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Carbon based films — Determination of optical properties of amorphous carbon films by spectroscopic ellipsometry

Films à base de carbone — Détermination des propriétés optiques des films de carbone amorphe par ellipsométrie spectroscopique

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Foreword

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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document provides a determination method and a classification for optical properties of amorphous carbon films by spectroscopic ellipsometry.

Amorphous carbon films have a structure, containing both sp^2 and sp^3 bonded carbon atoms and in several cases also hydrogen. There are graphite-like, polymer-like, glass-like and diamond-like carbon films. Because of their outstanding mechanical properties, amorphous carbon films are used in various hard coating applications on hard metals, e.g. as protective coatings against wear and corrosion in automotive parts. Biomedical and optical applications on alternative substrate materials such as silicon and glass have become more and more important. Currently, amorphous carbon films are classified into several types with regard to their chemical structures, and each type is selectively used according to its appropriate application. For an easy classification of amorphous carbon films, an optically quantified phase fingerprint with high accuracy is provided as a result of an international interlaboratory comparison.

The optical properties of refractive index *n* and extinction coefficient *k* determined by spectroscopic ellipsometry are the key quantities for the proposed classification of amorphous carbon films. The interlaboratory comparison demonstrated that a classification within the *n*-*k* plane is feasible for all types of amorphous carbon films. This will be beneficial for the identification of the coating type on alternative substrate materials (such as silicon and glass) and additional industrial applications. Spectroscopic ellipsometry as a fast and non-destructive analytical method can also be applied to quality control and development in industrial applications, given that smooth and well-defined substrate materials are used and appropriate modelling is applied.

This document is intended to implement recommended ellipsometric test conditions and the *n*-*k* plane classification scheme of amorphous carbon films on silicon wafers.

This document is useful for the complementary optical property classification and quality control of amorphous carbon films.

As amorphous carbon films show a huge diversity of structure and properties, it is crucial to select the appropriate type of amorphous carbon film to exploit their excellent properties in practical use. Therefore, carbon films are characterized by spectroscopic ellipsometry under reasonable conditions. This enables the classification of amorphous carbon films on silicon wafers within the *n*-*k* plane acting as a process fingerprint.

Carbon based films — Determination of optical properties of amorphous carbon films by spectroscopic ellipsometry

1 Scope

This document specifies spectroscopic ellipsometry for the determination of optical properties (refractive index n and extinction coefficient k) and the optical classification of different types of amorphous carbon films within the n-k plane.

It is applicable to amorphous carbon films deposited by ionized evaporation, sputtering, arc deposition, plasma-assisted chemical vapour deposition, hot filament techniques and others.

It does not apply to carbon films modified with metals or silicon, amorphous carbon films that have a gradient of composition/property in the thickness, paints and varnishes.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
refractive index
n
ratio of the velocity of propagation of electromagnetic radiation in a vacuum to the velocity of propagation of electromagnetic radiation in a medium

3.2
extinction coefficient
k
amount of absorption of electromagnetic radiation in a medium (substance)

4 Test specimen preparation

Amorphous carbon films on various substrates can be tested if the substrates are optically isotropic and optical models are available for them. The recommended test substrate is a Si wafer with a mirror surface. It can be used as substrates of the amorphous carbon films for testing depending on the requirements. Specimens shall be homogeneous amorphous carbon films. The thickness of the amorphous carbon films shall be 0,02 µm to 5 µm.

Any pertinent details of the specimens such as their dimensions, surface finish, material type, composition, microstructure and processing treatments shall be supplied.

5 Apparatus

5.1 Halogen lamp and blue LED for a light source, which shall have a spectral range of more than 450 nm to 950 nm.

5.2 Detector, for the acquisition of ellipsometric measurement data, which shall have a spectral range of more than 450 nm to 950 nm.

5.3 Spectrometer combined with photomultiplier tube (PMT)/charge coupled device (CCD)/ photodiode array (PDA), for detecting data.

5.4 Software, for obtaining optical constants and film thickness from ellipsometric measurement data.

5.5 Stage, for putting a sample.

5.6 Goniometer, to establish the angle of incidence to 70° or to a multi-angle.

5.7 Camera, for observation of the sample surface and positioning of the flat surface.

6 Procedure

6.1 Treatment of specimen before test

Wash the specimens ultrasonically in a high purity organic solvent for 10 min or longer, with the testing surface downward in the case of the disc specimen. Without allowing them to dry, the specimens should be rinsed with high purity organic solvent and then dried for 30 min or longer in an oven set at 120 °C. Organic solvent can be replaced with other solvents or deionized water as long as clean specimen surfaces are produced at the end of the procedure. The specimens shall be stored, in the same atmosphere as that used for the testing apparatus, until required.

6.2 Preparation for test

Place the specimen for measurement on the stand and set it in place to be irradiated with the light. Adjust the stand height to allow the signal to be as strong as possible for detection.

6.3 Testing conditions for test

Recommended test conditions are listed below, but these may be changed to suit the particular needs of the measuring process. All test conditions shall be described in the test report.

a) Wavelength in measurement: 450 nm to 950 nm.

b) Angle of incidence: 70°.

c) Spot size: over 500 µm × 500 µm.

d) Duration of measurement: 5 s.

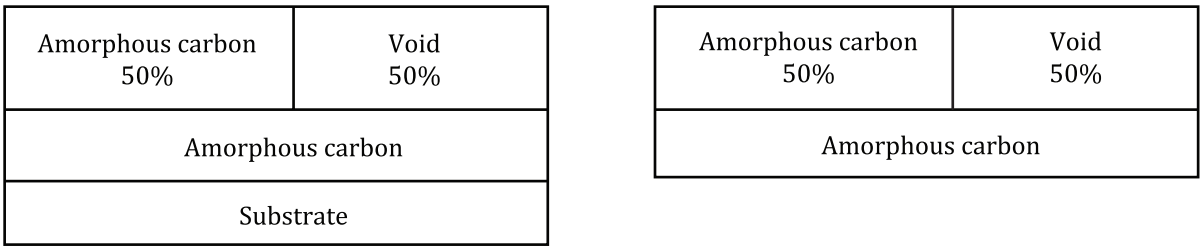
The test provides data for the angle of the ratio for the amplitudes of reflected p and s polarized lights, and the phase difference.

From the refractive index and extinction coefficient spectra, the values at or as close as possible to 550 nm should be used for amorphous carbon classification.

6.4 Optical model for analysis

For the first layer of amorphous carbon film, the Tauc-Lorentz dispersion formula shall be used. The second layer shows surface roughness as a mixture of amorphous carbon and voids using the effective medium theory model using Bruggeman's effective medium approximation (EMA).

Optical model 1, shown in Figure 1 a), shall be used for the first layer. Optical model 2, as shown in Figure 1 b), shall be used when the amorphous carbon did not exhibit transparency, meaning the light does not reach the substrate. The substrate model shall be removed. To apply the optical model properly, amorphous carbon shall be used as a substrate model for the calculations. The surface roughness layer shall be analysed using EMA for the first layer.



a) Optical model 1 **b) Optical model 2**

Figure 1 — Optical models for amorphous carbon film

Execute the measurement recipe, and analyse the resulting measurements and data.

The results of the data analysis show the thickness of the layer and the optical value (refractive index, n, extinction coefficient, k).

6.5 Number of test repeats

Repeat the test at least three times under the same testing conditions.

7 Classification of test results

The internal structure of amorphous carbon films shall be classified based on the refractive index and extinction coefficient determined by the spectroscopic ellipsometry at a wavelength of 550 nm in accordance with Annex A.

8 Test report

The test report shall contain the following information:

a) a reference to this document, i.e. ISO 23216:2021;

b) a description of the material or materials tested; the coating method shall be described if known;

c) the specification of the testing apparatus;

d) the testing conditions;

e) for each individual test:

   1) the testing temperature, humidity and their ranges;

   2) the dimension of the specimen;

   3) the optical model applied for the calculation;

4) the refractive index and extinction coefficient of the amorphous carbon film at a wavelength of 550 nm from the calculated curve;

5) any other relevant matters regarding the state of the test and the specimen after the test;

f) the method used;

g) any deviations from the procedure;

h) any unusual features observed;

i) the date of the test.