1 Laboratory class 1. - PECVD, e-beam evaporation, ALD

1.1 Goals and aims of excercise

In this laboratory class you will get hands-on experience with the deposition of thin films using three industrial devices, each using a different deposition method: plasma enhanced chemical vapor deposition (PECVD), electron beam evaporation and atomic layer deposition (ALD). 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.

1.2 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`

1.3 Tasks of the PECVD lab excercise

1. Explain the principles of CVD and PECVD methods. What is the difference between them? Give examples of both (providing typical deposition conditions).

2. Find information about the structure of DLC material (amorphous/crystalline, chemical composition, carbon bonding) and summarize it in the introduction of your lab report. Describe how DLC can be prepared (at least two methods), give typical deposition conditions for those methods (concentrate namely on necessary conditions in the case of PECVD). What are typical properties of DLC films, provide some numbers. What are the industrial applications of DLC films?

3. Understand the reactor PlasmaPro NGP 80 configuration, its pumping system, pressure measurements, gas supply system - describe it in your lab report by text and photographs.

4. Deposit DLC films in PlasmaPro NGP 80 for two deposition times on polished silicon (Si) and glass substrates. Place the Si substrates on the bottom electrode by three different ways and explain it in your lab report. Describe the steps and conditions of the procedure.

5. Estimate the thickness of DLC films on Si from the interference colors and compare it with the values measured by optical method. Calculate the deposition rates, discuss its variations on Si substrates placed on the bottom electrode by three different ways and speculate about the reasons. Comment on the absorption in the film looking at the film on glass, comment on the film adhesion and hardness.

1.4 Experimental setup of e-beam evaporation

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

1.5 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 other possible deposition methods. Explain in your report general principles of the e-beam evaporation and thickness (deposition rate) monitoring by quartz crystal microbalance (QCM) monitor. Report the Sauerbrey formula that is used to calculate the deposited mass by QCM.
2. Understand the set-up of the device for e-beam evaporation (system of valves, reactor configuration etc.). Describe the experimental set-up in the report by text and photographs.
3. Prepare Cu thin film, report on the specific conditions used during lab task.
4. The deposition rate is monitored by QCM system in which the type of deposited metal is set as Ti. Calculate the correct deposition rate and report the value.

1.6 Experimental setup of ALD

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

1.7 Tasks of the ALD laboratory excercise

1. Explain in your report general principles of ALD and the thermal and plasma-enhaced ALD of Al_2O_3 using trimethylaluminium (TMA) as Al precursor. What type of films are usually prepared by ALD and what are their potential applications?
2. Understand the device for ALD, the system of valves, reactor configuration, plasma source etc. - describe the experimental set up in the report by text and photographs.
3. Program the thermal and plasma-enhanced ALD of Al_2O_3 according to the standard provider (Fiji) receipts (see study materials). Check and report all the conditions including those specific for each step within one ALD cycle. Report the expected film thickness per cycle (deposition rate per cycle) that is provided in the Fiji receipts. Calculate the deposition rate per the total deposition time and the deposition rate per time of the active deposition process (exposure time to Al and oxygen precursors).

1.8 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.

2 Laboratory class 2. - Understanding PECVD in capacitively coupled plasmas

2.1 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 chemical vapor deposition (PECVD) processes and also for plasma 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.

2.2 Experimental setup R4

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.

2.3 Experimental setup R2

Pro depozici bude použit nerezový válcový reaktor R2 se dvěma horizontálně umístěnými rovnoběžnými vnitřními elektrodami (obr. 1). Výboj je buzen radiofrekvenčním (RF) generátorem Cesar 133 (Dressler) pracujícím na frekvenci 13,56 MHz s maximálním výkonem 300 W. Dodávaný a odražený výkon měřen wattmetrem generátoru. Vysokofrekvenční signál je ze zdroje veden přes přizpůsobovací LC člen, který obsahuje oddělovací kondenzátor (blocking condensator). Přizpůsobovací člen je spojen s elektronikou generátoru, která umožňuje automatické ladění minimálního odraženého výkonu a zároveň měří stejnosměrné předpětí. Schéma aparatury včetně vnějšího elektrického obvodu je na obrázku 1.

Horní zemněná elektroda reaktoru o průměru 38 cm je pohyblivá. Její vzdálenost od dolní elektrody je možné měnit v rozmezí 1.0 až 6.5 cm. V našem případě je použita vzdálenost 5.5 cm. Plyny jsou směšovány a promíchávány mimo reaktor ve směšovači a do reakční komory jsou vedeny přes otvory horní elektrody. Otvory jsou v elektrodě rozloženy středově symetricky v kruhu

o průměru 18 cm. Spodní RF buzená elektroda o průměru 40 cm slouží zároveň jako nosič substrátu. Reakční komora je čerpána systémem turbomolekulární a za ní umístěnou rotační vývěvou. Rotační vývěva slouží k dosažení tlaku kolem 1 Pa a je zároveň použita jako předčerpávající vývěva pro turbomolekulární vývěvu TURBOVAC 50 (Leybold Vacuum) těsněnou O-kroužkem. Mezní tlak této turbomolekulární vývěvy je 5×10^{-6} Pa. Aparaturu je možné čerpat buď přímo přes turbomolekulární vývěvu a nebo přes odbočku, ve které je umístěna vymrazovačka. Přímý vstup do turbomolekulární vývěvy je otevírán/zavírán deskovým ventilem (VAT) ovládaným pneumaticky. Odbočka je oddělena od čerpacího systému manuálním vlnovcovým ventil (VAT), který je také možné regulovat čerpací rychlost vývěv.

Tlak v aparatuře je měřen kapacitronem Leybold Vacuum Ceravac CTR 90 s rozsahem do 133 Pa a kombinovanou měrkou tlaku Balzers TPG 251A. Tlak během procesů se pohybuje od 1 do 100 Pa. Průtoky užívaných plynů jsou řízeny elektronickými regulátory průtoku Hastings HFC and Schaefer.

2.4 Relation between pressure increase and gas flow rate

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.

2.5 Tasks of the laboratory excercise

1. Explain the principles and processes of PECVD. What typical homogeneous and heterogeneous reactions that take place during PECVD?
2. Find information about CCP discharge and describe it in your laboratory report. Do not forget to explain what is dc self-bias.

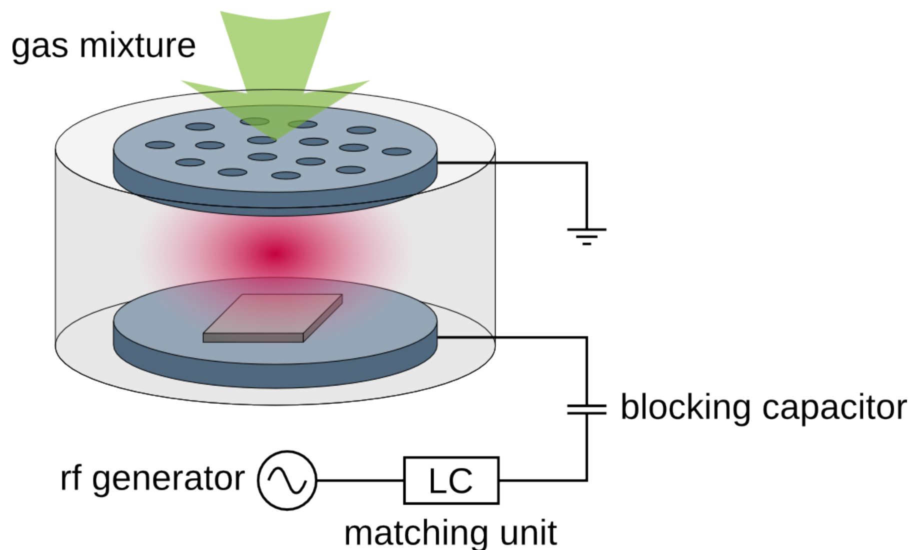

Figure 1: Scheme of the capacitively coupled PECVD reactor.

3. Understand the reactor configuration, its pumping system, pressure measurements, gas supply system, delivery of vapors from a liquid - describe it in your lab report by text and photographs.

4. 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.

5. Determine the volume of reactor using the known flow rate of argon regulated by the flow controller (for three flow rates). Describe the procedure (including what kind of gas and what gas correction factor has to be used if the flow controllers is calibrated for nitrogen). Report the experimental values, calculate the mean and its estimated standard deviation.

6. Determine the leak rate using the known reactor volume. Explain the procedure and report the experimental results determined at the beginning and at the end of laboratory task. Is it different and why?

7. Determine the relation between the flow rate and pressure in the reactor for 5 values. Plot the results, use linear approximation, determine correlation coefficient and discuss the validity of linear dependence, determine the slope and its error.

8. Determine the relation between the power from the generator and power delivered to the discharge. Is the delivered power lower? If yes, why? Plot the results, use linear approximation, determine correlation coefficient and discuss the validity of linear dependencies, determine the slopes and their errors. What is the effect of pressure?

9. Determine the relation between the power from the generator and self-bias for two different pressures. Plot the results, use linear approximation, determine correlation coefficient and discuss the validity of linear dependencies, determine the slopes and their errors. What is the effect of pressure? Imagine you would like to sputter the material on RF electrode. What pressure would you choose, low or high? Explain it in the lab report.

10. Determine the relation between the RF voltage amplitude and dc self-bias for two different pressures. Plot the results. What function can you use to fit the dependence? If it is linear determine the slopes and their errors.

11. Watch for the sheath and plasma expansion when changing pressure and power. Describe what do you see.