Appendix 2

The mineral garnet has been analyzed using the Quanta SEM with Inca EDS at WCU, and by WDS using the Cameca SX-50 electron probe microanalyzer (EPMA) at the University of Massachusetts, Amherst. The WDS-EPMA analyses are more precise and accurate, and the EDS-SEM data are compared with them.

The chemical composition of garnet is sensitive to external conditions of temperature and pressure during growth, resulting in chemical zoning in garnet crystals. For example, zoning in the abundance of Ca is shown in Figures 1 and 2. In both images, the hottest color (yellow center) represents the highest values (about 7 weight % CaO), while the coolest color (violet-blue) represents the lowest values (about 1.9 weight % CaO). Figure 1 was generated as a montage of elemental X-ray maps using the AUTOMATE function in the Inca EDS software on a carbon-coated thin section (operating conditions are given in the caption for Figures 3-5). Figure 2 was generated by WDS analysis on the same specimen using the Cameca SX-50 microprobe. The WDS map provides greater resolution, but the EDS map shows the essential zoning pattern. The EDS-SEM map was of good enough quality to guide the position of a traverse of individual points for detailed analysis by WDS-EPMA, shown as the line on Figure 2.

Figure 1. EDS-SEM map of Ca

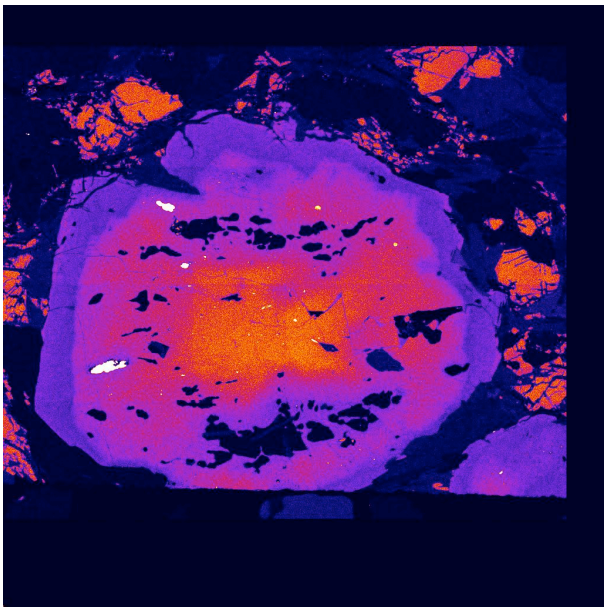
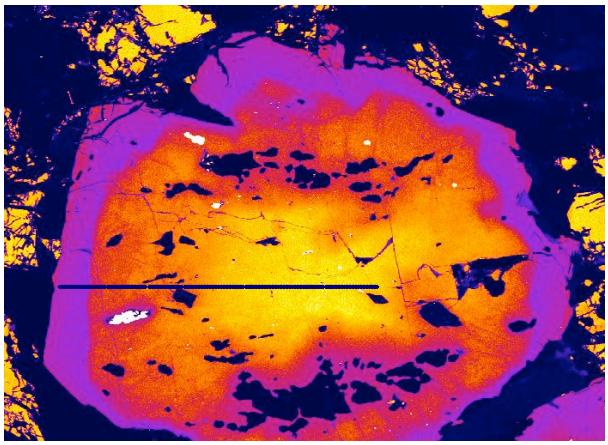


Figure 2. WDS-EPMA map of Ca



An advantage of the EDS-SEM map is that the X-ray spectra comprising the map are stored and can be retrieved using the Point and ID function of the Inca software. To compare the EDS with the WDS data, 50 small areas along a line traverse in the same location as the EPMA points were derived from the stored spectra, and the results are superimposed on the WDS-EPMA data in Figures 3-5. The data are shown in terms of the moles of Ca, Mn, Mg, and Fe in one formula unit of garnet. As can be seen in Figures 3-5, the data "mined" from the EDS-SEM map are a good match to the WDS-EPMA analyses.

Figures 3-5. Composition of chemically zoned garnet crystal.

Parameters for WDS-EPMA analyses (blue diamond symbols): Cameca SX-50, 15 kV accelerating voltage, 15 nA beam current, 1 µm beam diameter with magnification at 5000X, Ca analyzed with PET crystal, Mg analyzed with TAP crystal, Mn and Fe analyzed with LiF crystal. Over 200 individual point analyses were acquired along the line traverse shown in Figure 2.

Parameters for EDS-SEM analyses (magenta square symbols): X-ray spectra collected for elemental map using FEI Quanta SEM and Oxford INCA EDS: 30 kV accelerating voltage, spot size 6.5, acquisition rate ≈ 14 kcps, deadtime ≈ 30%, magnification at 300X, 200 µsec dwell time, process time 3, 10 frames, QuantOpt every 2 hours, ≈ 11 hour total run time. The Inca file containing the elemental maps was opened after analysis was complete, and the Point and ID function was used to select locations for data mining. The same line traverse shown on Figure 2 was divided into 50 square areas, each approximately 40 µm on each side. The acquired X-ray spectra were aggregated from within each square area to provide a single analysis.

Figure 3. Comparison of Fe.

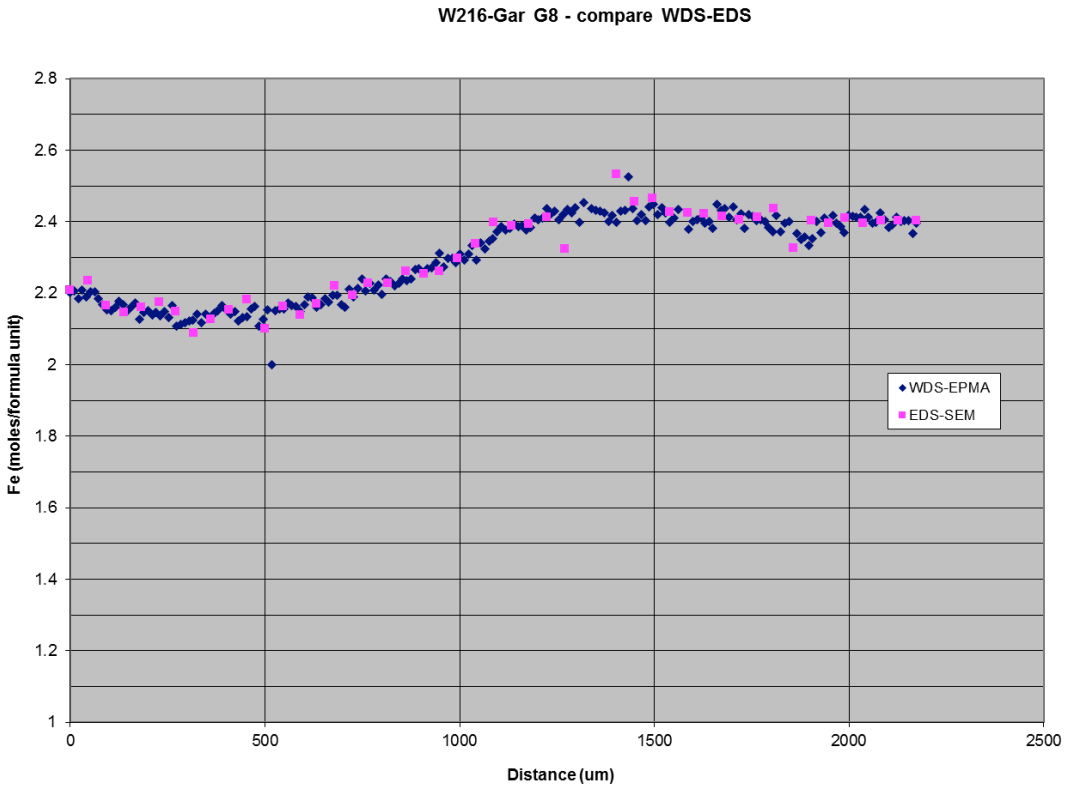


Figure 4. Comparison of Ca.

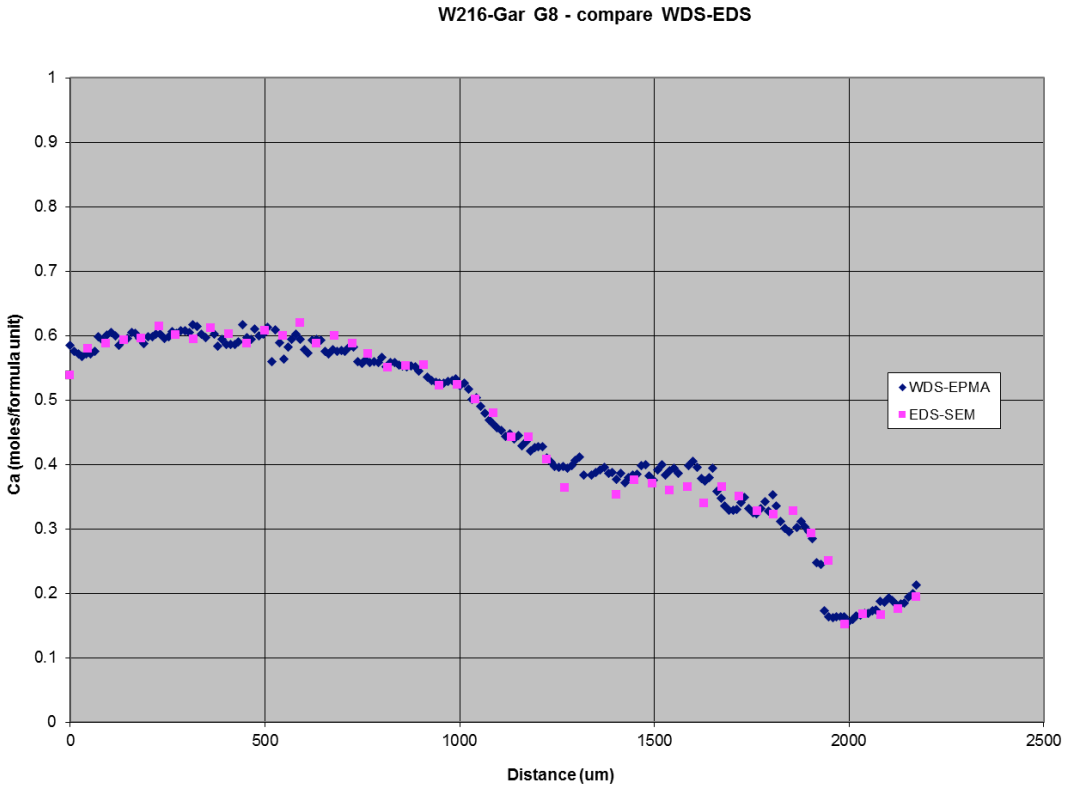


Figure 5. Comparison of Mn and Mg.