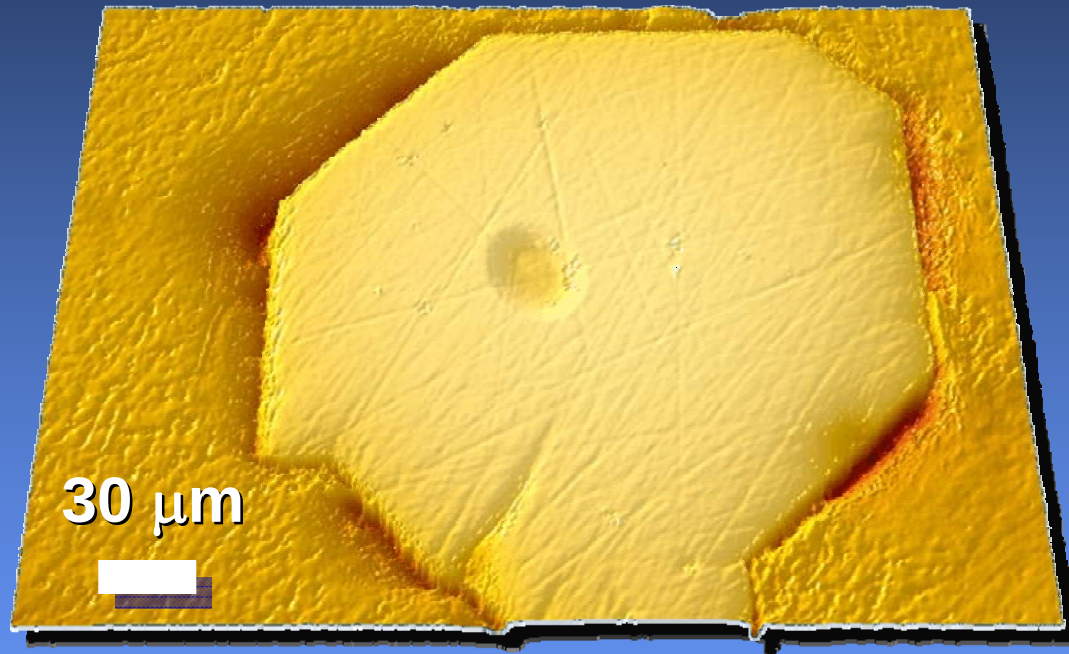


U-Pb (and U-Th) dating of micro-baddeleyite

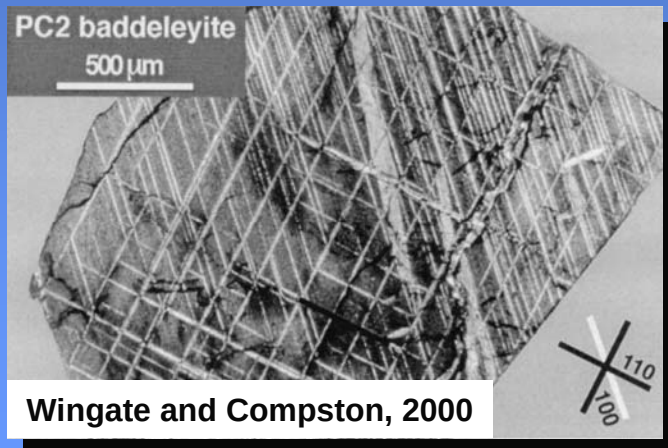
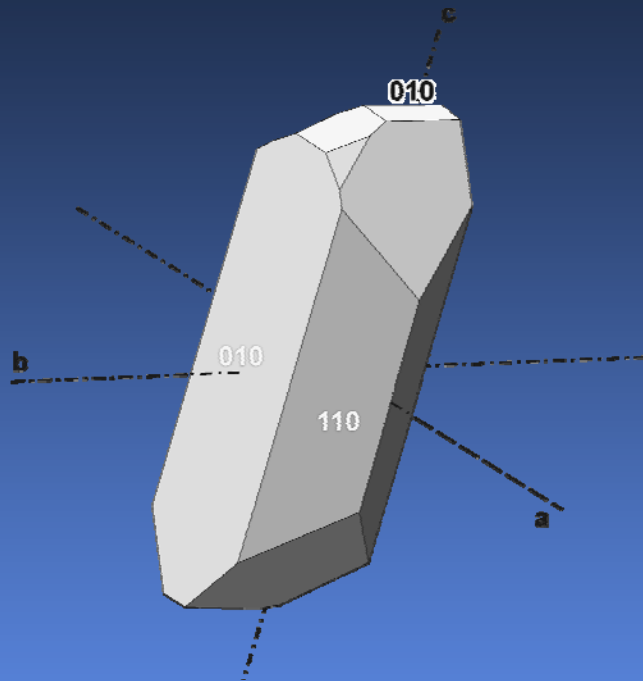


Axel K. Schmitt

UCLA SIMS, NSF National Ion Microprobe Facility

Collaborators: T. Mark Harrison (UCLA)
Kevin Chamberlain (University of Wyoming)

Baddeleyite (BAD-ü-LĒ-ite)* basics

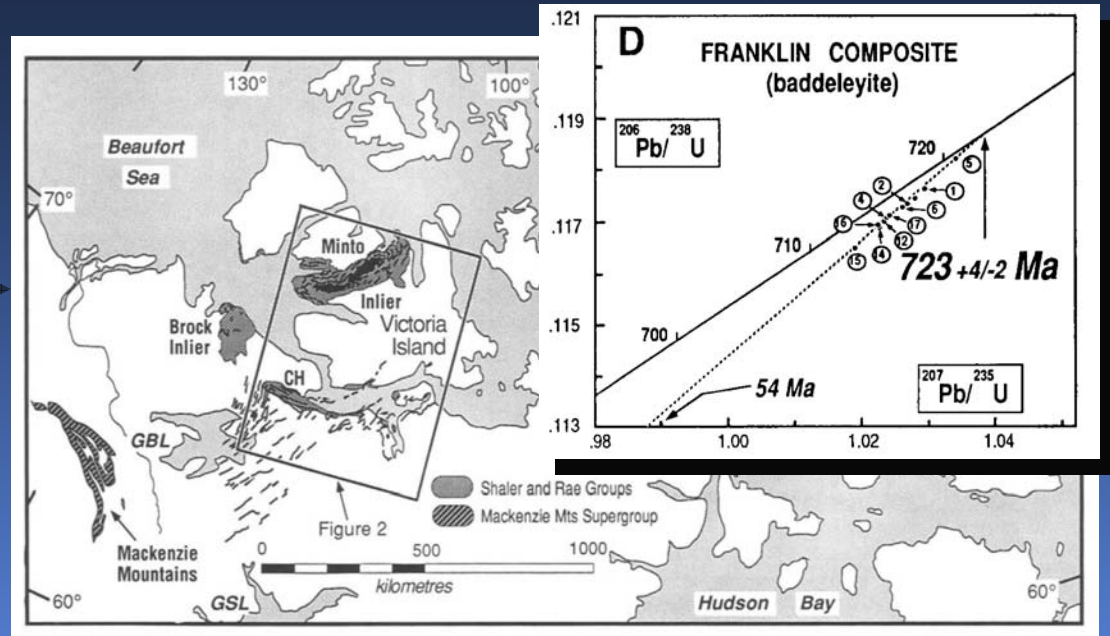


- chemical formula: ZrO_2
- monoclinic (commonly twinned)
- minor HfO_2 , TiO_2 , FeO , SiO_2
- U between ~200 – 1000 ppm
- low common Pb, $\text{Th/U} \ll 0.2$
- wide range of occurrences (terrestrial and extraterrestrial)
 - mafic and ultramafic rocks (basalt, gabbro, diabase)
 - alkali rocks (carbonatite, syenite)
 - mantle xenoliths (from kimberlites)
 - metacarbonates
 - impact-related rocks (tektites)

Baddeleyite dating: applications and examples

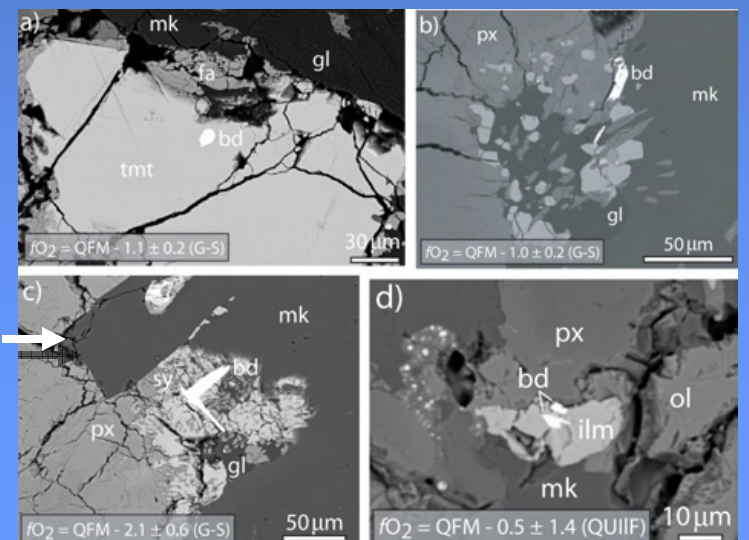
Bulk analysis (TIMS)

- Mafic dikes and layered intrusions (e.g., *Heaman et al., 1992*) →
- Detrital baddeleyite (e.g., *Bodet and Schärer, 2000*)



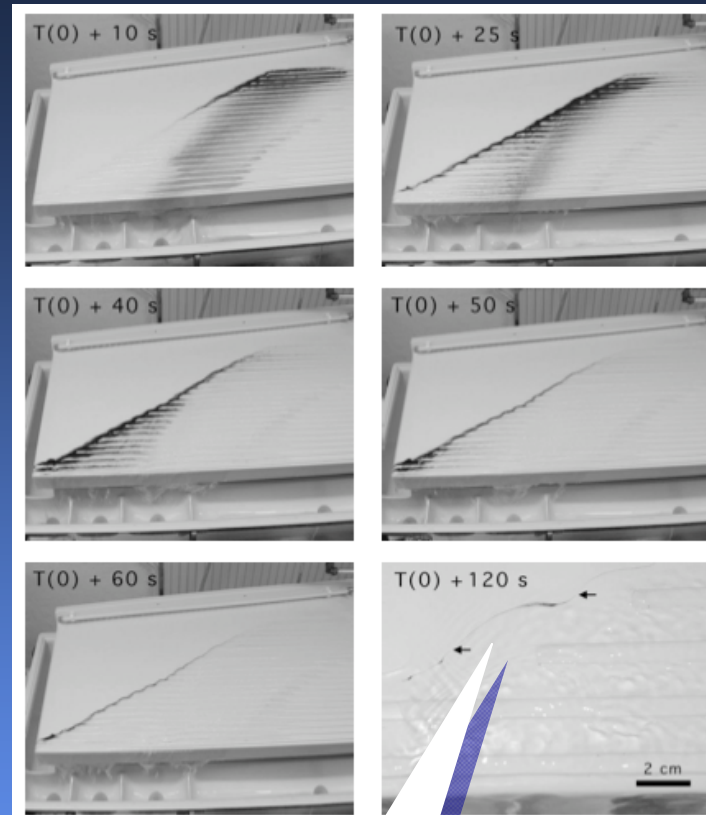
In-situ methods (SIMS, LA ICP MS, EPMA)

- Mafic dikes and gabbros (e.g., *Wingate et al., 1998; French et al., 2000*)
- SNC meteorites (Herd et al., 2007: $70 \pm 35 \text{ Ma}$ and $171 \pm 35 \text{ Ma}$) →



Micro-baddeleyite analysis: in-situ advantages

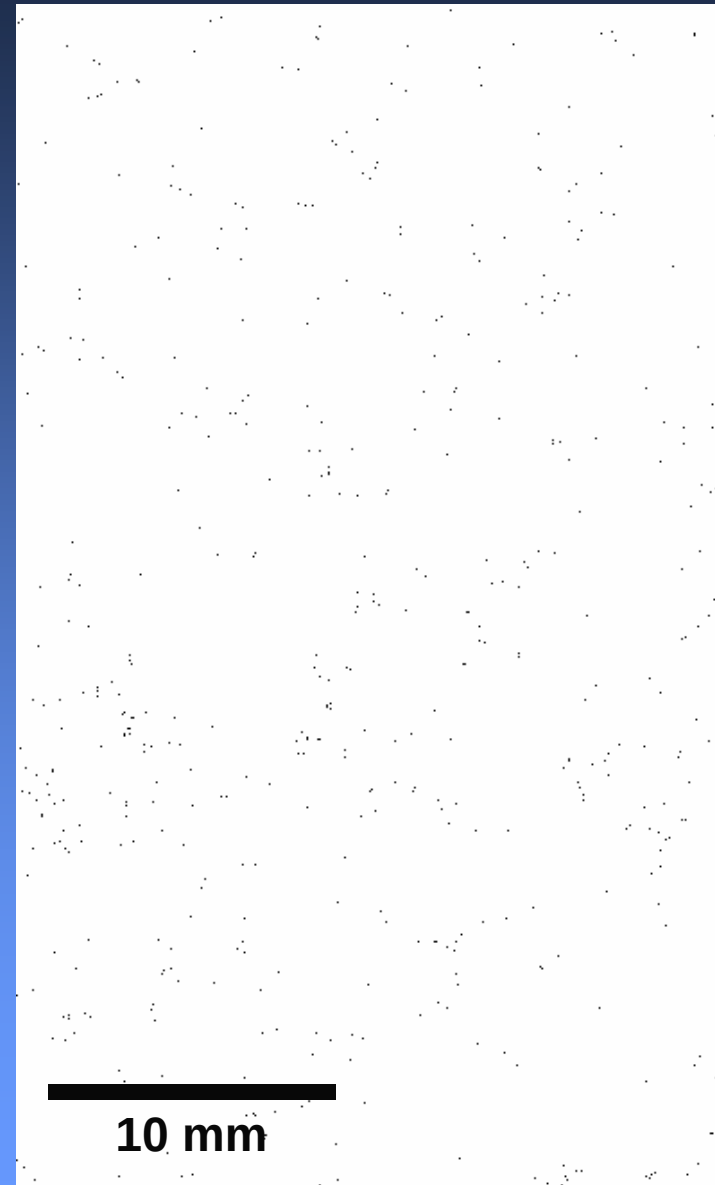
- Bulk analysis difficulties:
 - time-intensive, highly inefficient mineral separation
 - 100's of grains (<20 μm) required
 - contamination risk (though inheritance unknown)
 - abrasion impossible (Pb-loss)
- In-situ advantages:
 - grains can be located and analyzed in thin-section
 - no separation required
 - petrographic context preserved
 - limited sample size (e.g., drill samples, extraterrestrial)
 - screening for concordant grains



Söderlund and
Johansson, 2002

Micro-baddeleyite analysis: in-situ advantages

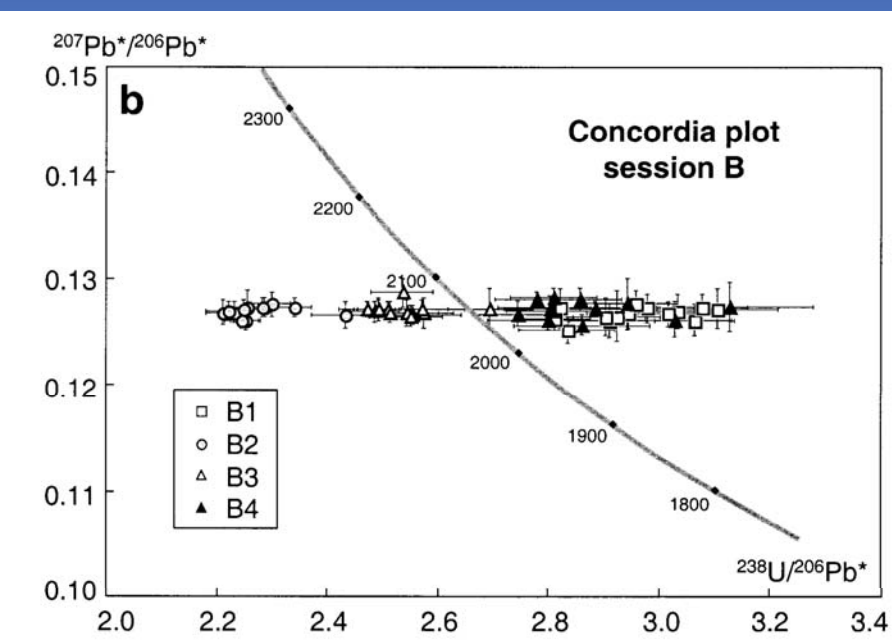
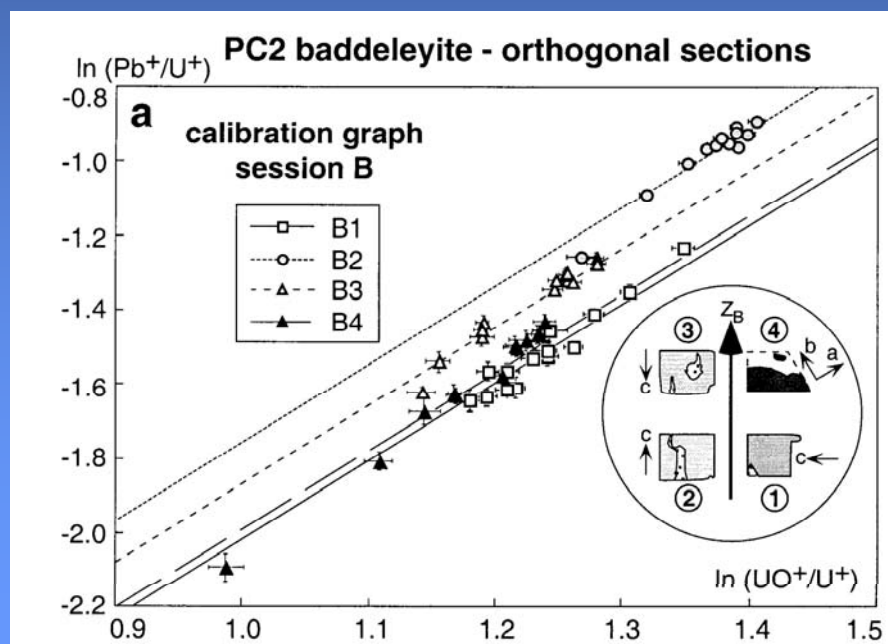
- **Bulk analysis difficulties:**
 - time-intensive, highly inefficient mineral separation
 - 100's of grains (<20 μm) required
 - contamination risk (though inheritance unknown)
 - abrasion impossible (Pb-loss)
- **In-situ advantages:**
 - grains can be located and analyzed in thin-section
 - no separation required
 - petrographic context preserved
 - limited sample size (e.g., drill samples, extraterrestrial)
 - screening for concordant grains



Zirconium X-ray map of mafic dike rock

Previous SIMS U-Pb dating of baddeleyite

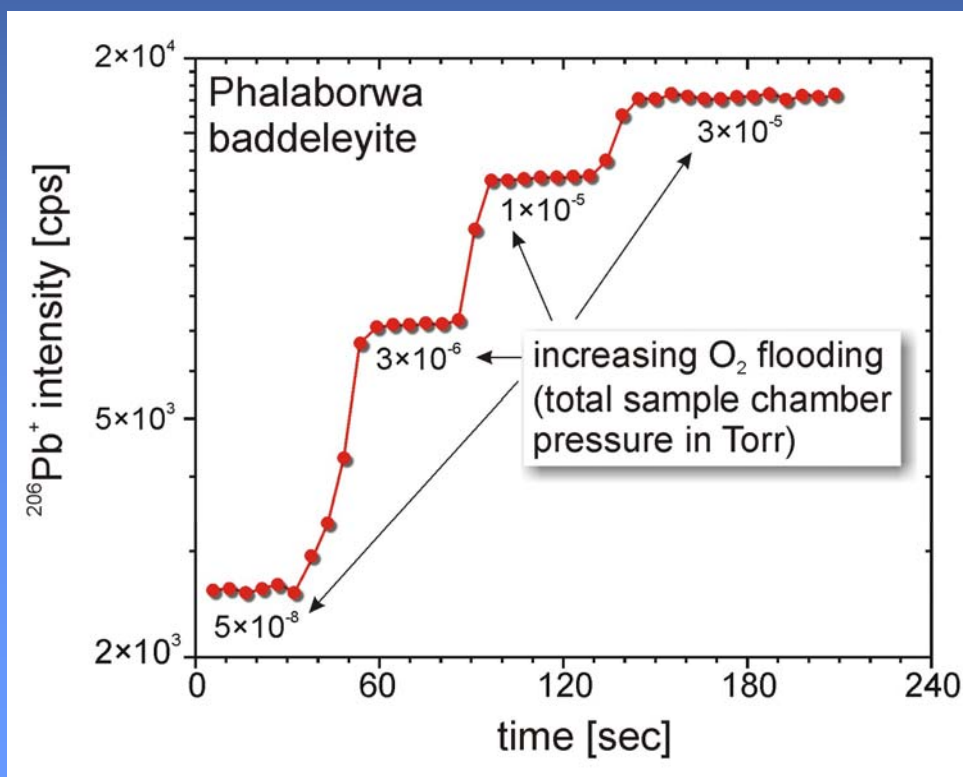
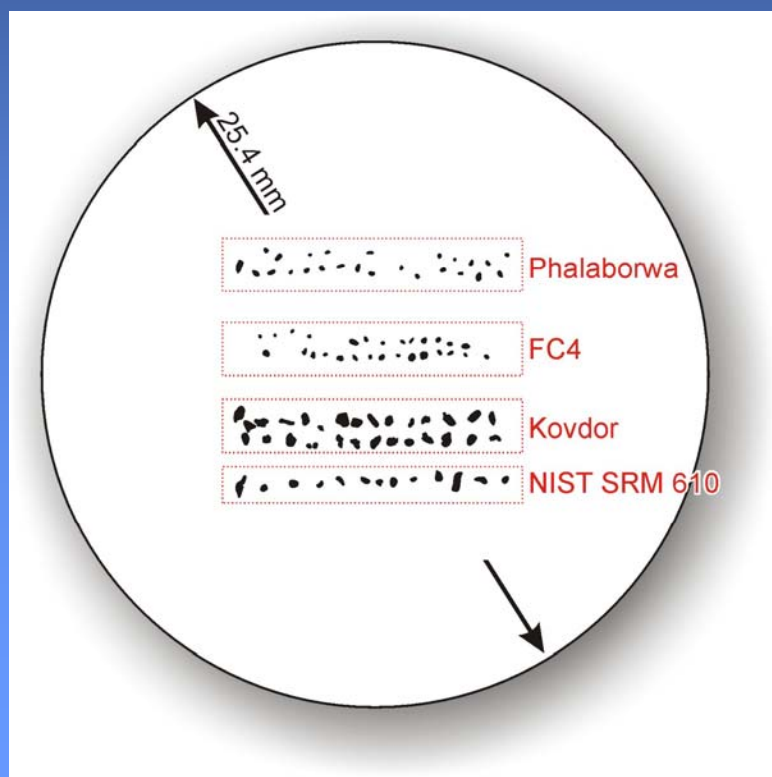
- SHRIMP II (Wingate and Compston, 2000)
 - Phalaborwa baddeleyite standard
 - $^{207}\text{Pb}/^{206}\text{Pb}$ ages accurate
 - $^{206}\text{Pb}/^{238}\text{U}$ ages deviate by up to -13% and +17%
 - crystal orientation affects U-Pb sensitivity
 - severe limitation on accuracy of U-Pb baddeleyite ages



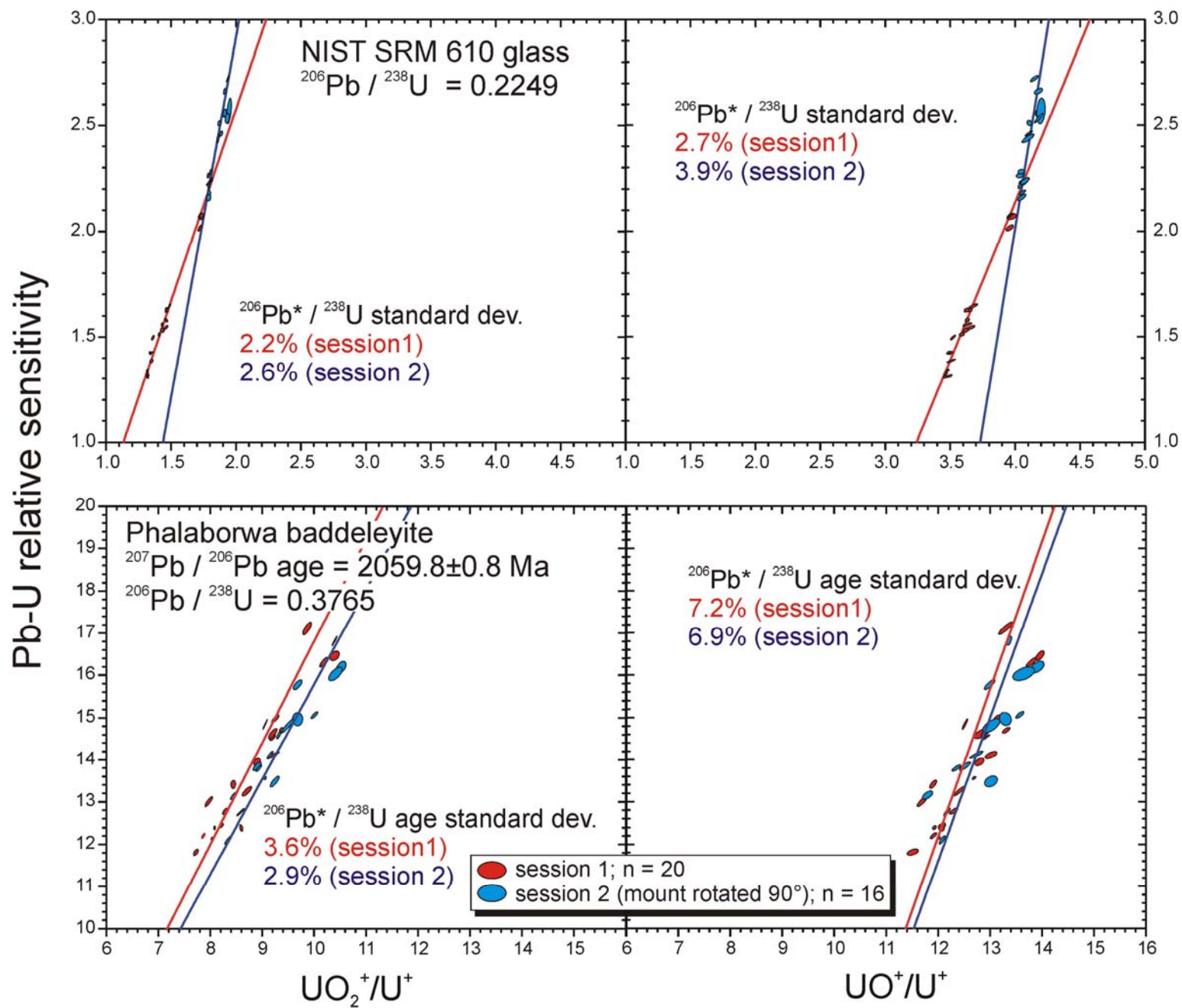
Wingate and Compston, 2000

Baddeleyite U-Pb analysis with the ims 1270

- cross-calibration of three baddeleyite standards
- randomly oriented grains, NIST SRM 610 glass on same mount
- 144 spot analyses total
- two sessions (overnight, fully automated beam centering and charge-compensation): rotate mount 90°
- $\sim 8\text{-}11\text{ nA } ^{16}\text{O}^-$ primary beam, $25\text{-}30\text{ }\mu\text{m}$ spot size, MRP = 4500
- O_2 - flooding (700% increase in Pb^+)

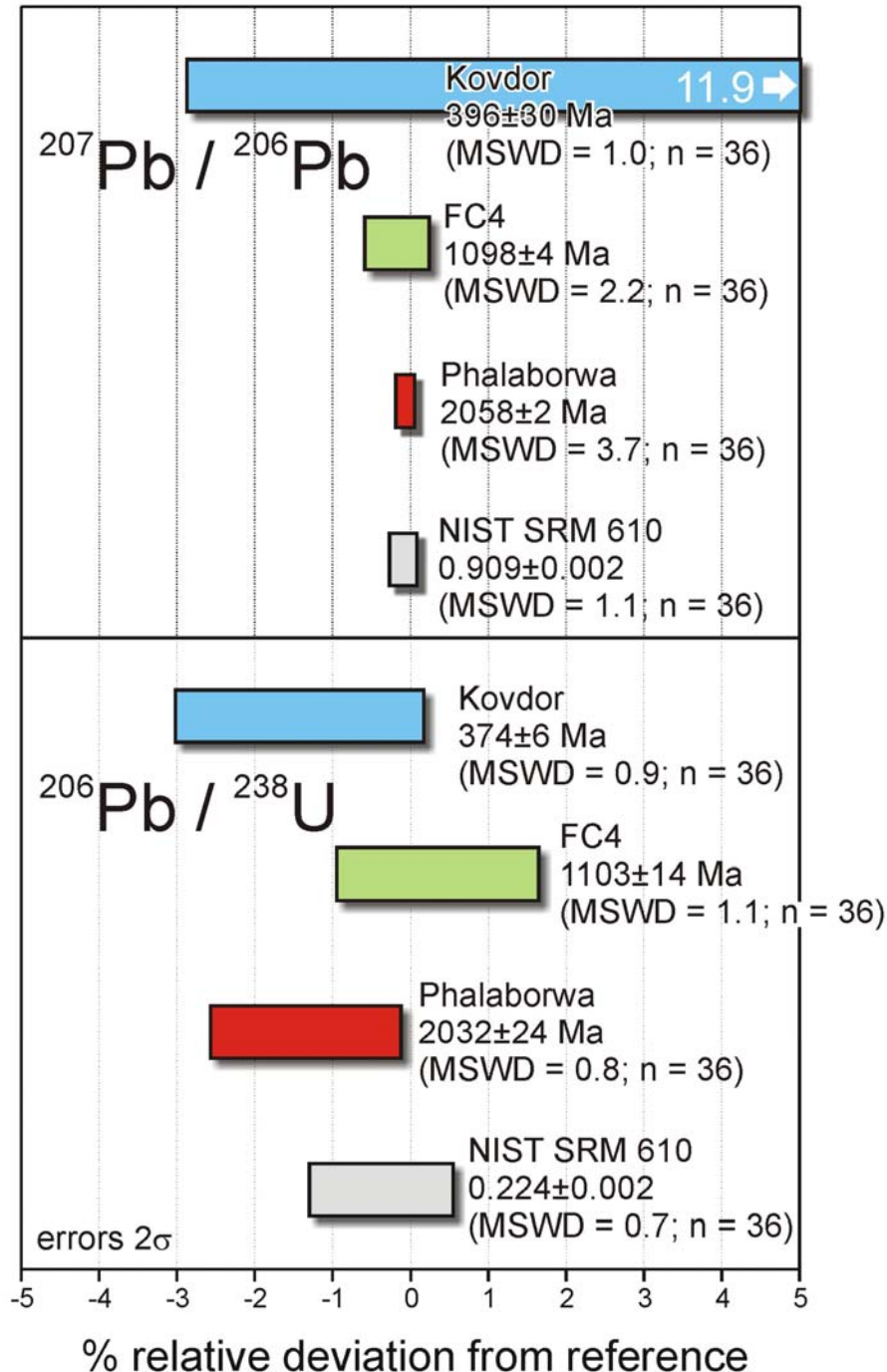


U-Pb calibration results



Standard intercomparison

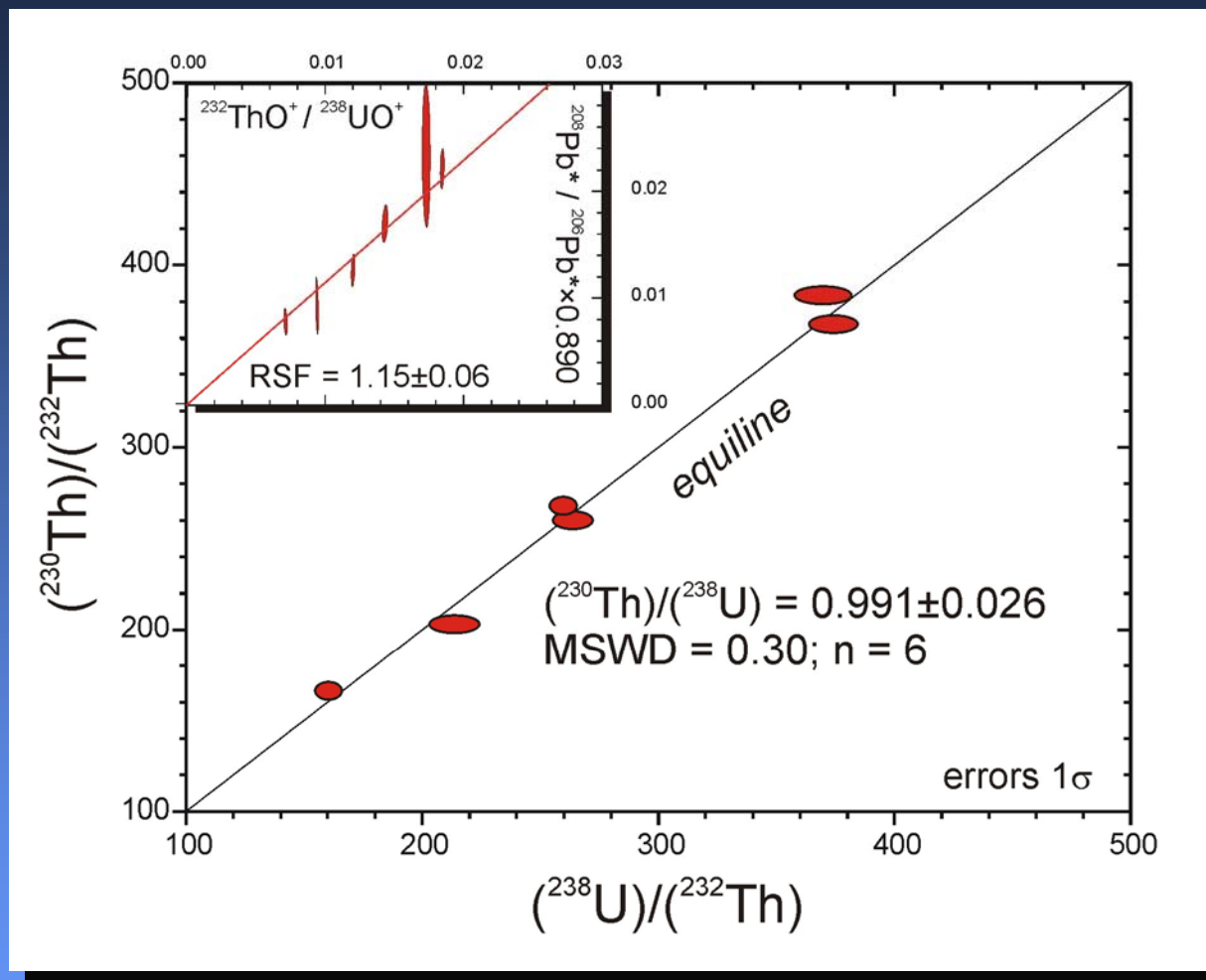
- new TIMS results for Kovdor and FC4 (Duluth)
- differences in calibration after rotation unrelated to crystal-orientation
- reproducibility on baddeleyite slightly poorer compared to glass
- highly accurate $^{207}\text{Pb}/^{206}\text{Pb}$ ages for Precambrian baddeleyite
- $^{206}\text{Pb}/^{238}\text{U}$ ages agree within measurement uncertainties ($\sim 1\text{-}2\%$ 2σ)



Phalaborwa: Heaman and LeCheminant, 1993

NIST SRM 610: Walder et al., 1993; Hirata and Nesbitt, 1995

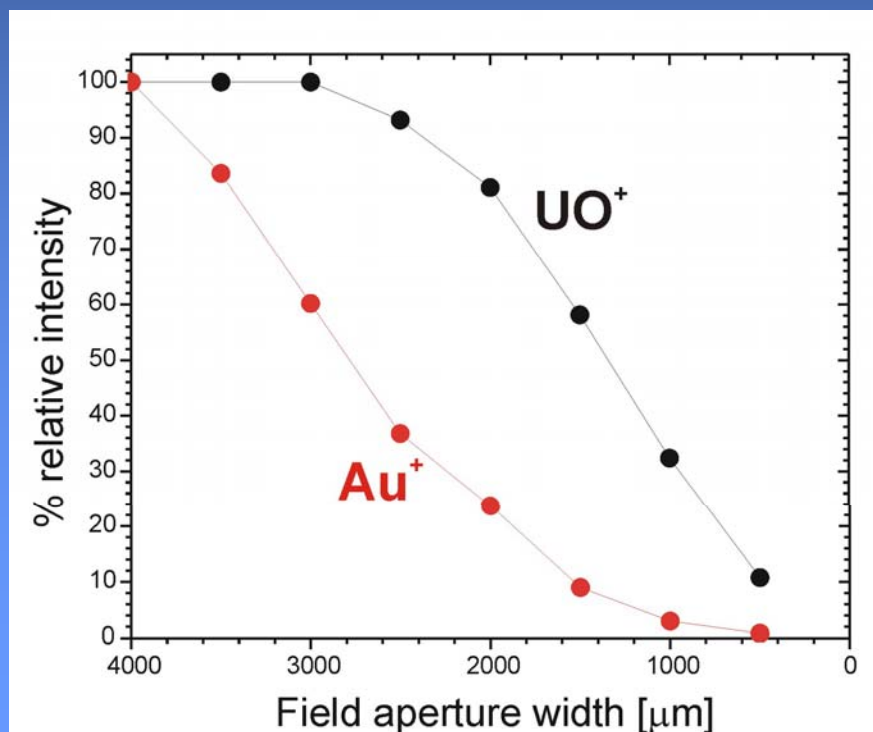
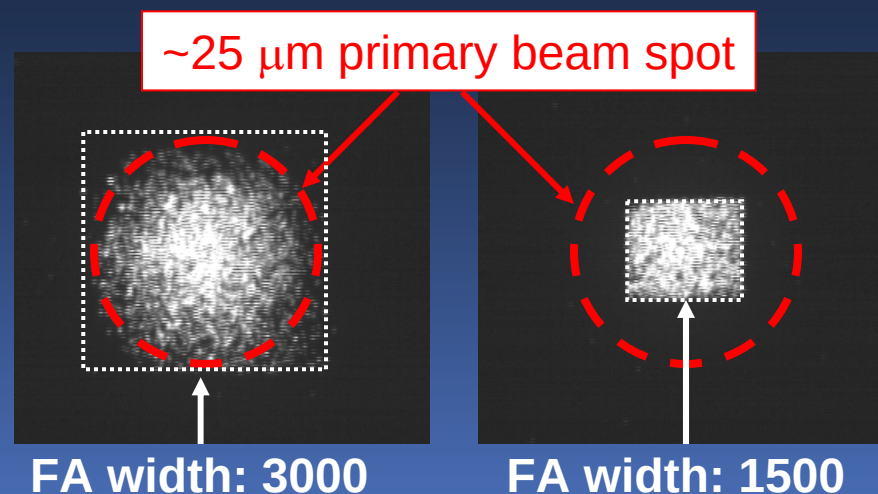
Baddeleyite U-Th dating



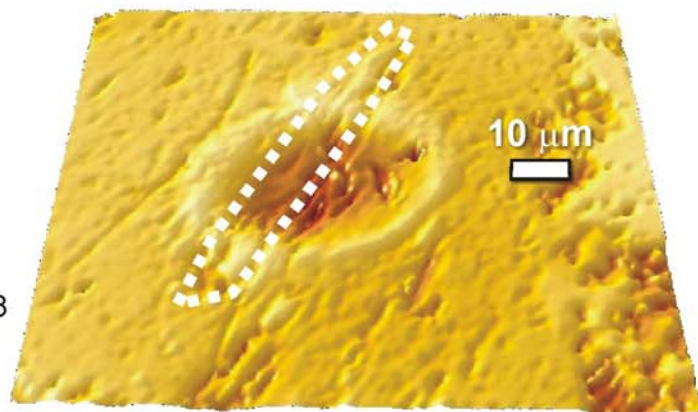
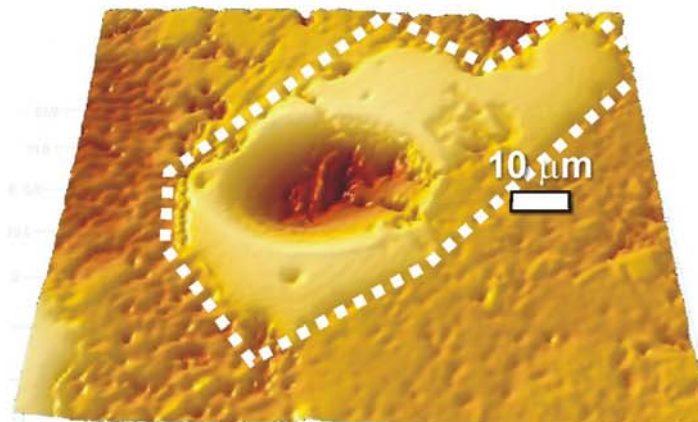
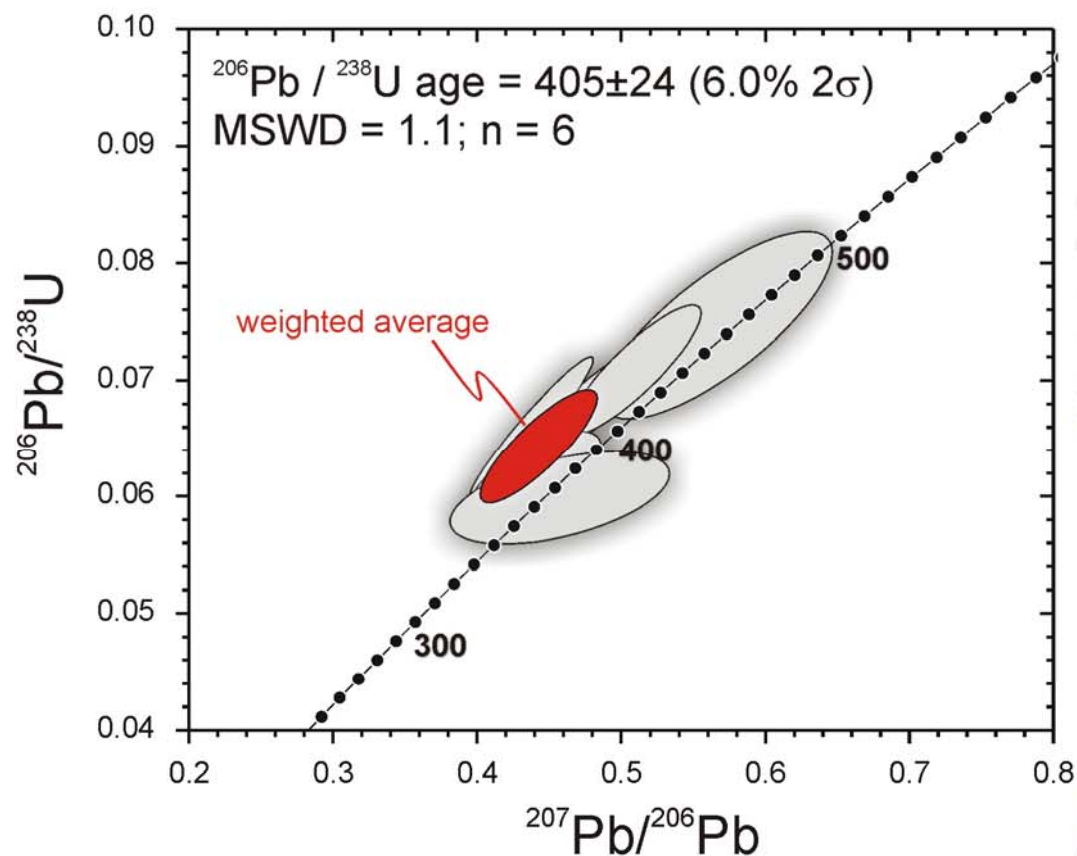
- Multicollection analysis of Phalaborwa baddeleyite (30 nA $^{16}\text{O}^-$ beam)
- U-Th relative sensitivity calibration comparable to zircon
- Yields equilibrium $(^{230}\text{Th})/(^{238}\text{U})$
- Potential for young mafic volcanics (e.g., Hawaii, Eifel)

Micro-baddeleyite: analytical parameters

- Pb useful yield ~0.4 % (similar to zircon)
- for 100 Ma baddeleyite, 500 ppm U, $^{206}\text{Pb}/^{238}\text{U}$ counting error of 1% requires $\sim 25\ \mu\text{m}^3$ baddeleyite
- In-situ thin-section analysis
- use field aperture to exclude common Pb from crater margins and matrix minerals



Micro-baddeleyite: preliminary results

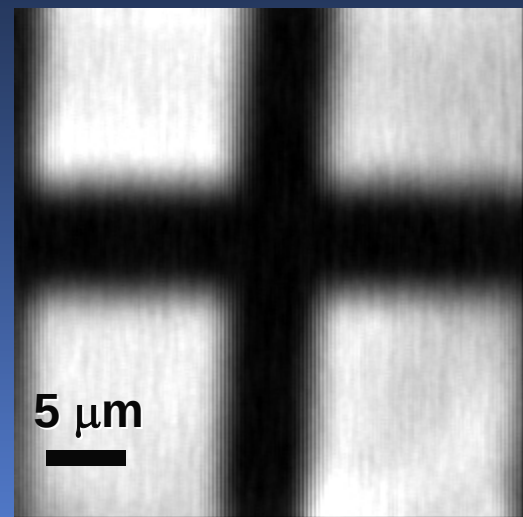


- baddeleyite from Sette Daban (N Siberia)
- mafic sills within carbonate platform sequence
- analyzed grains between 30 and 10 μm (short dimension), depth resolution $\sim 1\text{--}2 \mu\text{m}$

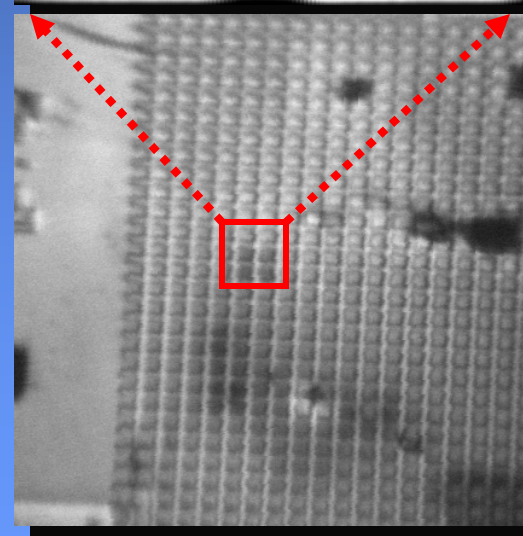
SIMS micro-baddeleyite geochronology: future developments

- more testing of crystal-orientation effects on Pb-U sensitivity (EBSD analysis underway)
- multi-collection of Pb isotopes to increase duty cycle
- implement sample maps (e.g., by SEM) into sample stage control
- explore potential of ion-beam generated secondary electron imaging (UCLA ims1270 features Ga liquid metal source, and new channeltron particle detector)

Secondary
electrons



Reflected
light



scanning electron images of Ta-Si grid, 2 pA Cs⁺ (1.5 μm lateral resolution)

Conclusions

- SIMS U-Pb dating of baddeleyite possible at ~3-4% (rel. uncertainty) level
- crystal-orientation effects under investigation, but appear to be minor/absent (O₂-flooding? secondary ion extraction geometry?)
- U-Pb ages accurate within internal reproducibility for randomly oriented crystals (e.g., in-situ baddeleyite in thin-section)
- U-Th calibration comparable to zircon
- potential for U-series dating of young mafic volcanics