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Standard Guide for Performing Sputter Crater Depth Measurements¹

This standard is issued under the fixed designation E 1634; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ This guide is under the jurisdiction of ASTM Committee E-42 on Surface Analysis and is the direct responsibility of Subcommittee E42.068 on STMS—Ion Beam Sputtering.

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1. Scope

1.1 This guide covers the preferred procedure for acquiring and post-processing of sputter crater depth measurements. This guide is limited to stylus-type surface profilometers equipped with a stage, stylus, associated scan and sensing electronics, video system for sample and scan alignment, and computerized system.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

E 673 Terminology Relating to Surface Analysis²

E 1162 Practice for Reporting Sputter Depth Profile Data in Secondary Ion Mass Spectrometry²

3. Terminology

3.1 Definitions:

3.1.1 Terms used in surface analysis are defined in Terminology E 673.

4. Significance and Use

4.1 Sputter crater depth measurements are performed in order to determine a sputter rate (depth/time) for each matrix sputtered during a sputter depth profile or similar in-depth type analyses. From sputter rate values, a linear depth scale can be calculated and displayed for the sputter depth profile.

4.2 Data obtained from surface profilometry are useful in monitoring instrumental parameters (for example, raster size, shape, and any irregularities in topography of the sputtered crater) used for depth profiles.

5. General Procedure

5.1 Upon completing a sputter depth profile, mark the crater for future identification (one can mark the exterior corner(s) of a crater with features, for example, lines, holes, etc., produced using an unrastered ion beam). Note the x - and y -position with respect to the rastered ion beam and sample geometry or suitable device feature(s).

5.2 Place the sample on the profilometer stage surface. If sample has an area of less than 1 cm², mount the sample onto another larger flat surface to prevent sample movement when profilometry is performed. The system should be reasonably leveled; for details on instrumental adjustments, see manufacturer's operational manual(s). Keep the environment as dust-free as possible and dust-off the sample surface with clean air/gas jet before performing the measurement.

5.3 Pre-select surface profilometer operational settings; computerized models are commonly used. Most surface profilometers commonly permit selection of the following parameters:

5.3.1 Stylus type (for example, diamond stylus),

5.3.2 Stylus radius (for example, 5 μm ; various stylus radii are available depending upon desired resolution of measurement, and to a certain degree the strength of the stylus tip for varying hardness of materials),

5.3.3 Stylus force (that is, force exerted on the analytical sample during operation, for example, 15 mg; this is an important variable when profiling sample with high hardness levels; damage to the stylus may occur, and hence damage to the instrumentation or errors in profilometry measurements, or both, may result),

² Annual Book of ASTM Standards, Vol 03.06.

5.3.4 Scan speed (for example, 50 $\mu\text{m/s}$; this value is dependent upon permissible noise levels, accuracy, etc., and is typically determined experimentally),

5.3.5 Scan length (one typically uses twice the crater size to allow for scanning over the level areas about the sputtered crater, and

5.3.6 Number of scans for signal averaging (for example, three repetitive scans averaged to improve the signal-to-noise ratio).

5.4 Lower the stylus in an area outside the sputtered crater, at a distance from the crater edge of approximately one-half the actual crater size, and in a reasonably smooth area to traverse the entire crater length. The scan path is typically chosen across the center of the sputtered crater in one direction with a repetitive measurement in the perpendicular direction, noting the symmetry with respect to the previously marked crater directions (see 5.1).

6. Interpretation of Results

6.1 In general, a plot representative of a sputtered crater will result (see Fig. 1). The data may then need post-processing, including leveling, rescaling, zeroing of surface depth, averaging top and bottom surface(s), etc. In the leveling process, one normally chooses a cursor position on the top left and right (outer surface of the crater). Upon leveling, rescaling, and zeroing, the full crater shape should be visible on the graph, with both top surfaces at the same zero point and the bottom surface of the crater relatively flat. Some systems permit cursor location at several points on the top and bottom surfaces of the crater. The computer would then average and calculate the differences to determine an accurate sputter crater depth measurement.

7. Modified Procedure for Large-Area Craters

7.1 For larger diameter ion beam sputter craters, such as are frequently used for Auger electron spectroscopy and x-ray photoelectron spectroscopy, it may be too difficult to define the original surface height and crater depth from the profilometer scan. In these cases, sputter crater depths have been successfully measured by sputtering the specimen through a metal mesh, such as a 3-mm TEM grid with 500 μm openings, then using the stylus profilometer to measure the heights of the replicated mesh pattern on the sputtered specimen surface (1). An example is shown in Fig. 2.

7.2 It is important for an accurate step-height measure of the original surface and the crater depth that the mesh grid be positioned directly onto the specimen surface, with no gap separating the two. Additionally, the mesh material must be selected so that sputtered contamination from the mesh does not interfere with signal from the elements of interest in the specimen material. This sputtered contamination from the mesh will also modestly alter the sputter rate of the specimen material, which may be a concern in some experiments.

8. Precision and Accuracy

8.1 Precision—The precision is determined by repeating measurements several times and reporting the standard deviation between values.

8.2 Accuracy—The accuracy of the measurement can be determined by measuring a calibrated depth standard typically supplied with commercial surface profilometers, and calculating a percent difference from the measured value. Bias often depends upon stylus limited point size, scan speeds/distances, vibration during measurement(s), condition of apparatus; calibration of surface profilometer equipment, etc., and should be considered carefully when measuring sputtered crater depth and reporting subsequent data.

89. Keywords

89.1 Auger electron spectroscopy; secondary ion mass spectrometry; stylus profilometry; surface analysis; x-ray photoelectron spectroscopy

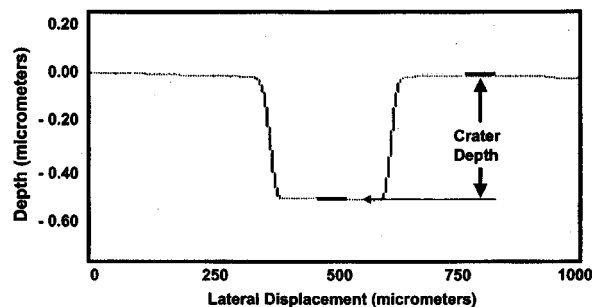


FIG. 1 A Typical Stylus Profilometer Scan of a Sputtered Crater

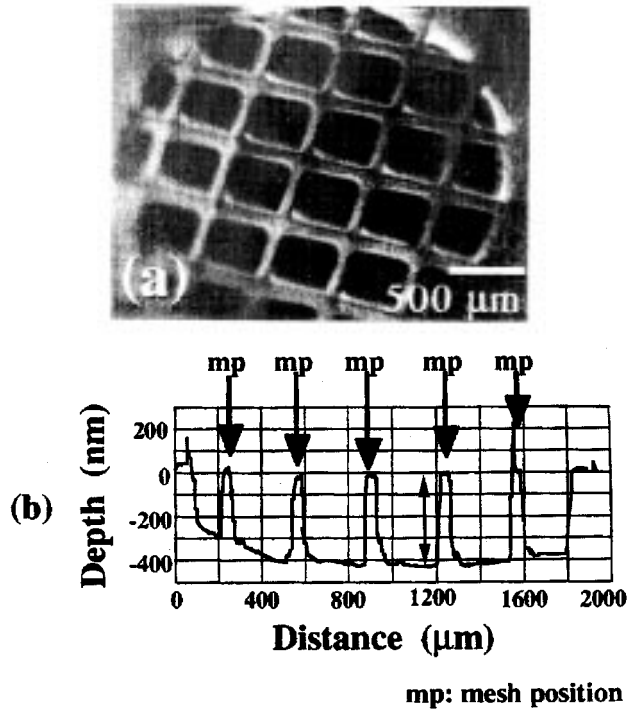


FIG. 2 (a) Scanning electron photomicrograph showing replication of mesh grid pattern on a specimen after sputtering. (b) Stylus profilometry scan across replicated grid pattern to measure sputtercrater depth. Crater depth is marked by double-headed arrow. From (1).

REFERENCES

- (1) Suzuki, M., Mogi, K., and Ando, H., "Technical Proposal for measurement of Sputtered Depth Using a Mesh—Especially for Auger Depth Profiling," *J. Surf. Anal.*, Vol. 5, 1999, pp. 188 – 191.

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