Age Dating of Gemstones, Pearls and Corals:

Fascinating Insights in their Formation, Origin and Historic Provenance

GemA
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Director
Swiss Gemmological Institute SSEF

Contents

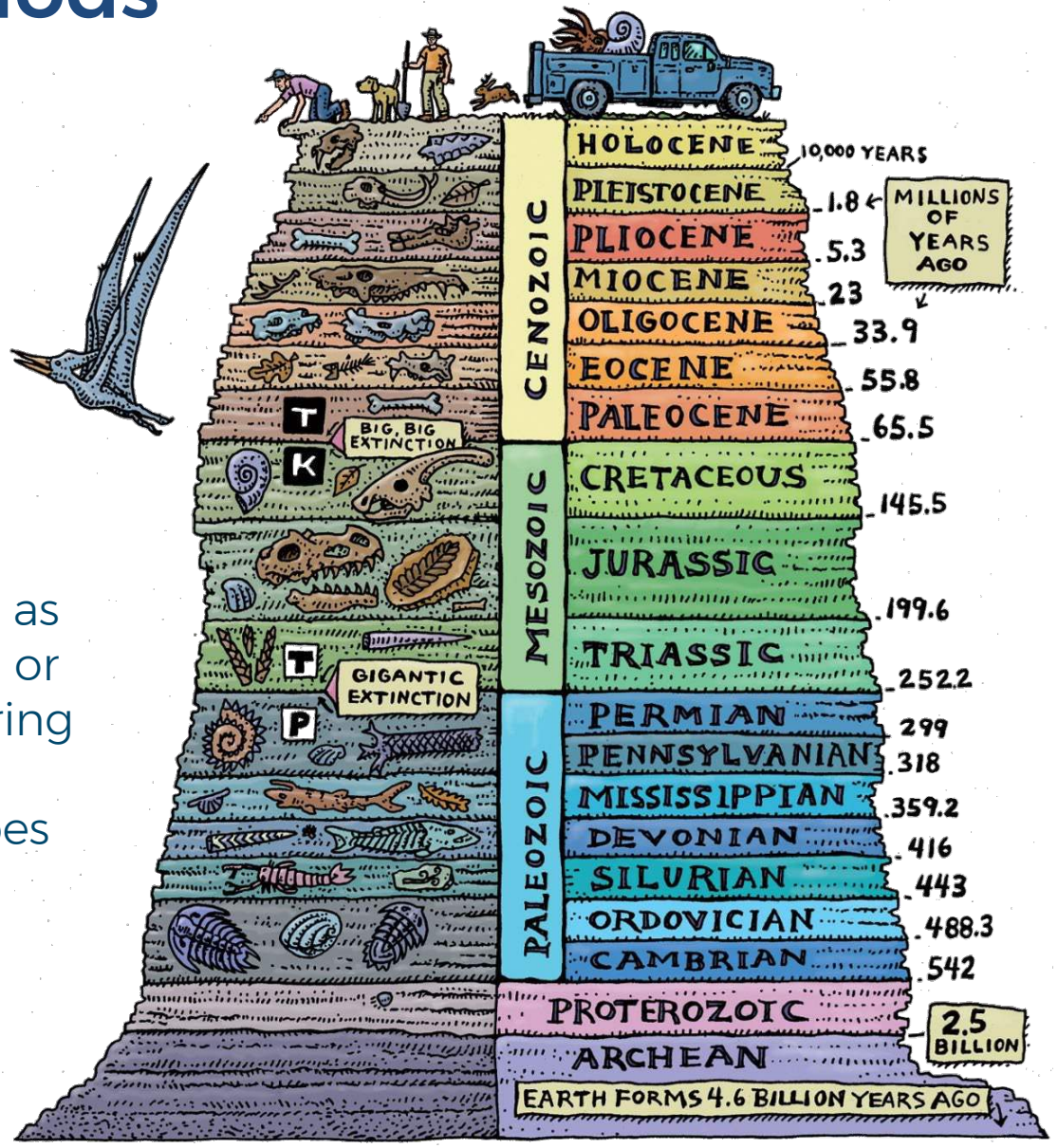
- Basics & Principles
- Instrumentation & Data plotting

- Age dating cases of:
- Pearls & corals
- Gemstones and gem-deposits

- Conclusions

Earth time periods

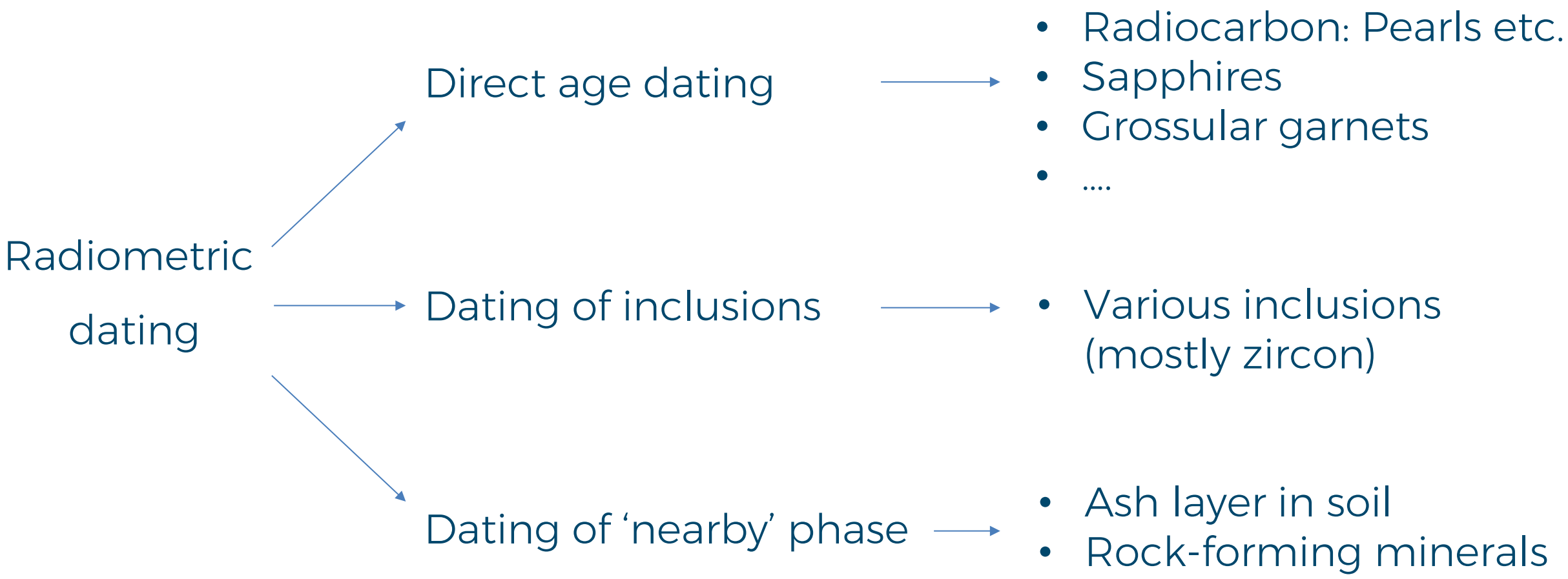
Radiometric age dating is used to date materials such as for example rocks, minerals, or historic artefacts by comparing the abundance of naturally occurring radioactive isotopes and their decay products.



Non-linear time axis

Scheme © Ray Troll

Options for radiometric dating



Reasons for radiometric dating in gemmology

Dating of geological event(s) and history, e.g. granite intrusion related to gem-deposit.

sample location and geological context exactly known!

Dating of mineral formation, e.g. zircon in mineral/gemstone.

may support origin determination if sample location is not known!

Dating to support historic provenance, e.g. pearls in historic jewellery.

to check if the documented provenance is reliable or not?

Dating as an additional information for clients.

offers the client an intriguing fact for story-telling about a gemstone!

Radiometric dating in gemmology

- **Age dating is not new in gemmology:**

Earth and Planetary Science Letters
Volume 94, Issues 1–2, August 1989, Pages 22-28

^{40}Ar–^{39}Ar laser probe dating of individual clinopyroxene inclusions in Premier eclogitic diamonds

R. Burgess [1], G. Turner [1], M. Laurenzi [2], J.W. Harris [3]

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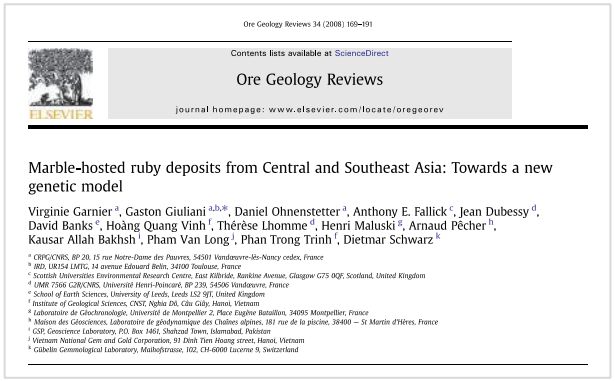
Abstract

The ages of seven individual clinopyroxene inclusions in Premier diamonds of eclogitic association have been determined using the ^{40}Ar—^{39}Ar dating technique. Syngenetic inclusions weighing between 10 and 130 μg were exposed on cleaved surfaces of the diamonds and analysed using a laser



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Marble-hosted ruby deposits from Central and Southeast Asia: Towards a new genetic model

Virginie Garnier [a], Gaston Giuliani [a,b,*], Daniel Ohnenstetter [a], Anthony E. Fallick [c], Jean Dubessy [d], David Banks [e], Hoàng Quang Vinh [f], Thérèse Lhomme [d], Henri Maluski [g], Arnaud Pêcher [h], Kausar Allah Bakhsh [i], Pham Van Long [j], Phan Trong Trinh [f], Dietmar Schwarz [k]

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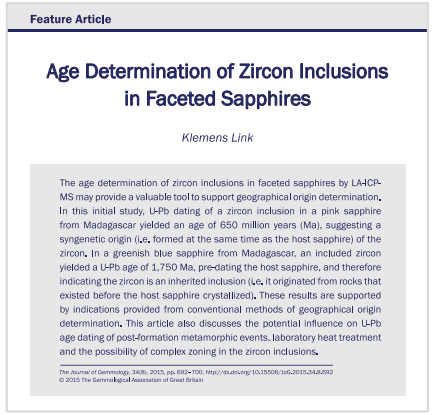

Feature Article

Age Determination of Zircon Inclusions in Faceted Sapphires

Klemens Link

The age determination of zircon inclusions in faceted sapphires by LA-ICP-MS may provide a valuable tool to support geographical origin determination. In this initial study, U-Pb dating of a zircon inclusion in a pink sapphire from Madagascar yielded an age of 650 million years (Ma), suggesting a syngenetic origin (i.e. formed at the same time as the host sapphire) of the zircon. In a greenish blue sapphire from Madagascar, an included zircon yielded a U-Pb age of 1,750 Ma, pre-dating the host sapphire, and therefore indicating the zircon is an inherited inclusion (i.e. it originated from rocks that existed before the host sapphire crystallized). These results are supported by indications provided from conventional methods of geographical origin determination. This article also discusses the potential influence on U-Pb age dating of post-formation metamorphic events, laboratory heat treatment and the possibility of complex zoning in the zircon inclusions.

inclusions sheds new light

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Abstract

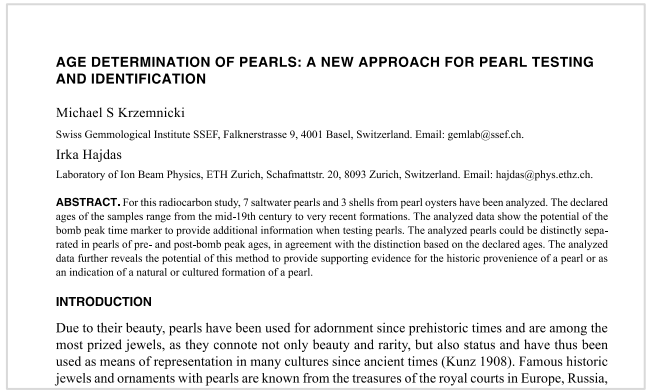
Uranium–lead isotope dating of two zircon inclusions in sapphires from the Central Province, NSW, gives ages of 35.9 ± 1.9 and 33.7 ± 2.1 million years (Ma). These ages fall within the range of basalt potassium–argon ages of 19 to 38Ma and zircon fission track ages of 2 to 49Ma for the timing of volcanism of the Central Province, NSW. These data, combined with the observation that corundum is found associated with many alkali basaltic provinces, indicate a genetic link between the growth of large corundum crystals and the processes involved in alkali basaltic magma generation. The reported failure of experimental attempts to grow corundum from a corundum-bearing basaltic composition, and more significantly, the abundance of incompatible elements such as U, Th, Zr, Nb and Ta in inclusion minerals indicate that the crystallization process is not simple. Corundum and the other minerals found as its inclusions (zircon, columbite, thorite, uranium pyrochlore, alkali feldspar etc.) could not have crystallized from most basaltic compositions. A more complex process must occur in which crystallization takes place when there are high proportions of incompatible elements and volatiles in the melt. These crystallization products are then carried to the surface by upward movement of later magmas. The extent of this process presumably determines whether a particular basaltic province carries sufficient corundum to be worked into economic concentrations of sapphire.

Keywords: sapphire, uranium–lead dating, inclusions, zircon, Central Province, Australia.

Introduction

A large number of important sapphire and ruby gemfields are associated with largely alkali volcanic terrains. The gem-quality corundum is com-

Keller, 1982; Gunawardene and Chawla, 1984); Bokeo Plateau, Xuan Loc Plateau, Cardomones Massif, Soloveus Plateau and Kassens Plateau in Kampuchea (Lacombe, 1969–70); Haut Chalong Plateau, Pleiku Plateau, Darlac Plateau



AGE DETERMINATION OF PEARLS: A NEW APPROACH FOR PEARL TESTING AND IDENTIFICATION

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ABSTRACT. For this radiocarbon study, 7 saltwater pearls and 3 shells from pearl oysters have been analyzed. The declared ages of the samples range from the mid-19th century to very recent formations. The analyzed data show the potential of the bomb peak time marker to provide additional information when testing pearls. The analyzed pearls could be distinctly separated in pearls of pre- and post-bomb peak ages, in agreement with the distinction based on the declared ages. The analyzed data further reveals the potential of this method to provide supporting evidence for the historic provenience of a pearl or as an indication of a natural or cultured formation of a pearl.

INTRODUCTION

Due to their beauty, pearls have been used for adornment since prehistoric times and are among the most prized jewels, as they connote not only beauty and rarity, but also status and have thus been used as means of representation in many cultures since ancient times (Kunz 1908). Famous historic jewels and ornaments with pearls are known from the treasures of the royal courts in Europe, Russia,

PERGAMON

Journal of Asian Earth Sciences 20 (2002) 841–849

Journal of Asian Earth Sciences

Sapphire crystallization, age and origin, Ban Huai Sai, Laos: age based on zircon inclusions

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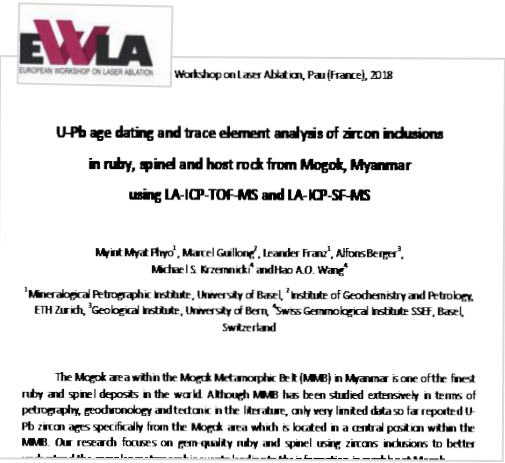
Abstract

Sapphire from the Ban Huai Sai alluvial gemfield, Laos, derives from local Cenozoic basalts. Syngenetic mineral inclusions in the sapphire include zircon, chromite, ulvöite and 'xenotime' (Y). Some of the high resolution ion microprobe (SHRIMP) analyses of zircon inclusions show enrichment in Hf, Y and rare-earth elements (REE), with REE ranging into the highest abundances in such zircons (up to 5785 ppm). This, with Eu depletion and the other inclusion mineralogy, suggests that the sapphire crystallized from highly evolved alkaline melts, before commencement of transport in basalt magma. U–Pb isotope (SHRIMP) determinations on zircon inclusions give measured zircon ages of 1.2 ± 1.3 * 0.3 Ma. This is taken to mark the time of host sapphire crystallization, within error of a basalt K–Ar age from provenance. The suggested age for Ban Huai Sai zoned sapphire crystallization matches that given by a zoned sapphire in a metasomatic xenolith from Khao Wua, Thailand (1 ± 2 Ma). These results favour sapphire genesis linked to processes associated with young alkali basaltic magmatism in South East Asia. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Sapphire; Zircon; Basalt; Cenozoic; U–Pb dating; Laos

1. Introduction

The corundum xenocrysts become concentrated in alluvial and alluvial deposits shed from the basaltic terrains. Corundum-bearing xenoliths are very rare compared to the most



EWLA EUROPEAN WORKSHOP ON LASER ABLATION

Workshop on Laser Ablation, Pau (France), 2018

U-Pb age dating and trace element analysis of zircon inclusions in ruby, spinel and host rock from Mogok, Myanmar using LA-ICP-TOF-MS and LA-ICP-SF-MS

Myint Myat Phyo[1], Marcel Guillong[2], Leander Franz[1], Alfons Berger[3], Michael S. Krzemnicki[4] and Hao A.O. Wang[4]

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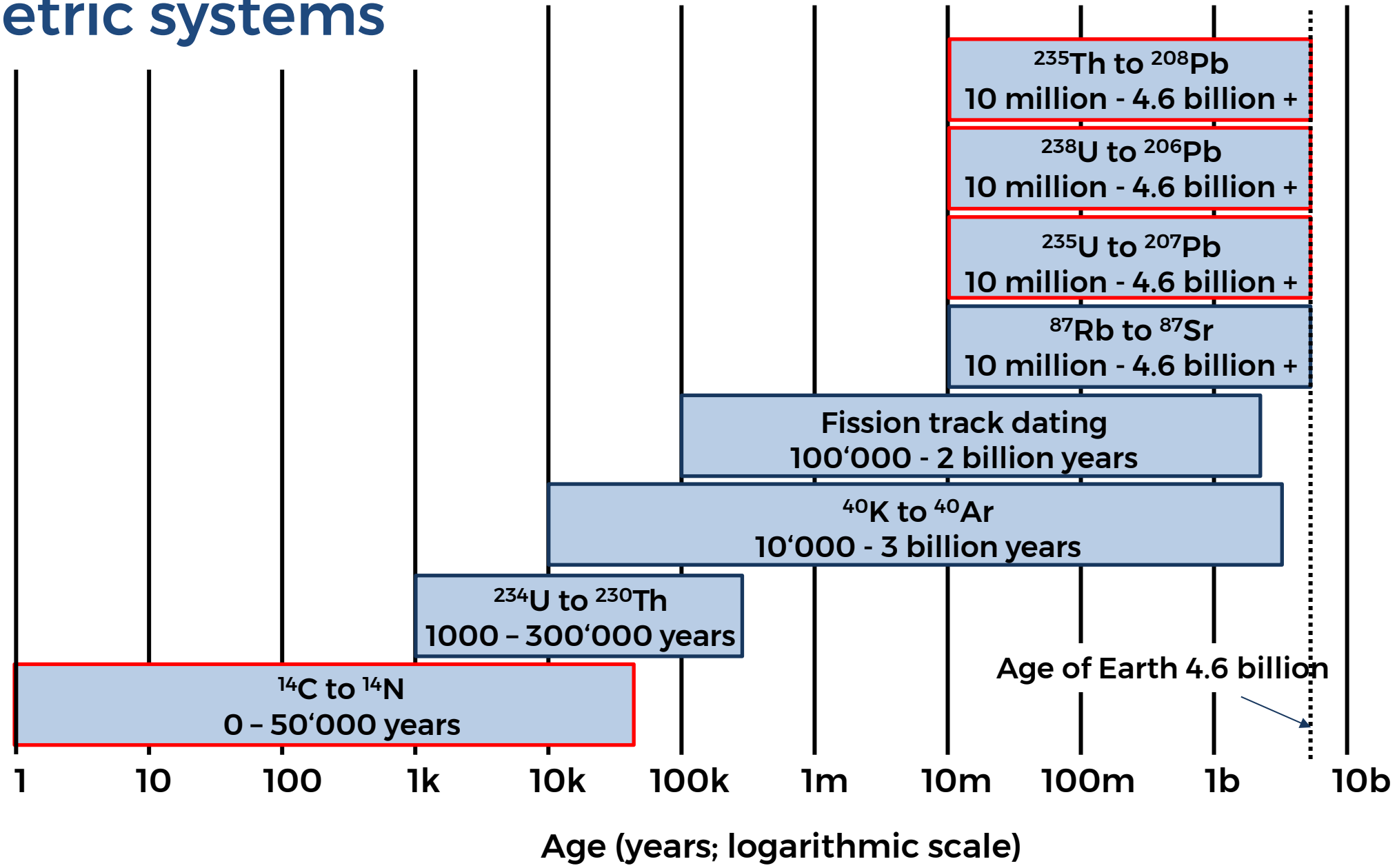
The Mogok area within the Mogok Metamorphic Belt (MMB) in Myanmar is one of the finest ruby and spinel deposits in the world. Although MMB has been studied extensively in terms of petrography, geochronology and tectonic in the literature, only very limited data so far reported U-Pb zircon ages specifically from the Mogok area which is located in a central position within the MMB. Our research focuses on gem-quality ruby and spinel using zircons inclusions to better

Radiometric systems



Radiometric systems

Principle:

- An instable (radioactive) isotope decays into a stable isotope (e.g. ^{238}U to ^{206}Pb).
- Often through a complex cascade of intermediate steps (alpha and beta-decays)
- Each radioactive decay path has a unique constant 'half-life'.
- Ratio of instable (radioactive) to stable isotope is used for radiometric age dating.

At SSEF, we mainly use three decay mechanisms:

- **^{14}C to ^{14}N: radiocarbon dating:**
  e.g. for dating biogenic or archaeological samples of 'young' age (< 60'000 years).

- **U-Pb dating** with two routes: ^{238}U to ^{206}Pb and ^{235}U to ^{207}Pb.
  well established in geoscience for dating of minerals (gems) and rocks.

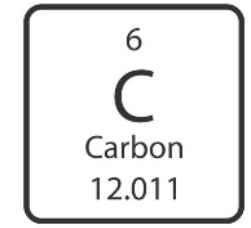
- **^{232}Th-^{208}Pb dating:**
  well established in geoscience for dating of minerals (gems) and rocks.

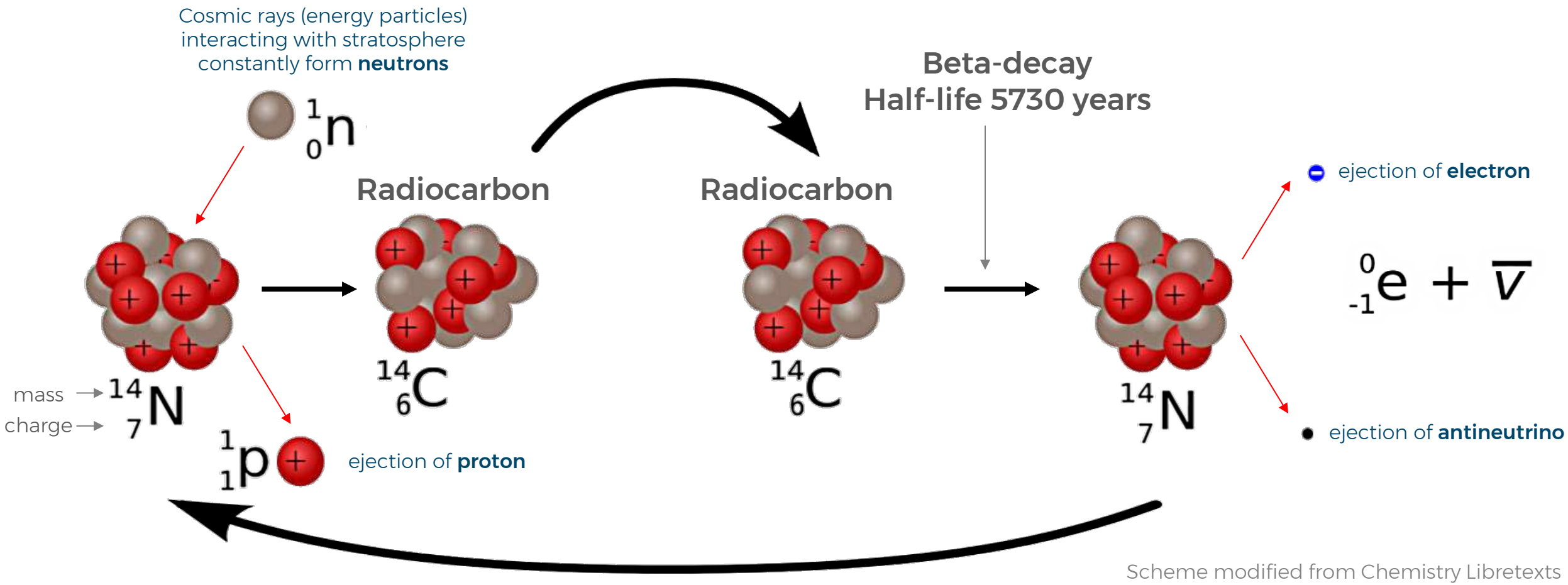
- Further geochronometers, such as for example ^{87}Sr-^{87}Rb are used so far only on specific research samples but are explored further on gems.

Radiocarbon cycle

- **^{14}C formation and decay to ^{14}N**

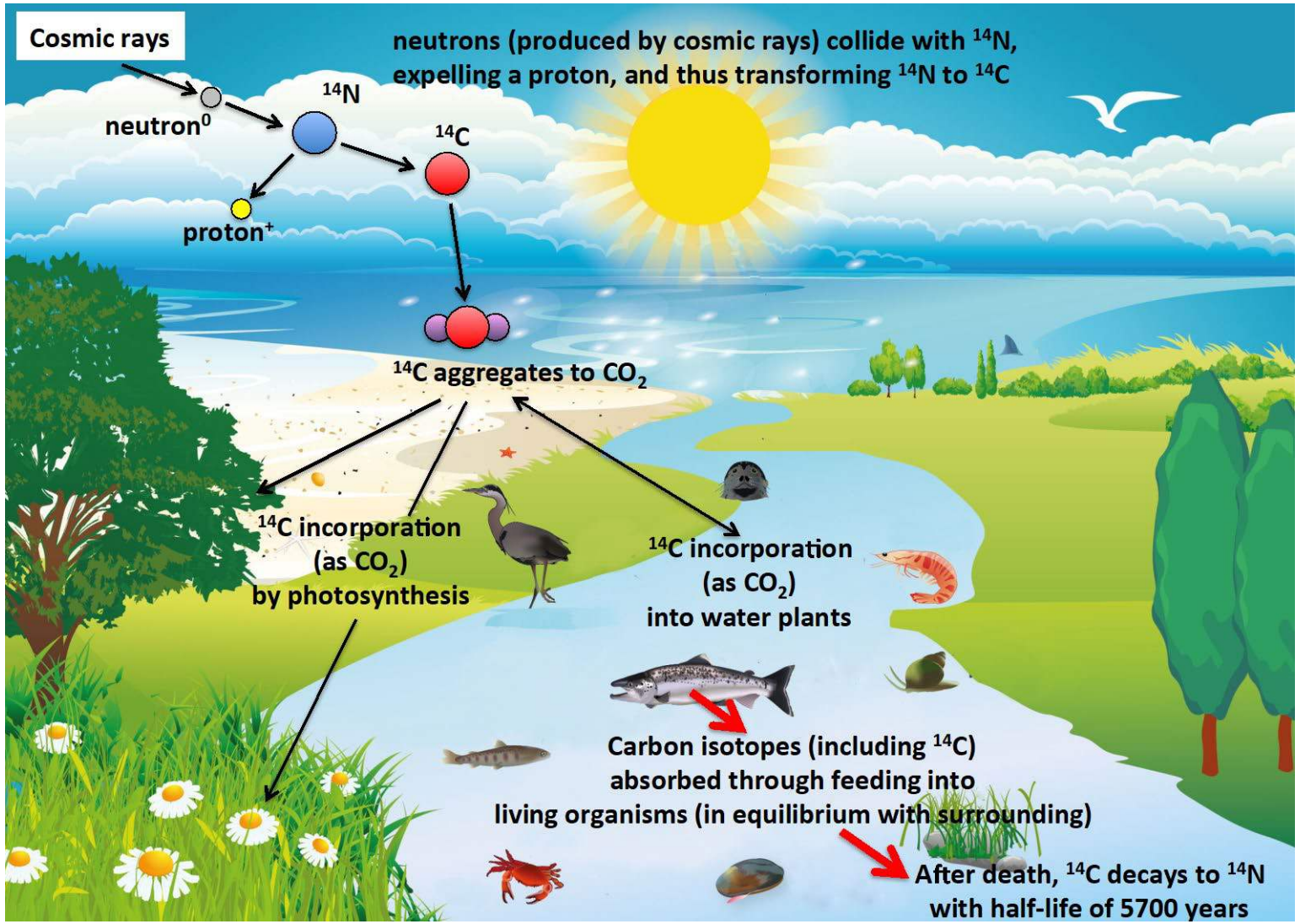
Carbon has three main isotopes:
^{12}C (stable; 98.9%),
^{13}C (stable; 1.06%),
and radiocarbon ^{14}C (instable; 1 ppt).





Scheme modified from Chemistry Libretexts

Radiocarbon cycle

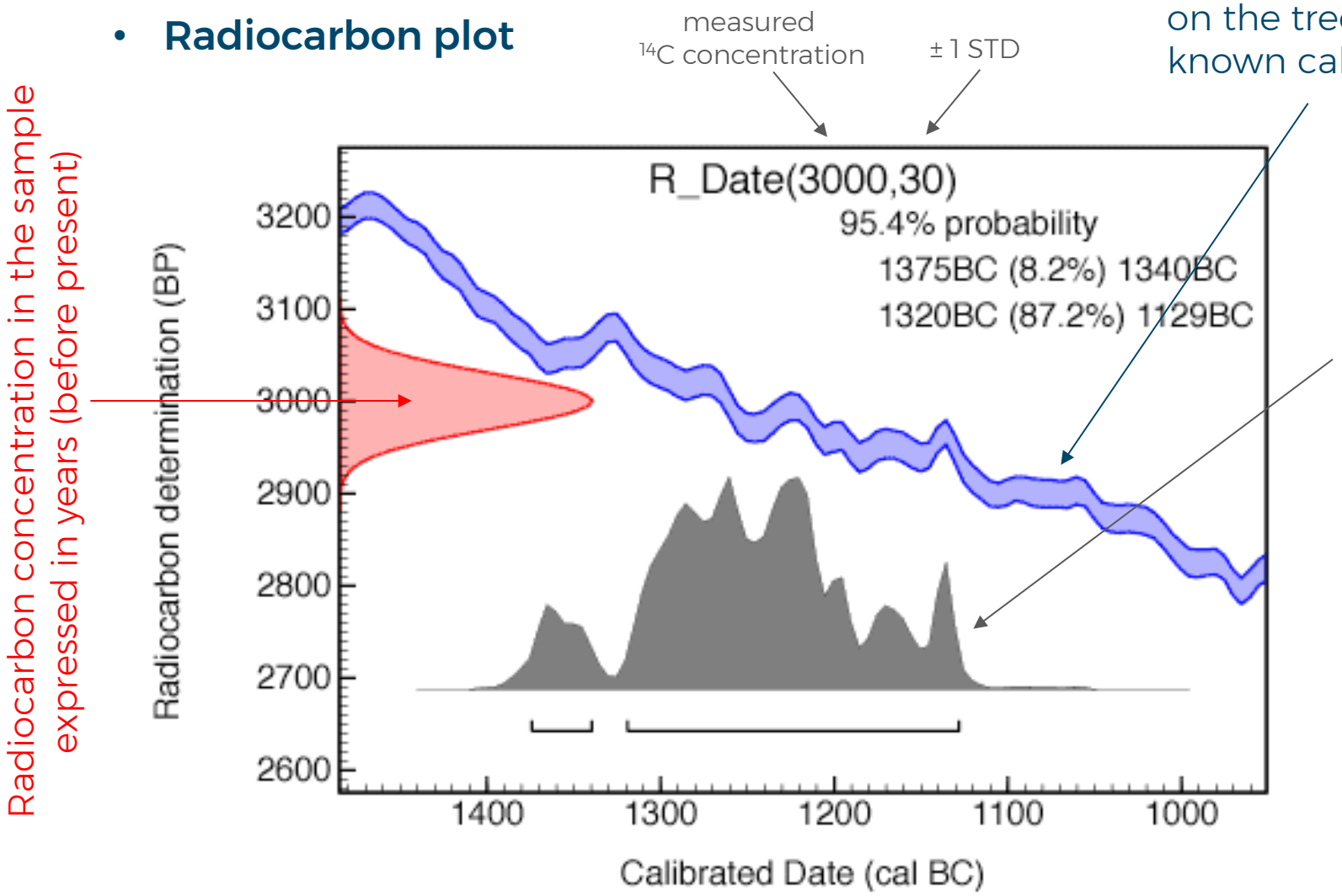


- Carbon needs to be present as main element in sample.
- Half-life of radiocarbon ^{14}C is 5730 years.
- Reliable dating only up to about 50’000 years ago.
- Dating method for rather “young” samples (e.g. pearls, corals, ivory).

Scheme
M.S. Krzemnicki, SSEF
using background illustration
from www.fisheriesireland.ie

Dating plots:

- **Radiocarbon plot**



Radiocarbon concentration in the sample expressed in years (before present)

Calendar years (derived from tree ring data)

radiocarbon measurements on the tree rings (± 1 STD) of known calibrated age.

The **grey histogram** shows possible ages for the sample (the higher the histogram the more likely that age is).
The results of calibration are often given as an age range.

In this case, we might say that we could be 95% sure that the sample comes from between 1375 cal BC and 1129 cal BC.

Scheme from Oxford University, slightly modified

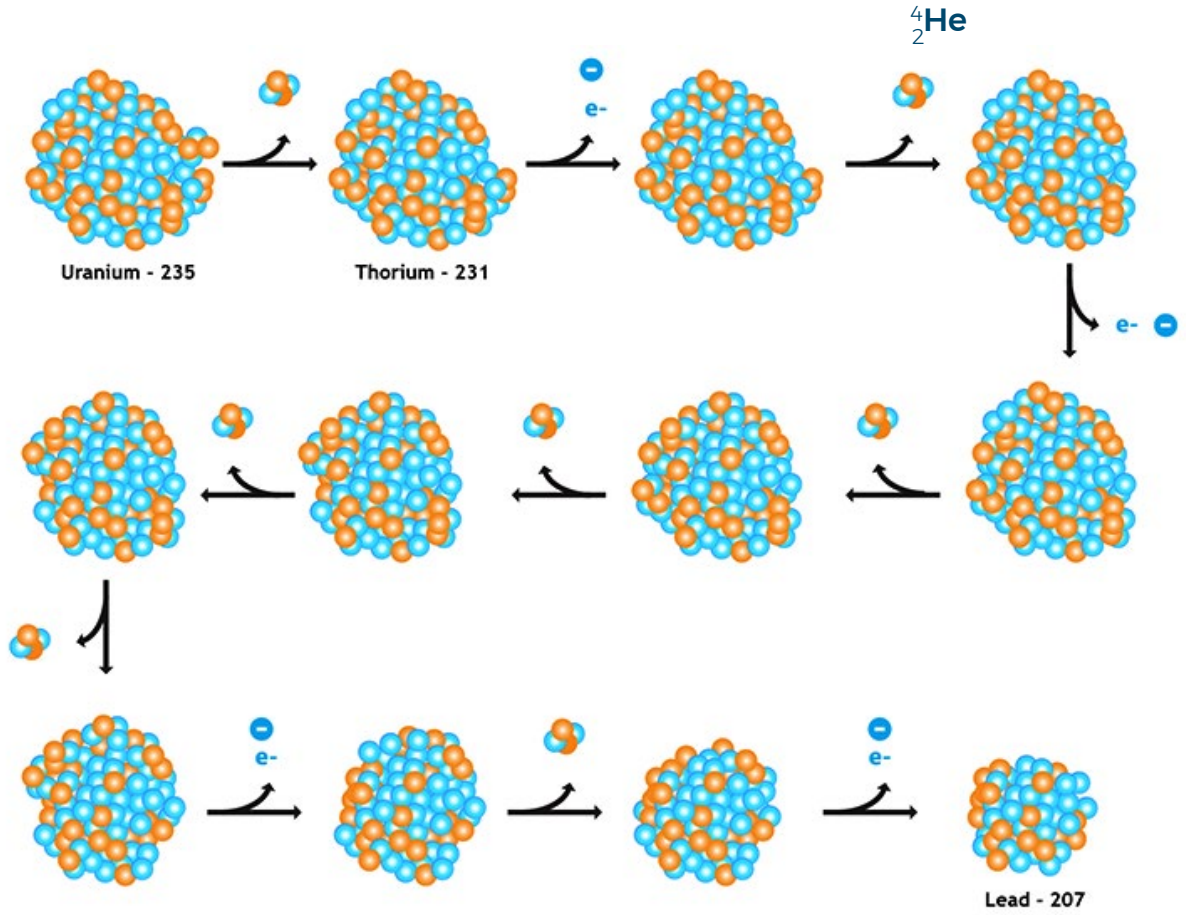
U-Pb and Th-Pb decay

- **Radioactive decay through complex alpha (^{4}He) and beta (e-) decay**

- **^{238}U to $^{206}Pb**
  Half-life: 4.468 billion years

- **^{235}U to $^{207}Pb**
  Half-life: 0.704 billion years

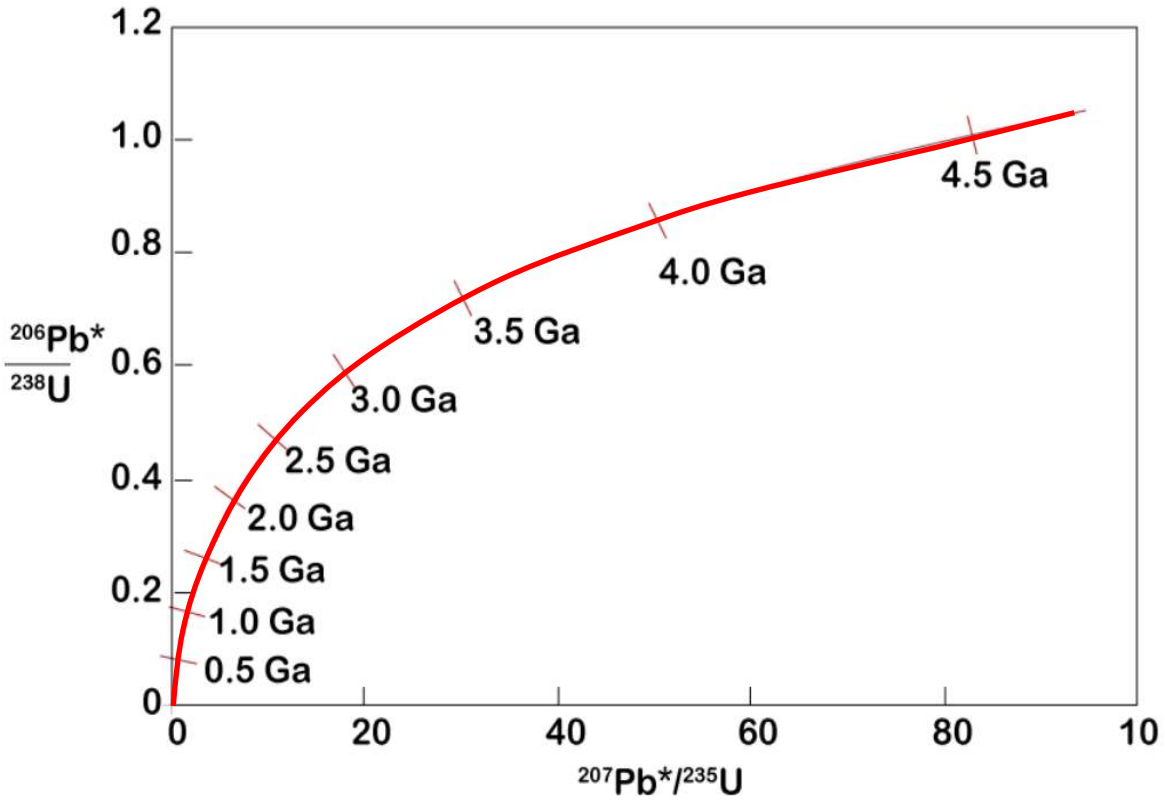
- **^{232}Th to $^{208}Pb**
  Half-life: 14.050 billion years



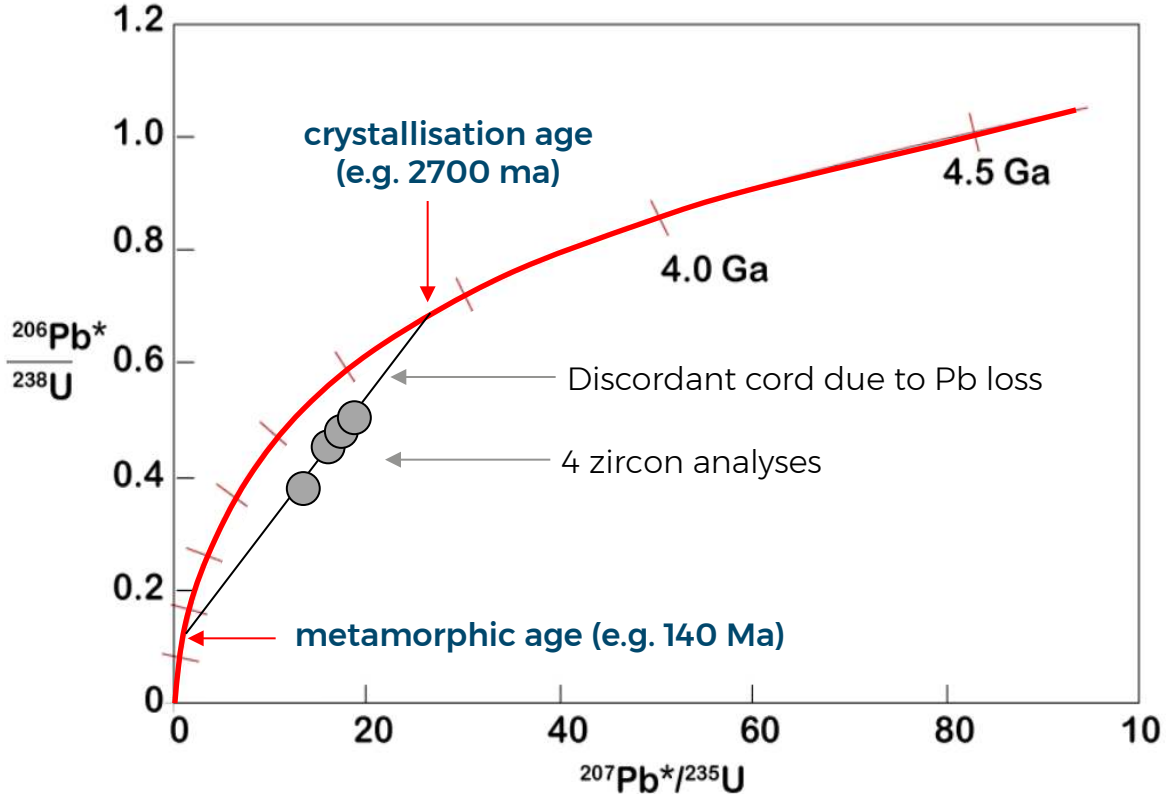
^{235}U to ^{207}Pb decay
Scheme from Berkeley University;
slightly modified

Dating plots:

- **The Concordia diagram for U-Pb radiometric dating**



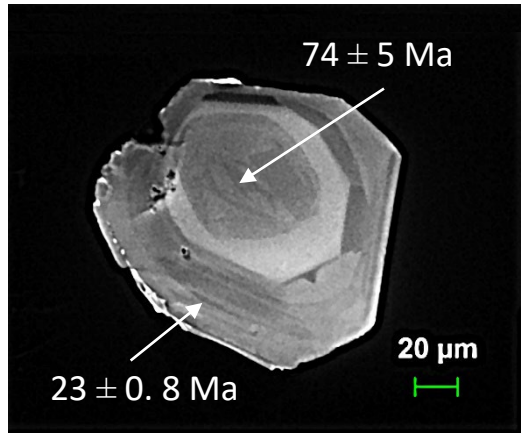
Pb*: radiogenic Pb



Schemes from Cornell University, USA, slightly modified

Special case: Detrital zircon inclusion ($ZrSiO_4$)

- Common U-Th-bearing accessory in igneous rocks, which form the main proto-sources of siliclastic sediments and many metamorphites.
- Very durable; undergoes minimal abrasion and chemical alteration.
- Often reintegrated in rocks and minerals (so-called **detrital zircons**)
- Considered **'time capsule'** carrying information about its igneous and metamorphic history.
- Zircon ages often open window to protogenetic events before the formation of the gemstone itself during a later metamorphic event.
- Often narrow and complex zoning: often 'mixed ages' (old and young zones ablated simultaneously with normal ablation spot (50-100 µm).
- For more detailed age dating of detrital zircon, a highly focused beam technique (e.g. SF-MS or Ion-beam) is required.



Analytical Methods used by SSEF

- **Accelerator mass spectrometer** (AMS)
  for radiocarbon dating
  of biogenic samples (e.g. pearls)

- **Time-of-Flight mass spectrometer** (LA-ICP-TOF-MS, aka GemTOF)
  for analysis and radiometric dating of
  geological samples (e.g. gemstones)

- **Sector-Field mass spectrometer** (LA-ICP-SF-MS)
  for high-resolution analysis of selected isotopes (multi-collector)
  and radiometric dating (e.g. zircon with zoning)

Images ETH Zurich & ionplus AG (AMS), TOFwerk, (TOF-MS) and Nu Instruments, Ametek (SF-MS)

Cases:

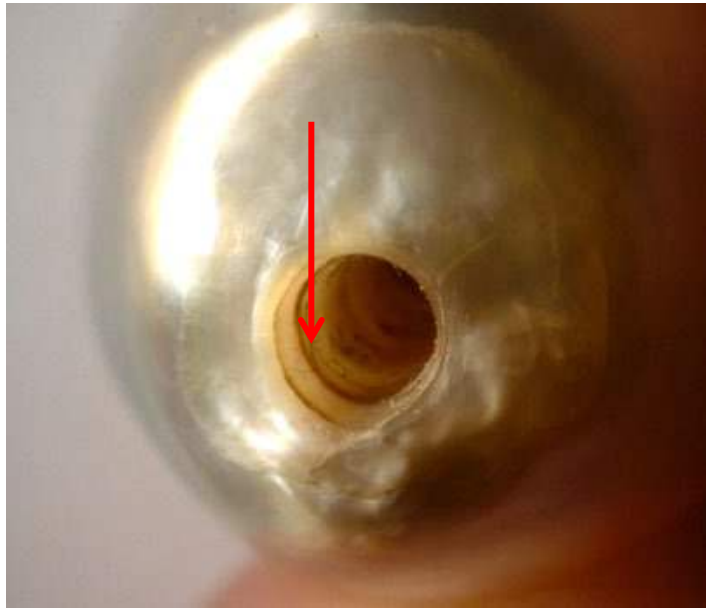
Radiocarbon dating : direct age dating

For biogenic material such as pearls, corals and ivory:

- Generally to investigate or confirm their historic or modern provenance.
- Specifically for pearls it may assist the identification of a natural pearl.
- For elephant ivory, to check for compliance with CITES and EU regulations.

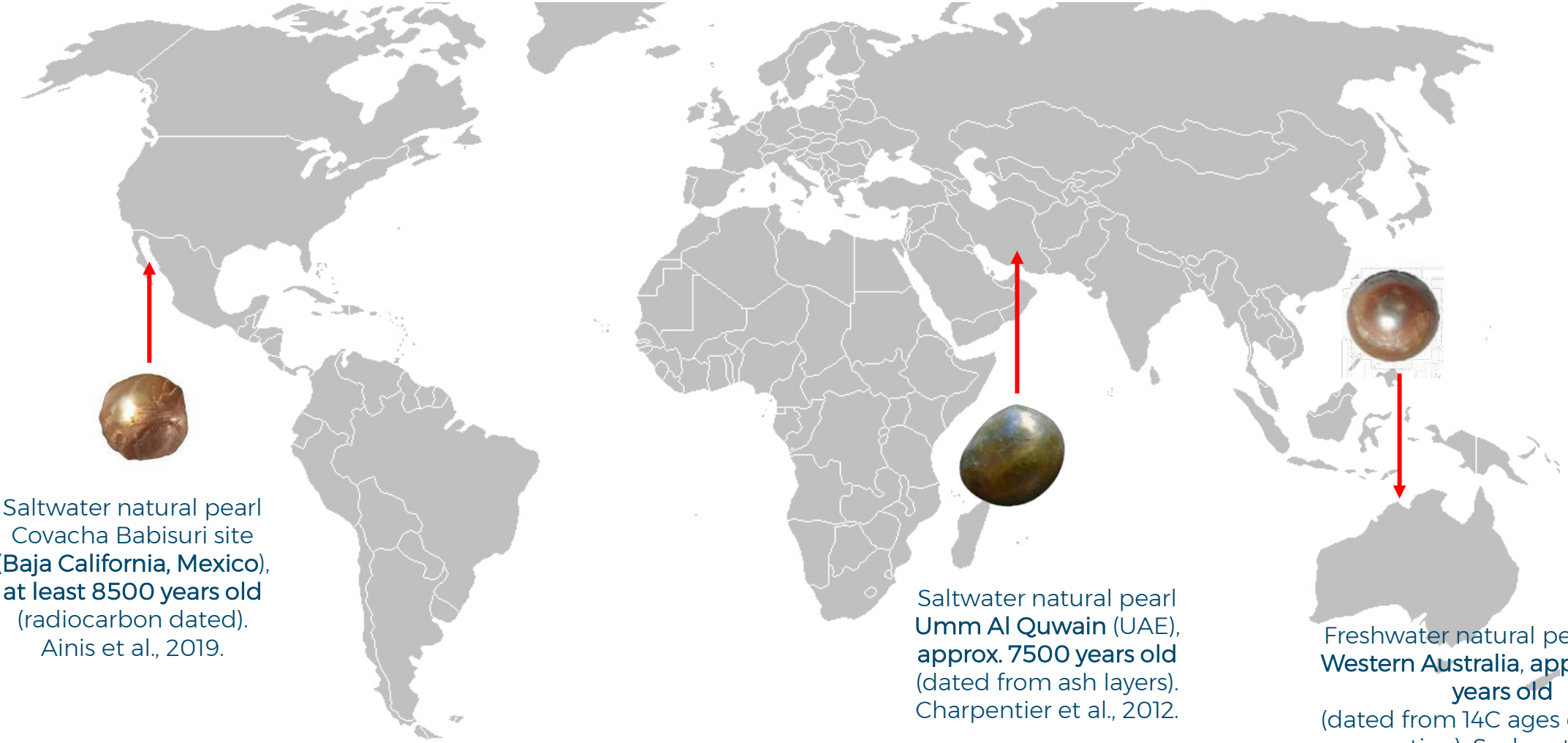
Radiocarbon dating at SSEF

- SSEF developed **quasi non-destructive sampling method**.
- Tiny sample (about 4 mg or 0.02 ct) is enough for radiocarbon dating.
- Sampling usually in the drill hole of pearls (or coral beads)
- Even possible for archaeological samples of cultural heritage.
- Radiocarbon dating **service for clients** offered in collaboration with specialized laboratory (Laboratory of Ion Beam Physics, ETH Zurich)



Pearls in history

Pliny, Historia Naturalis
"[Pearls] occupy the first rank…and the very highest position among valuables."



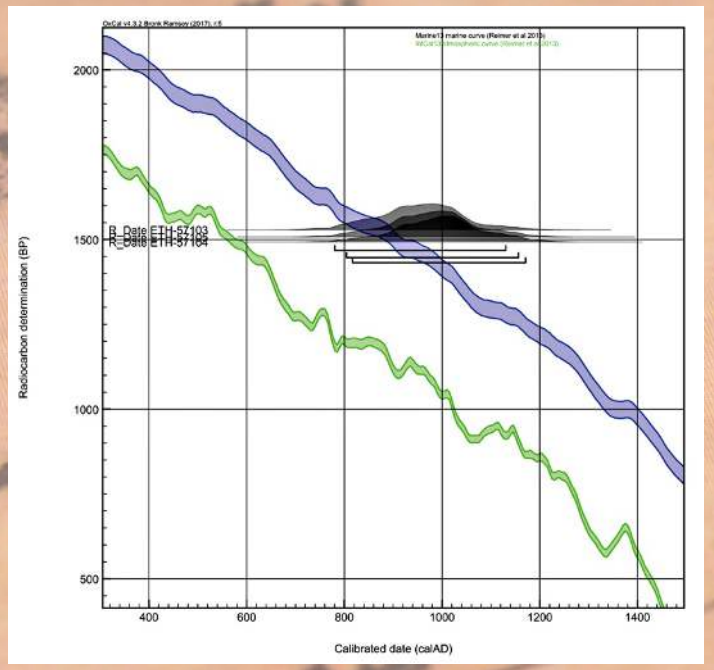
Saltwater natural pearl Covacha Babisuri site (**Baja California, Mexico**), **at least 8500 years old** (radiocarbon dated). Ainis et al., 2019.

Saltwater natural pearl **Umm Al Quwain** (UAE), **approx. 7500 years old** (dated from ash layers). Charpentier et al., 2012.

Freshwater natural pearl **North-Western Australia**, **approx. 2000 years old** (dated from 14C ages of shells in excavation). Szabo et al., 2015.

Map © Wikicommons

Pearls from the Cirebon Shipwreck



The ^{14}C ages perfectly fit with the archaeological dating of this shipwreck based on pottery and coins.

Feature Article

Radiocarbon Age Dating of 1,000-Year-Old Pearls from the Cirebon Shipwreck (Java, Indonesia)

Michael S. Krzemnicki, Laurent E. Cartier and Irka Hajdas

The 10th-century Cirebon shipwreck was discovered in 2003 in Indonesian waters. The excavation yielded an incredible array of archaeological finds, which included pearls and jewellery. Radiocarbon dating of the pearls agrees with the age of the shipwreck, which previously was inferred using recovered coins and ceramics. As such, these are some of the oldest pearls ever to be discovered. Based on this example, the present article shows how radiocarbon age dating can be adapted to the testing of historic pearls. The authors have further developed their sampling method so that radiocarbon age dating can be considered quasi-non-destructive, which is particularly important for future studies on pearls (and other biogenic gem materials) of significance to archaeology and cultural heritage.

The Journal of Gemmology, 35(8), 2017, pp. 728–736, http://dx.doi.org/10.15506/JoG.2017.35.8.728

Introduction

The discovery of the Cirebon (or Nan-Hai) shipwreck in the Java Sea in 2005 marks one of the most important archaeological finds in Southeast Asia in recent years (Hall, 2010; Liebner, 2014; Stargardt, 2014). Apart from ceramics, glassware and Chinese coins dating from the 10th century AD, the excavation of this ancient merchant vessel also produced a number of carved gastropod shells (presumably ritual objects, from *Turbinella pyrum*), jewellery (e.g. earrings with diamonds and sapphires), loose gemstones (e.g. sapphires, red garnet beads and rock crystal carvings) and a rather large number of small pearls (Tan, 2007; Liebner, 2010, 2014; Henricus, 2014). Of the more than 12,000 pearls that were recovered, most were less than a few millimetres in diameter (e.g. Figure 1).

Fishermen discovered the wreckage site accidentally in 2003, at depths greater than 50 m (Liebner, 2010) off the northern coast of Java, Indonesia, near the city of Cirebon (Figure 2). Excavation efforts were complicated due to legal uncertainties as to which companies/entities should be permitted to excavate the site, unfortunately leading to a period in which looting of the wreck occurred. Administrative, legal and diplomatic problems pertaining to the excavation, storage and ownership of recovered items continued in the following years (Tjoa-Bonatz, 2016).

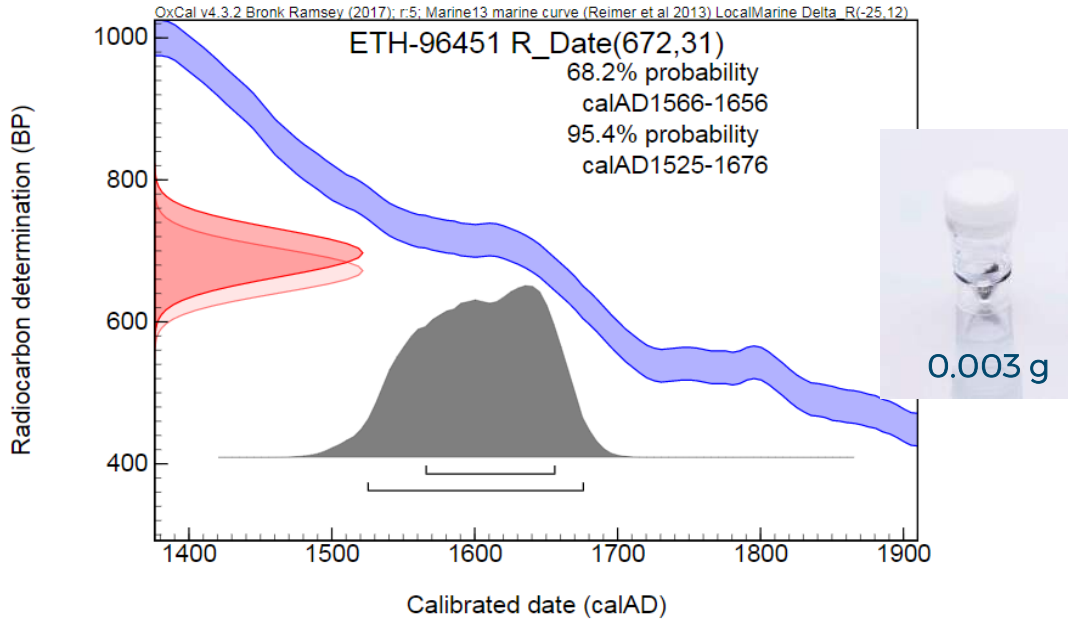
The exact route of the ship is still disputed in academic circles (Liebner, 2014), but there is ample evidence of strong trading ties between China and western Asia, which are supported by shipping routes along the Strait of Malacca between the Malay Peninsula and the Indonesian

Ana Maria Pearl

Provenance documented since mid 19th century:

Originally belonging to Ana María de Sevilla (1828-1861); probably fished during Hernán Cortéz' conquest of the Aztec empire in the 16th century .

The radiocarbon analyses (carried out by ETH Zurich) date this pearl to the 16th – 17th century, thus perfectly matching the documented provenance of this pearl.



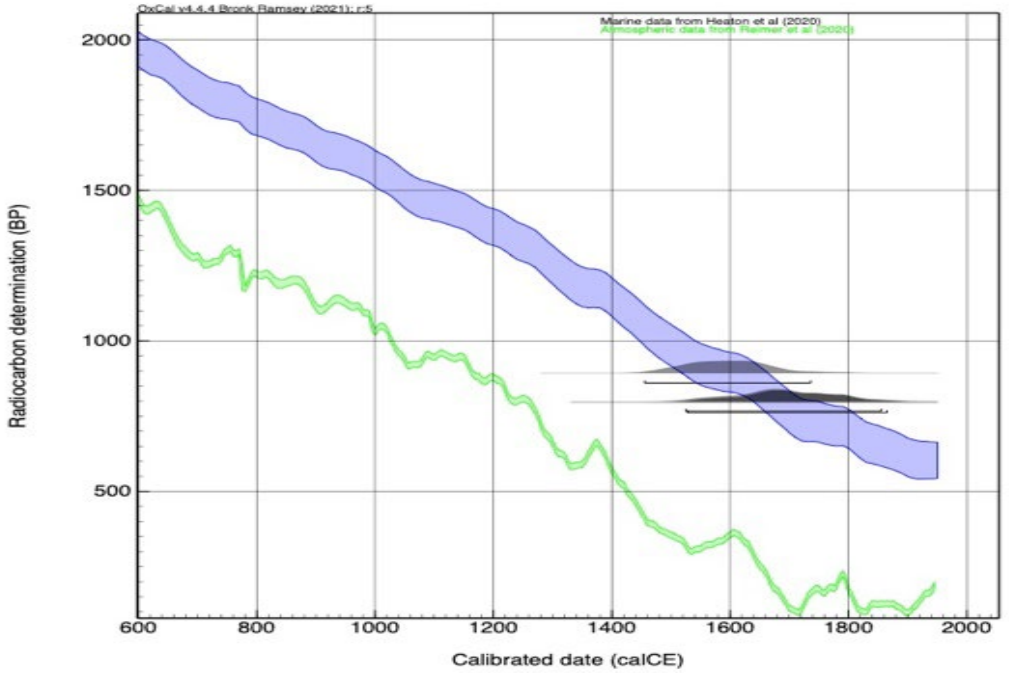
Historic pearl necklace

Radiocarbon dating on three randomly selected pearls confirmed their historic age (16th to 17th century).

Based on our DNA analyses:

- one pearl from *Pinctada radiata*
- and two pearls from *Pinctada persica*,
- Habitat of both species: Arabian Gulf.

To our knowledge, this is the first time that *Pinctada persica* is found in pearl jewellery.



Recent pearl formations

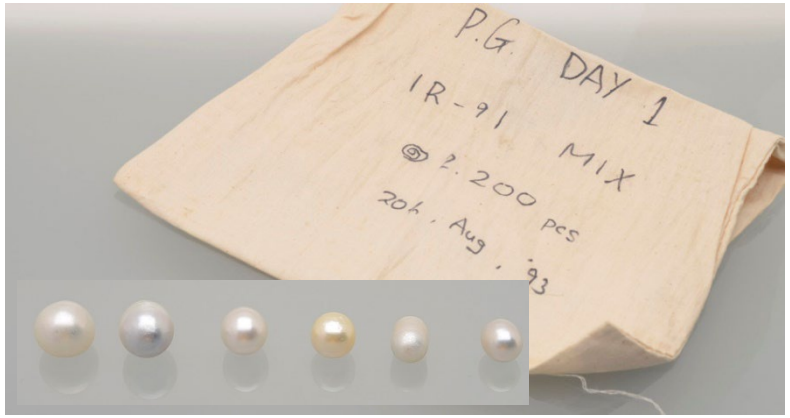
In the Paspaley pearl office in Darwin with Peter Bracher

Kuri Bay, in the Kimberley region of Northwestern Australia and location of Australia‘s first pearl farm by Paspaley (first production in 1958).

Recent pearl formations

Cultured pearls formed around the ‘bomb’ peak
Early production from Kuri Bay (Paspaley).

bomb peak
1966 (southern hemisphere)



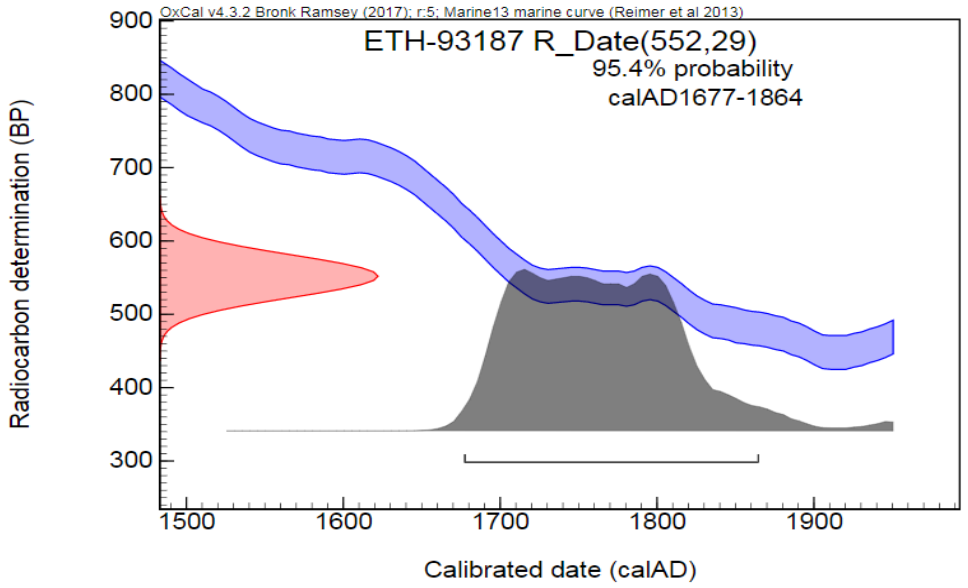
Cultured pearls from a test run by Paspaley: Seed grafting in 1991 and harvest in 1993.

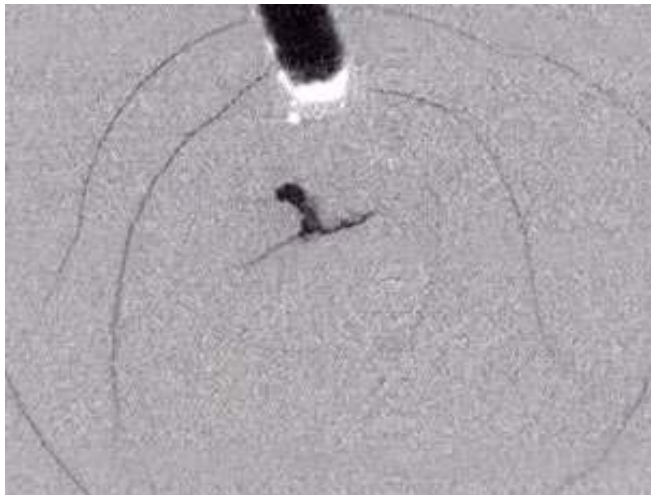
Natural pearl identification

Supporting evidence due to radiocarbon age dating

This saltwater pearl shows internal structures which are also known in beadless cultured pearls (Krzemnicki et al. 2010, Sturman 2009).

The radiocarbon analyses (carried out by ETH Zurich) date this pearl to the 17th – 18th century, distinctly pre-dating any pearl farming. The radiocarbon age thus supports the conclusion of natural pearl.





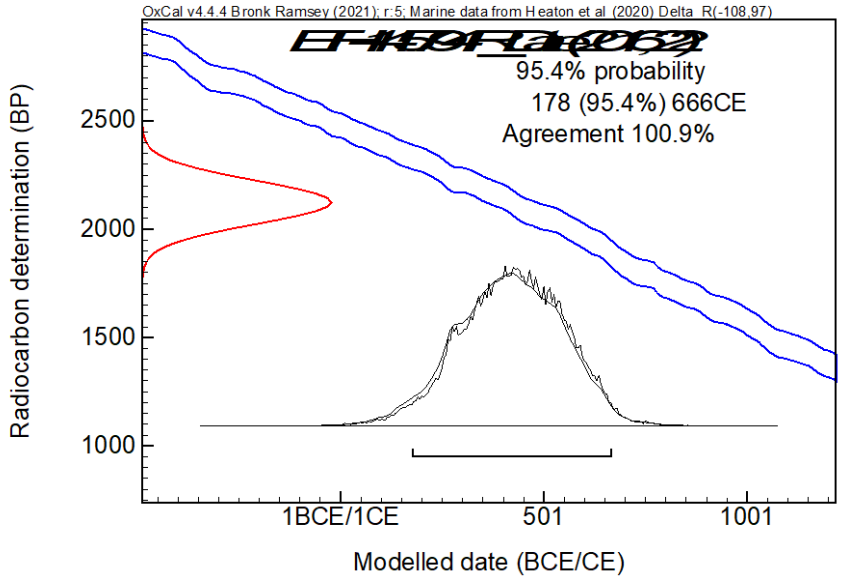
Micro X-ray tomography section of pearl centre.

Coral dating

Historic necklace with corals

- Offered at web-auction as a antique **Egyptian carnelian necklace**.
- In reality consists of **coral and one glass bead**.

- Radiocarbon: historic age of the corals.
- DNA testing: not conclusive but presumably *Corallium rubrum*



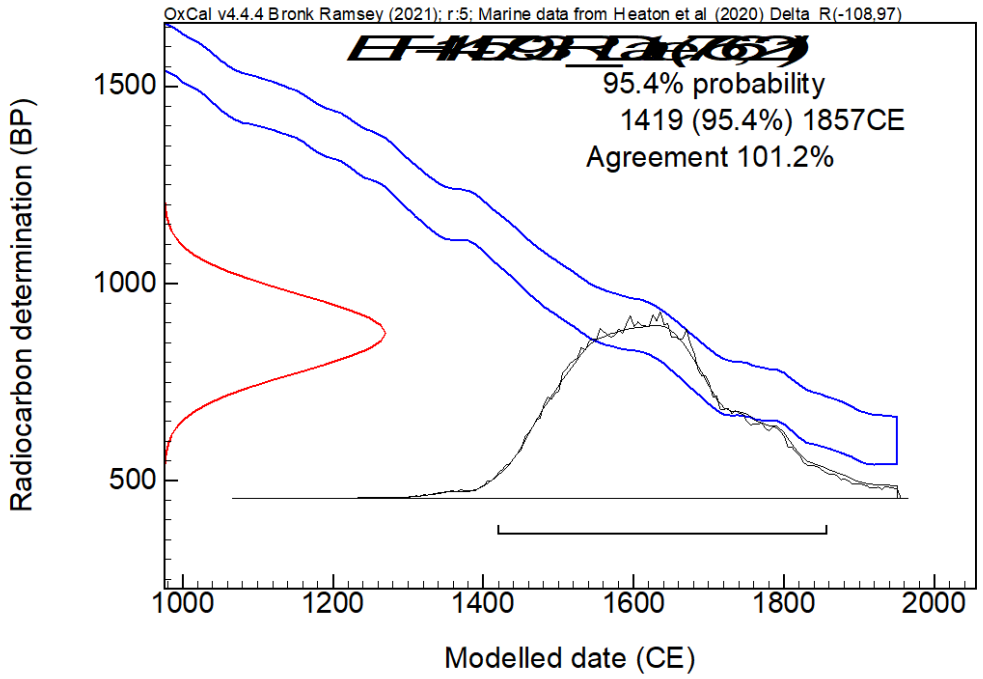
Detail of a coral necklace bought at auction

Coral dating

Historic multi-gem necklace with corals

- Presumably re-using even older drilled gemstones and coral beads.

- Radiocarbon: historic age of the corals.
- DNA testing: *Corallium rubrum*



Age dating of Gemstones

Rhino Ruby, Mozambique (22.04 ct),
ca. 550 million years old

Burma Ruby,
ca. 25 million years old

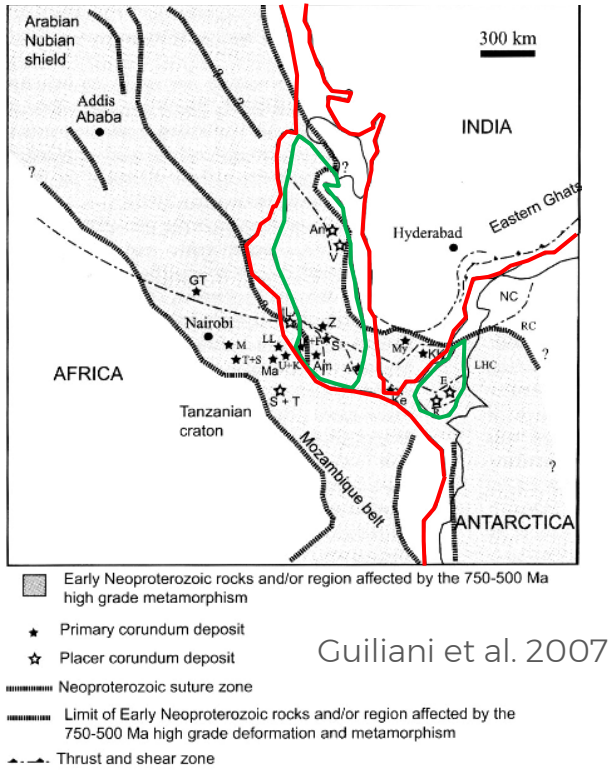
- Dating possible on inclusions or as direct dating of gemstones.

- May assist origin determination (geologically ‘young’ vs ‘old’ formation)
- Dating of gemstone and/or gem-deposit formation in research
- To offer client intriguing information about a gemstone: story-telling

Age dating of Gemstones

Three main global geological frameworks for many gem deposits:

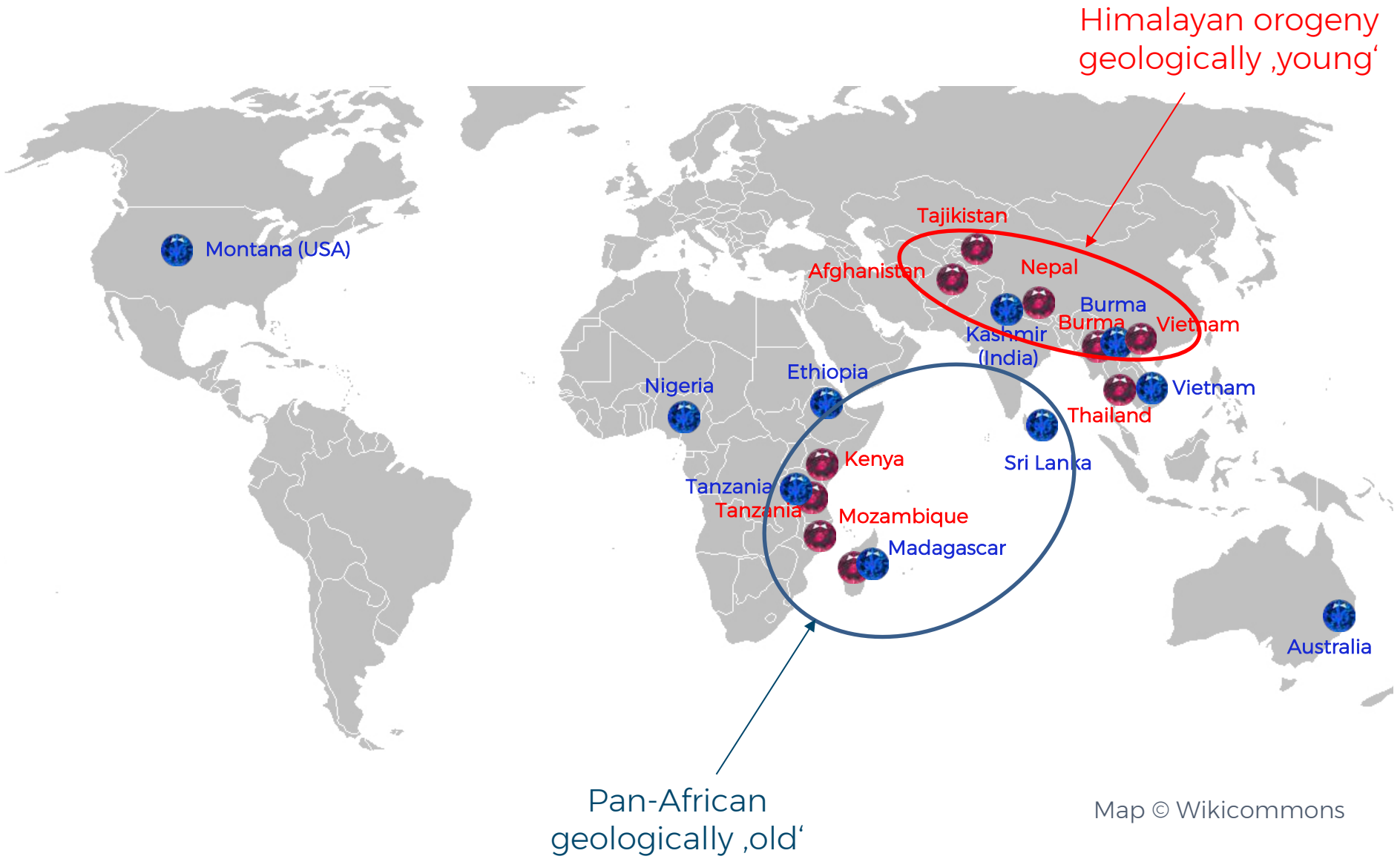
- Deposits related to Pan-African tectono-metamorphic events by the collision of eastern and western Gondwana about **750-450 ma** ago.
- Deposits related to the collision of the Indian and Eurasian plate (Himalayan orogeny) about **40-10 ma** ago.
- Volcanism (alkali-basalts), often related to extensional tectonics (e.g. Rift Valley in East-Africa), mostly about **4 ma ago and younger**.



Guiliani et al. 2007

Uluguru mountains, Tanzania. © Michael S. Krzemnicki

Age dating of Gemstones



Map © Wikicommons

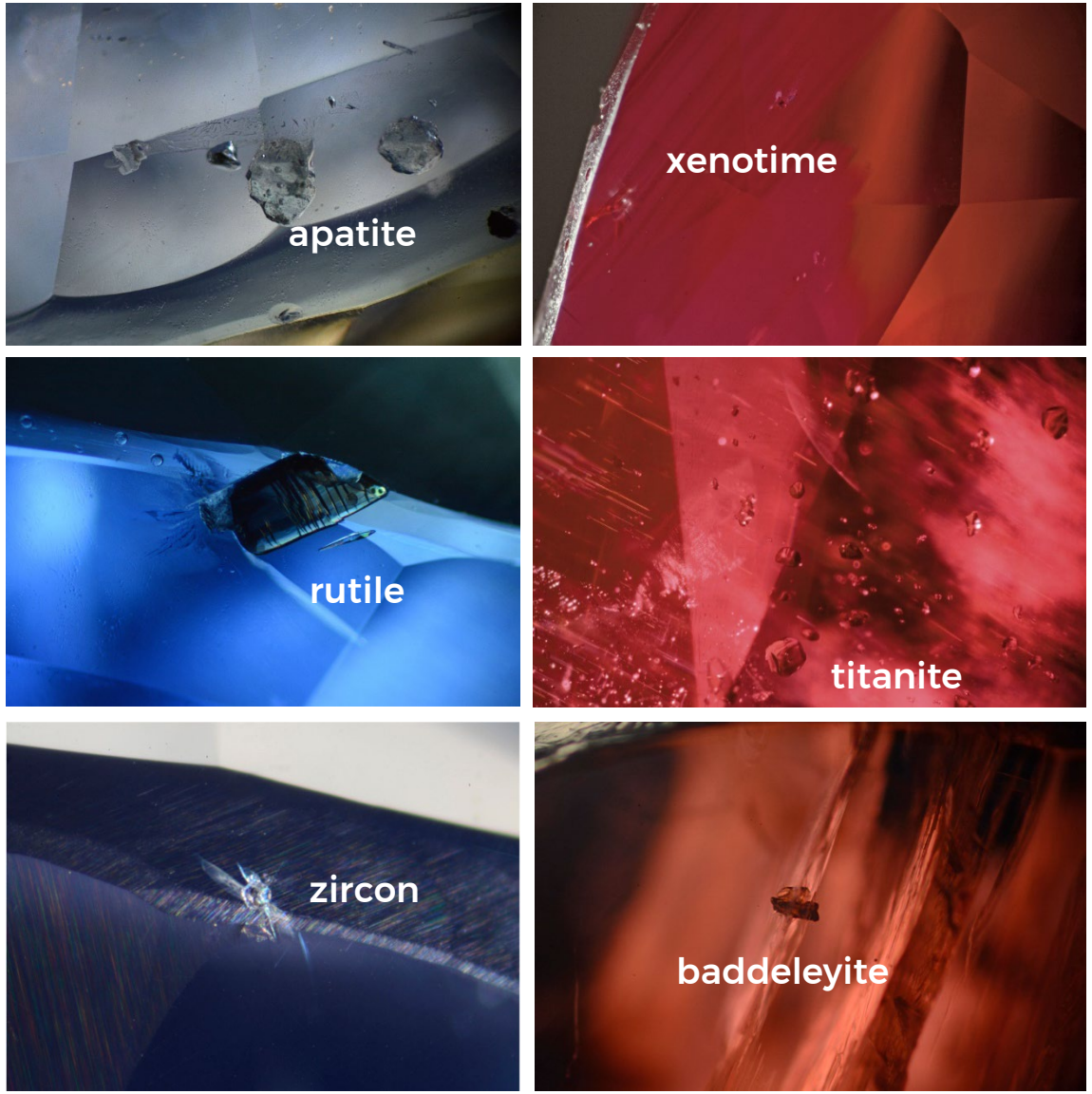
Age dating based on inclusions

The following inclusions have been used so far for U-Pb radiometric dating of gemstones at SSEF:

- Zircon
- Apatite
- Titanite
- Rutile (primary)
- Baddeleyite
- Zirconolite
- U-Pyrochlore
- Xenotime
- Chondrodite
- Monazite

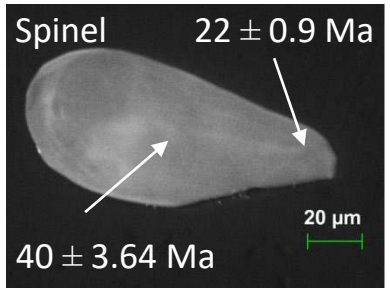
- Inclusion ages are either syn- or proto-genetic to gemstone formation.

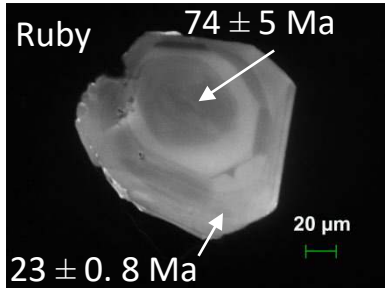
- A gemstone cannot be older than the age of its dated inclusions!

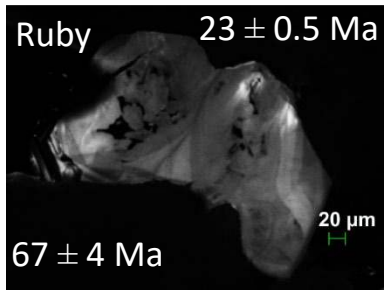


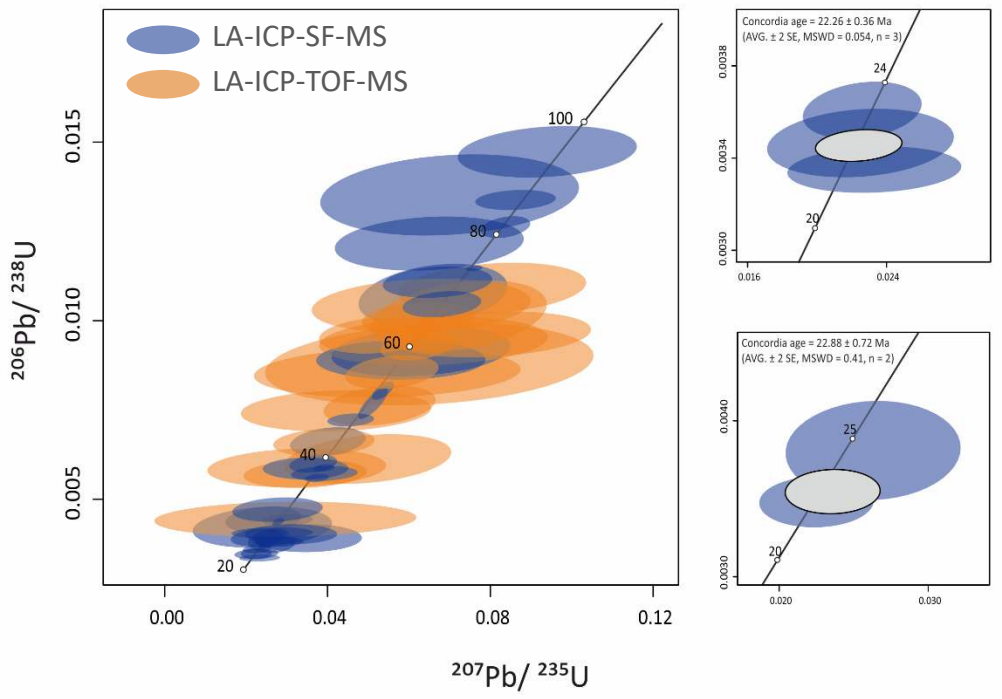
Geochronological Study: Mogok, Myanmar

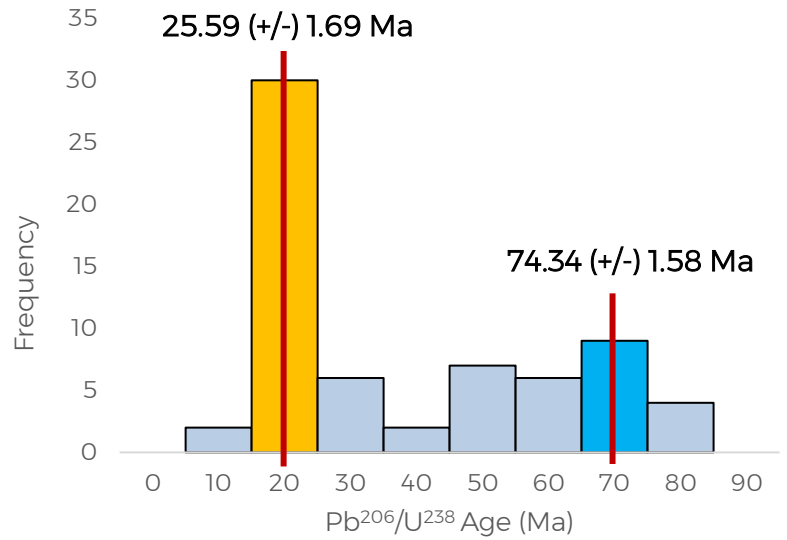
Zircon in Rubies and Spinel from Mogok (Himalayan orogeny)











22.88 ± 0.72 Ma (Spinel)

22.26 ± 0.36 Ma (Ruby)

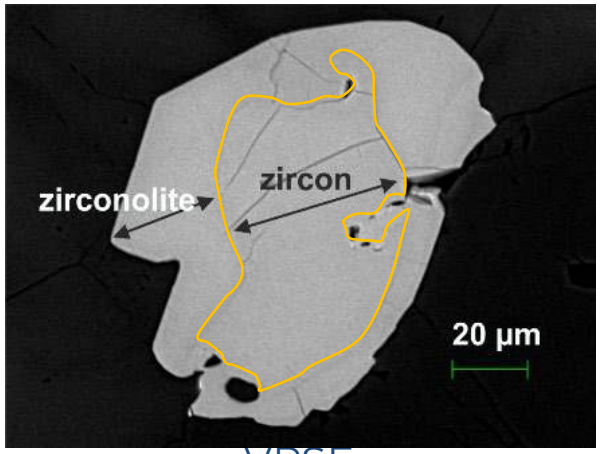
Youngest U-Pb ages of zircon inclusions indicate age of ruby and spinel formation in Mogok; Myanmar.

Data and figures from Myat Myint Phyo, (PhD thesis, University Basel)

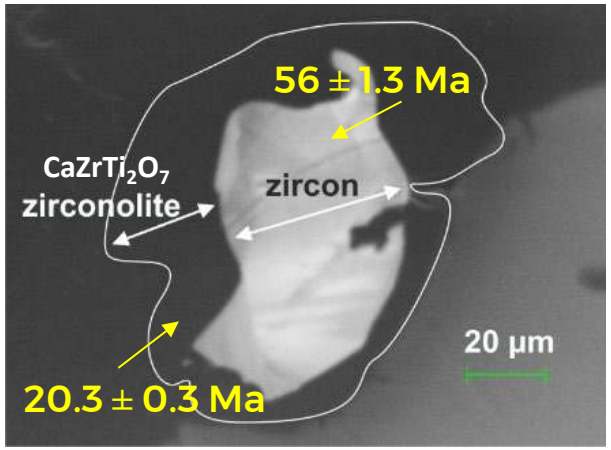
Geochronological Study: Mogok, Myanmar

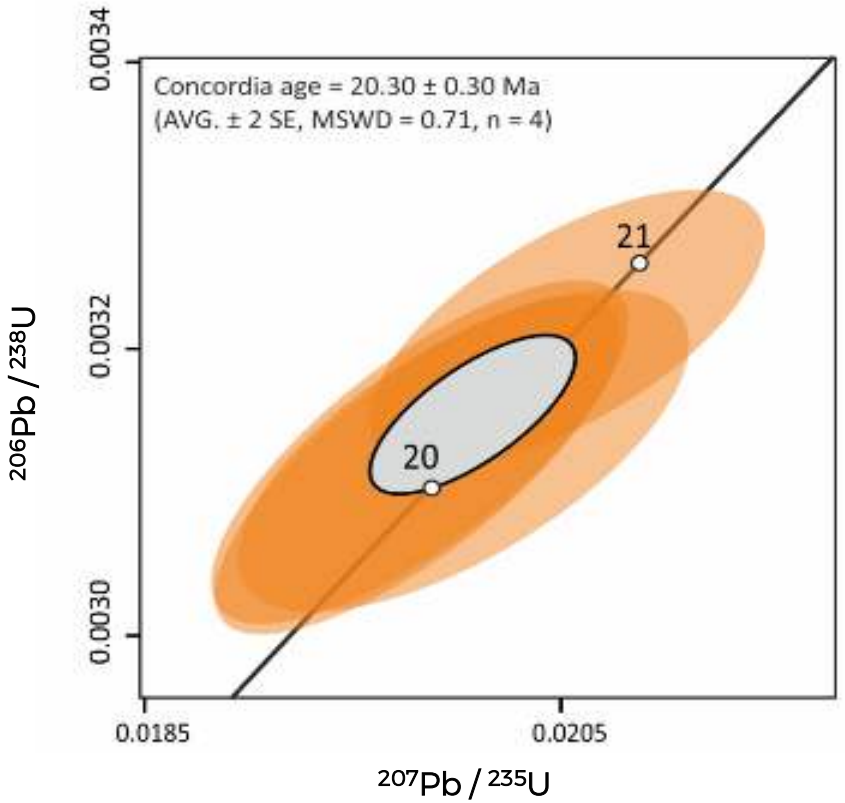
Zirconolite ($CaZrTi_2O_7$) rim grown around zircon inclusion in ruby from Mogok

BSE



VPSE

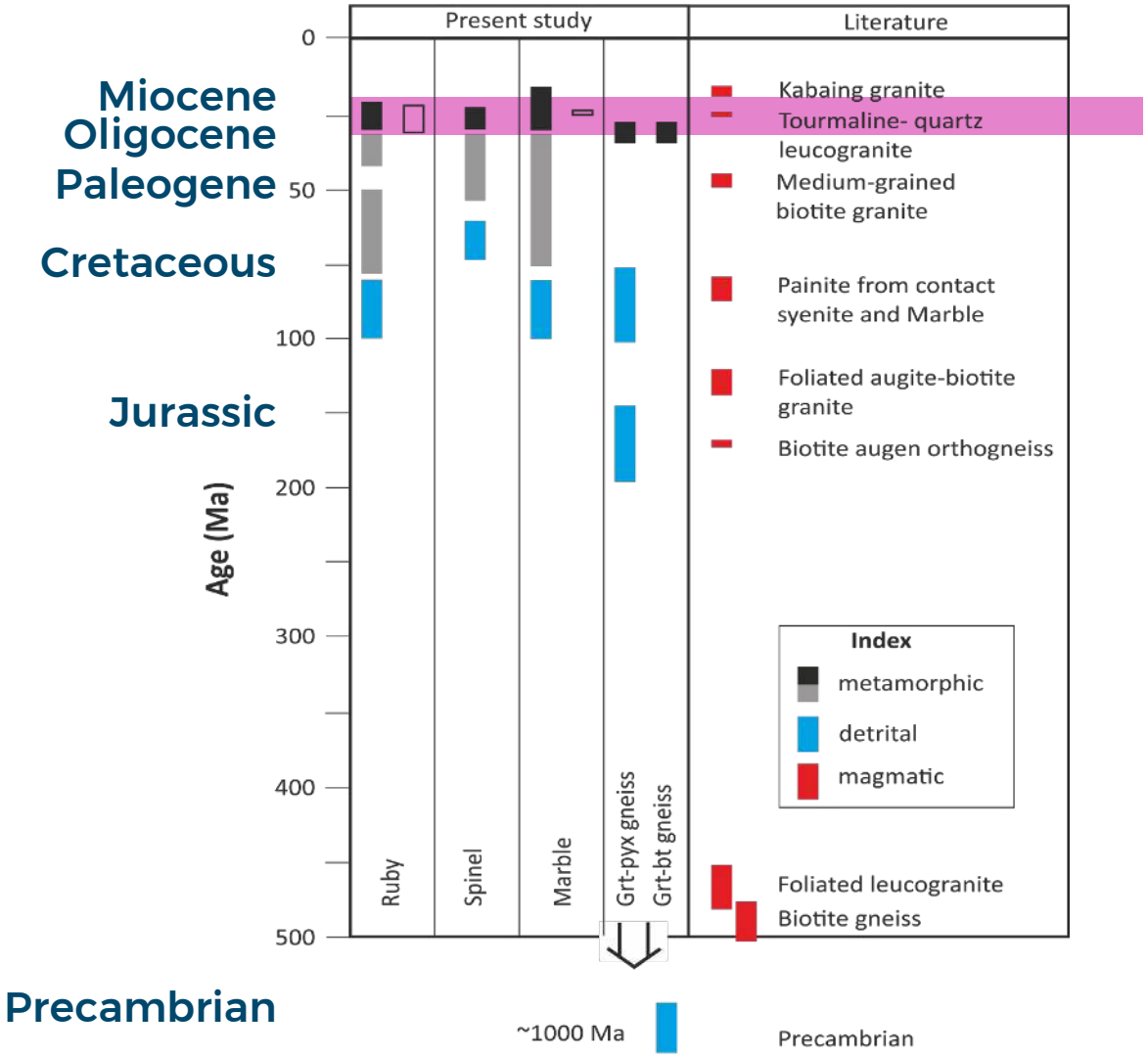




Ruby formation in Mogok at 20 ma years further confirmed by syngenetic zirconolite rim formed on detrital zircon grain.

Data and figures from Myat Myint Phyo, (PhD thesis, University Basel)

Geochronological Study: Mogok, Myanmar



Formation of marble-hosted ruby & spinel in Mogok

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Petrology, geothermobarometry and geochemistry of granulite facies wall rocks and hosting gneiss of gemstone deposits from the Mogok area (Myanmar)

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Garnet-nepheline gneiss
Subduction-related magmatism
Spinel-ruby gem deposit

ABSTRACT

The Mogok Metamorphic Belt (MMB) of Myanmar formed during the Paleogene collision between the West Burma block and the Shan-Thai block. The MMB is mainly composed of medium to high-grade metamorphic marble, calc-silicate rocks, gneiss, quartzite, peridotite and igneous rocks such as granite, syenite and gabbro. The Mogok area in the central part of the MMB is well-known for magnificent quality ruby, spinel, sapphire, and peridot. To unravel the metamorphic PT-conditions prevailing during the formation of spinel and ruby from primary marble deposits in the Mogok area, three different types of high-grade quartz-garnet gneiss from the neighbourhood of gemstone mines were investigated by electron microprobe. Geothermobarometry reveals granulite facies PT-conditions of 756–792 °C at 7.4–7.6 kbar, which is reproduced by Theriak-Domino modelling within the error of both methods at water activities of 0.34–0.4. Shoshonitic and high-K calc-alkaline mafic dykes occur within marble forming conspicuous garnet-nepheline and clinopyroxene-clinoamphibole gneiss. Petrologic and geochemical investigations of these metadykes verify their granulite facies metamorphism and classify them as subduction-related magmatic rocks, which intruded the marble sequences. These investigations as well as previous studies show that spinel and ruby in marble of the Mogok area may have formed not only by metasomatism around alkaline intrusions, but also by granulite facies regional metamorphism.

1. Introduction

Myanmar is well known for numerous deposits of world class precious gemstones. The Mogok area, also known as the Mogok stone tract, hosts the most important gemstone deposits in Myanmar (Iyer, 1953). Since the 15th century, this area is a major source of finest quality ruby, spinel and other gemstones (Iyer, 1953; Hughes, 1997).

In two previous studies we investigated ruby and spinel, which formed in marble and calc-silicate rocks of the Mogok Stone Tract. We found numerous high-grade mineral inclusions in spinel giving evidence for granulite-facies metamorphism (Phyo et al., 2019). Dating of zircon and zirconolite inclusions in spinel and ruby testified to the formation of the gemstones in Oligocene to Early Miocene time (Phyo et al., 2020). The present study is aiming to derive consistent PT-conditions of metamorphism and obtaining fluid conditions during the granulite facies metamorphism, which led to the blastesis of the gemstones. As the thermobarometry of the ruby- and spinel-bearing marble was hampered by the absence of suitable mineral assemblages, we chose three types of high-grade quartz-garnet gneiss from the vicinity of *gem* deposits Aunglan Taung, Bawpadan, Yadanar Kaday Kadar and Kinn in the central and western part of the Mogok area. Furthermore, we investigated mafic dykes and layers occurring in several deposits intercalated with the gemstone-bearing marble, now forming unique nepheline- and clinopyroxene-clinoamphibole gneiss. Here, the aim was to further examine their composition and to define the metamorphic grade these rocks experienced in order to investigate their possible influence on the formation of the gemstones.

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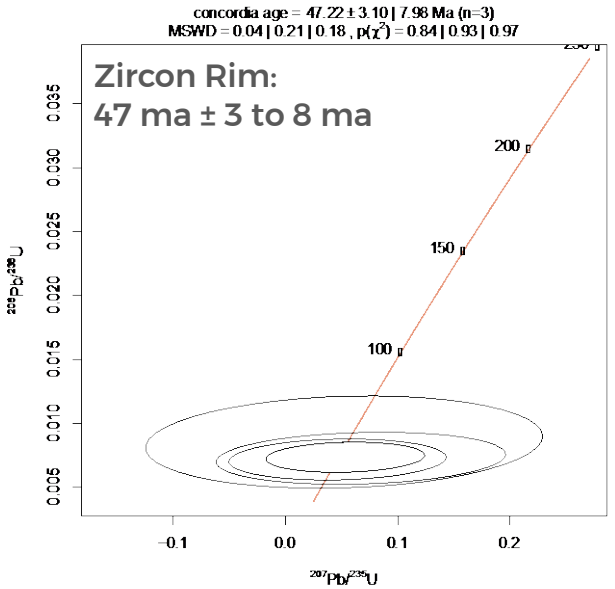
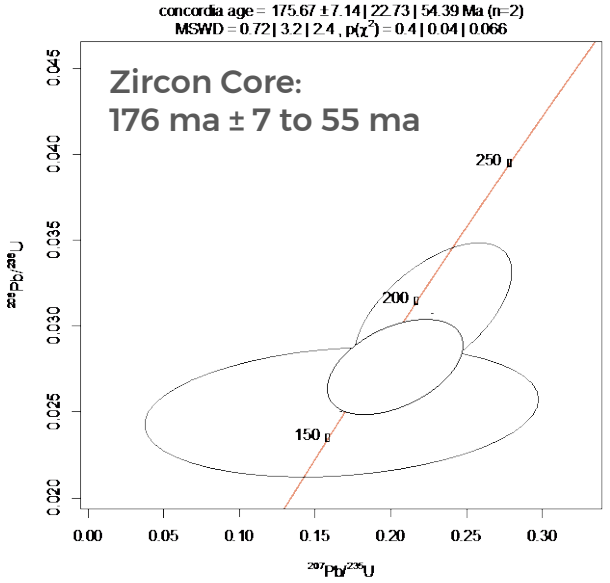
Data and figures from Myint Myat Phyo, (PhD thesis, University Basel) and Phyo et al. 2023

Cobalt-Spinel : Vietnam vs Tanzania

Vietnamese cobalt-spinel

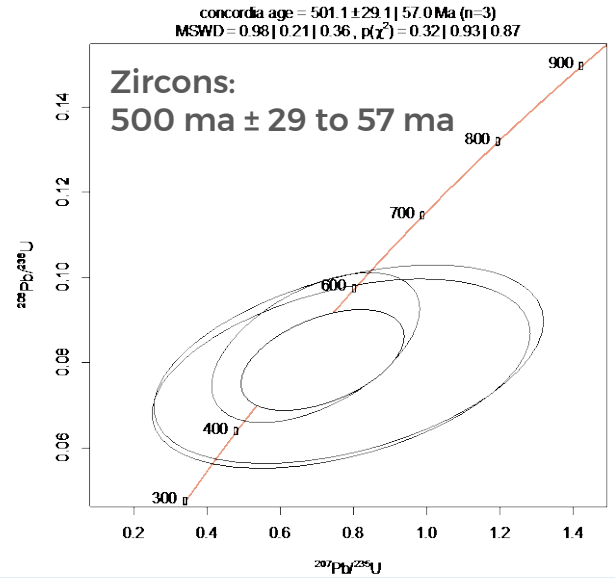
Zircon inclusion with two different ages.
rim: young
detrital core: older.





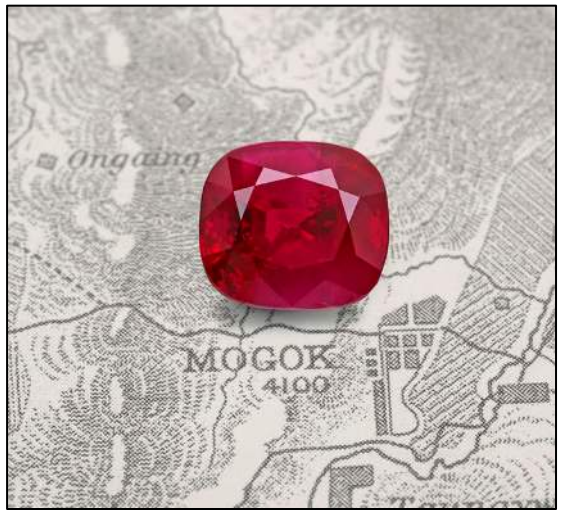
Tanzanian cobalt-spinel

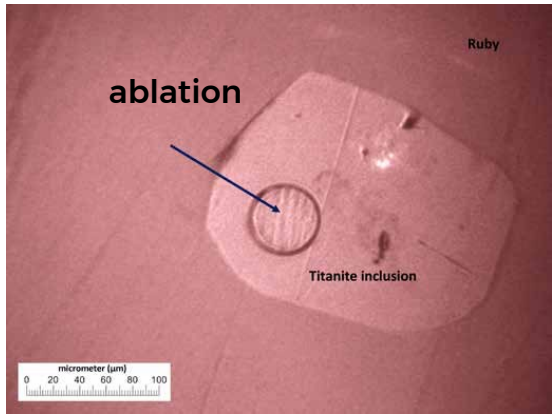
Several **zircon inclusions** analysed with only one ‘old’ age.



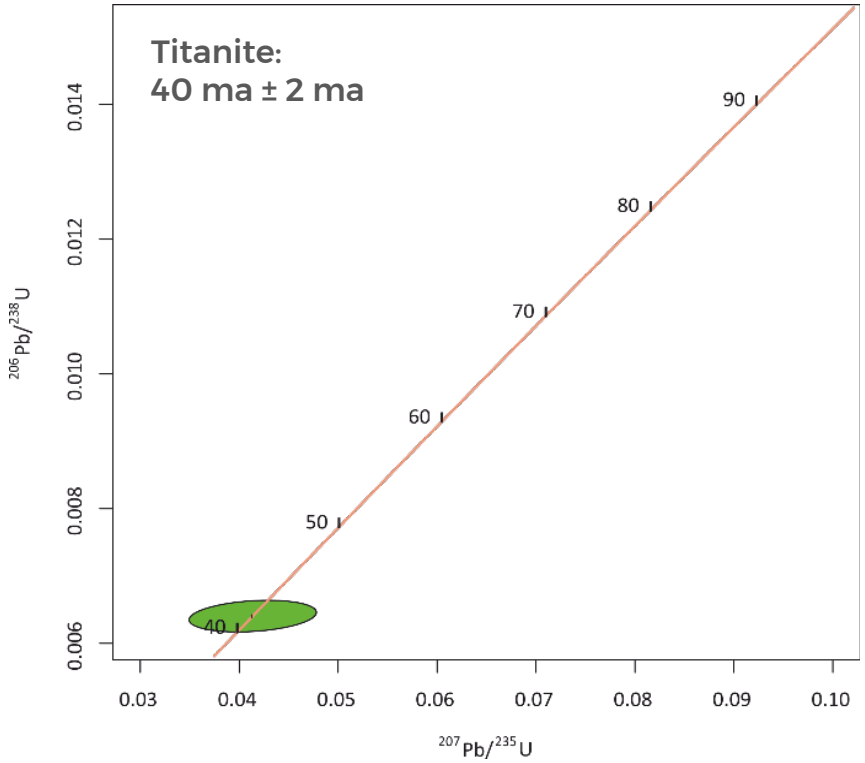
Ruby (marble type) : Burma vs East-Africa

Burmese ruby (12 ct) in Harry Winston necklace



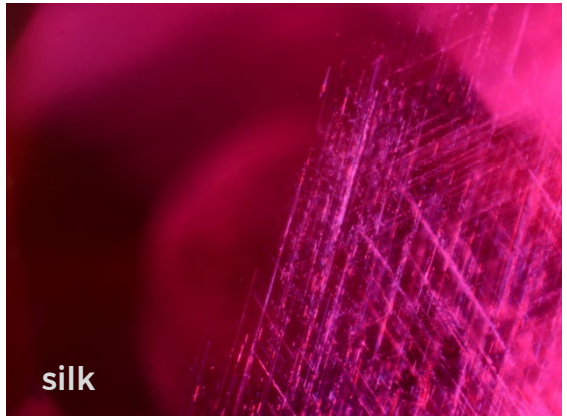


Titanite inclusion with 'young' age (possibly mixed age) predating ruby formation in Mogok.



Ruby (marble type) : Burma vs East-Africa

Ruby (5.1 ct) with zircon inclusion.

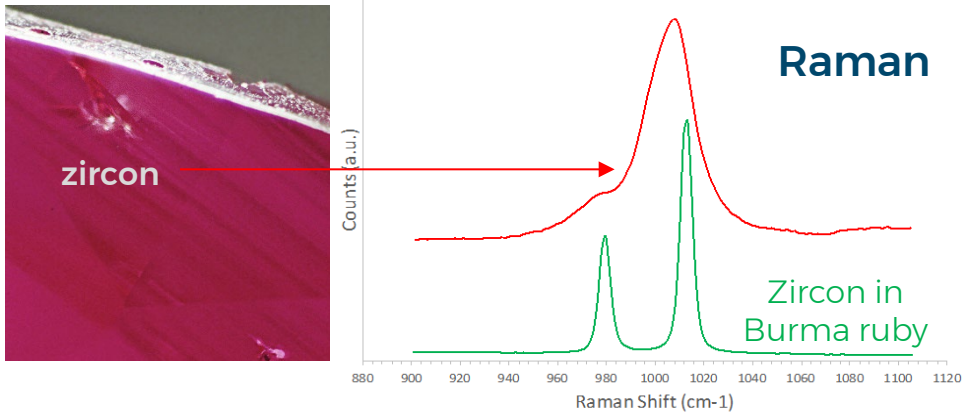


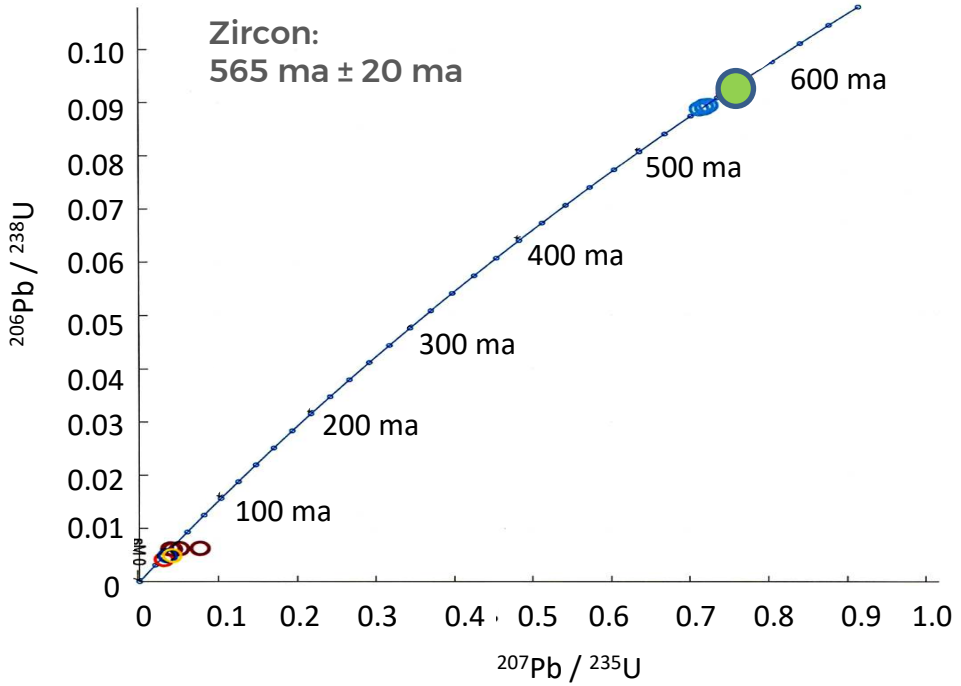
Ruby shows inclusion features similar to Burmese rubies, but **metamict zircon** inclusion (Raman).

Zircon inclusion with 'old' age fitting ruby formation during Pan-African tectonometamorphic events (450-750 ma).

Age support East-African origin of this ruby.

Metamict zircon in EA ruby
$FWHM_{1010}$ = 21.6 cm^{-1}

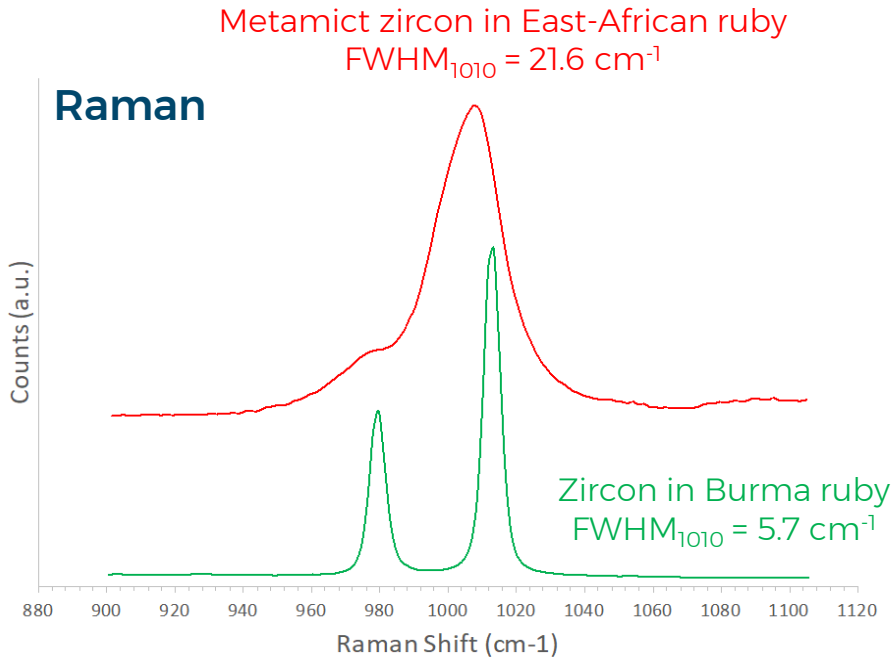


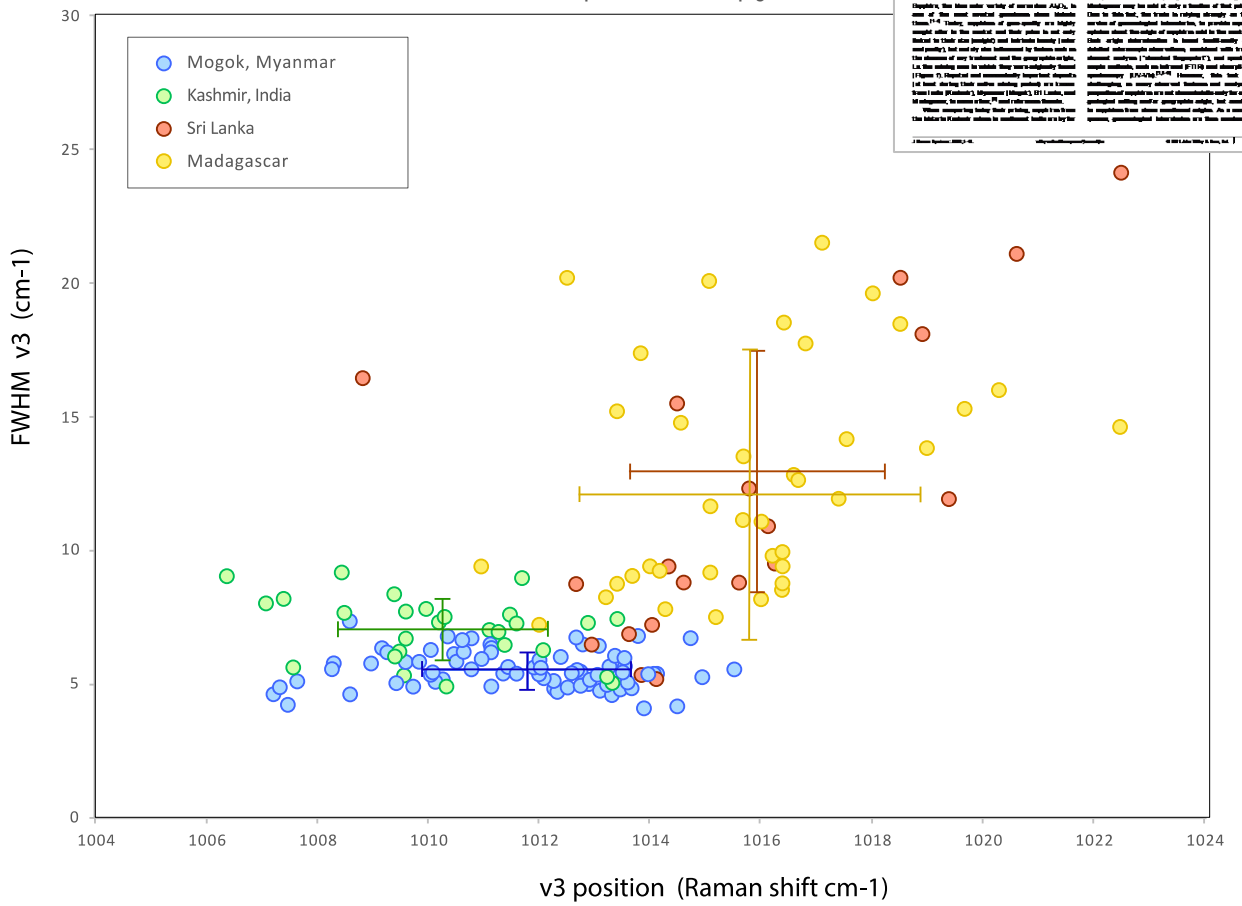


Raman spectra of zircon inclusions

- May assist heat treatment detection
- Provide information about origin
- May be used as indirect age dating (old vs young)

from Xu & Krzemnicki.
Journal of Raman Spectroscopy 2021



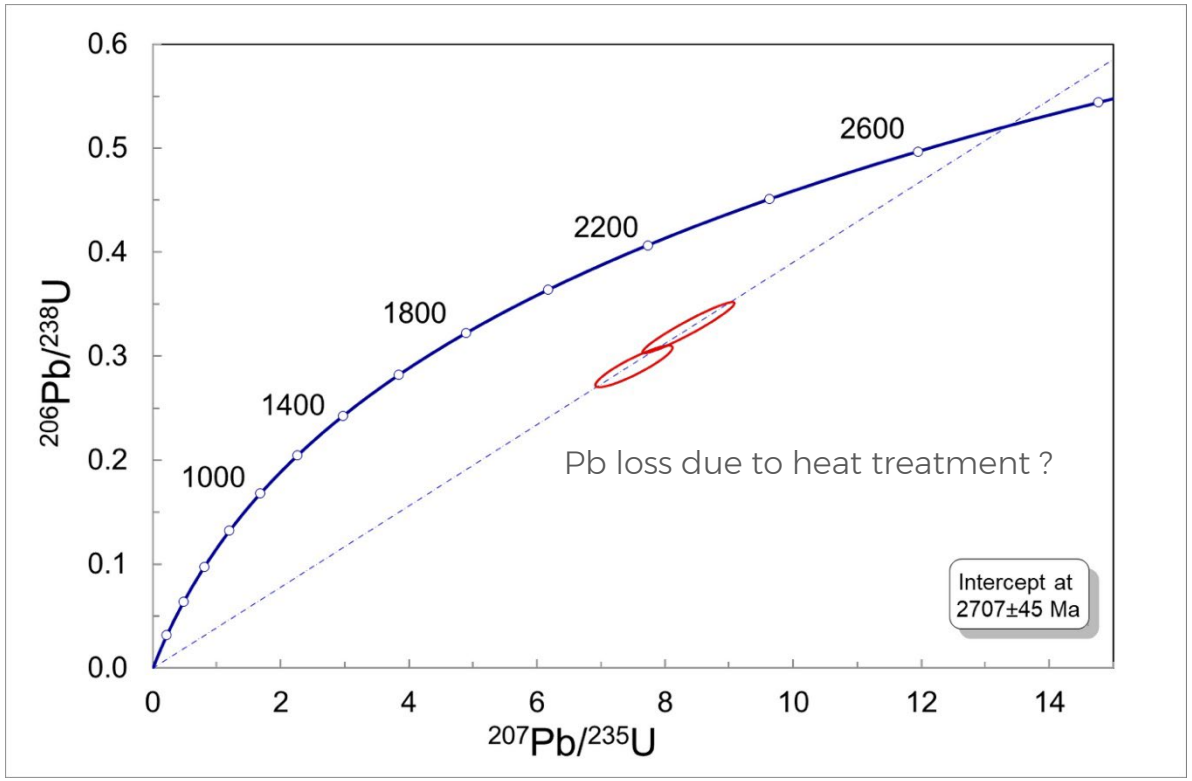


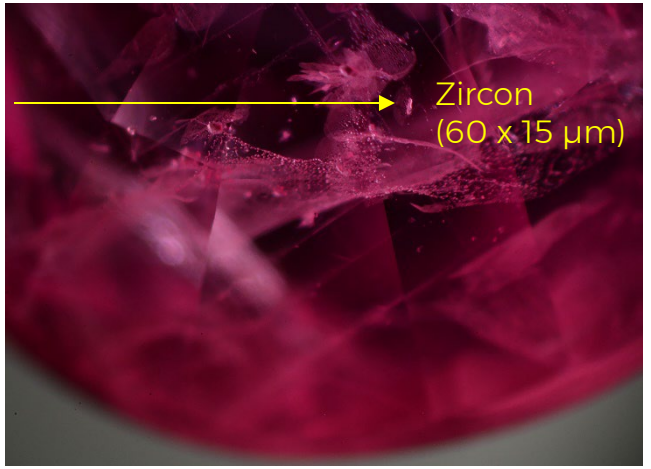
Greenland Ruby

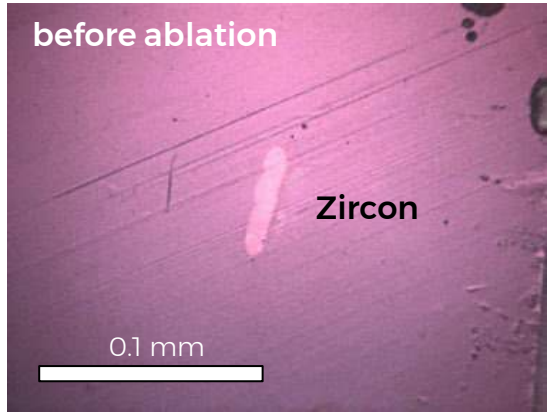
Aappaluttoq ruby deposit in W-Greenland.
Map: Krebs et al. 2019;
Photo: Greenland Ruby A/S.

Greenland ruby of 1.22 ct evident features of heat treatment at high temperature and with significant glassy residues.
Age dating was successful on a **tiny zircon inclusion**.

Greenland Ruby







The tiny zircon revealed an age of **2.7 billion years!**

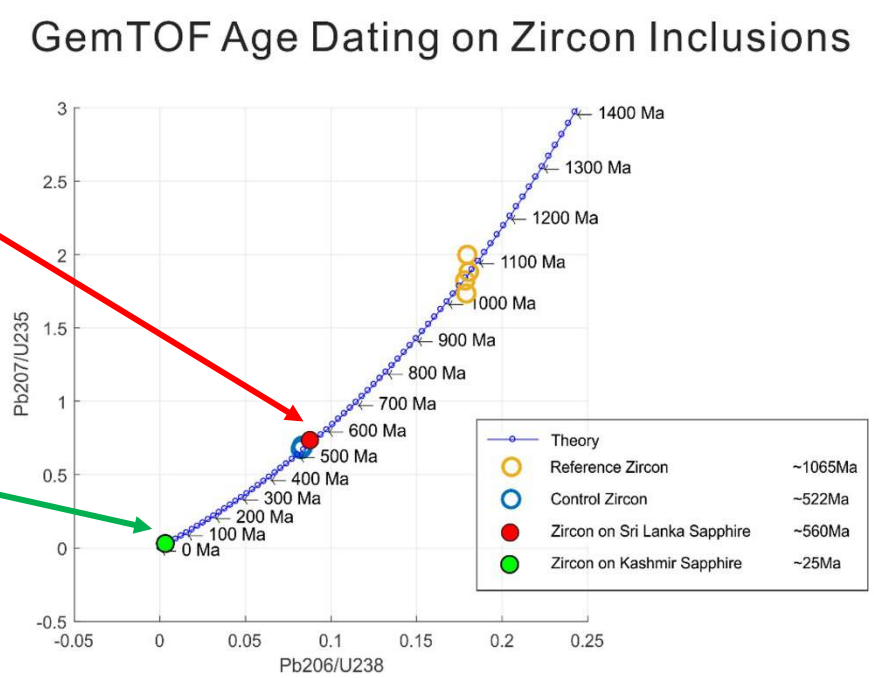
This is well in accordance with literature describing the rubies from Greenland as the oldest ones known on Earth (e.g. Krebs et al. 2019: Pb-Pb isochron age of 2686 +300/-74 ma).

Sapphire: Kashmir vs Sri Lanka or Madagascar

- **U-Pb dating supporting origin determination in the gem lab**

8.5 ct Sri Lanka

22 ct Kashmir

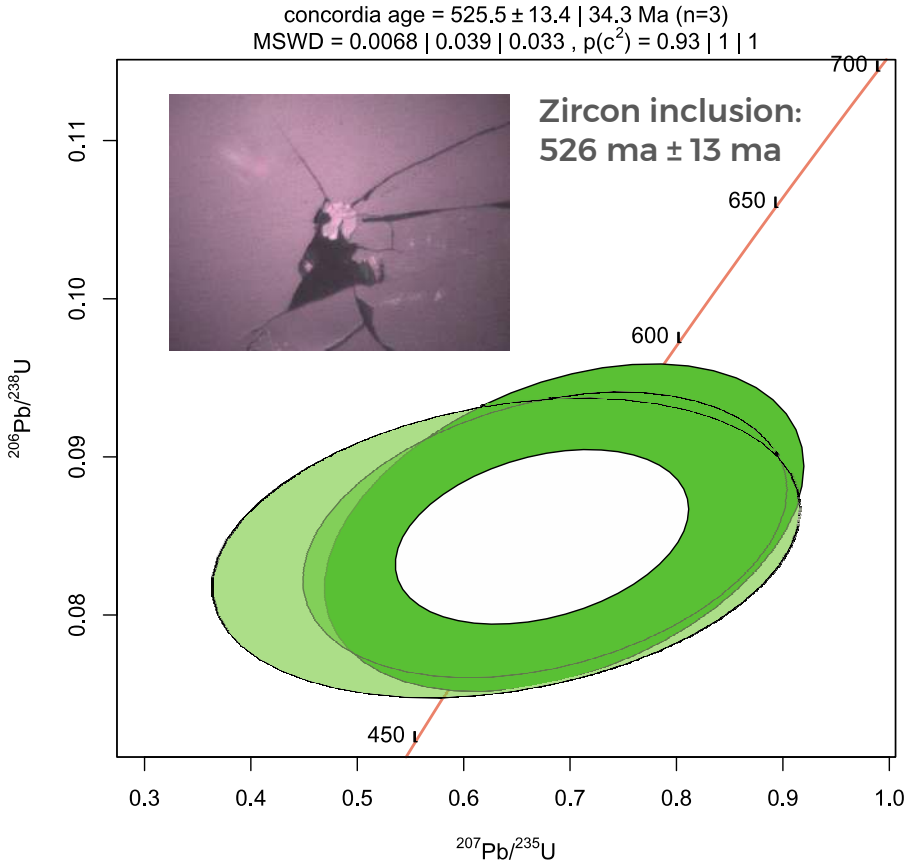


U-Pb dating of two sapphires with velvety blue “Kashmir-like” visual appearance…

Madagascar sapphire, showing a velvety blue colour similar to Kashmir sapphires of best quality!

Sapphire: Kashmir sapphire

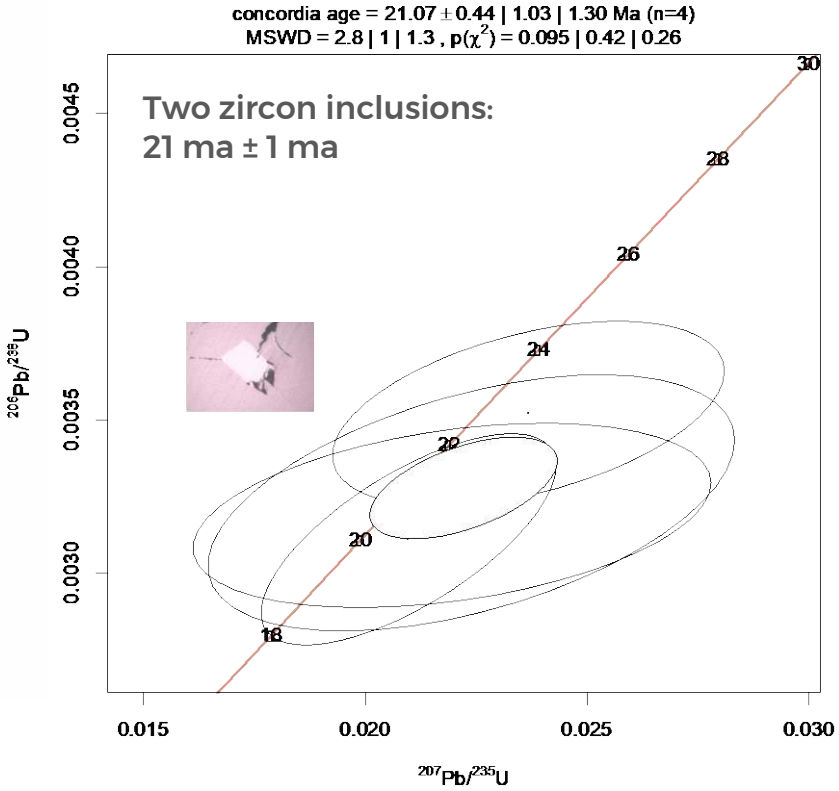
- **U-Pb dating supporting origin determination in the gem lab.**



U-Pb dating on **zircon inclusion** clearly supports Sri Lankan origin of this sapphire. It definitively excludes a Burmese formation.

Sapphire: Kashmir sapphire

- **U-Pb dating for 'story-telling'**



Kashmir sapphire of 43 ct, royal blue.
Sold for US$ 6 million at Christie's New York in December 2020

https://www.christies.com/en/lot/lot-6295931

Direct dating of corundum

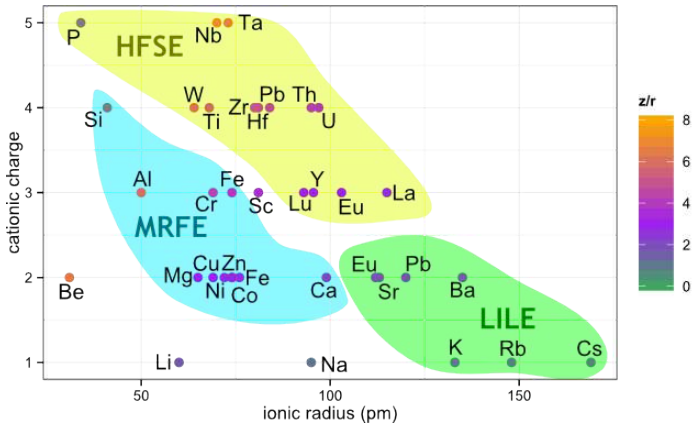
- **^{232}Th - ^{208}Pb decay**

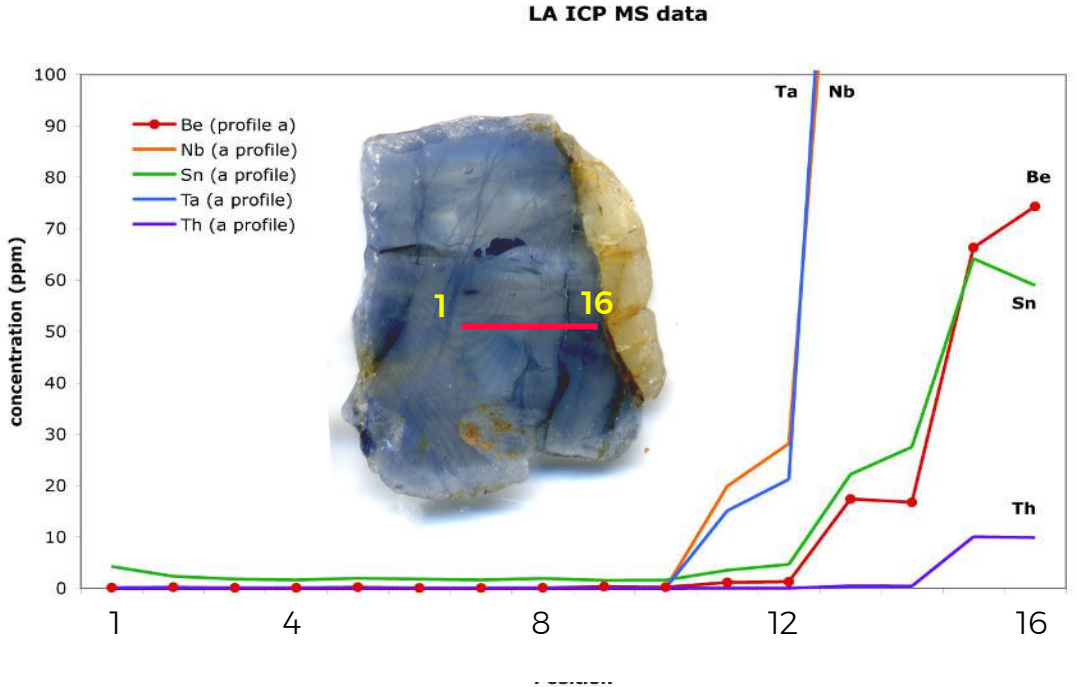
Metamorphic and basaltic sapphires occasionally contain traces of High-Field Strength Elements (HFSE) including Th and Pb.

They are commonly attributed to fine dispersed **nano-inclusions** resulting in “cloudy” appearance (Shen et al. 2007 and 2009, Baldwin et al. 2017).

A new study (Oto et al. 2023) reveals these nano-inclusions are possibly a “srilankite”-like mineral phase (srilankite: $ZrTi_2O_6$) epitaxially intergrown with the corundum matrix.

Th-Pb dating provides rather **rough estimate** of formation age: ‘old’ (e.g. Madagascar) vs ‘young’ (e.g. Kashmir).





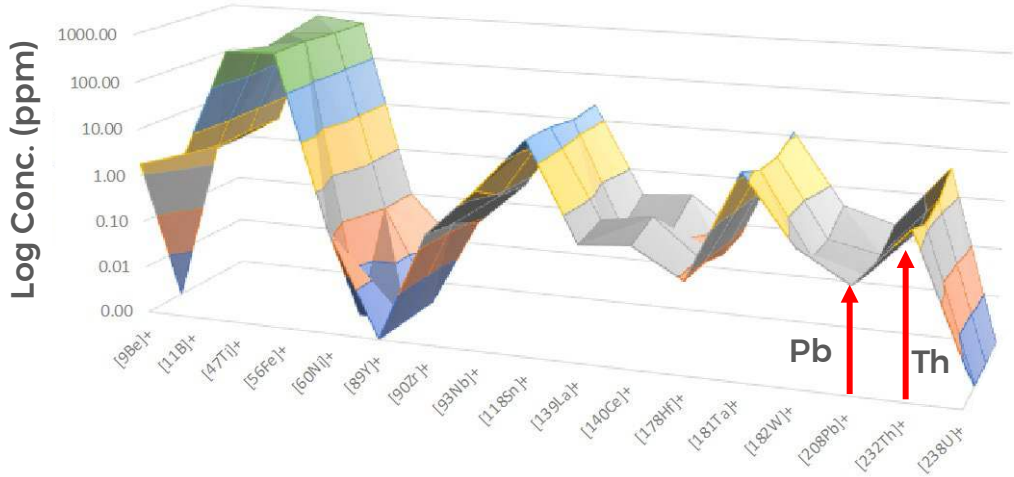
Chrysoberyl-Sapphire (basaltic) intergrowth. See M.S. Krzemnicki, 2008 (IGC Moscow)

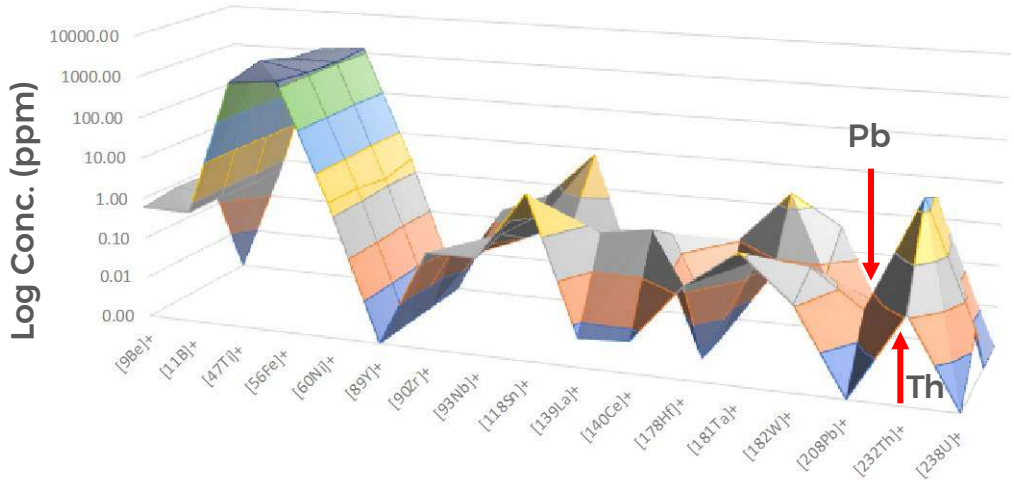
Direct dating of corundum

- ^{232}Th - ^{208}Pb decay

Th-Pb direct dating confirms **Madagascar** and **Kashmir** origin for these two sapphires.

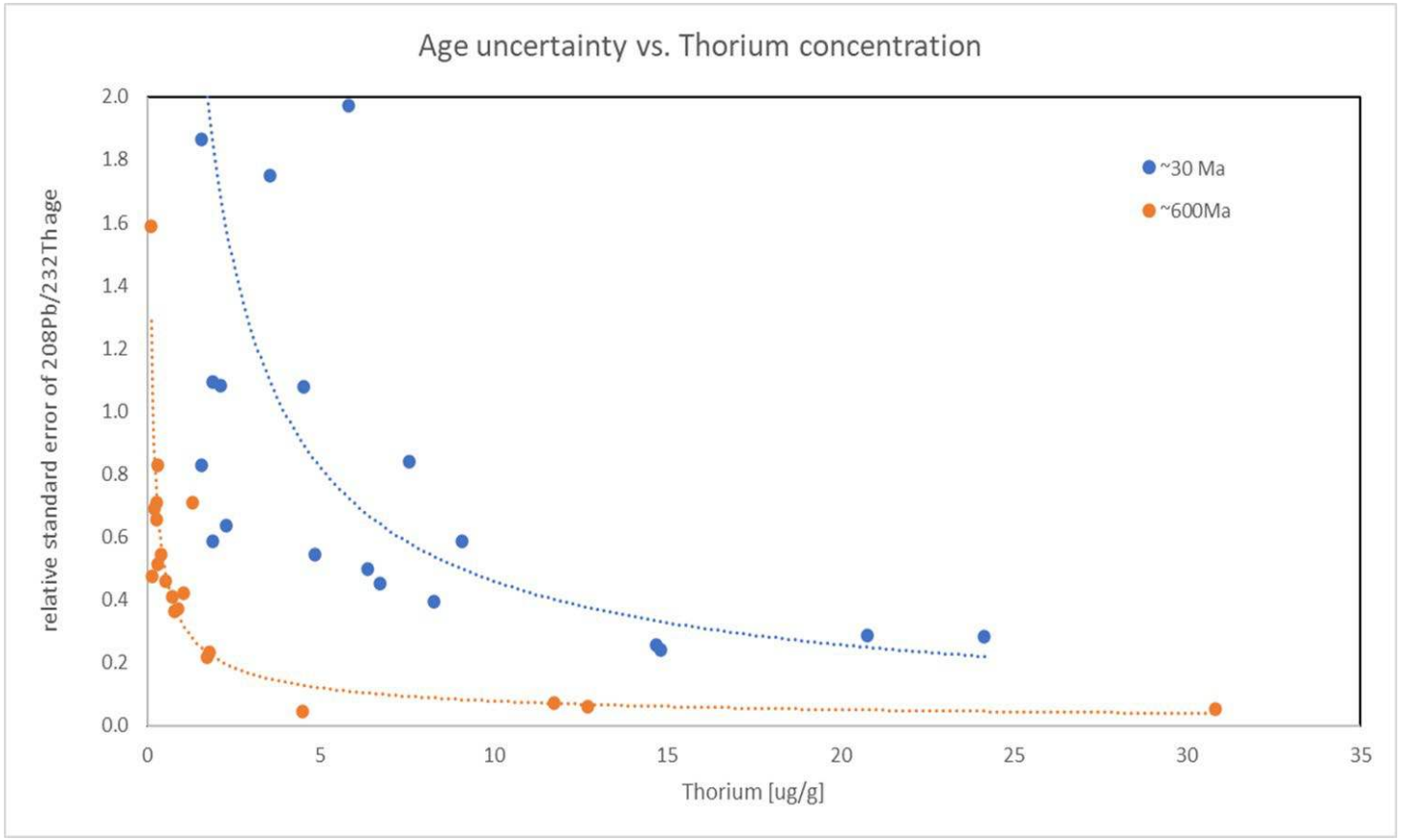
selected trace elements in these sapphires





Direct dating of corundum

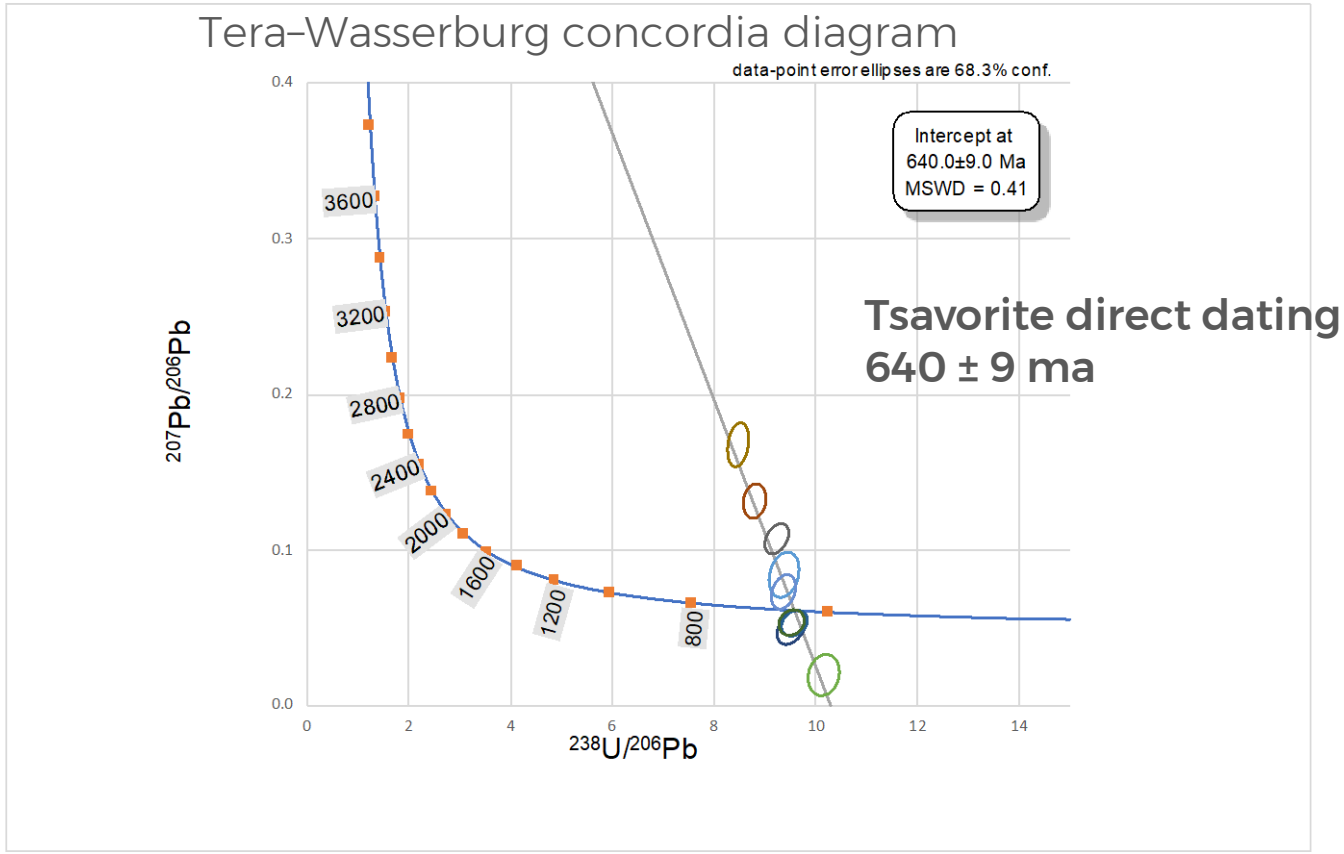
- ^{232}Th - ^{208}Pb decay



- About 2% of corundum samples tested at SSEF contain enough thorium to try direct dating.
- Mostly rough estimate of age ('young' vs 'old'). This may already support origin determination of gemstone.
- Research sample with ~3 ppm Th Pb/Th direct dating: 571 +/- 30 ma Zircon inclusion (Pb/U): 536 +/- 11 ma.

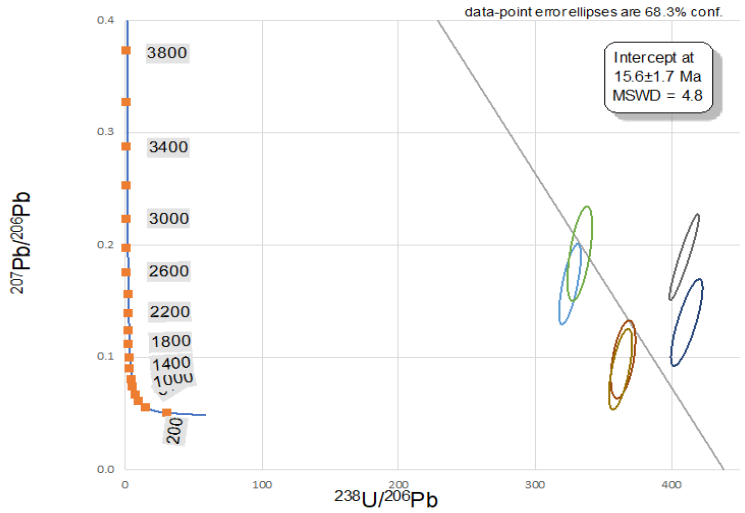
Direct dating of grossular garnet

- **Dating of several tsavorite samples from Gogogogo in Kenya.**

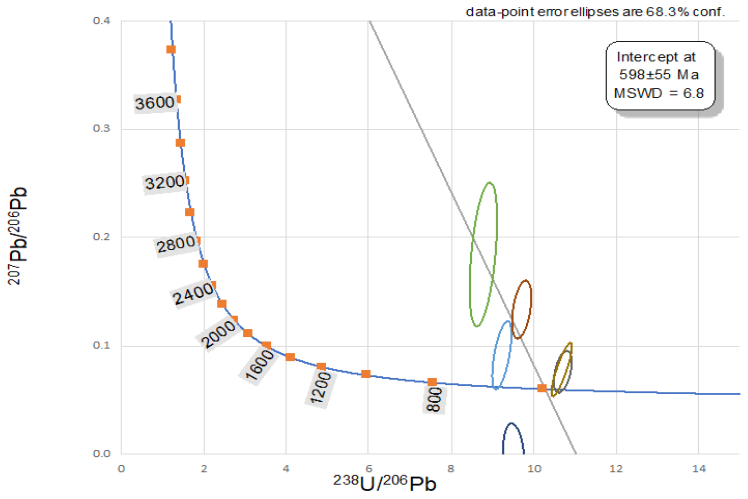


- The Ca-site in Grossular garnet $Ca_3Al_2(SiO_4)_3$ can accomodate large ions (substitutions), among them U, Th, Pb, REEs to name a few.
- Direct dating is possible based on U, Th, and Pb isotope ratios.

Direct dating of grossular garnet



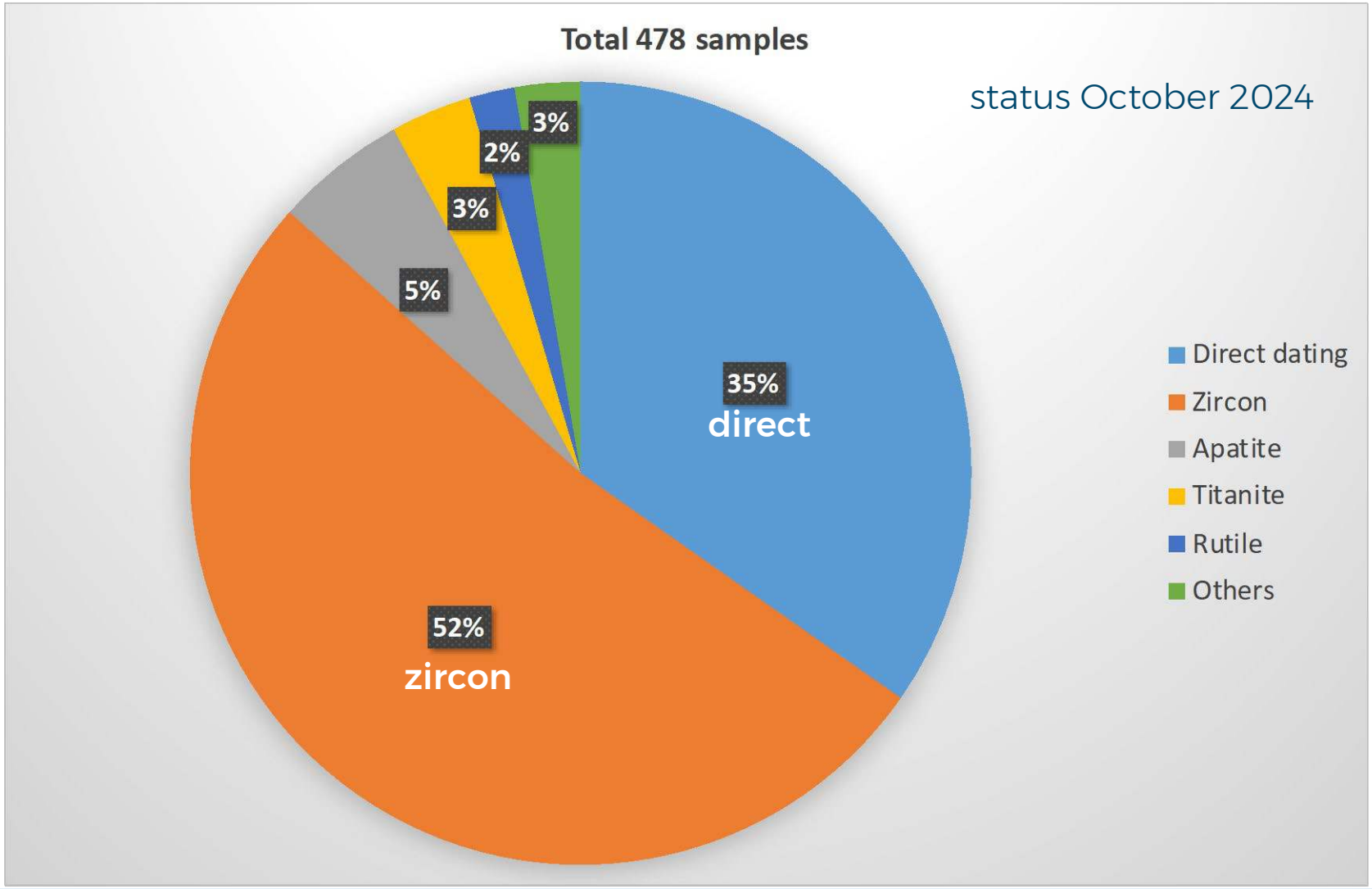
- Pink grossular from Mogok, Myanmar:
  Direct dating 15.6 ± 1.7 ma



- Grossular from Tanga region, NE-Tanzania
  Direct dating 598 ±55 ma

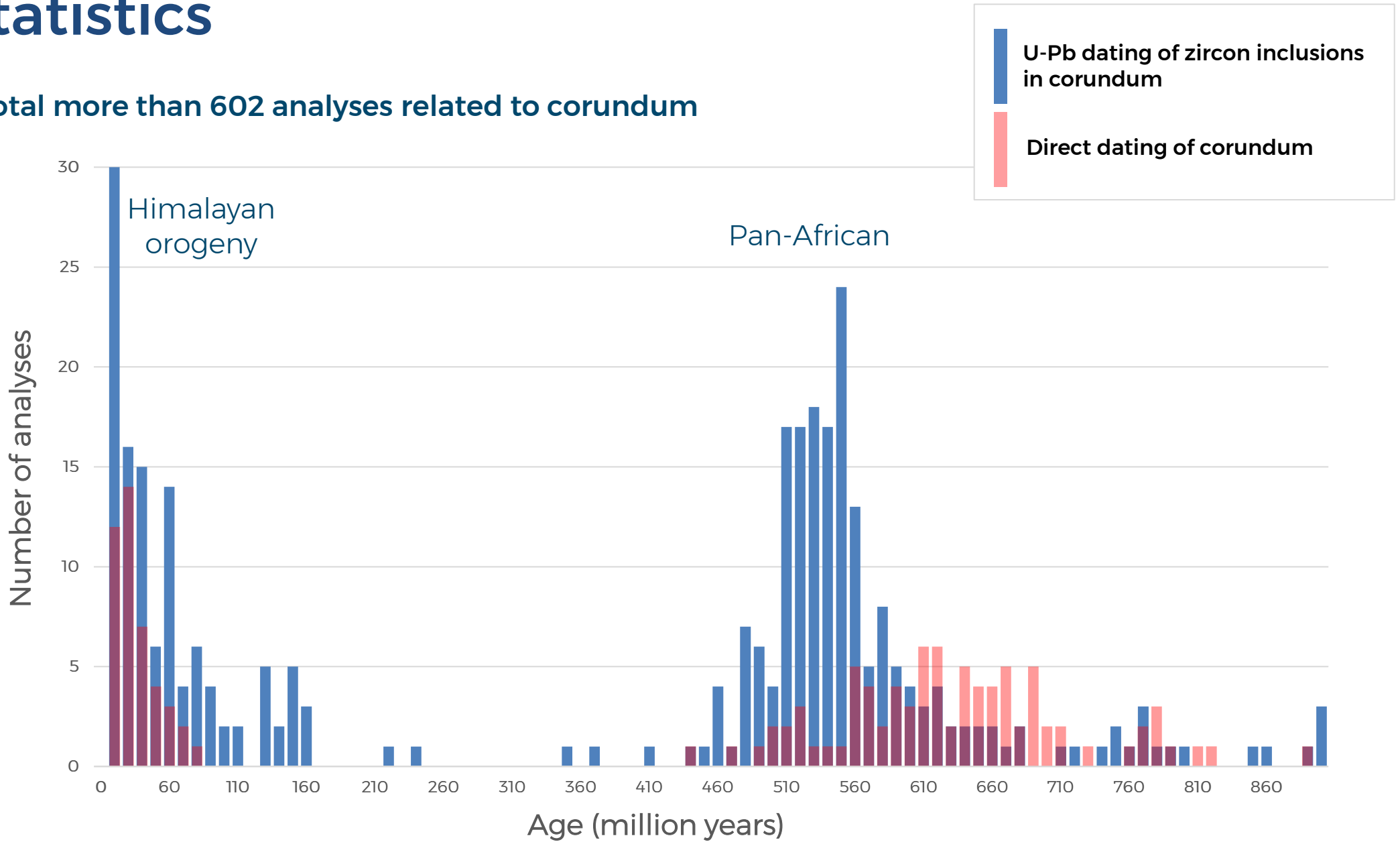
Statistics

- **Direct dating vs dating of inclusions**



Statistics

- **Total more than 602 analyses related to corundum**



Conclusions

- Age dating is applied at SSEF on client stones/pearls since 2017.

- **Radiometric dating** possible on specific **inclusions** (mainly zircon) or by **direct dating**.
  - but only if: inclusion at surface, or U, Th, and Pb present as traces in gemstone.

  - Consequence: not possible to offer as a standard client service.
    if done, then offered for free to client with no extra costs.

  - may help to elucidate geological history of gem deposit
  - may assist origin determination
  - may provide intriguing facts about a gemstone (story-telling)

- **Radiocarbon dating** is **direct dating**, possible for all carbon-rich **biogenic samples**.
  - restriction: not older than about 60‘000 years

  - Consequence: is offered as a client service by SSEF

Thank you for your attention

www.ssef.ch/ssef-facette