TECHNICAL REPORT

ISO/TR 15969

Second edition

Surface chemical analysis — Depth profiling — Measurement of sputtered depth

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

PROOF/ÉPREUVE

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO/TR 15969:2001), which has been technically revised.

The main changes compared to the previous edition are as follows:

— in the Scope, the applicable range of depth has been specified more clearly;

— Clause 3 has been revised according to the latest edition of the ISO 18115 series;

— in 4.2.2, the information on reference materials has been updated;

— Table A.1 bas been updated.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document 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 document provides guidelines for measuring the sputtered depth in sputtered depth profiling.

The methods of sputtered depth measurement described in this document 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. The depth typically determined by this approach is between 1 nm to 500 µm.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies. See also[1] and[2]

ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

ISO 22493, *Microbeam analysis — Scanning electron microscopy — Vocabulary*

ISO 15932, *Microbeam analysis — Analytical electron microscopy — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1, ISO 18115-2, ISO 22493 and ISO 15932 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
sputtered depth
distance z (in 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 Formula (1):

$$z = \frac{m}{A \cdot \rho} \quad (1)$$

where

m is the removed sample mass (kg);

A is the sputtered area (m^2);

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

4 Methods of determination of the sputtered depth

4.1 Crater depth measurement after sputter profiling

4.1.1 General description

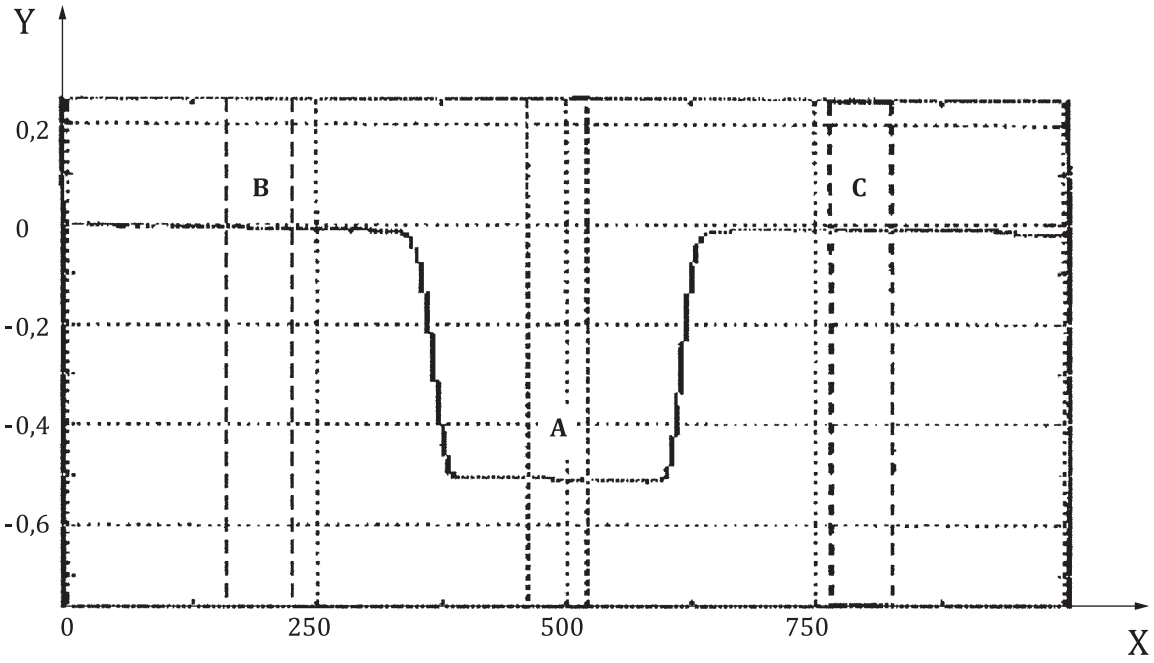
Usually, the result of sputter profiling is a signal intensity as a function of the sputtering time. The total sputtering time corresponds to the crater depth and the average sputtering rate is obtained by dividing the crater depth by the sputtering time. Crater depth measurements are usually performed by mechanical stylus profilometry[3] 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 can produce 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 can be as small as 10 nm, and the maximum can 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.



Key

X length (µm)

Y depth (µm)

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[3].

NOTE For the purposes of this document, 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

— it reveals the size, shape, and flatness of the crater bottom which are measures of the ion beam current density.

A disadvantage is that corrections can 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 are necessary 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 can produce 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) crosses 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 is 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 0,01 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.

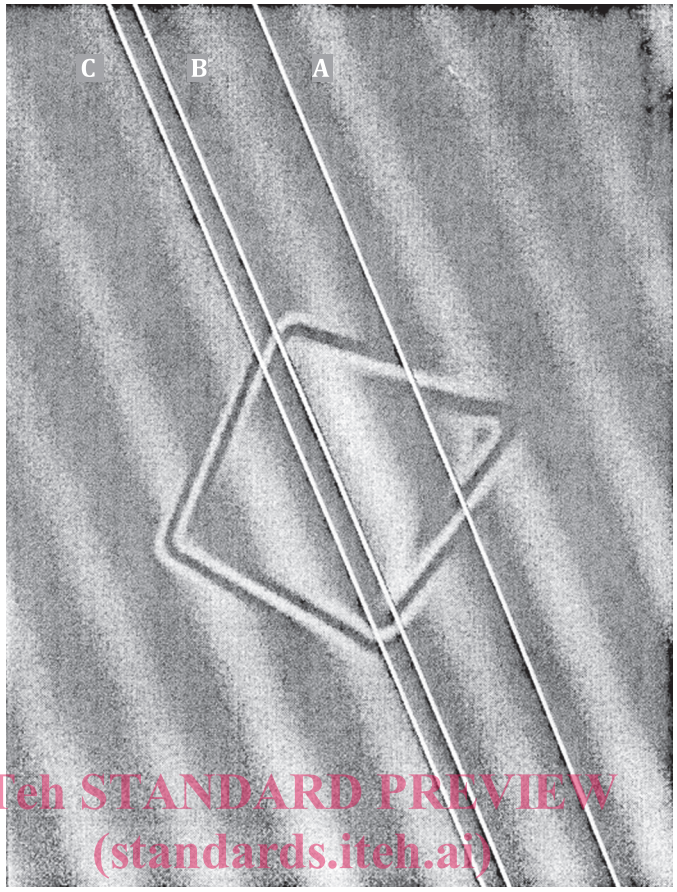


Figure 2 — Example photograph of optical interferometry crater depth measurement

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[4]. This error is of the order of the signal escape depth [electron: Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS); or ion escape depth: secondary-ion mass spectrometry (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 favourable conditions, a) and b) can 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