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ELEMENTAL MAPPING OF SEGREGATION IN STAINLESS STEELS WITH AN IMAGING FILTER

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1. INTRODUCTION

Elemental mapping in a TEM equipped with an imaging filter (Gubbens and Krivanek 1993, Mayer 1992) is expected to become an important microanalysis technique in materials science. The efficacy of the technique for studying solute segregation and composition gradients is not as well established as for mapping precipitate distributions. We have therefore undertaken a preliminary investigation of two types of solute segregation in 300-series austenitic stainless steels. Elemental mapping was performed at 200 kV with Gatan Imaging Filters (GIFs) attached to JEM 2010 and Philips CM20 electron microscopes operating with probe currents of > 100 nA. Further work will also use a GIF on a Philips CM30 at ORNL. Images were recorded (5-50 s) with the slow scan CCD camera of the filter for 30- or 40-eV-wide windows before and after ionization edges of interest, including Cr-, Fe-, and Ni-L₂₃ edges (Fig. 1), and were processed with Digital Micrograph software. Core-loss signal and jump-ratio elemental "maps" were produced by AE^r background extrapolation from two pre-edge windows (Egerton 1986) and by simple division of the post-edge image by a pre-edge one (Krivanek 1993), respectively, as illustrated in Fig. 2.

2. RESULTS

Elemental maps of Cr, Fe, and Ni were obtained for an edge-on grain boundary in a type 304L stainless steel sensitized by aging 15 h at 600°C, where Cr depletion occurs between intergranular M₂₃C₆ chromium carbides (Kenik 1992). Figure 3a shows a Cr-signal image and Figs. 3b and c show intensity profiles corresponding to the traces marked in Fig. 3a. Equivalent data for the Cr jump-ratio are shown in Figs. 3d-f. The M₂₃C₆ particles and the Cr depletion at the grain boundary are clearly visible in both elemental maps. Quantitative compositional data are easily obtained at resolutions approaching 1 nm.

In modified type 316 stainless steel (LS1A) irradiated with 4 MeV Ni ions to 1 displacement per atom (dpa) at 675°C, radiation-induced segregation (RIS) occurs at grain boundaries and dislocations due to coupling of solutes with the point-defect fluxes to sinks (Kenik 1986). Figure 4a shows an elastic bright-field image of a nearly edge-on Frank dislocation loop that intersects the two foil surfaces; the weak diffraction contrast has been enhanced by image processing. Figure 4b shows weak Cr depletion due to RIS at the loop. The expected Ni enrichment was less clearly revealed because of the much lower signal/background ratio (see Fig. 1).

3. CONCLUSIONS

These preliminary results have shown that a GIF can be used to study solute segregation in stainless steels, that image acquisition and processing are relatively straightforward and rapid, but that large probe currents and low specimen drift rates are essential. Further work is in progress on extracting quantitative compositions from jump-ratio images, on mapping of minor solutes, and on comparisons with spectrum images obtained with a FEG-STEM. Elemental mapping in a TEM

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should complement the more time-consuming spectrum imaging, which should have advantages for fine-structure mapping, complex compositions, and beam sensitive materials.

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Figure 1. (a) Typical energy-loss spectrum from stainless steel recorded with a GIF, (b) the corresponding intensity profile.

Figure 2. Procedures for producing elemental maps: core-loss signal, 3-(extrapolation from 1 and 2); jump ratio, 3/2 or 3/1.

Figure 3. Cr distribution at edge-on grain boundary in aged type 304L stainless steel. (a) Cr-signal, (b,c) intensity profiles (traces marked on 3a), (d) Cr jump-ratio image, (e,f) intensity profiles (traces marked on 3d).

Figure 4. LS1A irradiated to 1 dpa at 675°C (a) Elastic bright-field image (b) Cr-signal image.

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