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# Metamorphic zircon formation in gabbroic rocks – the tale of microtextures

VICTORIA BECKMAN

LITHOSPHERE AND BIOSPHERE SCIENCE | DEPARTMENT OF GEOLOGY | LUND UNIVERSITY 2018





Metamorphic zircon formation in gabbroic rocks  
– the tale of microtextures



# Metamorphic zircon formation in gabbroic rocks – the tale of microtextures

Victoria Beckman



**LUND**  
UNIVERSITY

Lithosphere and Biosphere Science  
Department of Geology

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Cover photo: Baddeleyite (light grey)  
with polycrystalline zircon rim (medium grey)  
and igneous zircon (medium grey) above.

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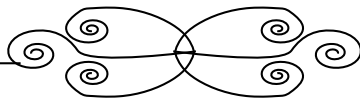
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To



Magdalena

&

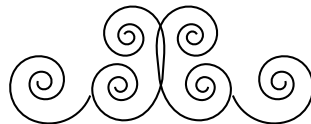
Karolina

*PRACTICALLY*

*PERFECT*

*IN*

*Every way*







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# Abstract

Dating of metamorphic events is crucial for the understanding and reconstruction of large-scale geological processes such as orogenesis. Zircon is one of the most commonly used minerals for dating of igneous and metamorphic events. Zircon incorporates uranium and excludes lead during crystallization, and with time the uranium decays to lead. The diffusion rates of both elements are slow, making zircon resilient to isotopic resetting. However, in order to date geological events, it is imperative to know exactly by which process the dated zircon formed. For example, regional metamorphism is a dynamic process taking place over millions of years. During tectonic burial and heating the rock gradually responds to the increasing temperature and pressure, giving rise to prograde mineral assemblages, whereas retrograde metamorphism takes place during cooling and exhumation. So, in a regionally metamorphosed rock, does the zircon age date the tectonic burial or the exhumation? The interpretation of how zircon formed has direct influence on the tectonic interpretation.

Zircon can form or recrystallize within a wide range of metamorphic pressures and temperatures and by several different processes. This means that, for meaningful interpretation of a metamorphic zircon age, the zircon growth needs to be linked to the mineral reactions in the rock. Due to the high closure temperature of zircon (the temperature below which zircon will not undergo isotope diffusion), zircon ages have traditionally been assigned to date the peak of metamorphism (the highest temperature). On the other hand, mass balance models suggest that, in mafic rocks, zircon dissolves during prograde and

grows during retrograde mineral reactions and therefore generally dates cooling and exhumation.

If hydrous fluids are not present, mafic igneous rocks may remain largely unaffected during a metamorphic event. Coarse-grained mafic rocks such as gabbro are the least permeable, and may record the gradual transition from pristine gabbro to its completely metamorphic recrystallized equivalent. Such metamorphic transition zones provide information about how metamorphic zircon formed. Two different metamorphic transition zones have been investigated in detail in this thesis: a) a gabbro to eclogite transition at Vinddøldalen in south-central Norway and, b) a gabbro to garnet amphibolite transition at Herrestad in South-central Sweden. The aim has been to link reaction textures to zircon growth and to obtain a direct U-Pb age of the metamorphic process. A third study investigates and reviews the zircon-forming textures in a number of metagabbro and metadolerite bodies metamorphosed at different pressures and temperatures.

The results in this thesis show that zircon formation is remarkably similar in all of the investigated metagabbroic rocks, and that zircon is mainly produced by the breakdown of igneous baddeleyite during prograde mineral reactions. The metamorphic mineral reactions and the associated zircon formation in gabbroic rocks are tightly linked to deformation and infiltration of hydrous fluids, and to a lesser extent dependent of variations in pressure and temperature. Therefore, in most gabbroic rocks, zircon formation will take place at the earliest stage of metamorphic recrystallization.

## List of papers

### Paper I

Beckman, V., Möller, C., Söderlund, U., Corfu, F., Pallon, J., & Chamberlain, K. R. (2014). *Metamorphic zircon formation at the transition from gabbro to eclogite in Trollheimen–Surnadalen, Norwegian Caledonides*. Geological Society, London, Special Publications, 390(1), 403-424.

### Paper II

Beckman, V., Möller, C., Söderlund, U., & Andersson, J. (2017). *Zircon Growth during Progressive Recrystallization of Gabbro to Garnet Amphibolite, Eastern Segment, Sveconorwegian Orogen*. *Journal of Petrology*, 58(1), 167-187.

### Paper III

Beckman, V & Möller, C. *Prograde metamorphic zircon formation in gabbroic rocks: the tale of micro-textures*. (Manuscript accepted to *Journal of Metamorphic Geology*).

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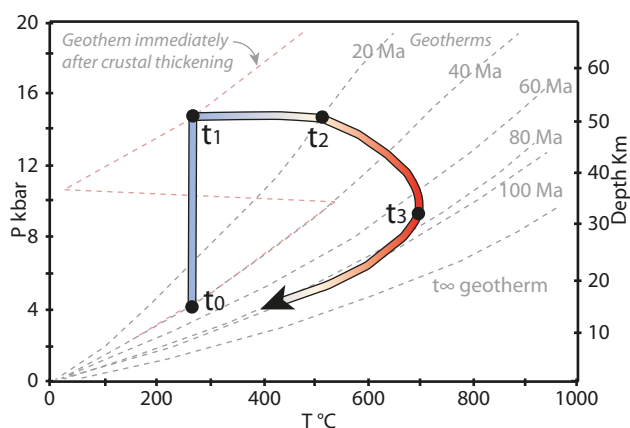
To my friends outside the department, Silvana, Paula, Kajsa and Anna, for cheering me on and to those who just have been looking forward to the dissertation party, sorry for letting you down on that one.

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Now only one remains: not faith, not hope but love, thank you Olof, for everything mentioned above and because “the difference between a lady and a flower girl is not how she behaves, but how she is treated” (Eliza Doolittle).

# Introduction

During plate-tectonic movements, the Earth's plates break up and collide, and the crust is exposed to changing physical conditions (notably pressure and temperature) which cause metamorphism: the transformation of mineral assemblages to a new chemical equilibrium under the new physical conditions. For example, during mountain building (orogenesis; caused by collisional processes), the crust is tectonically buried at great depth (increasing pressure) and increasing temperature. Parts of the tectonically buried crust later return to the surface by erosion or tectonic removal of the overlying rocks. During this tectonic cycle, rocks will gradually adjust their mineral assemblages to the changing conditions. "Metamorphic events" are seldom simple short heat pulses. More commonly, metamorphism begins with tectonic burial or subduction, and is followed by heating, uplift, and cooling over a period spanning several million or tens of million of years (cp. England and Thompson, 1984, Thompson and England 1984; Spear & Spear, 1993). Some rocks have experienced more than one metamorphic cycle, during completely separate thermal or orogenic events - these rocks are termed polymetamorphic. The P–T–evolution experienced by a rock can be visualized in a pressure vs. temperature diagram as a pressure–temperature–time path (P–T–t path; Fig. 1).



**Figure 1.**

Generalised clockwise P–T–t path (pressure–temperature–time path) after Peacock in Spear et al., 1991. Showing the evolution of geotherms with time in Ma. Pre-thrusting the bedrock situated at  $t_0$  becomes almost instantaneous buried to  $t_1$ , and the pressure increase due to double thickening of the crust. At  $t_1$  the temperature increase, as rocks adjust to the new conditions. After 20 Ma, erosion starts at  $t_2$ , and the pressure decline, although the rock is still heating. The maximum temperature is reached after c.70 Ma, at  $t_3$ .  $t_\infty$  the geotherm for double crustal thickening is never reached because of up-lift and erosion.

The specific shape of the P–T–t path will depend on the tectonic evolution, and in most cases the rates or durations of the different processes involved are unknown.

This is one reason why it is important in geochronology, the age determination of minerals, that each mineral age can be linked to a specific part of the P–T path. The techniques to link isotope ages to metamorphic conditions have evolved for the last two decades, mainly through the development of various micro-beam techniques, making it possible to extract textural, chemical and isotopic data with high accuracy and precision from small mineral domains, tens of microns in diameter. The integration of geochronology and petrology has been termed petrochronology, and has the aim to place the dated minerals in their petrological context via geochemistry, textures, and thermodynamic modelling (Engi et al., 2017).

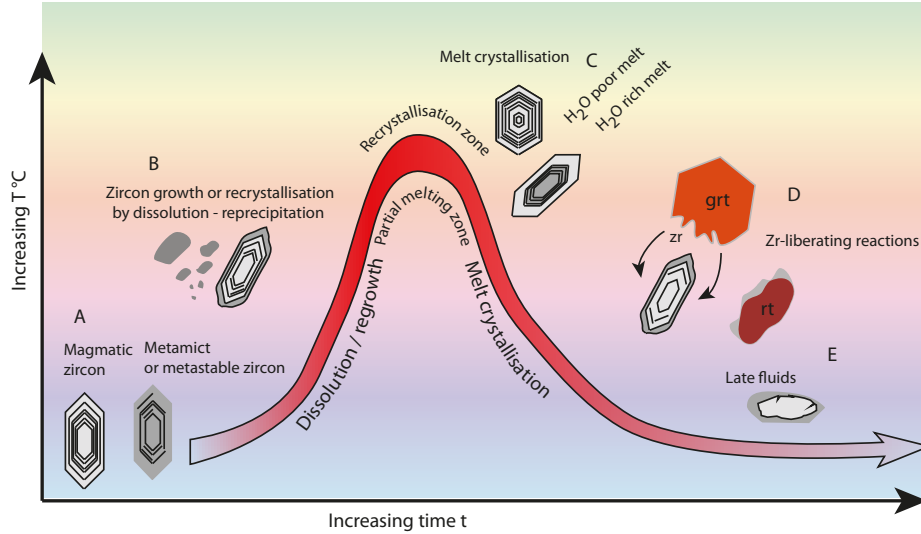
The most common method to determine the absolute age of an igneous rock is by radiometric dating of the mineral zircon ( $\text{ZrSiO}_4$ ), which incorporates small amounts of uranium (U) but excludes lead (Pb) during crystallization. U decays to Pb at a constant rate, and by measuring the amount of parent and daughter isotopes the age can be determined. The diffusion rate of Pb in zircon is slow, making it resistant to isotopic resetting at temperatures below 900 °C (Cherniak & Watson, 2001). Therefore, zircon can record and retain its isotopic system under a wide range of geological conditions, from low-temperature (Hay & Dempster, 2009) to high-temperature metamorphism (Harley et al., 2007).

This means that the U–Pb age of a zircon grain under most conditions (not ultrahigh-temperature metamorphism) reflects its crystallization age. This is different from many other isotopic systems (e.g.,  $^{40}\text{Ar}$ – $^{39}\text{Ar}$  ages of micas and amphiboles, U–Pb ages of rutile), which will be reset by diffusion at much lower temperatures (350–550 °C; (Parrish, 2001) and hence date cooling through the closure temperature of the specific system. In order to understand the meaning of the U–Pb age of zircon, i.e. the age of crystal growth, the zircon growth must be linked to a metamorphic reaction by which it formed. The means to “link age to stage” is by way of microstructures, microtextures (Johansson et al., 2001; Corfu et al., 2003; Harley et al., 2007), and trace element signatures (Rubatto, 2002; Hokada & Harley, 2004; Rubatto & Hermann, 2007; Rubatto, 2017). One of the difficulties is that zircon is an accessory mineral with subtle textural and chemical relations to the major minerals in the rock.

Metamorphic zircon ages have commonly been interpreted to date the peak or a retrograde metamorphic event. However, a zircon crystal can be partly or entirely dissolved, altered by low-temperature fluids, recrystallize in the presence of fluid or melt (e.g., Corfu et al., 2003; Kooijman et al., 2011; Rubatto, 2017). New zircon can form during metamorphism or metasomatism through the breakdown of minerals that contain trace amounts of Zr (Fraser et al., 1997; Bingen et al., 2001; Degeling

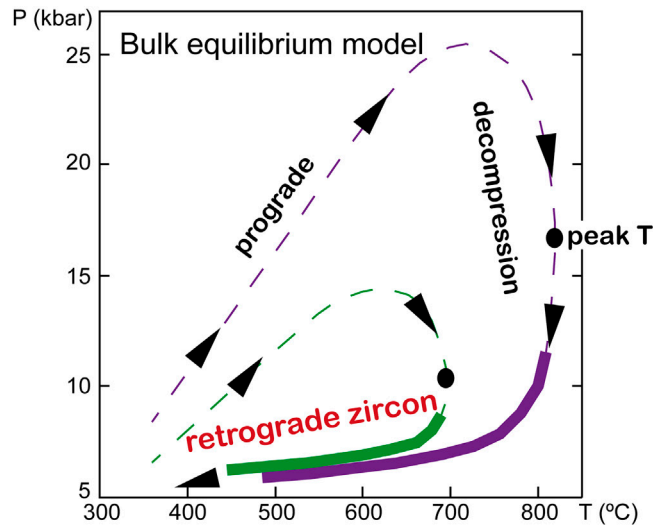
et al., 2001; Kohn et al., 2015) or through precipitation from a Zr-bearing fluid or melt (Williams et al., 1996; Liati & Gebauer, 1999; Rubatto & Herrman, 2003; Hansen et al., 2015). As reviewed by Harley et al. (2007) and Rubatto (2017), metamorphic zircon can in theory form at any stage of a P–T–t path (Fig. 2). In metamorphic rocks, especially if polymetamorphic, several gener-

ations of zircon might occur as new grains, overgrowths or as partly recrystallized grains in the same rock. With a careful choice of samples, it is possible to link a zircon age to a stage of the metamorphic cycle, and thereby (at least theoretically) to pinpoint the ages of several stages of the same P–T–t path (Hermann & Rubatto, 2003; Möller et al., 2003).



**Figure 2.** Schematic temperature–time path illustrating that zircon can potentially form at several different stages, in a hypothetical terrane. Modified after Harley et al., 2007. A) Illustrates that magmatic might survive increasing temperature, whereas metamict (radiation damaged) zircon is more prone to recrystallize or become modified during metamorphism. B) During heating and partial melting, small zircon grains might dissolve in favour of larger grains. C) Zircon crystallization from melt will generally occur at post peak conditions, at different intervals depending on the melts water content. Crystallizing as new zircon grains or as zircon overgrowths. D) Zircon growth from breakdown of Zr-rich minerals, such as Grt = garnet or Rt = rutile. E) At low-grade metamorphism, fluid alteration and solid- state replacement the main mechanism affecting zircon.

A recent attempt to understand zircon formation and dissolution by forward thermodynamic modelling (Kohn et al., 2015) suggests that, in metapelitic and metabasic chemical equilibrium systems, metamorphic zircon will not be produced during the prograde or peak metamorphic stages; instead pre-existing zircon is consumed. These models suggest that metamorphic zircon will be produced during decompression and cooling, from the breakdown of rutile or from crystallization of partial melt (Fig. 3). If this applies generally, it will have profound implications on the interpretation of metamorphic zircon ages.



**Figure 3.** Bulk equilibrium model with retrograde metamorphic zircon formation, as thick violet and green lines, in mafic rocks after Kohn et al., 2015. The temperature versus pressure diagram shows generalised P–T paths in dashed lines. Violet: P–T path reaching high temperature and pressure conditions corresponding to continental collision reaching ultra high pressure. Green: P–T path reaching moderate temperature and pressure corresponding to Barrovian type metamorphism (figure from paper III).

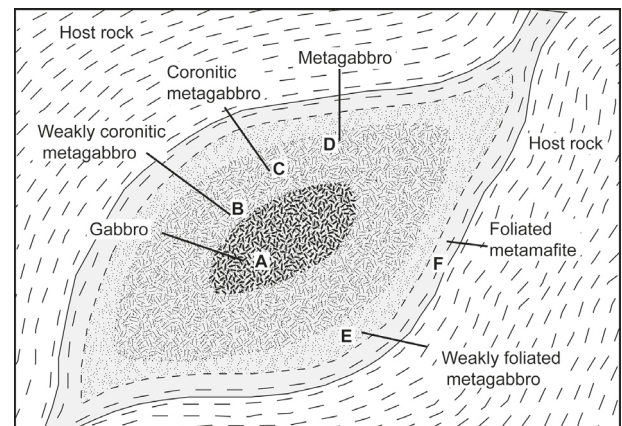
# Scope of the thesis

This thesis focuses on metamorphic zircon formation in mafic intrusions, specifically Fe-Ti-rich gabbro and dolerite, in granitic host rocks. The investigated bedrock represents what is commonly referred to as “continental crystalline crust” or “basement”, which was involved in orogenesis long after its original formation. The investigated rocks are part of the Baltic Shield or “Baltica basement nappes”, the latter in the Scandinavian Caledonides. The emphasis of this thesis is on microtextures, detailing the progressive formation of metamorphic zircon in rocks, which have undergone different degrees of metamorphic recrystallization. Metagabbro bodies may serve as natural laboratories, because they are competent rocks with a simple primary igneous mineralogy, in which zircon is absent or scarce. Metamorphic reactions are commonly incomplete where there has been a shortage or absence of fluids; in such cases, metagabbro preserves domains, which have partly escaped metamorphic recrystallization (Mørk, 1985; Rubie, 1998; Engvik et al., 2011; Fig. 4).



**Figure 4.** Scanned thin-section. Showing the gradual development of new mineral assemblages at increasing metamorphic recrystallization, from the Herrestad gabbro to garnet amphibolite transition.

These metamorphic transition zones provide a unique opportunity to study zircon-forming reactions in mafic rocks. Localities have been chosen where gabbroic rocks show gradual transitions in the scale of a few decimetres to several tens of meters, from well-preserved pristine igneous gabbro, over coronitic and partly recrystallized rock varieties, to fully recrystallized and deformed meta- mafite (Fig. 5).



**Figure 5.** Schematic illustration of metamorphic and deformational relations in a metagabbro lens. A: Preserved gabbro core with pristine igneous mineralogy. B: Weakly-coronitic metagabbro with well-preserved igneous mineralogy and texture. C: Coronitic gabbro where primary minerals to large extent replaced by metamorphic, although the igneous texture is still preserved. D: Metagabbro where metamorphic mineral replaced primary igneous minerals. E: Completely recrystallized and weakly-foliated metagabbro often forms light and dark domains revealing its primary mineralogy. F) Fully recrystallized and strongly deformed meta- mafite. Stage A: Unmetamorphosed rock. Stage B: Initial stages of metamorphic recrystallization. Stage C-D: Intermediate metamorphic recrystallization. Stage E-F: Advance stages of metamorphic recrystallization, with or without deformation (from paper III).

The main aims have been to (a) document reaction textures in which metamorphic zircon is formed, (b) elucidate the mechanism(s) for metamorphic zircon formation at different stages of metamorphic recrystallization, and (c) to propose, if possible, a generally applicable model for metamorphic zircon formation in gabbroic rocks.

Two case studies (papers I and II) document in detail the petrography, reaction textures, and zircon-forming mechanisms in different parts of metamorphic transition zones: a gabbro-to-eclogite transition at Vinddøldalen in the 0.4 Ga Scandinavian Caledonides, and a gabbro-to-garnet amphibolite at Herrestad in the 1.0 Ga Sveconorwegian orogen. Both papers include U-Pb analysis of zircon, pin-pointing the ages of the igneous protolith crystallization and the metamorphic recrystallization. Paper III has a broader approach, aiming at a compilation and comparison of zircon-forming textures in gabbro and dolerite at different metamorphic grades, from sub-greenschist to granulite facies.

Collectively, the rocks studied in this thesis show overwhelming evidence that metamorphic zircon in gabbroic rocks formed at the instance metamorphic fluids gained access to the rock, and that this is most commonly the case during prograde to peak metamorphic conditions. This is elaborated further below (chapter 6).



# Methods

Systematic sampling across the progressive recrystallization of gabbro made it possible to identify different types of zircon and relate them to reaction textures as well as to different stages of metamorphic recrystallization. A brief description of the methods used in these papers follows below and is divided into: textural analysis, geochemical analysis and geochronology.

## Textural analysis

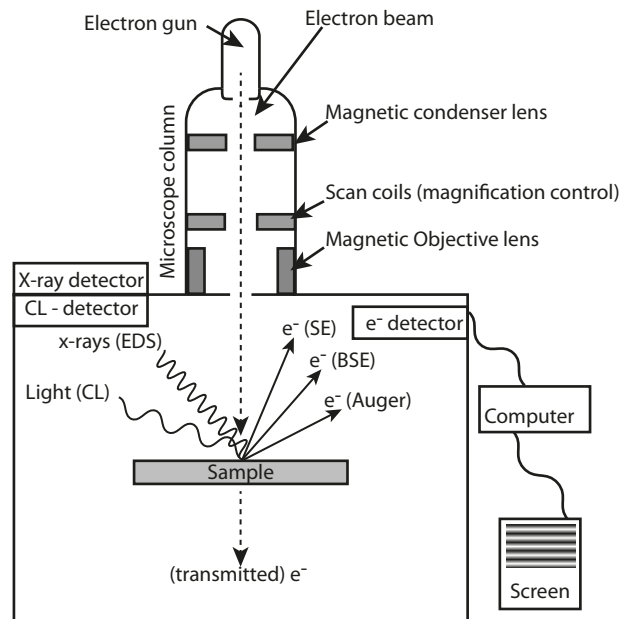
Petrographic description of rock samples was made by polarized light microscopy studies of 0.03 mm thick polished thin-sections (Fig. 4), using transmitted light for silicate minerals and reflected light for opaque phases. Polarized light microscopy is a fast and inexpensive method for the identification of minerals and their textural relations. The disadvantage of polarized light microscopy is where mineral crystals are small ( $< 40 \mu\text{m}$ ) and where the chemical composition needs to be determined. For identification and to localize zircon and baddeleyite grains, thin-sections were also examined using scanning electron microscopy (SEM). Detailed SEM imaging is a prerequisite for revealing the internal textures in zircon and baddeleyite. All imaging was performed on a Hitachi 3400N scanning electron microscope equipped with an Oxford EDS at the Department of Geology, Lund University.

## Principles of Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) is a powerful analytical tool for imaging the surfaces of solid materials at high resolution. It is a non-destructive method that focuses a primary beam of high-energy electrons on to the sample surface of a solid material. The impact of the primary beam onto the sample surface produces a secondary beam, consisting of electrons and photons of various energies, used to retrieve images or chemical information about the sample.

The SEM instrument (Fig. 6) consists of an electron source (electron gun), which generates the electron beam that is accelerated down through the microscope column, where an electromagnetic lens shapes, moves, focuses and regulates the current density of the beam onto the sample. The primary beam scans over the sample in a raster pattern to collect information over a defined area of the sample. Signals from the secondary beam are collected, amplified and converted into a voltage signal by the detector; the intensity is then converted into an image by a computer system so that each point on the sample corresponds to a point on the image. Magnifica-

tion is achieved by scanning a smaller area; in theory, an infinitely small scan should produce an infinitely high magnification (Skoog et al., 2017). The best resolution (down to about 5nm) is gained by secondary electrons (SE), which produce a topographic image of the surface. However, all secondary signals can be used for surface analysis or imaging; the three used here are backscattered electrons (BSE), photons or light (CL), and X-rays for chemical identification (EDS; see below). The greatest advantage of SEM compared with optical microscopy is higher resolution and that thin-sections are not required. The disadvantage is that samples must be coated with an electrically conductive surface layer (normally carbon), analysed under vacuum to avoid interaction with the beam and that the detector needs to be cooled (on older models) to cryogenic temperatures.



**Figure 6.** Schematic illustration of a scanning electron microscope (SEM). Dashed line illustrates, primary beam hitting the sample and producing various different secondary beams of electrons and photons.

## Backscatter electron (BSE) and cathodoluminescence (CL) imaging

Backscattered electrons are primary electrons that interacted with the sample atoms before bouncing back. Heavy elements backscatter electrons more strongly than light elements, which means that the intensity of the BSE signal corresponds to the average atomic number ( $Z$ ) in the sample and hence, yields a compositional image of the average atomic number. Zircon and baddeleyite appear very bright, in comparison to the main rock-forming

silicate-minerals in BSE images. The method therefore offers a quick means to identify and locate mineral grains of zircon and baddeleyite in the sample. However, different minerals with a similar average atomic number can appear the same under BSE e.g., pyrite is easily mistaken for zircon. Reducing brightness and enhancing contrast make differentiation between baddeleyite and zircon, as well as different compositions within zircon grains.

Cathodoluminescence occurs when photons are emitted from a sample and a SEM equipped with a CL detector registers the different wavelengths to produce a CL image. The CL emission from a mineral depends both on the crystal structure and on the chemistry (Vavra, 1990). In zircon, the foremost regulator of CL emission is the trace element content. As an example - in zircon, higher uranium concentration decreases CL emission and increases BSE emission (Rubatto & Gebauer, 2000). SE imaging has the greatest image-resolution, although BSE and CL imaging produces the best resolution concerning internal textures. Different zoning patterns (e.g., oscillatory or patchy zoning) and textures (e.g., overgrowths) are readily visible in CL and BSE and are used to interpret the origin of zircon grains and zircon domains (e.g., Corfu et al., 2003). BSE and CL imaging are consequently useful both for the location of individual zircon and baddeleyite grains and for the detection of igneous or metamorphic textures within the grains.

## Geochemical microanalysis

### Energy Dispersive X-ray analysis (EDS)

Electrons from the primary beam may eject tightly bound inner shell electrons from atoms in the sample; outer shell electrons fill the vacancies and release energy as X-rays that are characteristic of the atomic species. An energy-dispersive (EDS) detector is used to collect and separate the X-rays into an energy spectrum, i.e., a plot of x-ray counts vs. energy (keV). The different energy peaks correspond to the various elements in the sample. EDS can also be used in (semi-) quantitative mode to determine the chemical composition by the peak-height ratio relative to a standard. The minimum detection limit depends on the element and the matrix in which the element occurs, but about 1000 ppm or 0.1wt% is common. Elements in low concentration will generate X-ray peaks, which may not be resolvable from the background radiation (Reed, 1995). The Zr content of minerals was therefore mapped by PIXE (see below).

### Particle Induced X-ray emission (PIXE)

Particle induced X-ray emission (PIXE), works similarly to EDS, but instead of electrons, protons are used to

stimulate the emission of X-rays from the sample. The advantage is a much lower background radiation, hence a better peak to noise ratio. Therefore, PIXE has a higher sensitivity for trace elements than EDS, and trace element contents down to about 2 ppm can be detected.

## Geochronology

Absolute ages can only be obtained by radiometric dating of minerals. A suitable mineral for geochronology allows for some substitution of the parent isotope into its crystal lattice but excludes the daughter isotope during crystallization. Over time, if the system remains closed, the parent will decay and the daughter accumulate in the mineral. The ratio between the two can then be measured and will be a function of time. Different aspects, for example: crystallization, deformation, metamorphism or cooling, can be dated depending on the isotope system and mineral used. The choice of geochronometer and analytical technique is important in order to obtain accurate and precise ages. In this thesis, two mass spectrometry techniques were used to analyse the U-Pb system of zircon and baddeleyite. Zircon is used for determine the age of magmatic and metamorphic events, in contrast to baddeleyite, which records igneous crystallization.

### General principles of U-Pb geochronology

Natural lead has four isotopes:  $^{204}\text{Pb}$ ,  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$  and  $^{208}\text{Pb}$  whereof the latest three are radiogenic, formed through a series of intermediate radioactive daughter isotopes, by the decay of  $^{238}\text{U}$ ,  $^{235}\text{U}$  and  $^{232}\text{Th}$ , respectively. The parent isotope decay exponentially with time, and the rate of decay depends on the decay constant ( $\lambda$ ; the probability of decay for a specific isotope per unit time) and the half-life ( $t_{1/2}$ ; the time it takes for half of the number of parent isotopes to decay). As time passes, the ratio of mother and daughter isotope changes according to:

$$D = D_0 + N(e^{\lambda t} - 1) \quad \text{Eq: 1}$$

where D is the total amount of daughter isotope in the system at a given time.  $D_0$  is the presence of daughter isotope in the system from the beginning, N is the amount of parent isotope, and t is the time elapsed since the system closed and  $\lambda$  is the decay constant (Faure, 1995). The advantage of the U-Th-Pb geochronology is that it relies on three independent isotopic systems with identical chemical behaviour, but different decay rates. Hence, equation: 1 can be solved independently for each of the three systems, yielding three different age equations.

Although most zircons and baddeleyite, excludes lead at the time of crystallization small amounts of can still be present. Initial lead together with lead introduced

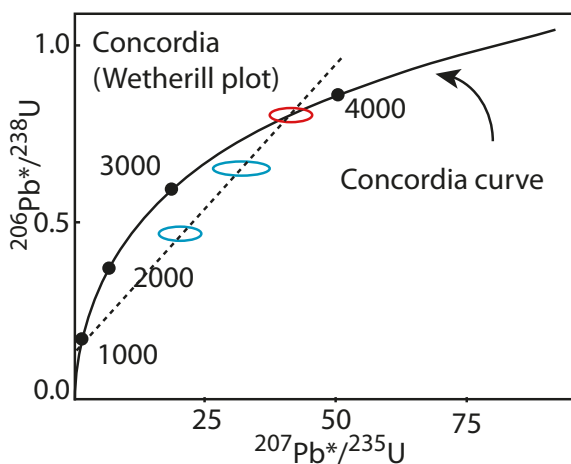
to the sample from other sources, is called common lead (Pbc), and needs to be corrected for. The only non-radiogenic isotope, <sup>204</sup>Pb, can be used to estimate the amount of common lead. If necessary, the initial isotopic composition can be assumed based on the bulk-Pb evolution model of Stacey & Kramers, 1975. If the initial lead negligible or Pbc is corrected for, equation 1 for the two U-Pb system can be simplified to:

$$(^{206}\text{Pb}^*/^{238}\text{U}) = (e^{\lambda t} - 1) \tag{Eq: 2}$$

$$(^{207}\text{Pb}^*/^{235}\text{U}) = (e^{\lambda t} - 1) \tag{Eq: 3}$$

where \* stands for radiogenic lead.

<sup>235</sup>U decays faster ( $t_{1/2} = 0.7083$  Ga) than <sup>238</sup>U ( $t_{1/2} = 4.468$  Ga) however, under ideal conditions, the two decay schemes, or “clocks”, should yield the same age. The two independent radioactive decay systems can be combined in a concordia diagram (Fig. 7). Each point on the concordia corresponds to a specific age, and any age of a closed system should plot on the concordia curve. Open system behaviour will cause a discrepancy between the two isotope systems, and cause discordance, i.e. deviation from the concordia curve. Such analyses will typically plot below the concordia curve. Providing multiple fractions are analysed and isotopic disturbance occurred at a single event (i.e. during metamorphism), then in the ideal case a straight line fitted through the discordant analyses will result in two intercepts along the concordia curve. In this case, the upper intercept will give the age of the zircon formation and the lower intercept the time of isotopic disturbance.



**Figure 7.** Normal concordia diagram also called Wetherill diagram. The black line is the concordia curve, where the <sup>206</sup>Pb/<sup>238</sup>U age equals the <sup>207</sup>Pb/<sup>235</sup>U age. Black dots on the concordia curve represent the age in Ma. Red circle is a concordant analyse plotting on the concordia curve, whereas blue circles are discordant and plot below the concordia curve. Dotted line fitted through the blue analyses; yield the upper and lower intercept age where it cuts the concordia.

### Isotope dilution thermal ionization mass spectrometry (ID-TIMS)

This method was used for age determination of zircon and baddeleyite (paper 1). As the name implies, the mineral to be analysed is dissolved before mass spectrometer analysis. Sample preparation starts with crushing and milling, and separation of heavy mineral fractions (including zircon and baddeleyite) using a combination of heavy liquids and magnetic separation techniques, or just a water-shaking table (see Söderlund and Johansson, 2002). Thereafter zircon or baddeleyite grains are hand-picked under the microscope. Zircon grains can be abraded (physically by air, or chemically) to remove damaged parts most prone to Pb loss (Krogh, 1982; Mattinson, 2005). Due to the fragile nature of baddeleyite, abrasion is not a feasible technique for baddeleyite (Rioux et al., 2010). Single grains or a number of mineral grains are dissolved in a mixture of HF:HNO<sub>3</sub> (10:1) and a small amount (usually 1 drop) of a tracer solution is added. The tracer (or spike) is of known chemical and isotopic composition and allows for precise measurement and calculation of U and Pb isotope concentrations in the sample. Prior to analysis in the mass spectrometer, the U and Pb can be separated by ion exchanges chemistry, but this is not necessary for small samples (single to a few grains). The sample is loaded onto an outgassed Rh filament and placed in the mass spectrometer. The filament is gradually heated to produce ions that are separated by their mass-to-charge ratio

### Secondary ion mass spectrometry (SIMS)

Secondary ion mass spectrometry (SIMS) is a high spatial resolution technique, for measuring isotopic and chemical composition of solid materials. A high-energy primary ion beam, typically 10-25 μm wide, is focused onto the sample, causing the surface layers of atoms to be stripped (sputtered) off. The ablation of the sample surface is typically only a few microns and SIMS is considered to be a non-destructive method. The small diameter of primary beam makes it possible to target small grains in thin-sections or specific domains within single crystals.



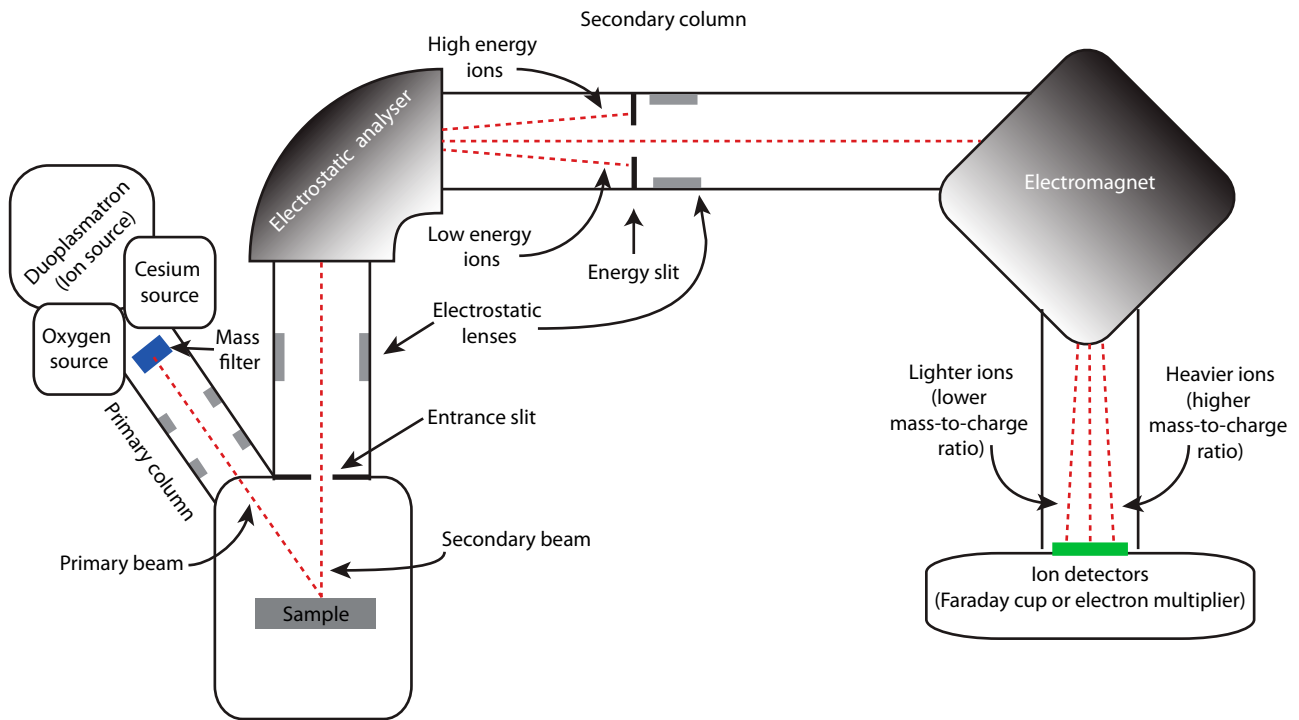
**Figure 8.** Thin-sections mounts for SIMS analysis.

The extraction of zircon is essentially the same as for TIMS, (standard separation techniques involving crushing, water-shaking-table, magnetic separation and hand picking of zircon grains) described above. Separated zircons and standard (91500 zircon standard) are mounted together, covered in epoxy and polished so that the centre of the zircon (or area of interested) is exposed. For in situ analysis in thin-section, zircon grains were first identified in the thin-sections using optical microscopy and BSE-imaging. Thereafter, domains of interest were cut

out using a diamond saw and mounted in epoxy together with standards (Fig. 8). Mounts are coated with a thin conductive gold surface.

For the study reported in paper II of this thesis, U-Pb geochronology was performed using a Cameca 1270-80 ion probe at the NordSIM laboratory, Museum of Natural History in Stockholm.

The SIMS instrument comprises an ion source, a sample chamber, a primary column, a secondary column, and ion detectors (Fig. 9).



**Figure 9.** Schematic illustration of a secondary ion mass spectrometer (SIMS). Dashed line indicate trajectory of the ion beam.

For analysis of positive ions (e.g., U and Pb) a primary ion source of oxygen ( $^{16}\text{O}^+$ ), which increases the yield of positive secondary ions, is used. The primary beam travels through the primary column, where it is mass filtered (to remove impurities in the beam) and focused by a series of electromagnetic lenses on to the sample. Secondary ions are extracted from the sample surface and accelerated into the secondary column. The first part of the secondary column also functions as an ion microscope and focuses the secondary beam into the mass spectrometer. The ions enter the mass spectrometer with a wide range of (kinetic) energies. In a double focusing mass spectrometer, the ions are separated in two steps: first by an electrostatic sector followed by a magnetic sector. The electrostatic analyser involves a curved electrostatic field that bends low energy ions more strongly than high-energy ions, so that an energy slit behind the electrostatic

analyser can be set to stop ions of unwanted energies (i.e., multi-atomic ions). The energy reduced ion beam is admitted to the magnetic sector where ions pass through a curved magnetic field that deflected ions by their mass-to-charge ratio. The dispersed ions fall on the detector device, commonly a multicollector system of electron multipliers and Faraday cups, that allows simultaneous measurement of several secondary ion signals (Skoog et al., 2017).

Compared with SIMS, TIMS is the most precise analytical technique, yielding better temporal resolution, although SIMS is better for composite zircon grains with multiple age domains. SIMS also requires an external standard against which the U/Pb ratio of the unknown zircon needs to be calibrated.

# Summary of component papers

Three papers form the core of this thesis. Their summaries are given below, arranged in chronological order. The authors' respective contributions are listed in Table 1.

## Paper I

*Beckman, V., Möller, C., Söderlund, U., Corfu, F., Pallon, J., & Chamberlain, K. R. (2014). Metamorphic zircon formation at the transition from gabbro to eclogite in Trollheimen–Surnadalen, Norwegian Caledonides. Geological Society, London, Special Publications, 390(1), 403–424.*

Rocks and minerals in high-pressure and -temperature (HPT) metamorphic terranes can preserve records of both burial and exhumation. U-Pb analysis of zircon is the most common method for age determination of HPT metamorphism, since this is one of few isotopic systems that are not reset at temperatures above 650°C. The challenge lies in assessing the stage of the metamorphic cycle during which zircon formed.

In this study, we investigated Zr-bearing accessory minerals at a gabbro to eclogite transition in allochthonous basement in the Trollheimen area, above the Western Gneiss Region, in Norway. Samples were collected along a profile from seemingly unmetamorphosed gabbro, via garnet-coronitic metagabbro, to undeformed eclogite with garnet forming pseudomorphs of the primary plagioclase laths, and further into foliated eclogite. The rock samples preserve igneous and eclogite-facies textures in which three different zircon types were identified: (I) igneous prismatic grains, (II) metamorphic polycrystalline rims and pseudomorphs after baddeleyite, and (III) minute (<20 µm) zircon grains arranged in a strings of bead-like manner. Zircon is scarce in the “unmetamorphosed” gabbro, but all three zircon types are present. In the coronitic metagabbro, bead-like zircons (type III) are abundant along the rims of igneous Fe-Ti oxide grain. These bead-like zircons become more abundant with increasing metamorphic recrystallization, forming up to 500 µm long strings of zircon beads at an increasing distance from the primary Fe-Ti oxide. In foliated eclogite, individual bead-like zircon grains are larger, although the strings are shorter (<350 µm) than in unfoliated eclogite. These zircon grains (type III) are still confined to rutile and ilmenite (former Fe-Ti domains). In eclogite, bead-like zircon grains occur in both high-pressure (garnet and rutile) and retrograde phases (plagioclase and amphibole). Thus, bead-like zircon dates the prograde to peak metamorphic stage (tectonic burial), although not necessarily the deepest or the highest temperature conditions.

Based on the consistent localization of metamorphic zircon around ilmenite and rutile, it was hypothesized that igneous ilmenite may have been the source of Zr. In order to test this hypothesis, the Zr-content of igneous Fe-Ti minerals and pyroxene were analysed by PIXE, revealing an overall low Zr concentration. Separated zircon grains from a sample of foliated eclogite gave an age of  $425 \pm 10$  Ma for the metamorphism (TIMS-analysis by F. Corfu, Oslo University). This age is interpreted to date the metamorphic transition from gabbro to eclogite in the upper basement of the Lower Allochthon in the south-central Scandinavian Caledonides. SIMS *in situ* (in thin section) dates of zircon and baddeleyite (analysis by K. Chamberlain, University of Wyoming) are comparable to separated baddeleyite grains dated by TIMS (analysis by U. Söderlund, Museum of Natural History in Stockholm), the latter yielding an igneous crystallization age of  $1457 \pm 11$  Ma for the gabbro. The results also show that remnant baddeleyite can survive eclogite-facies metamorphism if shielded from silica-rich fluids.

## Paper II

*Beckman, V., Möller, C., Söderlund, U., & Andersson, J. (2017). Zircon Growth during Progressive Recrystallization of Gabbro to Garnet Amphibolite, Eastern Segment, Sveconorwegian Orogen. Journal of Petrology, 58(1), 167–187.*

A challenging task in metamorphic terranes is to correctly identify or interpret the protolith, especially where excessive ductile deformation has eliminated all original textures and structures. A 10 km<sup>2</sup> large gabbro–metagabbro body at Herrestad, in the southeastern Sveconorwegian Province, offers the opportunity to study a gabbro intrusion, which has undergone different degrees of recrystallization and deformation under upper amphibolite-facies conditions. The gabbro–metagabbro body has several transitions from well-preserved gabbro to garnet amphibolite. Systematic sampling through these transitions made it possible to follow the zircon-forming processes in detail. The microtextures suggest that the majority of zircon formed at sub-solidus conditions, during the breakdown of baddeleyite in the presence of fluids. Internal textures of zircon (using CL) in garnet-porphyroblastic amphibolite reveal a polycrystalline origin also of the metamorphic zircon in completely metamorphosed varieties, hence also formed by reaction of baddeleyite to zircon. Zircon dating was performed using SIMS, at the Nordsim laboratory, Museum of Natural History in Stockholm. Five different zircon types were dated *in situ*

in thin sections. Igneous zircon grains gave  $1567 \pm 5$  Ma, within error similar to a baddeleyite TIMS age of  $1574 \pm 9$  Ma (Geological Survey of Sweden database). In situ dating of small zircon grains gave a vast range of apparent age, mainly caused by the analytical spot being larger than the target. Separated zircon from garnet amphibolite, sets the age for amphibolite facies recrystallization to  $970 \pm 7$  Ma.

lite, sets the age for amphibolite facies recrystallization to  $970 \pm 7$  Ma.

Table 1. Author contributions

	Paper I	Paper II	Paper III
Study design	C. Möller	V. Beckman C. Möller	C. Möller V. Beckman
Manuscript writing	V. Beckman* C. Möller* U. Söderlund F. Corfu K.R Chamberlain	V. Beckman* C. Möller* U.Söderlund J.Andersson	V. Beckman* C. Möller*
Manuscript illustrations	V. Beckman C. Möller	V. Beckman C. Möller	V. Beckman C. Möller
Field work and sampling	C. Möller	V. Beckman C. Möller	C. Möller U. Söderlund J. Andersson L. Johansson V. Beckman
Petrography	V. Beckman C. Möller	V. Beckman	V. Beckman
SEM	V. Beckman C. Möller	V. Beckman	V. Beckman
TIMS			
– sample preparation (Zrn)	U. Söderlund		
– analysis (Zrn)	F. Corfu		
– interpretation of analytical data (Zrn)	F. Corfu U. Söderlund V. Beckman		
– sample preparation (Bdy)	U. Söderlund V. Beckman		
– analysis (Bdy)	U. Söderlund		
– interpretation of analytical data (Bdy)	U. Söderlund V. Beckman		
SIMS			
–sample preparation	V. Beckman K.R Chamberlain	V. Beckman K. Linden	
– analysis	K.R Chamberlain	V. Beckman L. Levinsky M. Whitehouse	
– data reduction			
– interpretation of analytical data	V. Beckman K.R Chamberlain U. Söderlund	V. Beckman J. Andersson	
PIXE			
– measurement	J.Pallon V. Beckman		
– data reduction	J.Pallon		
– interpretation of analytical data	V. Beckman		

\*Main contributors

## Paper III

*Beckman, V & Möller, C. Prograde metamorphic zircon formation in gabbroic rocks: the tale of microtextures. (Manuscript submitted to Journal of Metamorphic Geology).*

Zircon is one of very few minerals, which can be used for dating metamorphic events at high metamorphic temperatures (>650 °C). This applies in particular to cases where mafic rocks such as eclogite, mafic granulite, and garnet amphibolite are key targets of investigation. For tectonic interpretation, it is crucial to understand whether the dated zircon formed during prograde metamorphism (heating), decompression, or retrogression (cooling). This paper presents zircon textures from six samples of gabbro and dolerite at different metamorphic grades, from sub-greenschist to granulite facies, in the Fenoscandian Shield. The documented textures reveal that, independent of metamorphic grade, the main mechanism of zircon formation is similar at initial stages of metamorphic recrystallization, and that baddeleyite is the major source of Zr. The zircon-forming reaction was dependent on the introduction of hydrous fluids into the dry gabbro body. Such infiltration is expected to take place during prograde metamorphism, from the dehydration of lower-grade and hydrous minerals.

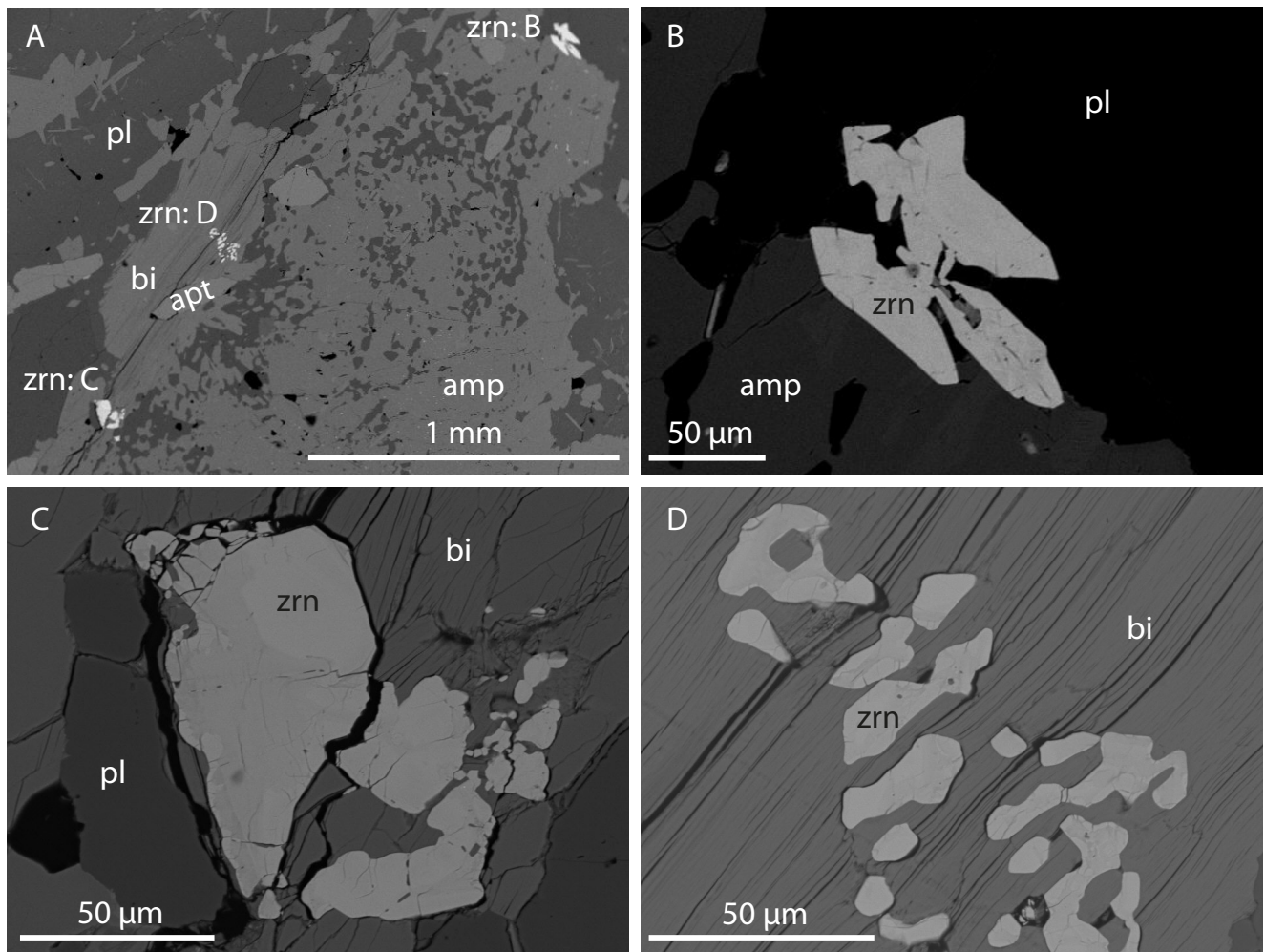
# Summary of zircon textures

In the papers presented in this thesis, different kinds of zircons have been defined based on their textural appearance. The age and origin cannot be proven for zircons crystals which have not been dated, but this study confirms that characteristic zircon types can be distinguished based on their morphology and internal texture. Below are a summary of the different zircon types and the interpretation of their petrogenesis.

### *Igneous zircon*

Igneous zircon grains are present in coarse-grained gab-

bro along with baddeleyite. The igneous zircon grains show a great variety in morphology and internal texture (Fig. 10). Most of them are unzoned, but some have faint broad-banded oscillatory zoning. Igneous zircon is typically CL-dark, and altered domains often comprise BSE-bright inclusions of U and Th. The low CL emission is possibly related to the high U concentration of igneous zircon (paper II). The formation of igneous zircon in coarse-grained gabbro is thought to reflect an increase of silica in the magma, at a late magmatic stage (Scoates & Chamberlain, 1995, Boehenke et al., 2013).



**Figure 10.** Illustrates the wide variety of igneous zircon morphology. A) BSE image overview of area in coarse-grained metagabbro, at stage C in fig. 5, (Herrestad; upper amphibolite facies; sample VV01b, paper II). B-D) are close up of zircon areas in A). All three zircon grains are dated and confirmed magmatic in origin (paper II).

A few zircon grains, texturally characterized as magmatic, carries frequent µm-sized inclusions of silicate minerals (Fig.11). This texture either formed as post-metamorphic alteration, perhaps due to later metamictisation or from zircon, high in non-formula elements, i.e., U and Th making the zircon more prone to alteration by coupled dissolution-precipitation during fluid ingress (Geisler

et al., 2007) generating inclusion rich or even porous zircon.

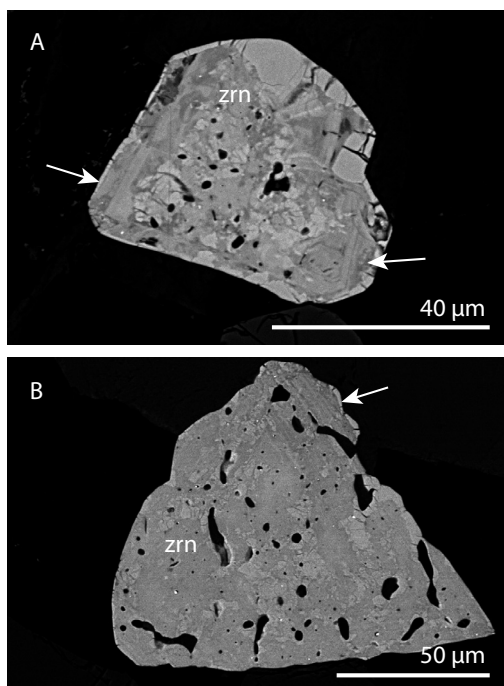


Figure 11.

Two examples of altered igneous zircon. A) from coarse-grained metagabbro, stage C in fig. 5, (Bassö; upper amphibolite to granulite facies, sample Bro01a, paper III) and B) from weakly foliated garnet amphibolite, stage E-F in fig. 5, (Herrestad upper amphibolite facies; sample VV30, paper II). Arrows indicate faint oscillatory zoning.

### *Inclusion rich zircon*

In one metagabbro sample (Paper II), inclusion zircon occurs (Fig. 12). The inclusion rich zircon grains also show faint CL emittance, in contrast to polycrystalline zircon, and are interpreted as altered igneous zircon (paper II). The inclusion rich zircon grains are only found in one sample, and are texturally similar to the hydrothermal zircons described by Rubin et al., (1989).

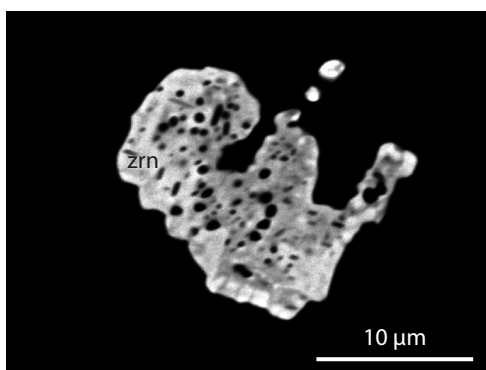


Figure 12.

Inclusion rich zircon from Herrestad metagabbro, stage D in fig. 5 (Herrestad upper amphibolite facies; sample VV31b, paper II).

### *Zircon overgrowth on baddeleyite*

Thick zircon edges or “rims” on baddeleyite in close proximity to igneous zircon indicate that magmatic zircon can form overgrowths on baddeleyite grains (Fig. 13a). Texturally,

these rims appear to be monocrystalline or consist of fewer and better-developed zircon crystals in comparison with most polycrystalline zircon rims on baddeleyite (described below). In mafic magmas, zircon saturation is reached during the late stages of crystallization (Hanchar & Watson, 2003; Boehnke et al., 2013), and baddeleyite and zircon (if present) are both thought to crystallize late. Theoretically, if Zr saturation is reached before silica saturation (Schaltegger & Davies, 2017), baddeleyite will crystallize before zircon.

### *Metamorphic zircon*

Three types of metamorphic zircon have been identified, and textures suggest that all three types formed from the breakdown of baddeleyite in the presence of a silica-rich fluid (cp. Davidson & van Breemen, 1998).

### *Polycrystalline zircon*

Polycrystalline zircon occurs as rims (Fig. 13b) on baddeleyite at the initial stages of metamorphic recrystallization and as polycrystalline pseudomorphs or aggregates after baddeleyite at the intermediate stages of metamorphic recrystallization (Figs. 14a, b, d). This type of zircon is common at the initial and intermediate stages of metamorphic recrystallization, although scarce or missing at the advanced stages of metamorphic recrystallization. The fate of polycrystalline zircon at advanced stages of metamorphic recrystallization is treated separately below (Metamorphic zircon).

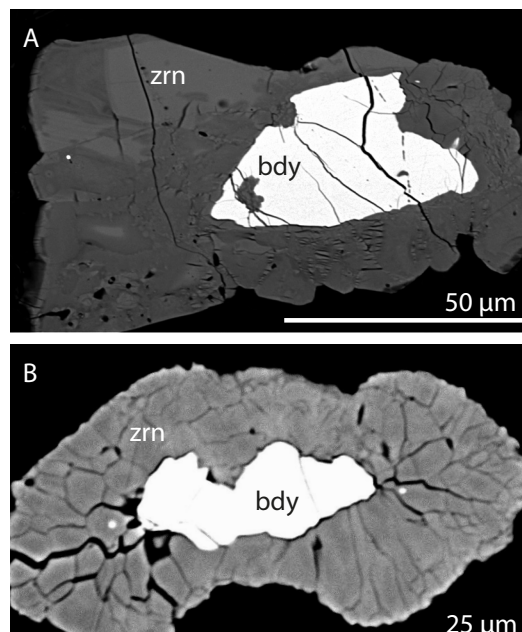


Figure 13.

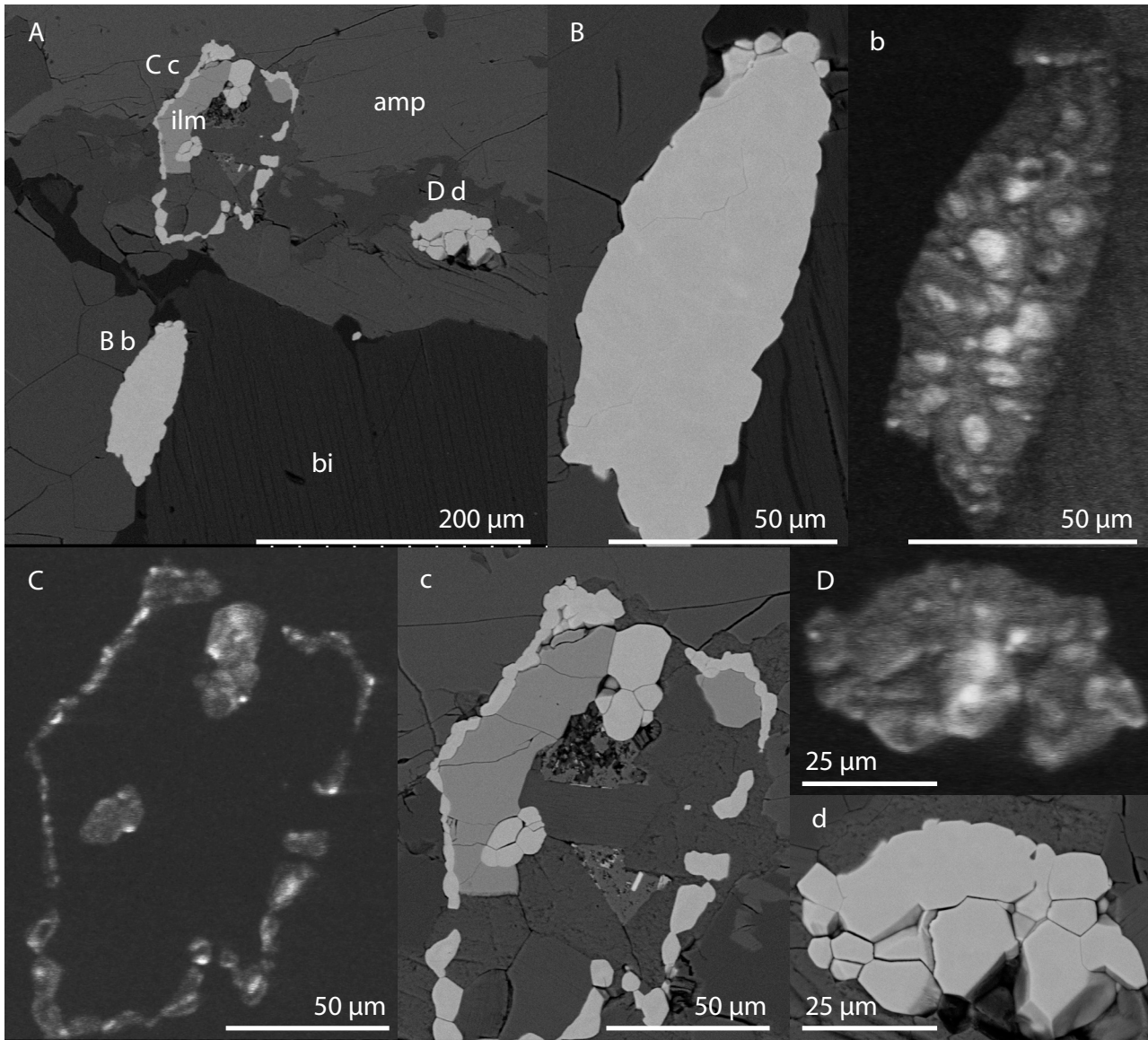
Comparison of baddeleyite with A) an interpreted additive overgrowth of magmatic zircon on baddeleyite in coarse-grained olivine-free metagabbro, stage A-B in fig. 5 (Herrestad upper amphibolite facies; sample VV01c, paper II). B) “normal” replacement of baddeleyite by metamorphic zircon (same sample as in A). Not also that the core of baddeleyite in A is more well-preserved compared with the core in B.



*Bead zircon*

Minute zircon grains, mostly about 2 µm in diameter, preferentially nucleating on the Fe-Ti oxide rims (Figs. 14a, c), appear simultaneous with the replacement of

baddeleyite rims by zircon. With increasing metamorphic recrystallization, the strings of zircon beads become longer and occur at an increasing distance to Fe-Ti oxides. Bead zircon is the most commonly occurring zircon type in completely recrystallized samples.



**Figure 14.**

BSE and CL images of polycrystalline zircon after baddeleyite and bead zircon in metagabbro, stage B in fig. 5 (Bassó, upper amphibolite – granulite facies; sample B7, paper II) A) Overview of zircon grains (BSE). A a, B b, C c and D d correspond to close up of zircon grains in BSE and CL. B) Polycrystalline zircon showing no internal texture in BSE but is clearly polycrystalline in b) CL-image. C) CL image of bead zircon only partly outlining small ilmenite. Note faint polycrystalline appearance of the largest zircon grain in top of the picture. c) Same area in BSE. D and d) Close up of grain D in A of smaller polycrystalline zircon aggregate.

*Metamorphic zircon*

At advanced stages of metamorphic recrystallization (Fig. 5), the zircon forming reactions are harder to follow, and polycrystalline zircon at first seemed to be missing. However, while polycrystalline zircon is missing, a population of somewhat larger, about 20 µm in diameter, rounded metamorphic zircon grains is present (dated in papers I and II). The latter metamorphic zircon grains are difficult to find in thin-sections but are plentiful (and up to

40 µm large) in heavy mineral separates from crushed samples (see papers I and II). The metamorphic zircon grains appear homogeneous in BSE images, but consist of multiple nuclei as revealed by CL images (papers II and III). Based on their multiple nuclei appearance in CL the metamorphic zircons are interpreted to have originated as polycrystalline zircon aggregates after baddeleyite, but under full fluid access and completed net-transfer metamorphic reactions.

# Discussion

The primary aim of this thesis is to document and interpret zircon formation in gabbroic rocks, by detailed textural observation, with the intention to link zircon ages to reaction textures. For this purpose, the studies have focussed on the characterization of microtextures related to the Zr-bearing phases baddeleyite and zircon, in gabbro and metagabbro “frozen” in various stages of metamorphic recrystallization. The principal conclusions are discussed below.

## The quest for zirconium

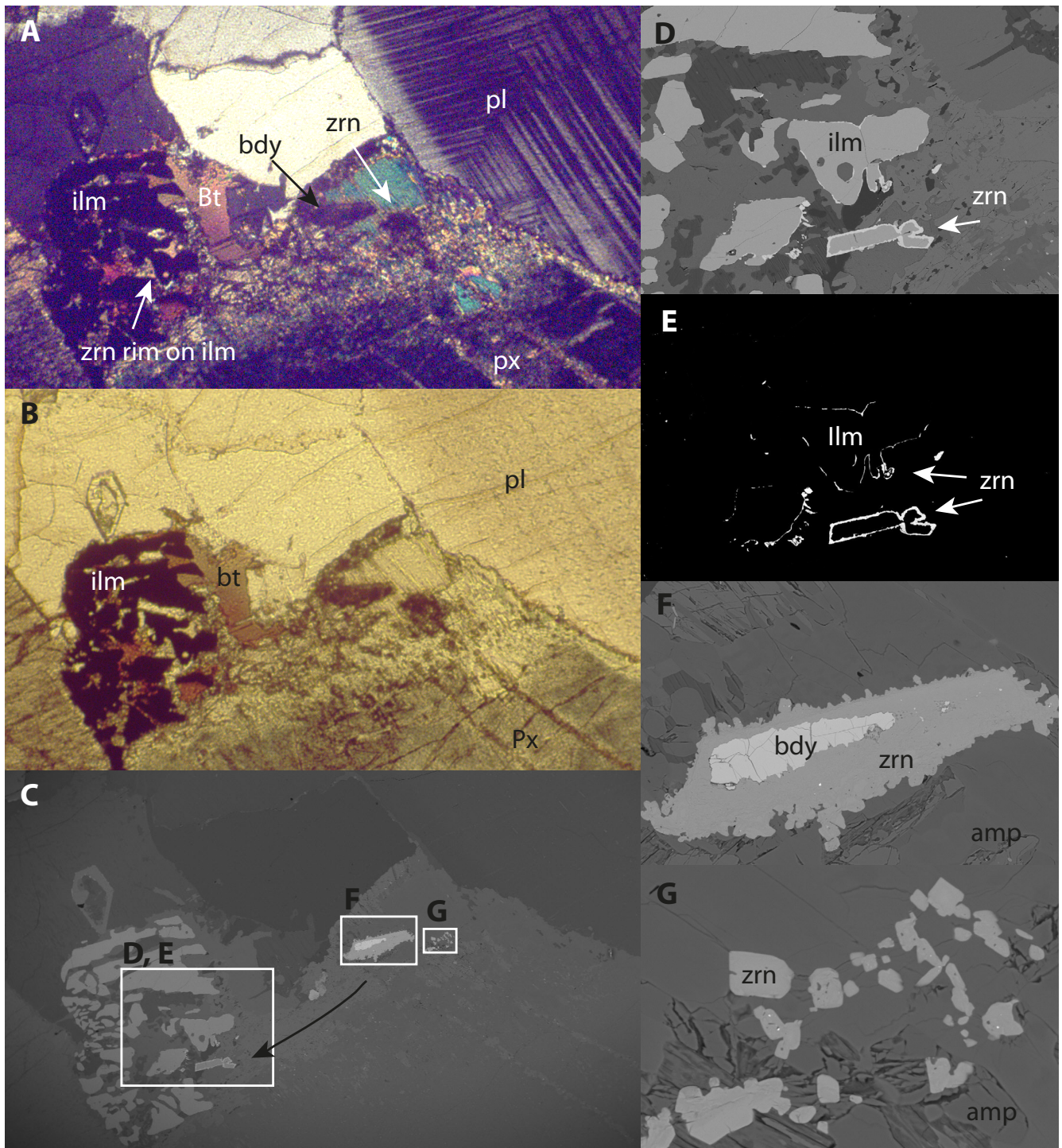
Metamorphic zircon can form through several different processes, alteration, and replacement/recrystallization of pre-existing zircon during metamorphism or new growth (Rubatto, 2017). In contrast to new growth, alteration or recrystallization process will not change the modal abundance of zircon in a sample. In mafic silica saturated or undersaturated rocks, igneous zircon; the primary carrier of Zr in felsic rocks (Bea et al., 2006) is scarce or absent (Boehnke et al., 2013). The alteration or recrystallization of igneous zircon is therefore not considered a major source for metamorphic zircon in mafic rocks. As a consequent, a source of Zr is required to form metamorphic zircon at subsolidus conditions in mafic rocks.

The consistent localisation of metamorphic zircon near Fe-Ti oxides (paper I), suggests that igneous Fe-Ti oxides are a potential source of Zr. Similar textures and interpretations have been described previously (Bingen et al., 2001; Söderlund et al., 2004; Charlier et al., 2007; Sláma et al., 2007). PIXE Zr-mapping of Fe-Ti oxides, plagioclase, and pyroxene (Paper I) revealed an overall low Zr content in igneous Fe-Ti oxide, except for a few irregular patches plausibly caused by zircon grains immediately beneath the surface (Paper I; and discussion in paper III).

Several minerals can contain a trace amount of Zr, such as garnet, rutile, hornblende, ilmenite, and pyroxene (Fraser et al., 1997; Degeling et al., 2001; Bea et al., 2006), and provide a source of zircon. The problem lies in that, with the exception of pyroxene and ilmenite, garnet, rutile and hornblende are not a significant primary constituent of the gabbroic igneous rocks investigated here. Instead, these minerals are part of the metamorphic assemblages formed during prograde metamorphism in gabbroic rocks. Mass balance modelling of mafic rocks shows that garnet, hornblende, and rutile can incorporate an increasing amount of Zr with rising temperature and would, therefore, provide a sink for Zr during prograde metamorphism (Kohn et al., 2015).

## Baddeleyite as the source

The contribution of Zr from major igneous minerals crystallized at high temperature, cannot be ruled out as a source of Zr (Rubatto, 2017), although their contribution to the Zr budget is probably far less than from baddeleyite. Baddeleyite is sensitive to metamorphism and easily reacts to form zircon as soon as free silica is available, according to the reaction  $ZrO_2 + SiO_2 = ZrSiO_4$ , and baddeleyite is commonly surrounded by a corona of zircon. This corona is a partial replacement of baddeleyite by zircon, caused by the influx of silica or a change in silica activity (Davidson & Van Breemen, 1988). There is only sparse information available on the redistribution of Zr during the replacement of baddeleyite to zircon. However, given the molar volume of baddeleyite (21,148 cm<sup>3</sup>/mol) and zircon (39,261 cm<sup>3</sup>/mol; Robie et al., 1967) unless Zr is removed in solution, the reaction must involve a significant increase in volume. Zircon rims on baddeleyite or zircon pseudomorphs left after baddeleyite is a commonly described phenomenon, although not (so far) associated with signs of volume increase such as expansion cracks. So, what happens to potential Zr in solution? At a micro-scale level, the baddeleyite-to-zircon reaction must be fluid-assisted, since it requires the addition of silica and the removal of Zr. Fluids play an essential role in zircon formation at sub-solidus conditions (i.e., Rubatto, 2017) and, although Zr traditionally has been regarded as an immobile element, several studies have shown that Zr can be mobile even at low temperatures (Rasmussen, 2005; Hay & Dempster, 2009). The textures reported in this thesis show that the Zr released during the breakdown of baddeleyite precipitates as tiny bead zircon preferentially nucleating on the rims of Fe-Ti oxide and as polycrystalline aggregates after baddeleyite. At the initial and intermediate stages of metamorphic recrystallization, fractures and thin recrystallization zones act as pathways for fluids. It is suggested that where these zones reach Fe-Ti oxide grains, they act as nucleation sites for zircon (Fig. 15). Interestingly, where shielded from fluids, baddeleyite may persist even under high P-T conditions (papers I, III). This indicates that baddeleyite might be stable up to at least granulite facies in silica-deficient environments (Schaltegger & Davies, 2017).



**Figure 15:** Thin-section images of (coarse-grained and olivine-free) gabbro, stage A-B in fig. 5. (Herrestad, upper amphibolite facies; sample vv01c). A-C) Same area, in cross-polarized light, plan-polarized light and as BSE image. Note the alteration at pyroxene borders. D-E) BSE images of areas in C, showing zircon and baddeleyite textures. Arrow indicates micro-fractures acting as fluid pathways. Note that zircon only nucleate at ilmenite edges facing the baddeleyite.

## Metamorphic zircon

The three types of metamorphic zircon; bead zircon, polycrystalline zircon, and metamorphic zircon are all interpreted as the breakdown product after baddeleyite. The amount of bead zircon increases with metamorphic recrystallization and bead zircon is the most numerous type of zircon in the samples, which have undergone

complete metamorphic recrystallization (papers I & II). Consequently, bead zircon is stable throughout progressive metamorphic recrystallization and occurs as inclusion in all prograde metamorphic minerals, such as rutile and garnet (Papers I, II & II), as well as in retrograde minerals, such as titanite (paper I). As baddeleyite decrease, polycrystalline zircon aggregates increase, with progressive metamorphic recrystallization until there is

no baddeleyite left. At complete metamorphic recrystallization, it appears to be a "bimodal" distribution with numerous small grains of bead zircon and larger rounded, less than 40  $\mu\text{m}$ , metamorphic zircon. The metamorphic zircon grains are homogenous in BSE images but have a complex appearance in CL imaging with multiple nuclei (Fig. 14; papers I & II), interpreted as originating from polycrystalline zircon aggregates after baddeleyite, but under full fluid access and complete net-transfer metamorphic reactions.

### Zircon formation at different metamorphic grades.

At initial and intermediate stages of metamorphic recrystallization, the zircon formation is easy to follow and is strikingly similar independent of metamorphic grade. However, at advanced stages, a subtle difference is indicated between different metamorphic grades. The strings of bead zircon reach their maximum length in fully recrystallized eclogite (paper I), although less abundant in deformed rocks, e.g., the foliated retro-eclogite described in paper I. Whereas in completely recrystallized garnet amphibolite (paper II) strings are slightly shorter in comparison with the metagabbro sample. These two samples were metamorphosed at similar temperatures (< 650° C), but different pressures. The difference is subtle and might be due to a different amount of primary baddeleyite in the different samples.

At the advanced stage of metamorphic recrystallization, except for the size, it is hard to distinguish between bead zircon and metamorphic zircon (unless imaged in CL), and it seems that metamorphic zircon is composed of fewer and larger nuclei at increasing metamorphic grade, making the differentiation even harder.

### Prograde, peak or retrograde zircon formation

The question of whether metamorphic zircon grows during the prograde, peak or retrograde stages of the metamorphic evolution is fundamental to geological interpretation. Zircon formation in gabbroic rocks metamorphosed under high pressure and temperature, such as eclogite and high-pressure granulites, is especially important since these rocks are used to reconstruct large-scale tectonic processes, including deep tectonic burial and exhumation during orogenesis.

Even if zircon can form at a wide range of conditions from low to high metamorphic grade, zircon formation is often assigned to date peak or retrograde metamorphism, most because the slow diffusional resetting. According to the equilibrium model (Kohn et al., 2015), zircon dissolves during prograde metamorphism

and grows during retrograde metamorphism through the breakdown of rutile or other mineral phases, which contain significant trace amounts of Zr. This is in contrast to the findings in this thesis (papers I, II & III), where the amount of metamorphic zircon increases with progressive metamorphic recrystallization, independent of metamorphic grade. The main reasons for the discrepancy between the mass balance modelling (Kohn et al., 2015) and natural gabbro (papers I, II & III) are that in the gabbroic rocks studied, the igneous minerals – notable baddeleyite is the primary reservoir of Zr and that the metamorphic recrystallization is dependent on fluid and deformation, leaving dry parts unaffected by metamorphism. The presence of a hydrous fluid is necessary for free silica to reach baddeleyite, sometimes referred to as "kinetic effects."

The abundance of small metamorphic zircon grains, 5-40  $\mu\text{m}$  in diameter, in metamorphosed gabbroic rocks makes it difficult to argue for the dissolution of zircon during progressive metamorphic recrystallization. Instead, metamorphic zircon seems to prefer nucleation above growth, resulting in abundant small grains of metamorphic zircon. It is clear that in the studied rocks, the Zr released from the accessory and major minerals during prograde metamorphism is not entirely taken up by other metamorphic minerals as the temperature increases. The growth of a second generation of metamorphic zircon during retrograde mineral reactions (i.e., the breakdown of rutile and garnet during decompression) cannot be ruled out, but has not taken place in the rocks studied here. Intrusions of water-deficient protoliths such as dolerites and gabbros are dependent on deformation and infiltration of hydrous fluids for metamorphic reactions to take place. External fluids probably emerged from the host rock (gneiss or granite) by dehydration reactions of the hydrous minerals (micas and possibly amphiboles) during prograde metamorphism. The tiny bead zircon and metamorphic zircon after baddeleyite thus seems to have formed by prograde metamorphic reaction and remained stable over the entire P-T evolution, indicating that the Zr became locked into metamorphic zircon during the prograde metamorphism.

### Possibilities and pitfalls for dating

Baddeleyite is a common primary constituent in many mafic undersaturated rocks (Keil & Fricker, 1974; Heaman & LeCheminant, 1993). Therefore, in baddeleyite carrying rocks similar to the Fe-Ti gabbros investigated here, zircon will form at an early stage of metamorphism, provided that the silica activity is high enough. It has been shown that baddeleyite, if shielded from fluids, can retain its igneous U-Pb age at metamorphic temperatures up to granulite-facies (Davidson & van Breemen 1988;

Söderlund et al., 2008, paper I, II). Hence, baddeleyite rimmed by zircon has the potential to yield both a primary magmatic age and the age of a later zircon-forming event. Theoretically, there is also a possibility for multiple zircon formation of metamorphic zircon. For example in a polymetamorphic setting, if baddeleyite was not completely consumed at the first event, the reaction can restart at the later event. Because baddeleyite is replaced by an inward progressing reaction (Davidson & van Breemen 1988, Amelin et al., 1999), the outer parts of the rim should yield the oldest age. The current refinement of technology has made it possible to date small zircon grains or micro-domains within grains, and baddeleyite grains only 3  $\mu\text{m}$  large have been dated successfully by SIMS (Chamberlain et al., 2010). In situ age determination of the tiny bead zircon grains will require analytical methods with higher spatial resolution than available today. When available, analysis of these small grains may open up new possibilities for petrogenetic characterization and dating of fluid infiltration in mafic rocks.

However, since the zircon formation is caused by the interaction of a silica rich fluid, it will date a fluid event and not necessary a metamorphic event. Discordant baddeleyite ages are often attributed to a thin zircon rim, and if a baddeleyite-to-zircon reaction has initiated, nano- or micro-scale inclusions of zircon in baddeleyite (or vice versa) will yield mixed ages (paper II). Mixed ages can also be caused by a zircon rim formed during two different events, for example the mixing of a late magmatic additive rim and a later metamorphic rim (paper I). The

origins of the different parts of such a rim could be indicated by crystal shape and differences in Th/U ratio, since magmatic zircon generally has higher Th/U ratio than both baddeleyite (Heaman & LeCheminant, 1993) and metamorphic zircon (Rubatto & Gebauer, 2000).

Linking of zircon ages to metamorphic mineral reactions remains the most critical step for the interpretation of metamorphic zircon ages. Therefore, the zircon forming mechanism and the drivers for zircon formation must be examined. Zircon geochemistry (such as Th/U ratios or REE partition) is often used to link zircon formation to metamorphic mineral reactions, for example in a garnet-bearing rock, zircon that grows simultaneous with garnet or after garnet will become depleted in HREE. Since garnet growth can take place under both prograde and retrograde metamorphism it does not necessarily zircon formation to a specific limit P-T condition. To discriminate between a magmatic and a metamorphic origin for zircon Th/U ratios are often used. However this is not always valid, since zircon formed from melt or at ultra-high temperature metamorphism, might have high Th/U values (Rubatto, 2017), or low Th/U values if formed together with a Th-rich phase such as allanite or monazite, or from baddeleyite. Therefore, zircon textures and textural relationships to other minerals situated in its original context strengthens the interpretation of geochemical and isotopic data and remains one of the most powerful tools to link zircon growth to specific mineral reactions and therefore P-T conditions.

## Svensk sammanfattning

Jordens platttektoniska rörelser leder till tektoniska skeenden vid plattgränserna, bland annat bergskedjebildning, då berggrunden utsätts för förändringar i tryck och temperatur. Vid ändrat tryck och ändrad temperatur förändras också bergarter: de ursprungliga mineralen konsumeras i kemiska reaktioner och i stället bildas nya mineral som är stabila vid de nya fysikaliska förhållandena - en process som kallas metamorfos. Under tektonisk förtjockning av jordskorpan ökar både tryck och temperatur, vilket ger upphov till prograd metamorfos (ökande temperatur); retrograd metamorfos sker då temperaturen minskar under avsvälning och upplyftning. Datering av prograd respektive retrograd metamorfos är avgörande för vår förståelse av storskaliga geologiska processer, så som bergskedjebildning.

Mineralet zirkon ( $\text{ZrSiO}_4$ ) är ett av de mest använda för datering av magmatiska och metamorfa skeenden. När zirkon bildas innesluts uran men inte bly i kristallgittret,

men bly bildas med tiden genom radioaktivt sönderfall från uran. Därmed fungerar uran- och bly-isotoperna i zirkon som en radioaktiv klocka. Diffusionshastigheterna för uran och bly i zirkon är båda låga, vilket gör att den radioaktiva klockan inte ställs om även om bergarten utsätts för hög temperatur.

Metamorfos under bergskedjebildning är en dynamisk process som äger rum över miljontals år. För en kunna tolka en datering med mineralet zirkon är det därför nödvändigt att förstå genom vilken process zirkonkristallerna bildades. Daterar zirkonåldern uppvärmning i samband med tektonisk förtjockning av jordskorpan (prograd metamorfos) eller sen avsvälning (retrograd metamorfos)? Tolkningen av vid vilken process zirkonkristallerna bildades har därmed direkt inverkan på den tektoniska tolkningen.

Metamorf zirkon kan bildas i flera olika geologiska miljöer, under olika tryck och temperatur och genom

flera olika processer. Detta innebär att för att kunna sätta en datering i sitt sammanhang måste man kunna knyta den zirkonbildande processen till de metamorfa mineralreaktionerna i bergarten. Ett vanligt antagande har varit att en zirkonålder daterar tidpunkten då den högsta temperaturen uppnåddes. Detta förenklade antagande beror delvis på att zirkon har en mycket hög blockeringstemperatur, c. 900°C, under vilken den radiometriska klockan inte nollställs. Å andra sidan visar massbalans-modeller att i basiska bergarter bör zirkon bör lösas upp under prograd metamorfos, för att i stället bildas under avkylning och upplyftning.

Om vattenrika fluider saknas under en metamorfos kan basiska intrusivbergarter, som gabbro och diabas, förbli i stort sett opåverkade. I synnerhet grovkorniga gabbrobergarter är vanligen motståndskraftiga mot deformation och associerad fluidinfiltration. Gabbrobergarter kan därför uppvisa gradvisa övergångar från välbevarad gabbro till en fullständigt metamorft omkristalliserad metagabbro. Sådana metamorfa övergångszoner ger värdefull information om hur metamorf zirkon bildades, och vid vilket stadium av den metamorfa utvecklingen.

I denna avhandling har två metamorfa

övergångszoner undersökts i detalj: en övergångszon från gabbro till eklogit i Vinddøldalen, södra Norge, samt en övergångszon från gabbro till granatamfibolit i Herrestad, Småland. Syfte med studierna har varit att systematiskt dokumentera reaktionstexturer och mekanismer för bildningen av metamorf zirkon, och att erhålla en direkt åldersbestämning metamorfosen. En tredje studie omfattar en undersökning och sammanställning av metamorf zirkonbildning i ett flertal olika gabbrobergarter, vilka har genomgått metamorfos vid olika tryck och temperatur. Sammantaget visar erhållna data att bildningen av metamorf zirkon är påfallande lika i samtliga fall, oberoende av tryck och temperatur, samt att den huvudsakliga zirkonbildningen skedde genom nedbrytning av det magmatiska mineralet baddeleyit ( $ZrO_2$ ). Detta betyder att metamorfa reaktioner och zirkonbildning i gabbrobergarterna i större utsträckning är beroende av fluidinfiltration (vanligen i sin tur beroende av deformation) än specifika tryck- och temperatur-förhållanden. Följaktligen bildas metamorf zirkon i gabbrobergarter generellt under det första tillfället av metamorf rekristallisation, vanligen under prograd metamorfos.

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