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APPLICATIONS OF THE ANALYTICAL ELECTRON MICROSCOPE TO MATERIALS SCIENCE

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In the last 20 years, the analytical electron microscope (AEM) has allowed investigators to obtain chemical and structural information from < 50 nm diameter regions in thin samples of materials and to explore problems where reactions occur at boundaries and interfaces or within small particles or phases in bulk samples. Examples of the application of the AEM to materials science problems are presented in this paper and demonstrate the usefulness and the future potential of the instrument.

The chemical composition of the specimen C_A, C_B .. can be calculated from the intensity I_A, I_B .. of elements A, B .. in the region of interest by using the ratio technique developed by Cliff and Lorimer [1] in which $C_A/C_B = k_{AB} I_A/I_B$, where the k_{AB} factor is a constant at a given operating voltage. The k_{AB} factor can be calculated but should be measured from a known standard if at all possible to increase the accuracy of the composition determination.

The measurement of reliable low temperature grain boundary and volume diffusion data has been limited by the spatial resolution of the instrumentation available. Because the AEM can be used to generate quantitative chemical analyses from diffusion couples or for grain boundary diffusion on a nanometer scale, diffusion coefficients four orders of magnitude lower than those obtained from the electron probe microanalyzer (EPMA) can be obtained. Figure 1 shows a TEM photomicrograph of a diffusion couple bond interface and the corresponding 2 micron long Ni compositional profile for a ternary austenite couple Fe-20Ni-P vs Fe-25Ni-P diffused at 6500C for 121 days. After the interdiffusion coefficients were measured, an order of magnitude increase in D Ni was observed at the same Ni

content due to the presence of P [2]. Figure 2 shows a symmetrical Cu composition profile around a stationary grain boundary in an Al-4 wt% Cu alloy aged at 2500C to produce θ precipitates on the boundary. Computer modelling of the solute profiles permits values of the grain boundary diffusion coefficient to be determined [3].

The major advantage of performing x-ray microanalysis in the AEM is the high compositional spatial resolution (R) and the elemental analysis sensitivity (MMF). The trade off between spatial resolution and MMF is shown in Figure 3 for measurements of Ni in an Fe-25 wt% Ni alloy [4]. The spatial resolution, R, approaches the beam diameter, d, in the thinnest specimens. The usefulness of the field emission gun (FEG) is clearly illustrated in Figure 3 where spatial resolution, R, can be improved by almost an order of magnitude from ~ 15 to ~ 1.8 nm for the same MMF and electron beam energy (100 keV). The MMF decreases (improves) continuously as the specimen thickness and spatial resolution increases. The best compromise in terms of improving x-ray spatial resolution and MMF is to use high operating voltage (300 to 400 kV), a FEG instrument, and thin specimens.

Figure 4 illustrates the type of spatial resolution available with present FEG instrumentation. A Ni composition profile across a planar 10 nm wide precipitate in the plessite region of the Grant iron meteorite is shown [5]. A spatial resolution of roughly 2.5 to 3.5 nm was obtained from the 20 nm thick sample analyzed for a 120s counting time in a Vacuum Generators HB-501 FEG AEM. This spatial resolution is one of the best (smallest) measured in real specimens.

With expected improvements in AEM instrumentation to reduce background and to improve peak to background and in improvements in the preparation of thin foils, it will be possible to detect the presence of single atoms in the analysis volume.From the standpoint of x-ray microanalysis, the AEM is still in the development stage. At the present time, it is not an instrument optimized for microanalysis. Using well developed EPMA concepts, a true "electron-probe-

nano-analyzer" will undoubtedly be available in the near future [6].

ACKNOWLEDGMENTS

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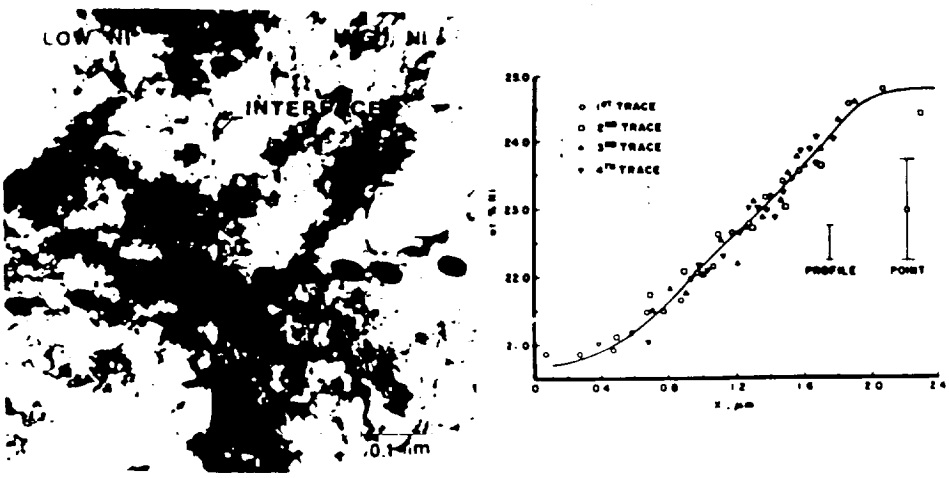

Fig. 1. (a) TEM photomicrograph of diffusion bond interface. (b) Ni concentration gradient in 20Ni-25Ni diffusion couple.

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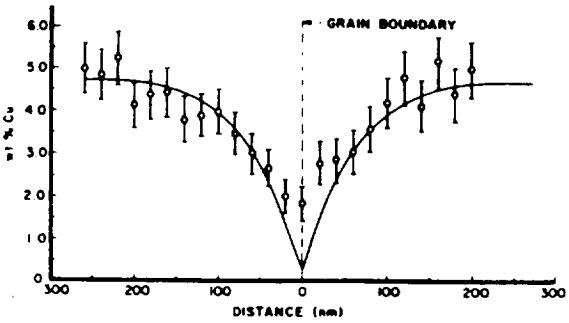

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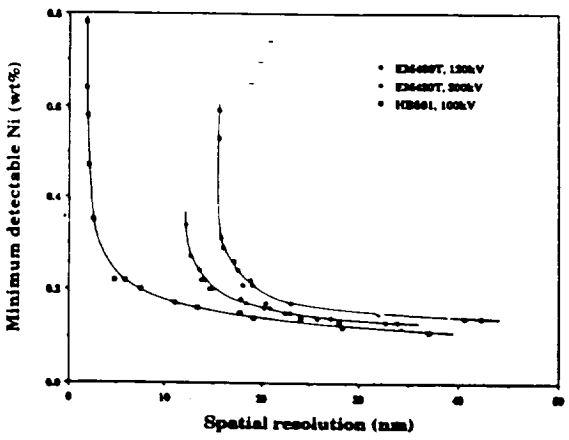

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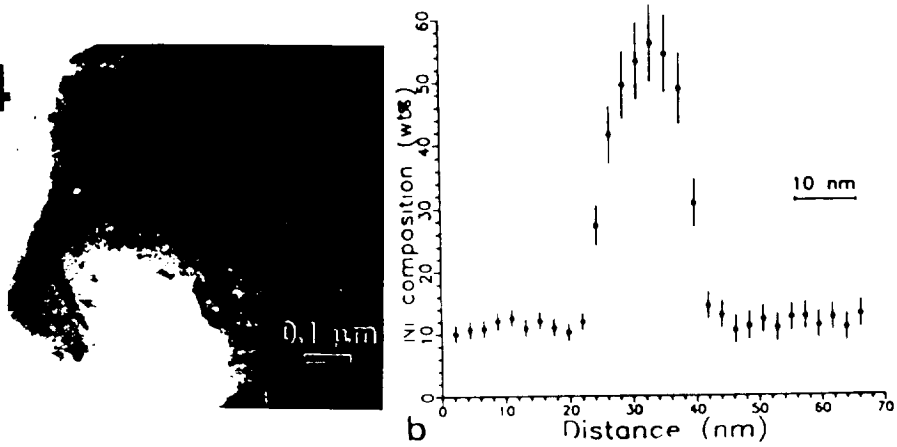

Fig. 2. Cu composition profile around a grain boundary in an Al-4 wt% Cu alloy aged at 250°C. Fig. 3. Relationship of spatial resolution and MMF measured by 3 AEM instruments. Fig. 4.(a) STEM image, and (b) Ni composition profile for a planar precipitate in the Grant meteorite: