

A Standards-Based Method to Determine Elemental Compositions using Energy Dispersive X-Ray Spectrometry for Compound Semiconductors

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Energy-dispersive X-ray spectrometry (EDX) is a widely used analytical technique in TEM mode for rapid identification of elemental composition at nanometer-scale spatial resolution [1]. The most popular, standard-based approach is the Cliff-Lorimer method, which relates composition ratios to intensity ratios via “*k*-factors”. An alternative approach is the “zeta-factor” (ζ -factor) approach introduced by Watanabe [2], which is sophisticated extension of the Cliff-Lorimer method. The ζ factors can be determined provided if the beam current, elemental compositions and foil thickness of specimen are known. However, measurement of foil thickness using TEM is not straightforward. This limits the application of this method in calibrating the composition of III-V materials.

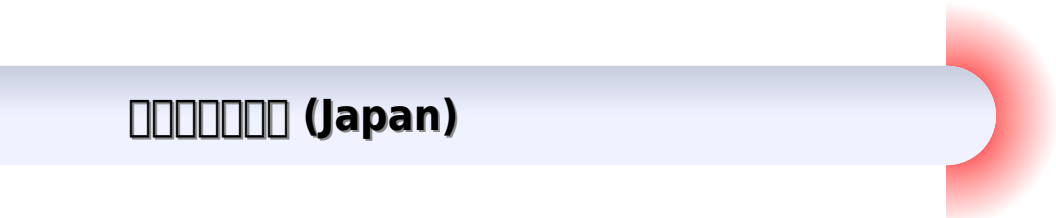
The method presented here is a standards-based modification and generalization of the ζ -factor method. The measured X-ray intensity at energy E can be expressed as:

$$I(E) = \alpha(E) \cdot n \cdot D_e \cdot T \cdot A(T, E) \cdot \sum_k w_k \cdot g_k(E)$$

where $\alpha(E)$ contains factors for detector solid angle and efficiency, D_e is the total electron dose, $A(T, E)$ is the absorption correction, and T is the foil thickness. The sum is over constituent elements k , with atomic fractions, w_k , and elemental, spectral generation rates, $g_k(E)$, which are convoluted with the detector response function. The method begins with a discrete set of related reference standards of known composition, applies multivariate statistical analysis to those spectra, and evaluates the compositions with a linear matrix algebra method to relate the spectra to elemental composition. The method requires no peak fitting and provides good accuracy in determining compound semiconductor alloy compositions. The composition is expressed as a vector $\vec{w}$ of dimension N_e . The absorption-corrected intensity $d(E) = I(E)/A(T, E)$ consists of a discrete set of N_e intensity data points (typically 4096), which can be written as a vector $\vec{d}$. A set of reference spectra from materials within the compositional space of interest are collected into a data matrix $\mathbf{D}$ of dimensions $N_e \times N_r$, which can be written as a product of $\mathbf{R}$ (row) and $\mathbf{C}$ (column) matrices $\mathbf{D} = \mathbf{R} \cdot \mathbf{C}$. Furthermore, PCA readily allows *compression* of these matrices by eliminating the factors with low significance, typically associated with noise. The factor loadings for an arbitrary test spectrum can be found with $\vec{c} = \mathbf{R}^L \cdot \vec{d}$ where $\mathbf{R}^L = (\mathbf{R}^T \cdot \mathbf{R})^{-1} \cdot \mathbf{R}^T$ is the left pseudoinverse of $\mathbf{R}$. The scaled composition vector $\vec{y} = n \cdot D_e \cdot T \cdot \vec{w}$ can be directly related to $\vec{y} = \mathbf{Z} \cdot \vec{c}$. Imposing the normalization condition $\sum_k w_k = 1$, the squared difference between calculated and known compositions can be minimized with respect to the components of $\mathbf{Z}$ within an overall scaling factor. This allows the $\vec{w}$ for all reference spectra. By considering one or more reference spectra of known thickness, the constant of proportionality β is found by minimizing the squared difference between $\vec{y}^{(r)}$ and $\beta \cdot \mathbf{Z} \cdot \vec{c}^{(r)}$. We then estimate the thickness for each reference spectrum, adjust the absorption

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