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Surface chemical analysis — Depth profiling — Measurement of sputtered depth

Analyse chimique des surfaces — Profilage d'épaisseur — Mesurage de l'épaisseur bombardée

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Contents

Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this Technical Report may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 15969 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 4, *Depth profiling*.

Introduction

This Technical Report is intended to be used as follows:

a) For the determination of the depth scale in sputter depth profiling where signal intensity is obtained as a function of sputtering time (or ion dose density). The sputtered depth per sputtering time is the sputtering rate (typically reported in nm/s).

b) To enhance the comparability of depth profiling data obtained with different instruments and to increase the reliability and use of depth profiling in industrial applications.

c) To serve as the basis for the development of International Standards on the measurement of sputtered depth.

Surface chemical analysis — Depth profiling — Measurement of sputtered depth

1 Scope

This Technical Report gives guidelines for measuring the sputtered depth in sputtered depth profiling. The methods of sputtered depth measurement described in this Technical Report are applicable to techniques of surface chemical analysis when used in combination with ion bombardment for the removal of a part of a solid sample to a typical sputtered depth of up to several micrometres.

2 Terms and definitions

For the purposes of this Technical Report, the following terms and definitions apply.

NOTE The terms used in this Technical Report follow basically the definitions in ASTM E 673-95c [1]. These definitions are to be modified to conform to those being developed by ISO/TC 201/SC 1 on *Terminology*. See also [2] and [3].

2.1
sputtered depth

distance z (m) (perpendicular to the surface) between the original surface and the analysed sample surface after removal of a measurable amount of matter as a result of sputter profiling, which is given by

$$z = \frac{m}{A \cdot \rho} \tag{1}$$

where

m is the removed sample mass (kg);

A is the sputtered area (m^2);

ρ is the density of the sample (kg/m^3)

2.2
crater depth

average distance (perpendicular to the surface) between the original surface and the region of a crater bottom from which the measured signal is derived

NOTE The crater depth is equal to the sputtered depth if primary-ion implantation and retention, which may cause enlargement ("swelling") of the sample in the direction perpendicular to the surface, is negligible [5]. If the sputtered depth is measured by crater depth measurement outside the analysis chamber, surface reactions (e.g. oxidation) may add to the swelling of the crater bottom, i.e. the crater depth is generally measured as being less than the sputtered depth.

3 Abbreviated terms

AES	Auger electron spectroscopy
AFM	Atomic force microscopy
EDS	Energy dispersive spectrometry
EPMA	Electron probe microanalysis
FIB	Focused ion beam
GIXR	Grazing incidence X-ray reflectivity
MEIS	Medium energy ion scattering
RBS	Rutherford backscattering spectrometry
SAM	Scanning Auger microscopy
SEM	Scanning electron microscopy
SIMS	Secondary-ion mass spectrometry
TEM	Transmission electron microscopy
XPS	X-ray photoelectron spectroscopy
XRF	X-ray fluorescence

4 Methods of determination of the sputtered depth

4.1 Crater depth measurement after sputter profiling

4.1.1 General description

Usually, the result of sputter profiling is a signal intensity as a function of the sputtering time. The total sputtering time corresponds to the crater depth and the average sputtering rate is obtained by dividing the crater depth by the sputtering time. Crater depth measurements are usually performed by mechanical stylus profilometry[6] or, less commonly in use, by optical interferometry. Optical instruments and scanned-probe microscopes give a two-dimensional view of the crater and its non-uniformities.

4.1.2 Mechanical stylus crater depth measurement

Mechanical stylus profilometers convert the deflection of a stylus in mechanical contact with the surface into a voltage that is amplified and then displayed directly on a strip chart, or digitized and processed in a computer. In some instruments, the stylus is scanned across the sample containing the crater, and in others the sample is scanned under the stylus. Profilometers typically produce one-dimensional line scans, though some modern instruments and scanned probe microscopes are capable of producing two-dimensional scans by making an automated series of closely spaced one-dimensional scans.

Stylus profilometry is appropriate for measuring the depths of craters in which the roughness of the original surface and that of the crater bottom are small compared to the crater depth. It is commonly used for craters made in semiconductors during SIMS depth profiling. The minimum depth that can be measured successfully depends on the acoustic and electronic noise of the profilometer as well as the surface roughness. In modern instruments the minimum depth may be as small as 10 nm, and the maximum may be as great as 100 µm.

To perform a crater depth measurement with a one-dimensional profilometer, a scan is made through the centre of the crater and over a sufficient distance of the unsputtered top surface on either side to establish an accurate baseline, as shown in Figure 1. Multiple scans are made over different traces through the crater centre to determine the repeatability of the crater depth measurement. The depth is measured on a computerized profilometer by determining the average height difference between a region in the centre of the crater at A and two regions of the reference surface on opposite sides at B and C. Figure 1 shows an example of a computerized profilometer trace of a sputtered crater in single crystal silicon approximately 0,5 µm in depth. The three pairs of vertical cursor lines indicate the regions over which the depth is averaged.

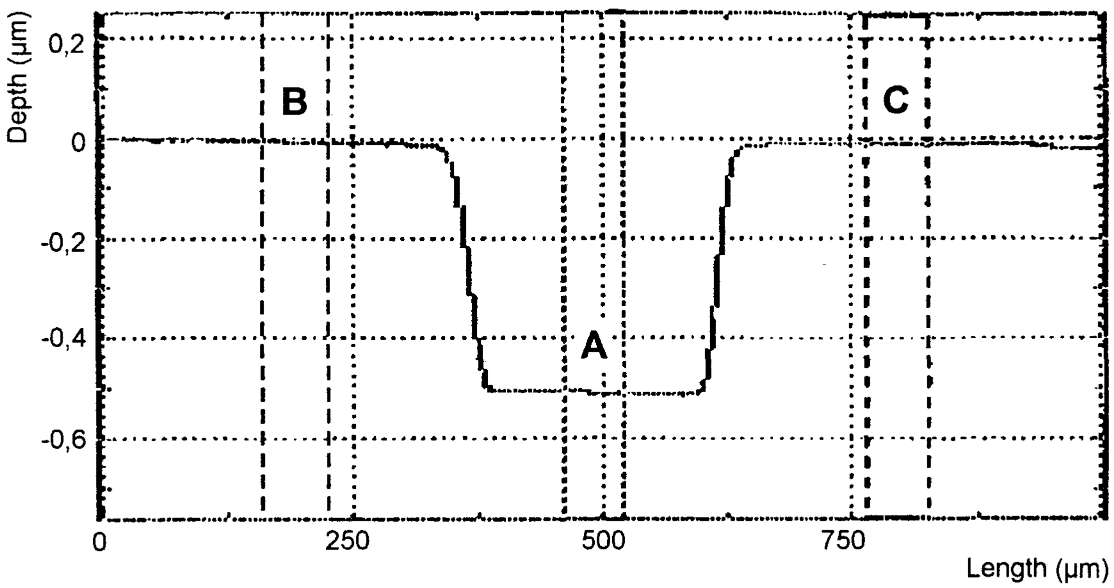


Figure 1 — Example of stylus profilometry trace of a 0,5 µm deep crater in silicon

The depth scale of the stylus profilometer is calibrated with standard step-heights or grooves that are traceable to fundamental length standards (wavelength of light). A typical calibration uncertainty is 1 % for a 1 µm standard gauge. The uncertainty of a crater depth measurement is a combination of calibration uncertainty and profilometer noise. In a recent round-robin experiment on craters in silicon, uncertainties ranged from ± 1,3 % for a 2 µm crater to ± 4,7 % for a 0,1 µm crater[6].

NOTE For the purposes of this Technical Report, typical uncertainties are given as one-standard-deviation uncertainties.

Advantages of stylus profilometry for crater depth measurements are that it is rapid, requires no sample preparation, and reveals the size, shape, and flatness of the crater bottom which are measures of the ion beam current density. A disadvantage is that corrections may be necessary to convert crater depth to sputtered depth in the case of non-negligible swelling or oxidation. In the case of layered structures with different sputtering rates, separate craters must be made for each interface so that the individual sputtering rates can be determined. Otherwise only an average sputtering rate is obtained.

4.1.3 Optical interferometry crater depth measurement

Optical interferometry is a simple and convenient non-contact method of crater depth measurement for which the equipment is relatively cheap to buy and easy to use.

This method utilizes a metallurgical microscope equipped with an interference attachment (Mireau or Michelson objective, sample tilting stage and monochromatic light source/interference filter) and is only applicable to smooth flat samples, for example flat glass, coatings on glass and semiconductor wafers. Generally, metal samples are too rough for this method to be suitable.

The crater to be measured is placed on the microscope sample stage, which usually is capable of producing a controlled tilting movement of the sample as well as the usual x-y translation. Using the interference objective or a normal objective, the crater of interest is located and placed at the centre of the field of view. This operation can be done with white light illumination. If a normal objective has been used, the interference objective is then put in place and the sample height adjusted to give white light interference fringes across the crater. The interference filter is put in place and the sample illuminated with monochromatic light. Using the tilting adjustment of the sample stage, the sample is tilted to spread the fringes to a suitable separation and/or to rotate them so that they produce a suitable contour map of the crater. Take care to ensure that there are no other craters on the sample near to the crater of interest that cause displacements of the fringes on either side of the crater that are to be used for the measurement. Produce a hard copy of the image.

Figure 2 shows an example: Using a straight-edged ruler draw two lines (A and B) through the centres of two adjacent fringes and measure the separation between them. Preferably one of these lines (A) should cross the crater. Draw a third line through the centre of a fringe running through the centre of the crater (C). Count the number of fringes intersected by the line (A) crossing the crater and estimate the fraction of a fringe spacing between that line and the line through the fringe in the crater (C). In the case of Figure 2, this fraction will be equal to the ratio of separation of lines B and C to that of A and B. Multiply this result by the half-wavelength of the light used for illumination to determine the crater depth.

This method is generally applicable to crater depths in the range 0,2 µm to 5 µm although, at the greater depths, surface roughening during profiling can cause problems. The errors associated with the measurement are: (a) the ability to count the fringes — getting this wrong usually produces an obvious error, (b) the uncertainty in estimating the fraction of a fringe — this should be less than 1/20 of the wavelength of the light used, and (c) the uncertainty in the wavelength of light used.

NOTE The greatest uncertainty comes from the estimation of the fractional fringe. This is an absolute amount, not a percentage. Consequently the percentage uncertainty is greatest for shallow craters and decreases with increasing depth. A total of 13 measurements by an experienced user on the crater shown in Figure 2 gave a crater depth of 325 nm and a standard deviation of 9 nm.

The optical image is also useful for showing the uniformity and any defects of the crater. Another optical method is confocal laser depth determination.

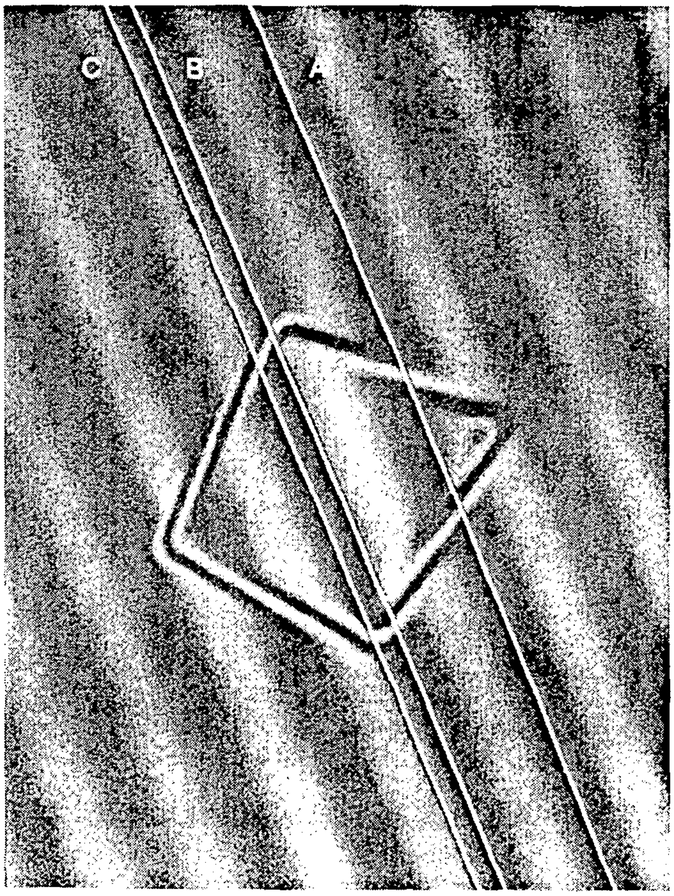


Figure 2 — Example photogragh of optical interferometry crater depth measurement

4.2 Comparison with sputter profiled samples having interfaces as depth markers

4.2.1 General description

A known depth of an interface or the depths of several interfaces can be used to determine the sputtered depth by comparison with the location of the 50 % drop of the plateau value on the sputtering time scale in the sputter profile. Errors involved are (a) the initial change of the sputtering rate (generally an initially slower sputtering rate is expected, caused by primary-ion implantation and the usual surface contamination layer, leading to typical errors of the order of 1 nm to 2 nm), and (b) a systematic shift of the 50 % plateau intensity (sputter profile interface location) to apparently lower depth as compared to the correct interface location[5]. This error is of the order of the signal escape depth [electron (AES, XPS) or ion escape depth (SIMS)] or the atomic mixing length, depending on the larger value. Under typical profiling conditions, the shift is of the order of 1 nm to 2 nm. Under favorable conditions, (a) and (b) may compensate and a linear relation between sputtering time and depth without a zero point shift is obtained. In multilayer profiling, both effects are similar at every interface and therefore always cancel in a first order approximation.

4.2.2 Reference materials

Any sample with one or several layers of known thickness can be used to determine the time needed to proceed from one interface to the other during a sputter profiling experiment with preset conditions for ion beam species, energy, incidence angle and ion formation chamber parameters determining the ion beam current density[7]. The latter can be given directly if the sputter yield for the sample material for the respective ion energy and incidence angle is known. For example, the certified reference material Ta_2O_5/Ta (BCR No. 261R)[8, 9], with certified oxide thickness $z(Ta_2O_5)$ of 30 nm and of 100 nm, yields immediately an "equivalent" thickness for the analysed layer if the sputtering time for the 50 % drop of the plateau intensity is used as a marker. By knowledge of the sputtering rate $\dot{z} = dz/dt$ of the analysed material M, $\dot{z}(M)$, relative to that of Ta_2O_5, $\dot{z}(Ta_2O_5)$, for the same ion beam conditions, the analysed layer thickness z is determined by the following relation:

$$z = z(Ta_2O_5) \cdot \frac{t(M)}{t(Ta_2O_5)} \cdot \frac{\dot{z}(M)}{\dot{z}(Ta_2O_5)} \qquad (2)$$

where $t(M)$ and $t(Ta_2O_5)$ are the sputtering times for the analysed layer and for the reference layer, respectively.

Owing to the build-up of the steady-state altered layer at the beginning of sputtering, the sputtering rate changes in a region of roughly two times the ion range impose a systematic error. Furthermore, sputtering-induced mixing as well as electron escape depth effects in AES and XPS shift the measured interface location as defined above in the direction nearer to the surface. With typical sputtering conditions for 30 nm layers, this shift is expected to be below 5 % and is proportionally less for thicker layers.

The advantage of the Ta_2O_5 reference material is that the data for equivalent thickness are readily available since it is frequently used to optimize depth resolution in AES sputter depth profiling.

Multilayer reference materials such as Ni/Cr[10] (NIST No. 2135b)[11] with alternating Ni and Cr layers of 63 nm and 53 nm thickness, respectively, can also be used to obtain the sputtered depth via equation (2). In this case, the thickness, relative sputtering rates and sputtering times of the Cr and Ni layers replace those of Ta_2O_5 in order to determine the sputtered depth of the analysed material (see also ISO 14606).

The advantages of the reference sample method are that no additional testing method is necessary, and that a direct depth calibration of samples with the same composition as well as a "relative" calibration of the ion gun is possible.

A disadvantage is that the sputtering rate of the reference sample generally differs from that of the profiled sample so that only an "equivalent depth" is obtained. A correction by equation (2) is possible but depends on the accuracy of available data.

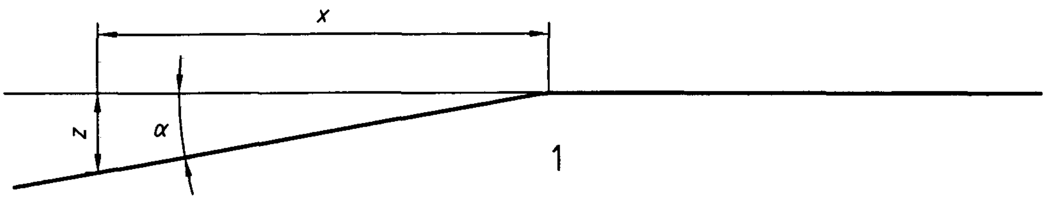
4.2.3 Interface depth determination for layered structures by independent measurements

4.2.3.1 Angle lapping, crater edge analysis or ball cratering and microscopy

Lapping of the surface with a small tilt angle, the lapping angle, allows the measurement of the thickness of thin-film structures by SEM imaging using the change in brightness at the interface[12]. Preferably, a line scan in SAM[13-18] is used. The relation between the measured length x between two interfaces and the respective distance z on the depth scale is given by the relation:

$$z = x \tan\alpha \tag{3}$$

where α is the lapping angle between the original surface and the surface generated by lapping (see Figure 3).



Key

1 Sample

Figure 3 — Angle lapping and crater edge profiling

Equation (3) applies also for the edge of a sputter crater and is the basis of crater edge profiling, where $\tan\alpha$ is typically in the range of 10^{-4} to 10^{-3}[13-15]. The angle α can be determined by profilometry (see 4.1). A well-defined constant value for the angle α can be obtained by changing the primary-ion dose linearly with the lateral coordinate x[16] or likewise changing the dwell time of a chemical reactant to form the angle α by chemical etching[17].

A special case of angle lapping is ball cratering with a rotating steel ball and an abrasive material, e.g. diamond paste[12]. The geometry of the generated crater is a spherical segment with diameter D in the surface plane (see Figure 4). Since the radius R of the ball is known (typically 1 cm to 3 cm), the depth z is given by the following equation as a function of the distance x from the crater edge:

$$z = \left[R^2 - (D/2 - x)^2\right]^{1/2} - \left[R^2 - (D/2)^2\right]^{1/2} \tag{4}$$

Since $R >> D$, a more convenient equation can be obtained:

$$z = \frac{D^2/4 - (D/2 - x)^2}{2R} \tag{5}$$

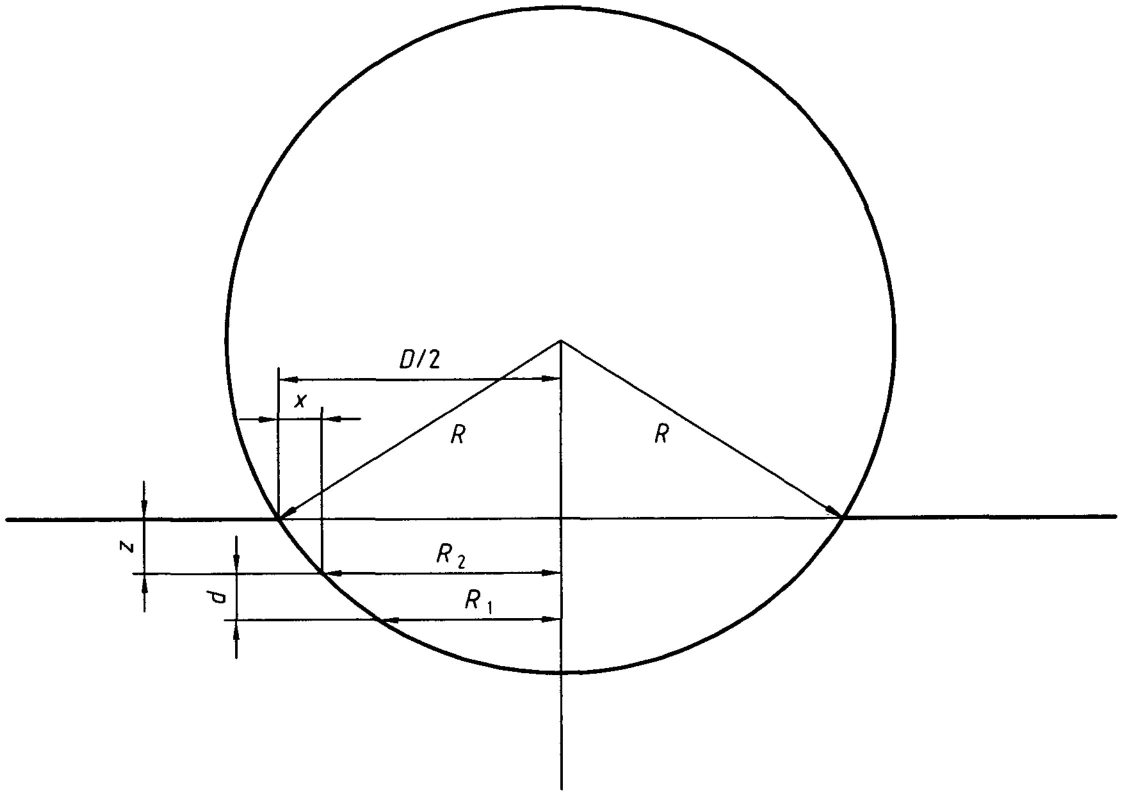


Figure 4 — Ball cratering

Measurement of the crater geometry with an optical microscope or a scanning electron microscope (SEM) allows direct determination of the thickness of a thin film between two interfaces characterized by an outer radius R_2 and an inner radius R_1 of the circular segment (see Figure 4). Considering the right-angled triangles formed by R and R_1 and R_2, respectively, generalization of equation (5) gives the approximate thickness d of the layer as the following equation:

$$d = \frac{R_2^2 - R_1^2}{2R} \tag{6}$$

Equations (5) and (6) are valid for the condition $R \gg D$ (accordingly, $R \gg R_2$) with an error smaller than $(R/64) \times (D/R)^4$. Usually R is larger than 1 cm and D and R_2 are both smaller than 1 mm, therefore the error is smaller than 15 nm or 1,5 % for a typical layer thickness of 1 µm.

The accuracy is limited mainly by the surface roughness induced by the abrasion process. For layers of 100 nm and above, typical uncertainties are ± (3 % to 7%), depending on the type of material and the original surface roughness [12, 18].

The advantage of angle lapping and ball cratering is that they are particularly useful and time saving methods for thick layer profiles in the range of several micrometres.

The advantages of ball cratering with respect to angle lapping are its easy application without the need for precise angle control and the fact that the slope $\tan\alpha$ is extremely small near the crater bottom.

4.2.3.2 Cross-section transmission electron microscopy or scanning electron microscopy

The thickness of thin-film structures can be measured by TEM [19] or by SEM [20] using cross-sectioned samples. Carefully prepared samples may be analysed with atomic resolution by TEM. After protection of the sample with an electrodeposited layer or a silicon wafer bonded to the sample surface by epoxy cement, the sample may be cut using a diamond saw or microtome and polished subsequently for study in the SEM directly. Resolutions in SEM are rarely better than 1 nm.

For TEM, to obtain sufficiently thin samples of the order, typically, of 100 nm thickness, the samples are usually thinned electrolytically and/or by ion bombardment [21, 22]. Focused ion beams (FIB) can also serve as a kind of microtome. The thickness of thin-film layered structures is measured on cross-section imaging micrographs obtained by TEM [22]. The inclination angle of the samples during the investigation by TEM should be considered for the final determination of the thickness. Care should be taken to ensure that the magnification of the instrument is calibrated. Depending on the quality of the interfaces and of the spatial resolution of the micrographs, the accuracy of film thickness measurements can be better than 1 % or one atomic layer.

Disadvantages of the TEM method are (a) that difficult and time-consuming preparation techniques are necessary and (b) that only a very small fraction of the sample is used, leading to results which may not be representative. SEM needs less sample preparation effort but has lower resolution, and the length scale cannot be as accurately calibrated as in TEM where it is done by using lattice images.

4.2.3.3 Rutherford backscattering spectrometry

In RBS, the areic atomic concentration (number of atoms per square metre) is measured as a function of the backscattering energy loss, from which the thickness can be determined provided that the density is known [23-25]. Therefore, for conversion of the data to a depth profile, the density of the thin-film material should be measured independently. Especially for multi-elemental thin films, the density calculated by summing the density of each element, normalized to its concentration, can be inaccurate by as much as 25 % (see the note 4.2.3.4). Accuracies with respect to the depth scale determination typically are 5 % to 20 %. The analysis depth is below 2 µm, with a depth resolution generally of 20 nm to 30 nm, but as low as 2 nm to 3 nm near the surface. Advantageous is the practically nondestructive analysis, which is insensitive to matrix effects.

NOTE Medium energy ion scattering (MEIS) has characteristics similar to RBS but, using ions with lower energy, it is confined to more shallow layers which can be detected with a depth resolution better by an order of magnitude than RBS. However, MEIS is not yet in a mature state, with not enough data being available for depth determination.

4.2.3.4 Electron probe microanalysis and energy dispersive spectrometry

EPMA and EDS can be used to determine the thickness of thin films in the micrometre region and below [26, 27]. This usage is based on common models of the depth distribution of electron-excited X-rays which depend on calculations for X-ray absorption, atomic number and possibly X-ray fluorescence correction.

For a known composition of the film, the thickness is obtained in terms of the quotient of mass m by the surface area A.

NOTE The mass per area m/A (areic mass) is usually given in µg/cm^2. Multiplication by 10^{-5} yields that value in the SI-unit kg/m^2. It is necessary to divide the latter quantity by the density ρ (kg/m^3) to obtain the thickness d (m) [see equation (1)]. As with 4.2.3.3, the accuracy depends on knowledge of the density ρ.

Typical thickness measurements with EPMA range from several nanometres to micrometres with an accuracy of ± 5 % and a precision of ± 0,5 %.

4.2.3.5 X-ray fluorescence spectrometry

For a layer thickness below about 100 nm, the thickness can be determined by the ratio of the signal intensity of the overlayer with that of a reference material of known composition [28, 29], e.g. an ion-implanted reference material [30].

For a layer thickness above about 100 nm, the thickness can be determined by the ratio of two signal intensities, one corresponding to the overlayer and the other to the substrate beneath, obtained at two sufficiently different take-off angles [29, 30].

4.2.3.6 Grazing incidence X-ray reflectivity measurement

In grazing incidence X-ray reflectivity measurements (GIXR) [31-34], the signal intensity as a function of the angle of incidence near total reflection allows the determination of layer thickness without reference materials. The period of the reflected X-ray intensity as a function of the incidence angle directly yields the thickness with high precision (± 0,1 nm) and accuracy (± 1 %) [33, 34].

4.2.3.7 Ellipsometry

Thickness measurement by ellipsometry with monochromatic light requires both the refractive index and the absorption coefficient to be known at the measurement wavelength [4, 35, 36]. The precision of this method is degraded by variations, over regions smaller than the light beam spot size, in substrate flatness, thickness and index of refraction.

4.2.3.8 Chemical analysis

Quantitative analytical chemistry can be applied to obtain the mass of a thin film of a given area, and from that data, with the density, the thickness of the thin film can be obtained [37].

4.3 Typical applications and uncertainties of the different methods

A comparative survey of the different methods with respect to typical applications and to typical uncertainties is given in Table A.1 (see also the note in 4.1.2).

Annex A

Survey of typical applications and uncertainties of the different methods

Table A.1 — Survey of typical applications and uncertainties of the different methods

Subclause	Method/technique	Typical application		Typical uncertainty	
		Depth range (nm)	Material/remarks	nm	%
4.1.2	Mechanical stylus	100 to 10 000	Hard	5	1 to 5
	AFM	2 to 700	Hard	2	2
4.1.3	Optical interferometry	200 to 5 000	Polished, reflective	10	0,2 to 5
	Confocal laser system	10 to 500 000	Non-transparent	10	2
4.2.2	Reference material	2 to 500	—	2	2
4.2.3.1	Angle lapping	100 to 50 000	Hard	5	1 to 5
	Crater edge analysis	20 to 10 000	—	2	1 to 10
	Ball cratering	500 to 50 000	Hard, layered structures; thick films	20 (depends on interface roughness)	3 to 7
4.2.3.2	Cross-section TEM	10 to 1 000	—	0,2	1
	Cross-section SEM	10 to 300 000	Change in atomic No.; contamination problems	5 to 10	2
4.2.3.3	RBS	100 to 30 000	—	5 to 20	5 to 20
4.2.3.4	EPMA and EDS	5 to 1 000	—	2 to 20	5
4.2.3.5	XRF	100 to 100 000	—	10 to 10 000	10
4.2.3.6	GIXR	1 to 1 000	—	0,1	1
4.2.3.7	Ellipsometry	1 to 5	Non-transparent	0,1 to 1	1
		1 to 10 000	Transparent	0,1 to 1	1
4.2.3.8	Chemical analysis	10 to 100 000	—	1 to 10	5 to 10

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