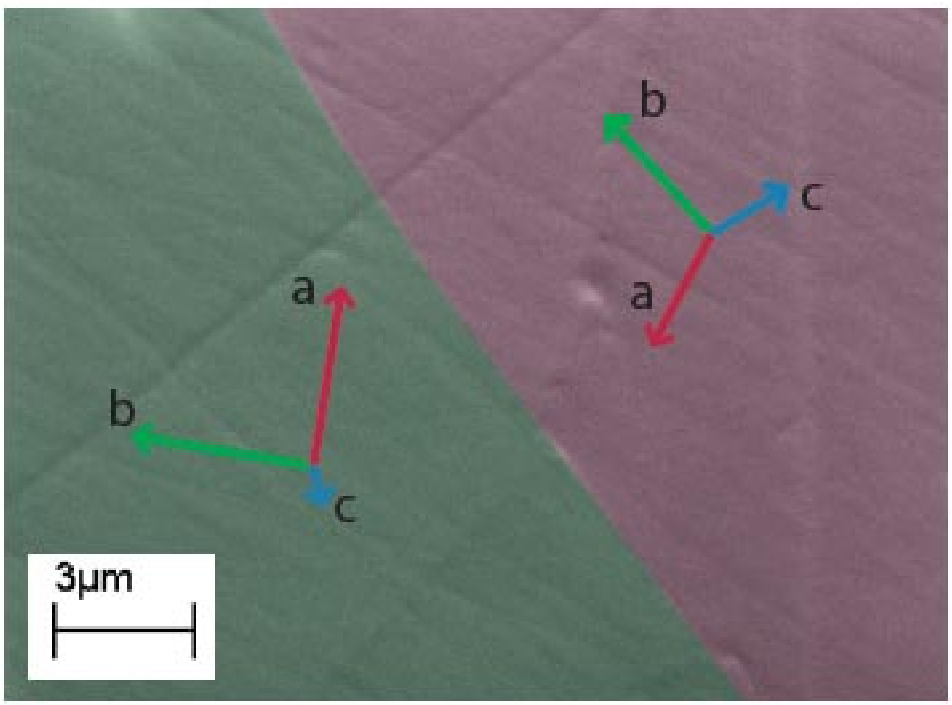
EA-4 SEM and TEM Characteristics of the Grain Boundary Region - discussion

An SEM image of a representative tight grain boundary is shown in figure EA-4-1. A very few grain boundaries showed “widths” approaching a few 100 nanometers in SEM, but these appeared to be late, rough, pull apart features rather than in situ grain boundaries. TEM preparation and analysis was performed at the Engineering Department at Caltech. This was very limited by the difficulty of sample preparation; most samples were too friable to isolate and image grain boundaries. Thus, TEM analysis was inconclusive and biased towards those rare ITM features which survived the ion milling. As an example, Figure EA-4-2 shows an apparent ~2.5μm wide amorphous region near a triple junction (with Ca-Si composition) imaged by TEM. We did not observe anything this thick with SEM and believe this to be an anomalous feature, not representative of the whole. Still, these are indications that some melt (albeit rare) may have existed in the ITM of the samples. SEM and TEM analysis suggests that grain boundaries are *dominantly* dry and melt free with discernible thicknesses no larger than 10s of nm – and probably much thinner in general.

EA-4-1. SEM image from thin section of sample EB036 (70 hour piston cylinder run). Example of a high-angle grain boundary with thickness smaller than can be resolved with scanning electron microscopy. The image consists of two large diopside grains, with a grain boundary running from left of center at top to right of center at bottom, visible as a sharp bright line in secondary electrons due to a small topographic step. Other linear features on the surface are polishing flaws and scratches. Electron Backscatter Diffraction mapping, shown by color overlay, identifies the orientations of the two grains (green and red, respectively), whose monoclinic crystal axes (indicated in projection by the red, blue and green arrows labeled a, b, c) are rotated relative to each other by >100° in two directions. There is no topotactic relation between these orientations. The orientation change is sharp at SEM resolution and there is no other phase, or opening, visible along the boundary.

EA-4-2. TEM image of sample EB027 (70 hour piston cylinder run). Thick amorphous layer at triple junction between grains. Image taken at 13600X magnification. Semi-quantitative chemical analysis of amorphous region indicates Si and Ca only.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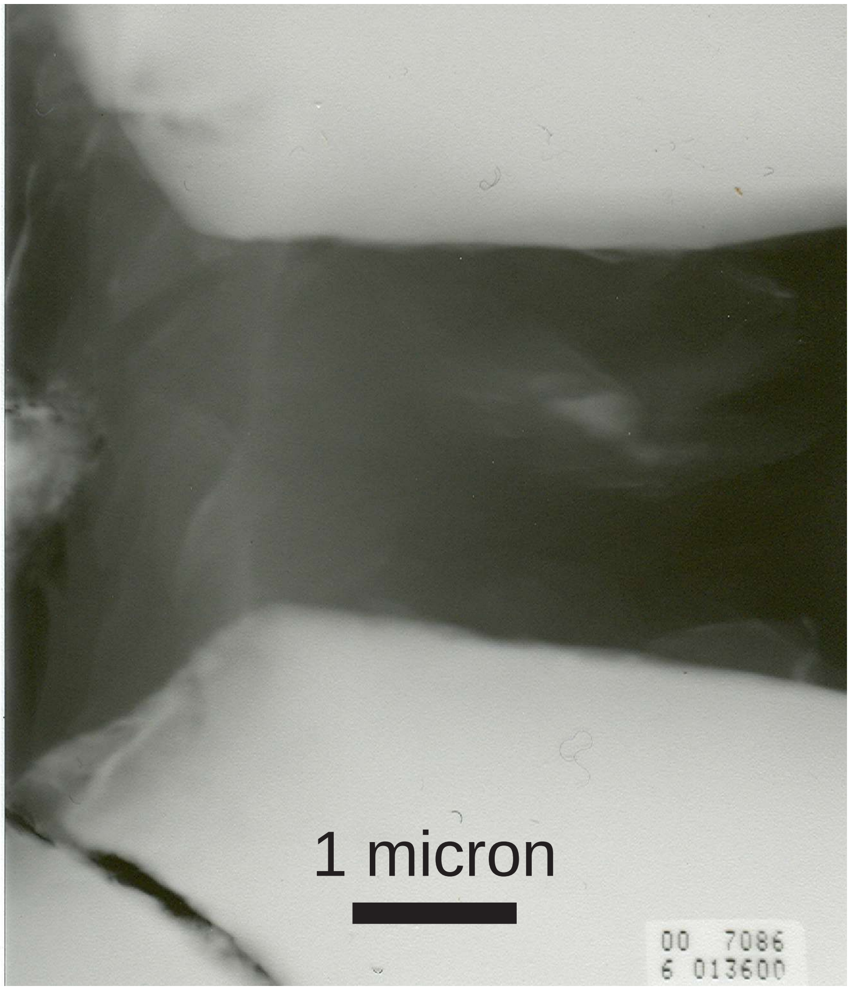
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