

Using Principal Component Analysis on Compositional Measurements of Multi-component Pu-alloys to identify potential minor phases

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Identifying and measuring the composition of minor phases in multi-component Pu-alloy is an important part of understanding the phase equilibria behavior and material properties of these alloys. Using wavelength-dispersive X-ray spectrometry (WDS) on the electron microprobe (EPMA) results in precise quantitative compositional data with a high spatial resolution (~ 1 micron), making it an ideal technique for this application. Analyzing the composition of small (< 5 microns) minor phases in various plutonium alloy compositions is a difficult task however, for reasons related to the generation and detection of characteristic X-rays using a focused electron beam. First, the excitation volume of the characteristic X-rays of interest can exceed the size of the grain, while X-rays from relatively lighter elements can be excited within the grain by heavier, higher energy X-rays coming from outside the grain (fluorescence), both of which lead to spurious signals that are difficult to quantify. There may also be peak interferences in the minor phases that are not present in the bulk material due to differing compositions and are therefore not accounted for. Short counting times (commonly used for broad area map surveys due to practical analysis time considerations) lead to poor counting statistics and detection sensitivity that can make phase identification difficult. Each of these difficulties can be overcome however with a dedicated analytical campaign designed specifically for a minor phase that has a particular composition. But this can be very resource intensive approach, particularly when the identities of the target phases are unknown.

We present here the results of applying principal component analysis on quantitative WDS map data that identifies correlations of the elements across varying bulk alloy compositions. This statistical method leverages the large number of data points in a map to overcome the difficulties listed above, and to point to the potential compositions and identity of minor phases present within the sample. This information can then be used to design a targeted analytical procedure that minimizes instrument time and analyst effort.