# 李秋立佐证材料

## 1. 10 篇代表性论文

| 序号 | 发表期刊 | 发表年度 | 影响因子 | 作者身份 |
|---|---|---|---|---|
| 1 | Earth-Science Reviews | 2021 | 12.4 | 第一作者 |
| 2 | Nature communications | 2021 | 14.9 | 通讯作者 |
| 3 | Chemical Geology | 2021 | 4.0 | 通讯作者 |
| 4 | Journal of Aanlytical Atomic Spectrometry | 2021 | 4.0 | 通讯作者 |
| 5 | Journal of Aanlytical Atomic Spectrometry | 2020 | 4.0 | 通讯作者 |
| 6 | Precambrian Research | 2020 | 4.7 | 通讯作者 |
| 7 | Lithos | 2020 | 4.0 | 通讯作者 |
| 8 | Geostandards and Geoanalytical Research | 2020 | 3.6 | 通讯作者 |
| 9 | Frontiers in Chemistry | 2020 | 5.2 | 通讯作者 |
| 10 | Journal of Aanlytical Atomic Spectrometry | 2018 | 4.0 | 第一作者 |

## 2. 5 项奖励

| 序号 | 奖项 | 排名 | 获得时间 |
|---|---|---|---|
| 1 | 国家自然科学奖二等奖 | 5 | 2010 年 |
| 2 | 中国科学院杰出科技成就奖 | 主要完成者 | 2014 年 |
| 3 | 侯德封青年科学家奖 | 个人奖 | 2014 年 |
| 4 | 中国科学院青促会优秀会员 | 个人奖 | 2015 年 |
| 5 | 第十二届孙贤鉌奖 | 个人奖 | 2018 年 |

## 3. 5 项专利

| 序号 | 类别 | 授权号 | 授权日期 | 排名 |
|---|---|---|---|---|
| 1 | 美国发明专利 | US 11101126 B2 | 2021.8.24 | 3 |
| 2 | 中国发明专利 | ZL 201911367225.X | 2020.10.16 | 2 |
| 3 | 中国发明专利 | ZL 202110057871.7 | 2021.11.16 | 2 |
| 4 | 中国发明专利 | ZL 201711126022.2 | 2018.12.21 | 3 |
| 5 | 中国发明专利 | ZL 201610023625.9 | 2017.9.1 | 2 |

Earth-Science Reviews 221 (2021) 103789

Contents lists available at ScienceDirect

Earth-Science Reviews

journal homepage: www.elsevier.com/locate/earscirev

Review Article

# Detrital zircon U—Pb age distributions and Hf isotopic constraints of the Ailaoshan-Song Ma Suture Zone and their paleogeographic implications for the Eastern Paleo-Tethys evolution

Qiuli Li [a,b], Wei Lin [a,b,*], Yin Wang [a,b,*], Michel Faure [c], Lingtong Meng [a,b], Hao Wang [a,b], Vuong Van Nguyen [d], Hoai Luong Thi Thu [d], Claude Lepvrier [e], Yang Chu [a,b], Wei Wei [a,b], Tich Van Vu [d]

[a] State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Innovation Academy of Earth Science, Chinese Academy of Sciences, Beijing 100029, China
[b] College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, China
[c] Institut des Sciences de la Terre d'Orléans, UMR CNRS 7327, Université d'Orléans, 45067 Orléans Cedex 2, France
[d] Faculty of Geology, Hanoi University of Science, Hanoi, Viet Nam
[e] Institut des Sciences de la Terre de Paris, UMR CNRS 7193, Case 129, Université Pierre & Marie Curie, 75252 Paris Cedex 05, France

ARTICLE INFO

*Keywords:*
Eastern Paleo-Tethys
Ailaoshan-Song Ma Suture
Detrital zircon
In-situ Hf isotopic content
Mélange
Tectonic evolution of SE Asia

ABSTRACT

During the Late Paleozoic to Cenozoic, with the closure of the Tethys oceans, the East and/or Southeast Asia was amalgamated by individual blocks. In order to understand the Paleogeography of the Eastern Paleo-Tethys, a systematic U—Pb dating on 11 samples have conducted to constrain the age and provenance of the elements forming the Ailaoshan-Song Ma mélange. These samples consist of the ophiolitic mélange zone (6 samples), the sedimentary cover of the Indochina block (4 samples), and the post-orogenic continental deposits (1 samples). The achieved detrital zircon age distribution, and the corresponding in-situ Hf isotopic recorder allow us to discuss the provenances of the materials involved in the tectonic processes during the subduction and collision. Three units (M1, M2, M3), which stand for the different segment of the suture zone, could be separated based on the differences in zircon age clusters and Hf isotopic values. These differences might be due to the variety of provenances. Combined with the previous works, especially our work on the Song Chay mélange zone, we concluded that both upper and lower plates are potential provenances for the mélanges between South China block and Indochina block. Along the suture zone, the component of the mélange presents a significant heterogeneity.

## 1. Introduction

Sedimentary provenance analysis is a popular method and an effective tool to study the evolution of an orogenic belt as well as the erosion, the transport and finally the deposition processes in the fore-deep basins (e.g. Fedo et al., 2003; Cawood et al., 2007, 2012; Dickinson and Gehrels, 2009). Ideally, the analyzed sample would completely represent geological history by including evidence of all the possible provenances. Detrital zircon geochronology has received particular attention as a single-mineral provenance tool because the abundance of zircon (in terms of numbers of grains) does not change drastically during sediment transport, owing to the inherent stability of zircon. The in-situ Hf isotope analysis of single zircon grains has proven to be a useful tool for reconstructing the tectonic evolution of continental blocks (Griffin et al., 2000; Condie et al., 2009). Therefore, the age spectra and related geochemistry, established from a large number of detrital zircon grains of igneous origin, could be used to constraint the ages of sedimentary sequences, to assess the provenance, and the routing of sediments through temporal/spatial dimensions. Such an approach requires a good knowledge of the magmatic, metamorphic, and tectonic events that took place in the source regions (Condie et al., 2009). Conversely, the analysis of detrital zircon age spectra might help to improve the

* Corresponding author at: State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Innovation Academy of Earth Science, Chinese Academy of Sciences, Beijing 100029, China.
*E-mail addresses:* linwei@mail.iggcas.ac.cn (W. Lin), wangyin@mail.iggcas.ac.cn (Y. Wang).

https://doi.org/10.1016/j.earscirev.2021.103789
Received 30 March 2021; Received in revised form 30 August 2021; Accepted 31 August 2021
Available online 6 September 2021
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nature COMMUNICATIONS

ARTICLE

https://doi.org/10.1038/s41467-021-26668-z OPEN

# Light oxygen isotopes in mantle-derived magmas reflect assimilation of sub-continental lithospheric mantle material

Jing-Yao Xu [1,2], Andrea Giuliani[3], Qiu-Li Li [1,2,4✉], Kai Lu[1,4], Joan Carles Melgarejo[5] & William L. Griffin [6]

Oxygen isotope ratios in mantle-derived magmas that differ from typical mantle values are generally attributed to crustal contamination, deeply subducted crustal material in the mantle source or primordial heterogeneities. Here we provide an alternative view for the origin of light oxygen-isotope signatures in mantle-derived magmas using kimberlites, carbonate-rich magmas that assimilate mantle debris during ascent. Olivine grains in kimberlites are commonly zoned between a mantle-derived core and a magmatic rim, thus constraining the compositions of both mantle wall-rocks and melt phase. Secondary ion mass spectrometry (SIMS) analyses of olivine in worldwide kimberlites show a remarkable correlation between mean oxygen-isotope compositions of cores and rims from mantle-like $^{18}O/^{16}O$ to lower 'crustal' values. This observation indicates that kimberlites entraining low-$^{18}O/^{16}O$ olivine xenocrysts are modified by assimilation of low-$^{18}O/^{16}O$ sub-continental lithospheric mantle material. Interaction with geochemically-enriched domains of the sub-continental lithospheric mantle can therefore be an important source of apparently 'crustal' signatures in mantle-derived magmas.

[1] State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, 100029 Beijing, China. [2] Innovation Academy for Earth Science, Chinese Academy of Sciences, 100029 Beijing, China. [3] Institute of Geochemistry and Petrology, Department of Earth Sciences, ETH Zurich, 8092 Zurich, Switzerland. [4] College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, 100049 Beijing, China. [5] Department of Mineralogy, Petrology and Applied Geology, Faculty of Earth Sciences, University of Barcelona, 08028 Barcelona, Spain. [6] ARC Centre of Excellence for Core to Crust Fluid Systems (CCFS) and GEMOC, Earth and Environmental Sciences, Macquarie University, Sydney, NSW 2109, Australia. ✉email: liqiuli@mail.iggcas.ac.cn

Chemical Geology 573 (2021) 120177

Contents lists available at ScienceDirect

Chemical Geology

journal homepage: www.elsevier.com/locate/chemgeo

# Erroneous determination of the duration of metamorphism from analysis of overlapping pre-sputtered areas during SIMS U–Pb dating of zircon

Hu Tang [a,b,c], Qiuli Li [a,b,c,*], Yu Liu [a,c], Yonghong Shi [d]

[a] State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China
[b] College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, China
[c] Innovation Academy for Earth Science, Chinese Academy of Sciences, Beijing 100029, China
[d] School of Resources and Environment Engineering, Hefei University of Technology, Hefei 230029, China

ARTICLE INFO

*Keywords:*
Metamorphic zircon
SIMS
U–Pb dating
Overlapping pre-sputtered areas
Age deviation

ABSTRACT

The duration of geological events has distinct implications for geological processes. Secondary ion mass spectrometry (SIMS) U–Pb dating of inherited zircon cores and metamorphic rims is the most common method used to determine the age of a protolith, and the timing and duration of metamorphism. This study investigates the age deviation induced by overlapping pre-sputtered areas during SIMS zircon core–rim U–Pb dating, using eclogite and country-rock granitic gneiss samples from the Shuanghe area in the Dabie Orogen, China. First, we analyzed only the zircon core or rim on each grain. Concordant ages of igneous zircon cores in two orthogneisses are 744 ± 10 and 768 ± 7 Ma, demonstrating that their protoliths were Neoproterozoic igneous rocks. The metamorphic zircon rims in the two granitic gneisses yielded a consistent weighted-mean $^{206}Pb/^{238}U$ age of 216 ± 2 Ma (2 SE; $n = 12$ and 17). Zircon grains in the studied eclogite are all metamorphic and yielded a weighted-mean $^{206}Pb/^{238}U$ age of 224 ± 2 Ma (2 SE; $n = 21$). The different metamorphic ages of the eclogite and orthogneisses suggest that they might be juxtaposed by later tectonic events. After being repolished, zircon grains with apparent core–rim structures from the two granitic gneisses were re-analyzed in the cores and rims of each grain. Due to the small size of the zircon grains, the pre-sputtered areas of the two spots in each zircon overlapped. Compared with the analyses in the first session, the overlapping pre-sputtered areas affected apparent dates for zircon rims by −8.2% to +13.2%. The age deviations of the second analyses correlate with the relative position of the overlaps. The apparent $^{206}Pb/^{238}U$ dates are younger when delta X < 0 (i.e., the X coordinate of the second analysis is smaller than the first analysis), while the apparent $^{206}Pb/^{238}U$ dates are older when delta X > 0. This study proves that erroneous SIMS U–Pb dates can be produced when dating metamorphic zircon rims after analyses of the cores with overlapping pre-sputtered areas. It will lead to an apparently longer and incorrect duration of a metamorphic event.

## 1. Introduction

Zircon occurs in most types of silica-saturated rocks, and commonly has high U and low common Pb contents (Watson et al., 1997). This mineral is characterized by slow diffusion of trace elements, including Pb and U (Lee et al., 1997; Cherniak and Watson, 2001), and is resistant to modification related to later tectono-thermal events (Harley and Kelly, 2007). As such, zircon has become the most important and widely used accessory mineral for dating. In high-grade metamorphic rocks, metamorphic zircon can grow as rims around inherited zircon cores (Rubatto, 2017 and references therein). Dating of zircon cores and rims can reveal the ages of the protolith and metamorphism, respectively. The duration of metamorphism can be estimated from the range of metamorphic zircon rim ages (Vavra et al., 1996; Rubatto et al., 1998; Katayama et al., 2001; Gao et al., 2011; Taylor et al., 2014). Variable durations for geological processes can be related to different tectonic evolution models. For example, fast and slow plate subduction–exhumation may represent the early and the late stages of subduction, respectively (Kylander-Clark et al., 2012). Two cycles of subduction and exhumation within a duration of < 20 Myr can be identified by detailed geochronological and mineral chemical analyses (Rubatto et al., 2011). Fast subduction–exhumation of ultrahigh-

* Corresponding author at: State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China.
*E-mail address:* liqiuli@mail.iggcas.ac.cn (Q. Li).

https://doi.org/10.1016/j.chemgeo.2021.120177
Received 17 November 2020; Received in revised form 11 March 2021; Accepted 12 March 2021
Available online 13 March 2021
0009-2541/© 2021 Published by Elsevier B.V.

JAAS

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View Article Online
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Open Access Article. Published on 19 March 2021. Downloaded on 6/10/2021 1:40:46 PM.
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Cite this: *J. Anal. At. Spectrom.*, 2021, **36**, 999

# $^{238}$U–$^{206}$Pb dating of U-series disequilibrium zircons by secondary ion mass spectrometry†

Yong-Shu Huang,[abc] Qiu-Li Li,[*abc] Yu Liu,[ac] Ping-Ping Liu,[d] Sun-Lin Chung[e] and Xian-Hua Li[abc]

Received 15th December 2020
Accepted 17th March 2021

DOI: 10.1039/d0ja00510j

rsc.li/jaas

Determining the timescales of magma accumulation and storage prior to large eruptions is fundamental to understand the pre-eruptive history of magma reservoirs. Zircon $^{238}$U–$^{230}$Th dating has been widely used to recover the timescales of magma reservoirs younger than 400 ka. However, theoretically and practically, the uncertainties for zircons older than *ca.* 150 ka, *i.e.*, two half-lives of $^{230}$Th, are large because ($^{238}$U)/($^{230}$Th) activities start to stabilize. On the other hand, the accumulation of radiogenic $^{206}$Pb begins to increase significantly after two half-lives of $^{230}$Th. Herein we theoretically demonstrate a calibration method for SIMS (Secondary Ion Mass Spectrometry) $^{238}$U–$^{206}$Pb dating within the timescales of U-series disequilibrium and apply it to zircons of the 75 ka Youngest Toba Tuff (YTT) in northern Sumatra, Indonesia. The $^{238}$U–$^{206}$Pb dates vary from *ca.* 665 to 75 ka after common Pb and initial $^{230}$Th disequilibrium corrections. This age range agrees with that obtained by previous studies using the $^{238}$U–$^{230}$Th method, but the precision of each age is improved by a factor of three or more. $^{238}$U–$^{206}$Pb and $^{238}$U–$^{230}$Th dating methods have advantages and disadvantages for zircons in U-series disequilibrium. The $^{238}$U–$^{230}$Th method is more suited to samples younger than 150 ka, but requires a large Th/U fractionation between the zircon and magma, and a high U content. The $^{238}$U–$^{206}$Pb method is more appropriate for samples older than 150 ka, but requires high U and low common Pb contents. Our approach improves the precision of *in situ* dating of zircon in U-series disequilibrium, which will contribute to investigations of the magmatic evolution beneath young volcanoes.

## 1. Introduction

Zircon is the most widely used U–Pb geochronometer because: (1) it contains abundant U and Th and does not incorporate Pb during crystallization; (2) it is very stable and resistant to disturbance by later hydrothermal activity and weathering; (3) it is widely distributed in most crustal rocks; (4) it can be used to investigate the history of the lithosphere together with a range of geochemical proxies. Given these properties, the U–Pb dating of zircon in young volcanic rocks can be used to investigate magmatic evolution in crustal magma chambers.[1–4]

For young samples within the time-scale of U-series disequilibrium, radiogenic lead is only a part of radiogenic daughters decayed from uranium because of a number of intermediate nuclides with varying half-lives. In a U-bearing magmatic system that has remained undisturbed for ~0.5 Ma, a state of secular equilibrium becomes established between the abundance of parent and daughter nuclides in the U-series decay chain, such that the decay rate (or "activity") of each intermediate daughter nuclide in the chain is equal to that of the parent.[5] During the crystallization of zircon in magma, nuclides in the U-series decay chain can become fractionated relative to one another, due to their different chemical properties or the lattice sites they occupy. This results in U-series disequilibrium, which can be utilized as dating tools. The $^{238}$U–$^{230}$Th system is most widely used for the study of igneous systems that are in U-series disequilibrium.[1,6,7] The applicable age range of $^{238}$U–$^{230}$Th dating is *ca.* 400 ka, based on the assumption that its activity will be within the error of secular equilibrium after five half-lives of $^{230}$Th. However, the dating uncertainty will increase gradually as the ($^{238}$U)/($^{230}$Th) activities begin to change less after two half-lives of $^{230}$Th (*ca.* 150 ka). Interestingly, the abundance of radiogenic $^{206}$Pb in zircon begins to surpass that of $^{230}$Th at that time. Though $^{238}$U–$^{206}$Pb dating was never confidentially applied to date zircons within the time scale of U-series disequilibrium, it has potential to improve

[a]State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China. E-mail: liqiuli@mail.iggcas.ac.cn

[b]College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, China

[c]Innovation Academy for Earth Science, Chinese Academy of Sciences, Beijing 100029, China

[d]Key Laboratory of Orogenic Belts and Crustal Evolution, School of Earth and Space Sciences, Peking University, Beijing 100871, China

[e]Institute of Earth Sciences, Academia Sinica, Taipei

† Electronic supplementary information (ESI) available. See DOI: 10.1039/d0ja00510j

JAAS

PAPER

Check for updates

Cite this: *J. Anal. At. Spectrom.*, 2020, **35**, 489

# Allanite U–Th–Pb geochronology by ion microprobe†

Xin Liao,[abc] Qiu-li Li,*[abc] M. J. Whitehouse,[d] Yue-heng Yang[ac] and Yu Liu[ac]

Received 13th December 2019
Accepted 6th January 2020

DOI: 10.1039/c9ja00426b

rsc.li/jaas

Allanite, an epidote group mineral, occurs as a common accessory mineral in igneous, metamorphic rocks and hydrothermal deposits. It contains radioactive elements Th and U, making it a valuable U–Th–Pb geochronometer for various geological processes. Due to the structural and compositional complexity of allanite, *in situ* dating method is a better choice when the allanite is heterogeneous or has crystallized in multi-stage events. The wide variation of chemical compositions, however, raises the potential for matrix effects and complicates the use of *in situ* methods for allanite U–Th–Pb chronology. To address this issue, we made a comprehensive investigation on allanite U–Th–Pb chronology using Secondary Ion Mass Spectrometry (SIMS). Five allanite samples (CAP, Daibosatsu, SQ-51, Toba OTT, TARA allanite) with variable elemental compositions and common lead concentrations have been used. Allanites with variations in FeO (from 12.8 to 16.1 wt%) and similar $ThO_2$ contents (around 1–2%) do not show significant matrix effects, as suggested by a previous study. While, obvious changes in calibration parameters ($UO_2^+/U^+$, $ThO_2^+/Th^+$) were noticed for allanites with a large range of Th contents (from 300 ppm to 2.0 wt%). By employing power law relationships between $Pb^+/U^+$ *versus* $UO_2^+/U^+$, $Pb^+/Th^+$ *versus* $ThO_2^+/Th^+$ with suitable exponentials, we do not observe obvious matrix effects with $ThO_2$ concentrations variations. This study demonstrates that allanite can be a good geochronometer for multi-stage mineralization of hydrothermal deposits, providing valuable complementary information to zircon and monazite.

## 1. Introduction

Allanite [$(Ca,REE,Th)_2(Fe^{2+},Al)_3Si_3O_{12}(OH)$] is a common LREE-bearing mineral in the continental crust, defined by Giere and Sorenson[1] with REE + Th content ≥ 0.5 atoms per formula unit (apfu), or CaO content < 20 wt%. As an epidote-group mineral, allanite has varied elemental and isotopic compositions, predominantly controlled by the crystallization environment (*e.g.* melt/fluid[2,3]). Allanite usually incorporates a high concentration of Th and moderate amounts of U into its crystal structure, which gives it potential for a useful U–Th–Pb chronometer despite its tendency to incorporate non-negligible levels of common Pb. Compared with other minerals suitable for U–Pb chronology, allanite can crystallize in a wider range of P–T conditions. This property is presumably due to its compositional variability and propensity to incorporate substantial trace elements.[1,4] For example, allanite occurs in most granites and pegmatites like zircon and monazite, but also in different types of mid-low grade metamorphic rocks and hydrothermal deposits.[2,5,6] Hence allanite could be a good geochronometer and supply complementary information to zircon and monazite.

Previous studies have been conducted on dating allanite using isotope dilution methods,[7–9] but in some cases, these results are inaccurate due to compositional and textural complexities. *In situ* analyses have also been conducted by LA-ICP-MS and ion microprobe to eliminate the intricate pretreatment processes and avoid the altered or damaged zones.[10–13] Normally, the ion microprobe method has potential to achieve a better spatial resolution and precision over LA-ICP-MS when applied to complex geological processes like multi-stage mineralization. There were different understandings about matrix effect during SIMS allanite Th–Pb dating. Catlos, *et al.*[10] conducted allanite Th–Pb dating using a CAMECA IMS 1270 and suggested a significant matrix effect due to the variation of Fe concentrations. In contrast, Gregory, *et al.*[11] performed a similar investigation using SHRIMP and concluded that no matrix effects were induced by Fe contents on Th–Pb ages. Moreover, there was no discussion about matrix effects because of variations in Th and U contents.

[a]*State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing, 100029, China. E-mail: liqiuli@mail.iggcas.ac.cn*

[b]*College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, China*

[c]*Innovation Academy for Earth Science, Chinese Academy of Sciences, Beijing, 100029, China*

[d]*Swedish Museum of Natural History, Box 50007, SE-104 05 Stockholm, Sweden*

† Electronic supplementary information (ESI) available. See DOI: 10.1039/c9ja00426b

Precambrian Research 342 (2020) 105695

Contents lists available at ScienceDirect

Precambrian Research

journal homepage: www.elsevier.com/locate/precamres

# Young ages from old garnet in the polyphase metamorphic terrane of the Limpopo Belt, South Africa

Tian Zhou[a,b,c], Qiuli Li[a,b,c,*], Chaofeng Li[a,b], Alfred Kröner[d]

[a] State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China
[b] Innovation Academy for Earth Science, Chinese Academy of Sciences, Beijing 100029, China
[c] College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, China
[d] Institute of Geosciences, University of Mainz, 55099 Mainz, Germany

ARTICLE INFO

*Keywords:*
Garnet
Sm–Nd isochron
*P–T–t* path
Limpopo belt
Polyphase metamorphism

ABSTRACT

The *P–T–t* path of high-grade metamorphic rocks is significant for understanding vertical motions and heating of the crust. In general, garnet is used to constrain *P–T* paths and metamorphic zircon is used to obtain *t* for different metamorphic stages. However, this approach may be complicated by polyphase tectonothermal events. The Central Zone of the Limpopo Belt in South Africa is a complex Precambrian metamorphic terrane that has experienced three distinct high-grade metamorphic events at 3.22 Ga ($M_1$), 2.62 Ga ($M_2$), and 2.02 Ga ($M_3$), based on zircon dating. A previous investigation of a metapelite enclave in the Bulai pluton in the Central Zone showed that metamorphic zircon in the enclave recorded mainly $M_2$ granulite-facies metamorphism and was "protected" by the Bulai pluton. However, titanite and rutile U–Pb ages of the same sample record the $M_3$ event. Here we investigated garnet Sm–Nd isotopic systematics of four metapelite samples from the Evelyn enclave in the Bulai pluton. We constructed single mineral Sm–Nd isochrons for garnet using micro-samples, in order to avoid the risk of isotopic disequilibrium between different minerals or whole-rocks. Metamorphic zircon with sillimanite inclusions has yielded a granulite-facies metamorphic age of ca. 2.6 Ga. However, the Sm–Nd isotopic data for the garnet record much younger ages of ca. 2.03 Ga. These ages are consistent with the $M_3$ event in this area, but were rarely identified in previous zircon dating studies. This suggests that the $M_3$ event reached sufficiently high temperatures to totally reset the rutile and titanite U–Pb and garnet Sm–Nd systems. Moreover, the decoupling of the age of garnet formation and the date it records needs to be carefully considered when reconstructing *P–T–t* paths, and again highlights the usefulness of garnet in the study of polyphase metamorphic terranes.

## 1. Introduction

Deciphering the tectonic–metamorphic evolution of high-grade metamorphic rocks is challenging, especially when such rocks have been subjected to polyphase metamorphism. Constructing a detailed *P–T–t* path makes it possible to quantitatively describe the heating events during metamorphism. Zircon geochronology is important in reconstructing such metamorphic events, due to its inert nature and high diffusion temperature for Pb (Cherniak and Watson, 2001). However, zircon may not form during late metamorphic events (Taylor et al., 2016; Rubatto, 2017 and reference therein), which makes it difficult to recognize all the metamorphic events that have affected a rock. Some accessory minerals, such as rutile and titanite, record cooling that is unrelated to the peak metamorphic event due to the relatively low closure temperatures of Pb in these minerals (Mezger et al., 1989; Cherniak, 2000; Li et al., 2003, 2011; Zack and Kooijman, 2017) or the influence of later fluid-assisted recrystallization (Frost et al., 2000; Romer and Rötzler, 2003). Furthermore, it is challenging to directly link zircon, rutile, and titanite age data to corresponding *P–T* conditions in a polyphase metamorphic orogen.

The Central Zone (CZ) of the Limpopo Belt in South Africa is a typical polyphase metamorphic terrane. This zone has experienced three metamorphic events at 3.22 Ga ($M_1$), 2.66–2.61 Ga ($M_2$), and 2.02 Ga ($M_3$) (e.g., Barton et al., 2006; Kramers et al., 2006; Zeh et al., 2010; Kröner et al., 2018). The $M_2$ event had previously only been identified at a few localities in rare zircon grains or zircon overgrowths (Millonig et al., 2008; Van Reenen et al., 2008). Its occurrence was controversial, given the fragmentary and indistinct geochronological evidence (Holzer

* Corresponding author at: State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China.
*E-mail address:* liqiuli@mail.iggcas.ac.cn (Q. Li).

https://doi.org/10.1016/j.precamres.2020.105695
Received 7 January 2020; Received in revised form 20 February 2020; Accepted 5 March 2020
Available online 06 March 2020
0301-9268/ © 2020 Elsevier B.V. All rights reserved.

Lithos 368–369 (2020) 105573

Contents lists available at ScienceDirect

Lithos

journal homepage: www.elsevier.com/locate/lithos

Research Article

# Multi-system geochronology of North Dabie eclogite: Ineffective garnet 'shielding' on rutile inclusions under multi-thermal conditions

Tian Zhou [a,b,c], Qiuli Li [a,b,c,*], Reiner Klemd [d], Yonghong Shi [e], Xu Tang [b], Chaofeng Li [a,b], Yu Liu [a,b]

[a] State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China
[b] Innovation Academy for Earth Science, Chinese Academy of Sciences, Beijing 100029, China
[c] College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, China
[d] Universität Erlangen-Nürnberg, Schlossgarten 5a, 91054 Erlangen, Germany
[e] School of Resources and Environment Engineering, Hefei University of Technology, Hefei 230029, China

ARTICLE INFO

*Article history:*
Received 30 March 2020
Received in revised form 9 May 2020
Accepted 10 May 2020
Available online 16 May 2020

*Keywords:*
Shielding effect
Rutile inclusions
Secondary ion mass spectrometry
U-Pb dating
Transmission electron microscope

ABSTRACT

It has been shown that certain host minerals can robustly shield mineral inclusions and prevent loss of petrochronological information during subsequent thermal events. Rutile inclusions in garnet may crystallize under a wide range of metamorphic conditions and thus rutile is expected to be a useful geochronometer for constraining different stages of a metamorphic P-T path. Hence, it is important to ascertain whether rutile inclusions shielded by garnet retain the original crystallization age information despite later retrograde cooling or subsequent new thermal events. In this study, we test the "shielding effect" of garnet on rutile inclusions from the Baizhangya eclogite in the North Dabie Zone (China), which underwent a multi-stage thermal evolution. Secondary ion mass spectrometry (SIMS) U—Pb dating of inherited zircon core domains constrains a Neoproterozoic formation age (~770 Ma) of the eclogite protolith. SIMS U—Pb dating of metamorphic zircon and complementary thermal-ionization mass spectrometry (TIMS) Sm—Nd isochron dating of garnet revealed Triassic eclogite-facies metamorphic ages (245–210 Ma). An Early Cretaceous SIMS U—Pb age of ca. 130 Ma, yielded by matrix rutile grains and rutile inclusions, suggests significant isotopic alteration since crystallization. This indicates that shielding was ineffective, since the U—Pb ages derived from the rutile inclusions, although hermetically armored by garnet, were influenced by a subsequent Early Cretaceous tectono-thermal event. Further transmission electron microscope (TEM) analysis revealed the presence of hematite microlites along the garnet-rutile boundaries, which may have acted as a potential reservoir to store the expelled radiogenic Pb from rutile. This study suggests that newly formed minerals located along mineral boundaries seem to be crucial for understanding the shielding effect.

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## 1. Introduction

Reconstructing the P-T-t evolution of a metamorphic (ultra)-high pressure (UHP) terrane is usually challenging since the correlation of prograde, peak and retrograde metamorphic ages with the evolution of the mineral assemblages is often difficult to achieve (e.g., Engi et al., 2017 and references therein). One of the particularly problematic issues is to accurately constrain the age of a prograde mineral assemblage, since this is often obscured by peak or later metamorphic overprinting. Mineral assemblages from polymetamorphic terrains are particularly susceptible to overprinting during later events, which makes the interpretation of age data ambiguous (Baxter et al., 2017). In order to gain reliable information on the timing of the prograde P-T path, the dateable mineral must have formed and always stayed throughout the entire metamorphic evolution below its closure temperature, or must have been armored by the host mineral to avoid diffusive loss of the daughter isotopes, which is described as the "shielding effect" (Engi, 2017).

The shielding effect that allows mineral inclusions to retain their original age information despite subsequent thermal overprinting has been documented in various studies (e.g., Bruguier et al., 2017; Catlos et al., 2002; de Meyer et al., 2014; DeWolf et al., 1993; Hoisch et al., 2008; Kelley et al., 1997; Kinny and Meyer, 1994; Montel et al., 2000; Zhu et al., 1997). For instance, zircon inclusions in natural diamonds from the Mbuji-Mayi kimberlite yielded a concordant U—Pb age of 628 ± 12 Ma (Kinny and Meyer, 1994), which is much older than that of matrix zircon megacrysts (~70 Ma), displaying the eruption age of the kimberlite (Schärer et al., 1997). This implies that the U—Pb isotopic system of the zircon megacrysts underwent open system behavior at mantle temperatures (Cherniak and Watson, 2001), while the diamond shielded the U—Pb system of the zircon from diffusive loss of Pb.

* Corresponding author at: State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China.
*E-mail address:* liqiuli@mail.iggcas.ac.cn (Q. Li).

https://doi.org/10.1016/j.lithos.2020.105573
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Vol. 44 — N° 3 09 20 P. 421 – 437

GEOSTANDARDS and GEOANALYTICAL RESEARCH

# Concurrent Determination of U-Pb Age and REE Mass Fractions of Zircon by High Mass Resolution SIMS

Yu **Liu** (1, 2), Qiu-Li **Li** (1, 2, 3)*, Guo-Qiang **Tang** (1, 2, 3) and Xian-Hua **Li** (1, 2, 3)

(1) State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China
(2) Innovation Academy for Earth Science, Chinese Academy of Sciences, Beijing 100029, China
(3) College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, China
* Corresponding author. e-mail: liqiuli@mail.iggcas.ac.cn

**A combined geochronological and geochemical investigation for the same domain of zircon provides valuable information on timing and genesis, particularly in the case of multi-growth metamorphic zircon. A high spatial resolution concurrent analytical method for zircon U-Pb age and rare earth element content was successfully achieved in this study, using a multi-collector secondary ion mass spectrometer (SIMS) at a ~ 8 μm diameter scale. Special instrument parameters were employed, including a high mass resolution of approximately 15000 applied to replace the previous energy filter method, and a dynamic multi-collector mode used to reduce the measurement time to 18 min per analysis. Six zircon reference materials yielded precise and accurate $^{206}Pb/^{238}U$ ages, which are comparable to those obtained by the ordinary mono-collector method, but with 2–3 times higher spatial resolution. All zircon grains measured in this study showed enriched heavy-REE (HREE) contents consistent with previously reported values determined by LA and solution ICP-MS methods. The light-REE (LREE) mass fractions measured using both SIMS and LA-ICP-MS methods in this study, although with quite different volume, show consistent results within uncertainties.**

Keywords: zircon, U-Pb geochronology, REE, high spatial resolution, concurrent determination.

**Received 31 Dec 18 – Accepted 13 May 20**

Zircon ($ZrSiO_4$) is a remarkable U- (and Th-) bearing mineral, occurring in most types of silica-saturated rocks. It is a well-known U-Th-Pb geochronometer because of its relatively high mass fraction of parent U and low mass fraction of daughter Pb as well as its relatively high U-Th-Pb closure temperature (Davis *et al.* 2003, Li *et al.* 2018). The trace element composition of zircon can also be used to constrain its geological significance, which is recorded in its various growth zones (Rubatto 2002). The utility of zircon lies in its chemical and physical durability, and remarkable resistance to high-temperature diffusive re-equilibration (e.g., Heaman *et al.* 1990, Mass *et al.* 1992, Hoskin and Black 2000, Belousova *et al.* 2002). Based on cathodoluminescence (CL) images and/or backscattered electron (BSE) images, multi-growth zones can be detected in a single zircon grain. Combining multiple sources of information on zircon (e.g., U-Pb age, REE pattern, Hf- and O-isotopic compositions) greatly enhances age interpretation, which has been successfully conducted to reveal multiple geological events recorded by single zircon grains (e.g., Rubatto 2002, Hoskin and Schaltegger 2003).

Theoretically, all of the combined data should be derived from the same analytical domain of the zircon grain. Therefore, *in situ* microanalysis techniques, including laser ablation inductively coupled plasma-mass spectrometry (LA-ICP-MS) and secondary ion mass spectrometry (SIMS), have been chosen as the preferred analytical techniques. Taking advantage of the rapid peak hopping of the quadrupole mass analyser, the U-Pb age and trace element compositions of zircon can be obtained simultaneously by LA-ICP-MS (e.g., Li *et al.* 2000, Yuan *et al.* 2004). This technique has been widely used for magmatic zircon analysis, at a spatial resolution of 25–40 μm (e.g., Buick *et al.* 2006, Ortiz *et al.* 2019). In general, the depth obtained in a normal LA-ICP-MS zircon U-Pb dating sequence shows a range of 6–20 μm, depending on the distinct laser configuration and ablation time (e.g., Bernal *et al.* 2014, Yuan *et al.* 2004). However, zircons that have

doi: 10.1111/ggr.12344
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frontiers in Chemistry

ORIGINAL RESEARCH
published: 03 December 2020
doi: 10.3389/fchem.2020.605646

# Impact and Correction of Analytical Positioning on Accuracy of Zircon U-Pb Dating by SIMS

*Yu Liu [1,2], Qiu-Li Li [1,2,3]*, Xiao-Xiao Ling [1,2], Guo-Qiang Tang [1,2], Jiao Li [1,2] and Xian-Hua Li [1,2,3]*

[1] State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing, China, [2] Innovation Academy for Earth Science, Chinese Academy of Sciences, Beijing, China, [3] College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing, China

Secondary ion mass spectrometry (SIMS) is one of the most important analytical tools for geochronology, especially for zircon U-Pb dating. Due to its advantages in spatial resolution and analytical precision, SIMS is the preferred option for multi-spot analyses on single zircon grain with complex structures. However, whether or how much the relative positions of multiple analytical spots on one zircon grain affect the U-Pb age accuracy is an important issue that has been neglected by most researchers. In this study, we carried out a series of investigation on the influence of relative analytical position during zircon U-Pb age analyses, using Cameca IMS 1280-HR instrument. The results demonstrated a significant influence on the second spot, with apparent U-Pb age deviation as high as around 10% especially on the left and right side with overlap in the raster area. Nevertheless, a linear correlation between a secondary ion centering parameter (DTCA-X) and age deviation in percentage terms was found, and a calibration method was established to correct this position effect. Four zircon standards (91500, M257, TEMORA-2, and Plešovice) were measured to prove the reliability of the established procedure. The original U-Pb apparent data show inconsistent deviation on four directions relative to the datum, while the final U-Pb age results is calibrated to be consistent with their recommended values, within uncertainties of ∼1%. This work calls for re-examination for the previous SIMS U-Pb dating results on core-rim dating strategy, and provides a calibration protocol to correct the relative position effect.

**Keywords: SIMS, zircon, U-Pb dating, positioning effect, metamorphic overgrowth**

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***Edited by:***
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*National Research Council Canada (NRC-CNRC), Canada*

***Reviewed by:***
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*Purdue University, United States*

****Correspondence:***
*Qiu-Li Li*
*liqiuli@mail.iggcas.ac.cn*

***Specialty section:***
*This article was submitted to Analytical Chemistry, a section of the journal Frontiers in Chemistry*

***Received:*** *12 September 2020*
***Accepted:*** *26 October 2020*
***Published:*** *03 December 2020*

***Citation:***
*Liu Y, Li Q-L, Ling X-X, Tang G-Q, Li J and Li X-H (2020) Impact and Correction of Analytical Positioning on Accuracy of Zircon U-Pb Dating by SIMS. Front. Chem. 8:605646. doi: 10.3389/fchem.2020.605646*

## INTRODUCTION

Secondary ion mass spectrometry (SIMS) is widely and routinely used in U-Pb dating of zircon and other accessory minerals. It can achieve a lateral resolution of 2–30 μm and a depth resolution of 1–2 μm, which makes it the first choice for *in situ* high spatial resolution zircon U-Pb dating. By using SIMS, multiple U-Pb dating analyses on different crystal domains of single zircon grain were routinely carried out to reveal its growth processes, and then the corresponding geological evolution history (e.g., Hermann and Rubatto, 2003; Whitehouse and Platt, 2003; Zhang et al., 2005; Tang et al., 2008; Qian et al., 2013). However, when seeking higher spatial resolution for zircon U-Pb age determination, it is necessary to understand whether and how much two analytical spots with overlapped pre-analysis sputtering area or even analytical position could affect the age accuracy. Although the instrument developers were aware of this potential influence (e.g., Ickert et al., 2008), no quantitative study has been reported on this issue.

JAAS

PAPER

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Cite this: *J. Anal. At. Spectrom.*, 2018, **33**, 1536

# Zircon Th–Pb dating by secondary ion mass spectrometry†

Qiuli Li,[ab]* Yu Liu,[ab] Guoqiang Tang,[ab] Kaiyi Wang,[a] Xiaoxiao Ling[a] and Jiao Li[a]

Zircon has been widely used as a geochronometer with the U–Pb decay system but rarely with the Th–Pb system. However, zircon from carbonatite contains very little U and has a high Th/U ratio, making Th–Pb dating preferable. As a one-dimensional system, a series of consistent Th–Pb ages can be used to date a geological event. In contrast, a wide variation in Th–Pb ages could result from Pb loss or multiple growth events, making it difficult to link to specific geological events. In this study, we described Th–Pb dating protocols on zircon and carried out Th–Pb measurements on seven zircon reference materials using a Cameca IMS 1280HR ion microprobe. The results demonstrated that these seven U–Pb zircon standards have similar absolute concentrations of common lead. The radiogenic $^{208}$Pb concentrations (depending on the Th content and age) determine the proportion of common lead and define the extent of variation in the Th–Pb system under certain analytical conditions. This relationship could be used as a criterion to evaluate whether it is a single population or not based on Th–Pb dating results of unknown zircons. By comparison, M257 and Qinghu zircons are suggested as the most suitable Th–Pb dating standards, with ID-TIMS U–Pb ages representing the best estimate of the Th–Pb reference ages. A zircon sample from the Wu dyke, an extremely rare earth element (REE)-rich carbonatite dyke in the Bayan Obo area, was dated with the established Th–Pb dating procedure and yielded an age of 1327 ± 20 Ma as the emplacement time. A pronounced relationship between apparent Th–Pb ages and corresponding Th contents (>1500 ppm) indicates that zircons with accumulated radiogenic doses of >1.7 × $10^{18}$ α per g may tend to lose radiogenic $^{208}$Pb.

Received 28th April 2018
Accepted 20th July 2018

DOI: 10.1039/c8ja00125a

rsc.li/jaas

## 1. Introduction

Zircon has been widely used as a U–Pb geochronometer because (1) it normally contains U and Th at concentrations well above its host rock average but discriminates strongly against the daughter element Pb during crystallization; (2) it is sufficiently robust to preserve its original parent and accumulated radiogenic Pb contents even through remelting events; (3) it is the most widespread of the common trace components in felsic rocks.[1] The U–Pb system offers an internal check for closed-system behaviour in that it contains two radionuclides ($^{238}$U–$^{206}$Pb and $^{235}$U–$^{207}$Pb), which means that two or three dates ($^{206}$Pb/$^{238}$U, $^{207}$Pb/$^{235}$U, and $^{207}$Pb/$^{206}$Pb ages) can be obtained simultaneously by one measurement. If the zircon has behaved as a closed system since the start of daughter ingrowth, the dates should be concordant. Even if the data are discordant, the coupled U–Pb decay systems could allow one to deduce the crystallization age from a series of discordant zircon analyses.[2]

The Th–Pb system ($^{232}$Th–$^{208}$Pb) is a similar system to U–Pb; however, it has seldom been applied to zircon due to two main reasons. First, the Th–Pb system has a half-life that is >3 times longer than that of the $^{238}$U–$^{206}$Pb system.[3] Therefore, in order for a zircon to have comparable radiogenic $^{208}$Pb and $^{206}$Pb, it must have a Th/U of >3. However, most zircon has a Th/U ratio of approximately 1 or less. Second, in many early thermal ionization mass spectrometry (TIMS) studies, it was found that the Th–Pb system was often decoupled from the U–Pb system.[4] This is because the altered zircon tends to lose $^{208}$Pb more readily than $^{206}$Pb, *i.e.*, the Th–Pb system tends to be disturbed more easily than the U–Pb system. In addition, it is relatively difficult to measure Th by TIMS because Th has poorer thermal ionization than U. Therefore, the $^{232}$Th–$^{208}$Pb system for zircon was rarely measured by the TIMS method. For the micro-beam analysis technique, the Th species is commonly measured to determine the Th/U ratio as a chemical characteristic and, sometimes, to evaluate the zircon crystallizing conditions. In general, the Pb/Th age is not measured independently but is calculated by means of $^{208}$Pb/$^{232}$Th = $^{208}$Pb/$^{206}$Pb × $^{206}$Pb/$^{238}$U × $^{238}$U/$^{232}$Th, which mainly relies on U–Pb analysis.

[a]*State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China. E-mail: liqiuli@mail.iggcas.ac.cn; Fax: +86-10-62010846; Tel: +86-10-82998535*

[b]*College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, China*

† Electronic supplementary information (ESI) available. See DOI: 10.1039/c8ja00125a



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# 国家自然科学奖

# 证　书

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项目名称：变质同位素年代学及华北与华南陆块碰撞过程

奖励等级：二等

获 奖 者：李秋立(中国科学技术大学)

中华人民共和国国务院

2010年[illegible]月29日

证书号：2010-Z-104-2-04-R05

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中国矿物岩石地球化学学会

2014年5月12日

证书编号：Y201526

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2018年10月12日

US011101126B2

(12) **United States Patent**
**Liu et al.**

(10) **Patent No.: US 11,101,126 B2**
(45) **Date of Patent: Aug. 24, 2021**

(54) **METHOD AND SYSTEM FOR MEASURING INERT GAS BY ION PROBE**

(71) Applicant: **INSTITUTE OF GEOLOGY AND GEOPHYSICS, CHINESE ACADEMY OF SCIENCES**, Beijing (CN)

(72) Inventors: **Yu Liu**, Beijing (CN); **Guoqiang Tang**, Beijing (CN); **Qiuli Li**, Beijing (CN)

(73) Assignee: **Institute of Geology and Geophysics, Chinese Academy of Sciences**, Beijing (CN)

( * ) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **16/754,629**

(22) PCT Filed: **Dec. 4, 2018**

(86) PCT No.: **PCT/CN2018/119204**
§ 371 (c)(1),
(2) Date: **Apr. 8, 2020**

(87) PCT Pub. No.: **WO2020/113426**
PCT Pub. Date: **Jun. 11, 2020**

(65) **Prior Publication Data**

US 2021/0057204 A1 Feb. 25, 2021

(51) **Int. Cl.**
***H01J 49/24*** (2006.01)
***H01J 49/14*** (2006.01)
(Continued)

(52) **U.S. Cl.**
CPC ..... ***H01J 49/24*** (2013.01); ***H01J 49/0463*** (2013.01); ***H01J 49/142*** (2013.01); ***H01J 49/147*** (2013.01); ***H01J 49/282*** (2013.01)

(58) **Field of Classification Search**
CPC .... H01J 49/0463; H01J 49/142; H01J 49/147; H01J 49/24; H01J 49/282
See application file for complete search history.

(56) **References Cited**

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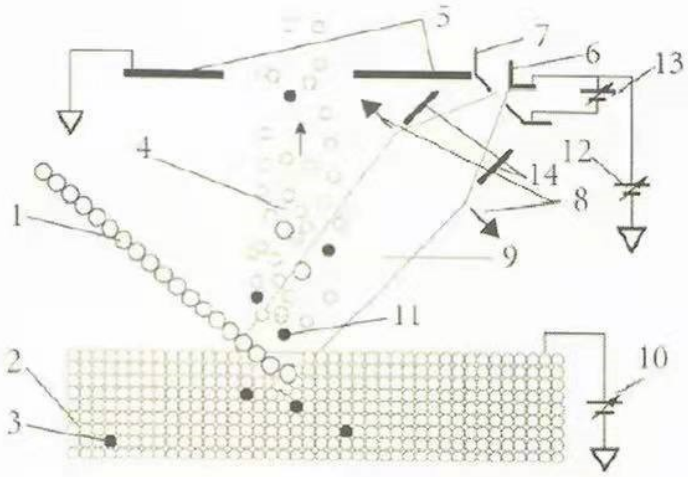
*Primary Examiner* — David E Smith
(74) *Attorney, Agent, or Firm* — Ohlandt, Greeley, Ruggiero & Perle, L.L.P.

(57) **ABSTRACT**

A method and system for measuring an inert gas by an ion probe. Embedding a to-be-measured sample into an epoxy resin, to obtain a sample target, where the to-be-measured sample includes an inert gas atom; after putting the obtained sample target into an analysis chamber of the ion probe, vacuumizing the analysis chamber, where the ion probe includes a primary ion source, an electron gun, a mass analyzer, and an ion detector; bombarding the sample target by using a primary ion beam formed by the primary ion source to release the inert gas atom in the sample target; ionizing the released inert gas atom by using an electron beam formed by the electron gun to form an inert gas ion; and analyzing a secondary ion containing the inert gas ion by using the mass analyzer and the ion detector to achieve measurement of the inert gas.

**20 Claims, 2 Drawing Sheets**



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发 明 名 称：一种离子探针的靶托组件及其样品靶制备的方法

发 明 人：李娇;李秋立

专 利 号：ZL 2019 1 1367225.X

专利申请日：2019年12月26日

专 利 权 人：中国科学院地质与地球物理研究所

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发明人：马红霞;凌潇潇;李秋立

专利号：ZL 2017 1 1126022.2

专利申请日：2017年11月15日

专利权人：中国科学院地质与地球物理研究所

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申请日时本专利记载的申请人、发明人信息如下：

申请人：

中国科学院地质与地球物理研究所

发明人：

马红霞；凌潇潇；李秋立

证书号第2605738号

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# 发明专利证书

发 明 名 称：一种基于二次离子质谱仪的测定氟碳铈矿样品的钍铅年龄的方法

发 明 人：凌潇潇；李秋立；李献华；刘宇；唐国强

专 利 号：ZL 2016 1 0023625.9

专利申请日：2016年01月14日

专 利 权 人：中国科学院地质与地球物理研究所

授权公告日：2017年09月01日

本发明经过本局依照中华人民共和国专利法进行审查，决定授予专利权，颁发本证书并在专利登记簿上予以登记。专利权自授权公告之日起生效。

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2017年09月01日