

## Lecture

# Sputter depth profiling of thin films by AES, XPS and ToF-SIMS

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Surface analysis techniques like Auger- and X-ray Photoelectron Spectroscopy (AES and XPS), respectively, together with Time-of-Flight (ToF-SIMS) are restricted to the outermost surface layers -  $\leq 5$  nm - due to either the low mean free path -  $\lambda$  - of the emitted electrons or the sample depth of ions under static secondary ion emission conditions. The sensitivity of the three methods ranges from below 1 % (AES; XPS) to ppm or ppb (ToF-SIMS) of a monolayer ( 1 monolayer corresponds to approx.  $10^{15}$  particles per  $\text{cm}^2$ ). By combining electron and/or ion spectroscopies with noble gas or reactive sputtering films of a thickness larger than a few nm as well as buried

interfaces can be analyzed. Depending on the film thickness different approaches are usually used as illustrated in fig. 1:

- (a) angular dependent measurements with AES and XPS [1]
- (b) lapping or ball cratering by mechanically polishing a crater and using a line scan with electrons or ions [2]
- (c) depth profiling with ions by combining electron ( $e^-$ ), photon ( $h\nu$ ) or ion bombardment with sputter removal of the surface which is the subject of this presentation

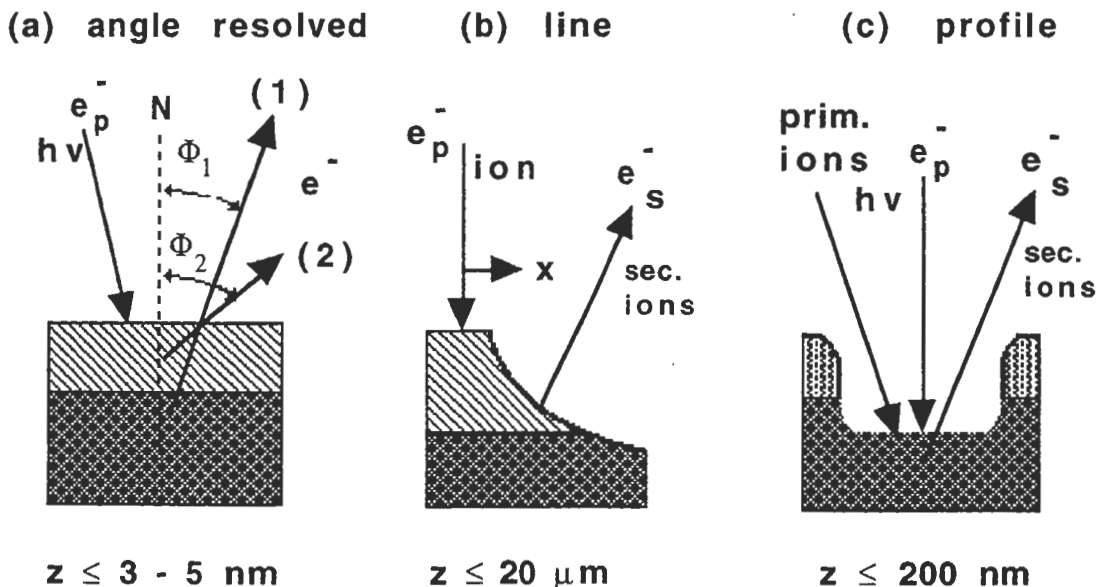


fig. 1. schematic representation of in-depth probing

**Part I** deals with the physical aspects of sputtering illustrated by AES and XPS depth profiles.[3-6]. We will use the thin film Ta<sub>2</sub>O<sub>5</sub>/Ta standard to discuss the basic definitions of depth profiling. The conversion of the sputter time axis is accomplished by using either the calibrated thickness of the Ta<sub>2</sub>O<sub>5</sub>/Ta standard and/or elemental standards for which Ar<sup>+</sup> sputter yields are known [7,8]. Their dependence on various parameters like energy and angle of incidence is shown allowing to convert to different experimental conditions different from 1 keV Ar<sup>+</sup> at normal incidence. Preferential sputtering in particular for metal alloys is discussed and how to correct for it. The interface width  $\Delta z$  is a criterion for the goodness of a profile. According to Hofmann [4]  $\Delta z$  can be described as the sum of several independent contributions like the ion beam roughening during sputtering, influence of the escape depth and geometrical factors dealing with the rastered beam area and crater edges. Practical examples [3,10,19] together with recent AFM results of sputtered surfaces will illustrate such contributions to the depth resolution. The improvement of depth resolution with the help of Zalar<sup>®</sup> rotation [9] is shown for AES and XPS profiles based on a round robin study. [10]

**Part II** illustrates the kind of chemical information that can be obtained from XPS profiles from applied work of our own laboratory. The improved adhesion of SiO<sub>x</sub> layers (1 ≤ x ≤ 2) on Polyethyleneterephthalate (PET) is correlated by the interface width of XPS depth profiles of the thin films after a plasma treatment of the PET surface [11]. SiO<sub>x</sub> does not exhibit preferential oxygen sputtering and maintains its stoichiometry which is identified and controlled by the chemical shift of the photoelectron binding energy. A second example demonstrates the usefulness of depth profiling for buried interfaces of Physical Vapor Deposited (PVD) layers used for micromechanical applications. The variation of the oxidation state is illustrated for this particular case. [12]

**Part III** illustrates ToF-SIMS profiles with and without postionization of the emitted particles. One example given is the investigation of non-homogeneous FeCrNb alloys for the study of corrosion behavior [13,14,20]. Imaging of the sample surface by ToF-SIMS allows one to select the area of interest with micron resolution and to perform a depth profile. The ionization probability of emitted particles from the metal substrate is in general low compared to the passive oxide film and reduces therefore drastically the signal intensity. Postionization of the emitted particles can help to overcome this problem. This is illustrated by a recent study on standard Ta<sub>2</sub>O<sub>5</sub> films with and without Excimer Laser postionization [15]. In addition, ultrahigh depth resolution with  $\Delta z \leq 1$  nm for 30 nm amorphous films is found for 12 keV Cs<sup>+</sup> bombardment. [16-18].

The presentation is concluded by a summary of the practical results together with an outlook for future improvements.

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