# 1 The reactive DC-Magnetron Sputtering Process

In our institute the optical functional coatings are produced in a dc-magnetron sputter process. This process offers the advantage of a homogeneous large area coating in the final production. For the production of suitable coatings favourable deposition parameters have to be identified. Their influence on the resulting layers is studied.

## 1.1 The Principle

Within the *sputtering process* gas ions out of a plasma are accelerated towards a target consisting of the material to be deposited. Material is detached ('sputtered') from the target and afterwards deposited on a substrate in the vicinity. The process is realized in a closed recipient, which is pumped down to a vacuum base pressure before deposition starts (see figure 1).

To enable the ignition of a plasma usually argon is feed into the chamber up to a pressure between 0,5...12 Pa. By natural cosmic radiation there are always some ionized $Ar^+$-ions available. In the *dc-sputtering* a negative potential $U$ up to some hundred Volts is applied to the target. As a result, the Ar-ions are accelerated towards the target and set material free, on the other hand they produce secondary electrons. These electrons cause a further ionization of the gas. The gas pressure $p$ and the electrode distance $d$ determine a break-through voltage $U_D$ [1, 5] — from which on a self sustaining glow discharge starts — following the equation $U_D = A \cdot pd/(\ln(pd) + B)$ with materials constants $A$ and $B$. Graphically spoken the ionization probability rises with an increase in pressure and hence the number of ions and the conductivity of the gas also increase. The break through voltage drops. For a sufficient ionization rate a stable burning plasma results, wherefrom a sufficient amount of ions is available for sputtering of the material.

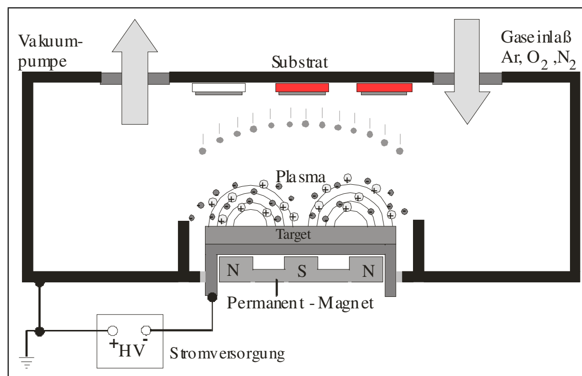


Figure 1: The principle of the sputtering process.

To increase the ionization rate by emitted secondary elecrons even further, a ring magnet below the target is used in the *magnetron sputtering*. The electrons in its field are trapped in cycloids and circulate over the targets surface. By the longer dwell time in the gas they cause a higher ionization probability and hence form a plasma ignition at pressures, which can be up to one hundred times smaller than for conventional sputtering. On the one hand higher deposition rates can be realized thereby. On the other hand less collisions occur for the sputtered material on the way to the substrate because of the lower pressure and hence the kinetic energy at the impact on the substrate is higher (see also below). The electron density and hence the number of generated ions is highest, where the **B**-field is parallel to the substrate surface. The highest sputter yield happens on the target area right below this region. An erosion zone is formed which follows the form of the magnetic field.

The bombardment of a non-conducting target with positive ions would lead to a charging of the surface and subsequently to a shielding of the electrical field. The ion current would die off. Therefore the *dc-sputtering* is restricted to conducting materials like metals or doped semiconductors. There are now two ways to produce dielectric films: In *rf-sputtering* (radio frequency) an ac-voltage is applied to the target. In one phase ions are accelerated towards the target surface and sputter material. In the other phase charge neutrality is achieved. Hereby also sputtering of non-conducting materials is possible.

Alternatively, for *reactive sputtering* other gases like oxygen or nitrogen are fed into the sputter chamber additionally to the argon, to produce oxidic or nitridic films.

## 1.2 The Sputter Parameters

The resulting film properties can be controlled by adjusting the following sputter parameters:

The *sputter current* $I_{sp}$ determines mainly the rate of the deposition process and hence the time which remains for the arriving particles during the growth process for either surface diffusion and agglomeration on existing growth centers or nucleation with other adatoms. The *applied voltage* determines the maximum energy, with which sputtered particles can escape from the target (reduced by the binding energy). Energies of the sputtered particles show a broad distribution with a maximum of the distribution between 1 $e$V and 10 $e$V [7, 4]. The applied voltage determines also the sputter yield, which is the number of sputtered particles per incoming ion.

The *pressure* $p$ in the sputter chamber determines the mean free path $\lambda$ for the sputtered material, which is proportional to $1/p$. Together with the *target-substrate* distance ($TS$) the pressure controls, how many collisions occur for the particles on their way from the target to the substrate. This can influence the

porosity of the films. But also the crystallinity and texture can be effected [2, 3].

Via the *gasmixture* one can control the stoichiometry of films, which are sputtered from a metallic target. The *oxygen flow* $q(O_2)$ is the parameter varied, whereas the desired total pressure is kept constant by regulation of the Ar-flow $q(Ar)$.

The *substrate temperture* can have a strong impact on the growth behaviour with respect to crystallinity or density of the samples. It can be adjusted between room temperature and 500° C. But even during sputtering without external heating the substrate temperature may rise considerably, especially during long sputtering times for the deposition of thick films [**?**, 6].

In principle a *bias-voltage* can be applied to the substrate up to $\pm 100$ V, which has the effect of accelerating electrons or ions towards the substrate or keeping them away. Both may have an influence on the layer growth as reported in the literature [9, 10].

Usually substrate and target surface are parallel to each other. A variation of the deposition angle (also: *sputtering under oblique incidence*) can be achieved by tilting the substrate. Thereby a new preferential direction for the film growth and potentially anisotropic films can be produced.

# 2 The Sputter System

All optical coatings under investigation were prepared utilizing a sputter system built by Stollenwerk [7] at the I. Institute of Physics. Up to six targets can be inserted into the base plate of the cylindrical vacuum chamber. Thus even complex layer stacks can be produced without breaking the vacuum. Due to the targets' relative small diameter of 3 inch, even rather exotic materials can be used at a reasonable financial expense. They are bonded to a water cooled copper carrier, ensuring both electrical and thermal contact. An aperture ensures that only one target at a time can be used for deposition, and prevents the other targets from being coated.

The rotatable substrate holder is attached to the top cover opposite of the targets. Up to 24 samples — typically 3×1 inch microscope slides, but also silicon wafers or other substrates — can be coated in one batch, resulting in a large number of samples under highly reproducable conditions. The maximum distance between target and substrate is limited to 78 mm by the chamber's dimensions, the minimum separation is approximately 50 mm, as for lower distances the plasma process is disrupted.

The substrate to be coated is rotated until a position directly above the target is reached. Then it remains stationary until the desired film thickness — $d = rt_S$, the product of deposition rate and time — is reached. For coating the next substrate, the sample holder is turned by one position. A second aperture directly below

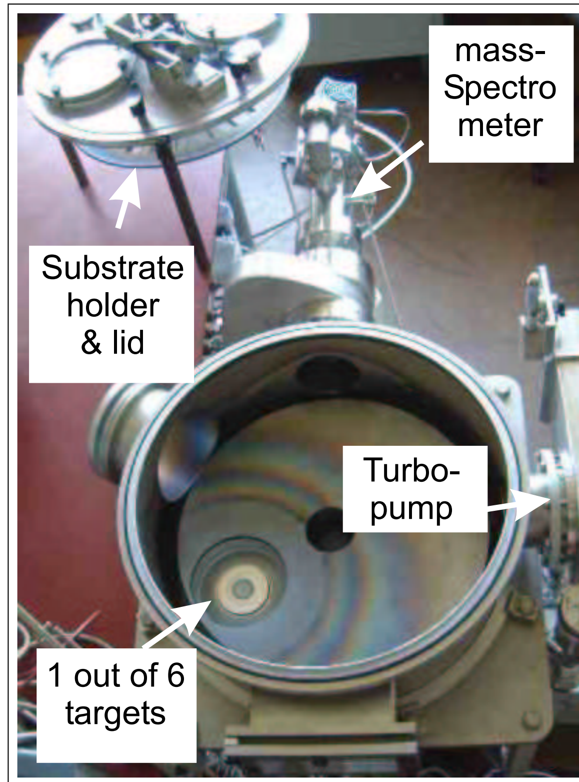


Figure 2: The opened sputter chamber with the target holder in the background.

the substrate holder ensures that only the sample opposite of the active target is coated. The sample movement can be done manually or under computer control. In the latter case the reproducibility is approximately 0,1 s [8].

Instead of coating an actual substrate, a quartz crystal balance can be used for the characterization of the target.

A plasma power supply with arc suppression produced by *ELAN* is used to apply high voltages (up to 600 V / 1,2 A) to the target. The films were produced in constant current mode. Constant power and constant voltage modes are also available. Additionally, a home built power supply can be used, which was initially developed for laser applications. Consequently, there is no arc suppression, but the maximum voltage of 3 kV enables the sputtering of target materials, where the ignition of a plasma is difficult.

The base pressure of the chamber is $3{\cdot}10^{-4}$ Pa, the chamber volume is 150 l. Up to 4 mass flow controllers (MFC's) are used to regulate gas composition and pressure during deposition. Usually the desired oxygen flow rate $q(O_2)$ is fixed and the total pressure (typ. 0,5. . . 12 Pa) is kept constant by varying the argon flow. The purity of both gases is 5N. Additionally the gas composition in the chamber can be monitored by a mass spectrometer (QMS) to determine the influence on the

film growth. Due to hysteresis effects when changing the oxygen flow, the oxygen content is an important parameter. Plasma monitoring can be performed by an optical emission spectroscopy (OES) setup. Analysis of the emitted spectral lines allows conclusions on the sputter process.

All of the process parameters mentioned are converted by an 12 bit digitizer card with 16 input ports and recorded and displayed by a home made computer program.

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