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# X-ray Analysis in the AEM with Angstrom-level Spatial Resolution and Single-Atom Detection

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In 1977 Joe Goldstein was the senior author on a paper entitled "Quantitative X-ray Analysis in the Electron Microscope" in which he outlined the principles of quantification of X-ray spectra, the absorption correction and, in collaboration with Stephen Reed, defined the single-scattering approximation limiting the spatial resolution of thin-foil analysis [1]. This paper was the driving force behind subsequent attempts to improve quantification and push the spatial resolution limit to the best possible levels. Over the last 30 years technological developments including intermediate voltage field emission gun AEMs, increased X-ray detector collection angles, computerized processing of noisy X-ray spectra and spherical aberration correction have all combined to make the possibility of atomic-level analysis a reality, as described below.

The crucial problem, common to all analysis techniques, is that as the spatial resolution improves, the analysis volume (and hence the generated signal quality) is degraded, so the spatial and detection limits compete with one another, as shown in figure 1 [2]. This figure is a modified version of a diagram from Lyman [3], which summarizes succinctly the 25-year progress in marrying these competing goals. All the instrument improvements listed above combine to either improve spatial resolution (e.g. smaller probe) or increase X-ray signal generation and collection efficiency (e.g. increased gun brightness, probe current) thus enhancing detection limits. A necessary corollary to all these improvements has been the development of AEMs with mechanical and electronic stability sufficient to permit atomic-level resolution to be maintained over the time necessary to accumulate X-ray spectra with sufficient counts for quantitative analysis. The progress to date is best shown in the X-ray maps in figure 2, which are the first X-ray maps with nanometer-level resolution in a Cs-corrected 300 kV VG HB 603. These fully quantitative maps still show nm-level instability during the ~3500s required to accumulate the maps at ~200 ms/pixel. Nevertheless, they represent a significant improvement from the qualitative dot maps with nanometer resolution first achieved via 100 kV VG 501 FEG STEMs almost 20 years ago and the quantitative maps with resolution levels of a few nanometers first achieved on the same VG HB 603 10 years ago (but without aberration correction) (e.g. [2]). Key elements in this progression reflect the initial impetus from Joe Goldstein's seminal paper that, in order to improve spatial resolution and detection limits, the main factors were higher kV and smaller probe sizes while maintaining high probe currents.

## References

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[3] C.E. Lyman, Physical Aspects of Microscopic Characterization of Materials (1987) Scanning Microscopy International, Suppl. **1**, 123.
[4] Research supported through NSF DMR-0304738 and Bechtel Bettis Laboratories

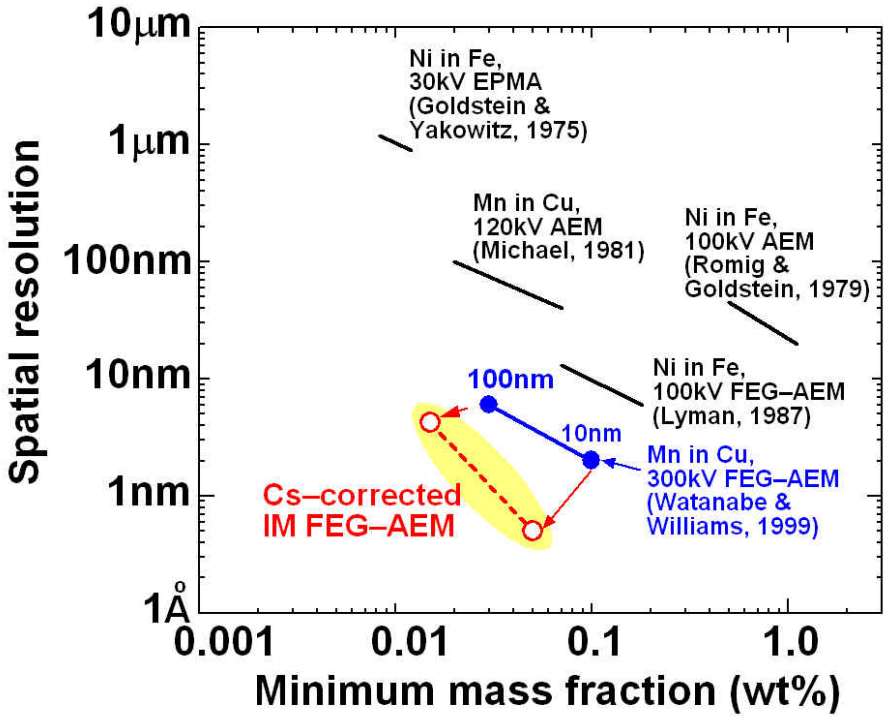


*Fig. 1.* A summary of the relationship between the spatial resolution and the minimum mass fraction for X-ray analysis in several electron-probe instruments (modified from Lyman [3] and Williams et al. [2]). The shadowed area represents the potential of the Cs-corrected, intermediate-voltage FEG-AEM.

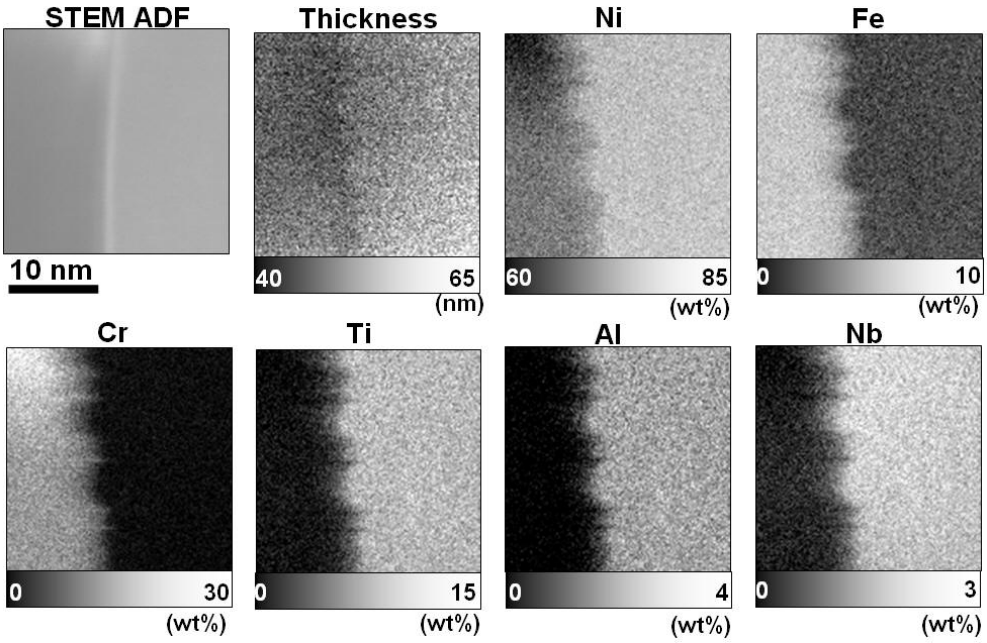


*Fig. 2.* The first quantified X-ray maps taken from a γ/γ' interface in a Ni-base superalloy by the Lehigh Cs-corrected VG HB 603 STEM.

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