The microtome: an innovative tool prerequisite for applying multiple high-precision/resolution analytical techniques to wafers of same sub-grain scale sample

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The modern high-precision/resolution analytical techniques call for single-grain samples, like individual zircon crystals. The problem is that even single zircon grains frequently demonstrate a complex, inhomogeneous nature due to the presence of chemically different phases, of structurally distinct crystal domains or of growth zones which may reflect diverse and sometimes independent stages of the history of the momentary host or precursor rocks.

The most reliable interpretation for the complex mineral grains can be achieved by applying all relevant analytical methods to the same mineral domain within a single mineral grain. However, up to now the conventional preparation of small samples for ion microprobe, electron microprobe/microscope, cathodoluminescence, radiography and other micro-analytical studies required mechanical polishing which usually results in irretrievable loss of at least half of the original sample. In addition, this way of sample preparation does not allow to retain the original material either for future cross-checking or for correlative investigations, such as studying the effects of chemical or mechanical treatment on the initial substance. Thus, the necessity of having closely equivalent portions of a specimen at hand is obvious. Crystal portions of such properties are most easily produced in the shape of thin wafers.

Our novel sub-grain scale technique based on a recently developed miniature wire-saw (microtome) expressly permits precise cutting of hard crystalline objects, in particular of submillimeter-sized crystals. The instrument drives a tungsten cutting wire, 4 to 13 micrometer in diameter, passing through a slurry of 1 to 0.5 micrometer diamond particles and can provide individual or sets of thin slices from single crystals, in any direction desired. The microtome has proved its worth in slicing mineral grains without damage to the original sample and without significant loss of material. The resulting cuts were found to be no wider than the diameter of the cutting wire. The exact and firm adjustment of the sample as well as the precise and continuously controlled guidance of the wire results in planar and strictly vertical cuts. Under the microscope, all the obtained mineral slices exhibit very light sawing tracks. These prove useful for the documentation of the microsampling process, such as orientation and identification of the slices and proper restoration of the grain. The sawing tracks have a negligible depth (fractions of micrometer) and - if desired - can be easily erased with a few passes in 1-micrometer diamond abrasive. This approach to producing equivalent wafers attains particular significance in creating standards for microanalytical work from existing rare and highly valuable reference materials.

An automated version of the prototype microtome described by Sergeev *et al.* [1997] has since been developed. This advanced instrument is now equipped with a slow-running electrical motor and an upgraded system for wire-tension control. This permits unidirectional and steadier motion of an extended portion of the cutting wire that further improves the quality of the cut surface.

The practicability of the microtome technique in zircon U/Pb analysis has been tested in two ways:

Firstly, we sliced a chip of the zircon standard, routinely used for calibration of the ion microprobe into three pieces. The two lateral parts were first checked by cathodoluminescence imaging for internal homogeneity and then placed onto the sticky tape with the cut surfaces down like the other unknowns before filling up the mount with epoxy resin. The remaining central part of the standard zircon was analysed by isotope dilution techniques in order to get precise calibration results for the standard material.

Secondly, with the aim of extracting a maximum of age and petrogenetic information from complex zircon grains we cut selected crystals along the c-axis to obtain two mirror-image sections using the 5-micrometer wire. One half-zircon (part A) was investigated by cathodoluminescence imaging and

prepared for electron microprobe study and high spatial resolution dating by the ion microprobe SHRIMP. After studying the cathodoluminescence images revealing the internal zircon structure, the second mirror half-grain (part B) was dissected into observed growth phases and crystal domains by either additional sawing or by mechanical cleaving. The fragments were then abraded and subjected to acid-washing to remove undesired or instable phases along margins. The resulting homogeneous zircon phases obtained from such morphological entities as rounded cores with reentrant boundaries, rhythmically zoned magmatic mantles or pyramidal terminations of the crystal, were analysed by isotope dilution techniques.

The combination of results based on the isotope dilution and ion microprobe analyses with those of the morphological study of the cathodoluminescence image suggests that the investigated zircons record a number of evolutionary events in form of distinct growth phases. Whereas the high-precision isotope dilution technique (on part A) was instrumental in establishing exact concordant ages for different generations of the zircon's zoned mantle or of the rim, the superb spatial resolution and high density of ion microprobe spot analyses (on part B) yielded indispensable information for distinguishing between cores of different age inherited from the precursor rocks as well as for the distribution of corresponding synchronous zircon phases within the complex zircon grains. We conclude that our miniature microtome is an indispensable and fundamental tool for applying the modern microanalytical techniques to sub-grain systems formed during different stages of a mineral's history.

References

Sergeev, S.A., Komarov, A.N., Bickel, R.A. and Steiger, R.H. (1997) *Eur. J. Mineral.*, **9**, 449–56.