1 Laboratory excercise - Understanding PECVD of plasma polymers in capacitively coupled plasmas

1.1 Goals and aims of excercise

In this laboratory exercise you will work with radio frequency (RF) discharge called capacitively coupled plasma (CCP). CCPs are used in many plasma enhanced CVD (PECVD) processes and also in etching of materials. The sketch of frequently used configuration of PECVD reactors is in Fig. 1. The aim of this excersise is to understand several relationships important for PECVD in CCP and obtain hands-on experience in setting the deposition parameters. After passing this excercise, you should be able to recognize all functional parts of PECVD reactor, measure the leak rate, understand the relation between the gas flow rate and pressure in the reactor and the relation between the power, delivered power and self-bias.

1.2 Experimental setup

The PECVD reactor used in this excercise is an experimental PECVD reactor (R4) constructed for plasma diagnostics of PECVD processes and used currently for the plasma polymerization of amine compound, cyclopropylamine (CPA). It is UHV metallic reactor with two parallel plate electrodes, one of them being capacitively coupled to driving RF voltage and the other is grounded. The R4 reactor utilizes RF generator Cito 136 (COMET) with nominal frequency 13.56 MHz and maximum power input of 600 W. The high frequency voltage is adjusted to the plasma impedance in matching unit, which also contains blocking capacitor in order to prevent DC current in the circuit. The matching unit is connected with the generator, which can automatically tune matching unit to minimal reflected power or self-bias. The delivered power is independently measured by Octiv VI probe (Impedans), which measures amplitudes of voltage and current and respective phase shift.

The upper grounded electrode supplies gases into the plasma chamber through set of holes, each 1 mm in diameter. It is separated from the lower driven electrode by gap of 55 mm. Diameter of both is 210 mm. The reaction chamber is pumped by system of turbomolecular (HiPace 300, Pfeiffer) and dry scroll pump (nXDS 15i, Edwards), enabling ultimate pressure of $\approx 5 \times 10^{-5}$ Pa. The pumping speed, and therefore also pressure, can be regulated by a remotely controlled disc valve (VAT). Pressure on the chamber is measured by capacitron (Baratron, MKS) and compact full range gauge (PKR 251, Pfeiffer). Gases are delivered into the by the general purpose mass flow controllers (MF1, MKS), while vapors can be delivered by vapor source mass flow controller (MFC 1150, MKS). Vapors of studied monomer cyclopropylamine (CPA) are delivered via a needle valve.

Gas flow Q can be calculated from increase of pressure Δp over time Δt after closing the pumping line. Resulting flow is given as:

$$Q = \frac{\Delta p}{\Delta t} \frac{V}{p_{\mathrm{atm}}}, \tag{1}$$

where V is the volume of chamber, and p_{atm} is the atmospheric pressure. Gas flow is often given in unit of standard cubic centimeters per minute (sccm), for which the volume must be expressed in cm^3 and time in minutes.

1.3 Tasks of the laboratory excercise

1. Find information about CCP discharge and describe it briefly in your laboratory report.
2. Understand the pumping system, the system of valves, gas supply system, delivery of CPA vapors, reactor configuration etc. in R4 reactor - describe the experimental set up in the

report by text and photographs.

3. Determine the zero settings of capacitron when the pressure in reactor is below 10^{-2} Pa - describe the procedure in the report and write down the value.
4. Determine the volume of reactor using known flow rate of argon regulated by the flow controller.
5. Determine the leak rate using known reactor volume.
6. Determine the relation between the flow rate and pressure in the reactor for 5 values.
7. Determine the relation between the power, delivered power and self-bias for two different pressures, watch for the sheath and plasma expansion.

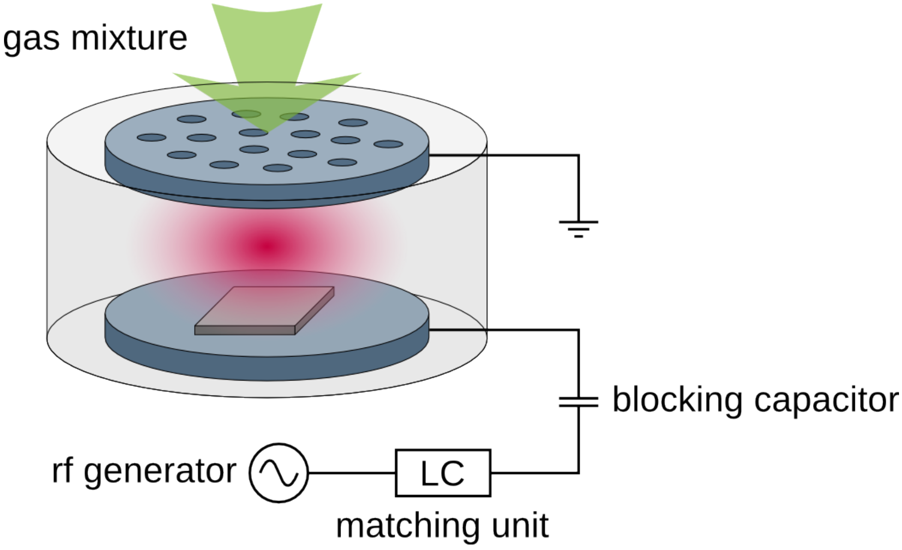

Figure 1: Scheme of the capacitively coupled PECVD reactor.

2 Laboratory excercise - PECVD, e-beam evaporation, ALD

2.1 Goals and aims of excercise

In this laboratory exercise you will get hands-on experience with the deposition of thin films using three industrial devices, each using a different deposition method: plasma enhanced CVD, electron beam evaporation and atomic layer deposition. The aim of this excersise is to understand the principal difference in the deposition methods concerning technical requirements, sets of materials that can be prepared with each method and differences in the deposition rates.

2.2 Experimental setup of PECVD

Deposition of DLC films will be carried out in CCP discharge using industrial plasma reactor PlasmaPro NGP 80 (by Oxford Instruments). The PlasmaPro NGP 80 is a modular plasma processing system, which is configured to carry out reactive ion etching (RIE), i. e. etching technology used in microfabrication, or deposition of hard coating. It can process a wide range of substrate sizes, from small wafer pieces up to 200 mm (8") diameter wafers, while the substrate temperature can be adjusted from -30 to 80 °C. The device can be used for etching of Si, SiO_2, Si_3N_4, TiO_2, W, Nb and deposition of DLC and CN films. The lower electrode is powered from an RF generator while the upper showerhead is grounded. The lower electrode acquires a DC negative self-bias, which attracts reactive ion species to the surface of the substrate.

Overview brochure:
`http://www.oxfordplasma.de/pdf_inst/PlasmaPro%20NGP80%20Brochure.pdf`
Detailed manual:
`https://www.london-nano.com/sites/default/files/u29/94-815831_IFU.pdf`

2.3 Tasks of the PECVD laboratory excercise

1. Find information about the structure of DLC material, its applications and methods of preparation (concentrate namely on necessary conditions in the case of PECVD) - summarize it in the introduction of your laboratory report.
2. Understand the pumping system, the system of valves, gas supply, reactor configuration etc. in PlasmaPro NGP 80 - describe the experimental set up in the report by text and photographs.
3. Deposit DLC film in PlasmaPro NGP 80 - describe the procedure steps.
4. Estimate the thickness of DLC film on Si from the interference color, comment on the absorption in the film looking at the film on glass, comment on the adhesion and hardness.

2.4 Experimental setup of e-beam evaporation

see pdf files in IS.MUNI Study materials - praktikum - e-beam evapor

2.5 Tasks of the e-beam evaporation laboratory excercise

1. Think about potential applications of metallic films and methods for their deposition, compare e-beam evaporation with possible deposition methods
2. Understand the device for e-beam evaporation, the system of valves, reactor configuration etc. - describe the experimental set up in the report by text and photographs.

3. Prepare Cu thin film - explain in your report general principles of e-beam evaporation and the specific conditions used during laboratories, estimate film thickness and deposition rate.

2.6 Experimental setup of ALD

see pdf files in IS.MUNI Study materials - praktikum - ALD

2.7 Tasks of the ALD laboratory excercise

1. Explain in your report general principles of ALD. What type of films can be usually prepared and what are their potential applications?
2. Understand the device for ALD, the system of valves, reactor configuration etc. - describe the experimental set up in the report by text and photographs.
3. Program the plasma-enhanced ALD of TiO_2 or SiO_2 thin films with the thickness of 25 nm.

2.8 Final task

1. In conclusion of your report, compare all three methods concerning the types of materials that can be prepared and the typical deposition rates.