LAACHER SEE REVISITED: HIGH SPATIAL RESOLUTION ZIRCON DATING IMPLIES RAPID FORMATION OF A ZONED MAGMA CHAMBER - DATA REPOSITORY

ANALYTICAL PROCEDURES

Ion microprobe U-Th measurements

Th-U dating was performed on individual zircons using the UCLA Cameca ims 1270 secondary ion mass spectrometer (SIMS, ion microprobe), modifying a technique from Reid et al. (1997). A mass-filtered $^{16}O^-$ beam was focused into a ~35 × 30 µm oval spot. Primary beam intensity was regulated to a current between 20 and 40 nA, in order to achieve $^{232}ThO^+$ count rates >10^6 cps. Secondary ions were accelerated at 10 keV with an energy bandpass of 50 eV and analyzed at a mass resolution of M/ΔM ~4,800 using an axial electron multiplier collector in peak jumping mode. Th/U relative sensitivity was calibrated from $^{238}UO^+/^{232}ThO^+$ and radiogenic $^{206}Pb/^{208}Pb$ measurements of concordant reference zircons AS3 and 91500 (Paces and Miller, 1993; Wiedenbeck et al., 1995): session 1 (June 07 2005): 1.12±0.01; session 2 (June 15 2005): 1.09±0.02; session 3 (September 21-22 2005): 1.11±0.03. Raw intensities were corrected for electron multiplied dead-time (25 nsec), and background corrections on $^{230}ThO^+$ were performed using the averaged intensities measured on two mass stations at 244.029 and 246.3 amu. Mass 244.029 ($^{232}ThC^+$) is also monitored as a proxy for $^{232}Th_2CO^{2+}$ isobaric interference (resulting from epoxy overlap) on $^{230}ThO^+$, but no elevated 244.029 intensities were found. Throughout analytical sessions, reference zircons were intermittently analyzed along with the unknowns and the following (^{230}Th)/(^{238}U) values were obtained: session 1: 0.991±0.056 (n = 1); session 2: 0.994±0.028 (MSWD = 1.2, n = 2); session 3: 1.010±0.016 (MSWD = 0.51; n = 11). U and Th abundances were calculated relative to $^{238}UO^+/^{92}Zr^{94}ZrO_4^+$ measured on zircon 91500 with a U content of 81.2 ppm (Wiedenbeck et al., 1994).

Ion microprobe U-Pb measurements

U-Pb ages were obtained using the UCLA CAMECA ims 1270 ion probe. Samples that had been coated with ~10 nm of Au were probed with a mass-filtered, ~10 nA $^{16}O^-$ beam focused to a ~20 × 25 µm diameter spot. Secondary ions were extracted at 10 kV with an energy band-pass of 50 eV. The mass spectrometer was tuned to a mass resolution of M/ΔM ~5,000 to resolve relevant molecular interferences in the mass range analyzed ($^{94}Zr_2O^+$, $^{204}Pb^+$, $^{206}Pb^+$, $^{207}Pb^+$, $^{208}Pb^+$, $^{238}U^+$, $^{232}Th^{16}O^+$, $^{238}U^{16}O^+$). Pb^+ yields were increased by a factor of ~2 by flooding the analysis surface with O_2 at a pressure of ~4 x10^{-3} Pa. The relative sensitivities for Pb and U were determined on reference zircon AS3 (Paces and Miller, 1993). Precision and accuracy of U-Pb ages is estimated from standard deviations of replicate zircon standard measurements and comparison with 91500 zircon ages to within 2 - 3 % (1σ). Th and U contents were calculated from $^{232}Th^{16}O^+/^{94}Zr_2O^+$ and $^{238}U^{16}O^+/^{94}Zr_2O^+$ ratios, respectively, compared to reference zircon 91500 (Th = 28.6 ppm; U = 81.2 ppm; Wiedenbeck et al., 1995). Ages were calculated following a ^{204}Pb-based common-Pb correction using anthropogenic Pb compositions (Sañudo-Wilhelmy and Flegal, 1994).

Ion microprobe oxygen isotopic measurements

Oxygen isotope measurements were made with the CAMECA ims1270 ion microprobe at UCLA. Two mounts were analyzed in subsequent sessions on August 24 (session 1) and August 25 2005 (session 2) after completion of U-Th analysis and regrinding and repolishing that removed several µm of the surface contaminated from previous $^{16}O^-$ ion beam bombardment. A ~15 × 20 µm oval Cs^+ primary beam with a primary intensity of ~3 nA was used. Secondary ion intensities for $^{16}O^-$ and $^{18}O^-$ were simultaneously collected in two Faraday cups (FC) set-up for a mass resolving power of ~2000, sufficient for resolving hydride interferences from the $^{18}O^-$ peak. In addition, a liquid nitrogen cold trap was used to suppress H-species in the vacuum of the sample chamber. Following a ~3 min pre-sputtering to remove potentially contaminated surface material, counts were accumulated for 100 seconds. Under these conditions, ~3.5 × 10^6 cps were typically detected for the minor isotope. Raw intensities were corrected for FC relative yields and backgrounds, which were monitored intermittently throughout the analysis period. Background corrections amount to typically <0.4‰. Instrumental mass fractionation (IMF) was calibrated from bracketing measurements of 91500 zircon ($\delta^{18}O$ = 10.1; Valley, 2003) where IMF = 1.007±0.005 (session 1; 1 standard deviation; n = 5) and 1.007±0.003 (session 2; n = 10). Since no drift or difference in IMF was found between both sessions, the average IMF was used for the correction of unknowns, and the standard deviation of all 91500 measurements (0.4 ‰; n = 15) was adopted as an estimate for the uncertainty of $\delta^{18}O$ of the unknowns.

Ion microprobe rare earth element (REE) measurements

REE measurements were performed using the CAMECA ims1270 ion microprobe at UCLA with a mass-filtered ~20 nA $^{16}O^-$ beam (October 28 2005). Sample mounts were slightly reground and repolished to remove Cs spots from previous oxygen isotopic analysis. The instrument was operated at moderate mass resolution (~1500) using a 100 eV energy offset with a 30 eV bandpass. REE intensities were normalized to $^{30}Si^+$ and corrected for electron multiplier dead-time (25 nsec) and interfering oxides. Relative sensitivities were calculated from NBS 610 glass using Pearce et al. (1995) values. For assessing accuracy and precision, averages and standard deviations of three replicate measurements of zircon 91500 are reported.

REFERENCES

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Table DR 1: U-Pb results for Laacher See zircons by ion microprobe analysis

Sample grain-spot	$^{206}Pb/^{238}U^*$	$^{207}Pb/^{235}U^*$	Correlation	$^{207}Pb/^{206}Pb^*$	$^{204}Pb/^{206}Pb$	^{206}Pb (% radiogenic)	$^{206}Pb/^{238}U$ (Ma)	$^{207}Pb/^{235}U$ (Ma)	$^{207}Pb/^{206}Pb$ (Ma)	Concentration U (ppm)	Th (ppm)
LS0501 - composite pumice											
3 - 1	0.212 ± 0.012	2.54 ± 0.15	0.94	0.0869 ± 0.0019	0.000291 ± 0.000093	99.5	1241 ± 63	1284 ± 44	1357 ± 41	90	40
4 - 1	0.348 ± 0.018	8.68 ± 0.46	0.99	0.181 ± 0.001	0.000074 ± 0.000017	99.9	1923 ± 86	2305 ± 48	2664 ± 13	270	60
5 - 1	0.267 ± 0.013	3.68 ± 0.19	0.97	0.100 ± 0.001	0.000107 ± 0.000025	99.8	1523 ± 66	1567 ± 40	1627 ± 22	340	130
6 - 1	0.0632 ± 0.0036	0.479 ± 0.030	0.94	0.0550 ± 0.0012	0.000293 ± 0.000061	99.5	395 ± 22	398 ± 21	414 ± 48	480	230
7 - 1	0.273 ± 0.014	6.01 ± 0.32	0.97	0.160 ± 0.002	0.000039 ± 0.000023	99.9	1556 ± 72	1977 ± 47	2452 ± 22	210	70
8 - 1	0.302 ± 0.018	4.19 ± 0.25	0.98	0.101 ± 0.001	0.000021 ± 0.000011	100.0	1702 ± 87	1673 ± 49	1636 ± 22	310	300
9 - 1	0.277 ± 0.016	3.88 ± 0.23	0.98	0.102 ± 0.001	0.000073 ± 0.000033	99.9	1576 ± 79	1609 ± 47	1653 ± 22	150	160
10 - 1	0.0617 ± 0.0031	0.455 ± 0.025	0.91	0.0535 ± 0.0013	0.000312 ± 0.000063	99.4	386 ± 19	381 ± 18	350 ± 53	630	470
11 - 1	0.0725 ± 0.0043	0.574 ± 0.038	0.93	0.0575 ± 0.0014	0.000221 ± 0.000056	99.6	451 ± 26	461 ± 24	509 ± 53	570	70
13 - 1	0.214 ± 0.014	2.27 ± 0.15	0.98	0.0770 ± 0.001	0.000101 ± 0.000034	99.8	1248 ± 72	1203 ± 45	1122 ± 27	210	100
14 - 1	0.264 ± 0.014	3.46 ± 0.19	0.97	0.0951 ± 0.0013	0.000026 ± 0.000012	100.0	1511 ± 69	1518 ± 43	1529 ± 26	340	160
15 - 1	0.256 ± 0.013	3.37 ± 0.17	0.97	0.0955 ± 0.0011	0.000065 ± 0.000023	99.9	1471 ± 66	1498 ± 39	1537 ± 22	260	180
20 - 1	0.306 ± 0.024	3.86 ± 0.33	0.97	0.0915 ± 0.0021	0.000067 ± 0.000036	99.9	1721 ± 116	1605 ± 70	1457 ± 44	440	140
21 - 1	0.336 ± 0.026	4.56 ± 0.35	0.97	0.0985 ± 0.0019	0.000112 ± 0.000051	99.8	1866 ± 125	1742 ± 64	1596 ± 37	180	90

Note: All errors 1σ.

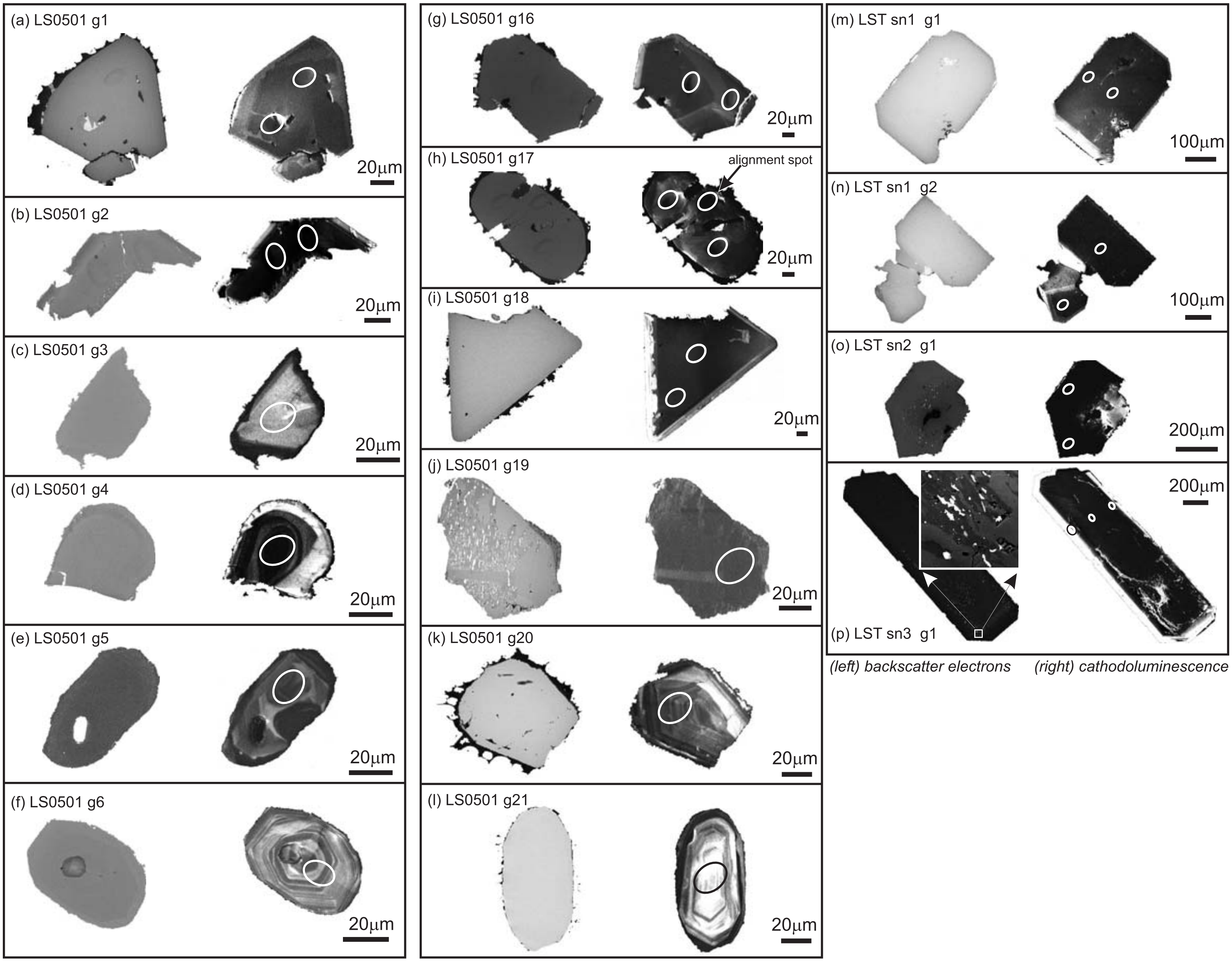
Table DR 2: Rare earth element results for Laacher See zircons by ion microprobe analysis

Sample	LS0501	LS0501	LS0501	LS0501	LST sn 2	LST sn 2	LST sn 3	LST sn 3	91500*
grain	1	2	16	17	1	1	1	1	average (n = 3)
spot	1	1	1	1	1	2	1	2	
La	0.14	29	0.27	0.05	3.2	1.2	0.66	2.4	0.028 ± 0.003
Ce	162	3047	415	27	992	1081	1107	845	2.8 ± 0.1
Pr	0.7	41	1.8	0.06	7.6	4.6	3.5	4.2	0.018 ± 0.001
Nd	7.4	287	19	0.7	67	48	37	41	0.20 ± 0.02
Sm	6.5	148	17	0.6	50	49	38	34	0.32 ± 0.03
Eu	1.7	53	4.4	0.25	18	20	16	13	0.18 ± 0.01
Gd	15	171	47	1.3	142	153	135	91	1.7 ± 0.02
Tb	10	91	26	1.3	53	75	71	46	0.75 ± 0.05
Dy	127	847	318	17	619	933	919	546	9.9 ± 0.4
Ho	57	268	140	8.3	258	412	417	234	4.4 ± 0.2
Er	286	1069	690	46	1258	2062	2113	1146	22 ± 0.2
Tm	74	220	171	13	326	551	557	298	6.0 ± 0.04
Yb	725	1433	1612	140	3226	5667	5484	2938	60 ± 0.6
Lu	156	149	335	35	643	1130	1092	583	12.8 ± 0.4

Note: all values in ppm

* average and standard deviation of three replicate measurements on 91500 reference zircon

DR Fig. 1: Backscatter and cathodoluminescence images of Laacher See zircons (ion microprobe U-Th spot locations indicated)



(left) backscatter electrons *(right) cathodoluminescence*