PUBLISHED DOCUMENT

PD ISO/TR
22335:2007

Surface chemical analysis — Depth profiling — Measurement of sputtering rate: mesh-replica method using a mechanical stylus profilometer

ICS 71.040.40

BSI
British Standards

National foreword

This Published Document is the UK implementation of ISO/TR 22335:2007.

The UK participation in its preparation was entrusted to Technical Committee CII/60, Surface chemical analysis.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 August 2007

ISBN 978 0 580 54014 1

Amendments issued since publication

Amd. No.	Date	Comments

TECHNICAL REPORT

ISO/TR 22335

First edition
2007-07-01

Surface chemical analysis — Depth profiling — Measurement of sputtering rate: mesh-replica method using a mechanical stylus profilometer

Analyse chimique des surfaces — Profilage en profondeur — Mesurage de la vitesse de pulvérisation: méthode par empreinte de grille au moyen d'un profilomètre à stylet mécanique

Reference number
ISO/TR 22335:2007(E)

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 22335 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 4, *Depth profiling*.

Introduction

This Technical Report has been prepared for the experimental determination of ion-sputtering rates for depth profiling using Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS), where ion sputtering is carried out over a region with an area between 0,4 mm^2 and 3,0 mm^2. For this Technical Report, a replica pattern is first formed on a specimen surface by ion sputtering through a grid mesh, of appropriate size, which is placed in contact with the specimen. The ion-sputtering rate is determined from the quotient of sputtered depth measured by a stylus profilometer and sputtering time by assuming a constant sputtering rate. This Technical Report provides a method to convert the ion-sputtering time scale in a depth profile to depth.

blank

Surface chemical analysis — Depth profiling — Measurement of sputtering rate: mesh-replica method using a mechanical stylus profilometer

1 Scope

This Technical Report describes a method for determining ion-sputtering rates for depth profiling measurements with Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) where the specimen is ion-sputtered over a region with an area between 0,4 mm^2 and 3,0 mm^2. This Technical Report is applicable only to a laterally homogeneous bulk or single-layered material where the ion-sputtering rate is determined from the sputtered depth, as measured by a mechanical stylus profilometer, and sputtering time.

This Technical Report provides a method to convert the ion-sputtering time scale to sputtered depth in a depth profile by assuming a constant sputtering velocity. This method has not been designed for, or tested using, a scanning probe microscope system. It is not applicable to the case where the sputtered area is less than 0,4 mm^2 or where the sputter-induced surface roughness is significant compared with the sputtered depth to be measured.

2 Terms and definitions

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

2.1
sputtering time
time for which the specimen surface is ion-bombarded

2.2
sputtered depth
distance (perpendicular to the surface) between the original surface and the analysed specimen surface after removal of a measurable amount of material as a result of sputter depth profiling

[ISO 18115:2001 [1]]

2.3
sputtering rate
quotient of sputtered depth and sputtering time

2.4
grid mesh
electroformed mesh, typically of 3 mm overall diameter, consisting of an array of mesh openings or apertures

NOTE A mesh of 75 lines per inch is recommended. [2]

3 Symbols and abbreviated terms

d sputtered depth

t sputtering time

R ion-sputtering rate

R_{ref} sputtering rate measured for a reference material

R_{rel} sputtering rate for the material of interest relative to that of a reference material

R_1 sputtering rate measured for material 1 of interest

AES Auger electron spectroscopy

SAM scanning Auger electron microscopy

XPS X-ray photoelectron spectroscopy

4 Principle

This procedure for measuring sputtering rates is separated into two parts:

a) the preparation of the specimen with the grid mesh followed by ion sputtering to form the replica pattern;

b) the sputtered-depth measurement.

The resulting sputtering rate R is calculated from a measurement of a sputtered depth d in a time t using Equation (1):

$$R = d/t \tag{1}$$

5 Procedure

5.1 Generating the replica pattern

5.1.1 General

To form the grid replica pattern, it is necessary to take a suitable specimen, place a grid on the surface and ion-sputter the specimen with the grid in position. These steps are discussed below.

NOTE Although many different grid materials can be used, the grid usually referred to is made from copper, which is commonly available and low in cost. Other grid materials have not been tested but are expected to behave similarly. Likewise, many different specimen materials can be used with this technique, but SiO_2 is used as an example.

5.1.2 Specimen surface preparation

This procedure requires both the specimen surface to be flat (within a few micrometres), such that there is good contact with the mesh grid, and a constant average ion-sputtering rate over the area to be analysed spectroscopically. The specimen flatness may be determined by a profile method using a stylus profilometer (that has been confirmed to be in proper working order for measurements at the 100 nm level) if there is concern about the roughness and waviness of the initial specimen surface. If the specimen surface is contaminated by small particles, they should be removed by an appropriate method such as blowing with an inert-gas jet, since non-uniform ion-sputtered areas may result, causing erroneous depth measurements. The

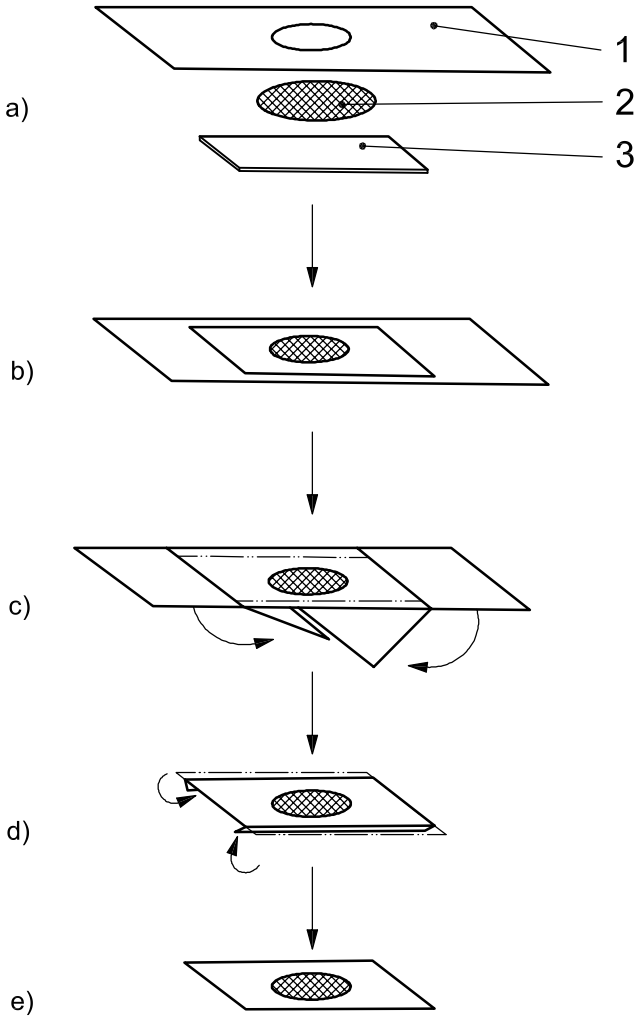
ion-sputtering rate uniformity within the grid mesh opening will be a significant factor for the repeatability of the measurements. The profilometer trace will reveal the shape of the sputter crater.

It is known that ion sputtering induces surface roughening on many polycrystalline specimens and that this roughening may be reduced by rotating the specimen during ion sputtering [3]. Rotation may reduce any uncertainties arising from the reduction in the sputtering rate that occurs as roughening develops, especially when profiling to significant depths in polycrystalline materials [4]. The mesh-replica method may be used with specimen rotation. In this case, it is important to align the analysed position at the rotational centre such that the axial "wobble" of the rotation axis is less than 10 % of the grid mesh opening.

5.1.3 Grid mesh specimen mounting procedures

5.1.3.1 The specimen is mounted under the grid mesh by one of the following methods or equivalent methods. It is important not to contaminate the specimen surface with dust particles in these procedures. Use, for example, dust-free gloves in a clean room.

a) A specimen-wrapping procedure [5] may be used to hold the mesh in place against the specimen (see Figure 1). This method is not recommended for specimens that are to be mounted vertically since the foil may not press the grid onto the specimen and slippage may occur. The grid mesh is first placed between the specimen and a thin metallic foil, such as aluminium foil, with a hole of a size which is smaller than the area of the grid mesh. It is important that the mesh and the hole in the aluminium foil are aligned well. The resulting sandwich, after wrapping, should have good electrical and mechanical contact. If the specimen feature to be analysed is smaller than one mesh opening, sometimes called the mesh aperture, proper alignment can be achieved by shifting the grid and viewing through an optical microscope. It is recommended that the wrapped specimen be inspected with an optical microscope to ensure that there is good contact between the specimen surface, the mesh grid and the wrapping foil. It is recommended that a flexible foil made from another material be used when the specimen contains aluminium or aluminium is of interest. Likewise, it is recommended that alternative grid materials be used if the specimen contains copper.



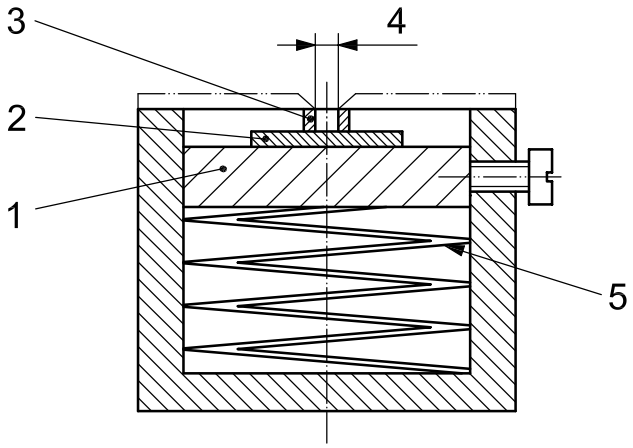
Key

1 foil
2 mesh
3 specimen

NOTE The hole in the foil is centred and placed over the mesh. Finally, the foil is folded, creating a sandwich, in accordance with Reference [5].

Figure 1 — Example of grid mesh specimen wrapping where the grid mesh is placed on the specimen and then covered by foil

b) A simple spring-loaded specimen holder shown in Figure 2 may also prove convenient. This specimen holder assembly gently squeezes the grid between a fixed aperture and the specimen that is supported by a base plate (platen). Good electrical and mechanical contact is made in this way. The bevel and sharpness of the fixed aperture edge must be considered in relation to the possible transference of material onto the specimen surface. This holder may be used for vertically mounted specimens.

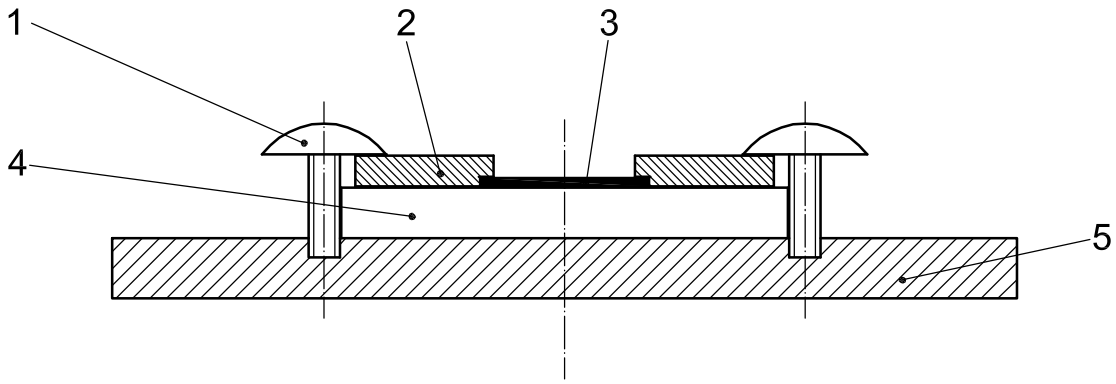


Key

1 cylindrical support base
2 specimen
3 3-mm grid (note that the grid thickness is greatly exaggerated in the drawing)
4 2,5-mm aperture
5 spring

Figure 2 — Cross-section of the spring-loaded specimen holder [2]

c) A simple screw-based specimen holder may also prove convenient (see Figure 3). This specimen holder assembly presses a mask onto the grid and then onto a specimen that is supported by a base plate (platen). The mask edge must be considered in reducing the ion-sputtered area and for the possible transfer of mask material onto the specimen surface.



Key

1 screw
2 mask
3 grid
4 specimen
5 platen

Figure 3 — Cross-section of the screw-based specimen holder

d) An electrically conductive adhesive (e.g. carbon or silver paint) is used to tack edges of the grid onto the specimen surface, as shown in Figure 4. This is a delicate procedure requiring practice. Complicating matters is the need to flatten the grid. To tack the grid down, small dots of wet adhesive are applied to the specimen and the grid is quickly placed in position before the adhesive dries. Alternatively, the grid can be held down and the adhesive applied over its edge. With either technique, the grid needs to be held down with a suitable instrument, such as tweezers, before the adhesive dries.

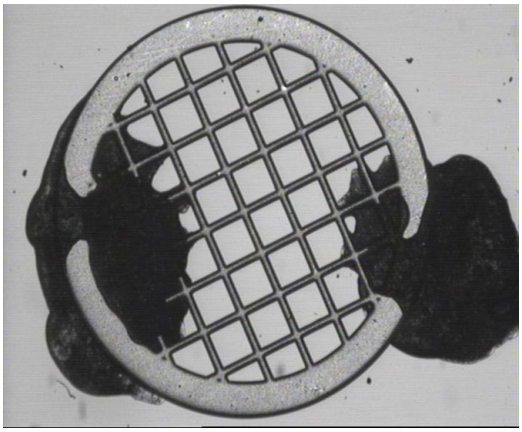


Figure 4 — Electrically conductive adhesive paint fixing the grid to the specimen

e) An electrically conductive carbon tape can also be used to fix the grid onto the specimen, as shown below. To accomplish this, small pieces of carbon tape with adhesive on at least one side are affixed to the edge of the grid sitting on the specimen to be sputtered. As with d) above, this technique requires some manual dexterity.

NOTE Outgassing of the carbon tape can be a source of contamination.

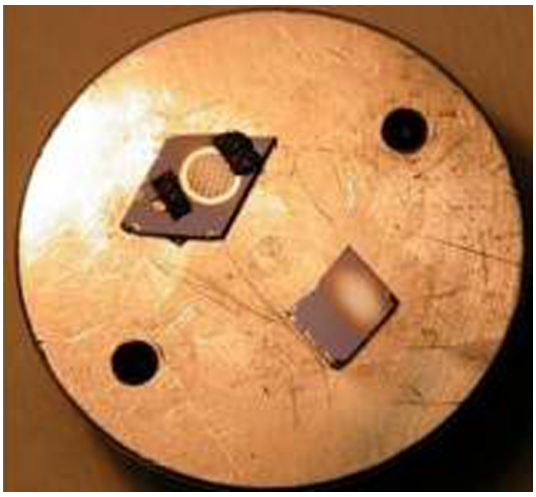


Figure 5 — Example of carbon tape used to hold the grid onto a specimen (upper) and an ion-sputtered specimen without the grid (lower)

5.1.3.2 Electroformed copper mesh is recommended for the grid since it conforms well to the specimen surface once sandwiched by the foil or restraining aperture. Copper mesh grids are mechanically flexible and make good electrical and mechanical contact with the specimen surface. However, if the specimen contains copper or copper is of interest, the user needs to select a different grid mesh material (several are available), since copper may be transferred by ion sputtering onto the specimen surface and then contribute to the spectrum.

NOTE Mesh grids are commonly available in copper from transmission electron microscopy supply houses. Grid meshes are also available in nickel and other metals.

5.1.3.3 In order to measure the sputtering rate using this mesh-replica method, a 75 mesh per inch grid is found best since larger meshes will expose too large an area to be uniformly ion-sputtered and a small mesh may result in grid material deposition over the sputtered area. The stylus profilometer used for measurement after ion sputtering needs to have a traverse range of at least 0,5 mm and a vertical range adequate to measure the crater depth, which is often in the range of 10 nm to 1 000 nm. The thickness of the mesh should be sufficiently thin to avoid shadowing in the ion-sputtering process and minimize sputter deposition from the grid mesh to the specimen surface and back onto the grid mesh. These issues are all discussed in Annex A.

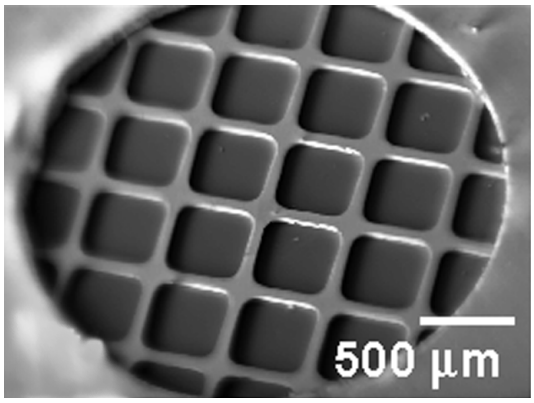
5.1.4 Ion-sputtering the specimen assembly

The specimen and grid assembly is set on the manipulator of the surface analysis instrument. Ion sputtering can be accomplished either using a stationary or raster-scanned ion beam. The geometry used should be the same as that normally used with the specific instrument. Guidance for the ion beam angle of incidence is given in Annex A. Sufficient sputtering time should be used to create a crater depth which can be accurately measured. When the specimen is a single layer on a substrate, the ion sputtering should be turned off in the first layer in order to get a precise sputtering rate. Record the total sputtering time t for estimating the sputtering rate.

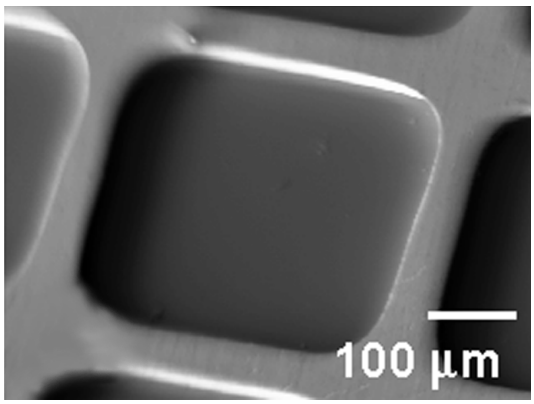
There are two general cases for ion sputtering, which depend upon the size of the analytical area. In both cases, the sputtering-rate measurement may need to be conducted on a separate specimen or position on the specimen other than that used for the depth-profiling measurement. In case b) below, it is occasionally possible to conduct a measurement with a stylus profilometer for the replica pattern which includes the actual analysed area or point. The application of case b) significantly depends on the instrumental geometry of the ion gun, primary beam source and electron analyser.

a) When the analysed area is larger than a mesh opening, as for many XPS instruments, the stylus profilometer measurements should include a larger area than that analysed to check the ion-sputtering rate uniformity.

b) For AES and small-spot-size XPS, the analysed area may be significantly smaller than the mesh opening [see Figure 6 b)]. After ion sputtering, the crater, including the masked part of the surface on both sides of the mesh opening, should be measured by the stylus profilometer procedure as described at 5.2.



a) Low magnification: the grid mesh is seen through the hole in the foil [a]



b) High magnification: the analysed area is selected to be in the centre of the mesh opening

[a] The specimen is below the grid mesh.

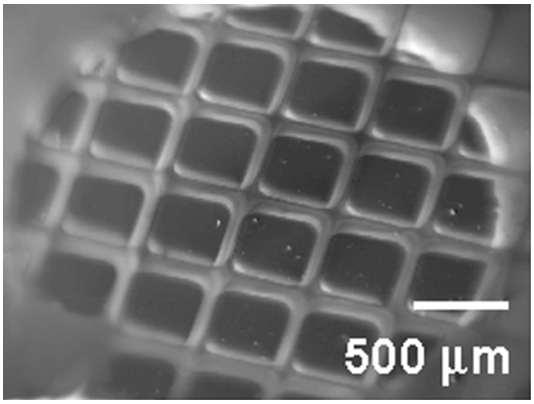
Figure 6 — Scanning electron microscope images before ion sputtering

5.2 Measurement of sputtered crater depth using a stylus profilometer

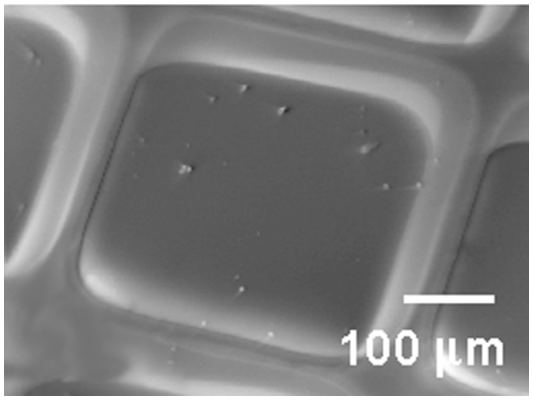
The stylus profilometer to be used needs to be calibrated by the manufacturer's recommended method before use or by one of the standard procedures given in the Note. The uncertainty of the measurement needs to be determined using a certified height reference standard which is in the range of the sputter crater depth.

NOTE ISO 5436-1 [6], ASME B46.1-1995 [7] or ISO 12179:2000 [8] may be used for the calibration of the stylus profilometer. Reference [9] gives additional guidance in the use of the stylus profilometer for measuring sputter craters.

Prior to the crater depth measurement, the specimen sandwich is separated and a replica pattern of the mesh on the specimen surface can be observed. The SEM image shown in Figure 7 is an example, although SEM observation is optional. The specimen is then mounted on the stylus profilometer stage and the profile of the mesh replica on the specimen surface measured.



a) Low magnification: the shadow pattern produced by the mesh can be clearly seen



b) High magnification: the sidewalls of the mesh, combined with the ion-sputtering geometry, is replicated into the specimen surface [a]

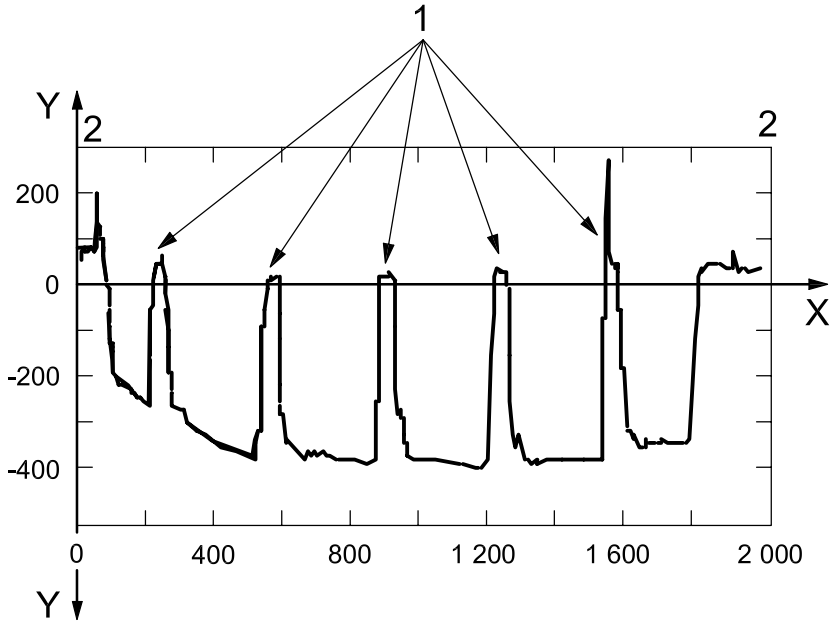
NOTE The ion beam was at 23° from the surface normal in the azimuthal direction from the lower left to the upper right corner in the picture.

[a] Small particle patterns in the mesh opening may be formed by small dust particles on the surface before sputtering.

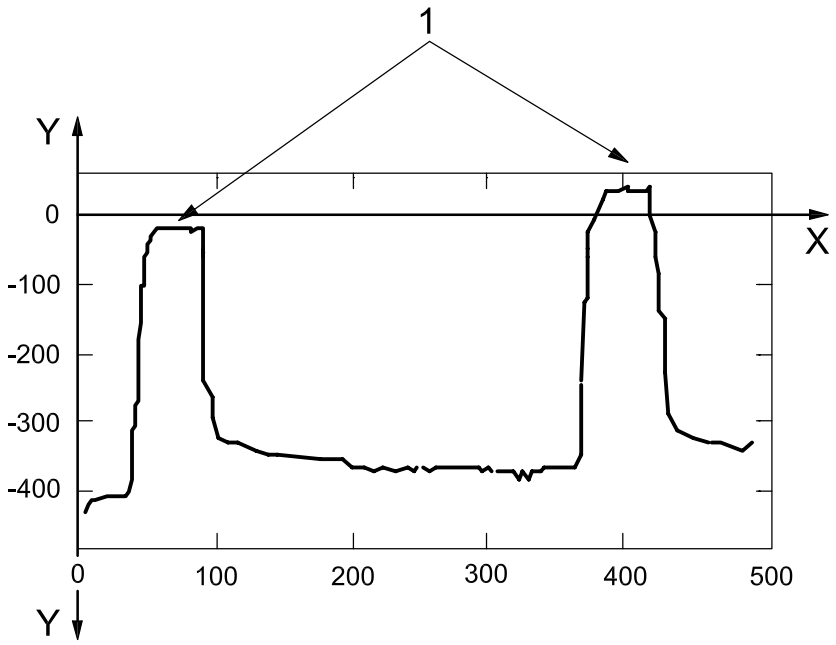
Figure 7 — SEM images of the grid mesh replica shown in Figure 6, after ion sputtering and removing the mesh

The sputtered depth d is measured as the distance between the original surface, defined by the height at the mesh bar positions, and the bottom of the crater as shown in Figure 8. It is recommended that the sputtered depth is sufficiently greater than the surface roughness for determining the original surface level and the sputtered depth. The sputtered depth is obtained from the mean line in the sputtered region, including sputter-induced roughness, for both the large analysed area [see 5.1.4 a)] and for the spot analysis [see 5.1.4 b)]. Positions measured with a stylus profilometer should be in the flat region and not shadowed by a mesh bar or affected by deposited sputtered material. The shadowing effect is considered in Annex A.

NOTE In order to obtain the mean or reference line, a method using the filtering process of the primary stylus profile may be used as described in Reference [10] or a least-square fitting method as described in Reference [11].



a) 2 000 µm trace [a,b,c]



b) Higher-magnification trace [d]

Key

X distance (µm)

Y depth (nm)

1 grid mesh bar positions

2 foil position

[a] The non-sputtered areas at both edges of the grid mesh shadowed by the aluminium foil and the non-uniformity of the crater are clear.

[b] If the analysis area covers several mesh openings, the average rate is determined as described in 5.3.

[c] Points 1 and 2 denote the ion-sputtering-shadowed areas due to the mesh bars and the aluminium foil, respectively.

[d] The sputtered depth for the analysed area is determined by averaging the depth at both sides of the mesh opening.

Figure 8 — Examples of stylus profilometer traces after sputtering

Micro-trenching structure(s) at the edge(s) of each sputtered crater may be observed within the mesh hole. These may be formed by specular re-emission of ions from sidewalls of the mesh that cause extra local sputtering of the specimen [12]. Examples of micro-trenches are shown in Figure B.1 in Annex B, at the right-hand edge of sputtered crater bases for the 75 and 100 mesh grids. When detecting a micro-trenching structure, the sputtered depth needs to be measured for the flat base region excluding the trench region.

5.3 Estimation of sputtering rate

The sputtering time t and sputtered depth d are obtained from 5.1.4 and 5.2, respectively. The sputtering rate R is calculated using Equation (1).

The sputtering rate is a function of sputtering conditions such as ion species, ion energy, scanning area, ion current, and so on, as described in ISO 14606 [13] and ISO/TR 15969 [14]. R is depth/time, and is usually expressed in nm/minute.

The sputtering rate may be used to determine the sputtering yield for the chosen conditions of the angle of incidence of the ion beam, the ion beam energy, the ion beam current density, and so on. However, it is often more convenient to work with relative sputtering rates, and so the concept of the relative sputtering rate is introduced instead of the absolute sputtering rate. The relative sputtering rate R_{rel} is expressed by the ratio of the rates R_1 for material 1 of interest and R_{ref} for the reference material as given in Equation (2):

$$R_{\text{rel}} = R_1/R_{\text{ref}} \tag{2}$$

The relative sputtering rate is useful in practice [5], [15] but requires the sputtering conditions to be the same for the analysed specimen and the reference specimen. This is because this value is applicable even when the sputtering conditions, for example ion current, ion density, and so on, are not precisely known but can be maintained unchanged.

When the analysis area covers several mesh openings, the relative sputtering rate R_{rel} should be determined as the average value for the same area as that of the analysis area. In this case, the sputtering rate R_1 for the material of interest, and the reference sputtering rate R_{ref}, should be measured for the several mesh openings corresponding to the position in the mesh of the analysis area. The values of R_1 and R_{ref} in Equation (2) are replaced by these averaged sputtering-rate values. If the sputtering rate varies significantly over these several mesh openings, the specimen will not be measured uniformly in depth.

6 Summary of round-robin results

In an interlaboratory study [16], 10 laboratories analysed this method using 75 mesh grids with argon and xenon ions in the energy range 1 keV to 3 keV and angles of incidence between 30° and 57°. These conformed to the geometry of Annex A. The 10 sets of results showed that the mesh increased the sputtering rate by between 0,937 and 1,020 times, with an average increase of 0,987 and a standard deviation of 0,025. These results were interpreted to show no significant change in sputtering rate arising from the presence of the mesh. These scatters illustrate the typical measurement repeatabilities of the laboratories involved.

For grids larger than 75 mesh, earlier interlaboratory studies had revealed significant transfer of the grid material (copper) during the sputtering process. The mechanism is not fully understood but it could arise from sputter re-deposition from the grid material.

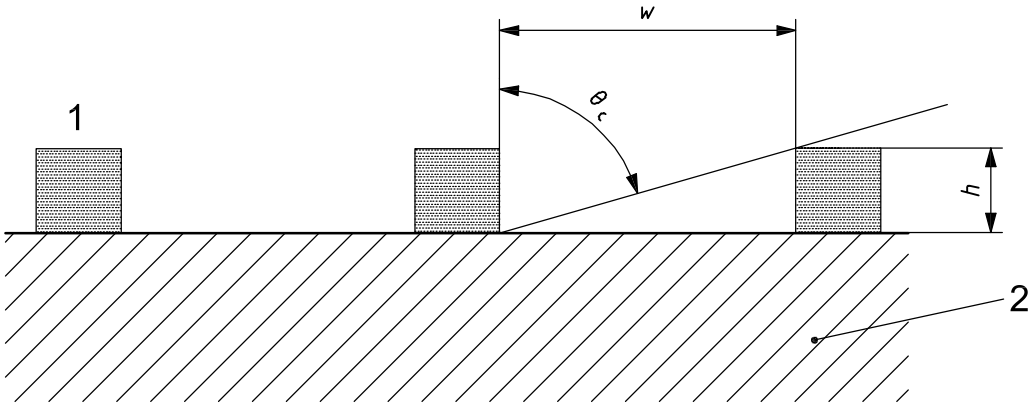
For grids smaller than 75 mesh, the grid opening is too large compared to the ion beam sputter uniformity within the mesh opening.

For these reasons, a 75 mesh grid is recommended.

Annex A
(informative)

Geometry of specimen surface and ion gun

In the mesh-replica method, the experimental geometry, such as the angle of the ion beam at the surface, is very important. When analysing a surface covered by a mesh grid with a scanning Auger microprobe, it is important to consider the respective angles of the specimen surface with the ion and electron beams. This is because an unsuitable geometry causes shadows on the specimen surface from the ion beam and/or the electron beam. This situation may also affect the amount of sputter deposition on the specimen surface from the grid material.



Key

1 cross-section of mesh grid
2 specimen
w mesh opening width
h mesh thickness (mesh height)
θ_c perspective angle from the bottom of a grid bar to the top of a neighbouring grid bar

Figure A.1 — Cross-sectional diagram of a mesh grid and a specimen surface for case 1

Assuming that the cross-sectional shape of the mesh grid is rectangular and that the specimen surface is flat, the wall of the grid makes a right angle with the surface as shown in Figure A.1. Here, the maximum bombardment angle θ_c is simply expressed by w and h as given by Equation (A.1):

$$\theta_c = \tan^{-1}\left(\frac{w}{h+l}\right) \quad \text{(A.1)}$$

where

l is the extent of any lift of the grid from the specimen surface [2] and should be kept as small as possible;

θ_c is defined as the maximum angle measured from the surface normal [2].

NOTE The parameter l is not shown in Figure A.1 but simply represents a space, generally less than h, between the grid bar and the specimen surface.

In order to avoid the shadowing effect caused by mesh grids, the ion beam angle of incidence θ needs to be set in the range 0 to θ_c. The same geometric conditions need to be adopted in the actual specimen analysis.

This is valid for case 1 where the primary excitation source (electron beam for AES, X-ray for XPS, and so on) and the ion beam source are within the angular range 0 to θ_c in the same azimuth. In the second case, case 2, where the primary excitation source is located on the opposite side of the ion beam source in respect of the surface normal, the incident angles of the primary beam and ion beam need to be less than the maximum angle θ'_c.

$$\theta'_c = \tan^{-1}\left(\frac{w/2}{h+l}\right) \tag{A.2}$$

This is shown in Figure A.2. Equation (A.2) is obtained assuming that the analysed point is symmetrically located at the central position in a mesh opening. It is recommended that the analyst estimate the acceptable angle of incidence range, considering the instrumental geometry and the shape and pitch of the mesh-grid used.

Table A.1 — Example list of parameters w and h which were used in the interlaboratory study [2] to define the angles θ_c and θ'_c

Mesh pitch	Mesh opening with	Mesh thickness [b]	Angle of incidence of ion beam [a]	
			Case 1	Case 2
	w μm	h μm	θ_c degrees	θ'_c degrees
50	450	29	85,0 (86,3)	80,2 (82,7)
75	283	29	82,2 (84,1)	74,6 (78,5)
100	200	29	79,0 (81,7)	68,7 (73,9)
150	117	30	71,1 (75,6)	55,6 (62,9)
200	85	31	64,2 (70,0)	46,0(53,9)
300	45	19	57,2 (67,1)	37,8 (49,8)
400	30	16	49,1 (61,9)	30,0 (43,2)

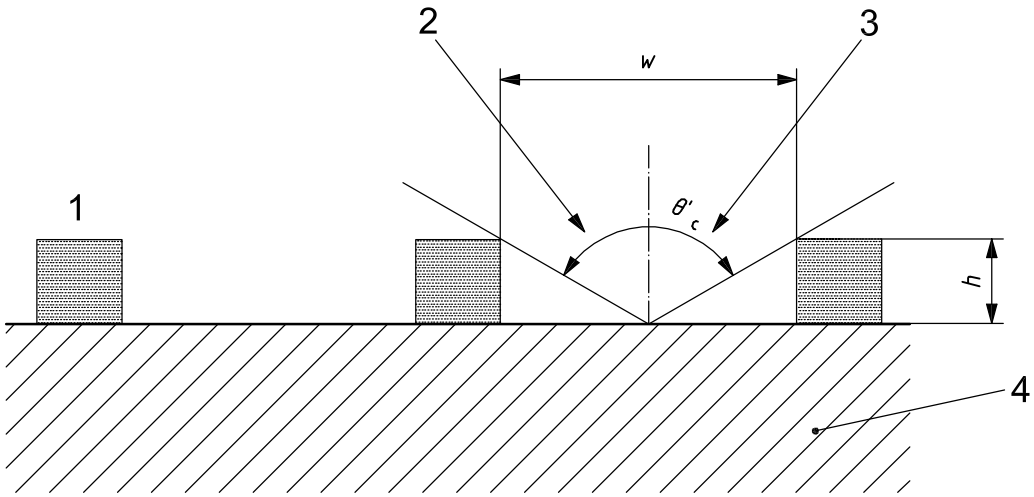
[a] The effect of grid lift-off is now included, using a value of 10 μm for the parameter l. The values in brackets show the original values.

[b] The mesh thickness h was measured using a micrometer.

Table A.1 shows the parameters w and h in Equations (A.1) and (A.2) as examples, which were used in the interlaboratory study [16]. A typical value of the lift-off l of the mesh from the specimen surface using method a) in 5.1.3.1 was found to be 10 μm, and this is used as an example in Table A.1. With methods b) and c), the lift-off may be significantly reduced, and then the values in brackets are applicable. When both axes of the ion and excitation beams are in the same azimuth, they need to be less than θ_c. For example, when using a 200-mesh, they are set at the angle lower than 64°.

When the axes of the ion and excitation beams are in the opposite azimuths, each incident of angle needs to be less than θ'_c. For example, when using a 200-mesh, they are set at the angle lower than 46°.

One may need to select the geometry where the angle of incidence of the ion beam is greater than θ_c or θ'_c in Table A.1. When surfaces are sputtered without rotation, it is recommended that the sputtered depth be measured with a stylus profilometer at a position between the point P_1 and the point P_2. Here, P_1 is the point at least $2h$ from the mesh grid bar in the azimuth away from the direction of the ion beam, and P_2 is the point at a distance $h\tan\theta$ from the opposing grid bar, where θ is the angle of incidence of the ion beam. When rotating the specimen during ion sputtering, it is recommended that the sputtered depth be measured at the centre of a mesh opening.



Key

1 cross-section of mesh grid
2 electrons or X-rays
3 ion beam
4 specimen

NOTE The axes of the excitation source and ion beam are in the opposite angles of view.

Figure A.2 — Cross-sectional diagram of a mesh grid and a specimen surface for case 2

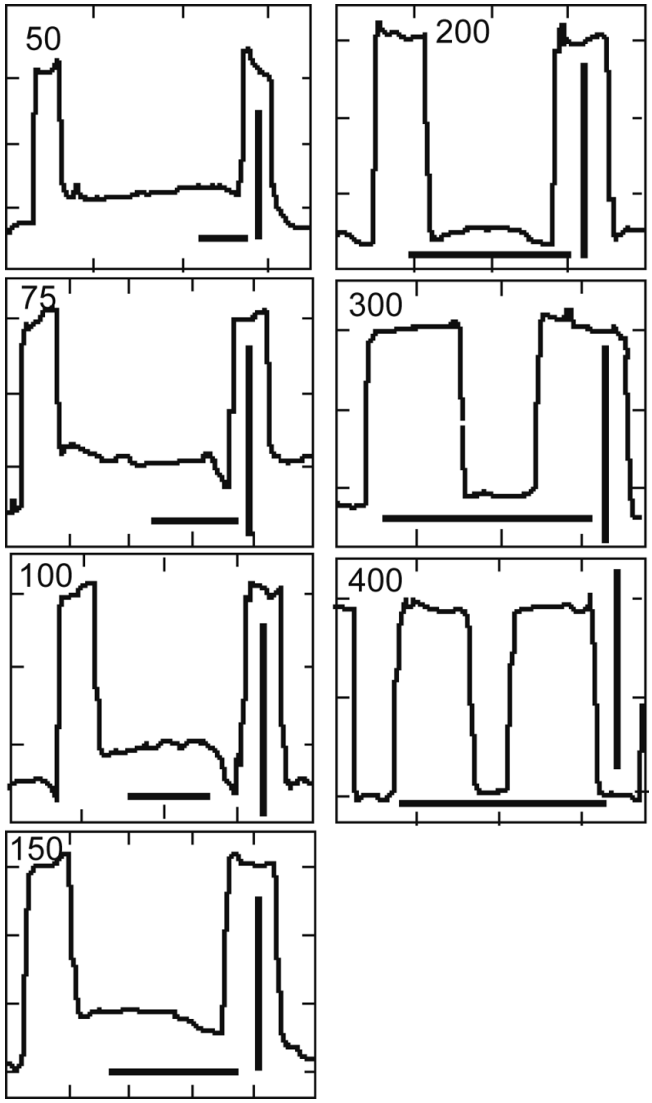
Note that Figures A.1 and A.2 and Table A.1 are made assuming an ideal situation. The cross-sectional shape of a mesh grid is in fact not rectangular, and the specimen surface is not always flat. In these cases, the values of θ_c and θ'_c are different from those in Table A.1. It is necessary to take into account the shape of mesh grid used and/or the overall tilt angle of the mesh grid on the specimen surface, especially when adopting a significantly high angle of incidence [2].

Annex B
(informative)

Dependance of replica patterns on mesh-opening size

An interlaboratory study was performed to examine an applicable limitation of mesh size (see Reference [16]). Here, as in the general system used industrially, the meshes are identified by the mesh number, i.e. the number of bars per inch. The mesh numbers used in this test were 50, 75, 100, 150, 200, 300, and 400, the meshes being mounted on bare silicon wafers. The examples of sputtered replica patterns measured by a stylus profilometer are shown in Figure B.1. All of the meshes were found applicable to the mesh-replica method to estimate the ion-sputtering rates. Though there are small amounts of sputter-deposited mesh material on the specimen surfaces for the 150 to 400 mesh, it does not seem to affect the sputtering rate of the specimen.

The replica patterns in Figure B.1 were obtained by ion sputtering without specimen rotation. Figure B.2 shows patterns with specimen rotation, reported in the interlaboratory study. The ion beam angle of incidence [1] was higher than 50°, and the ion energy was 3 keV. For the 50 mesh grid, the replica pattern is almost the same as that measured in the experiment without specimen rotation as shown in Figure B.1. On the other hand, for the 75 to 200 mesh grids, the side-wall shape is flask-like, which was due to the shadowing effects of the mesh bars. In these cross-sectional profiles, there are flat regions between the flask-like side-walls. In case a) in 5.1.4, it is possible to estimate the sputtering rate using these flat regions. For the 300 mesh grid, the replica pattern cannot be observed. This may be because the aspect ratio (mesh thickness/mesh-opening width) is too high to apply the mesh-replica method when rotating specimens.



NOTE 1 The vertical scale bars correspond to a distance of 100 nm, the horizontal scale bars to a distance of 100 μm.

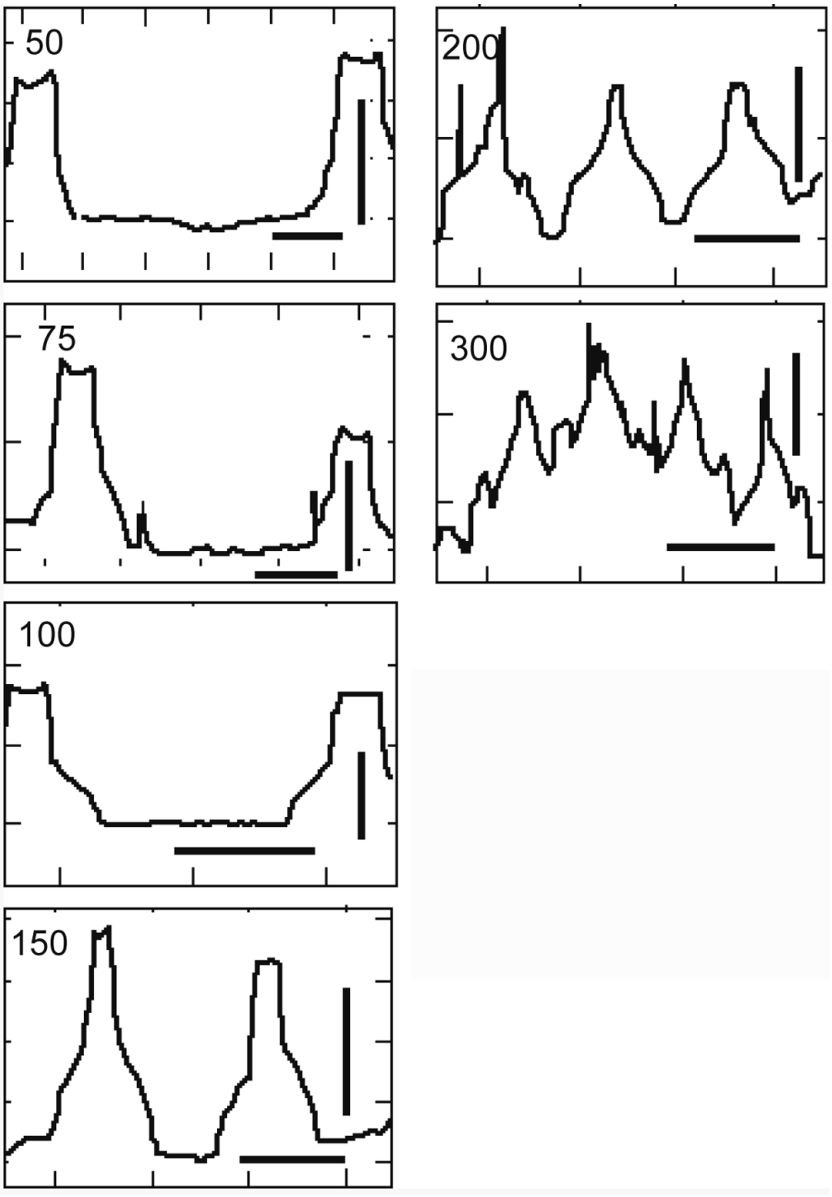
NOTE 2 The cross-sectional profiles of some of the mesh-replica patterns were measured using a stylus profilometer.

NOTE 3 The profiles cover one pitch from the bar to the neighbouring bar for 50 to 300 meshes, and two pitches for 400 mesh.

NOTE 4 In this example, the incident ion beam was at an angle of 23° from the normal to the surface and came from the left of these diagrams.

NOTE 5 The sputtering time was 20 minutes for all the meshes and the sputtering rate was measured to be about 4,5 nm/min.

Figure B.1 — Examples of mesh-replica patterns depending on the mesh-opening size [16]



NOTE 1 The vertical scale bars correspond to a distance of 100 nm for 50 to 200 meshes and 20 nm for 300 mesh.

NOTE 2 The horizontal scale bars correspond to a distance of 100 μm.

NOTE 3 The specimen surfaces were sputtered with specimen rotation.

NOTE 4 The specific feature is a “flask-type” side-wall formed by the ion-beam-shadowing effect caused by the mesh bar [2].

Figure B.2 — Examples of mesh-replica patterns reported in the interlaboratory study

Bibliography

[1] ISO 18115:2001, *Surface chemical analysis — Vocabulary*

[2] SEAH, M.P., GELLER, J. and SUZUKI, M., Accurate Measurement of Sputtered Depth for Ion Sputtering Rates and Yields: The Mesh Replica Method, *Surface and Interface Analysis,* Vol. 39, No. 1, pp. 69-78 (2007)

[3] ZALAR, A., *Thin Solid Films*, 124, 223 (1985)

[4] VEISFELD, N. and GELLER, J.D., *Ion Sputtering Yield Measurements for Submicrometer Thin Films*, J. Vac. Sci. & Tech., 6, 2077 (1988)

[5] SUZUKI, M., MOGI, K. and ANDO, H., *Technical Proposal for Measurement of Sputtered Depth Using a Mesh — Especially for Auger Depth profiling*, J. Surface Anal., 5, 188 (1999)

[6] ISO 5436-1:2000, *Geometrical Product Specifications (GPS) — Surface texture: Profile method; Measurement standards — Part 1: Material measures*

[7] ASME B46.1-1995, *Surface texture (Surface Roughness, Waviness, and Lay)*

[8] ISO 12179:2000, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Calibration of contact (stylus) instruments*

[9] VORBURGER, T.V., DAGATA, J.A., WILKENING, G. and IIZUKA, K., Chapter 4: "Characterization of Surface Topography" in *Methods of Surface Characterization*, Vol. 5, Beam Effects, Surface Topography, and Depth Profiling in Surface Analysis, edited by A. W. Czanderna, T. E. Madey, and C. J. Powell, Prenum Press (1998)

[10] ISO 13565-1:1996, *Geometrical Product Specifications (GPS) — Surface texture: Profile method; Surfaces having stratified functional properties — Part 1: Filtering and overall measuring conditions*

[11] ISO 13565-3:1998, *Geometrical Product Specifications (GPS) — Surface texture: Profile method; Surfaces having stratified functional properties — Part 3: Height characterization using the material probability curve*

[12] CALE, T.S., MAHADEV, V., TANG, Z., RAJAGOPALAN, G. and BORUCKI, L.J., *Topography Evolution During Semiconductor Processing*, in Plasma Processing of Semiconductors, edited by P. F. Williams, NATO ASI Series E: Applied Sciences Vol. 336, Kluwer Academic Publishers (1997)

[13] ISO 14606:2000, *Surface chemical analysis — Sputter depth profiling — Optimization using layered systems as reference materials*

[14] ISO/TR 15969:2001, *Surface chemical analysis — Depth profiling — Measurement of sputtered depth*

[15] INOUE, M. and SERD project group of SASJ (Surface Analysis Society of Japan), *Sputter Etching Rate Database (SERD)*, Project of SASJ, J. Korean Vac. Soc., 9, 103 (2000)

[16] SUZUKI, M., MOGI, K. and OGIWARA, T., *Investigation of Mesh Opening Size in Mesh-Replica Method toward Standardization of Depth Profiling Technique*, J. Surface Anal., 10, 144 (2003)

blank

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001. Email: orders@bsi-global.com. Standards are also available from the BSI website at http://www.bsi-global.com.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: +44 (0)20 8996 7111. Fax: +44 (0)20 8996 7048. Email: info@bsi-global.com.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002. Fax: +44 (0)20 8996 7001. Email: membership@bsi-global.com.

Information regarding online access to British Standards via British Standards Online can be found at http://www.bsi-global.com/bsonline.

Further information about BSI is available on the BSI website at http://www.bsi-global.com.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL