

# EarthScope Student Geochronology Research and Training Program Laboratory

## Overview

### University of Texas – UTChron (U-Th)/He and LA-SS-ICP-MS U-Pb Laboratory 03/11/16

#### Lab Description

The UTChron Geo- and Thermochronometry Laboratory at the Department of Geological Sciences of the Jackson School of Geosciences at the University of Texas includes both laboratory facilities for (U-Th)/He and  $^4\text{He}/^3\text{He}$  thermochronometry as well as for LA-ICP-MS geochronology and petrochronology. Training modules for Earthscope Geochronology Graduate Student Award graduate students will consist of theoretical lectures on methodology, hands-on analytical training, and data reduction and interpretation in one-on-one sessions. The laboratories house facilities for mineral separation, sample preparation, He noble gas, and solution and laser ablation ICP-MS analyses.

#### *(U-Th)/He and $^4\text{He}/^3\text{He}$ Thermochronology Laboratory*

The UTChron (U-Th)/He laboratory houses three state-of-the-art, all metal, ultra-high vacuum noble gas extraction and purification lines for measuring  $^4\text{He}$ . Two of the lines are used for total-fusion (U-Th)/He age dating and are equipped with PhotonMachines diode lasers, while a third line is employed in step-heating He diffusion kinetics experiments and equipped with four computer-controlled and automated light-bulb furnaces. UTChron also houses a Thermo Helix SFT magnetic sector mass-spectrometer with dedicated UHV extraction and purification line. The line is equipped with a diode laser for laser heating and two light-bulb furnaces for low-temperature step heating for  $^4\text{He}/^3\text{He}$  thermochronometry and a second dedicated PhotonMachine Analyte G.2 Excimer Laser Ablation system for laser-ablation He dating. The UTChron (U-Th)/He laboratory also contains a dedicated chemistry laboratory with 3 low-flow fume hoods, two laminar-flow workstations, acid distillation systems, water purification system, and sample drying environments. We use this dedicated facility extensively for sample dissolution following He degassing and sample spiking with various mixed  $^{230}\text{Th}$ - $^{235}\text{U}$ - $^{149}\text{Sm}$ -REE spike for ICP-MS based isotope dilution analysis of U, Th, Sm, and REEs using an Element2 HR-ICP-MS equipped with an ESI SC-E2 DX autosampler system and CETAC micronebulizer with 50 microliters/minute sample uptake to accommodate fast and automated small-volume sample analysis for U, Th, Sm for (UTh)/He dating.

#### *LA-SS-ICP-MS U-Pb Geochronology Laboratory*

The UTChron LA-ICP-MS U-Pb geochronology laboratory houses two Element2 HR-ICP-MS instruments. One is dedicated to in-situ laser ablation analysis for both U-Pb age dating and minor and trace-element fingerprinting and thermometry, mainly on detrital samples used for U-Pb-He double dating. The laboratory uses a PhotonMachines Analyte G.2 193nm ArF Excimer laser with a large-volume Helex cell to ablate material. We also routinely conduct split-stream laser ablation ICP-MS analyses on accessory mineral phases, simultaneously acquiring U-Pb and REE/Trace Element data while drilling into grains (depth profiling). This cutting-edge technology allows us to relate age information to petrological and geochemical boundary conditions, such as differentiating between magmatic and metamorphic protoliths, conditions of zircon growth, or metamorphic paragenesis during zircon formation. For data reduction, the laboratory has three dedicated workstations for data reduction and interpretation using Iolite software. Ablation rates and depths are calibrated using a Bruker Contour GT-1K 3D Optical Interferometer for improved down-hole fraction correction and laser ablation pit dimension and volume measurements.

#### Preparations and Expected Time Frame

For apatite and zircon (U-Th)/He analyses students should expect to spend a minimum of two weeks visiting our lab for preparation and analysis of a minimum of 10 samples (3-5 aliquots each). Sample preparation (handpicking, morphometric analysis, wrapping in Pt foil) commonly takes 2-4 hours per sample depending on sample purity and quality as well as skills and experience of visitors. There are 4 Nikon SMZ1000 stereo-microscopes available for picking of zircon, apatite, rutile, titanite, etc. Students are trained in all steps of the analytical work and are expected to carry out all analytical tasks under supervision from hand-picking, He analysis, sample dissolution, and ICP-MS based U, Th, and Sm analysis. All data (grain dimensions, He, U, Th, Sm) are uploaded to an online database. Software and online tools allow for management and reduction of data at UTChron or back at home institution. UHV lines for He analysis are fully automated (laser and extraction lines) and while students are expected to check their in-progress analyses, they can continue sample preparation during He analyses. The steps that each visiting student will learn and perform are as follows: (1) Select suitable minerals for analysis and measure grain dimensions for alpha-ejection correction using a binocular microscope, (2) Transfer measured single grains into Pt packets and upload sample name and grain dimensions to UTChron database, (3) Load individual samples (42 unknowns and 2 stds) into sapphire-window sample cell of UHV extraction line, (4) Load sample names from UTChron database and start automated laser heating and He analysis sequence, incl. He stds, blanks, and

samples, (5) Upload He data to UTChron database after completion of He measurements, (6) Retrieve degassed samples after He analysis and dissolve grains in clean lab for U, Th, and Sm ICP-MS analysis (due to university radiation safety regulations, all spiking of samples will be carried out by lab personnel), (7) Analyze U, Th, Sm abundances by solution ID-ICP-MS, (8) Reduce, interpret, and model data.

For zircon, rutile, and apatite U-Pb geochronology by LA-ICP-MS analysis, students should expect to spend a minimum of 3 days visiting our lab for preparation, analysis, and data reduction for 1-4 samples. Sample preparation involved either epoxy mounting and polishing (traditional) or tape-mounting (depth-profiling) for LA-ICP-MS analysis. Students are trained and supervised in grain and laser ablation spot selection (unknowns and standards) and queuing analyses for LA-ICP-MS analysis. Students are instructed and supervised in independent operation of LA-ICP-MS system. Sample sequences are started and run in automated fashion, but need to be checked by students. Software allows for live monitoring of data and data quality and it is the student's responsibility to check instrument vitals and liquid Ar and He carrier gas levels. After analysis, students are trained and supervised in data reduction using Iolite software and data interpretation. The steps that each visiting student will learn and perform are as follows: (1) Make sample mounts either as epoxy mounts or tape mounts on double-sided sticky tape, (2) Polish and CL image grains if desired, (3) Target samples and program laser ablation spots on unknown samples and primary/secondary standards, (4) Record sample mount image mosaics and annotated with sample numbers, (5) Tune and Optimize ICP-MS, (6) Creating and running of sample sequences by LA-ICP-MS analysis, (7) Data export, reduction, standardization, quality control, and interpretation using Iolite software.

### **Analytical Costs**

Students should budget the following for analytical work:

Apatite (U-Th)/He: \$320 per sample (3 aliquots);

Zircon (U-Th)/He: \$350 per sample (3 aliquots);

Apatite  $^4\text{He}/^3\text{He}$ : \$355 per sample (20 steps)

U-Pb LA-ICP-MS: \$7/spot (zircon, apatite, rutile, titanite)

REE/TR LA-ICP-MS: \$7 spot

Typically 30 spots for crystallization and 120 spots for detrital samples)

Prices include all consumables and supplies, use of equipment, training, and preliminary data reduction. Costs do not include mineral separation services. Cost schedule reviewed and approved by the University of Texas

### **Preparation for Visit**

Stockli is happy to work with students on design of best possible sampling strategy prior to field work or sample collection to optimize study success. UTChron is also happy to provide students with key literature prior to laboratory visit. Importantly, students should complete mineral separation prior to coming to UT Austin and mineral separates should be of reasonable purity and quality. Students who do not have access to mineral separation facilities should contact commercial mineral services, such as GeoSep Services (<http://www.geoseps.com/>). Mineral separation is a critical step and needs to be done as carefully as possible to ensure value and quality of analytical results. Mineral separation is time-consuming process and needs to be completed prior to UTChron laboratory scheduling. Note that not all samples or minerals are datable!

### **Laboratory Staff**

The UTChron (U-Th)/He and U-Pb geo- and thermochronometry laboratories are directed by Professor Daniel Stockli and managed by Des Patterson (Helium) and Lisa Stockli (U-Pb LA-ICP-MS). In addition, we have two full-time laboratory technicians (Yomayra Roman and Daniel Arnost) that help with sample preparation and training. Roman and Arnost will work with students on sample preparation and analytical efforts for (U-Th)/He dating, while Lisa Stockli will work with students on all LA-ICP-MS sample preparation and analytical efforts. Stockli will work with students on study design, sample strategy, analytical data quality control, data interpretation, and data modeling as well as one-on-one instruction in methodology and theory.

### **Laboratory Time Scheduling**

Students should contact UTChron Director and/or laboratory managers for scheduling 6-8 weeks prior to laboratory visit.

### **Contacts**

Daniel Stockli – Laboratory Director (STOCKLI@JSG.UTEXAS.EDU)

For more information visit: <http://www.jsg.utexas.edu/he-lab/>