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Multiple Analytical Instrumentation for Complete Materials Characterization

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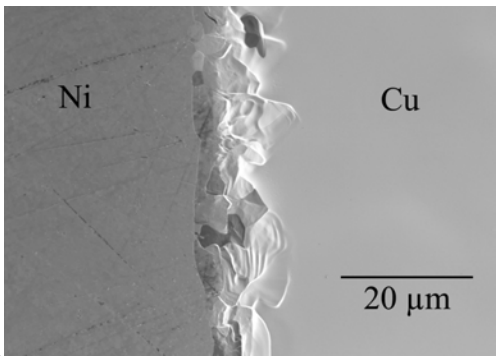
In many instances it is necessary to use multiple analytical characterization tools to obtain complementary information so that an entire materials problem may be understood [1]. Analytical instruments use different types of probes with varying spatial resolution and differences in mass resolution. Combining results from a focused ion beam (FIB) instrument, (scanning) transmission electron microscopy ((S)TEM) imaging, X-ray energy dispersive spectrometry (XEDS) and secondary ion mass spectrometry (SIMS) provides a range of imaging and elemental information. Use of these techniques enabled an understanding of the influence of grain boundary segregation on grain boundary diffusion in the Cu-Ni-Bi system.

FIGS. 1 and 2 are ion induced secondary electron FIB images of a Cu-Ni couple that was diffusion annealed at 600°C for 120 hours. Note that FIG. 1 shows the presence of diffusion induced recrystallization (DIR) at the interface, while FIG. 2 shows regions where DIR did and did not take place [2]. As interdiffusion studies are usually performed across flat polished interfaces, the observation of DIR vs. non-DIR regions was made possible using the ion channeling contrast that can be obtained with FIB imaging. Subsequent TEM specimen preparation of these DIR and non-DIR regions performed via FIB in-situ lift-out [3] enabled us to determine volume diffusivities for the Cu-Ni system via STEM/XEDS techniques [2].

FIG. 3 shows a SIMS profile of the concentration of Ni through a Cu $\Sigma 5/[100]$ twist boundary annealed at 650^{o}C for 200 hours [4]. The SIMS profiles were then calibrated (for specifics, see ref. 4) which provided the grain boundary contribution of the Ni diffusion through the Cu. The combination of the SIMS data in conjunction with the FIB/STEM/XEDS results was necessary to quantify the grain boundary diffusion of Ni through specific Cu bicrystals as shown in FIG. 4 [4].

FIG. 5 shows a high angle annular dark field STEM image of a Cu $45^{o}/[100]$ twist boundary showing Bi segregation at the boundary [5]. Note the bright band of contrast at the grain boundary that is indicative of the presence of the Bi. An XEDS Bi line profile across this interface shown in FIG. 6 yielded a maximum grain boundary coverage of 201 atoms/nm^2 for this boundary. Subsequent results on the effects of Ni diffusion down Bi-segregated Cu grain boundaries were also determined using the combined techniques discussed above [6,7].

In summary, multiple techniques were used to characterize volume diffusion, grain boundary diffusion, and grain boundary segregation effects on grain boundary diffusion in the Cu-Ni-Bi system. As shown above, complementary analytical techniques are often needed to fully characterize the microstructure, crystallography, and chemistry of a materials system [8].



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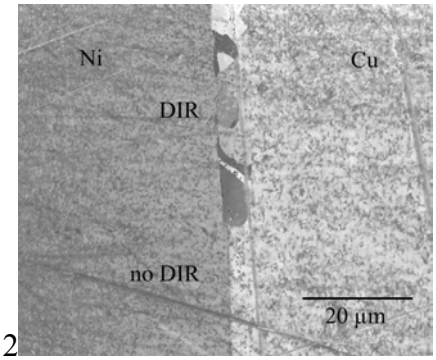

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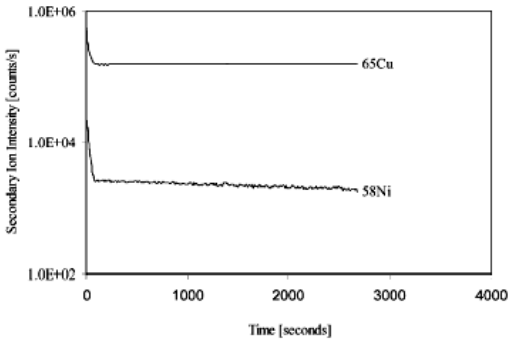

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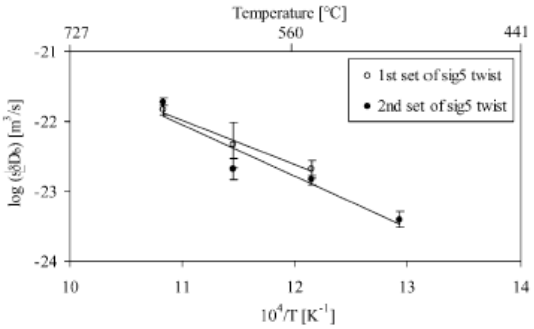

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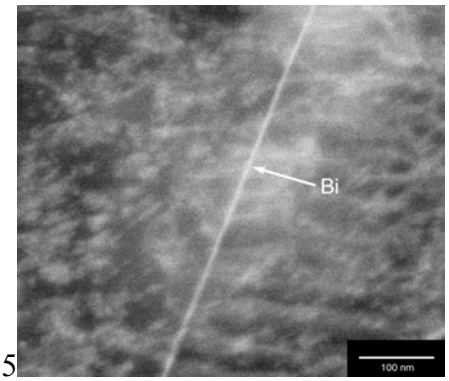

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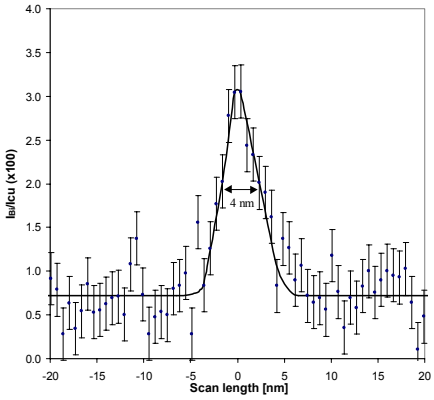

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FIG 1. FIB image of DIR region in a Cu-Ni diffusion couple [2].
FIG 2. FIB image showing DIR and non-DIR regions in a Cu-Ni diffusion couple [2].
FIG 3. SIMS profile of Ni through a Cu grain boundary [4].
FIG4. Arrhenius plot of diffusion of Ni through a Cu Σ5/[100] twist boundary [4].
FIG5. HAADF STEM image of Bi segregation to a Cu 45°/[100] twist boundary [5].
FIG6. XEDS line profile showing Bi concentration across the boundary in FIG 5 [6,7].

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