

Development of an Automated Phase Analysis Procedure for Multi-Component Samples in EPMA

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EPMA systems, with their high analytical accuracy, are ideal for the identification of intermetallic compounds, minerals, and other phases. In the past, phases were differentiated using a scatter diagram analysis on elemental map data. An intermetallic compound or a mineral can be recognizable as a cluster of compositional data points in the corresponding scatter diagram. The phase analysis program, previously used by EPMA, produced a phase map by plotting each pixel on the map, using a specific color for each cluster.

When using scatter diagram analysis for samples with multiple elements, choosing which elements to include in the analysis is of critical importance, as is the actual selection of the individual phases on the scatter plot. Our automated phase analysis system, Phase MAP Maker, automates these two operations using multivariate analysis.

For multi-element maps, the program uses principal component analysis to produce a score plot of the first and second main components of the sample. Cluster analysis of this score plot is then undertaken. Each cluster on this score plot represents a different phase. However, this principal component analysis method has some faults. Since it is a two-dimensional score plot, it is not really suited for a map area with multi-elements. Moreover, the first and second main components may not be the most suitable elements for defining all of the phases. Phase MAP Maker was designed to address these issues, to work with samples involving many elements, and to make the entire operation easy. The program automatically produces a scatter diagram of every combination of element pairs. The order that the diagrams are displayed is determined from the results of principal component analysis. As such, correlation plots for all the elements can be quickly scanned through, from the most important components to the least important components.

When performing the operation manually, only one scatter diagram is used. The scatter plot with the elements identified as the first and second main components of the system is automatically selected for manual analysis. This scatter diagram classifies the phases by color from the previous cluster analysis. The phase map, using the same colors defined in the scatter diagram, is then also displayed.

An example of the data processing is shown below, for a ceramic sample containing Fe, Al, Zr, Sn, Si, Ca and O. Figure 1a displays the elemental map data for the ceramic. By processing the map data with the Phase MAP Maker program, the results shown in Figures 1b and 2 are obtained automatically. Before the phase map can be generated, one needs to determine which elements represent the principle independent components. The program automatically displays all scatter diagram combinations, the recommended scatter diagrams to use, and the phase cluster delineations.

The Zr-Si diagram is among the scatter diagrams shown in Figure 1b. This scatter diagram differentiates a fourth phase not seen on most of the other diagrams. This is because of the Zr component. Though it is a minor element, it stabilizes an additional phase. Manual operation of this scatter diagram was carried out, and the additional phase was added to the phase map.

Utilizing this automated phase analysis program, complicated EPMA map data can be made more intelligible and can make a quick interpretation possible.

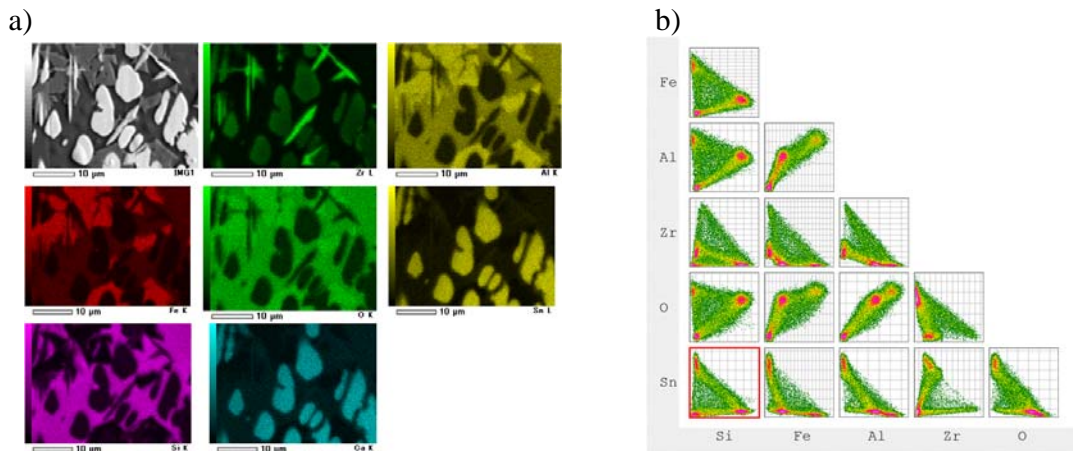


Figure 1. The element maps of a ceramic compound (a), and the scatter diagram automatically produced by the Phase Map Maker program (b).

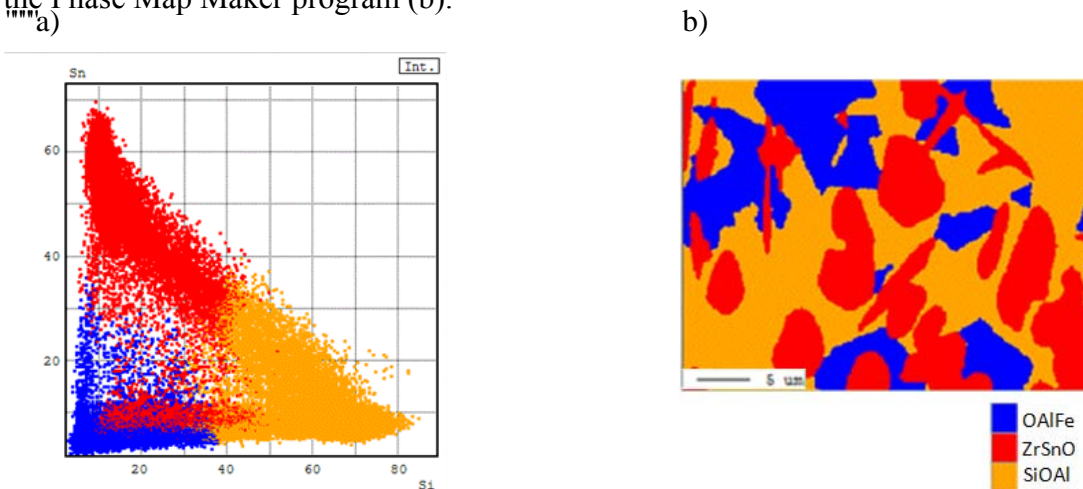


Figure 2. The phases were classified by color from principal component analysis and cluster analysis (a) and displayed on the phase map (b).