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Imaging atom probe microscopy for segregation studies*

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The measurement of low concentrations of elements segregated to or near grain boundaries with a spatial resolution of *ca.* 1 nm has recently become possible with the introduction of the imaging atom probe (i.a.p.). This development of the original atom probe field ion microscope uses a time-gated image intensifier as the detector of a time-of-flight mass spectrometer and displays an elemental map of ions desorbed from the surface of a field-ion specimen. The sensitivity of the analysis is uniform for both light (e.g. B, C, O) and heavy (e.g. Sn) elements, and concentrations down to 100 μg/g can be detected; accurate quantitative analyses are obtained by using the more conventional type of atom probe.

The i.a.p. has revealed a number of qualitative differences in the behaviour of segregants. For example, by using the conventional atom probe it was found that in a commercial molybdenum wire, oxygen was present at a grain boundary to a concentration of 1×10^{18} atoms/m^2. The i.a.p. showed the oxygen to be strongly localized, with 75 % of the oxygen in a band 1.5 nm wide along the boundary. Similar localization was observed for oxygen at a grain boundary node in the same wire: the oxygen concentration was approximately the same for the three interfaces, one of which contained a 4 nm step some 10 nm from the node. In contrast, carbon in a patented steel wire is observed to be spread over regions *ca.* 4 nm across near subgrain boundaries in the heavily deformed structure. On lightly annealing, the boundaries realign to form parallel low angle grain boundaries some 5 nm apart. Carbon is still detected over a *ca.* 4 nm region near these, and is non-uniform along the line of the boundaries, presumably as a result of interactions with their dislocation structure.

The segregation of tin to grain boundaries in N.P.L. pure iron is also being studied. As the grain size of this material is relatively large (*ca.* 40 μm), careful specimen preparation is needed to bring boundaries within the field of view of the f.i.m.: specimens are carefully electropolished and repeatedly examined in the transmission electron microscope until the boundaries are seen to be within *ca.* 100 nm of the tip. Boundaries examined in the conventional atom probe fall into two classes. One class shows a tin concentration at the boundary of *ca.* 15 %, compared with the bulk value of 0.22 %, while other boundaries show little tin enrichment but significant amounts of carbon (*ca.* 1 %), the bulk level being negligible. One such boundary examined in the i.a.p. unexpectedly showed a significant tin concentration in a region *ca.* 10 nm in diameter 5 nm from the boundary, which itself was almost free from tin.

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