Appendix 1

Chronology of Pluton Emplacement and Regional Deformation in the Southern Sierra Nevada Batholith, California-Supplementary Data and Discussions

Saleeby, J., Division of Geological and Planetary Sciences, California Institute of Technology, Pasadena CA 91125
Ducea, M.N., Department of Geosciences, University of Arizona, Tucson AZ 85721
Busby, C., Department of Geosciences, University of California, Santa Barbara CA 93106
Nadin, E., Division of Geological and Planetary Sciences, California Institute of Technology, Pasadena CA 91125
Wetmore, P.H., Department of Geology, University of South Florida, Tampa, FL 33620

Introduction

Supplementary data presented here include isotopic and concentration data for U/Pb zircon ages performed by isotope dilution and thermal ionization mass spectrometric techniques (ID-TIMS), and for feldspar common lead composition determinations used for nonradiogenic corrections of the zircon data (Tables SD1 and SD2, respectively). Supplementary discussions include an overview of the techniques employed for the ID-TIMS technique as well as the single crystal laser ablation ICP multicollector mass spectrometric technique (LA-ICP-MS).

Analytical techniques

Zircon Preparation

Batholithic zircon was separated from 1-5 kg rock samples by standard pulverizing, density contrast, and magnetic susceptibility techniques. Magnetic separation procedures were aimed towards maximum sample purity and selection of highest integrity grains. Non- or paramagnetic zircon was taken from Franz Isodynamic Separator runs with a front slope of 20° and a side slope of either 0° or -1°. After initial washing in 8N, and sieving in expendable nylon sieves, the fractions to be analyzed were picked to 100 percent purity by inspection of each grain. Only well-faceted clear euhedral grains lacking visible inclusions or cores were accepted. In the early stages of this study some silicic volcanic and hypabyssal samples from the Erskine Canyon sequence that ranged up to 50 kg were used for zircon separations. Separation procedures for these were the same as above, although handpicking was done at a level that could only assure ~99 percent purity, and inspection for grain integrity was not as rigorous. These early samples included E2, E3, E5 and E6.

Select samples for which there was concern for disturbance by superimposed thermal events (E5, D2 and D13) were mechanically abraded to a well rounded state prior to dissolution by techniques similar to Krogh (1982), but with the addition of fine pyrite as an abrading agent. These were given and additional concentrated HNO_3 wash prior to introduction into dissolution capsules. Feldspar separates were split from the separation procedures of samples B2, N4 and D10 for initial lead determinations.

U/Pb Zircon Isotope Dilution Analyses

Zircon separates were weighed into a TFE bomb capsule and then given an additional 15-minute warm wash with ultrapure concentrated HNO_3. Dissolution was performed in concentrated ultrapure HF with a drop of ultrapure concentrated HNO_3 at 225°C for 3 days. Following dissolution, the samples were evaporated and rebombed overnight in ultrapure 6N HC1 prior to spiking. Samples were then spiked with a mixed ^{205}Pb/^{235}U tracer, and equilibration was obtained in hot 6N HCL within a sealed PFA container overnight on a hotplate.

Dissolution and chemical extraction techniques for zircon entailed cycles of 2N, 3N and 6N HCl, similar to those described in Krogh (1973). Mass spectrometry was performed on a VG Sector multicollector instrument. Pb and U were run on outgassed Re single filaments with silica gel and graphite loads, respectively. Pb was ionized at ~1400°C and U at ~2000°C, yielding typical ion beam currents of 1 to 5 x 10^{-11} A. Regular inter-calibrations of the multiple detector system yielded stabilities at the 10-ppm level for time periods typically in excess of several hours, and thus Pb and U were run in a static multicollector mode. ^{206}Pb/^{204}Pb ratios were measured with the ^{204}Pb beam directed into a Daly deflection knob-photomultiplier system. The gain factor was stable within 5 per mil over the course of the Pb runs. A 0.13±0.05/amu mass fractionation correction was applied to both U and Pb runs based on replicate analyses of NBS 982, 983 and U500 standards. Samples E2, E3, E5 (non-abraded) and E6 were measured during the early stages of this study on the Caltech Lunatic II single collector thermal ionization mass spectrometer in peak jumping mode. Pb was ionized at ~1150°C yielding a stable ion beam current of 1 to 5 x 10^{-11} A for a period of 2-3 hours, and U at ~1700°C, yielding typical ion beam currents of 1 to 5 x 10^{-11} A stable for periods of ~ 2 hours. A 0.11±0.07/amu mass fractionation correction was applied to U, and 0.12±0.09/amu for Pb runs was applied based on replicate analyses of NBS 982, 983 and U500 standards.

Nonradiogenic lead corrections were based on 25 picogram blank Pb measured at 1:18:78:15:61:38:50 and initial Pb approximations from feldspar determinations (Table

SD2). The D1 initial lead values were used for the Erskine Canyon sequence and the Kern River suite samples. The B2 initial lead values were used for Bear Valley suite samples, the N4 values for Needles suite samples, and the D10 values for the Domelands and South Fork suite samples.

Decay constants used in age calculations are λ^{238}U = 1.55125 x 10^{-10} and λ^{235} U = 9.8485 x 10^{-10} (Jaffey and others, 1971). ^{235}U/^{235}U atom = 137.88 is after Chen And Wasserburg (1981). Uncertainties in 206Pb*/238U U and ^{206}Pb* are given as ± in last two figures. These uncertainties were calculated by taking the quadratic sum of the total derivatives of ^{238}U and radiogenic ^{206}Pb concentration, and the radiogenic ^{207}Pb/^{206}Pb equations with the respective error differentials defined as: 1. Isotopic ratio determinations from standard errors of mass spectrometer runs plus uncertainties in fractionation corrections based on multiple runs of NBS 981, 982, 983 and U500 standards; 2. spike concentrations from range of deviations in multiple calibrations with normal solutions; 3. spike compositions from external precisions of multiple isotope ratio determinations; 4. uncertainty in natural ^{238}U/^{235}U from Chen and Wasserburg (1981); and 5. nonradiogenic Pb isotope composition uncertainties in isotope ratio determinations of blank Pb and uncertainties in the isotopic compositions of initial Pb determinations (Table SD2). The error propagation encompasses systematic bias as well as precision factors. Concordia plots presented in Figures 4, 5 and 8 (after Ludwig, 2001) are presented as 2 sigma uncertainties which are nearly identical to the uncertainties shown in Table SD1, and calculated as above.

Feldspar Nonradiogenic Lead Analyses

Powdered feldspar separates were weighed into a TFE bomb capsule and given a warm wash for 15 minutes in ultrapure 4 N HNO_3. Dissolution was complete in concentrated ultrapure HF with 1 drop of ultrapure HNO_3 at 225°C in 24 hours. The solution was evaporated and the feldspar cake was treated with several drops of $HClO_4$ and warmed in the open capsule, evaporated, redissoloved in 6 N HCl and then aliquoted into PFA screw top capsules. One was spiked with a mixed ^{208}Pb-^{235}U-^{230}Th tracer and equilibrated on a hot plate over night. Both aliquots were evaporated, and then redissolved in 6 N HCl-1 N HBr solution. Lead extractions were performed by standard HCl and HBr cycles (cf. Chen and Tilton, 1991), and U-Th extractions were performed for spiked aliquots in HNO_3-HCl cycles. Lead, U and Th were analyzed on VG Sector multicollector thermal ionization mass spectrometer, as above, except Pb was analyzed in a dynamic multicollector peak switching mode while U and Th in static mode.

U/Pb Zircon Laser Ablation Analyses

Select zircon grains from coarse fractions (80-120μ) were mounted in 2.5 cm epoxy mounts and ablated with a New Wave DUV193 Excimer laser, operating with a wavelength of 193 nm and a spot diameter of 35–50 microns. Each grain analysis consists of a single 20-second integration on isotope peaks without laser-firing to obtain on-peak background levels, 20 one-second integrations with the laser firing at the center of each grain, followed finally by a 30-second purge with no laser firing to deliver the remaining evacuated sample. Hg contributions to ^{204}Pb were removed by taking on-peak backgrounds. Each excavation pit is ~20 microns in depth.

The ablated material is carried via argon gas into a Micromass Isoprobe, which is equipped with a flight tube of sufficient width that U and Pb isotopes are measured simultaneously. The measurements are made in static mode, using Faraday detectors for ^{238}U, ^{232}Th, $^{208–206}$Pb, and an ion-counting channel for ^{204}Pb. Ion yields are ~1 millivolt per ppm. Common Pb corrections are made by using the measured ^{204}Pb and assuming initial Pb compositions from Stacey and Kramers (1975). Analyses of zircon grains of known isotopic and U-Pb composition were conducted in most cases after each set of five or ten unknown measurements to correct for elemental isotopic fractionation. In some cases, the standard analyses were sufficiently stable to measure ten unknowns between standards. ^{207}Pb*/^{206}Pb* ratios for all samples were corrected for 2%–5% ± ~3% fractionation. Common Pb correction is performed by using the measured ^{204}Pb and assuming an initial Pb composition from Stacey and Kramers (1975) (with uncertainties of 1.0 for ^{206}Pb/^{204}Pb and 0.3 for ^{207}Pb/^{204}Pb). Measurement of ^{204}Pb is unaffected by the presence of ^{204}Hg because backgrounds are measured on peaks (thereby subtracting any background ^{204}Hg and ^{204}Pb), and because very little Hg is present in the argon gas.

For each analysis, the errors in determining ^{206}Pb/^{238}U and ^{206}Pb/^{204}Pb result in a measurement error of several percent (at 2-sigma level) in the ^{206}Pb/^{238}U age. The errors in measurement of ^{206}Pb/^{207}Pb are substantially larger for younger grains due to low intensity of the ^{207}Pb signal. The ^{207}Pb/^{235}U and ^{206}Pb/^{207}Pb ages for younger grains accordingly have large uncertainties, beyond the level of geologic meaning for samples of this study. Inter-element fractionation of Pb/U is generally <20%, whereas isotopic fractionation of Pb is generally <5%. The uncertainty resulting from the calibration correction is generally ~3% (2-sigma) for both ^{207}Pb/^{206}Pb and ^{206}Pb/^{238}U ages.

The pooled crystallization ages reported in this paper are weighted averages of individual spot analyses. The stated errors (2s) on the assigned ages are absolute values, and include contributions from all known random and systematic errors. Only ^{206}Pb/^{238}U ages are used in this study because errors of the ^{207}Pb/^{235}U and ^{206}Pb/^{207}Pb ratios are prohibitive. This is due primarily to the low intensity (commonly <1 mV) of the ^{207}Pb signal from these young, U^{235}-poor grains.

Supplementary references

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