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ROCKING-BEAM SPECTRUM IMAGES AND ALCHEMI OF $Ni_{50}Al_{40}Fe_{10}$

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A spectrum image can be acquired with a scanning transmission electron microscope (STEM) by translating a focused probe over a two-dimensional area of a specimen and acquiring a spectrum at an array of positions of the probe. Analogously, a rocking-beam spectrum image is formed when the incident electron beam is tilted relative to the specimen and a series of spectra is acquired from a single volume of the specimen. The orientation-dependence of such a series of spectra can be used to extract the atomic site-distributions of an ordered alloy or compound. One of the drawbacks of spectrum image acquisition is that the raw images are typically files of many megabytes, whereas the actual information of interest may be reducible to a fraction of this size. The dimension of the data set may be reduced by a number of methods. For example, Rossouw and coworkers have acquired rocking-beam X-ray maps for the characterization of atomic site-distributions in titanium aluminide intermetallic alloys.[1] These maps were formed by integrating the X-ray intensities in energy windows around characteristic X-ray peaks of the host and alloying elements composing the crystal. Such a method amounts to an *a priori* reduction of the size of the data. An alternative method for dimensional reduction of the data is multivariate statistical analysis of the series of spectra.[2] However, this method is computationally intensive and at present impractical for large spectrum images.[3] An intermediate approach is provided by summing spectra of similar orientation (θ/θ_B) relative to the systematic row or zone axis that exhibits site-discrimination. Such an *a posteriori* reduction of the data allows the analyst to choose a series of orientations that avoid nonsystematic low-order Kikuchi bands and average the effects of higher-index nonsystematic reflections.

A rocking-beam energy-dispersive X-ray (EDX) spectrum image was acquired near the [035] zone axis of a B2-ordered alloy of composition $Ni_{50}Al_{40}Fe_{10}$. The data were acquired with a Philips CM200FEG, equipped with an Oxford Super-ATW detector with XP3 pulse processor and an EMiSPEC Vision integrated acquisition system. The spectrum image was composed of 70 × 70 spectra × 2048 channels per spectrum. The dwell time per spectrum was 1 s, and the ~20 megabyte spectrum image was acquired in about 1.5 h. Images comparable to those acquired by Rossouw et al. were formed *a posteriori* by integrating the X-ray intensities in windows enclosing the Al-K, Fe-K_α, and Ni-K_α characteristic X-ray peaks for each pixel of the spectrum image. These images are shown in Fig. 1, along with a bright-field transmission channeling pattern (TCP), which records the signal from the bright-field STEM detector as the incident beam direction is varied with the beam-tilt coils, and an EDX spectrum from one pixel of the image. The range of orientations from which the spectrum image was acquired is indicated by the square superimposed on the TCP. The gray levels composing the X-ray maps were adjusted such that black corresponds to zero intensity and white corresponds to the most intense pixel of the image. Note that only the lowest order Kikuchi lines in the TCP can be discerned in the X-ray maps. At the [035] zone axis, the X-ray maps are dominated by the (200) Kikuchi band. The Ni and Al X-ray maps have opposite contrast in the (200) band, with the Ni map having a higher intensity at the center of the band ($\theta < \theta_{100}$) than in the side bands ($\theta_{100} < \theta < \theta_{200}$), and *vice versa* for the Al map. The greater similarity of the Fe X-ray map to the Al X-ray map indicates that the Fe alloying element predominantly occupies the 'Al'-sublattice in this alloy.

ALCHEMI (atom-location by channeling-enhanced microanalysis) was performed on a subset of the spectrum image using standard methods.[4] Spectra from a series of ~30 pixels along lines parallel to the (200) band were summed at each of 31 orientations relative to the band in the range $0 \leq \theta / \theta_{200} \leq 2.3$. Characteristic X-ray intensities of the K-shell X-rays of Ni, Fe, and Al were extracted from the 31 summed spectra with the simplex fitting procedure of the DTSA spectral analysis software. The fraction of Fe on the 'Ni'-site from this analysis, $p_{Fe'Ni'} = 23.8 \pm 2.1\%$, is in excellent agreement with $p_{Fe'Ni'} = 23.7 \pm 0.9\%$, which was determined by an analysis of a series of ten spectra acquired at orientations of the crystal carefully chosen so that the contributions of nonsystematic reflections are negligible.[5] The standard error of the present analysis is larger than that expected from Poisson counting statistics alone and more likely results from the lack of dead-time correction of the acquired spectra. The resulting count-rate dependence of the spectra would likely degrade the planar fit of the data and hence the standard error on the correlation coefficients.[6,7]

References

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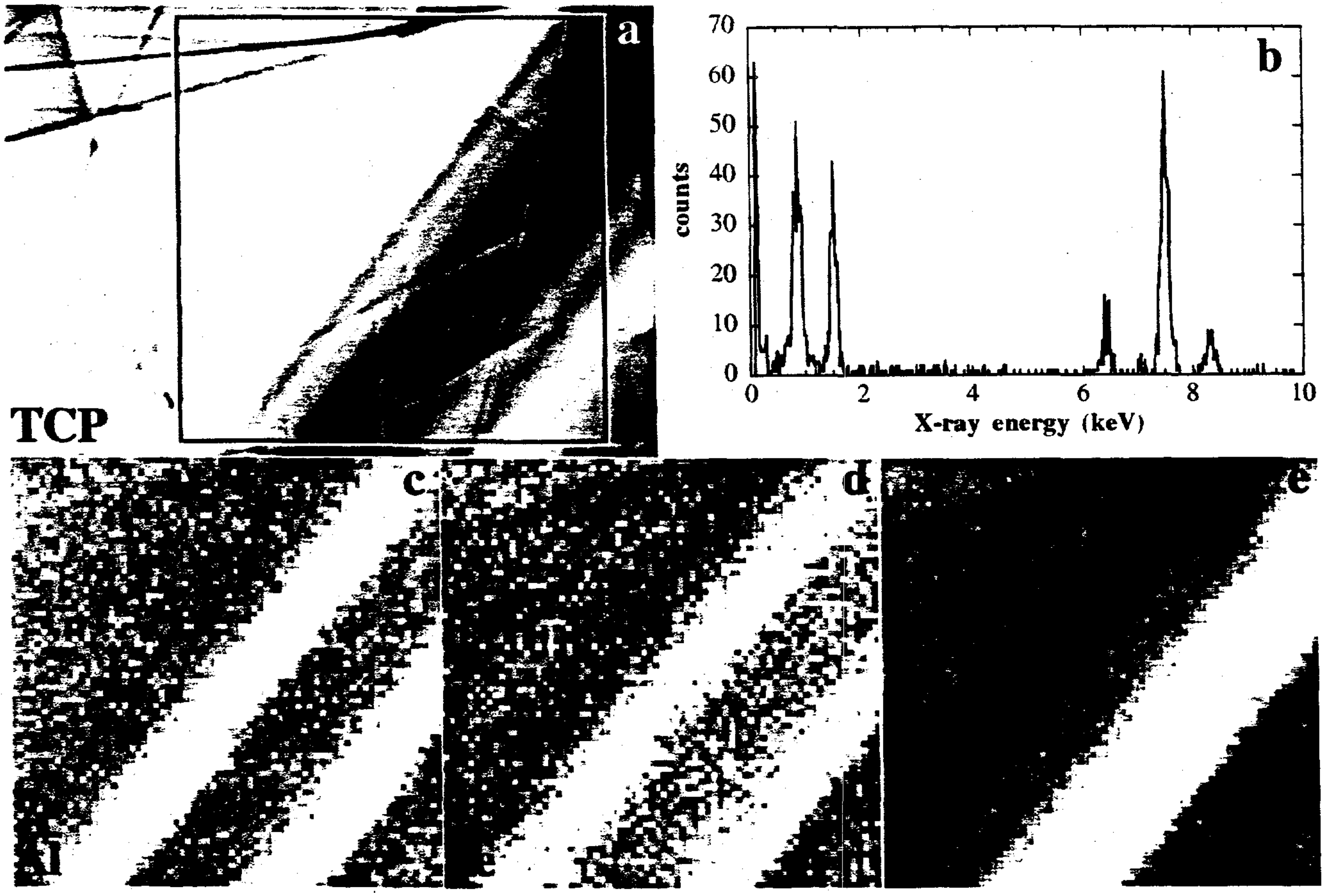

FIG 1 - Rocking-beam spectrum image acquired near [035] zone axis of B2-ordered Ni$_{50}$Al$_{40}$Fe$_{10}$. (a) Bright-field TCP with square showing area from which 70 × 70 spectrum image was acquired; (b) EDX spectrum from one pixel of the spectrum image; (c-e) X-ray maps corresponding to windows around the (c) Al-K, (d) Fe-K$_\alpha$, and (e) Ni-K$_\alpha$ peaks.

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