Proc Indian Natn Sci Acad **86** No. 1 March 2020 pp. 643-650

DOI: 10.16943/ptinsa/2020/49823

Status Report 2016-2019

New Initiatives to Bolster Analytical Facilities in India for *in situ* U-Th-Pb Geochronology, Hf and O Isotope Systematics in Zircon: A Focus on Laboratories at the IUAC,WIHG and CSIR-NGRI

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(Received on 25 September 2019; Accepted on 15 October 2019)

Introduction

Zircon U-Pb Geochronology

Over the last several decades, Geoscience research witnessed a paradigm shift from being an observational science to a more rigorous, multi-disciplinary science with an emphasis on quantification. Understanding the processes of crust-formation and crust-evolution through geologic time is fundamental to many issues in Earth System Science. In the area of geochronology and radiogenic isotope research, a very significant advancement concerns development of technologies and analytical methodologies for *in situ* measurement of elemental and isotopic compositions at high precision and spatial resolutions typically of the order of a micrometer directly in selected minerals (Compston, 1984; Ireland and Williams, 2003). Zircon ($ZrSiO_4$), a common accessory phase in rocks, is one of the most versatile minerals known to earth scientists. Presently, zircon studies and applications span an entire sub-discipline, *zirconology*, encompassing many aspects of Earth, Planetary, Environmental and Material sciences, most importantly, Geochronology and in tracing crustal processes involving interactions between Earth's mantle, crust, the hydrosphere and biosphere. In the studies on the formation and evolution of the earth's crust and lithosphere, zircon has been a preferred tool for precise age determinations through the time-honored U-Pb method (Faure, 1986; Davies *et al*., 2003; Dickin, 2005). Its application as a sensitive probe into Earth processes is based mainly on trace element, Hf, and O- isotopic compositions (Valley, 2003; Kinny and Mass, 2003; Bhaskar Rao, 2008; Condie and Aster, 2010; Belousova *et al*., 2010; Cawood *et al*., 2013; Griffin *et al*., 2014; Roberts and Spencer, 2015; Hawkesworth *et al*., 2017; Iizuka *et al*., 2017). In common rocks, zircon ranges in size from about 20 to 200μm, while very small zircons, < 20μm, may be present in volcanic rocks, some plutonic rocks and mudstones. In general, zircon is highly variable both in terms of external morphology and internal textures (Corfu *et al*., 2003). Commonly, these features reflect the geologic history of the mineral as well as its host rock in terms of the episode(s) of magmatic and metamorphic crystallization and/or recrystallization, effects of strain both due to external forces and internal factors such as volume changes caused by metamictization and chemical alteration. Commonly, zircon consists of distinct domains, which can be individually related to periods of zircon formation, recrystallization or consumption, often evident in internal textures revealed in imagery based on optical microscopy, X-Ray or electron scattering techniques (e.g., Corfu *et al*., 2003). The best resolution of internal textures is by cathodoluminescence (CL) or backscattered electron (BSE) imaging using CL-SEM and EPMA techniques, which has been widely applied

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over the last thirty years.U-Pb zircon geochronology (Faure, 1986; Davies *et al*., 2003; Dickin, 2005) is based on three distinct radioactive decay series involving the parent isotopes ^{238}U, ^{235}U and ^{232}Th, which produce their final daughter products - stable isotopes of ^{206}Pb, ^{207}Pb and ^{208}Pb respectively. Depending upon the geologic history, zircons may yield *concordant* or *discordant* U-Pb ages, that can be graphically visualized by plotting $^{207}Pb/^{235}U$ vs. $^{206}Pb/^{238}U$ ratios (U-Pb *concordia diagram*, Wetherill, 1956) and/or $^{238}U/^{206}Pb$ vs. $^{207}Pb/^{206}Pb$ ratios (Tera and Wasserburg, 1972). U-Pb zircon dating has been traditionally conducted by ID-TIMS (Krogh, 1982a, b; Parrish, 1987; Parrish and Noble, 2003) which undoubtedly yields the most precise and accurate ages, but the overall technique is cumbersome, destroys the sample, requires stringent clean chemical laboratories with Pb-blanks <10 pico-gram(pg) for a meaningful single-grain zircon dating. Most importantly, the TIMS technique is found to be inadequate for zircons with complex internal structures representing complex growth histories. For these reasons, zircon geochronology using *in situ* sampling techniques at high spatial resolution (~25µm) in individual crystals have been preferred. Indeed, laser spot sizes of ~ 10µm are now routinely possible with optimized analytical conditions (Mukherjee *et al*., 2019). Instruments such as Secondary Ion Mass Spectrometers (SIMS) and/or Laser Ablation (LA) devices connected to Quadruple Inductively Coupled Plasma Mass Spectrometry (Q-ICPMS) or Magnetic Sector Single Collector (SC) or Multi collector (MC) ICPMS instruments have been widely utilized *in situ* zircon geochronology to overcome the disadvantages of the TIMS U-Pb zircon techniques (Ireland and Williams, 2003; Kosler and Sylvester, 2003). Such techniques are being extensively deployed even for dating detrital and xenocrystic zircon grains, where the precursor host rocks are modified or nonexistent. A good example of application of detrital zircons relates to understanding the nature of Earth's primodial crust during the Hadean Eon (>4.0 Ga) where no rock record is preserved (Wilde *et al*., 2001; Harrison *et al*., 2008; Kemp *et al*., 2010; Iizuka *et al*., 2017; Sreenivas *et al*., 2019 references therein).

Facilities for Zircon Geochronology in India

With an extensively developed Precambrian and Phanerozoic terrains in the Indian subcontinent and thegrowing demand for laboratory facilities for *in situ* zircon geochronology, many Earth Science institutions in India established/augmented LA-Q-ICPMS and LA-MC-ICPMS facilities for zircon geochronology over the last decade. Some examples include CSIR-National Geophysical Research Institute (CSIR-NGRI), Hyderabad; Geological Survey of India (GSI), Kolkatta; Indian Institute of Technology (IIT) Kharagpur, Roorkee; Pondicherry University; Wadia Institute of Himalayan Geology (WIHG), Dehra Dun; Indian Institute of Science Engineering Research (IISER) Bhopal, Kolkatta; National Centre for Earth Science Studies (NCESS), Thiruvananthapuram. A very significant development in this regard is the establishment of the *National Geochronology Facility* (NGF) at the Inter-University Accelerator Centre (IUAC), New Delhi (formerly known as Nuclear Science Centre) with the generous funding from Ministry of Earth Sciences (MoES), Govt. of India. Notably, this centre is being equipped with state-of-the-art instruments including the Femtosecond Laser Ablation system connected to MC-ICPMS and Q-ICPMS and India's first large geometry High-Resolution Secondary Ion Mass Spectrometer (HR-SIMS).

This article presents an overview of the analytical facilities at the IUAC, WIHG and CSIR-NGRI, where the existing facilities are being augmented with new LA and Q-ICPMS instruments.

Inter-University Accelerator Center (IUAC) – National Geochronology Facility (NGF)

The IUAC is a national facility established under the aegis of the University Grants Commission (UGC). The centre is well known for ion accelerators facilitating wide-ranging low to medium energy (keV-MeV) ion-beam based experimental studies. In the past, the Earth Science fraternity has been greatly benefited from these accelerator facilities (Kumar *et al*., 2011, 2015), and further by utilizing 500 kV tandem ion accelerator-based Mass Spectrometer (AMS) (Sharma *et al*., 2018) primarily for precise measurements of cosmogenic radionuclides ^{14}C, ^{10}Be, ^{26}Al and associated isotopic ratio measurements and dating that has important Geological and Archaeological application. The main applications of ^{14}C, ^{10}Be, ^{26}Al dating include Quaternary lake and fluvial sediment studies (Lone *et al*., 2019; Singh *et* *al*., 2019) for tectonic and paleo-climates (Saini *et al*., 2019), erosion/denudation (Dhal *et al*., 2018) and exhumation history of the Himalayan mountain belt (Meenakshi *et al*., 2018), Paleo-seismology (Rajendran *et al*., 2018) and historic/prehistoric archaeological sites (Vahia *et al*., 2016). An additional AMS for medium and heavy mass radioisotopes (^{36}Cl, ^{41}Ca, ^{53}Mn, ^{59}Ni, ^{60}Fe, ^{93}Zr etc.) based on 6 MeV tandem ion accelerator is also planned for the near future. The IUAC-NGF is now being equipped with instruments and analytical protocols for geochronology and radiogenic isotope research involving parent-daughter decay systems with long half-lives 10^8 to 10^{11} y such as Rb-Sr, Sm-Nd, Pb-Pb and Lu-Hf and most importantly *in situ* zircon U-Pb geochronology with analysis of Hf and O isotope systematics. Several of the instruments have been installed, and the process of standardization is in progress. Major analytical facilities being organized at this centre include:

1. A Ti-sapphire crystal Femtosecond Laser Ablation system (Model: Excite Pharos, Teledyne CETAC Technologies, USA) connected to high-resolution magnetic sector field double-focusing ICPMS system -HR-ICPMS (Model: Thermo Fisher Scientific – Element XR) for *in situ* solid sample microanalysis. The laser system can be configured to 1028 nm, 257 nm or 206 nm wavelengths that is capable of delivering an energy density of 10 J/cm^2 (at 1028nm), 5 J/cm^2 (at 257nm) and 3 J/cm^2 (at 206nm) on sample surface making it suitable for wider applications. It is equipped with the class-leading HelEx II Active 2-Volume Ablation Cell.

2. Quadrupole-ICPMS (Model: iCAPQ, ThermoFisher Scientific): for multi-element trace or ultra-trace element analysis. This system shall also be interfaced with the LA system for rapid U-Pb zircon dating.

3. High Resolution Secondary Ion Mass Spectrometer (HR-SIMS, Model: IMS-1300 HR^3, CAMECA, France): capable of the high spatial resolution of the order of few microns for determination of ages and isotopic compositions of zircon and several other mineral grains. The machine is expected to be operational by early 2020. The machine has the highest possible salient features enabling excellent zircon age precision of ~0.3% at 10 µm beam size in 91500 zircon standard. The machine is equipped with two ion sources (Cs and O) and ten detectors (3 fixed in mono collection and seven on five movable trolleys).

4. Basic equipment for geochemical analysis, mineral characterization, and imaging such as WD-XRF, XRD, FE-SEM.

5. Facilities for sample pulverizing and heavy mineral extraction.

The geochronology facility at IUAC aims to facilitate researchers from Indian universities and research institutions to take-up challenging geoscience problems with easy access to a range of high-end instrumentation for geochronology, geochemical and isotopic research together with the sample preparation and imaging facilities.

Wadia Institute of Himalayan Geology (WIHG)

A laboratory dedicated to *in situ* zircon geochronology and Hf-isotope analysis has been set up at the Wadia Institute of Himalayan Geology (WIHG) during the last few years comprising LA-MC-ICPMS-Q-ICPMS. Basic analytical protocols and performance information is available in Mukherjee *et al*. (2017). A mixed collector configuration of MC-ICPMS is used for zircon geochronology. All Pb and Hg isotopes are measured on the Ion counters and ^{232}Th and ^{238}U are measured on faraday cups. Optimized laser spot sizes for zircon geochronology is 20 µm. Zircon standard 91500 is being used as a primary standard for instrumental drift, isotopic fractionation and mass bias correction and Plešovice is being used as a secondary standard for quality control. The laboratory has been utilised for research projects on understanding complex geological problems of the Himalaya and in studies addressing 1) sediment routing of Paleo-Floods (U-Pb Zircon Geochronology: as a provenance indicator) along the major rivers in Ladakh and Zanskar region, 2) Weathering Congruency and provenance of Teesta River basin, Indus River and other important river systems in The Himalaya using Li & Sr Isotopes, and 3) Zircon Geochronology of many gap regions in the Himalaya along Alaknanda-Dhauli Ganga valley, Kali river valley, Beas-Bhaga valley. Some significant results are summarized below:

New age constraints on the chronology of Ladakh migmatites (Sen *et al*., 2018a), granites and granite gneisses in the vicinity of Karakoram Fault Zone (Pundir *et al*., 2017) and age of rhyolites of the Khardung formation (Lakhan *et al*., 2019) in the Ladakh Himalaya. Based on U-Pb zircon age populations in fluvial deposits of Zhanskar River, Chahal *et al*. (2019) established a Pleistocene history of provenance and channel connectivity in the catchment. The intrusive Chor granite in the Paleoproterozoic Lesser Himalayan crystallines of the Jutogh group, Himachal Himalaya, has been dated between 806-1070 Ma (Singh *et al*., 2017). In Uttarakhand Himalaya, Mukherjee *et al*. (2017, 2019) and Jain *et al*. (*in press*) suggested a comprehensive model describing different terrains and litho tectonic units across Uttarakhand Himalaya based chiefly on zircon ages along with Sr and Nd isotopic constraints. The Inner Lesser Himalayan Berinag Quartzite and the Munsiari (MCT zone) mylonitic orthogneisses represent a Paleoproterozoic (1.89-1.95 Ga) magmatic arc-system about the Columbia Assembly.

In contrast, the Great Himalayan Sequence (GHS) is marked by the presence of younger Neoproterozoic zircons (1.05-0.85 Ga) with intrusive Palaeozoic granites during the Rodinia configuration. The overlying Tethyan Sequence is characterized by the first appearance of Early Palaeozoic (0.570-0.485 Ga) detrital zircons in this sequence. The augen gneisses of Munsiari Formation and granite gneisses of the Chiplakot crystalline of the Kumaun Himalaya of Kali River Valley were studied in the context of age and tectonic setting (Phukon *et al*., 2018). The study indicated Paleoproterozoic ages of 1970 and 1860 Ma suggesting that the granitoids, at the northern margin of the subcontinent were part of a continental arc associated with the Columbia supercontinent subduction system. Phukon *et al*. (2018) reported the U-Pb age of metapelite and granitic gneisses of the Higher Himalaya crystalline sequence (HHC) along the Kali river valley. Zircon ages from metapelite in VT ranges from ca. 500 to 2500 Ma with some young rim ages (~21-30 Ma) and for the granitic gneiss, the zircons have crystallization age of 455 Ma with some younger rim ages (~27-32Ma). From these young ages, it was inferred that the high-pressure partial melting of rocks from HHC (Kali Valley) took place in the time span of ~ 21 to 30 Ma, which is responsible for leucosome generation and melt migration towards the upper structural level of HHC. P-T conditions of peak metamorphism are at about ~720°C and ~9.2-9.9 kbar in the sample from a lower structural level.

CSIR-National Geophysical Research Institute

A Geochronology laboratory centered around TIMS was operational since 1986 and LA (Model: 213 Nd-YAG New Wave Research, USA) HR-MC-ICPMS (Model: Nu Plasma HR, Nu Instruments, UK) and Q-ICPMS (Model: Thermo XSeries^{II}, Germany) facility for *in situ* zircon dating and Hf-isotopic analysis was added in 2008 as a CSIR-DST-NIO-NGRI National facility. This laboratory was augmented with a 193nm excimer LA system (Model: Resolution-SE, Australian Scientific Instruments, Australia) in 2017 with project funding from MoES, Govt. of India, New Delhi. Significant results over the last four years from the laboratory are summarised below.

A detailed study including *in situ* U-Pb zircon dating and Hf-isotope analysis of charnockite orthogneisses from the Southern Granulite Terrain (SGT) south India is described in Vijaya Kumar *et al*. (2017). The charnockite orthogneisses from the Madurai, Trivandrum and Nagercoil blocks relate to a minimum of four distinct episodes of felsic magmatism centered around 2.62-2.46 Ga, ca. 2.05-1.84 Ga, ca. 1.0-0.9 Ga, and ca. 0.80-0.76 Ga, about the Siderian, Orosirian, and Tonian Periods. Hafnium isotope analyses of zircon grains from the charnockite gneisses suggest that the protoliths of the ca. 2.05-1.98 Ga gneisses from the Trivandrum and Nagercoil blocks and the ca. 1.0-0.9 Ga gneisses along the southeastern Madurai block involved a significant juvenile magma component, while the protoliths of charnockite gneisses elsewhere in the Madurai block formed mainly through recycling of older crust up to ca. 3.2 Ga. A regional granulite-facies metamorphic imprint during the Ediacaran-Cambrian marked an advanced stage in the amalgamation of the Madurai, Trivandrum, and Nagercoil blocks into the East African orogen and its collision with the Dharwar craton. These results significantly enhanced our understanding of the terrane assembly in southern India and their Gondwanic correlation.

Utilizing the drill cores from the Koyna Deep Drilling project (Gupta *et al*., 2017), the basement gneisses and granitoids beneath the pile of Deccan volcanic rocks at Rasati, borehole KBH-1, were studied to establish age of the basement and its correlation with the cratonic basement exposed around the Deccan volcanic province, India (Bhaskar Rao *et al*., 2017). Zircons from granodiorite and monzogranite samples yield consistent U-Pb ages of 2710±63 Ma and 2700±49 Ma (2s errors). The initial $^{176}Hf/^{177}Hf$ values indicate that the magmatic precursors of the KBH-1 gneisses represent juvenile magmatism around 2700 Ma. The data support the extension of the Eastern Dharwar Craton to the Koyna-Warna region.

Participating in the global quest for Earth's oldest materials to elucidate the planet's old geodynamic regimes, studies on the Singhbhum craton, eastern India indicated its potential as a new repository for Earth's oldest material. This follows from the discovery of rare Eoarchean zircon grains from the craton (Sreenivas *et al*., 2019). The study presented new U-Pb ages and Hf isotopic compositions of detrital zircon grains from ~2.9 Ga old quartzites and magmatic zircon from a 3.505 Ga old dacite from the Iron Ore Group. The detrital zircon grains range in age between 3.95 Ga and 2.91 Ga and the Hf isotope systematics suggest that the Eoarchean detrital zircons represent crust generated by recycling of Hadean felsic crust formed at ~4.3-4.2 Ga and ~3.95 Ga. A prominent shift in Hf isotope compositions at ~3.6-3.5 Ga towards superchondritic values is observed, which signifies an increased role for the depleted mantle and the relevance of plate tectonics. The Paleo-Mesoarchean zircon Hf isotopic record in the craton indicates crust generation involving the role of both depleted and enriched mantle sources. A tectonic model invoking a short-lived suprasubduction setting around ~3.6-3.5 Ga followed by mantle plume activity during the Paleo-Mesoarchean crust formation in the Singhbhum craton was proposed.

The ability to date very young zircon grains to constrain the age of Cu mineralization was evaluated in a study of samples from Chah-Firouzeh porphyry stock Cu-deposit in the Kerman Cenozoic Magmatic Assemblage (KCMA), SE Iran (Mohammaddoost *et al*., 2017). Zircon U–Pb dating of two representative samples from the Chah-Firouzeh porphyry stock yielded emplacement ages of 16.9±0.4 Ma and 16.5±0.2 Ma, respectively. The Early-Middle Miocene epoch marks the most critical period of porphyry Cu mineralization in the KCMA. During this period, many adakitic magmas intruded the Eocene volcano-sedimentary sequences and formed some of the largest porphyry copper deposits in Iran. Consistent with Re-Os dating of Molybdenite from the deposit, the U-Pb zircon ages tightly constrain the age of mineralization within 0.6 million years around 15.9 Ma.

With the creation/augmentation of the state of the art analytical facilities described in the preceding, it is evident that the research in the areas of isotope geochemistry and geochronology in India has entered an exciting phase offering new opportunities to address outstanding problems in Earth sciences.

Acknowledgment

YJB thanks the editors of this volume for their invitation to contribute this article and useful discussion with Prof. A K Jain in its preparation. Each of the laboratories depends on the technical support from many colleagues whose efforts are thankfully acknowledged. Funding from the MoES, DST and CSIR in the establishment and operation of these facilities is gratefully acknowledged. The authors also thank Directors of IUAC, WIHG, and CSIR-NGRI for continued support.

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