Multi-step dissolution of baddeleyite for ID-TIMS U-Pb dating

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Numerous studies have highlighted both the power and potential complexities of U-Pb baddeleyite geochronology. We present results from multi-step digestion experiments designed to understand and reduce the effects of Pb loss and secondary zircon growth in complex baddeleyites. The research was motivated by the recently developed chemical abrasion (CA) method for zircon U-Pb dating [1]. The experiments were carried out on both complex Neoproterozoic baddeleyites with zircon inter- and overgrowths and fragments of a large gem-quality Paleoproterozoic baddeleyite.

The first group of experiments focused on isolating the baddeleyite and zircon ages from complex grains. To discretely dissolve the two phases, we used a two step HCl-HF digestion procedure on annealed grains. Initial 48hr-220ºC HCl digestions completely dissolved baddeleyite, leaving behind intact zircon rims. The remaining zircon was dissolved in either a 48hr-220ºC HF digestion or in a multi-step chemical abrasion digestion. The data define two distinct populations. Baddeleyite (HCl) data plot along a statistically significant linear array with a zero age lower intercept. In contrast, the zircon rim analyses scatter to the left of the discordia, suggesting they may represent multi-component mixing between the baddeleyite crystallization age and younger non-zero age Pb loss or zircon growth. Single step digestion data from this sample produced significant scatter, reflecting mixing of the baddeleyite and zircon ages. The results demonstrate the potential for resolving both the igneous and metamorphic history of complex baddeleyites.

Multi-step chemical abrasion experiments on the two samples had more complex results. Annealed grains were subjected to a series of HCl digestion steps (120–220ºC) followed by a final HF digestion. The HCl digestion steps define discordia lines with upper intercept dates equal to the sample ages. However, individual digestion steps were both normally and reversely discordant suggesting that U and Pb were fractionated in the multi-step digestions. The data indicate that the CA method is not an effective way to reduce the impact of Pb loss in U-Pb baddeleyite geochronology.

[1] Mattinson (2005) *Chemical Geology* **220**, 47–66.

U/Pb-dating of calcite slickenfibres

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A new method for determining the age of brittle deformation is presented, which utilises U/Pb-dating of tectonic carbonates, i.e. synkinematically grown fibrous calcite such as slickenfibres and tension gashes. Brittle deformation typically occurs at relatively low temperatures, where only few *datable* minerals grow synkinematically. Tectonic carbonates form abundantly during brittle deformation, but have so far proven to be difficult to date.

Under favourable conditions, calcite can have high uranium and low lead concentrations resulting in elevated U/Pb-ratios, and calcite has been shown to be sufficiently resilient against secondary influences to make it suitable for dating [1, 2, 3].

To gain an overview of the range of naturally occurring U- and Pb-concentrations in tectonic carbonates, samples from a wide range of different host rock lithologies, tectonic settings and deformation ages were taken from several areas along the Alps.

Initial trace element profiling by LA-ICPMS reveals element distributions and concentrations, allowing for a quick overview of each individual sample. U concentrations of 0.4 – 3 ppm and approximate $^{238}U/^{204}Pb$-ratios of up to ~2000 – 10000 (with variations spanning 3 orders of magnitude) are comparable to published data of successfully dated calcites.

An attempt of in-situ U-Pb isotope ratio measurements by LA-ICPMS reveals the highly radiogenic character of some samples, but lower precision of in-situ analysis by a quadrupole ICPMS compared to more sensitive MC-ICPMS and an inherently high ^{204}Hg background prohibiting direct measurement of ^{204}Pb limits precision of such age information, even though those preliminary ages appear to fit into the respective geological framework.

Thus, selected samples are being microsampled, dissolved, followed by low-blank U-Pb ion exchange chromatography and isotope dilution MC-ICPMS; these results will be presented.

The new method not only yields insight in regional tectonic evolution, e.g. by providing 'absolute' age control for relative ages of fault sets obtained from paleo-stress analysis, but may also be applied to gain information on dynamics of single faults, like deformation rates and (minimum) durations.

[1] Woodhead *et al*. (2006) *Quaternary Geochronology* **1**, 22.
[2] Cole *et al*. (2005) *Geol. Soc. Am. Bull.* **117**, 276-287.
[3] Kelly *et al*. (2006) *Environ. Sci. Technol*. **40**, 2262-2268.