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TECHNICAL REPORT

colour inside

Nanotechnologies – A guideline for ellipsometry application to evaluate the thickness of nanoscale films

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NANOTECHNOLOGIES – A GUIDELINE FOR ELLIPSOMETRY APPLICATION TO EVALUATE THE THICKNESS OF NANOSCALE FILMS

FOREWORD

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IEC TR 63258, which is a Technical Report, has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems, in collaboration with ISO technical committee 229: Nanotechnologies.

It is published as a double logo document.

The text of this Technical Report is based on the following documents:

DTR	Report on voting
113/548/DTR	113/563/RVDTR

Full information on the voting for the approval of this Technical Report can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

Ellipsometry is a powerful optical technique to evaluate the dielectric properties of thin films. Ellipsometry can be used to characterize thickness, roughness, composition, crystalline nature, and other properties of nanomaterials, and is frequently used to warrant the quality and the performance of thin-film growth equipment. The signal depends on the change in the optical response of incident light that interacts with the nanomaterial being investigated.

Many current and emerging electrotechnical devices employ nanomaterials in the form of thin films. Therefore, it is important to develop a measurement protocol to evaluate the thickness of such films with sufficient accuracy. This document describes the practical considerations that need to be taken into account in using ellipsometry to evaluate the thickness of nanoscale films.

NANOTECHNOLOGIES – A GUIDELINE FOR ELLIPSOMETRY APPLICATION TO EVALUATE THE THICKNESS OF NANOSCALE FILMS

1 Scope

This document, which is a Technical Report, is focused on the practical protocol of ellipsometry to evaluate the thickness of nanoscale films. This document does not include any specification of the ellipsometers, but suggests how to minimize the data variation to improve data reproducibility.

This document includes

– outlines of the ellipsometry procedures,

– methods of interpretation of results and discussion of data analysis, and

– case studies.

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.

ISO/TS 80004-1, *Nanotechnologies –Vocabulary – Part 1: Core terms*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1 and the following apply.

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

- ISO Online browsing platform: available at http://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1 General terms

3.1.1
interlaboratory comparison
organization, performance and evaluation of measurements or tests on the same or similar items by two or more laboratories in accordance with predetermined conditions

[SOURCE: ISO/IEC 17043:2010, 3.4]

3.2 Terms specific to this document

3.2.1
polarization
direction of the electric field vector of an optical beam

Note 1 to entry: The plane of polarization is the plane containing the electric field vector and the direction of propagation of the beam.

[SOURCE: ISO/IEC 30193:2020, 3.28]

3.2.2
optical constant
refractive index $n(\lambda)$ and extinction coefficient $k(\lambda)$, as functions of wavelength λ

3.2.3
refractive index
n
ratio of the speed of electromagnetic wave in vacuum c to that in another medium v

$$n = \frac{c}{v}$$

Note 1 to entry: The refractive index shows how the speed of light is changing depending on media.

3.2.4
complex refractive index
N
index that determines the propagation of a plane electromagnetic wave in an isotropic absorbing medium expressed as

$$N(\lambda) = n(\lambda) + ik(\lambda)$$

where n and k are the real and imaginary parts, respectively

Note 1 to entry: The real n and imaginary k parts are called the refractive index and the extinction coefficient, respectively.

Note 2 to entry: Optics field convention is used for the definition of complex refractive index [1].

3.2.5
absorption coefficient
α
coefficient that describes the attenuation of electromagnetic wave intensity I_0 during propagation in absorbing media

Note 1 to entry: The electromagnetic wave intensity attenuates according to the following equation:

$$I = I_0 \exp(-\alpha x) .$$

where I_0 is the initial electromagnetic wave intensity and x is the propagation distance.

Note 2 to entry: Absorption coefficient α is related to extinction coefficient at a wavelength:

$$\alpha = \frac{4\pi k}{\lambda} .$$

3.2.6
complex dielectric constant
ε
value that indicates how atoms in a material respond when an outside electric field is applied to the material

Note 1 to entry: Complex dielectric constant is given by the equation

$$\varepsilon(\lambda) = \varepsilon_r(\lambda) + i\varepsilon_i(\lambda)$$

where ε_r, ε_i are the real and imaginary parts of complex dielectric function, respectively.

Note 2 to entry: Relationship between the complex dielectric constant and the complex refractive index obtained from Maxwell's equation is:

$$\varepsilon(\lambda) = N(\lambda)^2.$$

Note 3 to entry: Optics field convention is used for the definition of the complex dielectric constant [1].

Note 4 to entry: The terms complex dielectric function and dielectric function are used for the complex dielectric constant and dielectric constant when focusing on their wavelength or angular frequency dependence.

3.2.7
film thickness
d
distance between the top and bottom boundaries of the laminar film, where each boundary is determined as the interface at which the refractive index changes

3.2.8
Brewster's angle
angle of incidence at which there is no reflection of p-polarized light at an uncoated optical surface

4 Measurement of ellipsometry

4.1 General

The practical protocol of ellipsometry is well-established.

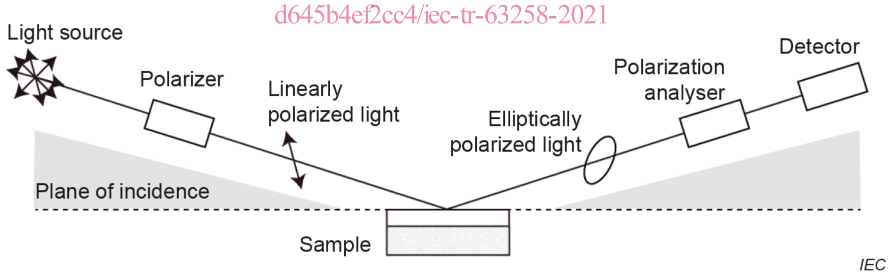

Figure 1 – Primary structure of ellipsometry measurement

Ellipsometry measures a change in polarization as light reflects from a sample. The polarization change is represented as an amplitude ratio, Ψ, and the phase difference, Δ. The basic components of the ellipsometry measurement are a light source, a polarizer, a polarization analyser and a detector, as shown in Figure 1. See references [2][1] and [3] for principles of ellipsometry.

1 Numbers in square brackets refer to the Bibliography.

4.2 Measurement procedure

4.2.1 Sample preparation for system check

Before the actual sample measurement is performed, it is necessary to check the system's accuracy. To make it possible, a reference sample with known thickness and/or refractive index should be used. The reference samples such as thermally oxidized SiO_2 on Si are available.

Ellipsometry is very sensitive to physical and chemical properties of the thin film material, its surface and the properties at the film–substrate interface.

4.2.2 Experimental procedure for system check

The general protocol of ellipsometry measurement is standardized to evaluate thin films.

– Step 1: Positioning of the reference sample on the stage.
– Step 2: Adjustment of the height and tilt.
– Step 3: Measurement of the reference sample.
– Step 4: Data analysis.
– Step 5: Result of thickness or refractive index should be within 1 % of the guaranteed values.
– Step 6: If the obtained result fulfils the condition of step 5, start to measure the test sample. If not, the system needs additional check.

It is advisable to check the system at the required angle of incidence.

4.2.3 Sample handling

Ellipsometry is very sensitive to physical and chemical properties of the sample's surface, so it is advisable to keep the sample in a clean and dry place after the preparation. Touching and scratching the surface should be avoided, because non-professional cleaning might affect the surface state and therefore change the result.

4.2.4 Experimental procedures

The general protocol of ellipsometry measurement is as follows.

– Step 1: Positioning of the sample on the stage.
– Step 2: Adjustment of the angle of incidence, height and tilt.
– Step 3: Measurement of the sample.
– Step 4: Data analysis.
– Step 5: Validation of analysis result.

NOTE This protocol is valid for non scattering and isotropic sample planes.

In order to minimize the data variation, the following practical recommendations apply.

1) The ellipsometry measurement should be done at an angle of incidence close to the Brewster's angle of the substrate.
2) The ellipsometry measurement should be done over a measurement wavelength range as wide as possible. For example, if there is absorption in the visible range, it needs to be measured including the near-infrared range.
3) The fitting analysis should be performed by changing the initial value of the film thickness and the type of dispersion formula at the time of analysis. The comparison should be done to confirm that equivalent results can be obtained. See Annex A.