mTorr. The measurements are done by employing conventional double probe technique. Two sets of measurements were performed in the presence and absence of magnetic and electric fields. Several current-voltage curves were obtained with respect to distance starting from the middle of the plasma quartz tube and ending at the substrate holder. Figures (4-5) show a comparison between the two sets of measurements. As it was expected in both cases the electron temperature increases with increasing distance towards the substrate while the plasma density decreases. But as the substrate and the chamber is biased, average energy of electrons and plasma density near the substrate cannot be measured. When using axial magnetic field, the plasma density increases considerably (Fig.3). Thus, if the plasma-surface reactions is to be performed inside the plasma, using a magnetic field is beneficial.

4. Conclusions

We report an inexpensive and versatile plasma reactor for solid surface modification. Deposition of thin films is possible both inside and outside the plasma region. By employing proper gas flow rates, substrate and chamber biasing, and choosing correct dimensions between the plasma and reactor chambers it became possible to keep energetic particles away from the substrate where only excited neutral species are allowed to reach the substrate. In addition, gas flow rates and their ratios are easily controlled by separating the gas mixing chambers from the other parts of the system. Nitrogen plasma was characterized and the results checks the design parameters. Right now experiments are in progress to deposite thin film layers on semiconductor and metal films.

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Fig. 1 - Schematic diagram of the plasma reactor.

Fig. 2 - A picture of the plasma reactor. The plasma chamber, the reactor chamber, the vacuum and gas supply systems are shown.
Fig. 3 - The plasma chamber is a quartz tube 15 cm in length and 5 cm in diameter which is held by feedthroughs and it is immersed in a magnetic field. A teflon coupling isolates this chamber from the reactor chamber.

Fig. 4 - Plasma density vs. distance in the absence and presence of electric and magnetic fields.
Fig. 5 - Electron temperature vs. distance in the absence and presence of electric and magnetic fields.

References