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DOI: 10.1017/S1431927606065238
Microsc Microanal 12(Supp 2), 2006

Atomic-Scale Characterization of Metals and Alloys Using Spherical-Aberration Corrected Scanning Transmission Electron Microscopy

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Identification and quantification of the nature of individual atomic configurations are the ultimate goals for the characterization of advanced nanoscale materials. It is now possible to perform such ultimate characterization by scanning transmission electron microscopy (STEM) involving electron energy-loss spectrometry (EELS) and X-ray energy dispersive spectrometry (XEDS) especially with the recently developed spherical-aberration (Cs) correctors. The Cs corrector for STEM permits refinement of the electron-probe dimensions down to the atomic level while maintaining the same amount of current as in the uncorrected probe. Using Cs-corrected STEM, therefore, image resolution has already reached sub-Ångstrom levels in high-angle annular dark-field (HAADF) imaging [1] and EELS analysis can be performed on individual atomic columns [2]. For XEDS, not only the spatial resolution but also the sensitivity of analysis can also be improved in the Cs-corrected instruments [3]. At Lehigh University, two types of Cs corrected instruments are available: a newly developed JEOL JEM-2200FS (200 keV) STEM/TEM integrated with a CEOS Cs-corrector system and an in-column Ω energy-filter, and the VG HB 603 (300 keV) dedicated STEM with a Nion Cs corrector system and an Oxford INCA Si(Li) XEDS system. The former instrument has been optimized for EELS analysis in the atomic-resolution ADF-STEM imaging mode. The latter is being used primarily for high-resolution XEDS analysis. In this study, both the Cs-corrected instruments were applied to the characterization of a Ni-base superalloy, which contains $L1_2$-ordered γ' precipitates in a γ matrix.

A high-resolution, phase-contrast image taken from a γ/γ' interface by the JEM-2200FS in the TEM mode is shown in Fig. 1. The primary XEDS analysis using the HB 603 shows that the Ti composition in atomic % is twice as high as Al in the γ' precipitates [3]. However, the precipitates still maintain the Ni_3Al- type $L1_2$ structure rather than the Ni_3Ti-type $D0_{24}$ structure. Figure 2 shows an atomic-resolution ADF-STEM image recorded from the same interface. In the (100) projection of the alloy, the atomic spacing is ~1.8 Å, which is clearly resolved in the ADF-STEM image as shown in Fig. 2. Furthermore, no local shift in atomic configuration was observed across the interface, although the contrast corresponding to the atomic-column positions clearly changes. From the ADF-STEM image, the γ' precipitates have a coherent interface with the matrix γ phase. Additionally, two types of major spots can be seen in the γ' precipitate region of the image. The brighter and darker spots correspond to face-centered (A site) and corner (B site) atom positions in the $L1_2$ structure, respectively. Figure 3 shows two EELS spectra around the Ti $L_{2,3}$ edge (~450 eV) recorded from the A (a) and B (b) sites, respectively. It is clear that the Ti $L_{2,3}$ edge intensity is much higher in the spectrum from the B site. These results suggest that the Ti atoms occupy preferentially the B-site, which agrees with previous studies using the ALCHEMI method via X-rays [4]. Using Cs-corrected STEMs, therefore, such site occupancies of alloying elements or even impurity elements can be determined directly at the individual atomic-column level, not by averaging information from broad regions as traditionally carried out using ALCHEMI. Characterization on the atomic column-by-atomic column scale can now, at

last, be achieved using state-of-the-art STEMs with Cs correctors.

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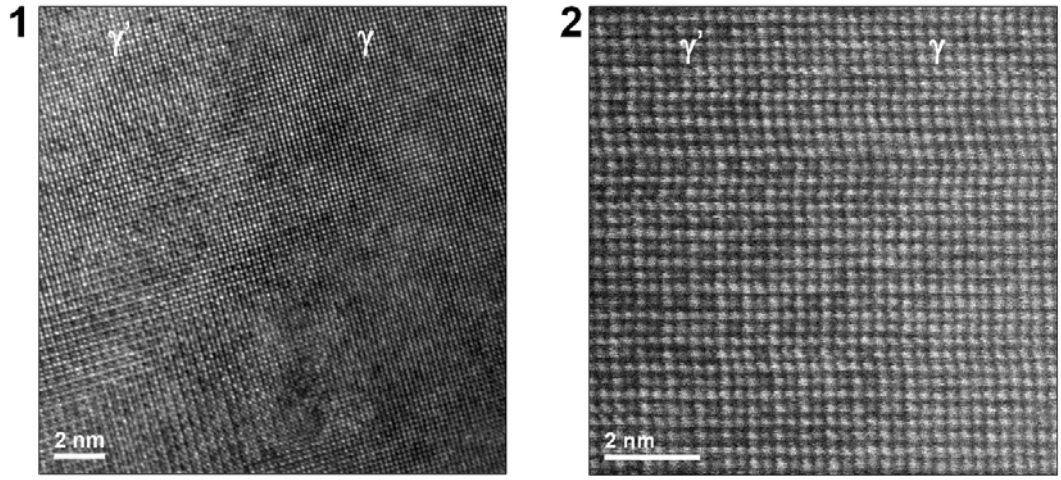

Fig. 1 A high-resolution, phase-contrast image taken from a γ/γ' interface by the JEM-2200FS in the TEM mode.
Fig. 2 A high-resolution HA-ADF image taken from a γ/γ' interface by the JEM-2200FS in the STEM mode.

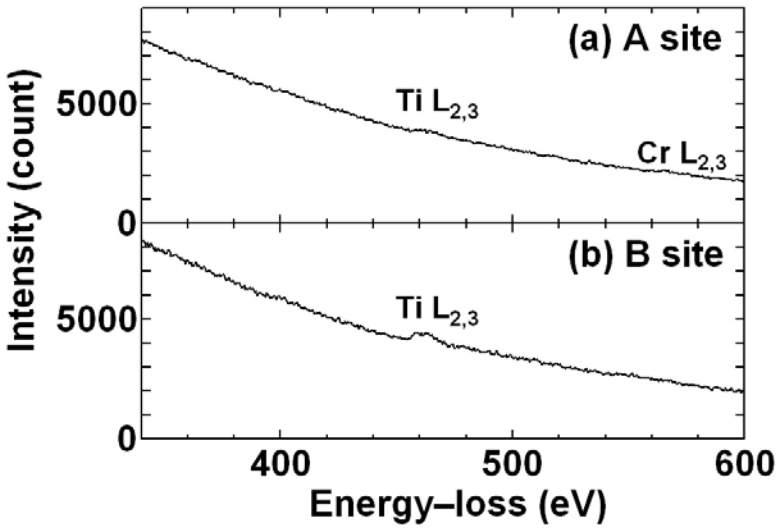

Fig. 3 EELS spectra taken from the A (a) and B (b) site positions in the γ' phase region by JEM-2200FS in the STEM mode.

https://doi.org/10.1017/S1431927606065238 Published online by Cambridge University Press