Cross-correlative Microscopy to Understand Nanocrystalline Stability

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The chemical partitioning of solutes to grain boundaries can provide a thermodynamic reduction of the boundary energy and/or a kinetic drag effect on the boundary's mobility. These mechanisms have been shown to help stabilized nanometer-scale grains from thermally driven growth. In large part, the solute segregation to these grain boundaries has been considered isotropic; however, the diversity in grain boundary energy and structure will make solute partitioning varied. In order to quantify this boundary specific partitioning, one requires high spatial resolution with high chemical sensitivity. To achieve these two requirements, the use of cross-correlative transmission electron microscopy (TEM) and atom probe tomography (APT) has been employed [1]. APT specimens were annularly ion milled into the required needle-like specimen tip, with the grain boundary misorientation measured by Precession Electron Diffraction (PED), Figure 1(a). The tip was then field evaporated via APT where the segregation can be quantified, Figure 1(b). Correlating the reconstructed atom map with the former PED map enabling the solute, as a function of grain boundary, to be linked, Figure 1(c). Using this technique, the stability in a Pt-11Au alloy was studied [2]. The alloy was magnetron sputtered deposited and then subsequently annealed at 700K and 1300K to facilitate partitioning. In the as-deposited film, the Au was in found to be in solid solution within the Pt matrix. Upon annealing at 700K up to 24h, the Au segregated into high angle grain boundaries. Interestingly, the interfacial excess (IE) mapping from the APT data [3] revealed variations of Au in same boundary, Figure 1(d). When the alloy was annealed to 1300K, grain growth from ~ 110 nm to ~215 nm was observed after 3 minutes indicating the loss of the stability. At this temperature and time, the Au was then found to cluster. This presentation will discuss how this level of quantification reveals intrinsic stability behavior in nanocrystalline alloys, including potential decomposition reactions within boundaries themselves and the evolution of the solute from decorating to clustering and its effect on boundary stability.

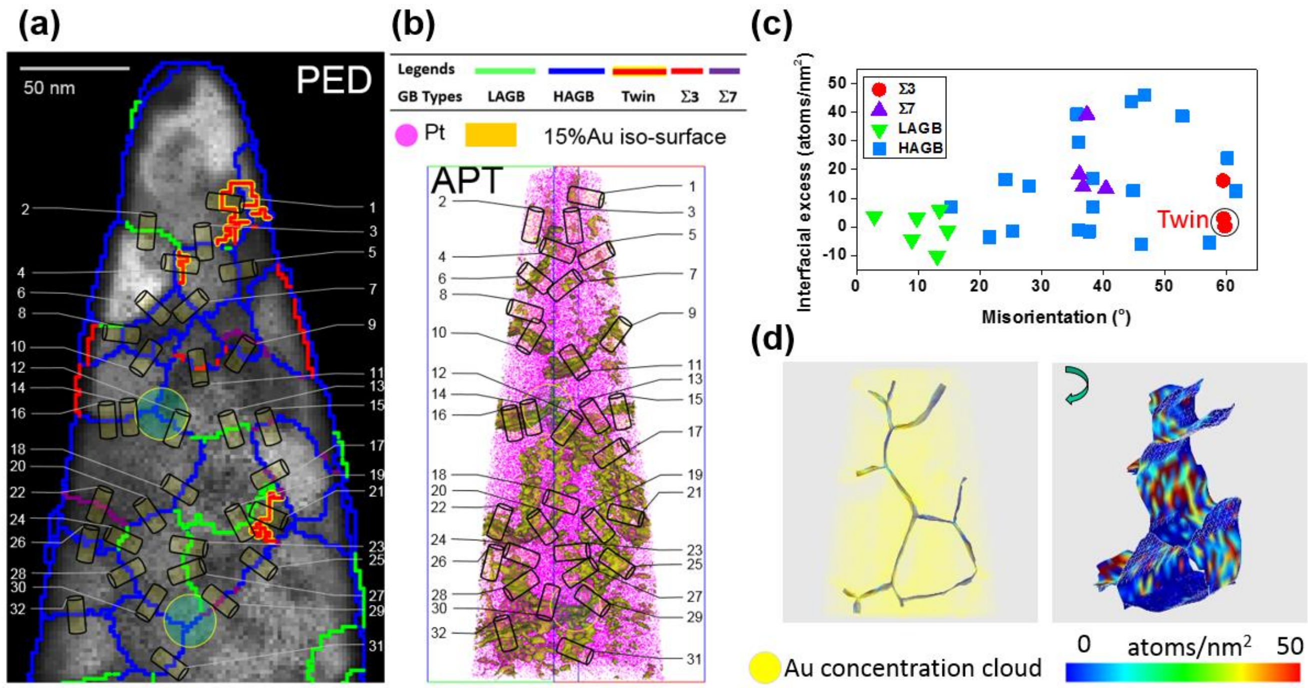

Figure 1: (a) PED map of the boundary segregation **(b)** APT reconstruction of (a) **(c)** IE values of different grain boundaries **(d)** visual IE map revealing variation of partitioning within the same boundary.

References:

[1] Zhou, X. et al., Scientific Reports **6** (2016) p. 34642.

[2] O'Brien, C. et al., Journal of Materials Science **53** (2018) p. 2911-2927.
[3] Felfer, P. et al., Ultramicroscopy **159** (2015) p. 438-444.