

## **DATA REPOSITORY ITEM 2003063:**

Sample descriptions, analytical methods, and xenolith compositions

### **SAMPLE DESCRIPTIONS**

**KX1-1 (Lace):** Sample KX1-1 is a 20 cm diameter xenolith, comprising a primary mineral assemblage of garnet, sapphirine, and quartz with subordinate rutile, sillimanite, graphite, and sulfide, and accessory kyanite, corundum, zircon, and monazite. The xenolith displays a coarse granoblastic texture with grain sizes of 2-5 mm for garnet and quartz, and 1-3 mm for sapphirine. Garnets are extensively fractured and have well-developed kelyphitization along these boundaries. Inclusions of quartz are common, concentrated toward the center of grains. Sapphirine is most commonly in grain contact with garnet, although it can be completely occluded by quartz. Quartz grains have a distinctive blue color (rutilated), and show patchy extinction, with the development of discrete subgrain domains separated by crenulated boundaries. Quartz also exhibits planar fractures in some grains, while most grains also contain parallel planar concentrations of minute inclusions, in addition to ubiquitous oriented rutile needles. Sillimanite occurs as rare discrete grains, and thin rinds between quartz and sapphirine. Rutile is found in two distinct settings: as elongate acicular micron-scale needles in garnet, sapphirine and quartz; and as larger rounded prisms and anhedral grains included in all major phases and on grain boundaries. The first morphology of rutile is crystallographically controlled, and best developed in quartz, where needles oriented on three planes can coarsen up to several microns in thickness and tens of microns in length.

**KX1-2 (Lace):** Sample KX1-2 is a 7 cm diameter xenolith which is slightly more altered than KX1-1, with the development of secondary phlogopite grains and pervasive grain boundary alteration. The mineralogy of sample KX1-2 is identical to that of KX1-1,

namely comprising garnet, sapphirine and quartz with minor rutile, sillimanite, sulfide and graphite, and accessory kyanite, corundum, zircon and monazite. Unlike KX1-1, garnet in this sample forms thin rims between some sapphirine and quartz crystals as well as large discrete crystals. KX1-2 is also coarser-grained, with garnets reaching over 1 cm in diameter and sapphirine up to 5 mm. This sample contains slightly more modal garnet than KX1-1, and sapphirine predominates over quartz. Both zircon and monazite are found as inclusions in garnet, sapphirine, and quartz, and as interstitial grains.

**KX1-8 (Lace):** Sample KX1-8 is a 10 cm diameter xenolith with an unaltered primary mineralogy more diverse than that of the previous sapphirine-bearing samples. In addition to garnet, sapphirine, quartz and rutile, this xenolith contains abundant sillimanite and rarer plagioclase feldspar grains. Textures are also more complex, with common reaction boundaries and ‘fingerprint’ intergrowths involving sapphirine, garnet, sillimanite and quartz. These coronae comprise single or double rinds of sillimanite ± garnet developed between primary sapphirine-quartz contacts, with sillimanite preferentially crystallized next to sapphirine and garnet next to quartz. Sillimanite rims sometimes merge into coarsely bladed crystals. Garnet is homogeneously distributed throughout the xenolith, with grain sizes of 3-7 mm, and the xenolith has an overall granoblastic texture. Kyanite is present as single bladed crystals and grain aggregates within discrete domains of fine-grained feldspar and quartz. Accessory minerals include rutile, graphite, sulfide, corundum, zircon, and monazite. Rutile follows the same morphologies and habit as the previously described xenoliths, and zircon is identified as inclusions in garnet and sapphirine.

**KX2-1 (Voorspoed):** Sample KX2-1 is a 15 cm diameter composite xenolith comprising a medium grained garnet + quartz + sapphirine + rutile assemblage, which has been infiltrated by leucocratic domains of milky white-colored antiperthitic feldspar and quartz. Garnet resorption and regrowth in contact with these leucocratic domains is apparent, accompanied by abundant retrogressive kyanite growth at the expense of sapphirine and quartz. Minor constituents include rutile, graphite, and sulfide, with accessory corundum, zircon, and monazite. The antiperthitic feldspar forms large 1-5 mm grains with pervasive micron-scale lamellae of K-feldspar. Kyanite comprises large blades, agglomerates of smaller crystals, and sheaf-like crystal sprays along the edges of leucocratic domains, and is in textural equilibrium with antiperthite. An indication of kyanite's late appearance is its relationship to rutile, which is commonly cleaved and disaggregated by clusters of small kyanite lathes. Rutile is also occluded by primary garnet, quartz, and feldspar, and forms anhedral interstitial grains. A second type of rutile is present in the form of oriented micron-scale needles in garnet and quartz. High relief inclusions identified as zircon or monazite are present in the aluminosilicates, garnet, and feldspar.

**KX2-2 (Voorspoed):** Sample KX2-2 is a 7 cm diameter xenolith consisting of equigranular granoblastic-textured submillimeter quartz, antiperthite and sillimanite with evenly distributed 2-3 cm porphyroblasts of garnet, and subordinate kyanite blades, euhedral rutile grains, red-brown titanian phlogopite tablets, graphite flakes, and sulfide blebs. While this sample does not contain sapphirine, it does contain the same rutile-zircon exsolution textures, which warrants placing this sample in the same UHT category. Orange-brown spinel is found as rare inclusions in the cores of some garnets, with quartz

and sillimanite. Exsolved rutile needles are present in garnet and quartz. Rutile is found as discrete grains occluded by garnet but more commonly within the quartz and feldspar domains. High-relief inclusions identified as zircon are observed in garnet, feldspar, sillimanite and quartz.

**KX27-1 (Star Mine):** Sample KX27-1 is an 8 cm diameter xenolith comprising a mineral assemblage dominated by garnet and quartz with minor plagioclase, and accessory graphite, sulfide, rutile, corundum, and rare zircon. The xenolith lacks a ferromagnesian phase other than garnet, and no monazite was obtained from mineral separates. Garnets range from 1-10 mm, with inclusion rich cores and relatively inclusion-free outer mantles. Quartz grains include large 3-5 mm crystals exhibiting patchy to undulose extinction, as well as smaller submillimeter interlocking grains with uniform extinction making up mosaic textured domains between larger crystals, along with plagioclase. Both garnet and quartz contain oriented rutile needles, and quartz has abundant trails of unidentified inclusions. Discrete rutile is the most abundant minor constituent, included in garnet and quartz and as interstitial grains.

**KX18-2 (Kaalvallei):** Sample KX18-2 is a very small, 3 cm diameter nodule comprising garnet, sapphirine, plagioclase, antiperthitic feldspar, quartz, sillimanite and rutile, with accessory graphite, corundum, sulfide, zircon and monazite. Garnet forms dispersed 3-5 mm porphyroblasts within a predominant matrix of 1-3 mm sapphirine, quartz, feldspar, and sillimanite crystals. Rutile is present as discrete submillimeter grains included in all major minerals and as interstitial crystals.

## **ANALYTICAL METHODS**

Zircon and monazite were isolated from xenoliths by standard crushing, heavy liquid, and magnetic separation techniques, and separated into different populations based on crystal morphology, color, and grain size. From these populations representative crystals were reserved for photomicrography. Grains were mounted in epoxy, polished, carbon-coated, and imaged by back-scattered electrons (BSE) and cathodoluminescence (CL) at the MIT JEOL 733 Superprobe electron microprobe facility. Both imaging techniques used an accelerating voltage of 15 keV, 10 nA beam current, and a 1  $\mu\text{m}$  beam spot size. High resolution gray-scale CL images were collected with the secondary electron detector mounted in place of the optical microscope ocular.

For U-Pb analysis, zircons were air-abraded with pyrite after the method of Krogh (1982), and acid rinsed in warm 3M  $\text{HNO}_3$  for 12 hours, followed by ultrasonication. All mineral fractions underwent ultrasonication in ethanol for two hours followed by rinsing in double-distilled acetone. Accessory mineral fractions were loaded into Teflon FEP microcapsules and washed again in 3M  $\text{HNO}_3$  (zircon) or high-purity water (monazite) at 50°C for 2-4 hours, followed by rinsing with several capsule volumes of water. Samples were spiked with a mixed  $^{205}\text{Pb}$ - $^{233}\text{U}$ - $^{235}\text{U}$  tracer and dissolved in 29M HF at 220°C for 48-120 hours (zircon), or 12M HCl at 180°C for 48 hours (monazite), followed by conversion to 6M HCl at 180°C for 18-24 hours. Pb and U were separated from the resulting mineral solutions using miniaturized HCl-based anion exchange chromatography procedures modified after Krogh (1973).

Pb and U were analyzed on the MIT VG Sector 54 thermal ionization multicollector mass spectrometer. Lead was loaded on single Re filaments with a dilute silica gel-0.1M  $\text{H}_3\text{PO}_4$  emitter solution and measured either dynamically with four high-

mass Faraday cups and an axial ion-counting Daly detector, peak-switching  $^{205}\text{Pb}$  into the axial position to obtain an internal Daly-Faraday gain calibration, or by peak-switching all ion beams into the Daly detector for very small amounts of lead. An ion beam of  $>0.1 \times 10^{-13}$  A was maintained for  $^{207}\text{Pb}$  during data acquisition. Uranium was loaded on single Re filaments either with colloidal graphite and measured as metal ions, or with silica gel and measured as  $\text{UO}_2^+$  ions by one of two methods: in static mode on three Faraday cups for  $^{238}\text{U}^+$  ( $^{238}\text{U}^{16}\text{O}_2^+$ ) ion-beam intensities of  $>0.5 \times 10^{-13}$  A, or by peak switching all ion beams into the Daly detector for smaller amounts of uranium. Details of fractionation and blank corrections are given in Table 2. Ages with propagated uncertainties were calculated using the methodology of Ludwig (1980).

Whole rock major element analyses were measured at the U. Massachusetts (Amherst) XRF laboratory using standard techniques, and trace element concentrations were obtained by solution ICP-MS on the VG PQ 2+S at M.I.T. For the latter, fifty milligrams of each sample powder were completely dissolved in a mix of HF-HNO<sub>3</sub>, in pressure vessels at 220°C for 5 days, converted to 7M HNO<sub>3</sub>, spiked with internal standards of Se, In, and Bi, diluted, and analyzed in triplicate. Following internal and external instrumental drift corrections, blank and isobaric interference corrections, concentrations were calibrated against USGS whole rock standards (BIR-1, BHVO-1, BCR-1, AGV-1) and are inferred to be accurate and precise to  $\pm 2$  to 5% based on replicate analyses of blind standards.

For Sm-Nd isotopic analyses, single crystals of monazite, or 50-100 mg of powdered whole rock were spiked with a mixed  $^{149}\text{Sm}$ - $^{150}\text{Nd}$  tracer and completely dissolved with 12M HCl or HF-HNO<sub>3</sub> respectively, in teflon pressure vessels at 220°C

for two or five days, followed by conversion to 6M HCl and fluxing at 120°C for 24 hours. The separation and purification of Sm and Nd were accomplished with a standard two-stage cation exchange-HDEHP reverse chromatography procedure. Sm was loaded on single Ta filaments with 1 µl of 1M H<sub>3</sub>PO<sub>4</sub> and analyzed as metal ions in static multicollector mode with a <sup>152</sup>Sm ion beam of 2.5x10<sup>-11</sup> A. Nd was loaded on triple Re filaments with 1 µl of 0.1M H<sub>3</sub>PO<sub>4</sub> and analyzed as metal ions in dynamic multicollector mode with a <sup>144</sup>Nd ion beam of 1.5x10<sup>-10</sup> A. Sm and Nd data were fractionation-corrected with an exponential law, normalizing to <sup>152</sup>Sm/<sup>147</sup>Sm = 1.783 and <sup>146</sup>Nd/<sup>144</sup>Nd = 0.7219, respectively. Details of data internal and external reproducibility are given in the caption to Table 3.

#### REFERENCES CITED

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TABLE DR1. WHOLE ROCK CHEMICAL COMPOSITIONS OF UHT GRANULITE XENOLITHS

| Sample                                      | KX1-1 | KX1-2 | KX1-8 | KX2-1 | KX2-2 | KX27-1 | Ave.<br>UHT | Ave.<br>Metapelite <sup>†</sup> | Ave.<br>Shale <sup>‡</sup> |
|---|-------|-------|-------|-------|-------|--------|-------------|---------------------------------|----------------------------|
| <b>Major elements (wt.%)</b>                |       |       |       |       |       |        |             |                                 |                            |
| SiO <sub>2</sub>                            | 52.41 | 46.44 | 60.42 | 47.30 | 64.26 | 52.38  | 53.87       | 60.50                           | 62.80                      |
| TiO <sub>2</sub>                            | 0.76  | 0.92  | 0.74  | 1.15  | 0.83  | 0.41   | 0.80        | 1.00                            | 1.00                       |
| Al <sub>2</sub> O <sub>3</sub>              | 21.87 | 24.10 | 19.87 | 24.87 | 18.55 | 18.71  | 21.33       | 17.60                           | 18.90                      |
| Fe <sub>2</sub> O <sub>3</sub> <sup>T</sup> | 12.30 | 13.67 | 9.99  | 13.91 | 7.06  | 15.50  | 12.07       | 7.84                            | 6.50                       |
| MnO   | 0.10  | 0.10  | 0.08  | 0.14  | 0.06  | 0.18   | 0.11        | 0.13                            | 0.11                       |
| MgO   | 11.29 | 13.47 | 7.35  | 9.39  | 4.51  | 8.41   | 9.07        | 3.12                            | 2.20                       |
| CaO   | 0.84  | 0.64  | 0.94  | 1.23  | 0.80  | 2.74   | 1.20        | 2.97                            | 1.30                       |
| Na <sub>2</sub> O                           | 0.08  | 0.09  | 0.26  | 0.89  | 1.35  | 0.90   | 0.60        | 2.40                            | 1.20                       |
| K <sub>2</sub> O                            | 0.52  | 0.47  | 0.68  | 1.35  | 2.68  | 0.85   | 1.09        | 1.99                            | 3.70                       |
| P <sub>2</sub> O <sub>5</sub>               | 0.03  | 0.03  | 0.01  | 0.06  | 0.02  | 0.03   | 0.03        | 0.12                            | 0.16                       |
| <b>Trace Elements (ppm)</b>                 |       |       |       |       |       |        |             |                                 |                            |
| V   | 273   | 356   | 243   | 346   | 164   | 286    | 278         | 129                             | 150                        |
| Cr  | 1608  | 2541  | 1126  | 1412  | 825   | 1224   | 1456        | 135                             | 110                        |
| Ni  | 633   | 965   | 434   | 336   | 224   | 261    | 476         | 52                              | 55                         |
| Rb  | 9.42  | 9.69  | 8.08  | 24.3  | 59.5  | 7.68   | 19.8        | 51                              | 160                        |
| Sr  | 48.1  | 34.3  | 115   | 108   | 135   | 128    | 94.8        | 331                             | 200                        |
| Y   | 33.9  | 29.4  | 30.4  | 32.3  | 23.1  | 46.3   | 32.6        | 44                              | 27                         |
| Zr  | 177   | 160   | 127   | 252   | 233   | 154    | 184         | 248                             | 210                        |
| Nb  | 12.8  | 11.9  | 18.9  | 13.2  | 15.2  | 7.2    | 13.2        | 21                              | 19                         |
| Cs  | 3.21  | 3.03  | 1.52  | 1.13  | 1.34  | 0.37   | 1.77        | 0.73                            | 15                         |
| Ba  | 150   | 171   | 252   | 273   | 390   | 218    | 243         | 730                             | 650                        |
| La  | 3.84  | 3.90  | 7.51  | 16.7  | 19.7  | 8.90   | 10.1        | 36                              | 38                         |
| Ce  | 8.22  | 6.51  | 11.1  | 34.3  | 37.1  | 13.7   | 18.5        | 71                              | 80                         |
| Pr  | 1.06  | 0.74  | 1.06  | 3.67  | 3.89  | 1.31   | 1.96        | 8.4                             | 8.9                        |
| Nd  | 5.87  | 3.82  | 4.31  | 15.09 | 13.45 | 5.58   | 8.02        | 32                              | 32                         |
| Sm  | 3.50  | 3.18  | 2.33  | 5.29  | 3.12  | 3.13   | 3.42        | 5.9                             | 5.6                        |
| Eu  | 0.52  | 0.48  | 0.96  | 0.77  | 0.83  | 0.76   | 0.72        | 1.48                            | 1.1                        |
| Gd  | 5.22  | 4.54  | 4.13  | 5.95  | 3.32  | 6.28   | 4.90        | 6.88                            | 4.7                        |
| Tb  | 0.95  | 0.82  | 0.79  | 0.99  | 0.60  | 1.28   | 0.91        | 1.05                            | 0.77                       |
| Dy  | 5.90  | 5.11  | 4.78  | 5.61  | 3.52  | 7.81   | 5.45        | 7.75                            | 4.4                        |
| Ho  | 1.20  | 1.03  | 1.04  | 1.08  | 0.77  | 1.65   | 1.13        | 1.6                             | 1                          |
| Er  | 3.30  | 2.84  | 2.96  | 2.90  | 2.20  | 4.71   | 3.15        | 4.5                             | 2.9                        |
| Tm  | 0.53  | 0.45  | 0.48  | 0.44  | 0.35  | 0.70   | 0.49        |                                 | 0.4                        |
| Yb  | 3.31  | 2.84  | 2.98  | 2.70  | 2.19  | 4.36   | 3.06        | 4.43                            | 2.8                        |
| Lu  | 0.50  | 0.43  | 0.46  | 0.40  | 0.34  | 0.61   | 0.46        | 0.67                            | 0.43                       |
| Hf  | 3.83  | 3.73  | 3.01  | 6.03  | 5.86  | 3.28   | 4.29        | 6.88                            | 5                          |
| Ta  | 0.84  | 0.77  | 1.16  | 0.65  | 0.91  | 0.36   | 0.78        | 1.3                             |                            |
| Pb  | 1.79  | 2.35  | 1.54  | 3.97  | 6.49  | 3.88   | 3.34        | 12.3                            | 20                         |
| Th  | 0.59  | 0.68  | 0.41  | 0.90  | 1.86  | 0.47   | 0.82        | 7.53                            | 14.6                       |
| U   | 0.12  | 0.22  | 0.03  | 0.32  | 0.42  | 0.47   | 0.26        | 0.58                            | 3.1                        |

*Note:* Major and trace elements measured by XRF and ICP-MS, respectively.

<sup>†</sup>Average metapelitic granulite composition from Rudnick and Fountain (1995).

<sup>‡</sup>Average post-Archean shale composition from Taylor and McLennan (1985).