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Electrons Rock: Multiscale Analysis of Subgrain Deformation Boundaries in Antarctic Garnets

Berit H. Goodge[1], Bryan R. Bandli[2], Lena F. Kourkoutis[1,3], John W. Goodge[4]

[1]School of Applied and Engineering Physics, Cornell University, Ithaca, NY, USA
[2]RJ Lee Group, Monroeville, PA, USA
[3]Kavli Institute at Cornell for Nanoscale Science, Cornell University, Ithaca, NY, USA
[4]Department of Earth & Environmental Sciences, University of Minnesota, Duluth, MN, USA

Electron probe techniques offer a rich and powerful variety of experimental approaches to study of materials ranging from those synthesized in a lab to those synthesized by the Earth. Each technique, however, is best suited to address specific questions: for example, diffraction experiments can provide extremely precise structural information, while spectroscopic methods instead probe elemental or chemical signatures. Similarly, these techniques are optimized for application across a finite range of length scales, whether microns or nanometers. Leveraging a combination of several disparate techniques through correlated experiments thus provides a route to study and understand materials comprehensively from the grain down to the atomic scale.

Garnet ($X_3Y_2Si_3O_{12}$) is a common mineral of Earth's crust and mantle often used as a geologic recorder of microstructures, mineral stability, metamorphic conditions, and orogenic timing. Due to its equant habit and relatively high shear modulus (G ~90 GPa), garnet grains in dynamically recrystallized metamorphic rocks normally resist crystal-plastic deformation during mountain building or other tectonic processes because strain is concentrated in weaker matrix minerals such as quartz and micas. Surprisingly, a quartzite specimen collected in the Transantarctic Mountains contains an abundance of almandine-rich (X_{Fe}=0.85-0.88) garnet grains which exhibit strongly anisotropic intracrystalline deformation despite being hosted by weaker quartz (G ~35 GPa). Figure 1 shows optical micrographs of two representative garnet porphyroblasts: one 'normal' garnet with typical equant habit and one 'deformed' garnet that is highly elliptical or lozenge-shaped. How did such severe deformation occur, especially in a quartz matrix more than three times weaker than the garnet?

Due to garnet's isometric crystal structure, lattice dislocations cannot be identified by light optics. Instead, electron backscatter diffraction (EBSD) was performed using on a JEOL 6500F field emission gun SEM equipped with an Oxford Instruments Symmetry detector to probe the crystalline structure of these deformed garnet grains. EBSD analysis reveals that many garnets contain several subgrains with different crystalline orientations separated by irregular subgrain boundaries with local lattice mismatches of up to 40°. Figure 1 shows the orientation contrast (OC) image and inverse pole figure (IPF)-X map of a distorted garnet grain in which the misorientation boundary between two subgrains (purple and pink) does not appear to correspond with any macroscopic disruption (e.g., fracture) of the crystal as observed by OC. Analysis of 43 individual grains shows random distribution of crystallographic orientations within the distorted grains.

To gain further insight to the character of these misorientation boundaries, we turn to another suite of electron probe techniques optimized for characterization at the nanoscale. Using a standard focused ion beam (FIB) lift-out procedure on a Thermo Fisher Helios G4 UX FIB, a cross-sectional specimen spanning this subgrain boundary was prepared for investigation by high-resolution scanning transmission electron microscopy (STEM). The subgrain boundary is immediately apparent by medium-

angle annular dark-field (MAADF)-STEM imaging: diffraction contrast in the MAADF-STEM image (Figure 2, top) highlights a sharp boundary running through the lamellae. Atomic-resolution high-angle annular dark-field (HAADF)-STEM imaging (Figure 2, bottom right) confirms the relative misorientation and reveals the abruptness of the subgrain boundary (~4 nm across). Uniform and regular crystal lattices are preserved in the domains to either side. The high magnification insets include overlays of almandine garnet ($Fe_3Al_2Si_3O_{12}$) atomic models showing good agreement with the identified [123] and [112] crystalline orientations. An elemental profile of the boundary was collected by energy dispersive x-ray spectroscopy (EDX) using a 1.8 steradian Dual-X EDX detector equipped on a Thermo Fisher Spectra 300 X-CFEG operating at 300 kV. The garnet grains in this specimen are nearly pure almandine with negligible variation in Fe concentration. The minor species Mg and Ca, however, show inverse concentration profiles across the subgrain boundary which itself is comparatively Ca-rich and Mg-poor.

Together, the integrated structural and elemental analyses help reveal the origin of these strongly deformed garnet grains: randomly oriented and distributed garnet porphyroblasts crystallized and were then deformed at high temperatures and high shear-strain rates ($T = 700°C$ and $\gamma \approx 5$, respectively) [1,2] in a quartz matrix, giving rise to crystal-plastic shape deformation and extremely abrupt intracrystalline dislocation boundaries within single grains. These boundaries allowed for crystallographic slip and acted as channels for aqueous fluid migration: progressive mineral reactions involving feldspar and apatite likely yielded Ca-rich fluids, which permeated the subgrain channels and diffused into the subgrain crystal on a scale of 10s of nm to either side. The combination of multiscale and multimodal analysis by a suite of analytical electron probes both reveals the geologic history of deformed garnets and demonstrates the power of uniting such complementary techniques [3].

References:

[1] Goodge, et al., *Tectonics* **12**, (1993).
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[3] We thank Dr. Nicholas Seaton at the University of Minnesota Characterization Facility for the EBSD measurement and analysis shown here. Supported by NSF (DMR-1654596, DMR-1429155, DMR-1719875, DMR-2039380, DMR-2011401) and NNCI (ECCS-2025124).

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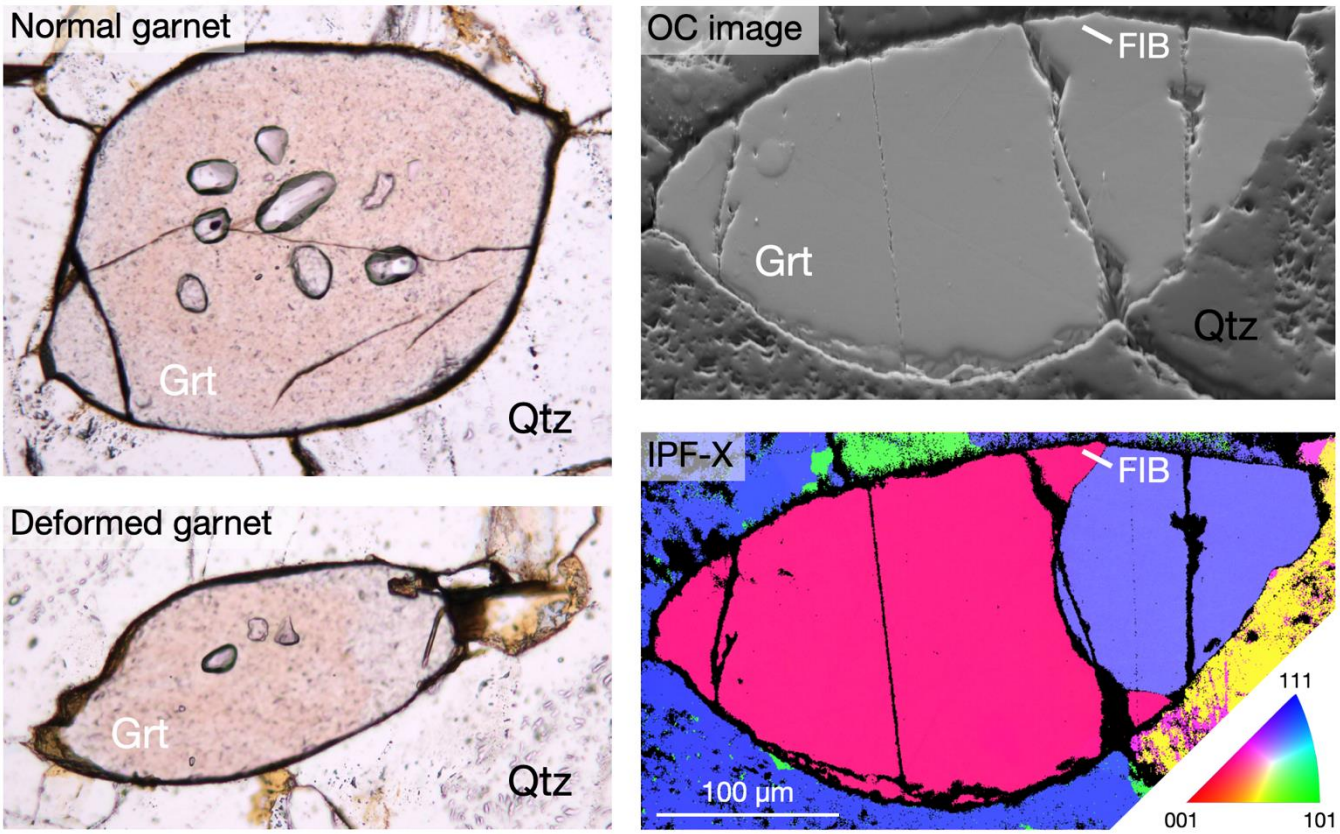

Figure 1. Macro- and meso-scale characterization of garnet (Grt) grains. In a weak matrix like quartz (Qtz), typical garnet grains exhibit round, isotropic shapes (top left). Geologic specimens collected from the Geologists Range in the Transantarctic Mountains show an abundance of strongly anisotropic or deformed garnet grains (bottom left). Typical grain sizes for both 'normal' and 'deformed' garnets are ~1 mm. EBSD mapping of a deformed garnet grain reveals an abrupt subgrain crystalline orientation boundary in the IPF-X map (bottom right) even where the OC image (top right) shows no sign of macroscopic division, e.g., cleaving or cracking. FIB lift-out was used to extract a cross section spanning this misorientation boundary for nano-scale investigation in the STEM.

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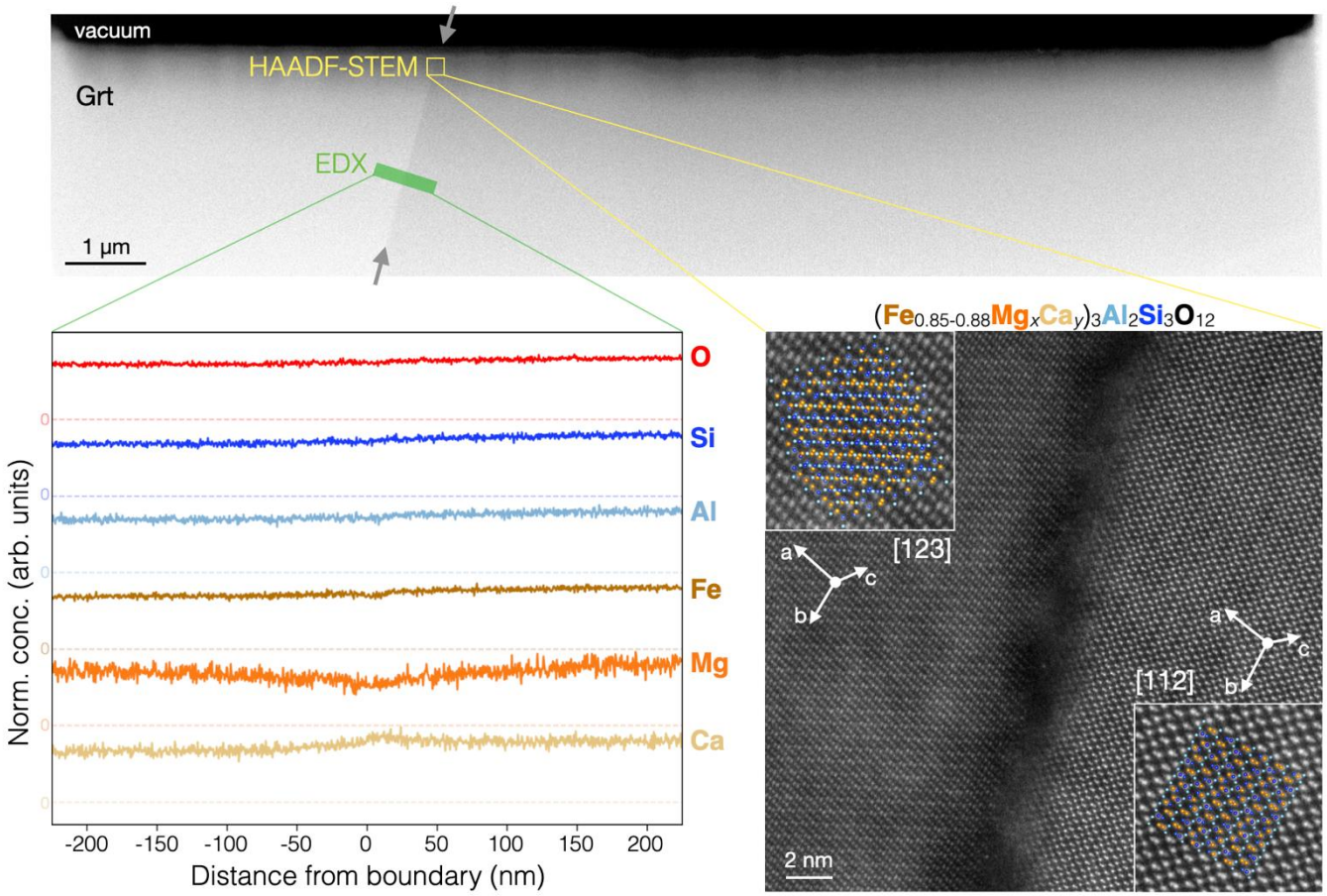

Figure 2. Nanocharacterization in the STEM confirms the abrupt change in crystalline orientation across the boundary identified by EBSD. Diffraction contrast in MAADF-STEM imaging highlights the boundary marked by gray arrows which extends throughout the full depth of the cross-sectional liftout (top). Atomic-resolution HAADF-STEM reveals the non-crystalline subgrain boundary and confirms the different orientations on either side, which can be matched to the [123] and [112] projections of almandine (bottom right, insets show atomic model overlays). Elemental analysis by STEM-EDX across the boundary shows a diffuse profile of increased (decreased) magnesium (calcium) concentration near the subgrain boundary (bottom left). Concentration profiles are normalized separately for each element and vertically offset for clarity; dotted lines in corresponding colors mark the zero-count level for each profile. The elemental profile of the subgrain boundary extends over tens of nanometers, but the structural modification is limited to only a ~4 nm channel.

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