XM-17410

SPECIMEN DECONVOLUTION PROGRAM

IXM-17410-1 (33500)
JAN2002-03220160
Printed in Japan

JEOL
Serving Advanced Technology

XM-17410

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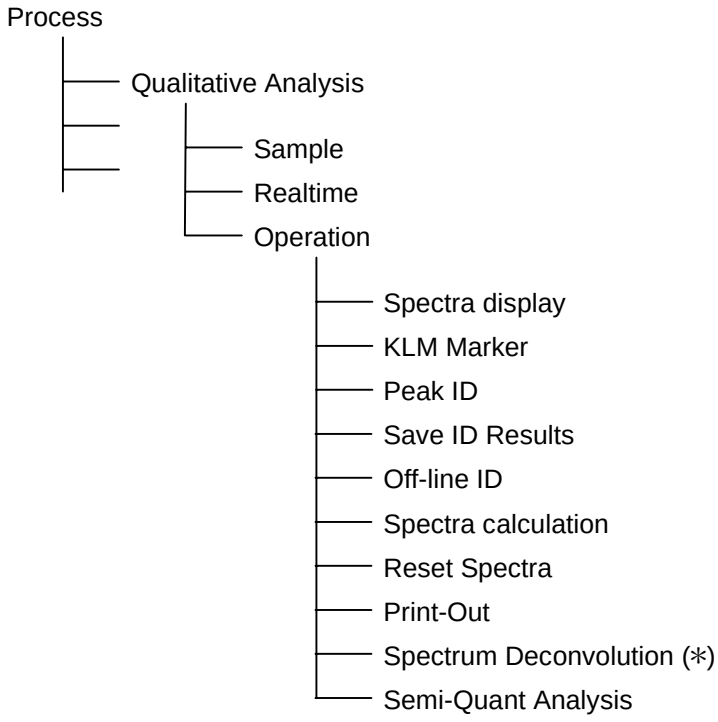
1 GENERAL

The spectrum deconvolution program is an optional program for the qualitative analysis program described in the separate manual “BASIC SOFTWARE 1”. In accordance with the spectral data on standard samples, it deconvolutes multiple elements’ overlapping characteristic X-ray peaks in an unknown samples spectrum selected from among the spectral data measured by the qualitative analysis program.

This program displays one spectrum of an unknown sample together with up to 7 spectra of standard samples. Then its spectrum deconvolution function carries out top hat filtering on each spectrum, allowing the mixing ratio of the standard sample spectra (k-Ratio) to be shown and the deconvoluted spectra of the standard samples to be displayed with the least square fitting method.

2 PROGRAM STRUCTURE

This program is included in “Spectrum Deconvolution” positioned under “Operation” of the Qualitative Analysis program.



* This item is effective when the optional software is installed.

3 OPERATIONS

This section assumes that the Qualitative Analysis Program has already acquired the standard sample spectra and unknown sample spectra.

✍ When carrying out spectrum acquisition with the qualitative analysis program, keep the following points in mind.

- To carry out high precision spectrum deconvolution, the same analyzing crystal in the same spectrometer channel must be used and the same measurement conditions must be set for measurement of unknown and standard sample spectra.
- When selecting a standard sample for an element, use a sample which includes no other elements to undergo spectrum deconvolution and which gives rise to no peaks of other elements around the X-ray peaks to be deconvoluted.
- The measurement range for spectra must be set so that their peak slopes are fully covered.

3.1 Displaying Spectra of Standard Samples and Unknown Samples

Click on the Process icon in the EPMA menu. A pulldown menu appears. Click on Qualitative Analysis in this menu to display the Qualitative Analysis window. Then click on the Sample button in this window to display the unknown or standard sample spectra.

✍ Display a spectrum of an unknown sample in the first place, then the spectra of standard samples.

✍ The spectrum deconvolution function does not operate when spectra are shown in keV. The message “No calculation for ‘keV’” will be displayed. On such an occasion, redisplay the spectra by selecting mm or nm for Display Axis after clicking on Spectra Display in the pulldown Operation menu.

✍ If no common measurement range (mm/nm) exists in the spectra of the unknown and standard samples, the message “Range unmatch” is displayed. On such an occasion, reset the measurement range for the unknown sample spectra or the standard sample spectra.

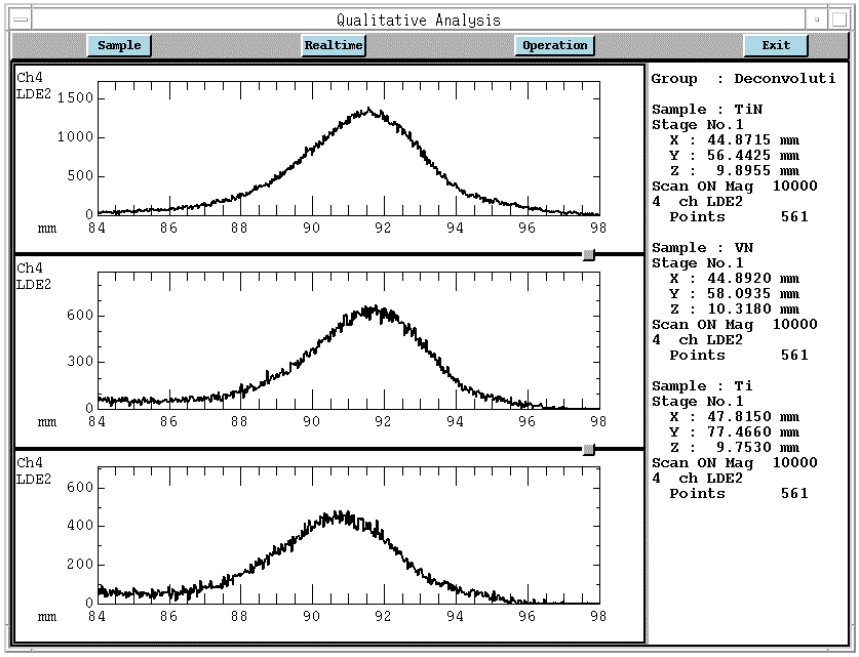


Fig. 1 Display of unknown and standard sample spectra

■ Displaying an unknown sample spectrum and standard sample spectra concurrently on the same coordinates axes

Click on Spectra Display in the Operation menu to display its submenu. Click on Mixed Spectra Display in the submenu, and the Mixed Spectra Display window will be displayed. In the window, select the Single and Absolute buttons by clicking on them.

Prior to performing spectrum deconvolution, get rid of backgrounds as necessary.

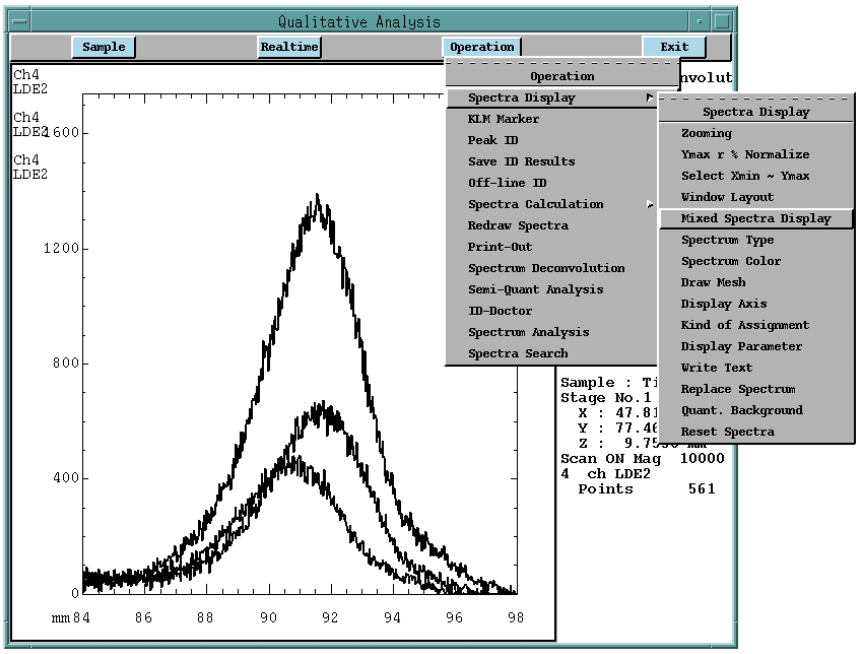


Fig. 2 Mixed Spectra Display window

3.2 Spectrum Deconvolution Window

Click on Spectrum Deconvolution in the Operation menu, and the Spectrum Deconvolution window (an operation window for spectrum deconvolution) will be displayed. Here, set the measurement conditions for spectrum deconvolution. The comment on and the coefficient and mixing ratio (k-Ratio) of the unknown and standard sample spectra are displayed in the window. If there are no previous values, the Comment area will be blank, and "1.000" and "−−−−−" will be set in the Coefficient area and the k-Ratio area, respectively.

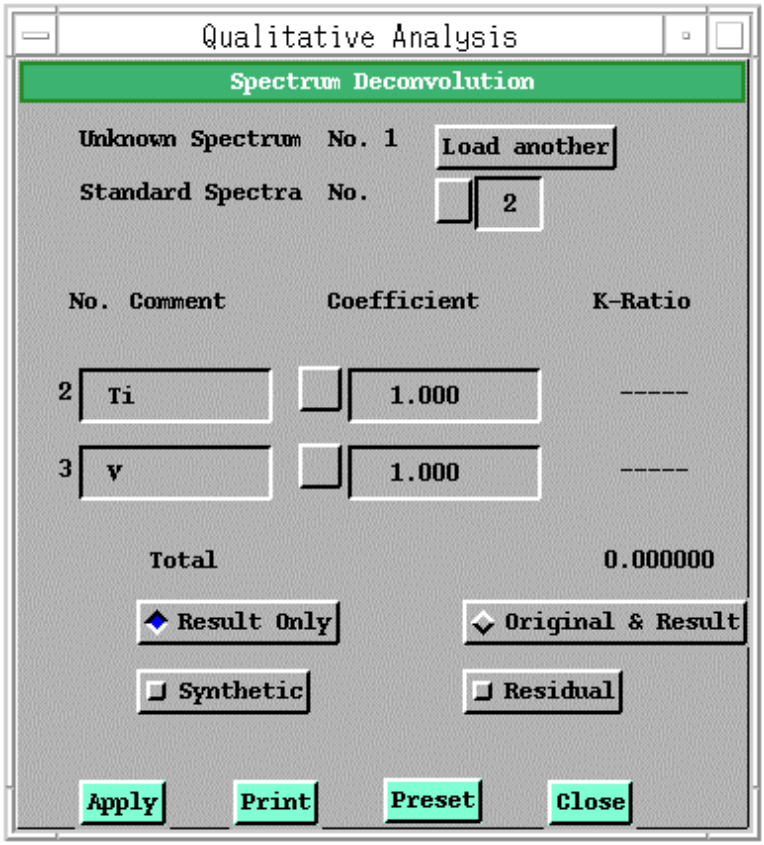


Fig. 3 Spectrum Deconvolution window

- Load another: The spectrum displayed at the top of the screen is the spectrum of the unknown sample. Clicking on the Load another button displays the sample selection window and allows you to replace the spectrum of the unknown sample with another sample.
- No. of spectra: Number of standard sample spectra.
- Comment: Comment entry area. Up to eight characters are enterable.
- Coefficient: An entry coefficient to adjust the resulted k-Ratio values. Normally, the concentration of the element in a standard sample is used. Enter 1.0 for a pure element sample.
- Synthetic: Displays a synthesized spectrum.
- Residual: Displays a residual from the synthesized spectrum.
- Apply: Clicking on the Apply button starts calculation of waveform deconvolution, and displays the mixing ratio of the standard sample spectrum to the unknown sample spectra.

- Print: Clicking on the Print button outputs the results after waveform deconvolution calculation to the printer. “Chi-Square” in the printout example shown in Fig. 5 denotes the square value of χ (chi). The closer to 1 the value, the better is the effect of fitting by the least square method.
- Preset: Used to carry out continuous spectrum deconvolution of multiple unknown samples.
- Variance: Dispersion of the k-Ratio variance calculated from the measured data. The closer to 0 the value, the higher is the reliability on the k-Ratio.

A fundamental concept on waveform deconvolution is described below.

If unknown sample spectrum f(u) is composed of overlapping standard sample spectra f(a) and f(b) of elements a and b, the relationship between these spectra is given by Equation (1).

$$f(u) = Ra * f(a) + Rb * f(b) \quad \text{..... (1)}$$

where Ra and Rb are given as a ratio of spectra f(a) and f(b) in unknown sample spectrum f(u) and calculated by the top hat filtering and the least square method.
Assuming that the concentrations of elements a and b in the standard sample are C_a^{STD} and C_b^{STD}, we obtain those in the unknown sample as Equation (2).

$$Ka = C_a^{STD} * Ra, Kb = C_b^{STD} * Rb \quad \text{..... (2)}$$

Ci and Ki (i = a, b) in Equation (2) represent the Coefficient and k-Ratio in the Spectrum Deconvolution window, respectively (refer to Fig. 3). Even when f(u) is composed of more than three spectra, the same relationship holds.

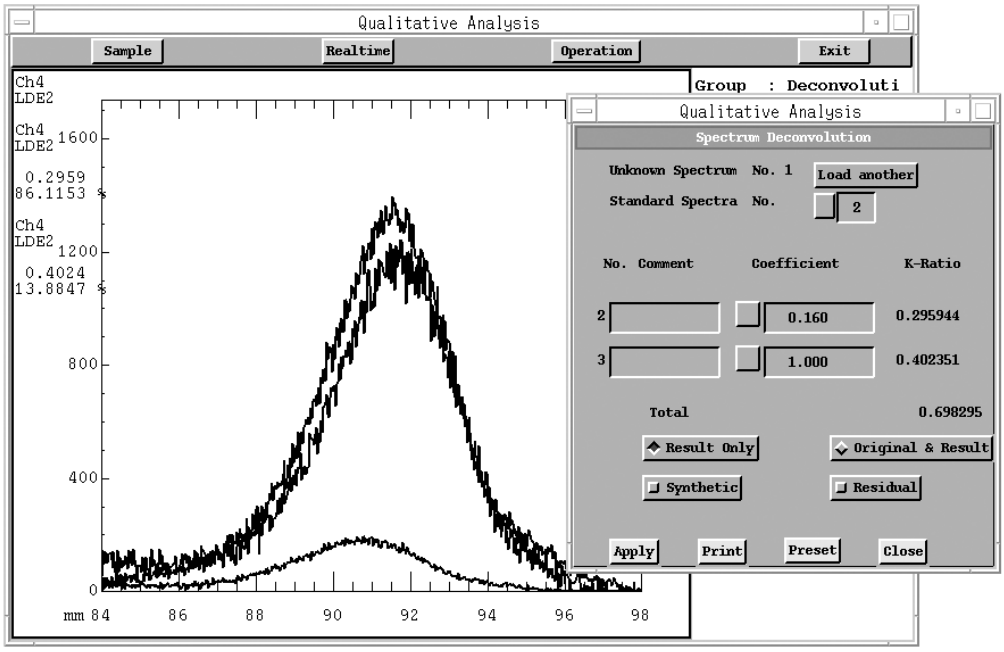


Fig. 4 An example of waveform deconvolution

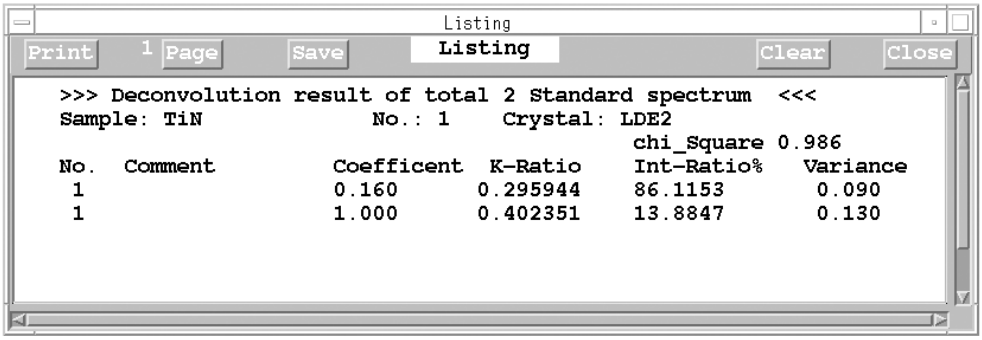


Fig. 5 A printout example

After waveform deconvolution, the spectra of standard samples are redisplayed in accordance with the mixing ratio. To carry out waveform deconvolution after changing the Coefficient, start with spectral display again.

Figs. 1, 2 and 4 show examples of waveform deconvolution of N-Kα and Ti-Ll spectra whose peaks overlap each other due to their adjacent peak positions. An unknown sample which are considered similar to TiN in composition was analyzed using 16%-N-containing vanadium nitrides (VN) and metal (Ti) as standard samples. In Fig. 1, the spectrum of the unknown sample is shown on the top, the N-Kα spectrum of VN in the middle, and the Ti-Ll spectrum of Ti at the bottom. Figs. 2 and 4 each show the unknown sample spectrum, N-Kα spectrum, and Ti-Ll spectrum which are displayed in the sequence of their peak intensity.