Etch selection and abrasion techniques applied to complex populations and revealed spurious ion probe ages

T. E. Krogh

Department of Earth Sciences, Royal Ontario Museum, Toronto, Ontario M5S 2C6, Canada

Earlier studies (e.g. Krogh, 1982) indicate that most single-stage zircon populations contain small amounts of concordant or near-concordant material that can be isolated by selecting gem quality crack free grains and abrading away geologically leached surfaces. In that study a 4% discordant multi-grain analysis was obtained for grains selected from a 17% discordant population. More recently Krogh, 1994 showed that etching in NaOH (room temperature saturated) for 3 hours at 220°C reveals radiation damage and allows selection of a few grains with minimum U from among several thousand. The bulk fraction had 650 ppm U (17% discordant) two abraded grains 230 and 175 ppm (1 and 3.4% discordant) and four etched abraded grains 152, 20, 58 and 154 ppm (0.4, 0.3, 0.3, and −0.2% discordant). Here we show that when applied to two-stage discordia (ie populations with metamorphic regrowth and diffusive Pb loss as well as primary growth) etching provides a rapid method for selecting grains that precisely define the mixing line (i.e. that do not have recent loss), and where core-overgrowth uranium contrasts exist reveals the low U tips or cores for analysis. Samples containing abundant metamorphic zircons such as metadiabase or melt-pod pegmatites provide an independent means for establishing the lower intercept. In most but not all cases data for the low U primary grains selected move up the mixing line obtained by abrasion but very few samples give concordant primary age results. Studies of these mixed-age populations, critical to the evaluation of potential laboratory leaching effects, prove that the selected etched abraded low U grains are not disturbed in the lab or by recent geological leaching.

Comparison of conventional and ion probe results

Primary emplacement ages are fundamental to geochemical studies of ancient rocks, which in many cases have a complex metamorphic history. Our tests on one of two outcrops of Uivak gneiss in Labrador for which an ion probe age of 3733 ± 9 Ma was previously published (Schiotte *et al.*, 1989) indicate magmatic emplacement at 3349 ± 10 Ma with a lower intercept amphibolite facies event recorded by meta diabase zircons at 2702 ± 1 and 2700 ± 4 Ma (0.9 and 1.3% discordant) and the primary grain discordia. No ion probe age for metamorphism is defined. We used HF and abrasion to release rounded and euhedral low U cores from metamict grains and data for three of these are colinear to 3350 Ma (the time of magmatism) and 3813^{26}_{-42} Ma (their source age) whereas two others gave data that is colinear from 3350 Ma to 3979 Ma. Data for two single grains plot on the line hence were inherited from the second sample described below. Ion probe data for a single core is given as 3863 ± 12 Ma. Most grains in this sample are highly altered which may explain why the ion probe primary age is based on only two grain interiors, one of which is described as euhedrally overgrown by highly zoned typical magmatic material. Given our magmatic age and core data it is clear that the ion probe procedure of selecting the highest overlapping 7/6 ages has led to an incorrect age reflecting that of a partly reset core.

Our second sample of Uivak gneiss is from an area that underwent a later granulite facies event for which an ion probe emplacement age of 3730 ± 8 Ma (based on 3 grains) was previously reported (Schiotte *et al.*, 1989). Our results indicate emplacement at 3592 ± 5 Ma and metamorphism at 2726 ± 12 Ma with data 5, 7, 10 and 99 and 102% discordant 86% probability of fit. Selection of metamorphic grains after etching in this case is facilitated by their extremely low U content (as low as 1 ppm U). By ion probe these are described as low U (114−142 ppm) with 6/4 ratios of 250−300 and 7/6 age errors of 30−50 Ma. Excess common lead in ion probe analyses is indicated because one of our grains has 152 ppm U, a 6/4 ratio of 4,500 and a 7/6 age error of ± 1 Ma (0.4% discordant). A second measure of the metamorphic growth is provided by a large single faceted tip from pegmatitic infil in a ruptured

ultramafic raft. This grain has 18 ppm U a 6/4 of 16,176 and an age of 2706±1 Ma (0.02% discordant). A single core from this sample gave data that falls on the 3813 Ma line. Data for some rounded cores by ion probe are reported as overlapping in age with their magmatic emplacement age and an abundance of *c.* 3600 Ma grains are noted. Given our new results it is certain that the ion probe age reported is that of a partially reset core. Our metamorphic ages of 2725 and 2706 imply that the ion probe age for low U metamorphic grains that cluster close to concordia with a mean 7/6 age of 2766 Ma may include a small amount of older material.

In general ion probe results plot below the mixing lines defined by isotope dilution and are correctly interpreted as reflecting metamorphic as well as recent lead loss and of no age significance. In both examples selection of the highest overlapping 7/6 ages (as minimum age values) has led to erroneous ages for emplacement. Examination of data reported below further indicates that this is a dangerous procedure because there may not be any material left that records concordant primary ages although assignment as a minimum age is valid. Our methods allow precise definition of two stage mixing lines and concentration of data at end members.

Further examples of selection etch abrasion results

Examples that demonstrate NaOH or HF etch techniques to isolate tips, cores, or high U zones or hot spots on otherwise perfect grains will be given. In one example 8 multigrain analyses where found to define a mixing line from about 400 Ma to 1570 Ma with 7/6 ages ranging from 480–1300 Ma. No concordant results could be found for tips, exterior fragments, etc. By treating a selection of grain tips in HF at 220°C for 3 hours tiny white seed xenochrysts where revealed - whereas the host grains with 10–15 ppm U were not affected. After abrasion 5 of 6 fractions are all concordant (0.1, −0.2, −0.2, 0.5 and 0.3% discordant) with a mean 6/8 age of 395.2 and a mean 7/6 age of 395.9 again the etch did not disturb the U-Pb system.

References

Krogh, T.E. (1982) *Geochim. Cosmoschim. Acta*, **46**, 637–49.

Krogh, T.E. (1994) *ICOG*, **94**, 180.

Schiotte, L., Compston, W., and Bridgwater, D. (1989) *Canad. J. Earth Sci.*, **26**, 1533–56.