Wyoming Space Grant Consortium
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Proposal title: Dating lunar and Martian meteorites using a new, non-destructive method for dating mafic rocks: in-situ U-Pb SIMS dating of micro-baddeleyite and zircon

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ABSTRACT. The research will apply and refine an innovative method to date mafic extra-terrestrial rocks, using U-Pb, secondary ionization mass spectrometry (SIMS) of micro-baddeleyite (ZrO_2) and zircon ($ZrSiO_4$) crystals measured in thin section. The new method is a major improvement for dating meteorites because 1) sample preparation is relatively simple, 2) there is little to no risk of losing the target minerals during preparation, and 3) the method consumes very little material (20 micron-diameter by 1 micron-deep pits). Current dating methods used for meteorites and lunar samples require painstaking crushing and physical mineral separations with dissolution and consumption of milligrams of sample. The new method has been established with Archean-aged, terrestrial rocks over the past year (Chamberlain et al., 2008) and we anticipate accurate $^{207}Pb/^{206}Pb$ dates from grains as small as 3x5 microns, with precisions as high as 0.2%. This will be the first application to extra-terrestrial samples.

The proposed research is a collaboration between UW, the University of Western Ontario (Canadian node of NASA's Lunar Science Network) and the Royal Ontario Museum, Toronto. Primary targets are lunar granulite meteorite NWA 3163, mare basalt NWA 032 and Martian shergottite DAG 476, which have all been loaned for research by a private collector and are in-hand. Lunar granulites were rare at Apollo sites but may represent a large portion of the mid- to lower lunar crust. Mare basalt NWA032 is chemically distinct from any collected by Apollo, and the age of Martian sample DAG 476 is in dispute. The recovery of precise age data from these rare samples will have direct implications for the magmatic evolutions of the Moon and Mars, timing of the late heavy bombardment of the inner planets, and planning for future manned missions. The project will firmly establish this new method for dating meteorites and returned extra-terrestrial samples.

Project Narrative: Dating lunar and Martian meteorites using a new, non-destructive method for dating mafic rocks: in-situ U-Pb SIMS dating of micro-baddeleyite and zircon.

I. DESCRIPTION OF THE PROPOSED RESEARCH.

Apollo and Luna samples have been critical in understanding lunar evolution and early solar system processes. For logistical and safety concerns, however, those missions were largely restricted to the mare regions, and the samples are dominated by 4.4 to 4.0 billion year old (Ga) mare basalts and anorthosites. Recent studies of meteorites from the moon have revealed that there is a much larger variation in lunar rock compositions (Gaffney and Borg 2008) and that magmatism continued to as recently as 2.86 Ga (Borg et al., 2004).

The proposed research will date lunar meteorites NWA 3163 (a granulitic impactite), NWA 032 (a mare basalt), and Martian meteorite DAG 476 (a shergottitic basalt), which have been loaned to the University of Western Ontario and the Royal Ontario Museum (Toronto) by a private collector. NWA 3163 is an anorthositic breccia that is believed to have been metamorphosd 10's of km deep within the Moon's crust (Irving et al., 2006). Deformation of the rock occurred much earlier in lunar history than the impact event that ejected it from the Moon (Irving et al., 2006). We aim to determine the magmatic age of the protolith from in-situ dates of micro-baddeleyite (e.g. Chamberlain et al., 2007, 2008) or zircon, and the timing of high temperature strain using zircon strain chronometry (Moser et al., 2009). Both of these techniques are innovative and non-destructive making them attractive to donors of this rare material. NWA 032 is geochemically and petrologically distinct from any Apollo or Luna sample and probably originated in a previously unsampled mare deposit (Fagan et al., 2002). It may also be one of the youngest lunar rocks at ~2.8 Ga. Our research will test this age. The ages of DAG 476 and other Martian meteorites are still in dispute, with Pb/Pb whole rock dates of 4.0 Ga (Bouvier et al., 2005) and ages of 0.35 to 0.18 Ga by other chronometers (Gaffney et al., 2007). Our combined application of detailed microstructural analysis and new micro-crystal dating on the U-rich, magmatic mineral, baddeleyite, will clarify this debate. Baddeleyite has been identified in several Martian meteorites including DAG 476 (Herd, 2005). The results of the research will firmly establish the methods for use with rare extra-terrestrial samples, add to our understanding of the early evolutions of the Moon and Mars, and help to constrain the timing of the late heavy bombardment of the inner solar system.

U-Pb radiometric system and Pb/Pb dates. The U-Pb system is unique among radiometric chronometers in that there are two independent decay chains from U to Pb, ^{238}U to ^{206}Pb and ^{235}U to ^{207}Pb. Robust dates can be determined by simply measuring the ^{207}Pb/^{206}Pb values of rocks and minerals and mathematically eliminating U from the age equations. Since uranium does not need to be measured, the ^{207}Pb/^{206}Pb method is extremely powerful as it avoids all potential inter-element analytical bias and preserves the age even if there has been recent chemical disturbance. The Pb/Pb method has been used to date the ages of the Earth, meteorites and the Moon (e.g. Patterson, 1956; Tatsumoto et al., 1973; Tera and Wasserburg 1972) and continues to be useful for high-precision extra-terrestrial dates (e.g. Amelin et al., 2002). Many of the early studies to determine the age of the Earth, Moon and meteorites relied on Pb/Pb data from whole rock dissolutions and had to correct the data for significant amounts of initial, non-radiogenic Pb (e.g. Tatsumoto et al., 1973; Tera and Wasserburg 1972). By targeting U-rich baddeleyite and zircon, new age determinations by the in-situ SIMS method will have much lower dependencies on the choices of initial Pb isotopic compositions and will potentially yield more accurate, higher-precision Pb/Pb dates.

Other radiometric systems have been used to date lunar rocks, including Sm-Nd, Rb-Sr, Ar-Ar, Re-Os and Lu-Hf (i.e. Aeschlimann et al. 1982; Carlson and Lugmair 1981; Nyquist et al. 1981, 2008; Edmunson et al., 2009). These methods are generally reliable although they can be affected by metamorphism and alteration, especially Rb-Sr and Ar-Ar. Each of these methods requires painstaking sample preparation and the destruction of milligrams of sample by dissolution, however. By contrast, preparation of a polished thin section for the in-situ method is straightforward, with little risk of lost sample, and the SIMS analysis only consumes ~4x10^{-7} mg (three to four 20 by 1 micron pits). Even the analyzed grains are largely preserved, as they are typically several microns thick in thin section.

Furthermore, the U-Pb method is the gold standard for geochronology as the uranium decay rates are widely considered to be the best determined (e.g. Schoene et al., 2006).

Baddeleyite U-Pb radiometric dating method. Baddeleyite (ZrO_2) has been a prime target for dating mafic rocks (e.g. Heaman and LeCheminant 1993), as it is a principal U-bearing mineral phase, it excludes Pb from its crystal structure initially, but retains radiogenic Pb well. It was first dated in 1972, from a lunar sample, using a low-resolution ion microprobe (Anderson and Hinthorne, 1972). U-Pb data from baddeleyite tend to be nearly concordant with only 1 to 2% Pb loss. Xenocrystic baddeleyite has been documented in only a few rare instances (e.g. Schärer et al., 1997), and the uranium concentrations are typically high (500 to several 1000 ppm, Heaman and LeCheminant, 1993), so analysis of a few grains of 100-200 micron long baddeleyite by dissolution and thermal ionization mass spectrometry (ID-TIMS) often leads to highly precise (0.1% or better), concordant dates on mafic magmatism.

Micro-baddeleyite crystals (<40 microns) are abundant in a wide variety of mafic rocks including lunar samples and anorthosites. Specialized concentration techniques have been developed to recover these micro-baddeleyite needles (e.g. Söderlund and Johansson, 2002) and high-precision U-Pb dates are possible by dissolution and ID-TIMS analysis of multiple grains (LeCheminant et al, 2007), but the in-situ method employed by the proposed research will avoid these difficult separations, lead to rapid, reliable dates and preserve much more of the sample, which then can be analyzed for other invaluable data such as oxygen isotopic values that can be tied directly to the ages (e.g. Valley et al., 2002). We have been able to analyze grains as small as 3x5 microns by the in-situ technique, much smaller than can be physically separated for dissolution.

Zircon strain chronometry. Magmatic zircon ($ZrSiO_4$) can also be found in anorthositic rocks, often co-existing with baddeleyite (Scoates and Chamberlain 1995, 1997, 2003). It is uranium-rich, with little initial Pb and retains radiogenic Pb. Zircon has been extensively dated by both ID-TIMS and SIMS methods as it occurs in a wide variety of rock types.

The zircon strain chronometer employs cutting-edge diffraction techniques (electron backscatter diffraction, EBSD, and microbeam X-ray diffractometry, μXRD) to measure crystal lattice-strain in zircon formed during high temperature crystal-plastic flow sometimes triggered by bolide impacts (Moser et al., 2009). The strain creates high diffusivity pathways that permit out-diffusion of radiogenic Pb and resets the U-Pb system in zircon. The timing of strain can be determined by SIMS spot analyses of the most highly-strained zircon domains. The magmatic age of the rock can often be determined as well, as the impact strain is typically inhomogeneous and some zircon inclusions can be armored by their host minerals.

In-situ dating method and attributes of the Cameca SIMS. The in-situ dating methods for both micro-baddeleyite and zircon start with polished thin sections (thin wafers of rock that are glued to glass slides, then cut and polished to a thickness of 30 microns). We will employ NASA protocols for meteorite sample preparation, and preserve all dust. The University of Western Ontario has all the necessary, approved equipment for thin section preparation of meteorites.

Zirconium abundances will be mapped in the thin sections by x-ray scanning on an electron microprobe (EMP) at UW, and then the baddeleyite grains will be further identified and imaged in back-scattered electron mode (BSE). At the UCLA SIMS lab, the target baddeleyite and zircon grains will be left in thin section and blasted by an oxygen ion beam that excavates a 20 micron-diameter by 1 micron-deep crater. Secondary ions generated by the excavation will be measured by a high-resolution mass spectrometer to determine Pb isotopic compositions and U-Pb abundances. The SIMS instrument is calibrated against baddeleyite standards of known ages and elemental compositions, alternately analyzed between unknowns.

The Cameca high-resolution SIMS instrument (*ims*1270) has several advantages over other brands, specifically the SHRIMP instruments. The ion optics of the Cameca instruments preserve the spatial relationships of the ion pit. Consequently, secondary ions from host mineral phases can be filtered out by an aperture in the flight tube, so even though the primary ion beam and sampling pit are 20 microns in diameter, the effective analytical area can be much less, a few microns (Figure 1). In addition, the Cameca instrument doesn't seem to suffer as much crystal-orientation bias in U/Pb fractionation as SHRIMP instruments during baddeleyite analyses (Wingate and Compston, 2000). Preliminary results show much less dispersion from the Cameca instrument compared to SHRIMP instruments (Figure 2). We will rely on $^{207}Pb/^{206}Pb$ dates for the proposed research, however, and there does not appear to be any Pb fractionation by SIMS analysis from either instrument (Wingate et al., 1998).

Based on our results from terrestrial rocks (Table 1), we anticipate that the Cameca SIMS instrument will produce $^{207}Pb/^{206}Pb$ weighted mean baddeleyite and zircon dates with precisions of 0.2% or better from extra-terrestrial rocks. Precisions on existing Pb/Pb dates on lunar and meteoritic samples range from 0.1% to 1% (e.g. Tera and Wasserburg, 1972; Tatsumoto et al., 1973; Jacobsen and Wasserburg, 1984; Carlson and Lugmair, 1988; Premo and Tatsumoto, 1992; Alibert et al., 1994), although some higher-precision dates have been reported recently (0.02%, Amelin et al., 2002).

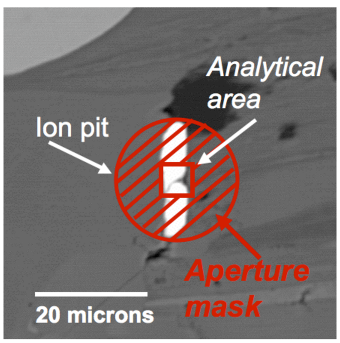

Figure 1. Backscatter image of micro-baddeleyite grain (bright) in thin section with sampling geometry of Cameca SIMS.

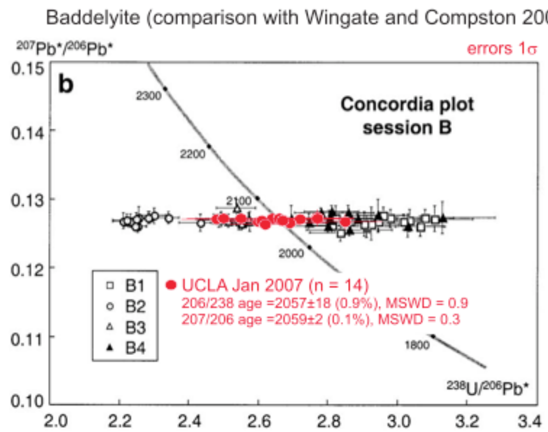

Figure 2. Concordia plot of data from SIMS analyses of Phalaborwa baddeleyite standard. Numbers on curve are ages in million years before present. SHRIMP data in black show more dispersion in $^{238}U/^{206}Pb$ (x-axis) than Cameca data (red-filled circles), but both have high precision $^{207}Pb/^{206}Pb$ dates (y-axis).

Table 1: Preliminary results from Cameca SIMS U-Pb dating of baddeleyite

Standards (n)	207Pb/206Pb Date in Ma	S.D.	206Pb/238U Date in Ma	S.D.	ID-TIMS Date (Ma)	ref
Phalaborwa:						
Jan. 2007 (14)	2059±2 (0.1%)	4 (0.2%)	2057±18 (0.9%)	83 (4%)	2060±0.5	1
Jan. 2008 (16)	2061±3 (0.2%)	11 (0.5%)	2043±43 (1.6%)	59 (2%)		
FC-4b (16)	1088±13(1.2%)	20 (1.0%)	1108±26 (2.6%)	45 (4%)	1099±0.2	2
Kovdor (16)	379±43 (11%)	101 (27%)	377.5±9.2 (2.4%)	16 (4%)	380±4	3
In-situ samples (n)						
07BGH02 (4)	2690±5 (0.2%)				none	
IP0763 (6)	1540±30 (1.9%)			none		
X705 (4)	1374±10 (0.75%)			none		
FC-4b (27)	1097±3 (0.3%)		1113±36 (3%)		1099±0.2	2
X04-29 (2)			451±12 (2.7%)		450±24	4

Ref: (1) Heaman, pers. comm. 2007, (2) Schmitz et al. 2003, (3) Bayanova et al. 1997, (4) Khudoley et al. 2001

II. RELATIONSHIP TO SPACE GRANT AND NASA GOALS. Although the proposed research is not a space-based technique, it broadly addresses five Strategic Objectives outlined in NASA's Direction for 2005 and Beyond. Strategic Objective 5 is to explore the universe to understand its origin, structure, evolution and destiny. The new dating technique will lead to a better understanding of the evolution of the Moon and early solar system through more precise chronology of lunar and Martian meteorites and timing of major impacts. Strategic Objectives 9 and 10 are extended human expeditions to the Moon and Mars. Precise dates of returned samples are a necessary scientific component to human expeditions. Baddeleyite has already been identified in several lunar samples from the Apollo project (Andersen and Hinthorne, 1972; Lovering and Wark, 1971; Smith and Steele, 1976) and Martian meteorites (Herd 2005). Objective 13 is to inspire and motivate the Nation's students and teachers. Evolution of the solar system and early Earth are first order scientific questions. Objective 14 is to advance the scientific knowledge of the Earth system. Dating major bolide impacts on the Moon has direct implications for the early evolution of the Earth as both bodies were undoubtedly struck with similar frequencies. Subsequent geologic processes on the Earth have largely removed the evidence for those early terrestrial impacts.

III. PRODUCTS. The proposal will cover the costs of preparation and material properties analysis and imaging of several thin sections from lunar meteorites NWA 3163, NWA 032 and Martian meteorite DAG 467, which are sourced from a formally typed meteorite collection. It will also fund 2 days of analytical time at the UCLA SIMS lab and associated travel. We expect to determine both the magmatic age and timing of major impact from NWA 3163, and magmatic ages from the other two. These results will lead directly to manuscripts and presentations.

The in-situ micro-baddeleyite dating method has generated considerable international interest. The PI presented preliminary results in an invited keynote Pardee talk at the 2007 annual GSA meeting (Chamberlain et al., 2007), and at the 2008 International Geochemistry conference in Vancouver, Canada (Goldschmidt 2008, Chamberlain et al., 2008). Two manuscripts on the method are in preparation. The results from the proposed research will strengthen future proposals to NSF and NASA to date extra-terrestrial basaltic rocks with implications for planetary and solar system evolution. There are also significant terrestrial applications of the micro-baddeleyite dating method including tests of supercontinent reconstructions and the evolution of plate tectonics in the early Earth, and evaluations of the evolution of the Earth's magnetic geodynamo. High-profile results, such as those from dating meteorites, will strengthen proposals to apply the method to Earth history as well.

IV. INVESTIGATORS AND TIMELINE. Dr. Kevin R. Chamberlain, Research Professor at the University of Wyoming, has vast experience dating both baddeleyite and zircon from anorthositic and basaltic rocks including those from the Laramie and Horse Creek Anorthosite complexes, WY and Buehls Butte, ID (Scoates and Chamberlain, 1995, 1997, 2003; Doughty and Chamberlain 2007). He has recently developed the in-situ micro-baddeleyite method for dating mafic rocks in collaboration with Dr. Susan Swapp, Senior Research Scientist at UW, and Dr. Axel K. Schmitt, Assistant Adjunct Professor at UCLA (Chamberlain et al., 2007, 2008).

Dr. Desmond Moser, Assistant Professor at the University of Western Ontario, also has vast experience with U-Pb geochronology and recently developed a zircon strain chronometer for dating impact events (Moser et al., 2009). By combining micro-structural techniques (electron backscatter diffraction, EBSD, and microbeam X-ray diffractometry, μXRD) with ID-TIMS and SIMS U-Pb geochronology, he was able to establish that the lower crust (30-40 km deep) of South Africa underwent regional, high temperature flow in response to the 2.02 Ga Vredefort impact. The techniques are non-destructive and can be applied to meteorites such as NWA 3163. A University of Western Ontario graduate student will also be involved in the project as will staff scientists from the Royal Ontario Museum in Toronto, one of whom is co-advising the graduate student.

Project will start in late spring 2009 and continue through the summer. Thin sections will be prepared at Univ. of Western Ontario using NASA-approved equipment. X-ray mapping, BSE, CL and EBSD imaging will be performed at UW and UWO, μXRD imaging will be done at Western Ontario in an ongoing collaboration between Moser and UWO XRD specialist Dr. R. Flemming. SIMS analyses at UCLA will be scheduled as soon as grain location and imaging is complete. Chamberlain, Moser and the University of Western Ontario graduate student will travel to UCLA for the SIMS dating, hopefully late summer or early fall 2009. The UCLA lab is in high demand as there are only 2 Cameca SIMS labs in North America (UCLA and University of Wisconsin), a third in development at the University of Alberta, and approximately 20 worldwide. Chamberlain has a long-standing working relationship with the UCLA lab, however, and we anticipate scheduling in a timely fashion.

Publications describing the results will be submitted in the fall of 2009. Follow-up proposals to NSF and NASA are likely in fall 2009 as well.

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SUMMARY PROJECT BUDGET

Project Title: Dating lunar and Martian meteorites using a new, non-destructive method for dating mafic rocks: in-situ U-Pb SIMS dating of micro-baddeleyite and zircon.

Applicant: Kevin R. Chamberlain, Research Professor, Dept. Geology and Geophysics, UW

			2. PROPOSED COST SHARE	
		1. Request	Non-Federal Cost-share	Federal Cost Share
3.Labor:				
Faculty/Applicant Labor- Salary		$8742	0	$5758
	Benefits (38%)	$5358	0	$3529
Univ. West. Ont. grad student		0	$5000	0
Univ. West. Ont. Faculty-Salary		0	$6000	0
4.Other costs:				
Travel	analytical trip to UCLA	$1300	$1200	0
Other	EMP mapping & imaging	$1200	0	$600
Other	EBSD imaging	$600	0	0
Other	µXRD & thin sect prep	0	$600	0
Other	SIMS use @$1400/day	$2800	0	0
5. TOTALS		$20,000	$12,800	$9,887

SUMMARY PROJECT BUDGET NARRATIVE

Labor: It is anticipated that the project will require at least 3 months of Applicant Kevin Chamberlain's time. He will be responsible for all aspects of the project, including imaging at UW and SIMS analysis at UCLA. The project will also require several months from a University of Western Ontario graduate student and their adviser, Desmond Moser. These costs are part of the non-Federal cost sharing.

Other costs: *Travel*. Project requires a 4-day trip to UCLA for final sample preparation and 2 days of SIMS analyses. Requested funds are estimated for Chamberlain's airfare, and lodging and food for Chamberlain and the two Canadians while in Los Angeles. Airfare and foreign travel expenses for the Canadians are listed as non-Federal cost share.

Analytical costs. Project requires x-ray, back-scattered electron and electron back-scattered diffraction imaging at UW with associated instrument use fees ($30 per hour inhouse). In our experience, the x-ray and BSE mapping costs are about $300 per thin section. We have budgeted for 6 sections. Thin section preparation and µXRD imaging will be performed at University of Western Ontario and are included as non-Federal cost sharing. X-ray mapping, color CL imaging and EBSD work may also be performed at the University of Western Ontario depending on how quickly their new FEG SEM is installed. It is scheduled for late summer. UCLA SIMS lab charges $1400 per day; 2 days are budgeted. Each spot requires nearly 1 hour of SIMS time, we anticipate 12 spots per sample for the micro-baddeleyite dates and 12 spots for the zircon strain chronometry work on NWA 3163.

Cost sharing: All of the Canadian costs are listed as non-Federal cost shares. These include salaries for the Canadian collaborator, Desmond Moser, plus his graduate student, airfare from Canada to UCLA, and costs of thin section preparation and μXRD imaging at Western Ontario. To keep the requested budget to $20,000, approximately one month of Chamberlain's salary and some of the analytical costs are listed as a Federal cost share. Chamberlain has an NSF proposal in final review with the Division of Instrumentation and Facilities to develop and refine the in-situ micro-baddeleyite dating method using well-dated terrestrial baddeleyite samples. Although the IF proposal is specifically limited to analyses of previously dated samples, one month's salary from that grant will be used on this project to refine the method for meteorite dating. If Chamberlain's NSF-IF grant is not funded, his additional salary will come from other sources.

5. Previous Space Grant funding: None