PROCEEDINGS OF THE TWENTY-EIGHTH ANNUAL KECK RESEARCH SYMPOSIUM IN GEOLOGY

April 2015
Union College, Schenectady, NY

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ISSN# 1528-7491

The Consortium Colleges   The National Science Foundation   ExxonMobil Corporation

KECK GEOLOGY CONSORTIUM
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ISSN# 1528-7491

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Funding Provided by:
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Funding Provided by:
Keck Geology Consortium Member Institutions
The National Science Foundation Grant NSF-REU 1358987
ExxonMobil Corporation

Keck Geology Consortium: Projects 2014-2015
Short Contributions— Tectonics of Prince William Terrane, AK Project

Funding Provided by:
Keck Geology Consortium Member Institutions
The National Science Foundation Grant NSF-REU 1358987
ExxonMobil Corporation

Learning Science Through Research
Published by Keck Geology Consortium
Short Contributions
28th Annual Symposium Volume
25th April, 2015
ISBN: 1528-7491

ANNEALING RADIATION DAMAGE IN PRECAMBRIAN ZIRCON IN WHALE BAY, ALASKA AND LABORATORY EXPERIMENT

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INTRODUCTION

The Chugach-Prince William (CPW) terrane in Alaska is a thick accretionary complex comprised partly of sandstones dominated by Mesozoic detrital zircon, but almost all units have a small fraction of Precambrian zircon (Hilbert-Wolf, 2012; Roe, 2013; Petiette 2013; Frett, 2014; Rick, 2014). Although rare, Precambrian detrital zircons are vital in our understanding of the provenance and thermal history of the complex because there have distinct sources along the continental margin. Raman Spectroscopy can be used to quantify the amount of radiation damage in zircons and the crystallinity can then be related to thermal history (Geisler et al., 2001; Nasdala et al., 2001; 2003). The primary goal of this research is to determine the temperature at which disorder in Precambrian zircons becomes annealed and then relate these findings to geologic settings. Radiation damage dating requires an understanding of the metamorphic history, because high temperatures anneal damage and change the apparent damage age. The overall study is aimed at understanding radiation damage in Precambrian grains in Alaska to better understand provenance of the zircon grains as well as the thermal history of the CPW complex.

BACKGROUND

Radiation damage in zircon is directly related to the uranium and thorium concentration in the crystal, the time that has elapsed, and annealing that may have occurred due to high temperatures. As a grain accumulates radiation damage, the crystal lattice becomes less rigid, more disordered, and less crystalline as uranium decays, thus uranium content and time directly affect crystallinity (Holland and Gottfried, 1955; Woodhead et al., 1991; Ellsworth, 1994; Zhang et al., 2000; Geisler et al., 2001; Nasdala et al., 2001; 2003). Crystallinity can be measured by Raman spectroscopy, and zircon has four different Raman-active modes (Geisler et al. 2001; Marsellos and Garver, 2010). For the ν_3SiO_4 Raman-active mode (the main peak we investigate), a wavenumber near 1007 to 1008 is undamaged highly crystalline zircon, whereas a Raman wavenumber near 1000 is a high-damage zircon, and very high damaged zircons have values below 1000. The crystallinity in zircon affects geochronolometric measurements, but is also related to the high-temperature thermal history of a zircon.

A crystalline zircon becomes amorphous, also called “metamict”, due to self-irradiation by actinides (U and Th), and recovery is determined by the microstructure within each zircon after radiation damage (Nasdala et al., 2002). For annealing of radiation damage, the onset temperature is the temperature at which the crystal begins to recover. In laboratory settings, little to moderately damaged zircons begin to recover around 500°C and are completely recovered by 1300°C, with a significant portion of structural the recovery occurring above 1000°C (Nasdala et al., 2002). Highly metamict zircons differ from moderate metamict zircons in the onset temperature because the lack of crystalline $ZrSiO_4$ makes low temperature reorganization impossible (Nasdala et al., 2002).

The two-step process of how partially damaged zircons recrystallize is described by Ewing el al. (2003). In the first step, lattice recovery begins to heal below ~727°C as seen through Raman spectroscopy and X-ray diffraction measurements. The result is a zircon with both crystalline regions and amorphous domains. In the second step, temperatures above ~727°C decrease the number of amorphous domains as seen through the decrease of the ν_3 Raman line-width and an increase in x-ray scattering intensity, signaling increased coordination of the Zr cations from seven-fold to eight-fold. At high temperatures, there is epitaxial recrystallization of partially damaged zircon due to the integrity of the microstructure (Ewing el al., 2003).

It is widely thought that above a critical threshold (~3.5 x 10^{15} α/mg), zircon become so damaged that different processes are required for annealing (Balan et al., 2001). Highly metamict zircons have more damaged microstructures and separation of phases, which may create another step in the recrystallization process. Reconstitution of highly metamict zircon involves an intermediate stage of decomposition into oxides as seen by a greater difference in enthalpy between highly radiation-damaged and crystalline zircon compared to metamict zircon and a mixture of tetragonal ZrO_2 and glassy SiO_2 (Nasdala et al., 2002). The temperature interval in which the intermediate oxide stage occurs appears to extend between temperatures ~800 to 950° and roughly 1100°C.

Recrystallization on geologic timescales is complex because it is difficult to accurately constrain the time, temperature, and rates of annealing. High temperatures may only be necessary in laboratory experiments, while lower temperatures across long geologic time may also recrystallize zircon (Ewing el al., 2003). Others suggest that geologic recrystallization only occurs at temperatures greater than 700°C for ~370 Ma (Geisler et al. 2001). The dominant mechanism for geologic settings may be purely thermal, diffusion-driven recrystallization (Ewing el al., 2003). However, there are practically no constrained examples of quantification of natural annealing under geologic conditions.

METHODS

The affect of temperature in annealing radiation damage has been examined in the natural setting, and in the laboratory. The natural annealing experiment involved rocks of the Chugach-Prince William terrane exposed in Whale Bay on Baranof Island collected in a transect with metamorphic grade increasing from prehnite-pumpellyite to the middle amphibolite facies (biotite + andalusite) adjacent to the 50 Ma Crawfish Inlet pluton. The laboratory experiment used the Sri Lanka Precambrian zircon which is a widely used standard, and these samples were heated between the temperatures of 400°C–1000°C to determine the temperature at which zircon becomes annealed.

Whale Bay Field Collection and U/Pb Dating

U/Pb detrital zircon ages were obtained from five samples collected along a SW-NE transect in Whale Bay, south of Sitka, Alaska (Rick, 2014). Zircons were separated using standard rock pulverization, magnetic separation, and density separation techniques using heavy liquids. Approximately one hundred zircons from each sample were randomly selected and individually dated using LA-MC-ICPMS at the Arizona Laserchron Center (Gehrels et al., 2008). Some samples have more than one hundred analyses because they had 100 traditional analyses, but also analyses from hand picking that targeted Precambrian grains. In the selection process, Precambrian grains were selected based on roundness and a pink color with the assumption that round grains have experienced significant transport and perhaps recycling, and a pink to purple color reflects accumulated radiation damage (Garver and Kamp, 2002). Analysis of the U/Pb data was conducted by Rick (2014) using IsoPlot (Ludwig, 2003) and Age Pick, an Excel macro provided by the LaserChron Center at the University of Arizona. The dated Precambrian grains are those analyzed in this study.

Raman Spectroscopy

Raman spectroscopy was used to measure the radiation damage of Precambrian zircons in samples WB13-4, WB13-15, and WB13-16 from Whale Bay, Alaska (Rick, 2014). Raman measurements were made using a 633 nm external He-Ne laser, and a Bruker Optics Senterra® Spectrometer coupled to an Olympus® BX51 reflected light microscope at Union College. The spectrometer includes a computer controlled three-grating turret with a spectral resolution up to 3 cm^{-1} and automatic laser and Raman frequency calibration. Samples were first located at 100x using bright field objectives, and the measurements were made with video camera and long working-distance dark field objectives at 500x or 1000x. The signal was captured by a low-noise 1024x256 pixel thermoelectric-cooled CCD detector. Measurements were made with a laser power of 20 mW, and an aperture of 25 x 1000 _m. An integration time of 15 to 60 s was used during acquisition of the Raman shift, and automated collection was done for background and monochromatic wavelength. For samples with a very strong peak to background ratio, a simple rubberband background correction was made, but for those with a more elevated background or a background broadly concave (likely due to slight fluorescence), a concave rubberband background correction was used. We then used FitYK® for peak fitting and we concentrated our efforts on the $\nu_1 SiO_4$ (~974 cm^{-1}) symmetric stretching and the $\nu_3 SiO_4$ (~1007 to 1008 cm^{-1}) antisymmetric stretching (i.e. Marsellos and Garver, 2010). We use a Gaussian/Lortenzian approximation using the Levenburge-Marquardt method. Each grain was measured twice in slightly different spot locations that tended to vary by less than 20 um, and grains were oriented with c-axis in a N-S position. The reported values for the Raman modes are the average of the two spot measurements. The average variation in measurements for the $\nu_3 SiO_4$ mode was ~0.2 cm^{-1}. For uranium and thorium determination, we use the numbers generated by the LAICPMS measurements, which is not ideal because they are not based on quantitative volume estimates and are assumed to have an uncertainty of about 30%.

Laboratory Experiment

To measure annealing of variation damage in a laboratory setting, a high damage zircon was progressively annealed in a laboratory oven. To accomplish this, 16 fragments of a single Precambrian Sri Lanka zircon (564 Ma) were analyzed in this experiment. Crystallinity of each fragment was first measured using Raman Spectroscopy. Two zircons were allocated for each temperature, and four allocated for the non-heated standards. After Raman measurements, samples were heated to 400°C, 600°C, 800°C, 900°C and 1000°C in an oven for 22,500 seconds. Note that a total of four samples were heated to 900°C due to an outlier zircon with low uranium. The Raman active modes where then again measured for each sample after heating. The uranium concentration of these fragments were not determined, but they are likely similar to the concentration of 518 ppm, which is the assumed concentration used in all analyses, although there does seem to be some variation.

RESULTS

Whale Bay, Alaska

The Raman active modes in the Precambrian zircon show a distinct trend with proximity to the Crawfish Pluton and metamorphic grade. Precambrian zircons from the southern units (WB13-16) away from the pluton have significant disorder in Prehnite-Pumpellyite grade rocks (≥300-350°C), they have less disorder in the biotite zone (~400°C) (WB13-04), and they are the most crystalline (least disorder), in rocks with andalusite + biotite (≥500°C) (WB13-15PC). This change is demonstrated by the observation that the $\nu_3 SiO_4$ progressively shifts toward 1008 cm^{-1}, as the metamorphic grade increases (Fig. 1).

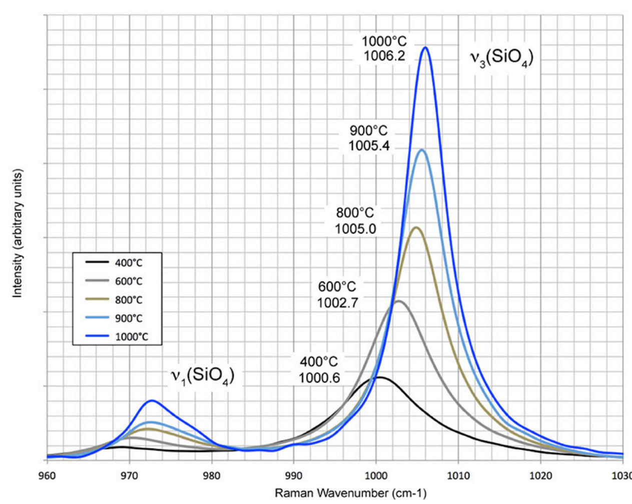

Figure 1. Raman wavenumber (n3SiO4) vs. effective uranium content (eU) for Precambrian grains along the Whale Bay transect. Shown in light blue are zircon grains from the Kodiak area, which are used as an un-annealed reference

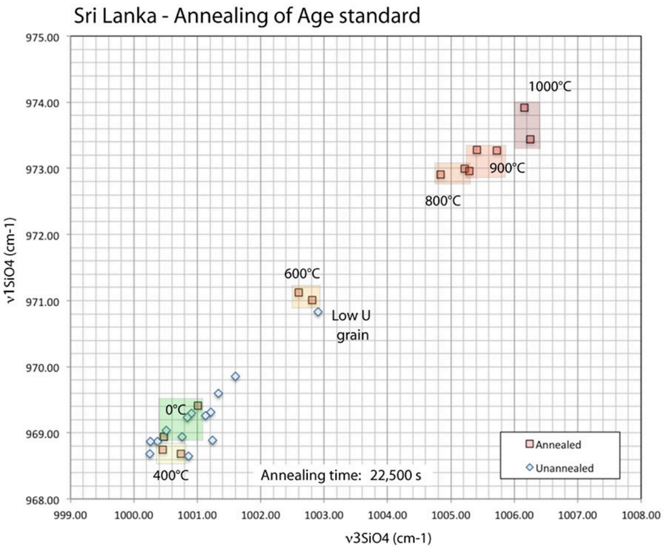

Figure 2. Raman wavenumber of the n3SiO4 and the n1SiO4 peaks with incremental heating steps.

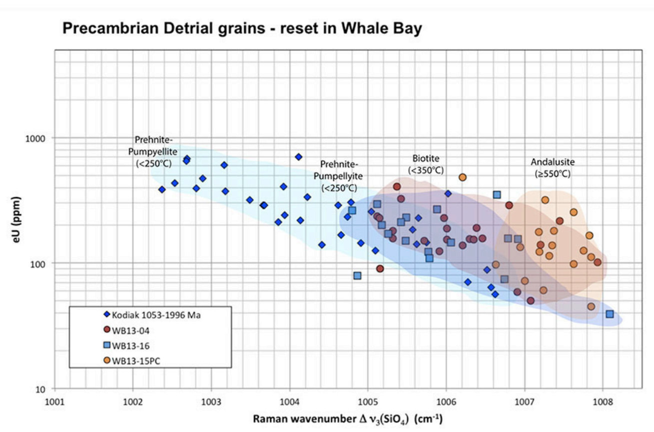

Figure 3. Raman n3SiO4 and n1SiO4 peaks from laboratory annealing experiments. Note how progressive annealing results in the shift in the position of the peaks, and a decrease in the width of the peaks (decrease in FHWM).

LABORATORY EXPERIMENT

The natural annealing process discovered in Whale Bay was recreated in the laboratory. The active Raman modes of Sri Lanka Precambrian zircons (Neoproterozoic) were measured, and determined to be highly damaged (ν_3SiO_4 at ~1000 to 1000.5 cm^{-1}). What is important in this experiment is the amount of structural recovery at different temperatures. At 400°C, no significant change in the radiation damage was present and the pre- and post-heating measurements were essentially identical. At 600°C, the damage was partially annealed (~1002.7 cm^{-1}). At temperatures between 800°C and 900°C, the damage became more fully annealed (~1005.7 cm^{-1}) (Figs. 2 & 3). Our highest temperature step of 1000°C resulted in ν_3SiO_4 of ~1006.2 cm^{-1}, a number that is the most crystalline in this study, but not fully crystalline (still below 1007 to 1008 cm^{-1}). The biggest and most dramatic annealing occurs between 400 and 800°C.

DISCUSSION

The crystallinity of a zircon is dependent on the amount of uranium in a crystal and time. The older zircons accumulate more radiation damage than younger zircons. Annealing occurs when damaged zircon becomes crystalline in high temperatures, and thus radiation damage decreases with increasing metamorphic grade. Our hypothesis is that the zircon crystals with high radiation damage begin the annealing process in greenschist facies (≥300-350°C) and radiation damage becomes fully annealed (or crystalline) in amphibolite facies (≥500°C). The results from the laboratory experiment suggests annealing at ~800°C, which supports the field evidence from Whale Bay.

Precambrian zircons are important in outboard terranes in Alaska because they are uncommon but useful to provenance analysis. They are also important because they record thermal histories of suites of rocks. In this case, the total radiation damage may be useful to understand how long a zircon has been in high crustal levels and accumulating damage, because this disorder may be annealed if the rocks are brought to high enough temperatures during metamorphism, such as those from the middle amphibolite facies rocks from Whale Bay.

Radiation damage dating may be a useful technique to indicate the provenance and thermal history of Precambrian zircon the Chugach–Prince William terrane. This provenance information may help in our understanding as to whether the units were translated northward since deposition (Cowan, 2003). Detrital zircon in the Yakutat Group (1047-1793 Ma) clearly record a heating event in recent history (~100 Ma), as seen through a much more crystalline structure compared to detrital zircon in the Kodiak-Prince

William Sound (1053-1996 Ma) area (Garver and Davidson, in press). A more thorough investigation of the relationship between uranium content and temperature in Precambrian zircons will allow us to solidify radiation damage dating as a confident method of dating. Further laboratory experiments will constrain the temperature at which a crystal anneals.

ACKNOWLEGMENTS

The fieldwork in Alaska was made possible by the Keck Geology Consortium and the National Science Foundation (NSF EAR 1116554 to John Garver, Union College; EAR 1116536 to Cameron Davidson, Carleton College EAR-1062720 to Robert Varga, Pomona College). The U/Pb dates were analyzed the Arizona LaserChron Center, which is organize and maintained by Mark Pecha and George Gehrels at the University of Arizona. Raman Spectroscopy conducted at Union College is made possible by NSF grants. Thank you to John Johnson and the Chugach Native Cooperation for allowing us to stay on Nucheck, as well as the Orca Adventure Lodge for their hospitality and transportation to Hinchinbrook Island. I can't thank Professor John Garver and Professor Cameron Davidson enough for their hard work and dedication to enriching our future with this incredible fieldwork and research experience. Thank you to the Union College Geology Department for their commitment to giving their students the best education possible.

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