Microscopic analysis of magmatic crystals – Part 2: A SEM study of the stability of accessory zircon under increasing metamorphic conditions

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Introduction

This contribution is a continuation of a previously published work in Microscopy Today that described the microscopic analysis of magmatic crystal growth by the example of accessory zircon(1). Zircon does not only represent a remarkable mineral phase concerning its crystallization out of the magmatic melt, but has also other interesting characteristics, one of which is the rather high physical stability of zircon allowing a determination of the mineral—even in high-grade metamorphic rocks. The changes of zircon from low- to high-grade deformation are very noticable and therefore offer an interesting operating field for electron microscopy. Since crystal microscopy and its specific fascination cannot often be found in a microscopy magazine, it is assumed that the article would awake the interest of the readers.

During the past 50 years, accessory zircon has represented a preferential mineral phase for mineralogical and petrologic studies. A major reason for the continuing interest of geoscientists in this accessory mineral is certainly its high resistance to any deformation processes in the near-surface earth crust. Due to this specific characteristic and, not less important, the ubiquitous occurrence of zircon in the terrestrial lithology, the mineral has found significant application to the petrogenetic classification of magmatic rocks(2) as well as the calculation of volume and mass balances in different types of crustal deformation zones(3).

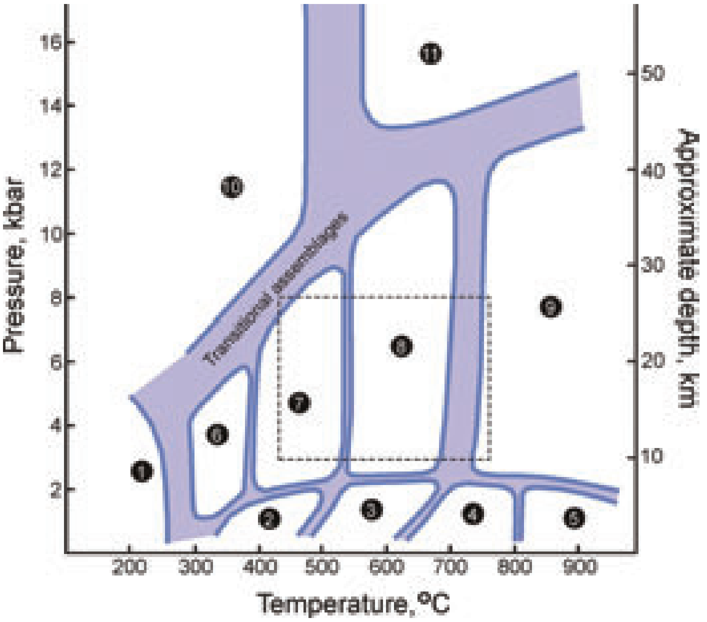

Figure 1. Pressure-temperature diagram with the areas of various metamorphic facies (1…zeolite, 2…albite-epidote-hornfels, 3…hornblende-hornfels, 4…pyroxene-hornfels, 5…sanidinite, 6…prehnite-pumpellyite, 7…greenschist, 8…amphibolite, 9…granulite, 10…blueschist, 11…eclogite)(11). The dashed frame indicates the PT field investigated in this contribution.

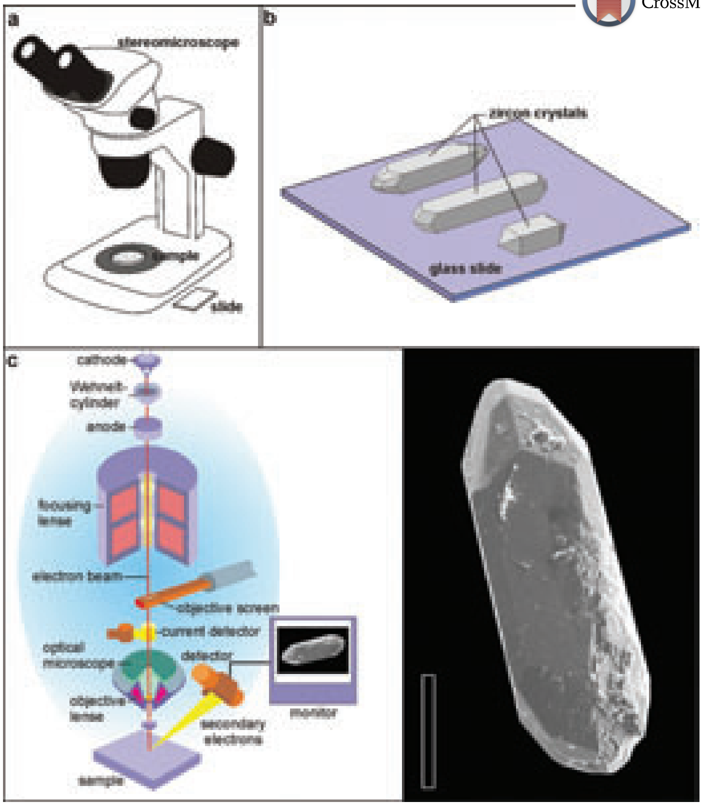

Figure 2. *Preparation procedure for the SEM investigation of zircon crystal surfaces: a) separation of representative crystals under the stereomicroscope, b) mounting of the crystals on a glass slide, c) electron microscopy (bar in the right image: 50 µm).*

The enhanced physical and chemical stability of zircon was already noticed in the 1950s and 1960s, when crystals of the mineral phase separated from magmatic, metamorphic and sedimentary rocks were subject to comparative light-microscopic investigations(4,5). With the help of these studies it was found that low- to medium-grade metamorphic deformation has only small effects on zircon crystals, resulting in the complete preservation of the mineral's internal structure. After high-grade metamorphic deformation, on the other hand, zircon undergoes processes of rounding, fracture, corrosion, recrystallization, and overgrowth, which not only cause a significant change of the outer crystal shape but also a modification of the inner morphology, the latter has to be considered in the case of geochronological measurements. More recent studies on physical and chemical damaging of zircon in metamorphic rocks were carried out on the basis of different electron microscopic methods (SEM, CL, BSEI) so as to understand micro-scale destruction processes on crystal surfaces such as the formation of small fissures or corrosion pits(3,6,7). These investigations also demonstrated the existence of a positive correlation between the amount of fluid phases infiltrating the deformation zone and the extent of crystal damage. Hence, accessory zircon from a medium-grade metamorphic rock with high fluid infiltration exhibits similar physical and chemical modifications as accessory zircon from a high-grade metamorphic rock characterized under 'dry' conditions. The enhanced physical and chemical resistance of accessory zircon was also established in part for high-pressure high-temperature shock metamorphism which is caused by the impact of an extraterrestrial rock body on the earth crust. Respective studies showed that impact-shocked zircon crystals are well rounded and form a granular surface texture due to extremely short-time melting processes(8).

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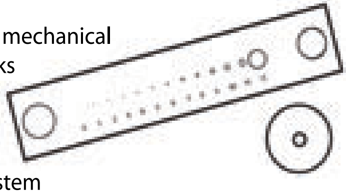

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In the contribution presented here, some excerpts of a previously conducted SEM study on accessory zircon in ductile deformation zones are described, thereby demonstrating external changes of zircon crystals that occur within a temperature range of 400 °C to 700 °C and a lithologic pressure varying from 4 kbar to 8 kbar (Fig. 1).

Crystal preparation and SEM imaging

The SEM investigation of zircon crystal damage due to rock deformation was preceded by the separation of the mineral phase from pre-selected rocks as well as the appropriate preparation of representative zircon grains. Determination of the concentration of accessory zircon was carried out according to a standard procedure(2,3) which among other includes the crushing, milling, and sieving of the rock material followed by a first rough mineral separation of the appropriate grain size fractions using the floatation method. Further separation of mineral phases was conducted in a magnetic field and a high-density liquid (tetra-brome-ethane, $\rho = 2.96\ \text{g cm}^{-3}$), resulting in the receipt of a concentrate of diamagnetic minerals with high specific weight (zircon, apatite, titanite, rutile, xenotime). To obtain a pure fraction of zircon crystals, respective grains were picked out of the concentrate under the stereomicroscope (Fig. 2).

Specimen preparation for electron microscopy was carried out by mounting selected grains on a glass slide with a drop of epoxy resin. The sample was then coated with an electron-conducting layer of carbon and examined in the electron microprobe (type JEOL JXA-8600) at the Department of Geology and Palaeontology, University of Salzburg. The following device setup was used: secondary electron mode, accelerating voltage of 15 kV, and a beam current of 3 nA (Fig. 2). Imaging of damaged zircon grains was performed using the analog microprobe camera producing photographs that were subsequently digitized.

Visualization of crystal damage under different grades of ductile deformation

Low-grade ductile deformation

As illustrated in Figs. 3 to 5, crystal surface and morphology of accessory zircon may be subject to significant changes with increasing grades of rock deformation and alteration. Rocks exhibiting a low-grade alteration (greenschist facies, Fig. 1) with temperatures and pressures not exceeding 500°C and 2-3 kbar, respectively, are often found at the margins of medium- to large-scale deformation zones. Zircon crystals separated from such weakly mylonitized lithologies are usually characterized by euhedral shapes with sharp and well defined edges between the crystal faces (Fig. 3). Due to this perfect preservation of the external crystal morphology, with its various prisms and pyramids, accessory zircon may be effectively used as a tool for the petrogenetic classification of the host rock according to the typology scheme introduced by Pupin(2). As far as detectable, damage of the zircon surface is limited to small scratches and roughening of single crystal faces or an insignificant rounding of the pyramidal tops. In the case of increased fluid infiltration of the alteration zone, first traces of crystal corrosion due to an interaction between a fluid phase and the crystal surface may be observed, leading to the formation of small pits and corrosive surface patterns (Fig. 3f). Zircon fracturing does not occur very frequently within the zircon population and therefore affects about 2 % of the investigated crystals.

Medium-grade ductile deformation

At medium grades of ductile rock deformation (lower amphibolite facies, Fig. 1), temperature usually ranges from 520 to 570 °C, and pressure varies between 4 and 5 kbar. These pressure-temperature conditions were established in the centre of ductile shear zones that formed at a depth of 15 to 20 km below the surface of the earth. Relics of such deformation zones are found in the Tauern Window and the Bohemian Massif in Central Europe(7). As depicted in Fig. 4, advanced changes of the zircon crystal surface are recognizable for this grade of deformation. Contrary to the zircon crystals found in weakly deformed rocks, medium grade ductile deformed single grains subject to metamorphic conditions of the lower amphibolite facies do not show euhedral crystal faces anymore due to an advanced rounding of the crystal edges. The pyramidal faces of single crystals are most affected by this mechanical process (Fig. 4a, b). The loss of perfect crystal shape with well-formed crystal faces reduces the value of the accessory mineral as petrogenetic indicators because exact typological classifications can not be made for non-euhedral grains. Strain forces having an effect on the crystals are expressed by a partial production of fissures and deep cracks along the directions of enhanced cleavage. In addition to the mechanical damage, mineral-fluid interaction increases in incidence, the result of which is the formation of larger corrosion pits, mainly along crystal fissures and cracks. These pits may reach diameters of several tens of micrometers and may penetrate deep into the crystals' interior (Fig. 4a, c, f). Crystal fracturing is significantly increased with respect to low-grade deformation, now affecting between 10 and 20 % of the whole crystal population.

High-grade ductile deformation

High-grade deformation of the upper amphibolite and granulite facies (Fig. 1) takes place at temperatures above 600°C and pressures between 5 and 8 kbar. Deformation zones having these conditions are for the most part formed in deep levels of the

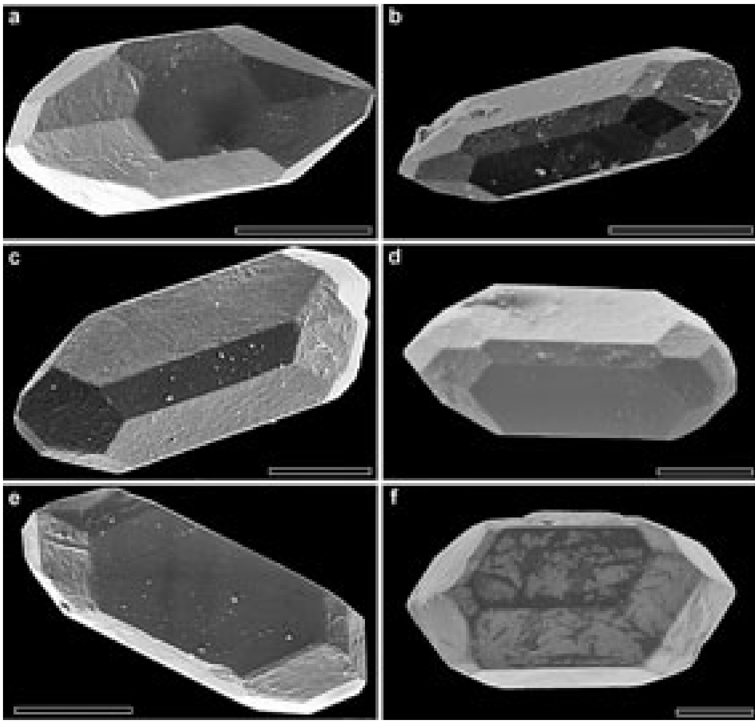

Figure 3. Zircon crystals separated from rocks affected by low-grade deformation (greenschist facies). Bars: 50 µm.

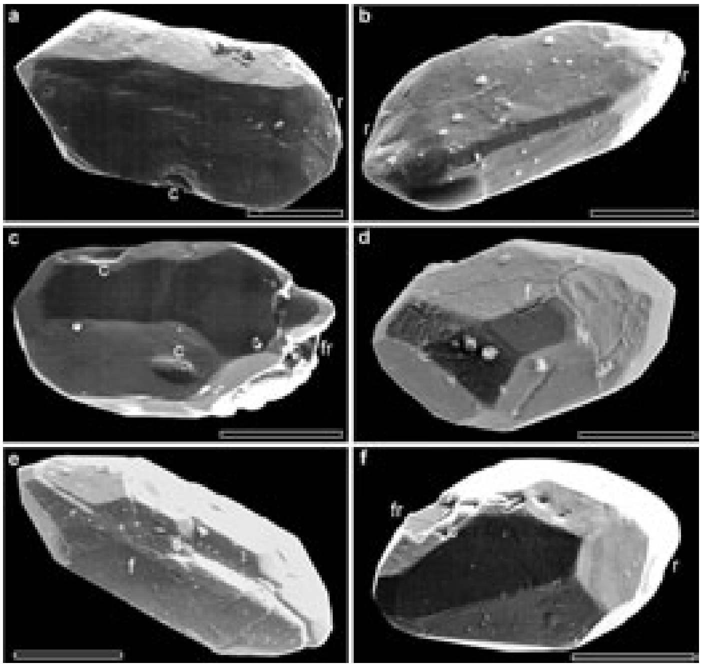

Figure 4. Zircon crystals separated from rocks that have undergone medium-grade deformation (lower amphibolite facies). Abbreviations: c… corrosion pits, f…fissures, fr…fracturing, r…rounding; bars: 50 µm.

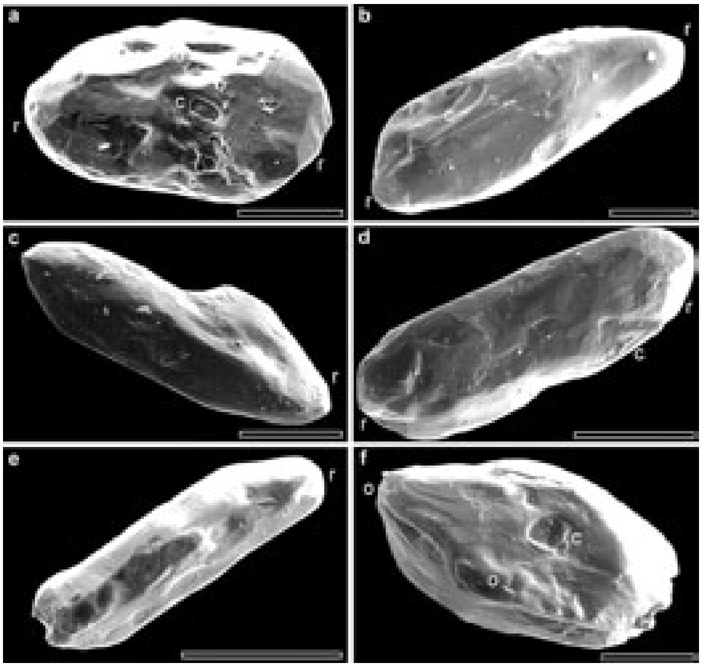

Figure 5. Zircon crystals separated from rocks which have been subject to high-grade deformation (upper amphibolite/granulite facies). Abbreviations: c…corrosion pits, f…fissures, fr…fracturing, o…overgrowth, r…rounding; bars: 50 µm.

earth crust (> 20 km). High temperatures and pressures result in an extensive alteration of the former mineral assemblages and the generation of new assemblages that are adjusted to the modified thermodynamic conditions. Regarding accessory zircon, high-grade ductile deformation causes a highly significant change in the crystals that is expressed by the near-complete loss of euhedral morphology due to extensive processes of rounding, corrosion, and, not less important under these conditions, overgrowth (Fig. 5). The impossibility of crystal face classification and related typological diagnosis(2) dramatically decreases the value of zircon as petrogenetic indicator or as a tool for an appropriate identification of the protolithic rock in the case of large and not fully accessible shear zones(7). Surfaces of numerous high-deformation zircon crystals are also characterized by a sequence of corrosion pits and overgrowths, resulting in a somehow battered appearance (Fig. 5a, d-f). Single corrosion pits may be extended over large parts of the crystal's surface, and overgrowths may lead to the formation of rather bizarre crystal shapes (Fig. 5f). Crystal fracturing is further enhanced with respect to medium-grade deformation and affects about 30 % of the zircon population.

Conclusions

As demonstrated by this study, accessory zircon reveals an enhanced resistance to rock metamorphism with respect to major rock mineral phases such as feldspar, quartz, biotite or comparable accessories. Deformational forces mainly affect the zircon crystal surface, causing a significant change of crystal shape at high-grade metamorphic processes. The rather high stability of zircon may be partly lead back to its hardness, weak cleavage, and small grain size reaching a maximum of a few hundred micrometers in granitic rocks(9,10). On the other side, zircon exhibits very low chemical reactivity with adjacent mineral phases due to the content of highly incompatible elements. Further studies of zircon stability will try to solve open questions in the future. ■

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