

## The Effects of Detector Efficiency on Distinguishing Solute Atoms in Random Solid Solution and Solute Clusters

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The introduction of foreign atoms into a crystal lattice impedes the motion of dislocation and leads to solid solution hardening. As the solute content increases, the number of solute-solute nearest neighbors increase and random clusters are formed. The percolation thresholds for solute atoms for the most common face and body centered cubic lattices have been found from Monte Carlo simulations to be  $p_c(\text{f.c.c.}) = 0.120$  and  $p_c(\text{b.c.c.}) = 0.180$  [1]. The addition of multiple types of solute atoms may also introduce additional populations of solute-solute clusters due to the interaction of the different solute species. The distribution of these solute clusters in a material is a key factor in understanding the properties of materials and in designing materials with improved properties

Ideally, an experimental technique that can determine the atomic coordinates and identity of all the atoms in the sample is required to quantify the distributions of both types of clusters. The detection efficiency (DE) of the single atom position-sensitive detector in the current generation of the local electrode atom probe (LEAP<sup>®</sup>) is typically  $\approx 40\%$ . Therefore, the influence of DE on cluster analysis has been simulated to establish the minimum DE for reliable analysis for this type of microstructural feature in the LEAP. As it is not practical to vary (i.e., increase) the DE of the detector over a wide range, series of simulations of atom probe tomography data [2] of  $\alpha$ -Fe crystal structure with a range of DEs, solute contents, and small deviations of the atoms from their lattice sites have been generated. The  $30 \times 30 \times 30$  nm volume shown in Fig. 1a contains 270 clusters with a nominal radius of 0.3 nm with a number density of  $2.7 \times 10^{23} \text{ m}^{-3}$ , a range of 6-13 atoms and an average of 9.5 atoms (100% DE) for a total solute content of 0.11 at. %. The effect of reducing the DE is shown for 0% matrix solute case in Figs. 1b-d for DEs of 80, 60 and 40%, respectively. In order to account for random solute clusters, volumes were also generated in which the identities of the atoms were randomized and analyzed. An example is shown in Fig. 1e for the 100% DE data.

The size distributions of the solute clusters were estimated by the maximum separation method [3] with the use of a maximum separation distance of 0.3 nm (approximately equivalent to the first nearest neighbor shell) and no minimum size cutoff. The change in the numbers of solute atoms collected as a function of cluster size for DEs between 40 and 100% and a trace matrix solute content of 0.003% is shown in Fig. 2. At 100 and 90% DE, separate peaks for the solute clusters and the solute in the matrix were evident. As the DE decreases, the distribution broadened and the position of the size distribution of the solute clusters progressively decreased due to the decrease in the number of ions collected and these two distributions merge. However, the Guinier radius of the solute clusters, as estimated from the spatial coordinates of the solute atoms with maximum separation method with a minimum cutoff of  $n_{\text{min}} = 2$  atoms (to exclude the contribution of the solute in the matrix), only progressively decreased from  $0.30 \pm 0.02$  to  $0.26 \pm 0.05$  nm over the 100 to 40% DE range. At this trace matrix concentration, the randomized distribution indicates that only isolated solute atoms and matrix clusters containing 2 solute atoms contributed to matrix component. Therefore, the small decrease in the Guinier radius from the correct value of 0.3 nm is due to the influence of the cut off of the size distributions of the solute clusters. No change was observed in the estimated number density of solute clusters.

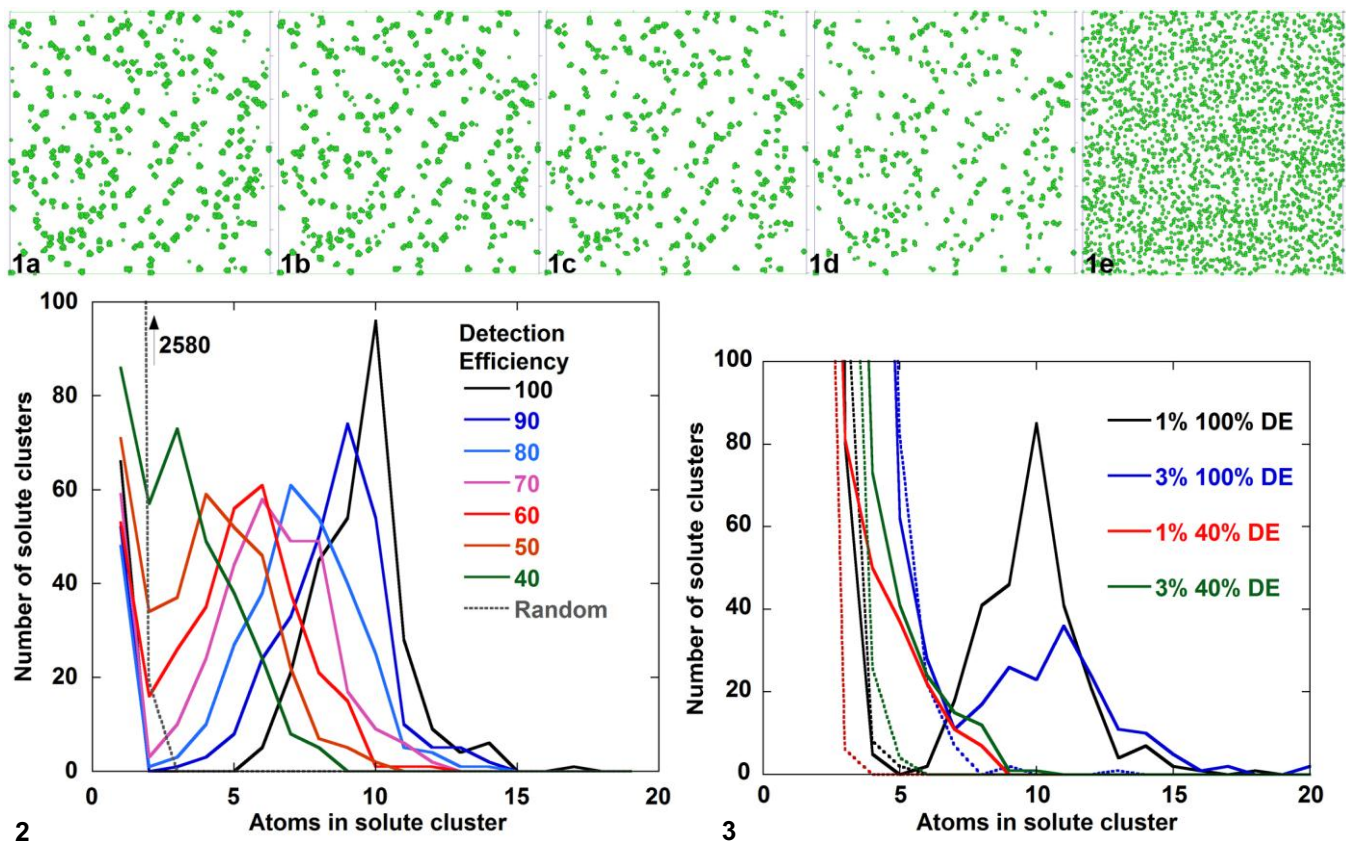
As the matrix solute content is increased, the maximum size of the cluster detected,  $n$ , increased from  $n=2$ , to  $n=5$  and 13 for solute contents of 1 and 3%, respectively (100% DE), and to  $n=3$  and 5 for 1 and 3% solutes, respectively (40% DE). The cluster distributions detected for 1 and 3% total solute for 100 and 40% DEs, Fig. 3, indicate that at these matrix solute levels, the sizes of the solute clusters in the 100% DE cases are shifted to smaller sizes in the 40% DE cases and are not easily deconvoluted from the matrix solute clusters. These simulations have indicated that detection efficiencies of greater than 90% are required for accurate analysis of clusters for matrix solute levels in excess of ~1%. [4]

[1] CD Lorenz and RM Ziff, *Phys. Rev. E*, **57** (1998) 230–236.

[2] MK Miller and JM Hyde, 11<sup>th</sup> Euro. Congr. Electron Microscopy, Aug. 1996, Dublin, Ireland, CD.

[3] JM Hyde and CA English, in: *Microstructural Processes in Irradiated Materials*, Eds. GE Lucas, L Snead, MA. Kirk, RG Elliman, MRS. Symp. Proc., Vol. 650 (MRS, Pittsburgh, 2000) p. R6.6.

[4] Research supported by the U.S. Department of Energy (DOE), Basic Energy Sciences (BES), Materials Sciences and Engineering Division, and through a user project supported by ORNL's Shared Research Equipment (ShaRE) User Program, which is also sponsored by DOE-BES.



**Figure 1.** 30 nm x 30 nm x 30 nm volumes showing the effect of DE on the solute clusters (0.003% solute in the matrix, 100% solute in clusters), a) 100%, b) 80%, c) 60% and d) 40% DE. e) Effect of randomizing the identities of the atoms (100% DE). **Figure 2.** Change in the number of solute atoms collected from 0.3-nm-radius clusters with DE for a matrix solute content of 0.003% and a cluster solute content of 100%. **Figure 3.** Effect of DE and matrix solute content on the number of clusters detected for each size. Dotted lines are randomized distributions of solute atoms.