

# An EBSD study of the damage to zircon crystals by U decay and the implications for SIMS U/Pb dating.

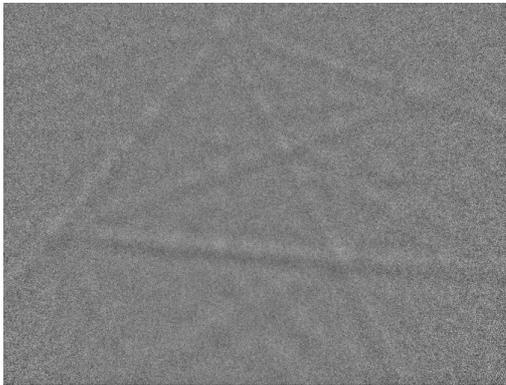
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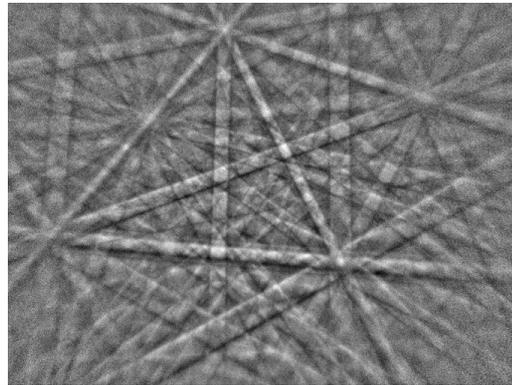
## Introduction

When analysing U-Th-Pb in zircon to determine its age, it is important to consider whether the crystal structure has been damaged by the natural radioactive decay of U and Th (Holland & Gottfried, 1955). If the crystal structure has become partly or fully amorphous, then products of radioactive decay (such as Pb) are more likely to have moved or been lost during heating or recrystallisation. This possible redistribution of Pb to other areas within a zircon grain has implications for the dating of such minerals as the method relies on accurate and original Pb/U isotopic concentrations. It is therefore important to know the state of the crystal structure to identify areas that may have been prone to Pb-loss. Since, ion beam damage will locally destroy much of this information, crystal structure determination must be made *before* any trace element, isotope or dating U-Th-Pb analyses.

Conventionally, scanning electron microscope (SEM) backscatter electron (BSE) and cathodoluminescence (CL) imaging have been used to characterise the growth histories and zoning within zircon grains prior to ion microprobe secondary ion mass spectrometry (SIMS) or other analysis. Typically, areas with a higher U content will appear brighter in BSE images and darker in CL images. Whilst these imaging methods give a good indication of the different areas within a particular grain they do not give any indication as to the state of the zircon crystal structure. However, electron backscatter diffraction (EBSD) is an SEM-based technique that utilises the diffraction patterns generated when an electron beam interacts with a crystalline sample. Using the EBSD technique, different areas of zircon grains can be analysed (non-destructively) at  $\leq 1\mu\text{m}$  intervals and the quality of the diffraction pattern assessed. Areas within the zircon grains that have been damaged by U and Th decay will be poorly crystalline resulting in weak, diffuse (see Figure 1a) or even no diffraction pattern, whereas areas that have the crystallinity preserved will give good, strong, clear diffraction patterns (Figure 1b).



**Figure 1a.** Weak diffraction pattern from a poorly crystalline area within a zircon grain.



**Figure 1b.** Good diffraction pattern from another area within the same zircon with good crystallinity.

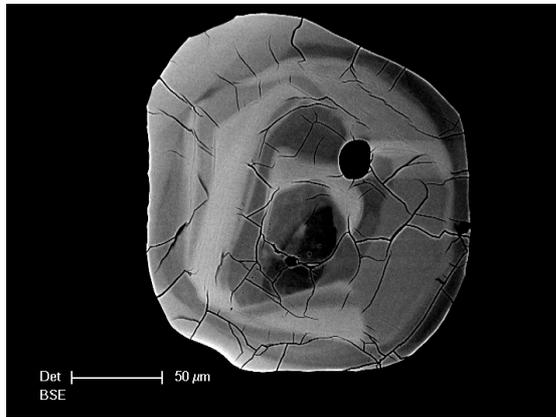
## Method

For the purpose of this study, a coarse grained leucogneiss (sample 49643) from McIntyre Island, Napier Complex, Antarctica was chosen as a suitable sample as it contained abundant zircon grains large enough to analyse and had been well characterised by previous petrographic work and SIMS analysis obtained using the Sensitive High Resolution Ion Microprobe (SHRIMP) at Curtin University of Technology, Australia. BSE and CL images were taken of 9 zircon grains and these were subsequently mapped using EBSD analysis to identify areas within the zircon grains according to changes in the degree of crystallinity (diffraction pattern quality). Trace element analyses were

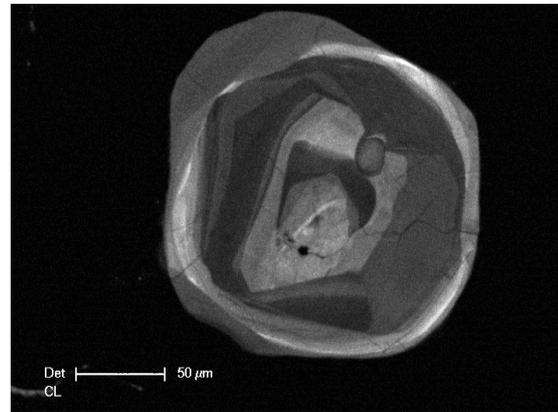
obtained using the Cameca IMS 4f ion microprobe and U-Th-Pb analyses using the Cameca IMS 1270 ion microprobe, both at the Ion Microprobe Facility, School of GeoSciences, University of Edinburgh.

## Results

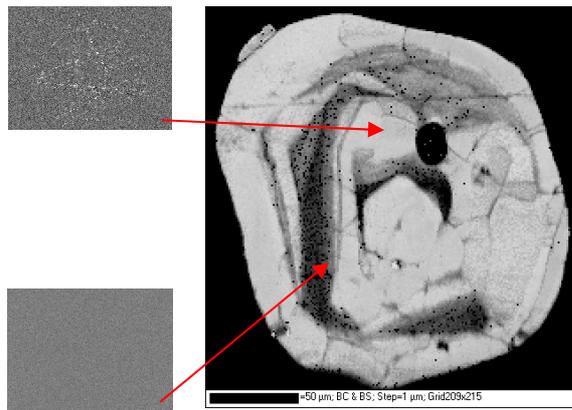
Figure 2 (below) shows that, in general, areas within the zircon grains that have a brighter BSE signal tend to be dark in the CL images. These same areas correspond well with areas that have poor diffraction patterns (poor crystallinity) in the EBSD maps.



**Figure 2a.** BSE image of one of the zircon grains studied.



**Figure 2b.** CL image of the same zircon grain as in Figure 2a. Note that areas that are dark in CL often correlate with areas that have a brighter BSE signal.



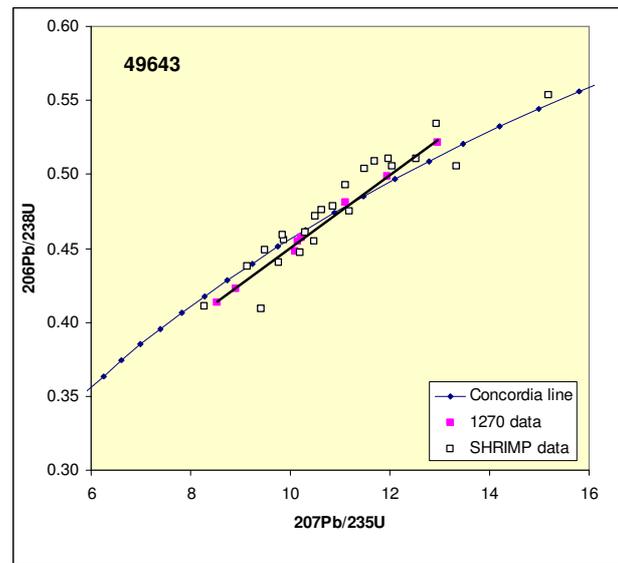
**Figure 2c.** The corresponding EBSD analysis map for the same zircon. The EBSD map shows variation in contrast according to the quality of the diffraction pattern as determined by changes in the band contrast (the relative intensity of the Kikuchi bands in the diffraction pattern) and the band slope (the sharpness of the diffraction image). The darker the pixel in the map, the poorer the crystallinity of the sample. Zero solutions (where the diffraction pattern was too poor to analyse) are shown as black. On the right are representative examples of good and weak diffraction patterns (as in Figure 1) for the appropriate areas within the zircon.

Trace element ion microprobe analyses for areas that have poor diffraction patterns in the EBSD maps show that these areas are characterised by higher U contents (>1400 ppm), whereas areas with good patterns have lower U concentrations (<1000 ppm). The concentration of Th and the other trace elements generally show little correlation with damage, therefore the Th/U ratio is generally defined by the U content and hence this ratio correlates with damage (0.05-0.07 for damaged areas compared to 0.17-0.36 for areas with good crystallinity).

The higher U content in damaged areas suggests that damage from U decay is a likely cause for the loss of crystallinity and there appears to be a U concentration threshold above which structural breakdown is always present. Once this limit is established for a particular suite of zircons, the U content could be used as an indicator of structural damage. In contrast, BSE and CL images do not always correlate with damage. A few areas within the zircons studied appear to exhibit similarly bright BSE and dark CL, but yet retain good crystallinity i.e. they have U concentrations below the threshold for obvious damage. The similarity of the bright BSE and dark CL signals in these areas is likely to be due to higher concentrations of other trace elements, especially Th, but further analyses are needed to confirm this. These areas might have been excluded from analysis initially if the BSE and CL images had been used without EBSD. It is also possible that such areas could have annealed

providing the temperatures and timescales were favourable for doing so, although it is not clear why some areas should anneal while others remain damaged.

The correlation between observed structural damage and the measured U-Th-Pb dates is complicated, in the samples studied, by the presence of at least two generations of zircon growth. On a Concordia plot (of  $^{206}\text{Pb}/^{238}\text{U}$  against  $^{207}\text{Pb}/^{235}\text{U}$ , see Figure 3) all points lie on a chord between  $2.25 \pm 0.05$  Ga and  $2.60 \pm 0.05$  Ga. A couple of the oldest points lie slightly above the Concordia line, but all lie well within the scatter of the previous SHRIMP data. Correlation along the chord requires further interpretation, but there does not appear to be a significant correlation between displacement along the chord and degree of damage. There is no evidence for lead loss after these dates (where the points would lie on a chord through zero), even in the most damaged areas.



**Figure 3.** Concordia plot illustrating the (1270) data collected in this study together with the previous SHRIMP data

## Conclusions

EBSA analysis is the only way of indicating areas of damaged / undamaged crystalline structure within zircon grains. BSE and CL images when used in conjunction with each other can suggest areas that are likely to be damaged, but they do not necessarily give a definitive picture of the extent or location of areas where the crystal structure has been damaged by radioactive decay. Checking zircon grains with EBSA analysis should therefore be an important consideration before trace element ion-microprobe analyses.

This study has only looked at a limited set of samples. A wider study of the correlation between BSE, CL, EBSA and trace element analyses on a wider set of samples from different geological settings is planned in order to investigate further how representative the above results are of zircons in general.

## References

Holland, H.D. & Gottfried, D. (1955). The effect of nuclear radiation on the structure of zircon. *Acta Crystallographica* **8**, p291-300.