Internal structures in detrital zircons from Hamrånge: a study of cathodoluminescence and back-scattered electron images

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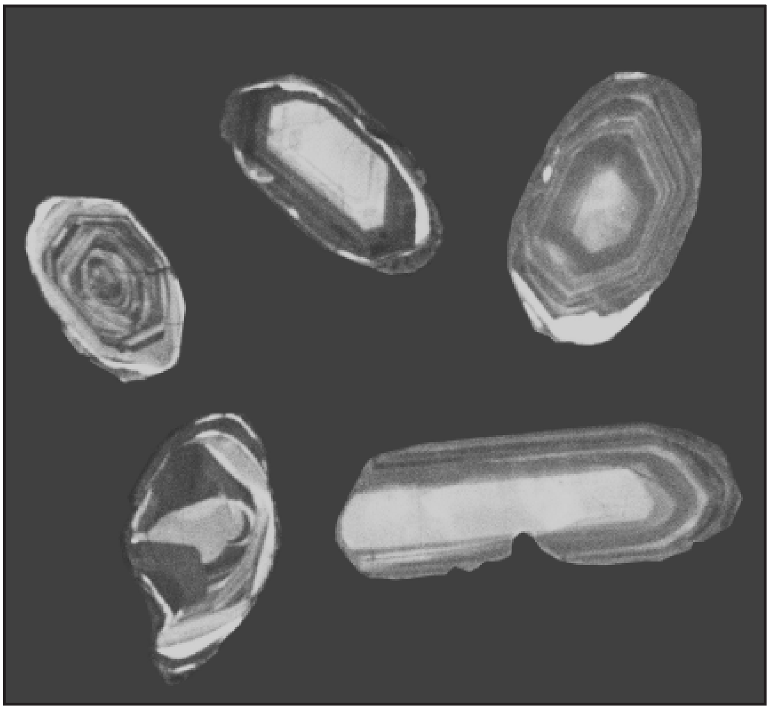

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Bachelor Thesis
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Cover Picture: Zircons from the Hamrånge area imaged with Cathodoluminescence.

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Abstract: The purpose of this study is to extract, study and investigate the internal structures of detrital zircons from schist samples in the Hamrånge area, north of Gävle, Sweden. Zircons were extracted from four different samples collected from three different localities. Zircon imaging was performed with optical microscope and scanning electron microscope, with back-scattered electrons and cathodoluminescence. Comparison between the samples were made and showed that although the structures of the zircons of the different samples varied there were also many similarities. The biggest difference between the groups was the degree of metamictisation of the grains and also the degree of abration of the grains. This also coincides with that the most abraded grains were hosted in the most coarse-grained unit. The inner structures did not vary to such extent that any conclusions about the provenance could be drawn without further analyses. Further provenance studies of the zircons will be performed by dating the crystals by spot analysis, i.e. by U-Pb-SIMS.

Keywords: zircons, internal structures, BSE, CL, Hamrånge

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Inre strukturer i detritala zirkoner från Hamrånge: En studie med katodluminescens och BSE

KAROLINA BJÄRNBORG

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Sammanfattning: Syftet med denna studie var att extrahera och undersöka de inre strukturerna hos zirkoner från skifferprover insamlade i Hamrångeområdet, norr om Gävle,. Fyra prover från tre olika lokaler valdes ut för undersökningen. Studierna genomfördes med optiskt mikroskop samt svepelektronmikroskop, där back-scatter och katodluminescens användes. Jämförelse mellan proverna visade att även om de inre strukturerna i zirkonerna varierade mellan proverna så fanns där även likheter. Den största skillnaden mellan proverna var graden av omvandlingar och metamiktisering samt graden av abrasion hos kornen. Detta stämde väl överens med att de mest rundade kornen fanns i det prov som ursprungligen hade den grövsta kornstorleken, det vill säga var mest sandig, och vice versa. Då de inre strukturerna inte varierade mycket kunde inga slutsatser angående provenans fastställas och fortsatta provenansstudier på zirkonerna kommer att utföras genom datering med U-Pb SIMS metoden.

Nyckelord: zirkoner, inre strukturer, zonering, metamiktisering, Hamrånge

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1 Introduction

Zircon is usually a small but interesting mineral that can be found in most rocks; sedimentary and igneous as well as metamorphic rocks. It is an important mineral in geochronology and it is widely used for dating geological events with the U-Pb method. Thanks to its durability it survives many of the influences that different geological events put it through and the different episodes can also give an imprint on the grain. By studying the internal structures in zircons it is possible to investigate the history of the crystal and thereby the history of the rock in which it is situated.

Studies on zircons have been made for a long time and methods used get better and better. Today it is possible to see even the smallest structures by using an electron microscope, but in spite of advanced technology there is still principles behind the formation of zircon growth structures that are not fully understood. The small grain has still many properties to explore.

1.1 Purpose and question of issue

In the Hamrånge area, north of Gävle in Sweden, metasupracrustal Svecofennian rocks are the dominating rocks, according to the geological maps (Fig. 1). Studies have been made on these rocks and the litostratigraphy is known. At the litostratigraphic bottom there is a sequence of schist, followed by acidic and basic volcanites overlain by quartzite. The acidic volcanites have been dated to 1888 ± 6 Ma and the minimum age for the quartzite is 1855 ± 10 Ma given by the age of the youngest zircon (Bergman et al. 2008). In the area folding and shearing has affected the rocks and therefore the chronostratigraphy is uncertain.

This study is a small part of a large project, which purpose is to investigate the relationship between the units of the litostratigraphy, which will be done by determining the age distribution of detrital zircons in the schist with U-Pb-SIMS (secondary ionisation mass spectrometry) method and studying the provenance of the zircons.

The purpose of this particular study is to investigate the extracted zircons from the immature metasediments and to investigate, describe and evaluate the internal structures of those zircons. The internal structures have been studied with back-scatter (BSE) and cathodoluminescence (CL) with a scanning electron microscope (SEM). Zircons from four samples collected at three different localities in the Hamrånge area have been investigated (Fig. 1).

The aim of this investigation and the following questions of issue were framed:

- What different internal structures appear in zircon?
- How has these structures formed and what kind of event or environment do they reflect?
- What internal structures can be found in the zircons collected for this study and what conclusions can be drawn from this?

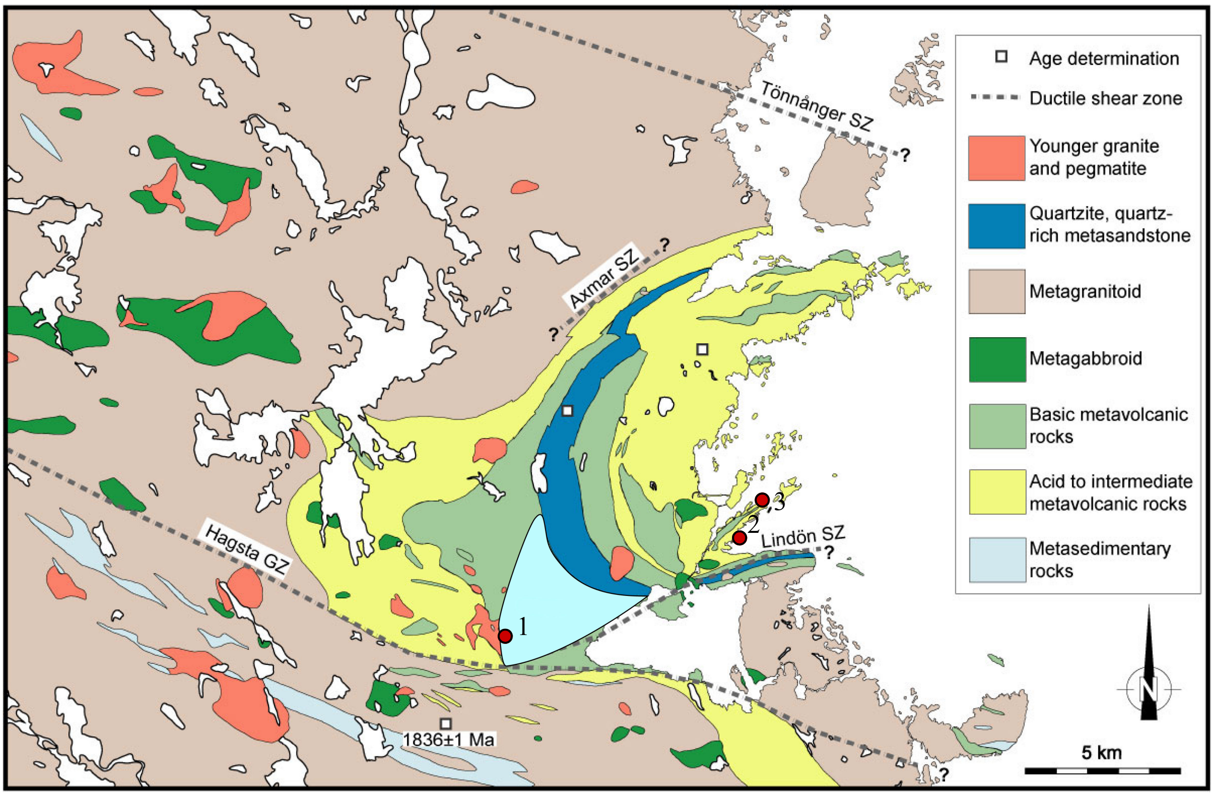

Fig. 1. Geologic map of the Hamrånge area with the localities marked. Locality 1 is E4, locality 2 is Lindön and locality 3 is Hundhällen. The map is modified from Bergman et al. (2008).

1.2 Localities

The localities where the samples were collected are shown in figure 1. Hamrånge is located by the coast to the Baltic Sea. All samples are collected from schists, interpreted to be the oldest unit of the supracrustal sequence. The locality Hundhällen is a schist with well developed foliations (S_0-S_2) that lies on the coast-line. The sample Lindön is collected from the Lindön shear zone and is situated on the peninsula of Lindön. E4 is a locality close to the highway near Hagsta. Two samples were taken from this locality – E4 1 characterised by a high sillimanite content, whereas E4 2 is a quartz-rich biotite-muscovite schist.

2. Background

Zircons have been studied for a long time and many scientists have contributed to the degree of understanding that we have today. The following section 3.1, comprise a summary of zircon mineralogy, morphology and internal structures common in zircons. Section 3.2 comprises a summary of the microscopy methods often used in zircon studies.

2.1 Zircon mineralogy and morphology

Zircon is an orthosilicate with the chemical formula $ZrSiO_4$. It is a common accessory mineral in most igneous rocks, but is also found in many sedimentary and metamorphic rocks. It has a hardness of about 7-8, a perfect tetragonal symmetry and has a high durability (Bulakh and Wenk 2004).

Zircon has a prismatic crystal habit with bipyramidal ends (Bishop et al. 1974). The ratio between length and width varies between different crystals and is believed to be an effect of the crystallisation rate, where higher length-to-width ratios reflect rapid crystallisation and lower ratios mean slow crystallisation. Also, the properties of the prismatic and bipyramidal shapes vary between different crystals in magmatic rocks and are thought to reflect the chemical composition and temperature of the magma (Corfu et al. 2003). Figure 2 A show a simple sketch of an ideal zircon crystal.

Natural zircon predominantly contains a number of elements other than zirconium (Zr), silica (Si) and oxygen (O). U, Th and Y often substitute Zr. Other common elements in zircon are Ce, Hf, Nb, Ta and Al (Bulakh and Wenk 2004). Hafnium is always present in zircon, in concentrations of around 1 percent, but the ratio between Zr and Hf can vary considerably between different rocks. Sometimes also phosphorous can replace Si. Iron is also often present, and occasionally Sn and Na, probably mostly as inclusions of other minerals (Deer et al. 1982).

The colour of zircon can vary from transparent to brown, from yellow to red, green or blue. The oxidation state of Fe is one possible cause of variations in colour, but it might also be influenced by the content of U and Th and other trace elements (Deer et al. 1982). The colour is sometimes correlated with the age and concentration of U (Heaman and Nudden 1991).

2.2 Internal structures

The zoning that can be seen in many zircon crystals is growth zoning and the chemical constituents of the zones reflect the growth environment (Hanchar and Miller 1993). Studies made on zircons show a connection between different zones and changes in concentrations of e.g. Hf, U, Y and rare earth elements (Hanchar and Miller 1993, Hanchar and Rudnik 1995, Hitchen et al. 1998).

The differences in concentration of the elements between the zones ranges from a zoning that has almost no change in composition at all, to a zoning that vary between two end-members – one with almost no trace elements and one with high concentration. Zoning with only a little change in composition tends to be very weak, while zoning with large differences in composition tend to be obvious (Corfu et al. 2003). Different types of zoning have different chemical properties and are subjected to different interpretations of formation.

2.2.1 Oscillatory zoning

Oscillatory zoning is a common structure in zircon (Fig. 2 B). It is an euhedral, concentric zoning that evolves during magmatic crystallisation as concentrations of trace elements incorporated in the zircon change during formation of new zones outside the already formed zircon. The formation of oscillatory zoning is not in equilibrium with the magma but reflects

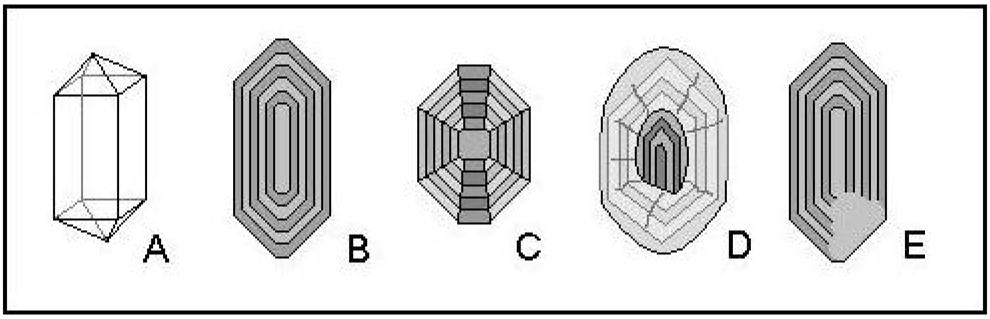

Fig. 2. Principle sketch of zircon morphology and structures. A) The crystal habit of zircon. B) Oscillatory concentric zoning. C) Sector zoning. D) Xenocrystic core with fractures going radially from the core. E) Recrystallised area in zircon.

local chemical gradients in the magma near the zircon (Benisek and Finger 1994).

The zoning might be disrupted by a new generation of zoning, where the outer part is resorbed and new zircon is crystallised with a slightly different zoning pattern. This might be due to periods of Zr undersaturation in the magma and it probably reflects a slowly crystallizing melt. (e.g. Corfu et al. 2003)

2.2.2 Sector zoning

Another type of zoning, although not as common as oscillatory zoning, is sector zoning. It is only visible in CL and BSE and shows sectors proceeding radially from the centre (Fig. 2 C). Concentric zoning is often also present, but the sectors with varying brightness are more prominent. Concentrations of Zr, Hf and Y correlate with the sectors. Darker sectors are enriched in Y and lighter are enriched in Hf (Hoffman and Long 1984). The formation of sector zoning has been debated, but is probably an effect of varying growth rates of different crystal surfaces during crystallisation (e.g. Corfu et al. 2003).

2.2.3 Xenocrystic cores

In zircons it is common to find xenocrystic cores, particularly in S-granites. When a previously crystallised zircon is surrounded by later crystallised zircon, possibly generated during late-stage crystallisation, the older zircon becomes a xenocrystic core in the newly formed zircon. The ability to see the whether a zircon has a xenocrystic core depends on the compositional differences between the core and the younger rim (Fig 2 D). An easier way to determine xenocrystic core in zircon is to study the truncation of zoning in the crystal. There can be more than one core generation in a zircon and these can be recognised by discontinuity in the zoning pattern (e.g. Corfu et al. 2003).

2.2.4 Recrystallisation

Recrystallisation of zircon grains creates discontinuities, with changes in chemical composition, in the zoning pattern. This is thought to occur after completion of magmatic crystallisation, and is visual as homogenous patches in the otherwise often oscillatory zoned crystal (Fig. 2 E). The recrystallised patches are often homogeneous and low in U, Pb and Th content, perhaps as an effect of chemical exchange with the surrounding (Pidgeon 1992).

Hitchen et al. (1998) suggest two different types of redistribution of zircon composition. The first type is that trace elements are focused to enriched bands by diffusion in a closed system. The second, where hydrous fluids are involved in the system, generates recrystallisation of areas of the zircon and loss of trace elements. The external morphology of the grain often remains more or less intact, but recrystallisation can sometimes affect the shape of the grain (Pidgeon 1992).

2.2.5 Metamictisation and Fracturing

Metamict zircon is an amorphous and less durable variety of zircon with lower density than the normal zircon. As mentioned before, U and Th are common trace elements in zircon. The spontaneous decay of these elements inside a zircon crystal leads to structural instability and breakdown of the crystal lattice due to the emission of α-particles, which move through the crystal and displaces lattice atoms (e.g. Deer et al. 1982). Metamictisation affect domains with a high-U content more than low-U domains and the transition into the metamict state often occurs when 1015-1016 α-decay events/mg is accumulated in the crystal lattice (e.g. Heaman and Nudden 1991).

The degree of metamictisation also depends on the time elapsed since the temperature, when the zircons crystallised, reached below the closing temperature (Mezger and Krogstad 1997).

As metamictisation leads to an increase in volume metamict parts of an e.g. zoned zircon exert mechanical stress on its surroundings and thereby cause fractures in the unaffected parts. This is a common feature in zircons that have a xenocrystic core, which eventually became metamict (Fig. 2 D). Fractures often set off radially from the intersection between the affected and the unaffected part.

Fractures can also be concentric and follow the borders between zones. They can also be caused by external parameters, for example external pressure, rapid pressure release or external stresses during mylonitisation or diagenesis (e.g. Corfu et al. 2003).

The radiation of U and Th decay in zircon can sometimes also affect the mineral in which it has been enclosed; normally this is visible as haloes of radiation damages in the host grain around the zircon (e.g. Deer et al.1982).

2.2.6 Alteration

Metamict and fractured grains are often affected by alterations, as fluids can easily pass through the grain. Alterations are visible as darker areas with bulbous or feathery structures. The zircon becomes hydrated and elements such as Fe and Ca are incorporated while Pb often leaches out, creating discordant U/Pb ratios (e.g. Corfu et al. 2003). This can lead to misinterpreted age determinations if altered zircons are used in such studies. Altered parts are more easily abraded than pristine parts, which often is seen when the partially abraded outer parts of a grain is altered (e.g. Heaman and Nudden 1991).

2.2.7 Other structures

Inclusions are common in zircon. Normally the minerals of the host rock such as quartz, feldspar and biotite are present as inclusions, but also inclusions of melt and fluids are possible to find.

Other common structures are over- and intergrowths of other minerals. One mineral that is fre-

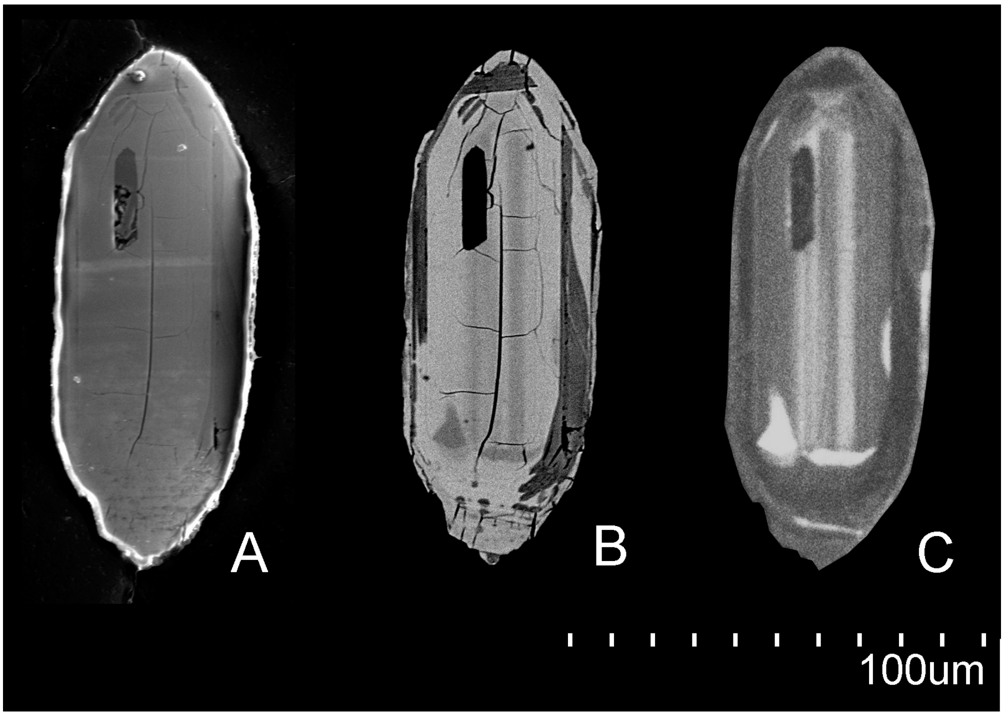

Fig. 3. Three different images on the same zircon, from E4 1 sample, imaged with A) Secondary electrons, B) Back-scattered electrons and C) Cathodoluminescence.

quently found as over-/intergrowths is xenotime, YPO_4, an isostructural mineral to zircon (e.g. Corfu et al. 2003). Xenotime might also be present as small inclusions in the zircon (Pidgeon 1992).

The structures described so far are characteristic and might be easy to distinguish in many crystals, but under other circumstances those structures can change into another. During medium to high temperature metamorphism the structures become more chaotic, old structures become distorted and new zircon zones might grow. The new zircon is often referred to as metamorphic zircon. Concentric zoning might be present, although more irregular than magmatic zoning, and recrystallisation of the grain might occur, both in the inner part and the outer part of the zircon (e.g. Corfu et al. 2003).

At high pressure metamorphism the zircons undergo recrystallisation and new growth of zircon, leading to complex structures. A number of structures might be present, including structures with new zircon growth oscillatory zoning resembling that of magmatic crystallisation and irregular patchy structures. Grains with two distinct phases are common; including relics of the old zircon grain surrounded and intruded with newly crystallised, often homogeneous zircon (e.g. Corfu et al. 2003).

Another feature related to metamorphic zircon but at low PT-condition is hydrothermal zircon, the difference lies in the temperatures of the environment during formation. Hydrothermal zircon precipitate from hydrothermal fluid or fluid-saturated residual melt at temperatures around 600°C in late magmatic systems and around 300°C in mesothermal ore-forming systems (Schaltegger 2007).

2.3 Microscopy

As the size of zircons often are around 10-100 µm studies are best performed by using a microscope. In the following text the most usual types and methods used during microscopy work are shortly described.

2.3.1 Optical Microscope

Optical microscope that has two separate eye pieces provides a 3D image. As visible light passes through the sample optical phenomenon due to anisotropic structures of the sample can be observed. There are both binocular and petrography microscopes. Binocular microscopes have lower magnifications than petrography microscopes. Only reflected light is used in binocular microscope and the best resolution is obtained when keeping the sample in ethanol. In petrography microscopes, both transmitted light and reflected light can be used. They show slightly different structures. Under reflected light colour, opacity, inclusions, fractures, alterations and external structure can be studied. Transmitted light is best used when the sample is in thin section or as a grain-mount. Zoning, inclusions and inner structures are visible, as well as degree of transparency, opacity and fractures. (Corfu et al. 2003)

2.3.2 Scanning Electron Microscope

The resolution of a microscope is proportional to the wavelength of the light used. This means that an optical microscope has an upper limit of magnifications at around 1000 times. Scanning electron microscope, SEM, use a beam of electrons that scans the sample, instead of visible light. The wavelength is thereby reduced and it is possible to get highly resolute magnifications up to 200 000 times.

As the electrons reach the sample, secondary electrons (SE) are emitted and can be detected and thereby provide a picture of the surface of the sample. In SEM only the surface of the sample can be studied, and thereby no structures at depth are visible (Bra Böckers Lexikon 1996). Figure 3 A shows a zircon imaged in SE.

When scanning the sample with electrons some electrons get back-scattered. The amount of electrons that get back-scattered depends on the average atomic number of the phase that is scanned, where areas with higher atomic numbers reflects more electrons than areas with lower atomic numbers. The intensity of back-scattering also depends on the topography of the sample as well as the orientation of the crystal.

A detector calculates the amount of back-scattered electrons and provides a picture where higher intensity of electrons gives lighter areas and vice versa, (Bulakh and Wenk 2004). Back-scattered electrons (BSE or BE) is a useful method for studying chemical variations in a mineral as well as over- and intergrowths. Figure 3 B shows a zircon imaged with BSE. The element that affects the amount of electrons backscattered most in zircon is Hf while U has a secondary effect (Hanchar and Miller 1993).

Cathodoluminescence is another method often used in SEM. When an electron beam in a cathodoluminescence mode reach the sample electrons get excited and as the electrons go back to normal state they emit photons. Different elements emit different wavelengths of light, depending on the energy level of the excited electron, and thereby it is possible to detect elements based on the light emitted (Marshall 1988). Mostly the luminescence is emitted not from the pure phases but from impurities, such as trace elements, in the crystal lattice (Marshall 1988).

There is no direct connection between emitters and phases and the principles behind CL-emitters in minerals are not fully understood. Although it is known that concentrations of trace elements affect the luminescence emitted and in zircon Dy^{3+} is considered to be the main factor while Tb^{3+} is a secondary factor (Hanchar and Rudnik 1995). Also Sm^{3+}, Eu^{2+}, Tb^{3+} and Y^{3+} might be important in this context (e.g. Hanchar and Miller 1993), but there is no linear relationship between cathodoluminescence intensity and trace element concentrations in zircons (Hanchar and Rudnik 1995). Figure 3 C show a zircon imaged with CL.

3 Method

3.1 Extraction- and study methods

The samples from each locality weighed approximately 0.5 kg. The weathered surface of the samples was removed by using a saw and the samples were then thoroughly washed to reduce the risk of contamination. All equipment used in the following extraction was washed before and after use. From each rock sample thin sections were prepared for petrology studies.

To extract the zircons the samples were first crushed into gravel-size and then grinded into dust. For extracting the heavier minerals a Wilfley separation table was used. This was done in two steps. In the first step, at a table dip of 8.5° and a water pressure of 0.25 bar, the heavier minerals were separated and collected. At the second step, at a table dip of 5.2° and a water pressure of 0.15 bar, the heaviest minerals, including zircon, were separated and collected (Johansson and Söderlund2002).

The samples were put in petri-dishes, magnetic minerals were extracted using a magnet and the water was evaporated. The zircons were handpicked in ethanol under a binocular microscope. A Franz magnetic barrier separator was used to extract garnets from the Hundhällen sample. The zircons from each sample were divided into two groups – one with zircons of high transparency and one with lower degree of transparency. Zircons of different properties were collected to ensure that as many types of different varieties as possible were represented. Thus metamict, opaque grains were as important and thereby as numerous as transparent, pale coloured grains. The zircons were mounted on a two sided tape, in groups according to locality and character. The mount was casted in epoxy and polished at the Nordsim laboratory, Swedish Museum of Natural History in Stockholm.

The sample was coated with carbon and the zircons were then studied under optical microscope using both reflected and transmitted light, and in scanning electron microscope, using BSE and CL. The instrument used was a Hitachi S 3400 N, located in the Department of Geology, Lund University. EDS analysis was used for the determination of the composition of some unidentified minerals.

All figures used in the text, except for figure 1, are made and arranged by the author.

3.2 The rock samples

Thin sections were studied in transmitted-light with an optical microscope. Observations about petrological and structural characteristics were made before the sample preparations. In the following section a short summary of each sample's attributes are presented.

3.2.1 Sample E4 1

The main characteristics of this rock is, as mentioned before, is the content of elongated sillimanite crystals. It's a medium grained schist with foliation and cleavages. The main minerals are quartz, biotite and sillimanite as fibrolite. No zircons were found in the thin section, but in the prepared this mineral was abundant. About 100 transparent and 100 non-transparent zircons were selected from this sample.

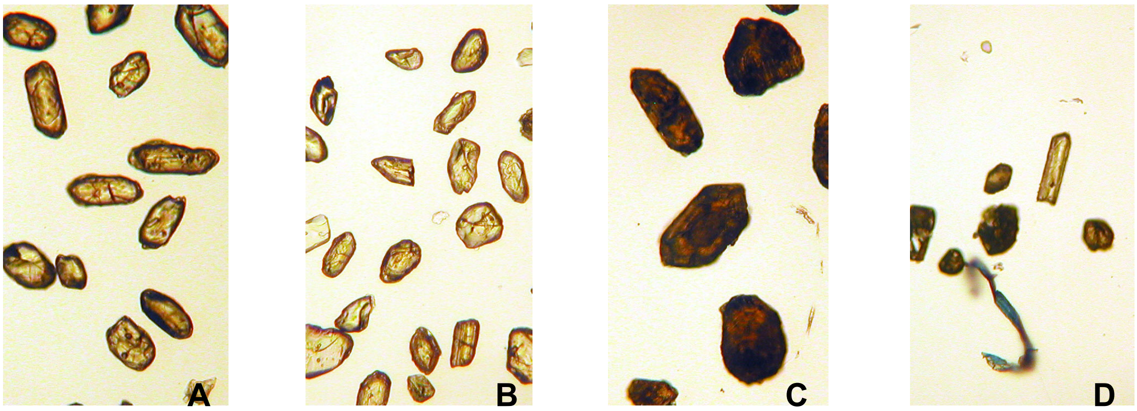

Fig. 4. Zircons from the samples studied in transmitted-light microscope. A) E4 1, B) E4 2, C) Lindön, D) Hundhällen. The zircons are around 60-200µm.

3.2.2 Sample E4 2

The sample is from a coarser grained unit interlayered with the sillimanite schist. It is fine- to medium-grained. Thin section shows that the main minerals are biotite and quartz. Other minerals present are chlorite, apatite and opaque minerals, which were indentified in the heavy mineral separate as pyrite and magnetite. The opaque minerals appear often adjacent to the biotite. Quartz is of equal size and subhedral. Zircons were abundant in this sample, but no zircons were found in the thin-section study. About 100 transparent and 100 non-transparent zircons were selected.

3.2.3 Sample Lindön

Also this rock is a fine grained schist, which is rich in mica and magnetic minerals. Petrography shows a pronounced foliation and the mineral composition is dominated by quartz, biotite and muscovite. Also apatite is abundant. During sample extraction pyrite and magnetite was observed.

In this sample zircon is rather rare, 50 transparent and 75 non-transparent zircons were selected.

3.2.4 Sample Hundhällen

The rock is foliated fine grained schist with a weak cleavage. The main characteristic of the rock is the high content of garnet. Thin section shows that the main minerals are quartz and biotite. The biotite grains are subhedral and have a random orientation. The quartz grains are also subhedral and of unequal size. Chlorite, apatite, garnet and opaque grains, probably pyrite and magnetite according to observations in the heavy mineral separate, were found. The thin section and the prepared sample contained very few zircons; only six possible zircons were recovered.

4. Results

4.1 External morphology of the zircons

The external morphology of the zircons was studied with optical petrography microscope in transmitted and reflected light. This is shortly described as a background to the main goal of this study.

4.1.1 Sample E4 1

The sample E4 1 has plenty of zircons. The zircons are around 50-150µm. The transparency of the zircons is often high and many grains are pale and clear. Many grains are, however, darker and some grains in the group of lower transparency are close to opaque. The colour of the grains is light pink to light brown. Fractures and inclusions are frequent among grains both with high and low transparency.

There are many rounded grains, but prismatic and broken grains are also present. The smaller prismatic, elongated grains are often less rounded than the shorter ones. They are also often more transparent than non-prismatic grains, although there are exceptions.

In transmitted light zoning is visible in many cases, but occasionally much of the zoning is concealed by the opacity (Fig. 4 A).

4.1.2 Sample E4 2

Of all samples the largest amount of zircons was recovered from sample E4 2. The zircons are also the most transparent and pale (Fig. 4 B) of all zircons studied. The colour of the grains is light pink to light brown and the average size of the grains is 50-100µm. The zircons populations are, as mentioned before, divided into two groups; transparent and less transparent, and the difference in transparency is great between the two groups in this sample. However, there are also different grades of transparency between grains within the same group. The less transparent grains have, to a great extent, more inclusions and fractures than the more transparent ones, although fractures and inclusions occur in both groups.

Most of the grains are rounded, though some grains still have a prismatic shape. Zircons of different shapes are present, both elongated and short ones. Some zoning is visible in transmitted light microscope.

4.1.3 Sample Lindön

Zircons are rarer in the Lindön sample, compared to the two samples mentioned above. The grains are less transparent, no pale grains are found, and the colour ranges from light pink to light brown. Surfaces of the grains are irregular and ragged and have dark coloured small grains attached to them. Inclusions and fractures are common and many grains in the group of lower transparency are nearly opaque (Fig. 4 C).

The grains are not rounded to any significant degree and many prismatic grains are found. Many of the grains are crystal fragments. The grains are of varying range of size, from 50µm-150µm, and are mostly either small or big. Prismatic grains are of all sizes. Zoning is visible in many grains, although the low transparency conceals these structures.

4.1.4 Sample Hundhällen

From six possible zircon grains only two turned out to be zircon. One is transparent, prismatic, with light pink colour. The size of this grain is about 100µm whereas the other grain is around 50µm. The other one is almost opaque, light brown and not prismatic. Inclusions and fractures are visible in the transparent zircon. Zoning is not clear in the grains of this sample. Figure 4 D show Hundhällen zircons, in transmitted light microscope.

4.2 Internal structures of the zircons

The SEM-study shows that most of the zircon structures are visible with both BSE and CL. The same structures are often revealed by the two modes but in inverted greyscale. However there are exceptions. One example of this is metamict structures, which are clearly visible and detailed in BSE but only grey and dull in CL. Topography such as fractures are also only visible in BSE. There are other structures that might be revealed by one mode only, one example is zircon A in figure 7 where the recrystallised part is visible in CL but only slightly indicated in BSE. This emphasises the need for using both BSE and CL when imaging internal structures prior to further studies, such as dating zircon with spot analysis.

From the BSE and CL images, eight to nine representative zircons per sample were chosen for a more detailed study, with exception for Hundhällen which only contained two zircons. The selected zircons are imaged both in CL and BSE.

4.2.1 Sample E4 1

The zircons from E4 1 are shown in figure 6 A-H. They yield a range of different internal structures which cannot be seen in optical microscope. Many of the zircons with low transparency are metamict to some degree and most of the transparent zircons show distinct structures (Fig. 6).

Most of the zircons have a concentric, oscillatory zoning. This is, however, sometimes concealed due to metamictisation and that many grains are only fragments of crystals. In this sample no zircons with sector zoning are found. Zircon A has a concentric zoning that is visible in both CL and BSE. The zoning is interrupted in places and a slight change in growth direction can be discerned, possibly due to recrystallisation during the formation of the crystal. In the lower left of the crystal an embayment appears as a homogenous area penetrating the oscillatory zoning. Zircon B has also concentric zoning, which is more visible in CL than in BSE. The concentric zoning of zircon H has not been interrupted and is visible in both BSE and CL. The zoning of zircon G is very faint in BSE but in CL interrupted growth is visible. Zircon C has a strange zoning pattern which irregularly encircles around a core in the central part. The core appears to be a fragmented crystal. Also zircon D shows a strange zoning pattern which can neither be referred to as concentric or sector zoning in combination with embayments.

Irregularity in the zoning pattern might be due to the presence of xenocrystic core. Zircon A has a core, which most probably is not xenocrystic. It is rounded and has a zoning that is different from the zoning outside and is most likely a synmagmatic feature. A similar feature is visible in zircon G. The core in zircon B is on the other hand is xenocrystic. This is visible in BSE but more distinct in CL. The core appears to be a fragment and has a partially angular shape. A xenocrystic core is possible also in zircon F, indicated in the BSE image.

As mentioned before, some zircons with concentric zoning has interrupted zoning pattern (Fig. 6 A and D), which are possible recrystallised areas. In zircon A, a large homogenous area cut the zoning, which perhaps is of an younger age. Also zircon F has homogenous areas surrounding BSE dark metamict domains.

Grains of low transparency are often partially metamict. This structure yields dark areas in both BSE

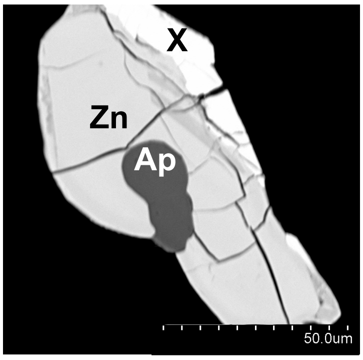

Fig 5: Zircon with both apatite and xenotime areas. The dark area is apatite whereas the bright is xenotime.

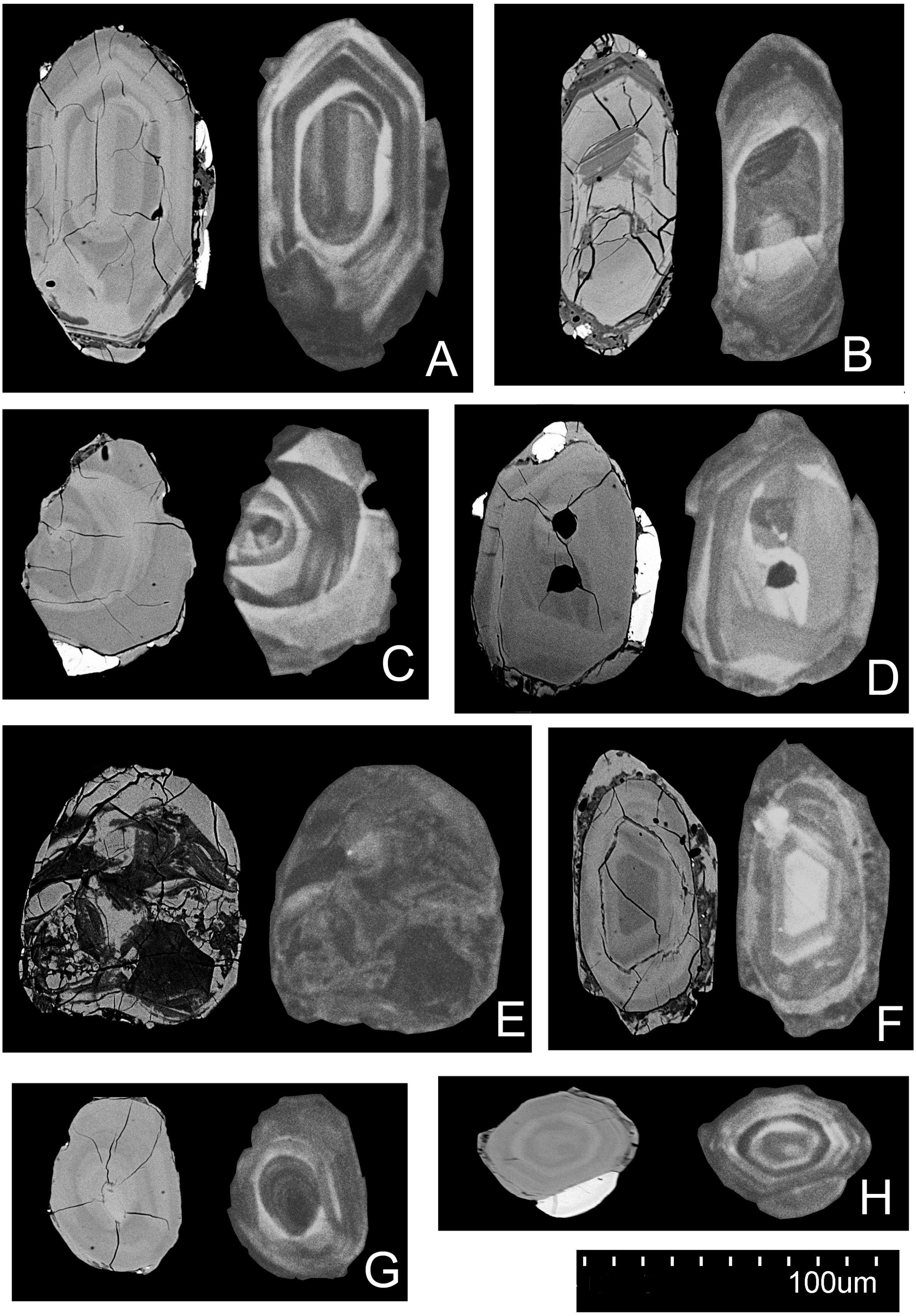

Fig. 6. Zircons from E4 1. Picture A-H are all in both BSE and CL, where BSE is to the left and CL to the right, for comparison of structures visible.

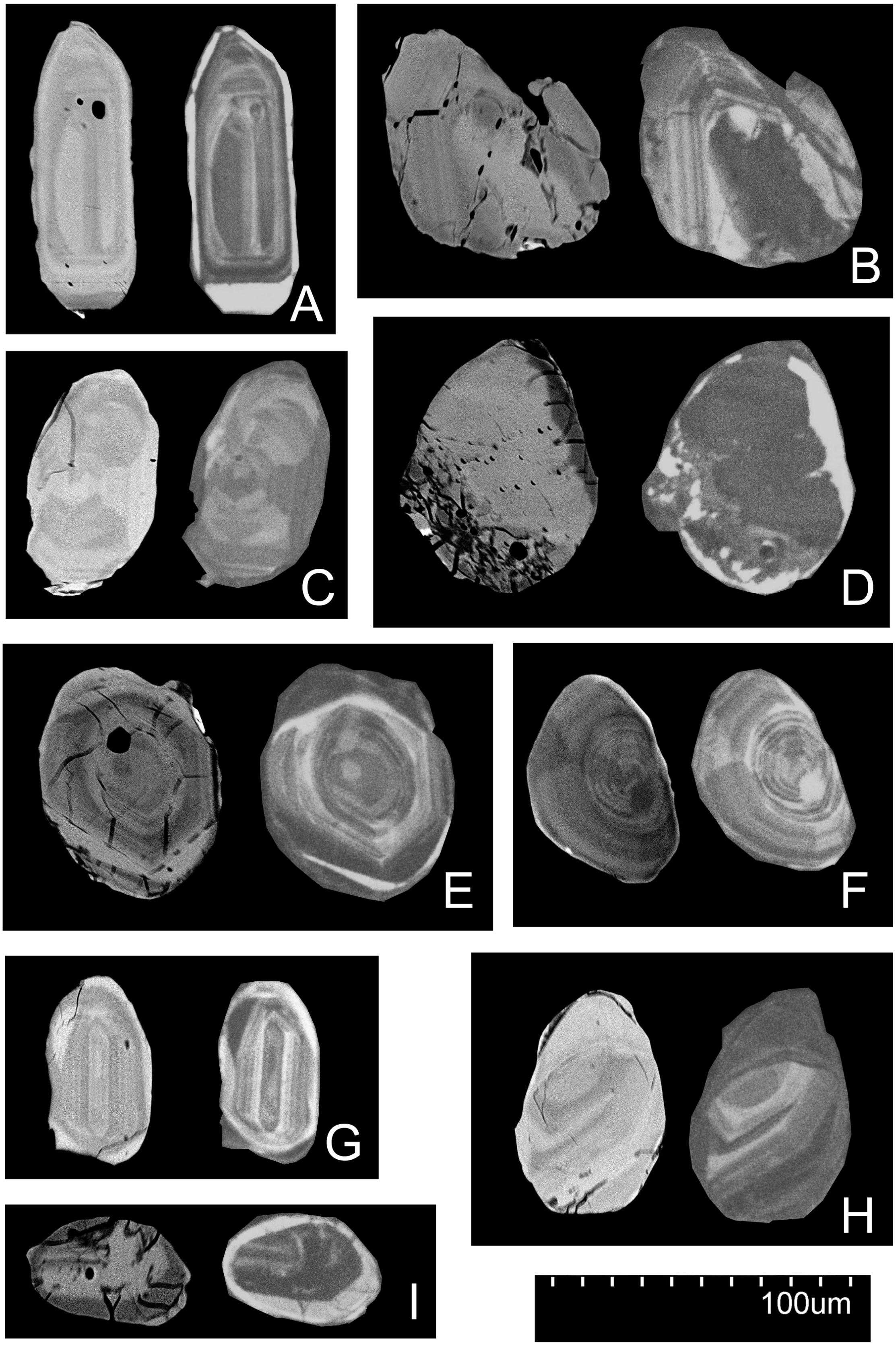

Fig. 7. Zircons from E4 2. Picture A-I are all in both BSE and CL, where BSE is to the left and CL to the right, for comparison of structures visible.

and CL, often along the zoning pattern, as the outer parts of zircon A and B. In the more severely metamict grains the structures are irregular and cover the entire grain, as in zircon E. Zircon F might as well have slightly metamict inner parts, where zoning is weak and the CL image of the grain is dark and blurry in contrast to the central part of the grain.

Some grains have a metamict rim regardless of the crystallinity of the central parts of the grain. The rims are often irregular and partly rounded. Some grains, like zircon F, have sharp boundaries between the central part and the metamict rim, perhaps indicating that the rim might be recrystallised or newly formed zircon, which has later become metamict. The boundary mentioned above is not always present, as in zircon B.

Many grains are fractured, often around possible xenocrystic cores (zircon B). Zircon B and G show such fractures, emerging from the rim of the core radiating out through the crystal. Zircon F has fractures that run along the zoning, possibly indicating an inner part that has different trace element composition than the outer part. Less metamict parts in metamict zircons often have irregular fractures, as zircon E, which penetrates the whole grain.

The grains are often slightly rounded, but they have to a large extent irregular shape. The uneven shape is often due to over-growth of xenotime, which appear bright in BSE but dark in CL. Small xenotime areas have formed on most zircon, either on the outer parts of the grain (zircon H) or together with recrystallised or newly formed zircon (zircon D). Other grains have areas that look like missing parts of the grain, which actually consists of apatite, which is dark in BSE but light in CL. Apatite occurs also as large inclusions or morphological embayments in the zircon (Fig. 5). In the E4 1 sample there are also monazite which is a heavy mineral that resembles zircon. Monazite is light in BSE but dark in CL.

4.2.2 Sample E4 2

Also in the E4 2 sample many different zircon structures can be identified. This sample hosts the most transparent grains and the least amount of metamict zircons. Figure 7 (A-I) show different varieties of zircons found.

Oscillatory zoning is common in this sample, many grains yield a clear and thin-banded zoning, as zircons A, E and G. The zoning is often as clear in CL as in BSE. Also sector zoned grains are present. Zircon C shows a typical sector zoning, whereas zircon F is a concentrically zoned grain with indications of sector zoning.

Xenocrystic cores are not very common in this sample. Indications of xenocrystic cores although exist, as in zircon E where fractures and a slight difference in zoning pattern are factors pointing in that direction. However incorporation of various amounts of trace elements during magmatic growth cannot be ruled out.

Zircon G has a homogenous area within the zoning close to the margin, light and clear in CL but weak grey in BSE, which is an embayment that probably consists of recrystallised zircon. Another homogenous area, dark in CL but light in BSE is also present. This does not cut the zoning as the embayment does, but is probably also recrystallised zircon.

Recrystallised areas also occur as in zircon B, where a homogenous area disrupts the zoning. In this zircon these structures are only discernable in BSE. The CL image reveals a dark homogenous area situated in a likewise homogenous bright area, enclosed by the zoning on two sides. Many zircons also show homogenous areas around a zoned central part. There are two varieties. The first variety is dark in BSE and bright in CL, as in zircon A and I, whereas the second type is light in BSE but darker in CL, as zircon E, G and H. The homogenous part is not as obvious in BSE as in CL, and is most apparent in zircon A. The inner zircon is in most cases slightly rounded, except for zircon A which has a angular shape. The homogenous areas are probably recrystallised zircon but late magmatic growth cannot be ruled out.

The zircons of this sample do not show much metamictisation. Zircon D is a homogenous grain with fractures and areas that appear very light in CL, but dark in BSE, which probably are recrystallised domains. The BSE-dark area in the lower left might be metamict, but the structures might as well reflect other conditions. The metamict rims found in the E4 1 sample are not found and the metamict zones in the zircons are more rare than in the other samples. Xenotime is also present on some grains, but not to the same extent as in the E4 1 sample.

Fractures are not very common. There are a few, randomly oriented fractures, as in zircon B and C, or fractures developed due to a possible xenocrystic core as in zircon E. The few metamict grains are also often severely fractured. Most grains are well rounded and have smooth rims, for example zircon F and H which are very rounded.

4.2.3 Sample Lindön

The zircons of the Lindön sample have the most numerous grains of low transparency and are also the most metamictised. Zircons from both groups (high and low transparency) are often more or less metamict. Different structures present in the zircons of this sample are shown in figure 8. Xenotime found in both E4 1 and E4 2 is very rare in this sample and only a few very small inclusions have been recognised.

The internal structures of the zircons are weaker due to a higher degree of metamictisation and perhaps also of other reasons. The unaffected grains often show concentric zoning, weak in both BSE and CL, as in zircons A and E. In both of these zircons the width of the zones varies, from very thin to wide. The wide bands might represent recrystallised zircon, at

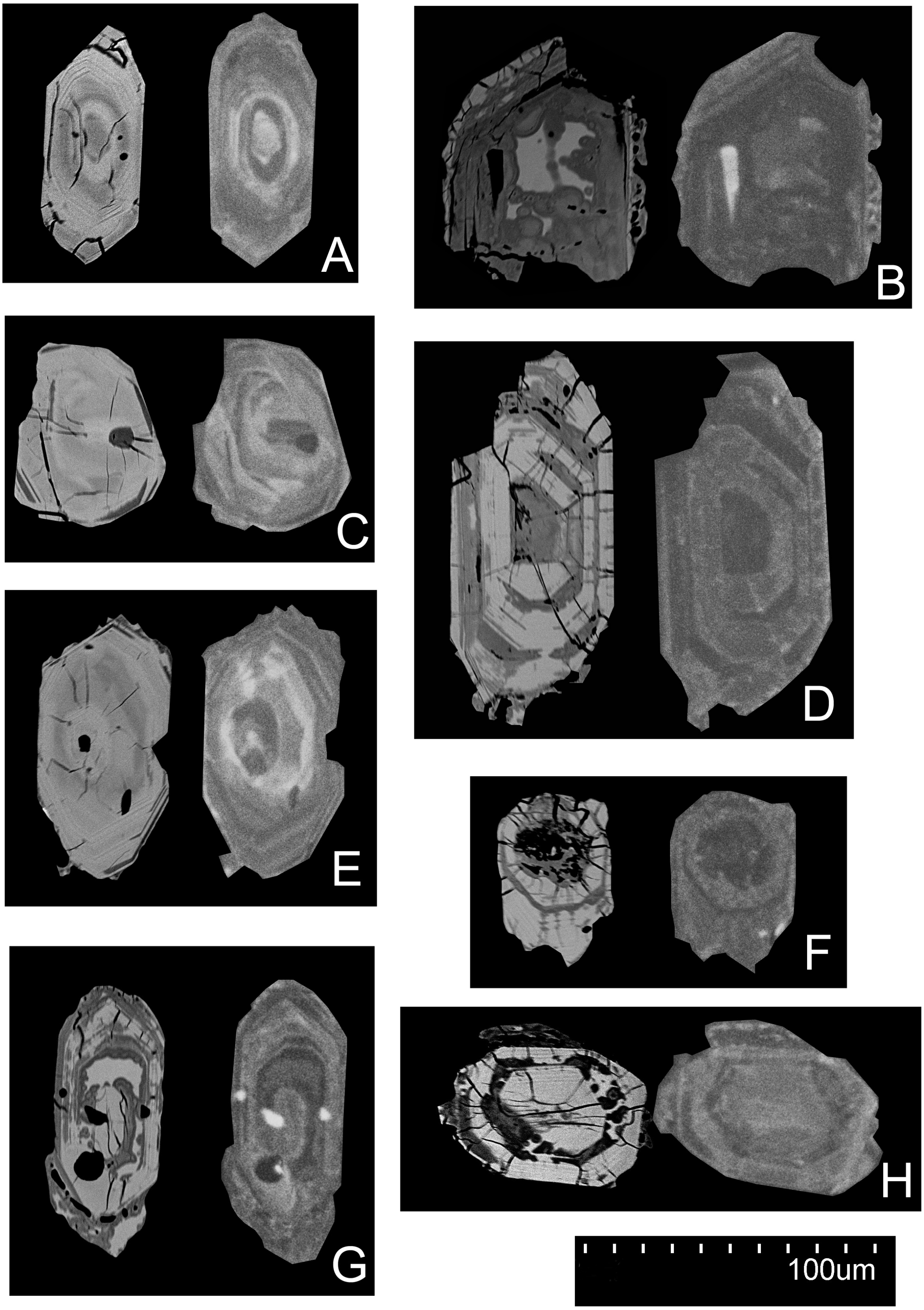

Fig. 8. Zircons from Lindön. Picture A-G are all in both BSE and CL, where BSE is to the left and CL to the right, for comparison of structures visible.

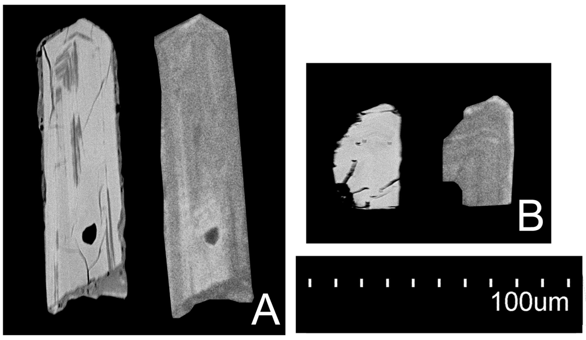

Fig. 9. Zircons from Hundhällen. Picture A and B are both in BSE and CL, where BSE is to the left and CL to the right.

least in zircon E. Zircon C is almost homogenous in BSE but in CL a clear zoning, although irregular, is visible. The zoning encircles a central part and the zones cut each other generating a chaotic pattern.

Many zircons have indications of xenocrystic cores, as zircon E where an area which is light in BSE but dark in CL differs from the surrounding zircon. There are also fractures emerging from the boarder of the rounded core.

As mentioned before, many grains from this sample are metamict. There are different degrees of metamictisation among the grains, from zircons having metamict parts (zircon G) to zircons with metamict zones (zircon D, F and H) to zircons that are more or less completely metamictised (zircon B). The metamict grains are best studied in BSE as CL images appear dark grey and reveal little information. The metamict grains are often fractured, where fractures are going radially through less affected zones, as zircon D, F and H. In zircon D a metamict zoning pattern is visible with altering metamict and unaffected zones. Zircon F has a severely metamict core, which has created fractures in the surrounding zircon, whereas zircon H has both unaffected zones and metamict zones, where the central parts are crystalline and fractured, and the BSE-dark domains are metamict. There are also BSE bright bulbous shapes connected with metamictisation, which are interpreted as recrystallised areas. Zircon G has similar structures in its inner parts as zircon B.

Like the zircons in sample E4 1, the zircons of this sample have metamict and partially abraded outer rims, visible in zircon B and G. The shapes of the zircons are often not rounded but angular and irregular. Many grains have angular fragmentation, which is due to sample processing. The main causes of the abundant fractures are either a U-Th rich xenocrystic core or metamictisation.

4.2.4 Sample Hundhällen

The two zircons of the Hundhällen sample are shown in figure 9. Zircon A shows a weak zoning. Parts of the crystal zoning are dark in BSE, probably due to metamictisation. In CL the crystal is grey and dull, similar to the metamict zircons in the other samples, which also gives indication of some degree of metamictisation. The zoning is truncated on the left side of the grain by an area of homogenous zircon enclosing the inner part, which is light in BSE but dark in CL. This might consist of recrystallised zircon. Outside this, the grain has an outer, slightly abraded metamict rim that encloses most of the crystal. The zircon has fractures revealed by BSE and an inclusion visible in both BSE and CL.

Zircon B is a small crystal fragment with a very weak zoning. The zoning is barely visible in BSE but is discernible in CL. The grain is fractured but shown no signs of metamictisation.

5. Discussion

Many of the zircons in all the samples have been affected by metamictisation. Lindön has the most numerous and severely metamict zircons whereas the least metamict ones are found in E4 2. It was obvious already during the handpicking of zircons and during studies by optical microscopy that the Lindön zircons were the most altered. These grains are the least transparent and they are often more coloured than the light coloured transparent crystals from the two E4 samples. As metamictisation depends on the content of uranium and thorium in the zircon, it would be interesting to examine zircons of the different samples to evaluate how the content of these elements varies.

It is also interesting to note that all samples contained zircons with outer metamict and perhaps altered rims, probably as the rims are more affected by the surrounding fluids. The fluids are first to penetrate the outer rims of a grain, before reaching the inner parts. Lindön and E4 1 have the most altered rims. The rims are often partly abraded, irregular and unequally distributed around the grains. Some altered rims are also found at the grains from E4 2, but they are rare and often insignificant. Zircons from E4 2 are often also rounded and had smooth exterior, whereas the zircons from the other samples are irregular, angular and with an uneven exterior. This agrees well with the mineralogical composition of the host rocks. E4 1 and Lindön are both schists with higher content of mica than the host rock of E4 2. The latter is more compact and has a higher quartz content and originate from a more mature sediment. Abration of the grains during deposition of sandy rock has probably lead to smooth, rounded grains. These zircons might have had a higher degree of altered rims preceding the abration.

Zircons with probable xenocrystic cores are present in all sample. Some probable cores are more likely to be xenocrystic than other. In many cases the cores are surrounded by fractures which radiate from the cores, indicating that the inner core have expanded, perhaps due to metamictisation as the volume increases. Between the samples, the ratio of grains with xenocrystic cores versus grains without did not vary much. The biggest difference is that the cores are more or less unaffected in the E4 1 and E4 2 and show zoning patterns discordant to the surrounding zoning of the grains, while the zircons from Lindön have cores that often are severely metamictised with no inner primary structures preserved.

In some cases the cores are not as distinct and could represent disrupted zoning and thereby reflect a change in the zoning pattern. This might be the case for the zircons in figures 6 C, 7 E and 8 C. Another possibility is that they in fact are xenocrystic cores but this is although very uncertain and is based mostly on the existing fractures around the central part. The fractures might however be an irregular fracture pattern of other causes. All three zircons show a changing zoning pattern, the zircons in figures 6 C and 8 C exhibit the most chaotic zoning. This might be a primary pattern that was formed during crystallisation in a magma or be an effect of metamorphism.

Some grains are recrystallised, which has produced homogeneous areas in the crystals. These types are most common in E4 1 (Fig. 6 A) and E4 2 (Fig. 7 G), and not as common in the sample from Lindön. The absence of recrystallised domains in the latter sample might be an effect of the severe metamictisation that continuously would destroy such structures. As recrystallisation occurs both during late-magmatic and metamorphism episodes, further conclusions about the formation of the recrystallised parts cannot be done without more detailed studies. The zircons from E4 2 have also large homogeneous rims, dark in CL, around a zoned central part. As described in the background, metamorphism can result in homogenous rims on pre-existing zircon. Whether this is the explanation to the wide rims on these zircons is uncertain. In the same sample there are zircon with CL-bright rims (Fig. 7 D, E and I). The formation of these rims might have similar explanation as the zircons previously discussed.

Inclusions and overgrowths of other minerals are present in all samples. Xenotime overgrowths of the zircons are common in the E4 1 sample, in addition to apatite which also occurs as inclusions. Probable xenotime is also present in E4 2 and Lindön samples, but not to the same extent.

Overall the zircons are best preserved in the E4 2 sample, where the transparency is high and metamict and altered grains are few. Most of the crystals from this sample are abraded and but still some have the primary form preserved. In Lindön and E4 1 the zircons are more altered and metamict but nevertheless have retained their original shapes. Fragmented zircon are also present in all samples. Metamict and altered grains are less durable to mechanical processes and thereby these grains probably have been crushed during the abration. This is probably the case for the E4 2 sample, while E4 1 and Lindön samples have experienced less abration and thereby have more altered, but less eroded zircons.

The Hundhällen sample contain non-rounded zircon similar to the E4 1 rather than the E4 2 sample. As only two zircons were extracted from Hundhällen, discussions and comparisons are hard to do. The infinitesimal number of zircons in that sample might depend on the source rock. The heavy minerals in a sedimentary rock are accumulated in certain strata and also the type of heavy mineral is dependent on the source rock/s. If zircon is less abundant in these so will the sediment. Also, the distribution of accessory minerals as zircon is not homogenous in rocks, and there is a possibility that the sample chosen contained extremely few zircons compared to the rest of the rock.

The internal zircon structures vary, both in and between the samples, but not to such a large extent that it is possible to make any conclusions whether they reflect different sources for the zircons. There are contrast differences between comparable zones among zircons from the same sample. The explanation to this might be that there are different sources of the zircons. Another explanation, perhaps more likely, is that there were variations in concentration of different trace elements in the original magma, leading to different concentration of these elements in the zircons even though they are from the same source. There might be only one, but also as several sources for the zircons, and only by further studies of the provenance, for example age determinations, can spread a light on this issue.

The BSE and CL images of the internal structures in the zircons would have been more accurate if the grains were unbroken and cut along the same crystallographic axis, perpendicular or parallel to the c-axis, and thereby the structures would have been easier to compare. It is important to note that many of the structures in the zircons in this study might be distorted due to sections oblique to the crystallographic axis. However, the purpose of this study was to investigate what types of different structures present. It was more important to find different types than finding a perfect section of the grains. A detailed zirconologic investigation is beyond the scoop of this limited study, which could otherwise have been made. To obtain more detailed results it would also have been necessary to make analyses on the compositional variations of the zircons, and to compare this with the zoning to establish whether they are correlated or not.

Studies of zircon zoning and other internal structures is very interesting and a lot of information might be extracted from such investigations. However the theories behind the development of different structures are still not fully understood and misinterpretations can easily be made. Structures formed in different environments might still be very similar.

In this study the BSE images are often of better quality than the CL images. According to most studies CL images use to reveal more information than the BSE, even though they often are less sharp This is further relevant in this study where only a mini-GATAN CL-detector has been used. In addition, as the CL detector is not used routinely, the optimal settings are not known and this part of the project has been a pilot study. Perhaps many of the zircons did not have enough variations to be revealed in CL and thereby giving very low contrasts. According to Hanchar and Rudnik (1995), the results with CL imaging depends on the size of the zircons, and zircons smaller than 60µm are difficult to image. Many zircons in this study are approximately of that size and that could be a contributing reason. The quality of the images also decreased with time and electric charging might have contributed to the poor resolution of some pictures.

6. Conclusions

The following conclusions can be drawn:

- Zircons can have numerous different structures. The types of structures that are present in a grain depend on the environment in which the grain has formed and also in which environment it has been affected during time.
- The zircons studied showed many different internal structures, but no obvious relationship between sample and zoning structures could be concluded.
- There were, however, a clear relationship between degree of alteration and sample, where E4 2 had the most transparent and also least altered zircons whereas Lindön had the least transparent and most altered zircons. E4 1 was an intermediate between those two.
- Further, E4 2 had the most abraded grains, which can be correlated to the originally high sand content. The zircons from the other twp samples had thick, unevenly abraded, altered rims, which the E4 2 zircons probably had lost, due to the abration.
- There are too little differences between the structures within the zircons of the different samples to make conclusions about the provenance of the zircons. Further analyzes are needed for this matter.

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