Designation: E1438 – 11

Standard Guide for Measuring Widths of Interfaces in Sputter Depth Profiling Using SIMS[1]

This standard is issued under the fixed designation E1438; 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. Scope

1.1 This guide provides the SIMS analyst with a method for determining the width of interfaces from SIMS sputtering data obtained from analyses of layered specimens (both organic and inorganic). This guide does not apply to data obtained from analyses of specimens with thin markers or specimens without interfaces such as ion-implanted specimens.

1.2 This guide does not describe methods for the optimization of interface width or the optimization of depth resolution.

1.3 *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:*[2]

E673 Terminology Relating to Surface Analysis (Withdrawn 2012)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 See Terminology E673 for definitions of terms used in SIMS.

4. Summary of Guide

4.1 This guide will allow interface widths to be calculated from plots of SIMS secondary ion intensity versus time that are acquired during sputtering of layered specimens. It assumes that a primary ion beam with a stable current density is being used. Briefly, these plots are obtained in the following fashion: an ion beam of a particular ion species, ion energy, and angle of incidence is used to bombard a sample. The beam is rastered or defocused so as to attempt to produce uniform current density in the analyzed area, that is defined by means of mechanical or electronic gating. The intensity of one or more secondary ions is monitored with respect to time as sputtering continues.

4.2 The interface width is then determined from the secondary ion intensity versus time data according to an arithmetic model described in the Procedure section. A measurement of the thickness of the layer overlying the interface is required. This measurement may be performed by another analytical technique.

5. Significance and Use

5.1 Although it would be desirable to measure the extent of profile distortion in any unknown sample by using a standard sample and this guide, measurements of interface width (profile distortion) can be unique to every sample composition (**1, 2**).[4] This guide, describes a method that determines the unique width of a particular interface for the chosen set of operating conditions. It is intended to provide a method for checking on proper or consistent, or both, instrument performance. Periodic analysis of the same sample followed by a measurement of the interface width, in accordance with this guide, will provide these checks.

5.2 The procedure described in this guide is adaptable to any layered sample with an interface between layers in which a nominated element is present in one layer and absent from the other. It has been shown that for SIMS in particular (**3, 4**) and for surface analysis in general (**5, 6**), only rigorous calibration methods can determine accurate interface widths. Such procedures are prohibitively time-consuming. Therefore the interface width measurement obtained using the procedure described in this guide may contain significant systematic error (**7**). Therefore, this measure of interface width may have no relation to similar measures made with other methods. However, this does not diminish its use as a check on proper or consistent instrument performance, or both.

[1] This guide is under the jurisdiction of ASTM Committee E42 on Surface Analysis and is the direct responsibility of Subcommittee E42.06 on SIMS.

Current edition approved Nov. 1, 2011. Published December 2011. Originally approved in 1991. Last previous edition approved in 2006 as E1438 – 06. DOI: 10.1520/E1438-11.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] The boldface numbers given in parentheses refer to a list of references at the end of this guide.

5.3 This guide can be used for both elemental and molecular depth profiles, provided that the materials have constant sputter rates throughout the depth of the overlayer, and minimal interlayer mixing is occurring. For more detailed information regarding measurements of interface widths during organic depth profiling, please see Mahoney (**8**).

6. Apparatus

6.1 The procedure described in this guide can be used to determine an interface width from data obtained with virtually any SIMS instrument.

6.2 Use of the interface width measurement from a layered specimen as a check on proper or consistent instrument performance, or both, does not assume that the sample of interest contains no interface roughness. Rather, it assumes that the interface roughness is consistent from one analyzed area to the next. Any layered sample that meets this criterion is suitable for the use intended by this guide.

7. Procedure

7.1 This procedure for measurement of depth resolution is based on the amount of time required for the signal of one of the major elements of the layer overlying the interface to be reduced from 84 to 16 % of its average intensity in the overlying layer. The ratio of this interface sputtering time to that of the sputtering time of the overlayer is equated to the ratio of their respective thicknesses in order to obtain the measurement of interface thickness. This is expressed in the form of the following equation (refer to Fig. 1):

$$\Delta z = (\Delta t/t)z \quad (1)$$

where:

Δz = interface width,
Δt = sputtering time for decrease from 84 to 16 % of the signal intensity,
t = time required to sputter through overlayer, from $t = 0$ until t is equal to the time at which the major element reaches 50 % of its value in the overlayer, and
z = thickness of overlayer.

7.1.1 The measurement of sputter time (t) for the interfacial region and for the overlying layer thickness should be acquired graphically from the plot of secondary ion signal versus time.

7.1.2 The thickness of the overlayer (z) is commonly obtained from a post-profile crater measurement and assuming a constant sputter rate. The thicknesses of layers in a multilayered profile are only approximated by this procedure. A more accurate thickness value for the overlayer is obtained by halting the sputtering at the interface, and obtaining a crater measurement. Alternatively, other techniques may be used to measure the overlying layer. Some possible techniques that may be used include optical interferometry, ellipsometry, selective chemical etching followed by profilometry, and spectroscopic methods such as Rutherford Backscattering Spectrometry (RBS), X-ray Fluorescence (XRF), and Angle Resolved X-ray Photoelectron Spectroscopy (ARXPS).

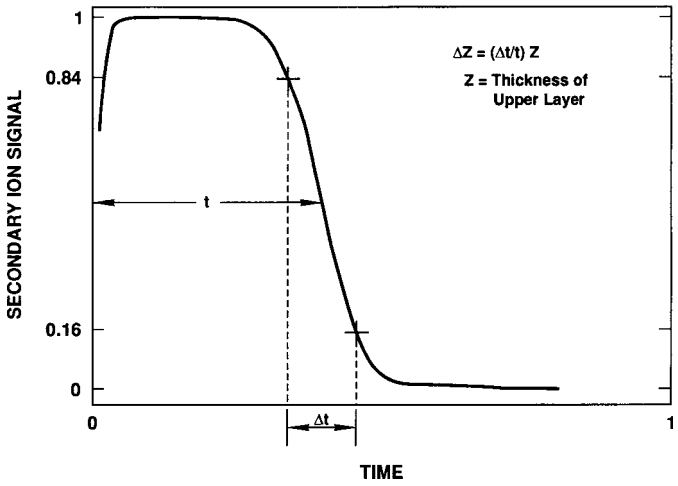

FIG. 1 Measurement of Interface Width, Δz, from a Plot of SIMS Intensity Versus Ion Bombardment Time

7.1.3 Additional considerations for molecular depth profiling: (*1*) interfaces of molecular samples on silicon (Si) substrates are ill-defined due to the large differences in sputter properties between organic/inorganic interfaces. These differences can cause primary ion recoil and Si rebounding. This can cause increased damage or drastic changes, or both, in sputter rates of the organic material at the interface. Therefore, it is impossible to determine accurate sputter rates in this region, unless they are measured directly. It is recommended that any discussion of interface widths in such systems should be for relative comparisons only. (*2*) For organic samples, one may use either the polymer signal decay or the rise in the substrate signal to describe the interface width, depending on the particular system, experimental conditions, chemistries and artifacts observed for your selected organic system. For more information, see Mahoney (**8**).

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