Deformation and metasomatism recorded by single grain apatite petrochronology

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A. SAMPLE INFORMATION

Sample	IGSN	Lat (North)	Long (East)	Elev (m)
15-SB-05	IEDGS5605	42.814645°	1.697385°	1004
16-SB-13	IEDGS5613	42.830028°	1.736155°	1940
16-SB-14	IEDGS5614	42.830101°	1.744736°	1904

Sample 15-SB-05 is from the high-grade granulite facies gneiss in the footwall. The sample is comprised of quartz, plagioclase feldspar, garnet, biotite, sillimanite, and cordierite (Fig. S6). Sample 16-SB-14 is from the Main Mylonitic Band and is comprised of quartz, feldspar, biotite, and sillimanite (Fig. S7).

SCANNING ELECTRON MICROSCOPY METHODS

Representative apatite grains were mounted in epoxy and polished to expose an internal surface parallel to the grain's long axis. Cathodoluminescence (CL) imaging and electron backscatter diffraction (EBSD) analysis was conducted at the University of Nevada, Reno using the JEOL JSM-7100FT field-emission scanning electron microscope with an Oxford NordlysMax2 EBSD camera. Grain mounts were polished down to 0.05 micron colloidal with a vibratory polisher. EBSD data was collected at a working distance of 25 mm and 70-degree tilt using a beam energy of 25–30 kV and probe current of 18 nA. Table S1 summarizes detailed EBSD settings and acquisition parameters.

C. LA-ICP-MS MAPPING METHODS

U-Pb and TREE maps were conducted in the UTChron facilities at the Jackson School of Geosciences, University of Texas at Austin. Analysis was done on a PhotonMachine Analyte G.2 Excimer laser with a large-volume Helex sample cell and a Thermo Element2 ICP-MS. Select trace elements (Sr, Y, La, Ce, Eu, Yb, and Lu) and U-Pb were collected using LA-ICP-MS mapping techniques. Protocols are detailed in Tables S4 (TREE) and S5 (U-Pb). U-Pb ages and TREE concentrations are plotted as 2D surface interpolations using Matlab (Fig. 3, 4 main text).

For TREE analysis, a grid of spots was placed over the polished apatite grains using an 8 μm spot size, 10 Hz, and 30 shots. Elements ^{87}Sr , ^{88}Y , ^{139}La , ^{140}Ce , ^{152}Eu , ^{173}Yb , and ^{175}Lu were measured. The NIST 610 glass standard was used to correct for instrumental drift with ^{42}Ca as the internal elemental standard. TREE data reduction was performed using the 'X_Trace_Elements_IS' DRS in Iolite (Paton et al., 2011). Data can be found in Table S2 and is plotted 2D surface interpolations using Matlab (main text Fig. 3, 4).

For U-Pb analysis, a grid of spots was placed over the grains using a 12 μm spot size, 10 Hz, and 30 shots. MAD apatite (Thomson et al., 2012) was used as the primary reference standard (in-house TIMS ages of 472.4 ± 0.7 Ma) and McClure Mountain (523.5 ± 1.5 Ma; Schoene and Bowring, 2006) as a secondary reference standard. U-Pb data was reduced using Iolite data reduction software and VizualAge_UcomPbine (Chew et al., 2014). Single spot ages were corrected for Pb_c using a ^{207}Pb correction with an initial $^{207}\text{Pb}/^{206}\text{Pb}$ composition based on

the Pb evolution model of Stacey and Kramers, 1973. Data can be found in Table S3 and is plotted 2D surface interpolations using Matlab (main text Fig. 3, 4). Single grain Tera-Wasserburg plots show similar behavior to bulk sample trends (Fig. SF1, SF2 and main text Fig. 1). 15-SB-05 grain 321 yields a lower intercept U-Pb age of 103.5 ± 7.5 Ma (MSWD = 1.8; Fig. SF1), consistent with the bulk sample age and single analysis spots. 16-SB-14 grain 220 exhibits similar patterns to the bulk sample with a spread in data between the 300 and 100 Ma intercepts (Fig. SF2 and main text Fig. 1) that is characteristic of partial recrystallization (Odlum and Stockli, 2020). The single grains record the same ages as the bulk sample, including capturing the complexity of the two age domains and partial recrystallization in 16-SB-14 grain 220.

D. TABLES

Tables ST2-ST3 are attached as Excel files.

Table ST1: EBSD settings

EBSD system: Oxford NordlysMax2 Detector - Aztec				
Grain	15-SB-05 grain 334	15-SB-05 grain 321	16-SB-14 grain 202	16-SB-14 grain 220
Figure	2A, 3B	3E	2D, 4B	4E
Binning	4x4	4x4	4x4	4x4
Gain	1	1	1	1
Hough resolution	100	100	60	60
Band detection	12	12	8	8
Average mean angular deviation	0.34	0.31	0.57	0.59
Step distance (μm)	0.5	0.5	2	2
Carbon coat (nm)	6	6	6	6
Acc. Voltage (kV)	25	25	25	25
Working distance (mm)	25	25	24.5	24.5
Tilt (degrees)	70	70	70	70

Table ST4: LA-ICP-MS TREE mapping protocol

LA-ICP-MS TREE Mapping protocol	
Laboratory name:	UTChron, The University of Texas at Austin
Sample type/mineral:	Apatite grain mount
Sample preparation:	Conventional mineral separation, 1" round polished epoxy
Laser ablation system	
Make, Model & type:	Photon Machines Analyte G2
Ablation cell & volume:	HelEx cell, dual volume
Laser wavelength (nm):	193 nm
Pulse width (ns):	<4 ns
Fluence (J.cm-2):	~1.24 J/cm-2
Repetition rate (Hz):	10 Hz
Ablation duration (secs):	~3 secs
Ablation pit depth / ablation rate:	~3 µm pit depth
Spot diameter (µm) nominal/actual:	8 µm
Sampling mode:	Static spot ablation
Carrier gas:	100% He in the cell
ICP-MS Instrument	
Make, Model & type:	ThermoFischer, Element 2, HR-ICP-MS
Sample introduction:	Ablation aerosol
RF power (W):	1100-1250 W
Make-up gas:	flow Argon controlled by MFC set at 0.5 LPM
Detection system:	second electron multiplier (SEM)
Masses measured:	87, 88, 139, 140, 152, 173, 175
Data Processing	
Gas blank:	5 second on-peak zero subtracted
Calibration strategy:	NIST 610 glass with ⁴² Ca as the internal elemental standard
Reference Material info:	USGS NIST 610 (https://www.nist.gov/srmors/certificates/610.pdf)
Data processing package used /Correction for LIEF:	Iolite (Paton et al., 2011) and 'X_Trace_Elements_IS' data reduction scheme (Paton et al., 2011).
Mass discrimination:	Concentrations normalized to reference material
Common-Pb correction:	N/A
Uncertainty level & propagation:	Uncertainties of concentrations are reported tables S2 at 2-sigma.

Table ST5: LA-ICP-MS U-Pb mapping protocol

LA-ICP-MS U-Pb Mapping protocol	
Laboratory name:	UTChron, The University of Texas at Austin
Sample type/mineral:	Apatite grain mount
Sample preparation:	Conventional mineral separation, 1" round polished epoxy
Laser ablation system	
Make, Model & type:	Photon Machines Analyte G2
Ablation cell & volume:	HelEx cell, dual volume
Laser wavelength (nm):	193 nm
Pulse width (ns):	<4 ns
Fluence (J.cm ⁻²):	~1.24 J/cm ²
Repetition rate (Hz):	10 Hz
Ablation duration (secs):	~3 secs
Ablation pit depth / ablation rate:	~3 µm pit depth
Spot diameter (µm) nominal/actual:	12 µm
Sampling mode:	Static spot ablation
Carrier gas:	100% He in the cell
ICP-MS Instrument	
Make, Model & type:	ThermoFischer, Element 2, HR-ICP-MS
Sample introduction:	Ablation aerosol
RF power (W):	1100-1250 W
Make-up gas:	flow Argon controlled by MFC set at 0.5 LPM
Detection system:	secondary electron multiplier (SEM)
Masses measured:	202, 204, 206, 207, 208, 232, 235, 238
Data Processing	
Gas blank:	5 second on-peak zero subtracted
Calibration strategy:	MAD apatite as primary; McClure mountain secondary standard
Reference Material info:	MAD apatite (Thomson et al., 2012) was used as the primary reference standard (in-house TIMS ages of 472.4 ±0.7 Ma); McClure Mountain (523.5 ±1.5 Ma; Schoene and Bowring, 2006) was used as a secondary reference standard to monitor data quality
Data processing package used /	Correction for LIEF: Iolite (Paton et al., 2011) and VizualAge_UcomPbine data reduction scheme (Chew et al., 2014)
Mass discrimination:	207Pb/206Pb and 206Pb/238U normalized to reference material
Common-Pb correction:	207Pb correction applied using values from Stacey & Kramer (1975)
Uncertainty level & propagation:	Uncertainties of isotope ratios and ages are reported in tables S3 at 2-sigma. Age uncertainties are reported with only internal (measurement) errors

E. SUPPLEMENTARY FIGURES

Supplemental Figure S1

Supplemental Figure S1: Additional plots of EBSD data for apatite grains presented in the main text; Forescatter diode images show surface texture of grains. Cathodoluminescence (CL) images show internal zoning patterns. Kernel average misorientation (KAM) maps show local misorientations within grains. Grain reference orientation deviation (GROD) maps show the misorientation of each point relative to the mean orientation of the grain. Low-angle (2-10°) misorientation inverse pole figures (IPF) show the orientation of misorientation axes in crystal coordinates.

Supplementary Figure S2

Mylonitic footwall gneiss

Supplementary Figure S2: EBSD data for additional apatite grains from 15-SB-05, the mylonitic gneiss, analyzed in this study. Forescatter diode images show surface texture of grains. Cathodoluminescence (CL) images show internal zoning patterns. Kernel average misorientation (KAM) maps show local misorientations within grains. Grain reference orientation deviation (GROD) maps show the misorientation of each point relative to the mean orientation of the grain. Low-angle (2-10°) misorientation inverse pole figures (IPF) show the orientation of misorientation axes in crystal coordinates.

Supplementary Figure S3

Main Mylonitic Band

Supplementary Figure S3: EBSD data for additional apatite grains from the Main Mylonitic Band (samples 16-SB-14 and 16-SB-15 from this study and Odlum & Stockli, 2020) analyzed in this study. Forescatter diode images show surface texture of grains. Cathodoluminescence (CL) images show internal zoning patterns. Kernel average misorientation (KAM) maps show local misorientations within grains. Grain reference orientation deviation (GROD) maps show the misorientation of each point relative to the mean orientation of the grain. Low-angle (2-10°) misorientation inverse pole figures (IPF) show the orientation of misorientation axes in crystal coordinates.

Supplementary Figure S4

Supplemental Figure S4: Single grain Tera-Wasserburg plot for 15-SB-05 grain 321. Single grain age from U-Pb mapping techniques is consistent with bulk sample ages (109.8 ± 2.8 Ma; main text Fig. 1D).

Figure S5

Supplemental Figure S5: Single grain Tera-Wasserburg plot for 16-SB-14 grain 220. Single grain age from U-Pb mapping techniques is consistent with bulk sample patterns and age ranges (main text Fig. 1).

Supplemental Figure S6

Supplemental Figure S6: Thin section photo micrographs of 15-SB-05 in PPL (A) and XPL (B).

Supplemental Figure S7

Supplemental Figure S7: Thin section photo micrographs of 16-SB-14 in PPL (A) and XPL (B).

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