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Citation for published version (APA):
Smit, M. K., & Verhoof, J. W. (1990). A bounded-error approach to accuracy analysis in ellipsometry. *Mathematics and Computers in Simulation*, *32*(5-6), 545-551. https://doi.org/10.1016/0378-4754(90)90010-G

DOI:
10.1016/0378-4754(90)90010-G

Document status and date:
Published: 01/01/1990

Document Version:
Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:

• A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
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Mathematics and Computers in Simulation 32 (1990) 545–551
North-Holland

A BOUNDED-ERROR APPROACH TO ACCURACY ANALYSIS IN ELLIPSOMETRY

M.K. SMIT and J.W. VERHOOF

Delft University of Technology, Department of Electrical Engineering, Laboratory for Telecommunication and Remote Sensing Technology, P.O. Box 5031, 2600 GA Delft, Netherlands

A nonlinear Bounded-Error Estimation method is applied to the case of ellipsometric measurement of film properties. It is shown that this method can be used with advantage for estimating the magnitude of the measurement errors including systematic errors.

1. Introduction

Ellipsometry is a fast and nondestructive method for measuring optical film properties [1,2]. The method is based on measuring the complex ratio of the reflection coefficients of a film-covered substrate for light polarized parallel to and perpendicular to the incidence plane respectively. From this ratio the thickness and the refractive index of the film can be computed, if the substrate index is known. If the latter is not known, or the refractive index has a nonnegligible imaginary part, additional measurements are necessary to determine the unknown parameters.

Additional measurements can also be used to increase the measurement accuracy. They can be obtained by measuring at multiple angles of incidence (MAI), multiple wavelengths (MW) or both (MAW). We analyzed the accuracy improvements which can be obtained with commercially available measurement equipment operating at a single wavelength (633 nm) with three fixed incidence angles (30°, 50° and 70°).

Accuracy analysis of MAI-measurements has been reported with Least-Squares Estimation (LSE) methods [3,4] and parameter correlation analysis [5]. In this paper the potential of the bounded-error approach for analysing measurement accuracy, also in strongly nonlinear regions, will be demonstrated. A more detailed description of the results is given elsewhere [6]. Further, the potential of the bounded-error approach for estimating the magnitude of random and systematic errors will be shown.

2. Measurement method

Optical film and substrate properties are calculated from the measured complex ratio

$$\rho = \rho^p / \rho^s \tag{1}$$

of the reflection coefficients ρ^p and ρ^s for light polarized parallel and perpendicular to the plane

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of incidence respectively. Usually ρ is represented by the parameters Δ and Ψ, which are defined as

$$\Delta = \arg(\rho), \tag{2a}$$

$$\Psi = \arctan(|\rho|). \tag{2b}$$

The parameters Δ and Ψ can be written as functions of the (complex) film and substrate refractive indices n_f and n_s, the film thickness d, and the incidence angle ϕ_0 (for details see [1]):

$$\Delta = \Delta(n_\mathrm{f}, n_\mathrm{s}, d, \phi_0), \tag{3a}$$

$$\Psi = \Psi(n_\mathrm{f}, n_\mathrm{s}, d, \phi_0). \tag{3b}$$

If n_s is known, and the film absorption (i.e. the imaginary part of n_f) can be neglected, n_f and d can be determined from a single measurement of Δ and Ψ using equations (3a) and (3b). With these formulae the influence of measurement errors in Δ and Ψ on the calculated values for n_f and d can be analysed.

The accuracy can be improved by measuring Δ and Ψ at n incidence angles instead of at a single one. This gives us $2n$ equations (the real and imaginary part of equations (3a) and (3b) for each of the incidence angles) with two unknowns. The usual approach to finding n_f and d from this set is made by applying an LSE method. Loescher et al. [3] and Humlícek [4] analyzed the accuracy in the parameter estimates which is obtained if an LSE method is applied. This analysis assumes the measurement errors to be normally distributed and described by their covariance matrix. From this matrix the errors in the unknown parameters can be inferred if equations (3a) and (3b) can be written in matrix form, i.e. can be linearly approximated around the observation point.

In strongly nonlinear regions a more complicated approach is necessary to estimate the parameter errors. To avoid this problem we applied a Bounded-Error (BE) approach, as introduced by Schweppe [7] and Witsenhausen [8], instead of a conventional LSE method. An additional advantage of the BE-approach is that it offers an easy means of testing the validity of the assumptions made about the actually occurring observation errors, which are often difficult to validate.

A recent survey of Bounded-Error estimation techniques has been made by Walter and Piet-Lahanier [9]. Most authors in this field work with linear approximations. Walter and Piet-Lahanier [10] describe a method based on minimizing the number of outliers in the measured data, which also applies to nonlinear models. Norton [11] maps the error bounds of each individual observation into the (two-dimensional) parameter space, and determines the set of parameters which are consistent with the noise-corrupted observations as the region enclosed within all bounds. We applied a closely related approach which will be briefly described below.

For the present problem the observation is represented as a hypercube B in a six-dimensional space set up by the six measurement entities (Δ and Ψ at 30°, 50° and 70° incidence angles). The length of the edges is determined by the magnitude of the errors in the different entities. Because the possible values of Δ and Ψ are determined by only two parameters n_f and d, the model response surface (defined by equations (3a) and (3b)) is two dimensional. *Figure 1* illustrates the foregoing for a three-dimensional observation space (a six-dimensional one being difficult to draw). The (nonlinear) response plane represents the subspace of observations which

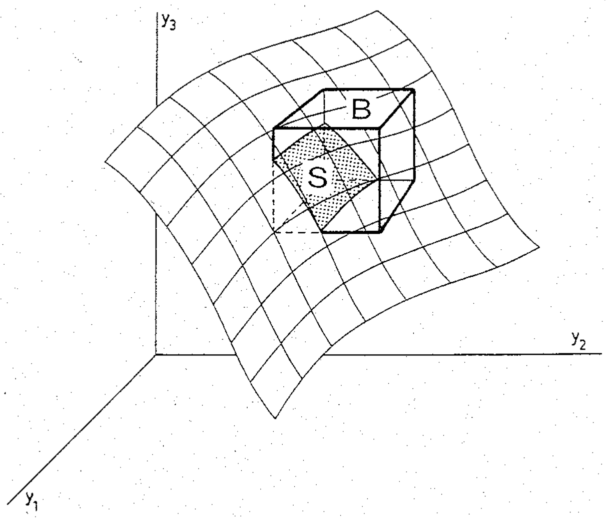

Fig. 1. Cross-section (shaded region) of a two-dimensional model response surface with a three-dimensional observation uncertainty region.

are physically possible, the orthogonal lines indicating lines of constant n_f and d. The model membership set S, i.e. the region of values of n_f and d which could have caused the actual observation, given the possible observation errors, is the shaded part of the response plane, falling within the cube.

To determine the size and the shape of the model membership set S we applied the boundary-detection algorithm described by Freeman [12]. In this algorithm a rectangular grid is defined in the parameter (n_f, d) space. The boundary contour is computed by determining between which two of the eight grid points, surrounding the previously found contour point, the boundary is located. The one of these two points which is a member of S (i.e. for which the model response is a member of B), is the next contour point, and the procedure is repeated until the contour is closed. Application of this algorithm avoids the need of inverting the model description (equations (3a) and (3b)) in order to determine the parameter bounds. The computation time of the algorithm is linear in the number of dimensions of the observation space so that it is capable of handling long measurement records without excessive computation times. The method is implemented into a computer program, and described in more detail by Smit and van Vliet [13,14].

Figure 2 shows an example of the model membership set computed in this way. From a plot like this the maximal error in n_f and d can be read directly. Further, the effect of a priori knowledge about one of the parameters, for example n_f, on the measurement accuracy of the other is immediately seen: the membership set is reduced to the slice falling within the error bounds of n_f (the striped region).

If the measurement errors are estimated too optimistically, or if the description of equations (3a) and (3b) is not exact (e.g. due to film anisotropy or the presence of a thin film of adsorbed water) no intersection between the cube B and the response plane may occur at all. This gives us a means of testing the validity of our assumptions about the actual measurement accuracy for Δ and Ψ. By computing S, as shown in Fig. 2, for a considerable number of independent

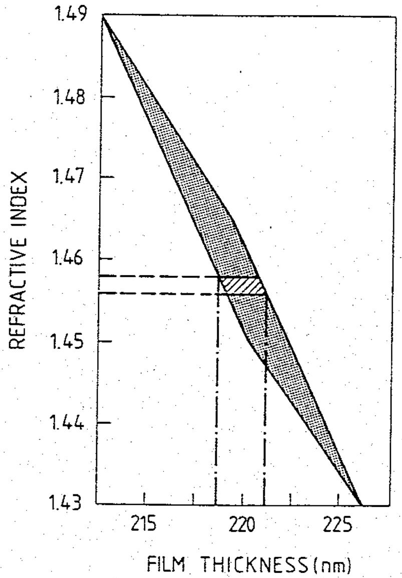

Fig. 2. Membership set for n_f and d, with (striped) and without (dotted) a priori knowledge about the refractive index.

observations, the smallest error-value for Δ and Ψ for which all observations yield nonempty sets may be considered as an indication of the actual measurement accuracy, as will be discussed in Section 4.

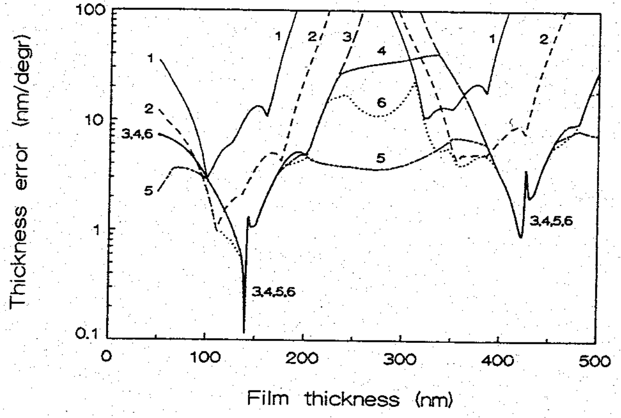

Fig. 3. The measurement errors in the film thickness resulting from a measurement error of 1° in Δ and Ψ:
Curve 1: 30° incidence angle, no restriction on n_f,
Curve 2: 50° incidence angle, no restriction on n_f,
Curve 3: 70° incidence angle, no restriction on n_f,
Curve 4: 70° incidence angle, $\delta n_f < 0.01$,
Curve 5: 70° incidence angle, $\delta n_f < 0.001$,
Curve 6: 30°–50°–70° combined, no restriction on n_f.

3. Accuracy results

To determine the accuracy of the single- and three-angle measurements, we analyzed the effect of measurement errors in Δ and Ψ on the estimated values of n_f and d for a thermally oxidized SiO_2 layer on a silicon substrate.

Figure 3 shows the maximum error in the film thickness d, for thickness values ranging from 100 to 400 nm. The error is computed as the half width of the projection of the membership set S onto the d-axis, as shown in Fig. 2. The curves show the effect of an error of 1° both in Δ and Ψ. The first three curves represent the accuracy of results based on a single-angle measurement, for 30°, 50° and 70° incidence angle respectively. The fourth and the fifth curve show the effect of a priori knowledge about the refractive index. The sixth curve represents the results of combining the observations made at 30°, 50° and 70° incidence angle.

The single-angle curves show singularities in the range from 200 to 300 nm. These singularities correspond to $\frac{1}{2}\lambda$ film thickness (i.e. a half wavelength). The 70° curve (3) shows a singularity at $\frac{1}{4}\lambda$ wavelength. This singularity occurs at the pseudo-Brewster angle, which equals 70° for an oxide-covered silicon substrate. In these regions linear approximations may yield erroneous results.

4. Accuracy analysis

To test the validity of the applied method we measured a number of records, each consisting of Δ and Ψ measured at 30, 50° and 70°, on three silicon substrates covered with thermal oxide films with 145, 195 and 235 nm thickness respectively (i.e. 0.25λ, 0.35λ and 0.4λ). The 0.25λ-substrate was measured at 9 different positions. The other substrates, which showed less spread in the observations, were measured at 6 positions. The number of measurement records thus totalled 21 three-angle records or 63 single-angle records.

The random variation of Δ and Ψ over the substrates was found to amount to $\pm 0.15°$ for Δ and $\pm 0.4°$ for Ψ. We tested the significance of these figures by substituting them as the maximum observation errors for all measured records. We found no solutions for n_f and d (i.e. all membership sets S were empty) for any of the 21 records. The error value had to be increased to 0.4° for both Δ and Ψ for the first nonempty set to appear. On a further increase to 0.6° all but three empty sets disappeared, which suggests that the systematic errors are considerably larger than the random ones. The remaining three empty sets all occurred for the 0.25λ-film, the last one disappeared on increasing the measurement error to 1.7°.

These results illustrate that in a practical measurement situation the errors may be considerably greater than those which are usually presented in the literature (0.01°-0.1°) for comparable measurement equipment. The discrepancy may be due to systematic errors occurring in the observation of Δ and Ψ (the error structure, see Belforte et al. [15]), but more probably to the deviation of the experimental measurement configuration from its idealized description (the model structure, equations (3a) and (3b)), as may occur due to adsorption of a thin water layer, depolarization due to surface scatter or birefringence due to tension in the film, to mention just a few examples.

Systematic errors (both in the observations and the model structure) will cause a displacement of the model response surface and the (hyper)cube, as shown in Fig. 1, relative to each other. If this displacement is directed along the response surface, the systematic error is not detectable with the present approach. If it is directed perpendicular to this surface, however, the lowest value of the observation errors (i.e. the edges of the hypercube) for which none of the measurement records yields an empty membership set S, is indicative for the total effect of both random and systematic errors (a fixed ratio between the observation errors has been assumed). By computing the membership sets S corresponding to the observation errors so found, we obtain an estimate of the magnitude of parameter errors resulting from both systematic and random errors (except, of course, for the worst-case record on which the error estimate was based).

Assume that the number of measurements is much larger than the number of parameters. The projection on the response surface of the displacement induced by systematic errors is then likely to be small in relative terms. The error estimates produced by the above algorithm are therefore unlikely to be much too optimistic.

To investigate the practical value of the algorithm we proceeded as follows. For all measurements we computed the refractive index value as the centre of the uncertainty interval. The error in this value was determined by comparing it to the literature value for fused silica (1.4573, [16]) which is assumed to be close to the value of thermal oxide. In this way maximum errors were determined for both single and three-angle measurements, for the three film thicknesses investigated.

Of a total of approximately 60 independent measurements we found only two cases in which the real measurement error, i.e. the difference between the literature value and the estimated value, exceeded the one predicted according to the above algorithm. In the worst of them, the real error was twice the one predicted. Both discrepancies occurred for the 0.25λ film, for measurement sets of which the computed parameters coincided with the singularity observable in Fig. 3 (at 140 nm). If we exclude these measurements, the maximum errors found for the different measurement records were within 15–80% of the predicted errors, and for 60% of the series within 40–80%.

Although no general conclusions can be drawn from these experiments, the results indicate that in the field of ellipsometry the above algorithm may be applied with advantage to analyse the magnitude of both random and systematic errors.

5. Conclusions

Film parameters may be determined from ellipsometric measurement results both with a Least-Squares Estimation method or a Bounded-Error Estimation method. The latter method yields reliable accuracy information also in parameter regions where the dependence of the observations on the parameters is highly nonlinear. Further, it provides a tool for analyzing the maximal measurement errors, both random and systematic, which occur in the applied measurement procedure.

References

[1] R.M.A. Azzam and N.M. Bashara, *Ellipsometry and Polarized Light* (North-Holland, Amsterdam, 1977).

[2] F.L. McCrackin, E. Passaglia, R.R. Stromberg and H.L. Steinberg, Measurement of the thickness and refractive

index of very thin films and the optical properties of surfaces by ellipsometry, *J. Res. Nat. Bur. Standards* 67A (1963) 363–377.

[3] D.H. Loescher, R.J. Detry and M.J. Clauser, Least-squares analysis of the film-substrate problem in ellipsometry, *J. Opt. Soc. Amer.* 61 (1971) 1230–1235.

[4] J. Humlícek, Sensitivity extrema in multiple-angle ellipsometry, *J. Opt. Soc. Amer. A* 2 (1985) 713–722.

[5] H.W. Dinges, Determination of the optical properties of GaAs and InP by multiple angle of incidence ellipsometry, *J. Physique, Coll. c10, Suppl. au No.* 12, *Tome* 44 (1983) 33–43.

[6] M.K. Smit and J.W. Verhoof, Accuracy analysis in multiple angle of incidence ellipsometry, *Thin Solid Films* 189 (2) (1990) 193–203.

[7] F.C. Schweppe, Recursive state estimation: unknown but bounded errors and system inputs, *IEEE Trans. Automat. Control* AC-13 (1968) 22–28.

[8] H.S. Witsenhausen, Sets of possible states of linear systems given perturbed observations, *IEEE Trans. Automat. Control* AC-13 (1968) 556–558.

[9] E. Walter and H. Piet-Lahanier, Estimation of parameter bounds from bounded-error data: a survey, in: *Proc. 12th IMACS World Congress on Scientific Computation*, Paris, Vol. 2 (1988) 467–472.

[10] E. Walter and H. Piet-Lahanier, Robust nonlinear parameter estimation in the bounded noise case, in: *Proc. 25th IEEE Conference on Decision and Control*, Athens (1986) 1037–1042.

[11] J.P. Norton, Problems in identifying the dynamics of biological systems from very short records, in: *Proc. 25th IEEE Conference on Decision and Control*, Athens (1986) 286–290.

[12] H. Freeman, Boundary encoding and processing, in: B.S. Lipkin and A. Rosenfeld, eds., *Picture Processing and Psychopictorics* (Academic Press, New York, 1970).

[13] M.K. Smit, A novel approach to the solution of indirect measurement problems with minimal error propagation, *Measurement* 1 (4) (1983) 181–190.

[14] M.K. Smit and C.H. van Vliet, OMTP: Fortran program for optimally solving indirect measurement problems, *Measurement* 1 (4) (1983) 209–211.

[15] G. Belforte, B. Bona and V. Cerone, Identification, structure selection and validation of uncertain models with set-membership error description, *Math. Comput. Simulation* 32 (5&6) (1990) 561–569 (this issue).

[16] *American Institute of Physics Handbook* (McGraw-Hill, New York, 1982).