

XM-17460

**THIN FILM ANALYSIS
PROGRAM**

For the proper use of the instrument, be sure to read this instruction manual. Even after you read it, please keep the manual on hand so that you can consult it whenever necessary.

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THIN FILM ANALYSIS
PROGRAM

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1 GENERAL

This thin film analysis program is option software for the quantitative analysis programs described in the separate “BASIC SOFTWARE 1” instruction manual. It is intended for thin film analysis using the Philibert & Tixier method* and also the Reuter method**.

In actual analysis, this program is used in combination with the quantitative analysis program.

The following correction methods are used with this program.


- **Philibert & Tixier method**

This is a method of performing quantitative analysis of thin films using a bulk standard specimen. It enables the mass/concentration ratio of all elements in a thin film to be obtained when the thin film alone is maintained in a vacuum (the final results are normalized to 100%).

 This method requires that the film be sufficiently thin.

- **Reuter method**

This method is used in combination with the Philibert & Tixier method when the thin film is on a substrate and the substrate contains none of the elements that are in the film. In this method, an X-ray intensity correction term is added to take account of excitation by the backscattered electrons from the substrate.

 This method requires that the substrate does not contain any of the elements that are in the film.

* Philibert, J and Tixier, R. (1968). Brit. J. Appl. Phys., 1, 685.

** Reuter, W. (1972). Proc. 6th. Int. Conf. on X-ray Optics and Microanalysis, eds. Shinoda, G. et al., Univ. of Tokyo Press, Tokyo, 121.

2 CORRECTION METHOD

2.1 Philibert & Tixier Method

The specimen is a thin film without a substrate that is maintained in a vacuum.

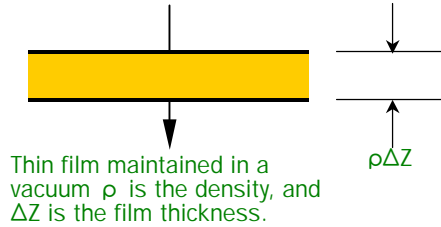


Fig. 1 Philibert & Tixier method

Assuming that the standard specimen is a bulk sample which consists of a pure element i , the intensity of the X-rays is determined according to the following equation.

$$I_i^{100A} = \frac{W_i \times N_i \times R_i}{A_i \times F_i} \quad (i = 1 \sim n) \quad \dots\dots\dots (1)$$

On the other hand, if the thickness of the thin film is assumed to be ΔZ cm, the intensity of the X-rays emitted from the thin film for element i is determined according to the following equation.

$$I_i^F = C_i \cdot \frac{W_i \times N_i}{A_i} \cdot \psi_i \rho \Delta Z \quad \dots\dots\dots (2)$$

where

- | | |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <p>W_i: Fluorescence absorption rate of element i</p> <p>N_o: Avogadro's number</p> <p>A_i: Atomic weight of element i</p> <p>Ψ_i: Ionization cross-sectional area</p> <p>R_i: Back scattering coefficient of element i</p> | <p>F_i: $1 / (\int_{E_{ki}}^{E_o} \frac{\Psi_i}{S_i} dE)$</p> <p>$\rho$: Density</p> <p>$E_o$: Electron beam accelerating voltage</p> <p>E_{ki}: Absorption edge of element i</p> <p>S_i: Stopping power of element i</p> |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|

Assuming that the film is sufficiently thin, the X-ray absorption by the film can be ignored. Consequently, if only the absorption of the bulk standard specimen is taken into account, the ratio of the X-ray intensity for element i between a standard specimen and a thin film is obtained using equations (1) and (2), as follows:

$$K_i = \frac{I_i^F}{I_i^{100A}} = C_i \cdot \frac{\psi_i \times F_i}{R_i} \cdot \rho \Delta Z \cdot \frac{1}{f(x)_i} \quad \dots\dots\dots (3)$$

If, now, K_j is calculated for element j , the ratio of the X-ray intensities is determined according to the following equation.

$$\frac{K_i}{K_j} = \frac{C_i \cdot \psi_i \cdot (F_i / R_i) \cdot (1 / f(x)_i)}{C_j \cdot \psi_j \cdot (F_j / R_j) \cdot (1 / f(x)_j)} \quad \dots\dots\dots (4)$$

Thus $\rho\Delta Z$ can be eliminated. In other words, the resulting equation does not depend upon the thickness of the film. Next, re-arrange equation (4) as follows:

$$\frac{C_i}{C_j} = \frac{K_i}{K_j} \cdot \frac{(1/\psi_i) \cdot (R_i/F_i) \cdot f(\chi)_i}{(1/\psi_j) \cdot (R_j/F_j) \cdot f(\chi)_j} \dots\dots\dots (5)$$

Here,

$$\psi_i \times E_{ki}^2 = 7.92 \times 10^{-14} \ell n U_0(i) / U_0(i)$$

where

$$U_0(i) = E_0 / E_{ki}$$

Therefore,

$$G_i = (E_{ki} / \ell n U_0(i)) \cdot (R_i / F_i) \cdot f(\chi)_i$$

Consequently, equation (5) can be re-written as follows.

$$\frac{C_i}{C_j} = \frac{K_i}{K_j} \cdot \frac{G_i}{G_j} \dots\dots\dots (6)$$

Here,

$$C_1 + C_2 + \dots + C_n = 1$$

Therefore,

$$\frac{C_1}{C_j} + \frac{C_2}{C_j} + \dots + \frac{C_n}{C_j} = \frac{1}{C_j}$$

$$\therefore C_j = \frac{1}{\sum_i (C_i / C_j)}$$

$$\therefore C_i = \frac{K_i}{K_j} \cdot \frac{G_i}{G_j} C_j$$

Consequently, the concentration of all elements in the thin film can be obtained.

2.2 Reuter Method

This method assumes that none of the elements in the thin film are contained in the substrate.

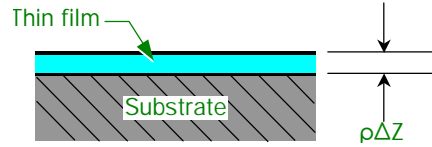


Fig. 2 Reuter method

Reuter assumed in 1972 that the X-ray intensity for element i contained in the thin film is intensified by a factor of R_0 by the backscattered electrons from the substrate compared to the case where the film is maintained in a vacuum.

R_0 is calculated using the following equation.

$$R_0 = 1 + 2.8 \left(1 - \frac{0.9}{U_0(i)}\right) \eta$$

where η is the reflection coefficient of the substrate for the electron beam. It is determined using the following equation.

$$\eta = -0.0254 + 0.016Z - 0.000186Z^2 + 8.3 \times 10^{-7} Z^3$$

In the case of a compound,

$$\eta = \sum_i C_i \eta_i$$

In other words, if the specimen is on a substrate, equation (2) can be re-written as shown below.

$$I_i^F = C_i \cdot \frac{W_i \times N_o}{A_i} \cdot \psi_\lambda \cdot \rho \Delta Z \cdot R_{oi} \dots \dots \dots (2)'$$

In other words, equation (3) can be re-written as follows.

$$K_i = C_i \cdot \frac{\psi_i \times F_i}{R_i} \cdot \rho \Delta Z \cdot \frac{1}{f(x)_i} \cdot R_{oi} \dots \dots \dots (3)'$$

Equation (4) can be re-written as follows.

$$\frac{K_i}{K_j} = \frac{C_i}{C_j} \cdot \frac{\psi_i \cdot (F_i / R_i) \cdot (1 / f(\chi)_i)}{\psi_j \cdot (F_j / R_j) \cdot (1 / f(\chi)_j)} \cdot \frac{R_{oi}}{R_{oj}} \dots \dots \dots (4)'$$

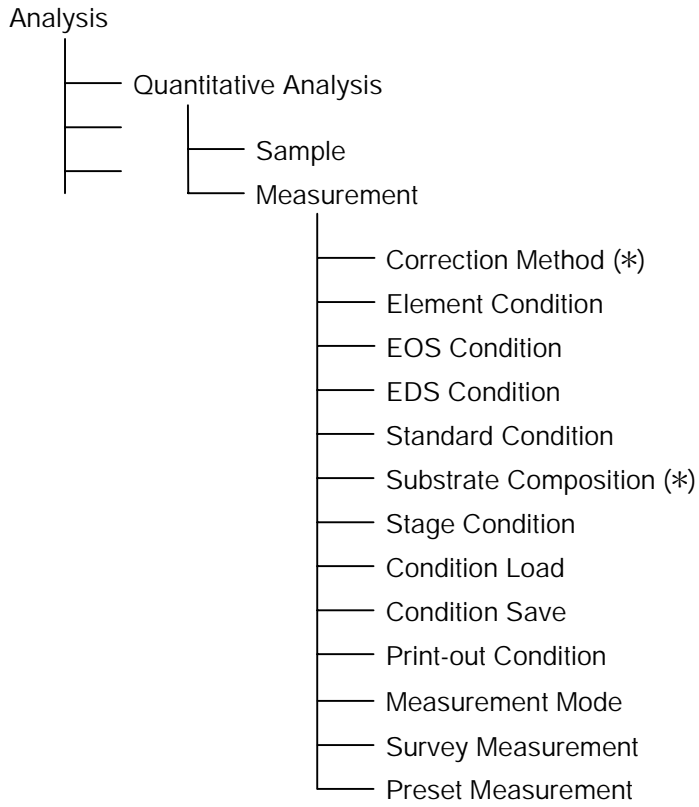
Also, the following relationship holds.

$$\frac{C_i}{C_j} = \frac{K_i}{K_j} \cdot \frac{G_i}{G_j} \cdot \frac{1 / R_{oi}}{1 / R_{oj}} \dots \dots \dots (5)'$$

Thus the concentration of all of the elements in the thin film can be obtained.

3 PROGRAM STRUCTURE

The thin film analysis program is contained in the Measurement directory under the Quantitative Analysis program.



* Changed/added for thin film measurement.

4 OPERATION

The basic operation of this program is the same as that of the quantitative analysis program. The following operation sequence describes parts that differ from the quantitative analysis program and also parts that have been newly added for thin film analysis.

☞ For a description of operation of the quantitative analysis program, refer to the separate “BASIC SOFTWARE 1” instruction manual.

4.1 Quantitative Analysis Menu Display

Click on the Analysis icon in the EPMA menu, then click on the item “Quantitative Analysis” in the sub-menu. The Quantitative Analysis window will appear. Next, click on the Measurement button in the window. The Measurement menu will appear. The items “Correction Method” and “Substrate Composition” have been added by the thin film analysis program.

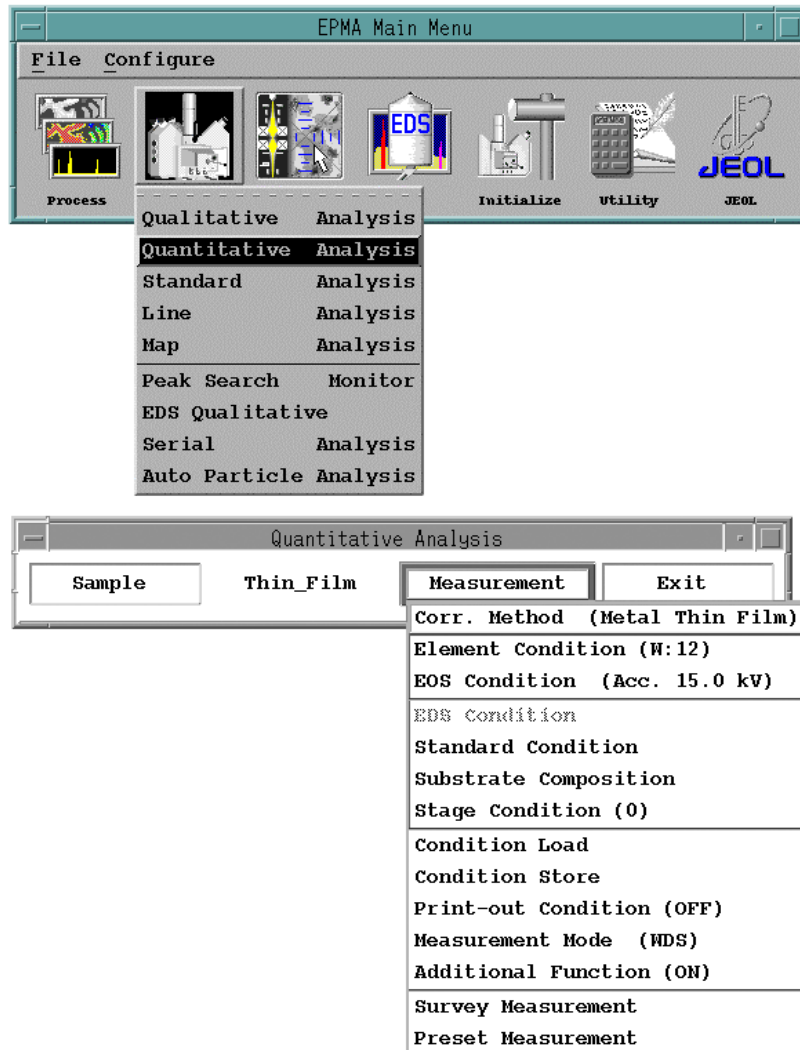


Fig. 3 Quantitative analysis menu display

4.2 Correction Method Window

When performing thin film analysis, click on the item “Correction Method” in the Measurement menu to display the Correction Method window (refer to Fig. 4), then click on the Thin Film button of Correction Method in this window.

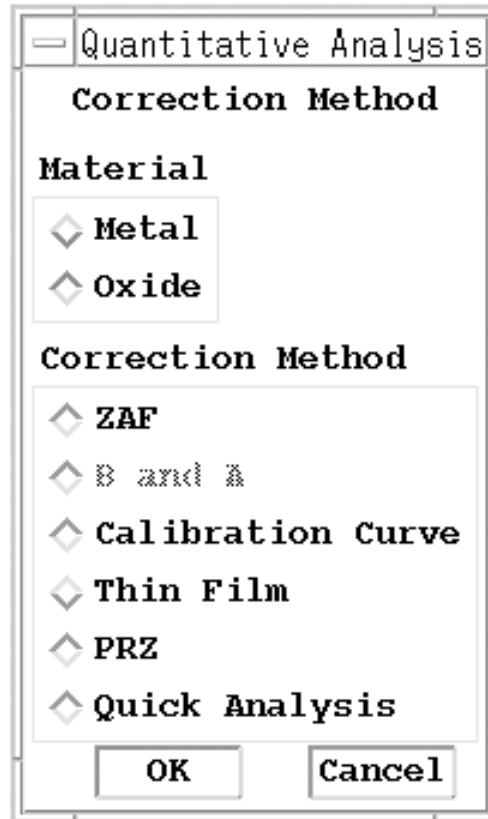


Fig. 4 Correction method window

4.3 Setting the Accelerating Voltage

When performing thin film analysis, the film must be sufficiently thin. In many cases, this assumption will not hold if the film thickness exceeds 1000 to 2000 nm. In such a case, if the accelerating voltage is increased, the X-ray emission area will increase, enabling the film to be considered relatively thin. Based on this fact, there is a method of thin film analysis in which the accelerating voltage is set higher than normal in order to provide better results.

4.4 Setting a Standard Specimen

In thin film analysis, a normal bulk standard specimen is used, so specify the standard specimen in the normal way.

4.5 Substrate Composition Window

By clicking on the item “Substrate Composition” in the Measurement menu, the Substrate Composition window (refer to Fig. 5) will appear, enabling a thin film on a substrate to be analyzed.

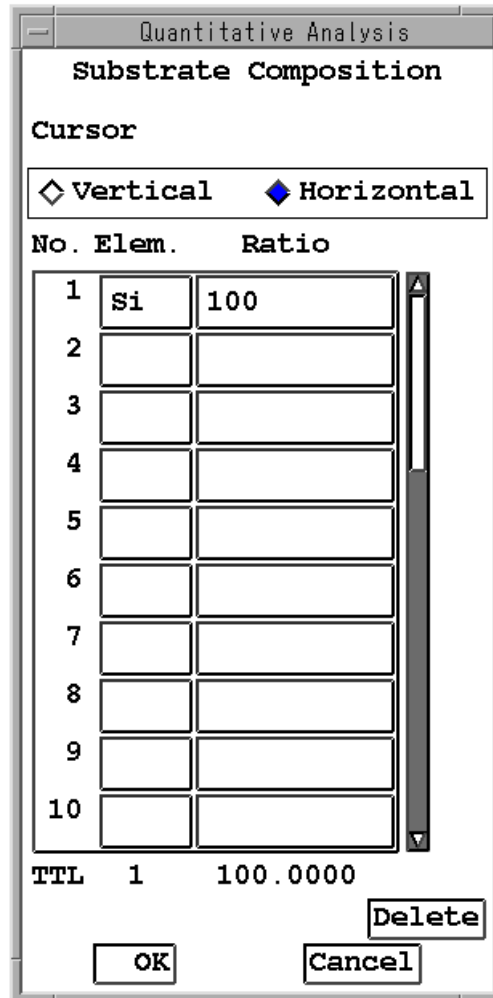


Fig. 5 Substrate Composition window


Set the Substrate Composition window using the following procedure.

1. Enter the elements to be analyzed in the Element column of the table in the Substrate Composition window.
2. Enter the mass concentration (%) in the Mass % column.

If the thin film analysis is performed according to the above procedure, the contribution degree, R_0 , due to back scattering from the substrate will be calculated by the Reuter method, and the result displayed in the Listing Window (refer to Fig. 6). When analyzing a thin film without a substrate, there is no need to make an entry to the Substrate Composition window.

4.6 Executing Thin Film Correction

If each of the above settings is carried out and quantitative analysis performed, thin film correction will take place after measurement, and the results will be displayed in the Listing window (see Fig. 6).

 The mass concentration is always 100% normalized.

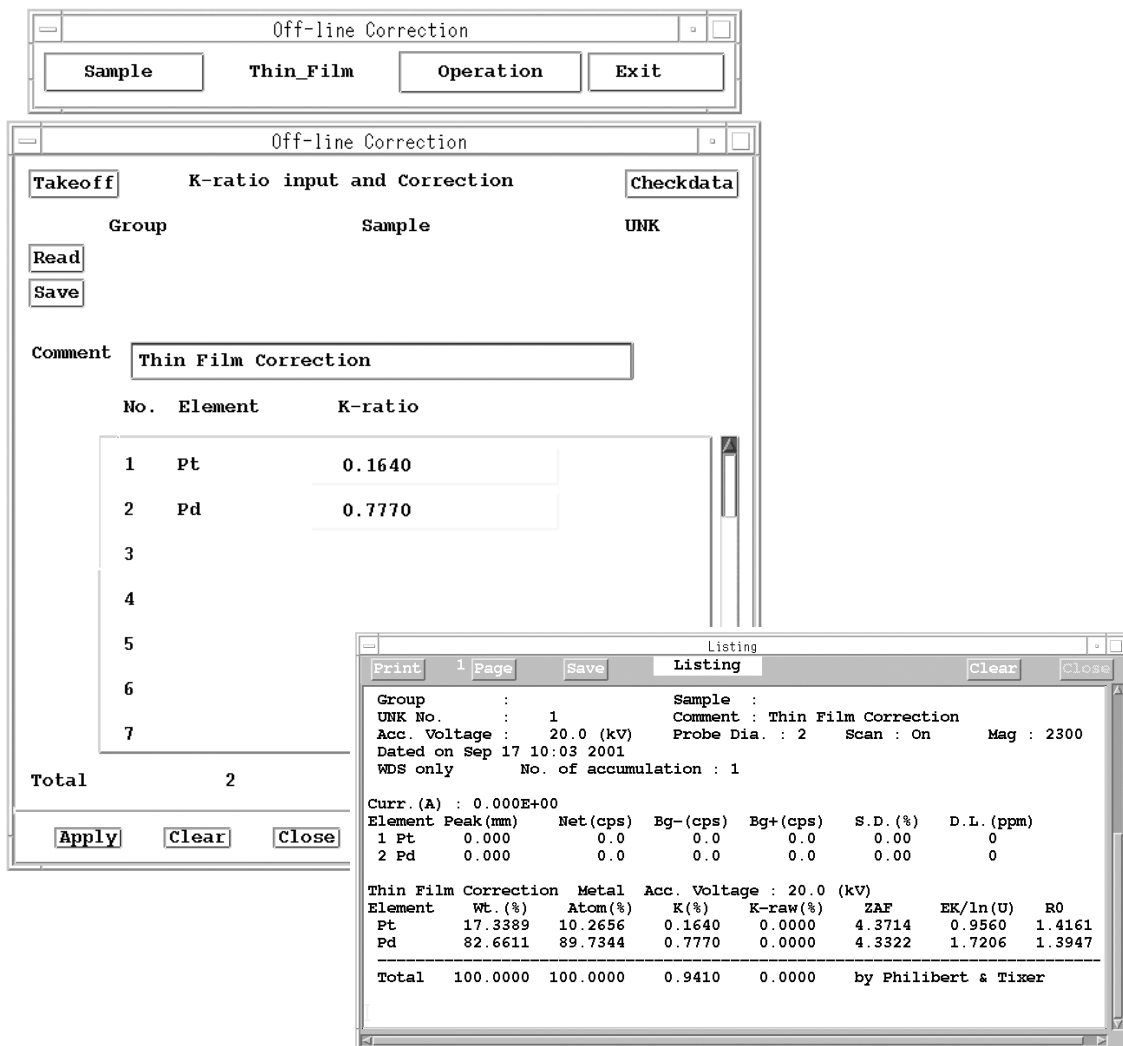


Fig. 6 Listing window

The contents of the displayed items are as follows.

- EK/ln (U): Term of the X-ray generation function for thin film in the Philibert & Tixier method.
- R0: Correction term related to the influence on the X-ray intensity due to backscattered electrons in the Reuter method.
- ZAF: (STD Factor) · EK/ln (U)/R0
By multiplying this value by the K-ratio, the mass concentration will be obtained.