Feature mapping protocol for grain selection in preparation for *in-situ* U-Pb geochronology: Shales and phyllites from the COSC-2 deep borehole

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Dating of minerals such as monazite, zircon, apatite, etc. by microbeam techniques (LA-ICP-MS, SIMS, EPMA), where it is required that the grains remain *in-situ* within their host rock, presents challenges for locating such very small objects and finding a sufficiently large grain population. *In-situ* analysis may be necessary to preserve the textural context of the grains or if the sample is small and/or precious a minimally destructive analytical approach is demanded. Manual searching is laborious, time-consuming and expensive. Fine-grained rocks such as argillaceous sediments or low-grade metapelites are particularly challenging. We were confronted with such issues during dating of monazite in mudrocks and slatey metapelites in core samples from the ICDP project COSC-2 in the Swedish Caledonides (Lorenz et al. 2022), for which we used the electron microprobe chemical U-Th-Pb method. Monazite grains were mostly very small (up to a few microns) and a non-destructive approach was preferred due to the high value and rarity of the samples and the need to preserve textural relationships to depositional, authigenic, metamorphic or vein fabrics. Our method was based on the Oxford Instruments AztecFeature™ software. Polished thin sections were scanned in the Quanta FEG 650 SEM at Heriot-Watt University, Edinburgh using back-scattered electron (BSE) mode generating an array of tiles, which were then stitched to make a high-resolution image of the whole sample area. Sub-areas of the image were inspected under high contrast to identify grains of possible interest for dating (monazite, zircon, apatite, rutile, allanite, titanite) from their EDX spectra. A grey-scale intensity range for each was established for each mineral species, which was used to set threshold values to as a basis for grain classification. This was applied to detect grains of the selected types across the entire image and establish their co-ordinate positions. For each identified grain its size and shape were determined and an EDX spectrum was taken to determine its chemical composition. The workflow is automated but can be interactive, for example, if the system discovers a new grain type it can be added to the search. The output from an automated run can be presented as a spreadsheet that can be sorted and filtered. For the COSC-2 samples we filtered to select grains of suitable size for EMP analysis. Further filtering could, for example, select for composition such as EDX-derived Th or U concentration. After an automated run of 3 days length, a scan of a typical COSC-2 polished section detected 2358 zircons, 3884 monazites, 1475 Ti-oxides, 1048 ilmenites, 2196 apatites and 23 titanites. For monazites 1066 grains had sizes judged to be suitable for EMP analysis (length >10µm). An X-Y graph can be plotted to produce a map with the locations of all these grains. With a little trial and error such maps can be superimposed upon the base image used in the electron probe micro-analyser. Grains can then be located to acquire detailed BSE images and more precise and accurate wavelength dispersive WDX mapping and analysis as a basis for final age data acquisition by EPMA, LA-ICP-MS or SIMS.

While the protocol described here is relatively expensive in terms of SEM machine time, this can be balanced against time saved in locating grains for analysis and the more representative grain populations yielded by the automated, rapid and systematic feature analysis method. Challenges that remain are that some lithologies with abundant non-datable grains exhibiting bright BSE intensity (e.g., pyrite in pelites) yield large numbers of failed grain classifications; also, a more robust and precise method of matching the grain map to base images would be beneficial.

References

Lorenz, H., Rosberg, J.-E., Juhlin, C. and 14 others, 2022: COSC-2 – drilling the basal décollement and underlying margin of palaeocontinent Baltica in the Paleozoic Caledonide Orogen of Scandinavia. *Sci. Dril., 30,* 43–57.

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