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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

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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is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.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 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.

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):

$$R = d/t \quad (1)$$

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

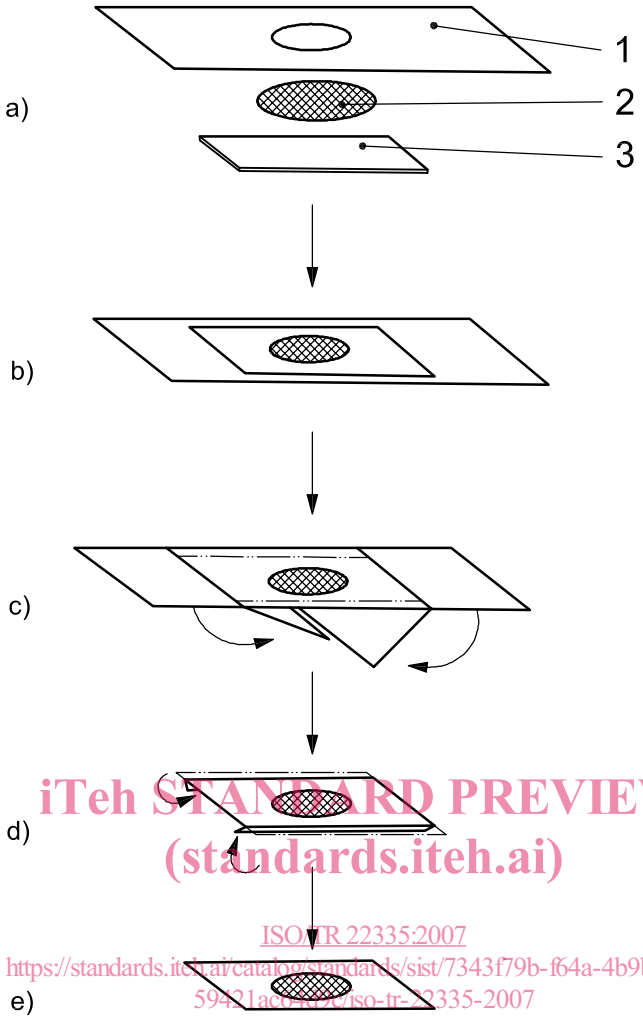
ion-sputtering rate uniformity within the grid mesh opening will be a significant factor for the repeatability of the measurements. The profilometer trace will reveal the shape of the sputter crater.

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.



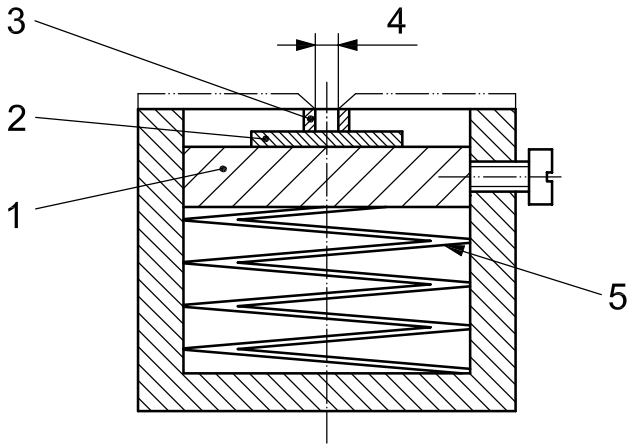
Key

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.

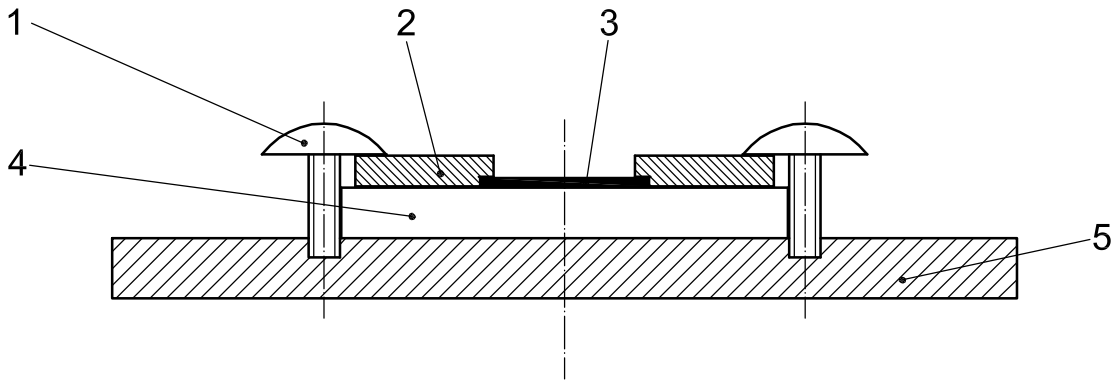


Key

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