Elemental Mapping with SDD-EDS: Solving the Hard Problems

Dale E. Newbury and Nicholas W. M. Ritchie

National Institute of Standards and Technology, Gaithersburg, MD 20899-8370

Elemental mapping has been one of the most powerful and popular modes of x-ray microanalysis throughout the history of electron-excited x-ray spectrometry in the scanning electron microscope (SEM) [1]. Mapping was first possible with wavelength dispersive spectrometry (WDS) and later with the introduction of the Si(Li)-EDS. The advantages of higher spectral resolution and x-ray throughput of WDS compared to Si(Li)-EDS have made WDS the obvious choice for elemental mapping when difficult x-ray spectrometry situations exist, such as mapping elements with interfering peaks and mapping minor ($0.01 \leq C \leq 0.1$) and trace ($C < 0.01$) constituents, where C is the concentration (mass fraction). The emergence of the silicon drift detector (SDD)-EDS with its greatly increased throughput, a factor of 50 or more for the same spectral resolution compared to Si(Li)-EDS, has substantially changed this situation [2]. In fact, the geometric and quantum efficiencies of SDD-EDS offer significant advantages over WDS when mapping beam-sensitive samples. The SDD-EDS x-ray throughput, which can exceed 1 MHz at 145 eV for a four-detector SDD array, enables new capabilities for attacking the difficult mapping problems that have historically been the exclusive domain of WDS. While spectral resolution is important in mapping, the total accumulated x-ray count per unit electron dose and per unit of real time is often a critical factor. EDS mapping in the x-ray spectrum imaging (XSI) mode also has the advantage of recording the entire spectrum at each image pixel, enabling the discovery of unexpected constituents which can elude WDS. Mapping minor and trace constituents demands careful attention to correction of the spectral background because of the impact of the compositional dependence of the x-ray continuum upon low intensity x-ray peaks. Background correction can be directly calculated from the full spectral information embedded in the XSI, while operation with a single WDS requires time-consuming sequential peak and background scans, or when multiple WDS are available, one WDS can be devoted to background [3]. Figure 1 shows an example of the correction of background to remove a continuum artifact in the mapping of a minor (C ~ 0.04) Fe constituent in Al-Ni (Raney nickel), where the uncorrected image shows apparent Fe contrast that actually arises from changes in the major Al and Ni concentrations. More challenging for EDS is the case of severe spectral interference, such as that encountered in mapping Pb-Cd-Sn solder, where the uncorrected contribution from CdL$\beta_{1,2}$ in the SnLα peak region creates a contrast artifact, as shown in Figure 2 (note regions marked "1" in (b) and (c)). When the Sn is viewed as a concentration image after the XSI is processed with multiple linear least squares peak fitting in NIST DTSA-II, this contrast artifact is removed, correctly revealing the trace level solubility of Sn in Cd [4].

References
[1] J. Goldstein et al., *Scanning Electron Microscopy and X-ray Microanalysis*, 3rd ed. (Springer, New York, 2003) 485.
[2] D. Newbury, Microsc. Microanal. 12 (2006) 26
[3] R. Myklebust, D. Newbury, and R. Marinenko, Anal. Chem., 61 (1989) 1612.
[4] N. Ritchie, Microsc. Today, (19) 2011 26-31; DTSA-II is available for free download at www.cstl.nist.gov/div837/837.02/epq/dtsa2/index.html
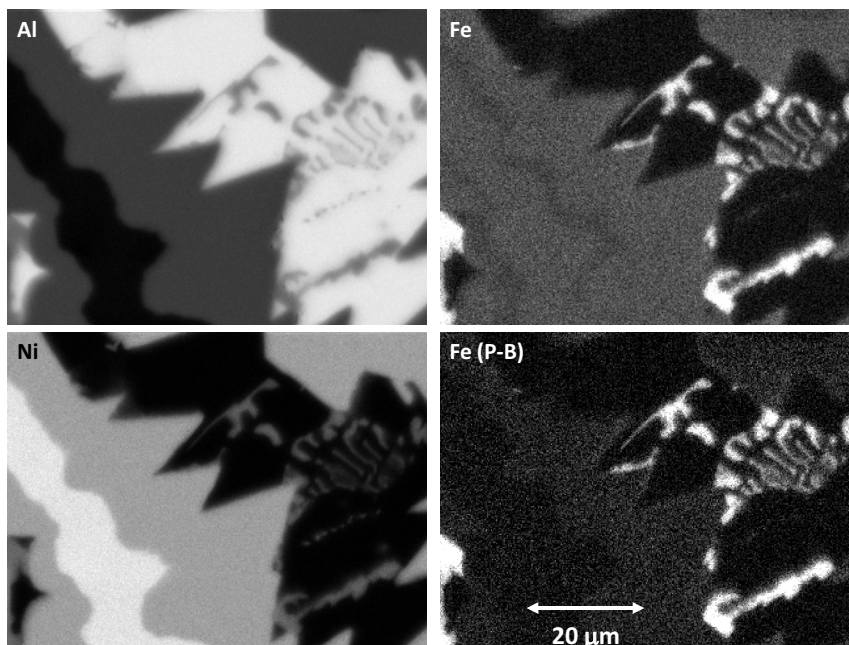
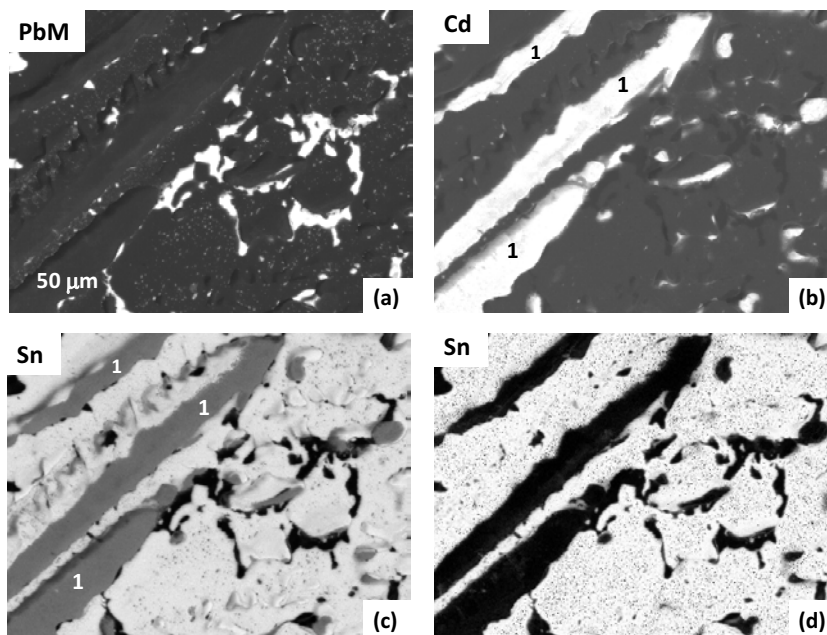Figure 1 SDD-EDS XSI: raw, uncorrected and background corrected Fe maps in Raney nickel.

Figure 2 PbCdSn solder: intensity maps for (a) PbM; (b) CdL; (c) SnL; (d)SnL compositional map.