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Unraveling alteration timing in serpentinites and associated ultramafic rocks with magnetite (U-Th)/He geochronology

Emily H. G. Cooperdock[1] and Daniel F. Stockli[1]
[1]*Department of Geological Sciences, University of Texas at Austin, Austin, TX 78712-1692, U.S.A.*

MAGNETITE TRACE ELEMENT MEASUREMENT

Trace elements (Mg, Al, Ti, V) were measured on magnetite grains from three 60 μm-thick thin sections of the serpentinite. Laser spot analyses were measured on all visible magnetite grains of sufficient size from each thin section as either transects across the grain (7 grains on one thin section of the serpentinite) or as random points (16 grains on two thin sections of the serpentinite). The data from these measurements are reported in the manuscript Fig. 3. (See Supplement Table DR1 for LA-ICP-MS data and Supplement Fig. DR1 for additional grain transect data). Measurements were made on a single collector ThermoFisher Element II HR-ICP-MS with an attached Photonmachines Analyte G2 193 nm ArF excimer laser ablation system. The laser settings included a 70% laser energy, 6.35 J/cm^2 fluence, and 6 mJ energy. Spot sizes of 65 μm were employed, and all spots were pre-ablated. GSD, the primary standard, was run between every four unknowns, and GSC was run between every 12 unknowns as the secondary standard. Analyses for each element in GSD (n = 29) and GSC (n=11) are reproducible within < 5% over the experiment, with the exception of V (25%) in GSC. Concentrations were calculated using Fe as an internal standard on Igor Pro Iolite software.

MAGNETITE PREPARATION AND (U-TH)/HE MEASUREMENT

Whole rock samples were crushed and magnetite crystals were separated from the crushed whole rock separate by hand magnet. Individual magnetite grains were picked based on size and morphology using an optical microscope (see Supplement Fig. DR2 for photomicrographs of thin sections). Single, euhedral magnetite grains greater than one millimeter were selected from the chlorite schist and crushed with mortar and pestle. Internal fragments without clear crystal faces were selected for CT-scanning. Since grain sizes ranged from hundreds of micrometers to over one millimeter in the serpentinite, two grain treatments were employed. Magnetite grains over a millimeter were crushed with mortar and pestle and internal fragments with no clear crystal faces were selected for CT-scanning. Magnetite grains between ~300-600 μm were hand picked based on euhedral morphology. These sub-mm grains were physically air abraded by placing 20-40 similarly sized grains into an air abrasion vessel at once and abrading at 6 psi for about 4 hours following the procedure reported in Blackburn et al., (2007). The abrasion conditions employed in this study are based on previous experiments that calibarated the pressure and time to effectively remove ~ 20 μm from the outside of magnetite.

Internal fragments and abraded grains were scanned using the X-Radia Micro-CT scanner at the University of Texas High-Resolution Computed Tomography Facility (UTCT). In order to fit within the 5 mm^3 scanning volume, 10-50 grains or internal fragments were scanned in a single run at ~ 6 μm resolution. The X-Ray CT data were used to screen grains and fragments for attached matrix material (serpentine or chlorite), and inclusions, which are otherwise impossible to see on a 3D scale in opaque minerals. (See Supplement Fig. DR3 for an example of CT data).

Individual aliquots were constructed using 2-7 internal fragments (from a single large crystal when possible), or abraded grains of similar size. The fragments or abraded grains that were clear of inclusions were wrapped into platinum tubes and laser heated with a Photonmachine Diode Laser at a pre-specified pyrometer temperature for 10 min. ^{4}He amounts were measured on a Blazers Prisma QMS-200 quadrupole mass spectrometer (QMS) by "spiking" with a ^{3}He internal isotopic reference standard. Gas was purified in a Janis cryogenic trap, and a SAES NP10 getter before entering the QMS. Aliquots were reheated until the He yield became ≤ 2x blank values measured after each unknown. Final ^{4}He amounts were calculated using a calibration against a manometrically-determined ^{4}He standard. Gas blanks were measured between each aliquot to determine the background and drift during the run. Final gas amounts are all blank corrected corresponding to the blank following gas extraction of an unknown.

Samples were dissolved for U, Th, and Sm measurement using a two-step HF-HNO_3 and HCl hot plate dissolution procedure. After aliquots were unpacked from platinum tubes into Savillex beakers, a ^{238}U, ^{230}Th, and ^{149}Sm spike in 5% HNO_3 was added. A 5:1 concentrated HF - 7N HNO_3 mixture was added to the beakers, which were tightly sealed and heated at ~180°C overnight. Samples were dried down to a small bead and 200 µl of concentrated HCl was added to the beaker, which was tightly sealed and heated overnight at 180°C. After chloride conversion, the solution was dried down to a small bead and 100 µl of 7N HNO_3, followed by 500 µl of MilliQ H_2O were added in preparation for analysis on the Thermo Element 2 HR-ICP-MS. Final U-Th-Sm concentrations were calculated using isotope dilution with a mixed spike calibrated against a gravimetric 1 ppb U-Th-Sm standard solution.

SUPPLEMENT TABLES

TABLE DR1. LA-ICP-MS SPOT ANALYSES ON MAGNETITE GRAINS FROM SERPENTINITE SAMPLE

Grain	Grain size (µm)	# of spots	Mg (ppm)	SD*	Ti (ppm)	SD*	V (ppm)	SD*	Al (ppm)	SD*
Random Spot Analysis										
1	523	2	455	52	78	1	427	59	23	3
2	597	2	470	4	84	3	475	1	24	1
3	726	2	512	87	86	3	537	1	27	5
4	313	1	436	19	73	4	388	17	19	2
5	296	1	345	16	83	4	324	14	17	2
6	504	2	416	16	69	7	438	49	17	2
7	1039	3	575	53	183	89	543	36	35	7
8	506	1	466	68	55	1	420	5	18	1
9	768	2	490	18	54	.4	358	47	20	2
10	4202	11	755	123	178	91	591	56	60	16
11	606	2	652	136	72	5	453	55	27	1
12	949	3	643	98	190	58	582	32	37	6
13	1218	4	806	221	123	56	549	71	43	22
14	5734	11	736	199	172	81	597	59	64	18
15	619	2	551	30	85	7	497	28	27	4
16	681	3	523	44	89	18	492	41	23	5

Grain-Spot #	Grain size (µm)	Mg (ppm)	SD*	Ti (ppm)	SD*	V (ppm)	SD*
Grain Transect Analysis							
1-1	1858	333	18	59	8	315	14
1-2		635	51	180	42	505	25
1-3		497	29	131	6	607	30
1-4		862	45	267	43	671	37
1-5		1063	52	167	19	715	34
1-6		644	26	153	24	647	23
1-7		612	61	80	11	535	36
2-1	487	365	17	52	5	309	8
2-1		589	26	48	4	384	12
3-1	1518	536	64	65	7	359	24
3-2		461	19	67	4	442	14
3-3		483	22	101	11	528	17
3-4		446	20	132	19	580	15
3-5		472	19	153	31	620	17

Grain-Spot #	Grain size (µm)	Mg (ppm)	SD*	Ti (ppm)	SD*	V (ppm)	SD*
3-6		644	29	180	42	652	20
3-7		875	32	140	13	688	21
3-8		991	31	124	9	703	20
3-9		1339	57	122	8	707	22
3-10		1030	43	157	20	719	25
3-11		960	34	137	11	687	24
3-12		904	40	132	11	673	24
3-13		743	35	143	15	620	22
3-14		529	22	107	12	568	18
4-1	5500	679	57	197	47	641	20
4-2		554	26	110	16	663	24
4-3		752	25	114	6	70	21
4-4		1053	39	139	12	717	30
4-5		1125	40	150	21	747	26
4-6		1118	51	137	19	764	32
4-7		1151	47	107	8	773	31
4-8		1191	50	188	73	794	36
4-9		1136	34	234	78	780	23
4-10		1132	35	117	9	768	22
4-11		1092	28	236	82	749	12
4-12		961	39	140	13	735	21
4-13		956	28	298	67	716	22
4-14		869	32	188	85	723	21
4-15		904	39	179	49	715	29
4-16		829	48	116	8	700	36
4-17		658	35	140	17	645	32
4-18		1260	120	71	6	401	23
5-1	2021	432	20	73	8	473	22
5-2		573	32	152	49	557	26
5-3		484	26	118	8	610	24
5-4		520	24	117	7	622	25
5-5		599	29	138	16	638	28
5-6		950	260	100	9	608	39
5-7		707	37	116	9	639	28
5-8		665	27	106	9	634	30
5-9		585	28	198	54	646	30
5-10		689	25	115	8	627	26
5-11		609	33	625	40	549	27
5-12		475	22	91	7	495	22
6-1	796	480	24	79	5	539	23
6-2		985	38	305	27	635	24
6-3		1075	38	148	10	682	22
6-4		1006	34	672	38	663	21

Grain-Spot #	Grain size (µm)	Mg (ppm)	SD*	Ti (ppm)	SD*	V (ppm)	SD*
6-5		604	27	129	8	549	19
7-1	2003	452	24	80	6	471	21
7-2		599	30	364	83	595	30
7-3		690	29	130	10	620	29
7-4		807	37	290	150	653	24
7-5		698	25	223	24	639	21
7-6		616	30	163	14	648	26
7-7		521	28	223	73	594	29
7-8		427	25	104	6	554	23

* Reported SD errors are the standard deviation of averaged laser spot analyses. If n=1, reported errors are standard error on the individual analyses.

TABLE DR2. (U-Th)/He AGE DATA FOR INDIVIDUAL ALIQUOTS

Aliquot name	Age (Ma)	U (ppb)	Th (ppb)	Sm (ppb)	^{4}He (nmol/g)	Th/U	Mass (μg)	# of grains or fragments
<u>Serpentinite</u>								
Multi-grain aliquots of individually abraded grains (300-600 μm)								
mg13KA03-1	3.3 ± 0.1	4	247	11	0.0011	56	736	3
mg13KA03-5	3.4 ± 0.1	5	488	32	0.0022	90	516	3
mg13KA03-7	2.9 ± 0.5	5	18	2	0.0001	3	946	3
mg13KA03-8	4.3 ± 0.4	3	50	3	0.0004	15	824	4
mg13KA03ab-1	2.7 ± 0.3	6	156	12	0.0006	27	825	4
mg13KA03ab-4	1.8 ± 0.7	8	17	4	0.0001	2	948	3
mg13KA03ab-7	2.4 ± 0.2	7	65	10	0.0003	9	1114	3
mg13KA03ab-11	3.6 ± 1.6	3	9	4	0.0001	3	960	2
mg13KA03ab-14	3.3 ± 0.6	6	54	7	0.0003	9	724	7
Internal fragments from single large grain (≥ mm)								
mg13KA03ab-28	8.8 ± 0.8	3	32	12	0.0005	11	2377	4
mg13KA03ab-29	15.3 ± 1.8	2	7	2	0.0003	3	1078	4
mg13KA03ab-30	6.8 ± 1.2	11	39	17	0.0007	4	2487	7
<u>Chlorite schist</u>								
Internal fragments from large grains (≥ mm)								
mg07SY09-1	13.0 ± 1.8	6	6	698	0.0010	0.9	315	4
mg07SY09-3	21.0 ± 1.1	26	15	1045	0.0043	0.6	826	3
mg07SY09-8	13.2 ± 0.5	27	53	1425	0.0037	2	576	3
mg07SY09-11	20.1 ± 0.7	21	16	174	0.0028	0.8	1452	3
mg07SY09-14	16.0 ± 0.2	43	217	3480	0.0106	5	705	3
mg07SY09-17	13.5 ± 0.1	84	412	6186	0.0170	5	582	3
mg07SY09-21	15.5 ± 0.6	5	15	828	0.0013	3	894	2
mg07SY09-20	43.7 ± 0.5	27	13	1479	0.0099	0.5	223	3
mg07SY09-22	45.0 ± 0.5	13	12	592	0.0051	0.9	2205	7
mg07SY09-23	30.5 ± 0.9	3	6	144	0.0009	2	1302	2

Mass refers to the mass of the aliquot, not individual grains. *# of grains or fragments* refers to the number combined into a single aliquot.

SUPPLEMENT FIGURES

Fig. DR1: Additional LA-ICP-MS transects measuring Mg, V and Ti across four magnetite grains from the serpentinite. Each symbol represents a laser spot (65 µm spot size).

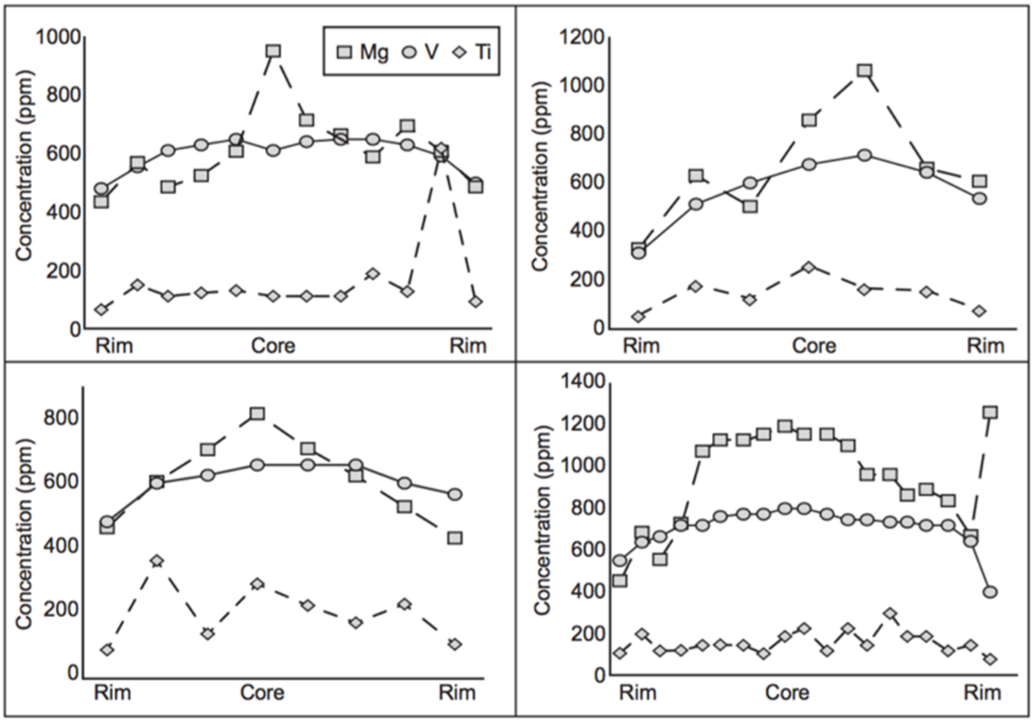

Fig DR2.: Photomicrographs of serpentinite and chlorite schist thin sections. A- C are serpentinite, D is chlorite schist.

A) Plane polarized light through talc-rich section of serpentinite. General mineralogy: serpentine, talc, magnetite and minor chlorite.

B) Cross-polarized light for the same thin section as A

C) Second scanned thin section of serpentinite in less talc-rich section

D) Chlorite schist thin section. Large magnetite are euhedral. Other opaque mineral is ilmenite. General mineralogy: chlorite, epidote, rutile, ilmenite, magnetite, apatite and < 6 µm zircon.

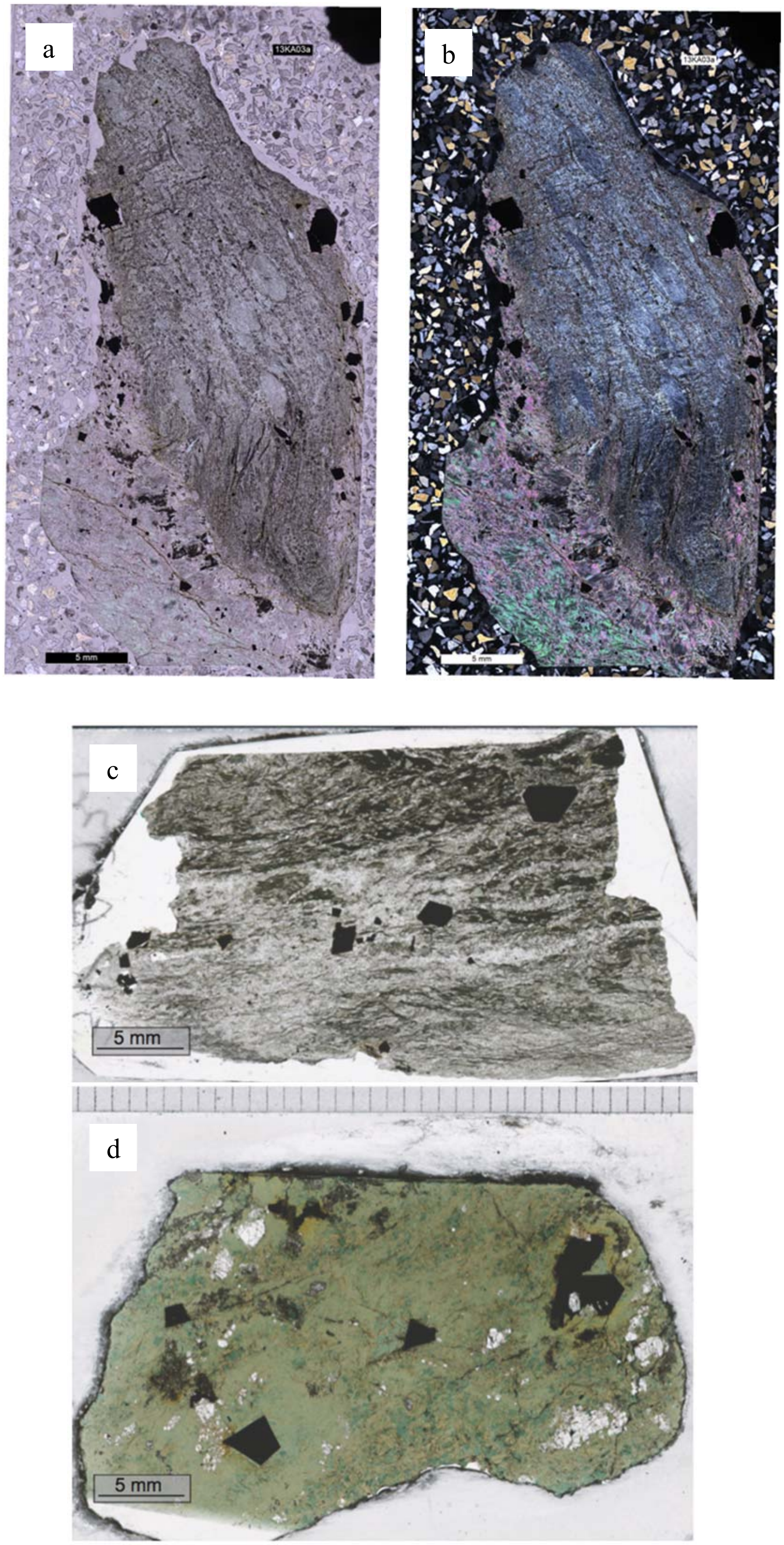
a
13KA03a
5 mm
b
13KA03a
5 mm
c
5 mm
d
5 mm

Fig. DR3 (scale bar is 1 mm): Example of CT Data: a) Optical microscope photograph of unabraded magnetite grains mounted on double-sided sticky tape in prepartation for CT scanning. b) 3-D isosurface rendering of magnetite grains constructed using the X-Ray CT data and Avizo software. c) Volume rendering of magnetite grains with inclusions that have a different density from the magnetite. Inclusions are highlighted in bright pink. The grains with these inclusions were avoided for analysis.