

Thin Film Technology

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1 INTRODUCTION

A thin film is a layer of material ranging from some nanometers to several micrometers in thickness. The controlled synthesis of materials as thin films, known as deposition, is a fundamental step in many modern applications. Physical and chemical approaches have been recognized as the primary pathways for thin film deposition, with several techniques categorized under these two broad routes, as shown in Fig. 1 (based on [1]). This report focuses on the two processes called evaporation and sputtering which fall under the category of the so called physical vapor deposition (PVD) techniques, the common feature of which is the vapor-phase transport of the source material which makes up the film material. Both of these techniques play a central role in modern micro- and nanotechnology fabrication. For instance, evaporation is instrumental in producing precisely controlled coatings for optical devices, such as anti-reflective layers on lenses, which enhance visual clarity and device efficiency [2]. On the other hand the presence of sputtering techniques in the semiconductor industry can hardly be understated, creating the intricate layers necessary in integrated circuits, thereby enabling the continued miniaturization and performance enhancement of electronic devices. A more recent example of its importance is its application in component manufacturing for solid oxide fuel cells, contributing to increasing energy conversion efficiency while minimizing material usage [3]. Moreover, in the realm of data storage, a recent development, so called phase-change memory applications rely on sputtering techniques [4]. In the following we will outline the basic theory behind these methods and apply them by fabricating two different kinds of thin films using both evaporation and sput-

tering respectively. We will then investigate the films by means of reflectometry and profilometry.

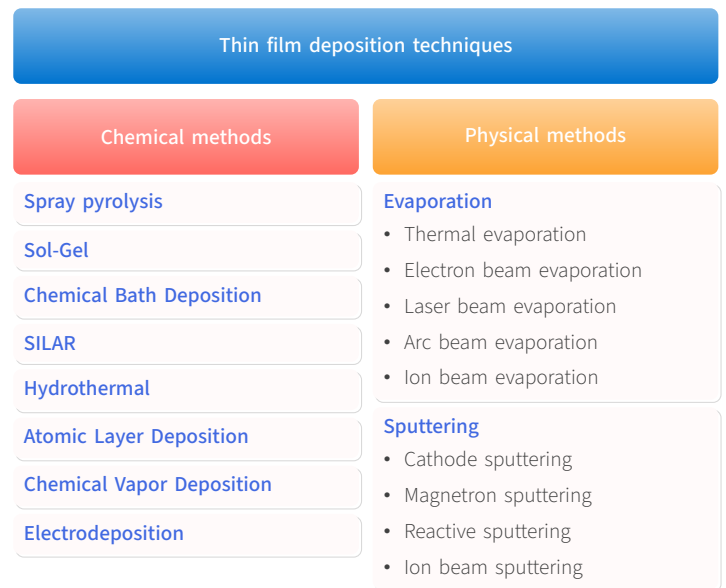


Fig. 1 | Overview of thin film deposition techniques. Based on [1].

2 OVERVIEW OF THE TECHNOLOGICAL EXPERIMENTS

The different experiments which have been carried out are outlined in Tab. 1.

3 SPUTTERING

Sputtering is one of the most widely adapted methods to fabricate thin films nowadays. There is a large number of different sputter deposition methods which range from classical cathode sputtering to more sophisticated techniques such as reactive magnetron sputtering. Figure 3 depicts a schematic representation of a basic sputtering process. The process which takes place inside an evacuated chamber (typically around 1 to 10e-6 mbar) uses an inert gas (e.g., argon) which is placed inside an electric field established between two conductive electrodes. The applied electrical field accelerates free electrons present in the vacuum chamber. These

Experiment (in order of execution)	Short description
Sputtering	Si substrates are coated with TiO ₂ in a sputtering system.
Evaporation	An Ag layer is produced on previously cleaned glass substrates in a thermal vapor deposition system.
Profilometer	In this experiment, the layer thickness of the Ag-layer vapor-deposited on glass is measured using a profilometer. To do this, the silver is scraped off the surface of the sample in a straight line.
Reflektometry	The TiO ₂ layer sputtered on a silicon substrate is determined using a reflectometer.

Tab. 1 | Experiments.

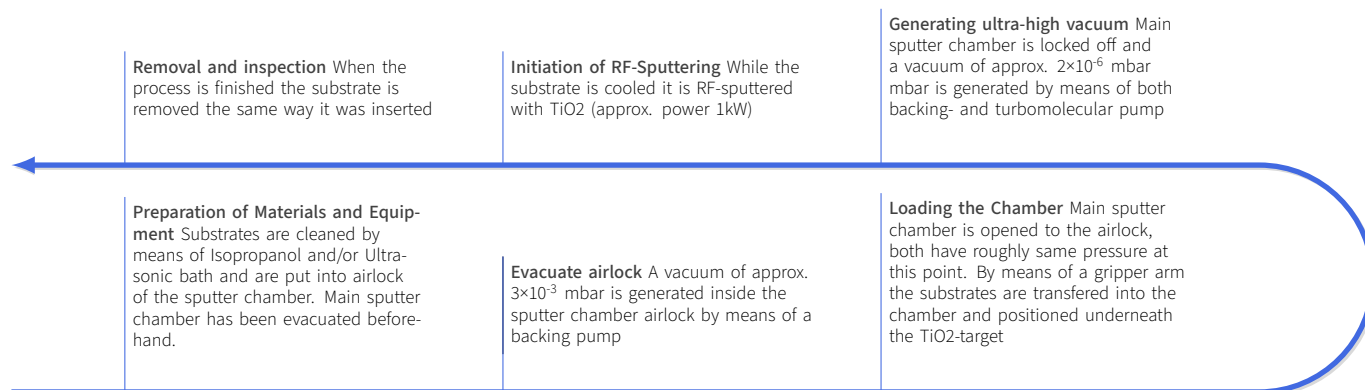


Fig. 2 | Process steps undertaken in the fabrication of a TiO₂-layer using sputtering.

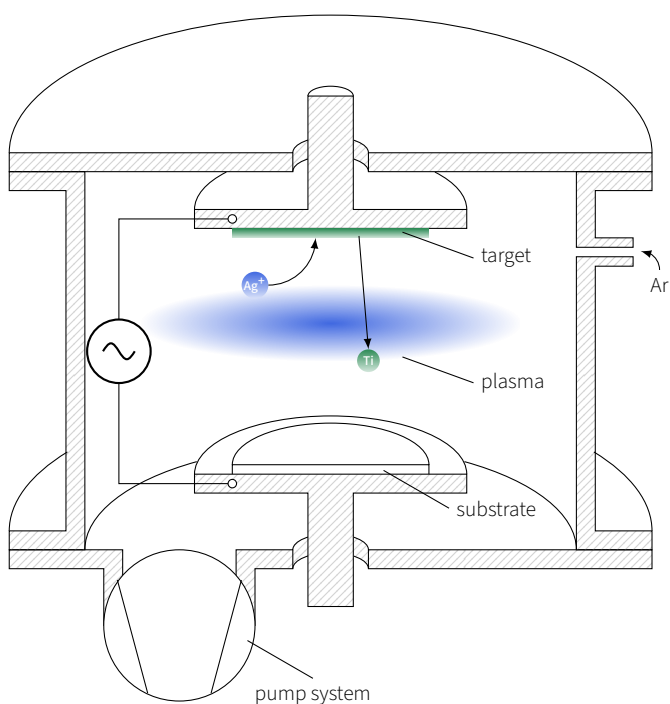


Fig. 3 | Schematic representation of a sputter reactor.

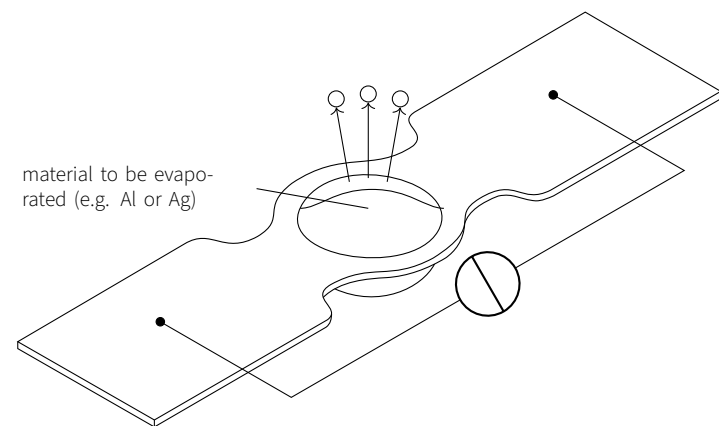
energetic electrons collide with argon atoms, ionizing them (removing electrons to create Ar⁺ ions) and forming plasma, which is a mixture of electrons, ions, and neutral atoms. The positively charged Ar⁺ ions are thereby accelerated towards the (negatively charged) cathode at which a so called target, consisting of the material to be deposited is placed. Collisions of these ions with the surface release atoms from the target material. Opposite the target the substrate is placed towards which the released atoms diffuse and deposit on. This presumes that the target material is electrically conducting, otherwise the charge of the positive ions impinging on the non-conductive surface will cause it to charge up and eventually suppressing the bombardment by further ions.

Sputtering with non-conductive material is still possible by replacing the DC-voltage with an AC-voltage at RF-frequencies (typically ranging from ~10kHz to ~10MHz). At such frequencies, the heavy ions are not able to follow the fast changes of polarity whereas the mobility of the electrons is excellent, they can follow the field changes without any delay. As a result a steady amount of electrons will accumulate on the insulating target causing a DC bias. Magnetron sputtering employs magnets near the target to manipulate ion trajectories, enhancing sputtering efficiency, however this shall not be discussed further. It should be added that by swapping the polarity of the electrodes the plasma can be used to remove material from the substrate itself (plasma etching). For instance, this can be useful when removing an oxide layer from the substrate before depositing a different material. The energy from the plasma further influences the properties of the deposited layer, affecting factors like adhesion and surface mobility. Although the deposition is generally non-directional, adjustments can be made to coat complex structures thoroughly. Parameters such as substrate voltage, temperature, chamber geometry, and introducing reactive gases can be optimized to control the film's characteristics [5, 6, 7, 8].

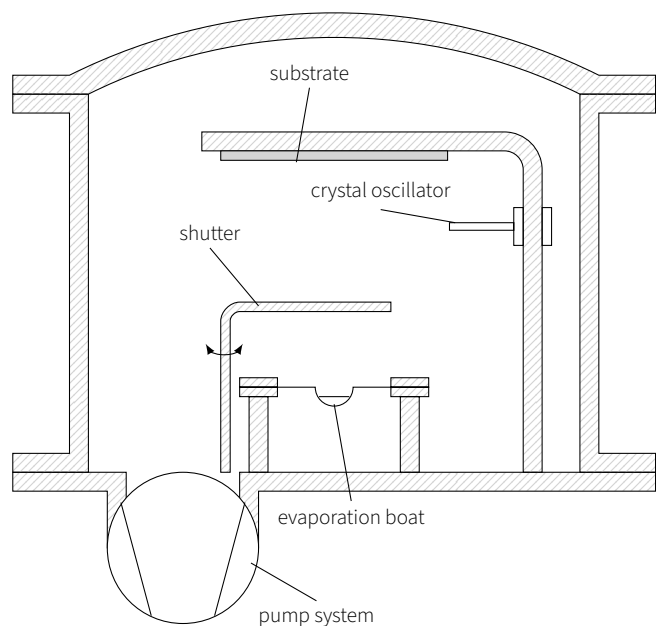
Experimental In our experiment we aim to fabricate a thin layer of TiO₂ onto a Silicon substrate. Since TiO₂ is an electrically insulating material the RF-sputtering technique is applied. The process steps which have been carried out are outlined in the flow diagram in Fig. 2.

4 EVAPORATION

Another PVD-process which shall be discussed here is the so called (thermal) evaporation method. Evaporation is one of the oldest methods for depositing thin films on many different substrate materials, including polymers, metals and glasses. The layers can consist of metals, insulators and semiconductors. The basic principle of the method is the vaporization of the material in a



(a) Evaporation boat. Typically, tungsten is the material of choice for these.



(b) Schematic representation of an evaporation reactor.

Fig. 4 | Evaporation.

heated source, which then condenses on all exposed (cold) surfaces within the mean free path of the gas phase. The source can take the form of a filament or an electrically heated crucible containing the material to be vaporized. The evaporation rate is determined by the vapor pressure of the source material at the evaporation temperature and can reach a high value using tungsten or molybdenum crucibles, where temperatures of up to 2800K can be achieved. Even higher temperatures can be achieved by heating the material to be deposited with a focused electron beam, which also avoids contamination from the crucible. The process is carried out in an evaporation chamber under high vacuum ($<10^{-2}$ Pa) to ultra-high vacuum ($<10^{-6}$ Pa). The better the evacuation of the chamber, the higher the quality and purity of the deposition. The most important process parameter for thermal evaporation is the substrate temperature, by changing which layer properties such as porosity, adhesion, surface roughness and crystallinity can be controlled. In Fig. 4b shows a schematic representation of the thermal evaporation process. A predefined layer thickness can be achieved through controlled usage of a crystal oscillator and a shutter.

The main advantages of thermal evaporation are a high deposition rate and high thickness of the layer that can be produced. A major disadvantage is that while flat surfaces can be coated effectively,

the sides and bottoms of deeper or more complex structures may not be sufficiently covered or may not be reached at all. An alternative can be the above discussed sputtering, both methods are compared in Tab. 2, taken from [6]. It should be noted that there are many other PVD-methods which include cathodic arc deposition (widely used in industry), ion plating, laser beam ablation and thermal spraying. In addition to PVD, there are a number of other coating methods. These include, for example, chemical vapor deposition (CVD) or epitaxial layer growth. The choice of method can depend heavily on the materials used. On the other hand, requirements (e.g. layer thickness, uniformity, purity) and costs should always be compared appropriately [6, 7, 8].

	Evaporation	Sputtering
Depositionrate	up to $\mu\text{m/s}$	$<1\text{nm/s}$
Choice of material	limited	fast unlimited
Purity	very high at high vacuum	impurities
Heating of substrate	little	could be high
Stoichiometry	difficult (even with multiple sources)	easier
Changing the film material	easy	expensive
Decomposition of material	high	low
Scaling up	easy (e-beam difficult)	easy
Costs	low	high
In situ thickness control	easy	difficult
Lift-Off Masking	easy	difficult
Step coverage	poor	good, could be controlled

Tab. 2 | Comparison of thermal evaporation with sputtering. Taken from [6]

Experimental In our experiment we aim to fabricate a thin layer of Ag onto a Silicon substrate. The deposition of Ag is preceded by cleaning the surface using nitrogen plasma. The different process steps are outlined in detail in Fig. 5. It should be noted that in our case we neither use a shutter nor a crystal oscillator to control the film growth. The deposition process is therefore finished once the Ag has been completely evaporated.

5 MEASUREMENTS

5.1 REFLECTOMETRY

A reflectometer is used to measure the layer thickness of the sputtered TiO₂ film. A reflectometer directs a beam of light onto the surface of a sample and measures the intensity of the reflected light. The basic underlying theory is illustrated in Fig. 6. When the light hits the film applied to the wafer, some of the light is reflected by the surface and some of the light passes through the film and is reflected at the interface between the film and the substrate below. The light rays reflected from the surface and the interface interfere with each other and, depending on the film thickness, result in different measured intensities, which can be converted into a film thickness. This presumes that the material to be measured is transparent for the light-source used and its (complex, wavelength dependent) refractive index is known. Moreover, it should be noted that combination of materials with

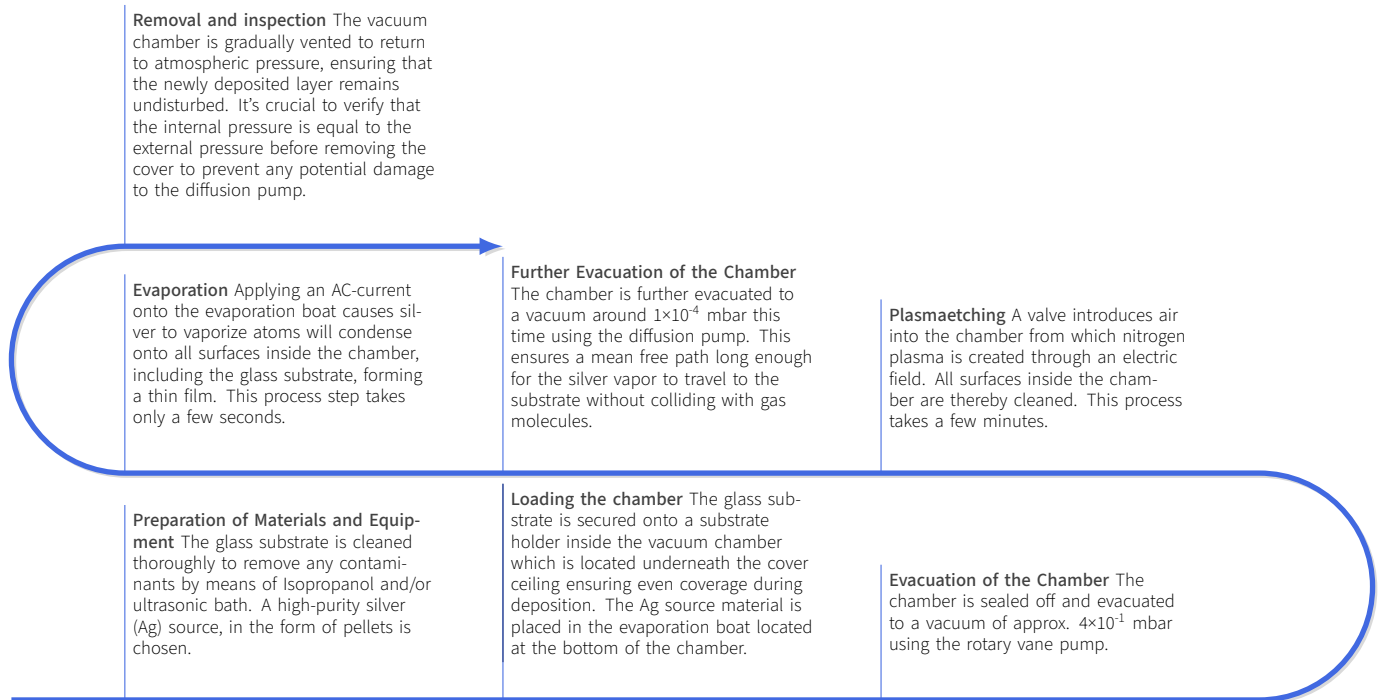


Fig. 5 | Deposition of Ag onto a glass substrate by means of evaporation.

very different reflectivity coefficients can impede the measurement significantly. An advantage of this method is that it leaves the sample undamaged.

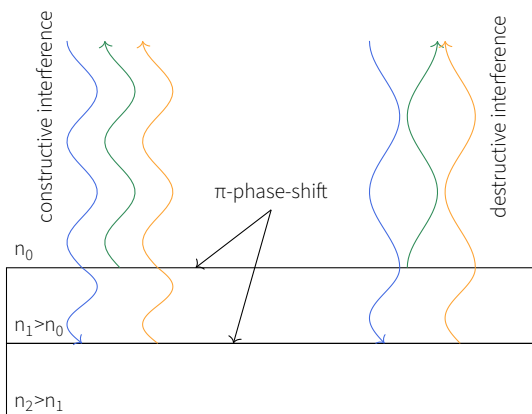


Fig. 6 | Basic operating principle of a reflectometer.

Experimental From the underlying theory (which shall not be discussed here) reference measurements are required to eliminate unknown parameters such that a fitable function can be obtained. For this, we use a wafer fabricated specifically for this purpose consisting of silicon the surface of which is partly coated with a SiO₂ layer with known thickness. After measuring the reflectivity of the bare silicon, the SiO₂ surface as well as the intensity of the ambient light (a special prism is used here), the program predicts the thickness of the SiO₂ layer. If the predicted value is in good agreement the value provided (which was the case), the program is calibrated. After the calibration, the program is informed that the material of the film to be measured is TiO₂. Subsequently, a measurement series of five different locations on the substrate is carried out. The curves obtained from the measurements are depicted in Fig. 7b, along with their respective goodness of fit values. The extremes of the curve correspond to constructive or destructive interferences, i.e. phase differences between incident and reflected rays of even or odd multiples of π .

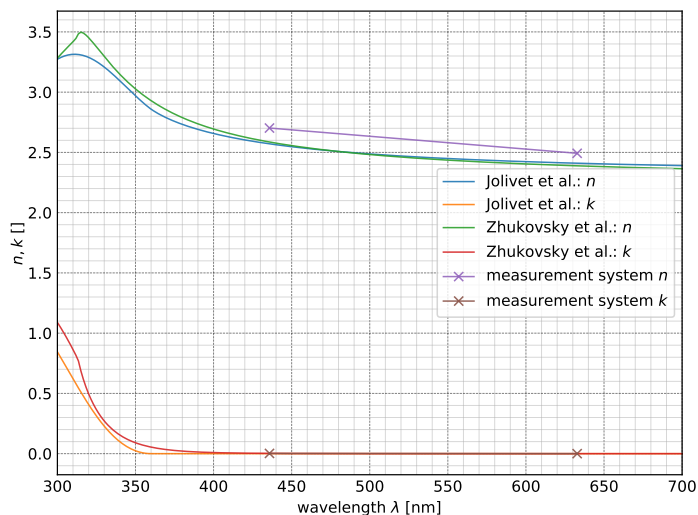
Discussion As stated above, to accurately predict the value of the film thickness we need the correct wavelength dependent refractive index of TiO₂. At the time of measurement this value (n and k) was only given for two different wavelengths. The program used for obtaining the fitted parameters, interpolates between these values as is depicted in Fig. 7a. We investigate the accuracy of this approach by comparing the interpolation with values found in recent literature. For amorphous TiO₂, Jolivet et al. [9] and Zhukovski et al. [10] provide credible data. As can be seen in the figure the value for n used for the measurement deviates slightly (roughly 5%) from the nearly matching literature values. Therefore, it is reasonable to assume a system error from the measurement by 5% since n corresponds directly to optical distance. Our measurement therefore yields for the thickness d_{TiO_2} of the TiO₂-layer:

$$d_{\text{TiO}_2} = \bar{d} \pm \sqrt{\sigma_d^2 + (0.05\bar{d})^2} = 144\text{nm} \pm 7\text{nm},$$

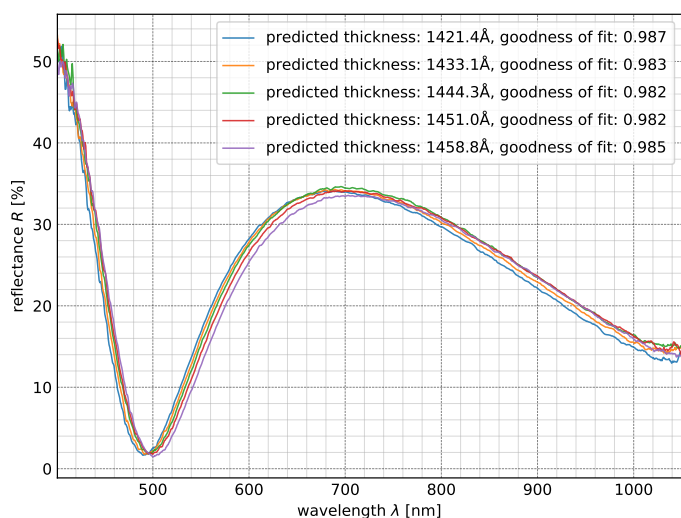
where σ_d denotes the standard deviation of the measurement series.

5.2 PROFILOMETER

As silver is impenetrable to visible light, the Ag-layer on our glass substrate cannot be measured using reflectometry. It is therefore determined with the help of a profilometer. A profilometer is an instrument for measuring the surface profile, roughness and thickness of thin layers and structures on a substrate. The surface is touched with a needle attached to an arm. The needle moves in a straight line across the surface of the substrate. If the needle detects changes in the surface topography (e.g. at the edges and top of a structure), the arm with the needle moves up and down (see Fig. 8a). The vertical movement is recorded and a profile of the surface features along the measurement path is obtained from the recorded data. To measure the thickness of a structure, the profilometer can measure the distance between the base plane of



(a) Real refractive index and extinction coefficient of TiO₂ with respect to wavelength. Data taken from [10, 9].



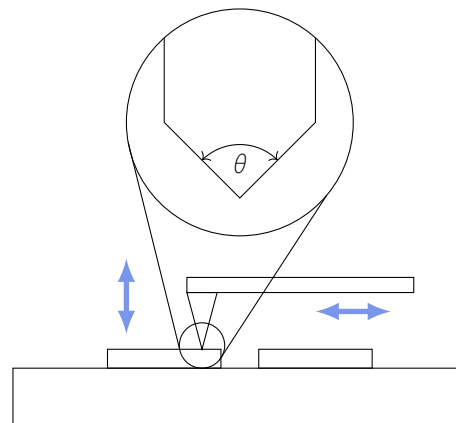
(b) Reflectance curves measured through reflectometer.

Fig. 7 | Reflectometry data

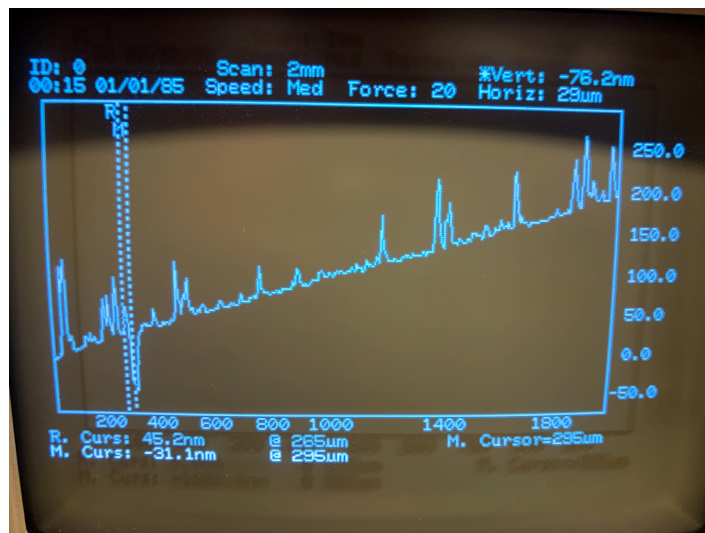
the surface (substrate) and the top of the structure lying on the substrate. In cases where the shape of the topology of the surface is of interest, the angle θ of the needle tip has to be taken into account, when analyzing the measured data-points. Mathematically it represents the convolution of the shape of the needle tip and the surface topology. A clear advantage of this method from reflectometry is that the properties n, k do not have to be known. However, in many cases the needle of the measurement device damages the surface. This can become a problem if the surface consists of devices that can easily be destroyed by very small forces.

Experimental In our case, we scratch off the Ag in a straight line at an arbitrary position of the substrate creating the desired topology for the measurement. The recorded layer thickness for one sample can be seen in Fig. 8b. The measurement is carried out by placing the two cursors in an appropriate position. The difference of both y-values gives the measurement result.

Discussion It should be noted that the resolution of this particular device is poor and we only made two measurements. Therefore, the results should only serve as an approximate value for the film thickness. As seen in the figure the value for the thickness of the film at this particular location reads 76.2nm. For the second



(a) Basic operating principle of a profilometer.



(b) Measured data over a scratch of the Ag-surface.

Fig. 8 | Profilometer.

measurement we get 82.3nm. We think it is reasonable to assume that the thickness roughly ranges from 60nm to 90nm. Large variations of the thickness were to be expected since in our experiment the distance from substrates to the evaporation boat varied and thus the amount of Ag that deposits on each substrate.

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