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THE EFFECTS OF LOCAL MAGNIFICATION AND TRAJECTORY ABERRATIONS ON ATOM PROBE ANALYSIS

M.K. Miller
Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6376, U.S.A.

ABSTRACT - The effects of the difference in local magnification between phase and trajectory aberrations at interfaces and boundaries on atom probe and imaging atom probe analysis has been investigated using field-ion and ungated desorption micrographs. Local magnification differences of 2 to 5 times between phases were measured in Fe-Cr and Fe-Cu alloys. Significant trajectory aberrations at interphase interfaces and grain boundaries were also detected. The influence of these effects on the quantification of atom probe and imaging atom probe analyses is discussed.

INTRODUCTION

In many metallurgical investigations, a precise estimate of the spatial extent of compositional fluctuations is an essential parameter in the determination of the processes that have occurred. However, it is well known that trajectory aberrations and variations in the local magnification of different phases influence the accuracy of this type of analysis. Small aiming errors have been reported by Brenner and McKinney when aligning the probe aperture with the field-ion image. It has been shown by Waugh et al. that the rings in the low-coverage field desorption images are not the same as those in field-ion images.

Previous computer simulations of imaging atom probe desorption images by Delargy and Miller have shown that the technique is relatively sensitive to local changes in composition between phases and solute enrichment at boundaries. However, both these studies assumed that there was no differences in local magnification between the phases and that there were no trajectory aberrations. In a preliminary investigation, Miller et al. observed that the effective diameter of the cylinder of analysis in an Fe-30 wt. % Cr alloy aged 5200 h at 470°C was not constant and that the linear magnification changed by more than 40% between the chromium-enriched α' phase and the iron-rich α phase.

In this paper, an estimate of the magnitude of the differences in local magnification between phases and the magnitude of trajectory aberrations at boundaries and interfaces has been performed using a combination of field desorption images and distance-monitored atom probe composition profiles.

EXPERIMENTAL

The difference in local magnification between phases was investigated in a spinodally decomposed binary Fe-Cr alloy that exhibited a two phase isotropic interconnected microstructure and in an Fe-Cu alloy that contained small precipitates. These two high purity materials were selected since the lattice parameters of the two phases in the decomposed microstructure were similar and therefore did not introduce an additional parameter into the desorption images due to differences in atomic density. The trajectory aberrations at high angle grain boundaries were investigated in both a D1₃-ordered Ni₄Mo intermetallic alloy and a boron-containing L1₂-ordered Ni₅Al alloy.

Field desorption images were recorded in the imaging atom probe section of the ORNL atom probe.
Each desorption image was obtained during the evaporation of approximately a monolayer of material. All desorption images were recorded at a total pressure of $2 \times 10^{-6}$ Pa. Composition profiles were obtained in the energy-compensated time-of-flight mass spectrometer section of the same instrument in the presence of approximately $1 \times 10^{-6}$ Pa of neon so that the field-ion image could be visually monitored.

RESULTS AND DISCUSSION

A typical desorption image of the (001) region of a pure aluminum specimen is shown in Fig. 1. This type of image for a pure element is characterized by dark and light lines which coincide with the projection of crystallographic zones, as previously described. These light and dark features are not generally observed in alloys. Other contrast mechanisms and aberrations operate in two phase structures. Another common feature observed in these images is a bright or dark center to poles. Several mechanisms have been proposed to explain these features in desorption images.

The contrast between phases in field-ion images is a result of small differences in the field evaporation behavior of the phases and is composition dependent. When a phase field-evaporates at a lower than average field, the surface of the specimen will be slightly recessed in this area, as shown in Fig. 2(a). Therefore, the ionization probability of an image gas atom above this phase will be lower and, hence, it will appear dark in the field-ion image. In desorption images, each micrograph is obtained during the field evaporation of a thin layer of atoms from the specimen, Fig. 2(b). The relative contrast in these images is again due to differences in the field evaporation behavior of the two phases which, in this case, produces a convergence of the trajectories of the ions that originate from the recessed phase, Fig. 2(b). Therefore, the dark phase in the FIM image will appear as a locally higher concentration of dots, i.e. brighter, in the desorption image. A field-ion image and the complementary ungated desorption image of the two phase microstructure in an Fe-45 wt. % Cr alloy aged 485 h at 525°C are shown in Fig. 3. It is evident that the dark regions in the field-ion image are exactly reproduced as the brightly imaging regions in the desorption image. Similar results were obtained in the Fe-1.5 wt. % Cu alloy aged 4 h at 650°C where the copper-rich precipitates image darkly in the field-ion image and brightly in the ungated desorption image, as shown in Fig. 4. The difference in local magnification between each phase may be determined from the apparent concentration of atoms per unit area. However, the relatively high density of atoms in these images makes these determinations difficult. Alternatively, the same information may be determined more accurately from distance-monitored composition profiles.

A distance-monitored atom probe composition profile for the Fe-45 wt. % Cr alloy aged 485 h at 525°C is presented in Fig. 5(a). Visual evidence revealed that the darkly imaging regions in the field-ion image were the chromium-enriched regions, i.e. the $\alpha'$ phase. The average number of atoms collected per (110) plane evaporated at a constant voltage from the $\alpha$ and $\alpha'$ phase regions was 19 and 51 respectively. The data from this composition profile is replotted in Fig. 5(b) using a fixed block size of 50 ions. A comparison of the two composition profiles reveals that considerably more atoms are collected per unit depth from the darkly imaging regions than from the brightly imaging regions. Therefore, size estimates of the phases based on the average number of atoms removed per unit depth obtained from calibration experiments are subject to considerably error.

The desorption images in Figs. 3 and 4 indicated a relatively uniform density within each of the different phases. This uniformity was not always observed as shown in Fig. 6 for both the Fe-Cr and Fe-Cu alloys. In these desorption images a distinct high density rim was evident at the interface between the second phase and the matrix indicating more extensive trajectory aberrations. In this Fe-45 wt. % Cr alloy (and other binary Fe-Cr materials in which isolated islands of $\alpha'$ were present), the rim was observed in only a few out of several hundred micrographs, whereas, in the Fe-Cu alloy, approximately 10% of the precipitates exhibited this effect.

Trajectory aberrations at grain boundaries were also observed in single phase alloys as shown in Fig. 7 for a Ni$_3$Al specimen. It should be noted that care has to be exercised in obtaining desorption images in that an appropriate amount of material has to be removed while ensuring that the boundary does not move significantly during the exposure. The spatial extent of the bright region in Fig. 7(c) was estimated to be 2 nm. In many cases both bright and dim parallel lines were evident along the boundary. It is difficult to precisely define the plane of the boundary in these desorption micrographs and it is very time consuming to obtain a field-ion image to correspond to each desorption image.

Trajectory aberrations were also evident even when the line of the boundary is difficult to discern in
the field-ion micrograph. A grain boundary in Ni₄Mo grain boundary is shown in Fig. 8. The contrast observed in the desorption micrographs is very different from one region of the boundary to another even when the desorption micrographs were taken sequentially approximately an atomic layer apart. In one case the contrast changes from bright to dim on crossing the boundary at the indicated position, Fig. 8(b), and in the other the reverse is observed, Fig. 8(c).

The reason for these trajectory aberrations may simply be an extension of the explanation of the bright/dim lines observed in pure elements in that the trajectory of the atom as it leaves the surface of the specimen is strongly influenced by its neighbors. The local atomic configuration and therefore the influence of the neighbors on an evaporating ion change abruptly for a very small amount of material removed at a boundary and explains the contrast switch in Fig. 8(b) and 8(c).

These aberrations seriously effect the quantification of time-gated imaging atom probe elemental maps. Unfortunately, the current generation of imaging atom probes are only capable of recording one mass-to-charge species, usually an element, at a time and the remainder of the atoms are not detected. It is therefore not possible to take account of these trajectory aberrations without additional information. The difference in local magnification may be determined, as indicated above, from depth-monitored composition profiles. This information and the gated desorption image may then be used to examine the solute distributions. However, this procedure should only be regarded as semi-quantitative.

It is not sufficient to infer that there is segregation to a boundary based only on an increased density in the desorption image, except in the special case where there is a low solute content in the matrix.

These effects also influence conventional atom probe analyses particularly with respect to interphase interfaces and other boundaries. Since all elements are collected simultaneously, the effects are not as severe as for time-gated elemental maps. However, the standard practice for determining a composition profile as a function of distance from a boundary by repeated positioning of the probe aperture at a variety of distances away from the boundary should be used with care since the distance scale close to the boundary plane is non-linear. The optimum method for accurate depth scale, when the specimen geometry permits, is to use composition profiles normal to the boundary plane where the evaporation of a known pole is visually monitored in the field-ion image to estimate the depth of material removed.

SUMMARY

Significant differences in local magnification of 2 to 5 times were measured between phases from composition profiles. It is recommended that distance-monitored composition profiles are obtained if a precise measurement of the spatial extent of a phase is required. Trajectory aberrations were observed at grain boundaries in single phase materials and at interphase interfaces. These aberrations make the distance scale non-linear and the extent of composition gradients difficult to measure.

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REFERENCES

Fig. 1. Ungated field desorption image of a pure aluminum specimen.

Fig. 2. Schematic diagrams of (a) field-ion and (b) field desorption imaging.

Fig. 3. Comparison of (a) field-ion and (b) desorption micrographs of an Fe-45 wt. % Cr alloy aged 485 h at 525°C. The dark regions in the field-ion micrograph and the bright regions in the desorption micrograph are the chromium-enriched α' phase.

Fig. 4. Comparison of (a) FIM and (b) desorption micrographs of an Fe-1.5 wt. % Cu alloy aged 4 h at 650°C where the copper-rich precipitates are dark and bright, respectively.
Fig. 5. (a) Distance-monitored atom probe composition profile in an Fe-45 wt.% Cr alloy aged 485 h at 525°C. (b) data replotted in 50 ion blocks. Note the difference in the relative apparent width of the two phases.

Fig. 6. Trajectory aberrations at interphase interfaces in (a) Fe-45 wt.% Cr and (b) Fe-1.5 wt.% Cu.
Fig. 7. Trajectory aberrations at a grain boundary Ni₅Al. (a) FIM, (b) desorption image, submonolayer, (c) desorption image, 1 monolayer.

Fig. 8. Trajectory aberrations at a grain boundary in Ni₄Mo. (a) FIM, (b & c) desorption images taken one layer apart.