

## Exercise 1. Thermal oxidation of silicon wafers. Ellipsometric measurement of oxide thickness.

### Oxidation

Silicon dioxide ( $\text{SiO}_2$ ) is a main insulating material used in microtechnology. The most common technique utilised to produce insulating oxide layer is thermal oxidation performed in a furnace. There are two methods of oxidation:

- ◆ dry oxidation, when the wafer is exposed to an oxygen at  $\sim 1000^\circ\text{C}$ , high quality oxide for MOS gate insulation is produced in this manner
- ◆ wet oxidation, when mixture of high-purity oxygen and hydrogen at  $\sim 1000^\circ\text{C}$  is burned, giving water vapour environment; the advantage of this procedure is higher growth rate, although “wet” oxide features not so good quality and may be used only as a masking layer for instance.

An aim of the exercise is to grow the layer of the oxide and measure its thickness employing an ellipsometer. The wet oxidation method shall be used for growth; however, due to safety regulations, hydrogen must not be used in the lab. Alternatively, oxidation will be performed in an atmosphere of oxygen or nitrogen saturated with water vapour.

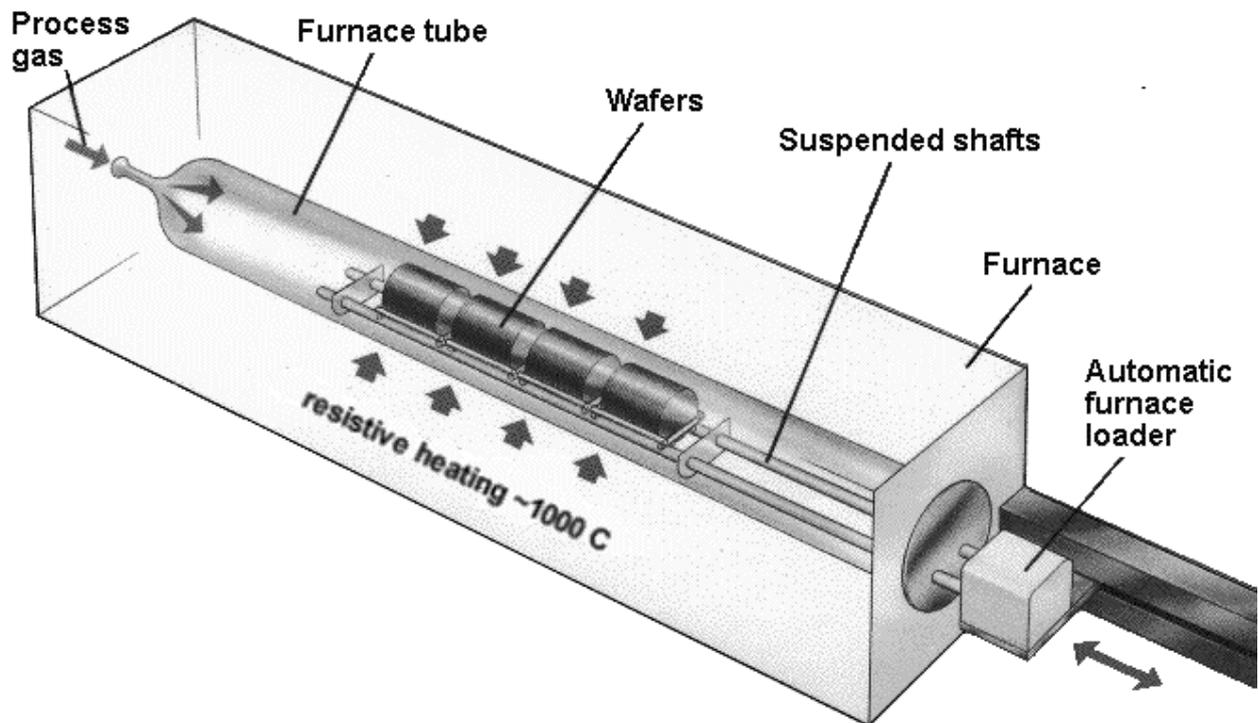


Fig.1 An internal view of the heating section of the furnace



*Fig.2. The wafers in a quartz boat*

To perform an oxidation one should:

1. Turn the furnace on and heat it up. This operation will be performed by the lab staff before an exercise as heating up takes 1-2 hours.
2. Set the process temperature 1000°C on the temperature controller. Use the thermocouple calibration table to do this.
3. When the set-point temperature has been reached, put the wafers into a quartz boat and load it into the tube utilising a furnace loader (loading speed 30 cm/s). **When working in the vicinity of the tube, always wear heat-resistant, protective gloves!**
4. During heating up, pure oxygen or nitrogen flows through the processing tube. To start process, put the gas flow through the water bubbler – this is where gas saturation with water vapour takes place. Use the gas system scheme shown on the gas section of the furnace.
5. After 1 hour, unload the wafers from the tube (use the 20 cm/s speed). Leave the wafers for 10 minutes to cool down.

**Any of your actions must not be performed without the supervision of the lab staff. This is for your safety only!**

Now, you can start the oxide thickness measurement.

## Ellipsometric measurement of SiO<sub>2</sub> layer on Si substrate

Ellipsometry is an optical technique, which enables measurements of thickness and optical properties of thin films. In microtechnology it is commonly used to evaluate thickness of silicon oxide, silicon nitride, polysilicon, photoresist. An idea of ellipsometry is based on reflection of polarised light from the sample under study. It can be shown (and it is intuitively obvious) that polarised light changes its polarisation state upon reflection and that this change is determined, among other things, by thickness and optical properties of the measured film (see fig.1). After the measurement has been done, calculations of the thickness and refractive index should be performed. Unfortunately, equations of ellipsometry are, in general, analytically insoluble, so numerical methods and computer program must be applied.

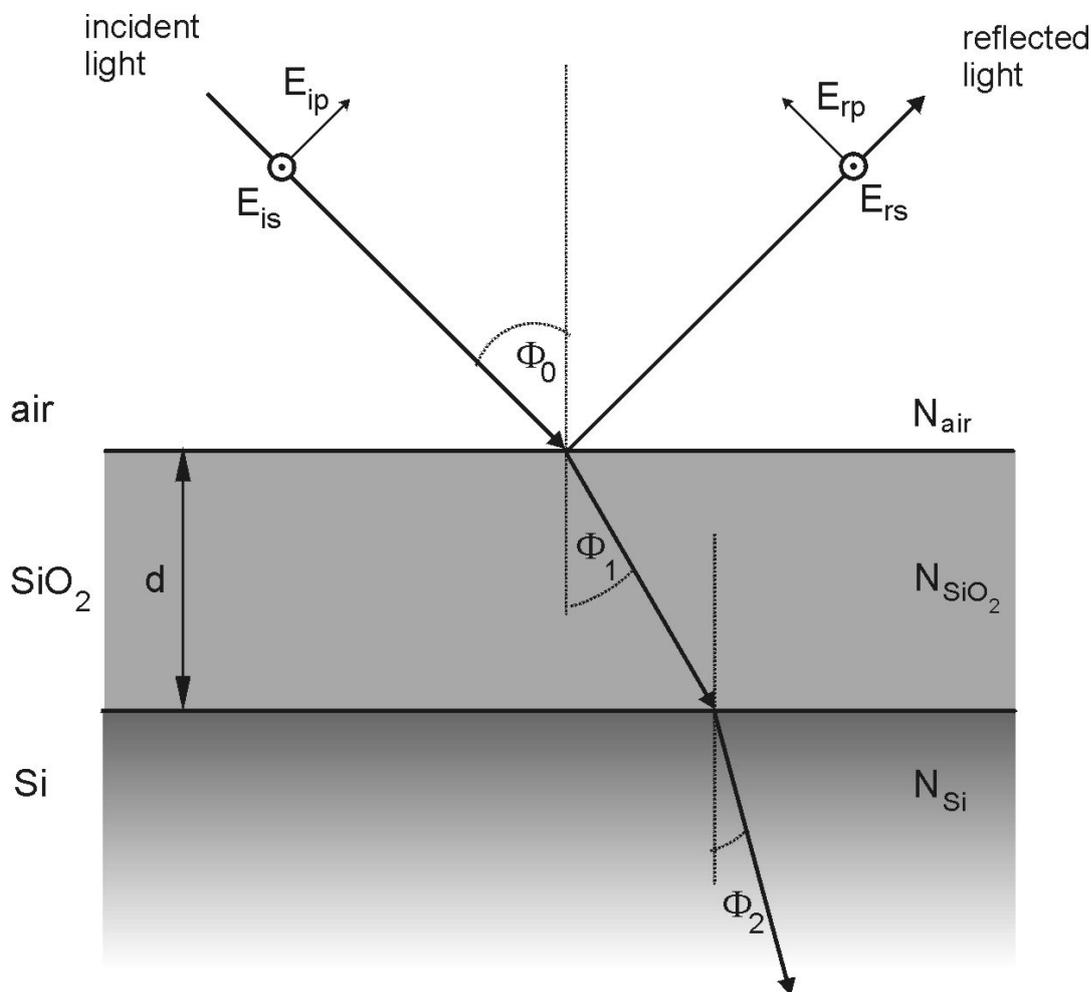


Fig.1. Reflection of light at SiO<sub>2</sub> layer thermally grown on Si substrate. Indices "s" and "p" describe electric field components (incident and reflected) perpendicular (s) and parallel (p) to an incidence plane.

## Measurement

Figure 2 shows configuration of so called null-ellipsometer. Light emitted from mercury lamp LS (wavelength 546.1 nm) passes through linear polariser P, quarter-wave retarder Q (which shifts phase between  $E_p$  and  $E_s$  components) reflects at the sample S, passes through the second polariser (analyser) A and enters a photomultiplier PM. An aim of the measurement is to set polariser and analyser angles in such a way that intensity of light entering the photomultiplier is minimised (null condition).

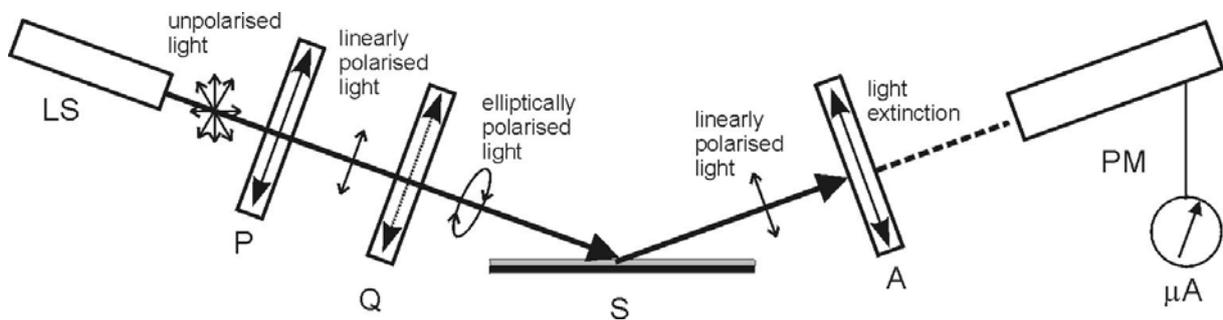


Fig.2. Principle of operation of null-ellipsometer

To perform measurement, one should:

- switch the device on
- place the sample on the stage
- switch the photomultiplier on (PM button)
- set an angle of incidence  $\Phi_0$  of light beam, measurements for angles 50, 60, 70 degrees are recommended
- for given  $\Phi_0$ , rotate polariser and analyser until extinction condition (minimum of photomultiplier current) is found
- read polariser and analyser azimuth angles,
- correct readout by subtracting  $\Delta P = 212^\circ$  and  $\Delta A = 172.5^\circ$  from polariser and analyser angles respectively; the correction is necessary because of internal calibration of the instrument.
- **not rotate** quarter-wave retarder, which is fixed

When the first pair of angles,  $P_1$  and  $A_1$  has been found, it is easier to find the second one because for perfect optical elements, the following relations hold:

$$P_2 = P_1 + 90^\circ, A_2 = -A_1 \quad (1)$$

but **please note** that there is an ambiguity in  $P$  and  $A$  measurements:  $P$  and  $P + k * 180^\circ$ ,  $A$  and  $A + k * 180^\circ$  ( $k=1,2,\dots$ ) cannot be distinguished!

Then, the following parameters, called  $\Delta$  and  $\Psi$  must be calculated according to relations:

$$\Delta = 2 * P_{1,2} \pm 90^\circ, \Psi = \text{abs}(A_{1,2}) \quad (2)$$

Again, the same ambiguity occurs, so one should feel free to add or subtract  $k * 180^\circ$  until the following conditions are fulfilled:

$$0^\circ < \Delta < 360^\circ, 0^\circ < \Psi < 90^\circ \quad (3)$$

Two values of  $\Delta$  differing by  $180^\circ$  and one value of  $\Psi$  are the result of these considerations. They have straightforward physical interpretation:  $\Delta$  is the phase shift which is introduced by the sample upon reflection,  $\text{tg}(\Psi)$  describes ratio of reflection coefficients of  $p$  and  $s$  component of light.

Both pairs of  $\Delta$  and  $\Psi$  are entered into computer program and film thickness and its optical properties can be calculated.

It is recommended to repeat the whole procedure for more than one angle of incidence of light - this will improve precision of the results substantially.

Instructions how to use an ellipsometric software will be provided by the teacher.