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激光剥蚀电感耦合等离子体质谱副矿物U-Th-Pb定年新进展

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摘 要: 激光剥蚀电感耦合等离子体质谱(LA-ICP-MS)副矿物U-Th-Pb定年技术为精确厘定地质演化历史、探讨成岩成矿等重要地质作用过程提供重要的时间参数,是地质年代学快速发展的重要技术支撑.总结了LA-ICP-MS副矿物U-Th-Pb定年技术在元素分馏校正、非基体匹配分析、标准样品研发、普通铅校正、高空间分辨率及高效率分析等方面取得的重要进展.展望未来,需更进一步深入探讨和研究元素分馏及基体效应机理,研发更多种类的高质量副矿物标样,建立更多更准确、更精密、更高空间分辨率及更高效的LA-ICP-MS副矿物U-Th-Pb定年新方法和新技术,以实现从更微观和更精细的角度探讨地质问题,并持续为高效解决地球与行星科学研究领域重大科学问题提供关键支撑.

关键词: 激光剥蚀电感耦合等离子体质谱;U-Th-Pb年代学;元素分馏;基体效应;标准样品;普通铅校正;年代学.

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Recent Advances in U-Th-Pb Dating of Accessory Minerals by Laser Ablation Inductively Coupled Plasma Mass Spectrometry

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Abstract: Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) U-Th-Pb dating technique provides crucial temporal constraints to accurately determine geological evolution history and explore important geological processes such as diagenesis and mineralization, which is an important technical support for the rapid development of geochronology. In this study, it summarizes the important progress of LA-ICP-MS U-Th-Pb dating technique in elemental fractionation correction, non-matrix matched analysis, reference material development, common lead correction, high spatial resolution and high efficiency analysis. Looking into the future, further investigations on the mechanism of elemental fractionation and matrix effect, characterization of more kinds of high-quality reference materials, and development of more accurate, more precise, and more efficient LA-ICP-MS U-Th-Pb dating methods with higher spatial resolution are needed. These new established methods and techniques will help the geoscientists investigate the geological problems from a more microscopic and delicate perspective, and those will continue to provide critical supports for the advances and innovations in earth and planetary science research.

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Key words: LA-ICP-MS; U-Th-Pb geochronology; elemental fractionation; matrix effect; reference material; common-Pb correction; chronology.

0　引言

同位素年代学研究可提供各种地质体形成的准确时间,为研究各类地质事件及追溯地质演化历史提供精确的年代信息,一直是地学研究中的热点.U-Th-Pb定年技术作为最经典的同位素年代学方法之一,已被广泛应用于各类含U、Th副矿物定年(Schoene, 2014;Condon *et al.*, 2021;Fanka *et al.*,2021;麦源君等,2021;涂城等,2021).用于副矿物U-Th-Pb同位素分析的方法主要有热电离质谱法(TIMS)、二次离子探针法(SIMS)和激光剥蚀电感耦合等离子体质谱法(LA-ICP-MS)(Schaltegger *et al.*, 2015).热电离质谱法是当前副矿物U-Th-Pb年代学分析准确度和精密度最高的方法,不同实验室间$^{206}Pb/^{238}U$年龄重现性可优于0.05%(Condon *et al.*, 2015;McLean *et al.*, 2015),是副矿物U-Th-Pb年代学分析的基准方法.但该方法需要复杂的化学前处理流程,一般只能提供样品的混合年代学信息.离子探针法在20世纪70年代开始用于锆石U-Th-Pb年龄分析,其空间分辨能力高,离子束直径约为2~30 μm,纵向空间分辨率可达亚微米级(Liu *et al.*, 2011a, 2011b, 2020;Jeon and Whitehouse, 2015),可实现超高空间分辨率下的副矿物U-Th-Pb年龄准确分析(Che *et al.*, 2021;Li *et al.*, 2021;Zhao *et al.*, 2022),但离子探针分析过程中严重的基体效应和其高昂的运行成本限制了该技术的普及.激光剥蚀等离子体质谱技术具有样品前处理简单、分析速度快、基体效应相对较低、运行成本低等优点,已广泛应用于副矿物U-Th-Pb年龄分析.Web of science核心数据库统计结果表明,2011~2021年由激光剥蚀技术支撑发表的U-Th-Pb相关的论文在以上3种方法中所占比例呈上升趋势,由2011年的50%提升到2021年的70%左右(图1a),这表明LA-ICP-MS技术在支撑副矿物U-Th-Pb年代学发展中发挥着越来越重要的作用.从2011~2021年由激光剥蚀技术支撑发表的U-Th-Pb相关论文的地域分布如图1b所示,论文数量排名前三的国家为中国、美国和德国(图1b),中国论文数量以57%的比例远高于其他国家,说明我国已成为LA-ICP-MS副矿物U-Th-Pb年代学研究的重要支撑力量.

为推动激光剥蚀技术在副矿物U-Th-Pb年代学及元素、同位素分析中的发展和应用,高山教授等人于2005年西北大学发起和组织了"亚太地区激光剥蚀和微区分析研讨会",该会议现已发展成为微区分析技术及应用领域的专业盛会,极大地推动了中国乃至亚太地区激光剥蚀等微区分析技术的快速发展.此外,值得指出的是,中国学者在LA-ICP-MS副矿物U-Pb年龄和微量以及同位素同时测定方面取得了世界领先的成果(李献华等,2022).如我国学者率先提出锆石U-Pb年龄和微量元素同时测试技术(Liang *et al.*, 1999;Li *et al.*, 2000)以及锆石/斜锆石年龄-微量元素-Hf同位素多机

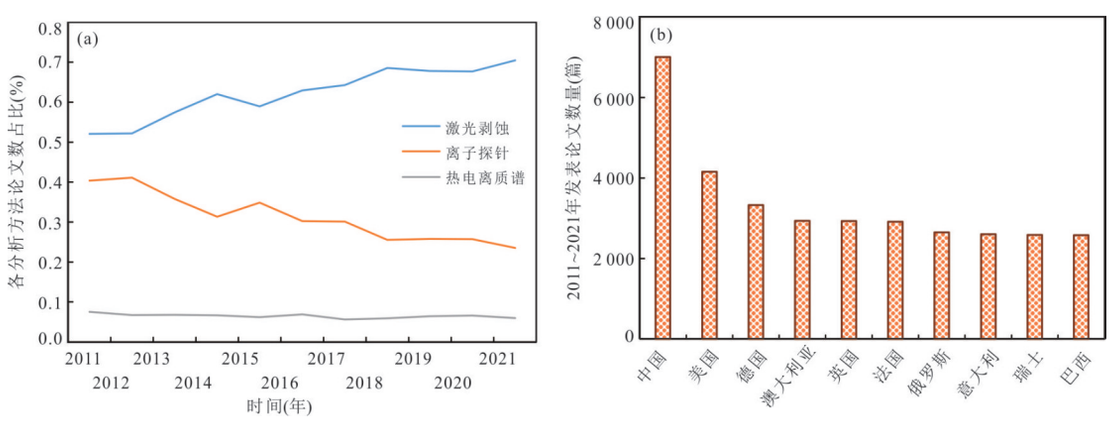

图1　2011—2021年发表与U-Th-Pb定年相关论文数据统计结果

Fig. 1　Articles related with U-Th-Pb geochronology published from 2011 to 2021

数据源于Web of science核心数据库. a.三种测试方法支撑发表论文数对比;b.由激光剥蚀技术支撑发表U-Th-Pb相关论文数量前十的地域分布情况

联测“一石三鸟”分析技术(Xie *et al.*, 2008; Yuan *et al.*, 2008).该多机联测技术具有广阔的应用前景,可广泛应用于其他副矿物年龄和同位素及矿物多同位素同时分析(Goudie *et al.*, 2014; Huang *et al.*, 2015; Zhang *et al.*, 2021a, 2021b).

激光剥蚀电感耦合等离子体质谱广泛应用于副矿物U-Th-Pb年龄分析(刘勇胜等, 2013, 2021; 陈意等, 2021; 李献华等, 2022).自Feng *et al.*(1993)和Fryer *et al.*(1993)最初进行LA-ICP-MS锆石U-Pb年龄分析以来,随着仪器硬件的改进和分析水平的提高,该技术在数据结果准确性和精密度、标准样品研发、空间分辨能力等方面取得了诸多的进步和发展.现可开展多种副矿物U-Th-Pb年龄测试,如锆石、斜锆石、榍石、独居石、蛋白石、钙钛矿、磷灰石、褐帘石、磷钇矿、沥青铀矿、异性石、金红石、锡石、碳酸盐、氟碳铈矿、铌铁矿、赤铁矿、石榴石、葡萄石、黑钨矿、孔雀石、绿帘石、萤石和符山石等(Liang *et al.*, 1999; Li *et al.*, 2000; Yuan *et al.*, 2004, 2011; Amelin and Back, 2006; Bayanova, 2006; Cox and Wilton, 2006; Storey *et al.*, 2006, 2007; Alexandre *et al.*, 2007; Gregory *et al.*, 2007; Klötzli *et al.*, 2007; 柳小明等, 2007; 李怀坤等, 2009; Li *et al.*, 2009, 2014; Liu *et al.*, 2010; Wu *et al.*, 2010, 2022; Zack *et al.*, 2011; 周红英等, 2012, 2018; Yang *et al.*, 2014a, 2014b, 2018, 2020; Che *et al.*, 2015; 李艳广等, 2015; 王倩和侯可军, 2015; Zong *et al.*, 2015; Bao *et al.*, 2016; Courtney-Davies *et al.*, 2016; Burisch *et al.*, 2017; 崔玉荣等, 2017; Deng *et al.*, 2017; Luo *et al.*, 2019; 赵令浩等, 2020; Kahou *et al.*, 2021; Lenoir *et al.*, 2021; Lv *et al.*, 2021; Peverelli *et al.*, 2021; 杨岳衡等, 2021; Zhang *et al.*, 2021a, 2021b; Wei *et al.*, 2022).尽管使用LA-ICP-MS方法进行U-Th-Pb年代学分析的副矿物种类丰富,但实际分析时仍存在元素分馏效应、基体效应、高质量标准样品匮乏、普通铅校正困难、空间分辨能力较弱以及微区分析效率低等影响分析结果的重要问题.本文将对LA-ICP-MS副矿物U-Th-Pb年龄分析仍存在的主要问题及近期研究进展进行阐述.

1 元素分馏

元素分馏是LA-ICP-MS开展固体样品元素及同位素分析过程中存在的共性问题,可发生在激光样品相互作用过程、样品传输过程和分析物在ICP离子化等过程(Günther and Hattendorf, 2005)(图2a).由于元素物理化学性质(如氧化物熔点、50%冷凝温度等)的差异,激光样品相互作用时会产生尺寸大小不均、化学组成不同的气溶胶颗粒(Günther and Hattendorf, 2005; Hu *et al.*, 2008; Luo *et al.*, 2015).这些不同大小的气溶胶颗粒化学组成与原始样品有较大差异(Koch *et al.*, 2004),如亲石元素倾向富集于大尺寸气溶胶颗粒,而亲铜、亲铁元素倾向富集于小尺寸颗粒(Hu *et al.*, 2008; Luo *et al.*, 2015);这些不同大小的气溶胶颗粒在传输时也可能部分丢失(Koch *et al.*, 2002);大尺寸气溶胶颗粒在ICP等离子体中可能不完全离子化(Kuhn *et al.*, 2004),这些过程中均会造成元素分馏(Günther and Hattendorf, 2005).激光波长、束斑大小、能量密度、剥蚀方式、脉冲宽度等剥蚀参数及样品基体性质均会影响剥蚀产生的样品气溶胶颗粒大小和化学组成(罗涛, 2018).此外,载气类型(Luo *et al.*, 2018a)及激光剥蚀采样点气体流速大小(Luo *et al.*, 2015, 2018b)也会影响纳秒激

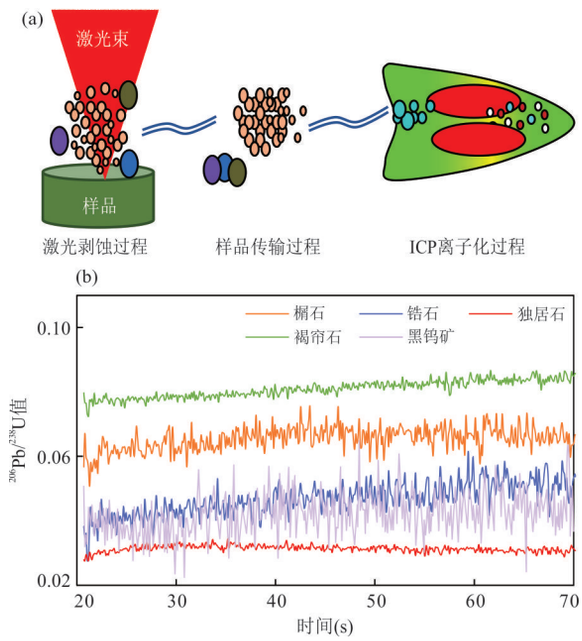

图2 LA-ICP-MS分析过程中元素分馏及基体效应示意图(a)和193 nm准分子激光ns-LA-ICP-MS分析不同副矿物时Pb/U比值瞬时信号图(b)

Fig.2 Schematic diagram of elemental fractionation and matrix effect in LA-ICP-MS analysis (a) and time-resolved Pb/U ratio of different accessory minerals analyzed with 193 nm excimer ns-LA-ICP-MS (b)

图2a修改自罗涛(2018)

光剥蚀产生的气溶胶颗粒尺寸大小.采用氦气替代氩气作为样品气溶胶的载气,或在高气体流速下剥蚀采样可减小大尺寸气溶胶颗粒的产生(Luo *et al.*, 2018b).通过降低等离子体氩气流速或引入辅助气(氮气、水蒸气等)等方式提高ICP的蒸发、离子化效率可减小ICP离子化不完全引起的元素分馏效应(Luo *et al.*, 2015, 2018b).

对于LA-ICP-MS副矿物U-Pb年龄分析而言,挥发性元素Pb和难熔元素U之间的分馏效应是影响分析结果的主要因素.Pb/U分馏一般可以分为2类:(1)激光样品相互作用引起的化学分馏(Košler *et al.*, 2005;Hergenröder *et al.*, 2006;Kuhn *et al.*, 2010),如锆石热分解过程中生成斜锆石ZrO_2和SiO_2,而U和Pb分别进入ZrO_2和SiO_2相,因此导致Pb/U元素分馏;(2)与激光剥蚀时间有关的元素分馏,即随着单点激光剥蚀的进行,剥蚀坑纵横比逐渐增加,Pb/U比值也逐渐增加的现象(Kosler and Sylvester, 2003;Jackson *et al.*, 2004),这也是通常所谓的与剥蚀时间有关的"downhole"分馏(Eggins *et al.*, 1998)."downhole"分馏本质上也是由于激光剥蚀过程中产生不同化学组成、不同尺寸大小气溶胶颗粒导致.在研究初期,学者们通过优化激光剥蚀参数以降低U-Pb分析时的"downhole"分馏,如活动聚焦(Hirata and Nesbitt, 1995)和逐步增加剥蚀能量的软剥蚀(Hirata, 1997)等方法,因这些方法操作繁琐,没有被广泛采用.通过激光线扫描的剥蚀方式降低分馏效应也可获得准确的副矿物U-Pb定年结果(Li *et al.*, 2001;Horstwood *et al.*, 2003;Sláma *et al.*, 2008),但这种剥蚀模式严重限制了激光微区分析的空间分辨能力.采用"截距法"计算出Pb/U比值进行校正也可以很大程度降低"downhole"分馏效应的影响(Košler *et al.*, 2001, 2002;Košler and Sylvester, 2003;Chew *et al.*, 2011).当前应用最为广泛的是通过数学模型校正以降低Pb/U"downhole"分馏效应,Horn *et al.*最早提出可采用线性回归方程校正元素分馏以获得准确的$^{206}Pb/^{238}U$比值(Horn *et al.*, 2000).随后的研究表明,在特定的仪器条件下,分析不同矿物时的"downhole"分馏不仅仅为简单的线性关系,也可能为指数或更复杂的数学形式.如图2b所示,激光单点剥蚀过程中不同副矿物间的Pb/U分馏行为呈现显著差异.但这些不同的分馏模式均可采用数学模型进行校正(Paton *et al.*, 2010;Ver Hoeve *et al.*, 2018).需要注意的是,只有标准物质与待测样品分馏模式一致时,才能通过数学模型校正元素分馏以获得准确的分析结果.

2　基体效应

LA-ICP-MS副矿物U-Th-Pb年代学分析时通常需要采用基体匹配的标准样品与待测样品间插的方式进行元素分馏、质量歧视和仪器漂移校正.由标样和样品间基体变化引起的Pb/U和Pb/Th分馏行为差异会导致副矿物U-Th-Pb年龄测试结果相对其TIMS参考值的系统偏差,即为基体效应(Allen and Campbell, 2012; Luo *et al.*, 2018b).高质量的基体匹配微区分析标准样品极度缺乏,严重制约着副矿物U-Th-Pb年代学的广泛应用.为突破这一瓶颈,学者们广泛尝试使用基体不匹配的副矿物标样进行外标校正,但通常会观察到显著的基体效应.如使用锆石作为外标分析榍石和褐帘石时获得的U-Pb年龄结果出现约7%～15%的系统偏差(El Korh, 2013, 2014).Liu *et al.*(2011a)分别采用锆石和独居石标样作为外部标样校正磷钇矿BS-1,结果表明采用基体不匹配的外标分析时观察到显著的基体效应,需采用基体匹配的磷钇矿标样才能获得准确的数据结果.Sun *et al.*(2012)采用锆石91500作为外标分析榍石OLT-1的结果比推荐值年轻约12%.Yang *et al.*(2014a, 2018)分别研究了锆石和氟碳铈矿以及锆石和石榴石之间U-Pb年龄分析的基体效应,发现以锆石91500为外标分析氟碳铈矿获得的年龄比其推荐值年轻约10%,而获得的石榴石结果比其推荐值偏老约11%.飞秒激光脉冲宽度短,激光与样品相互作用过程中的热效应小(Fernández *et al.*, 2007),且剥蚀产生的气溶胶颗粒细小,被认为可以有效地降低LA-ICP-MS分析过程中的元素分馏和基体效应(Koch *et al.*, 2005;Li *et al.*, 2015b).但Wohlgemuth-Ueberwasser *et al.*(2018)研究结果表明,采用fs-LA-ICP-MS以锆石为外标分析斜锆石时仍存在显著的基体效应.笔者实验室分别采用纳秒和飞秒激光以NIST610玻璃为外标分析锆石、独居石、磷钇矿和榍石的结果表明:纳秒激光剥蚀时,各副矿物分析结果出现约9%～23%的系统偏差;飞秒激光剥蚀时,定年结果呈现约9%～18%的系统偏差,两种激光剥蚀采样时均会出现显著的基体效应(图3).因此,基体匹配的标准物质是准确开展纳秒和飞秒激光定年工作的前提.

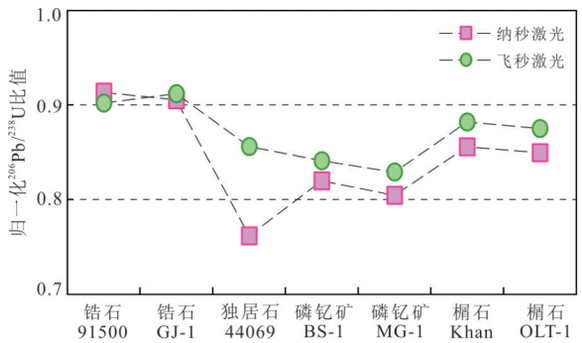

图3 采用纳秒和飞秒激光分别以NIST610玻璃为外标分析锆石、独居石、磷钇矿和榍石结果

Fig.3 Normalized $^{206}Pb/^{238}U$ ages of different accessory minerals by calibration against NIST 610 with ns- and fs-LA-ICP-MS

归一化$^{206}Pb/^{238}U$比值由各矿物测试$^{206}Pb/^{238}U$值除以其推荐值

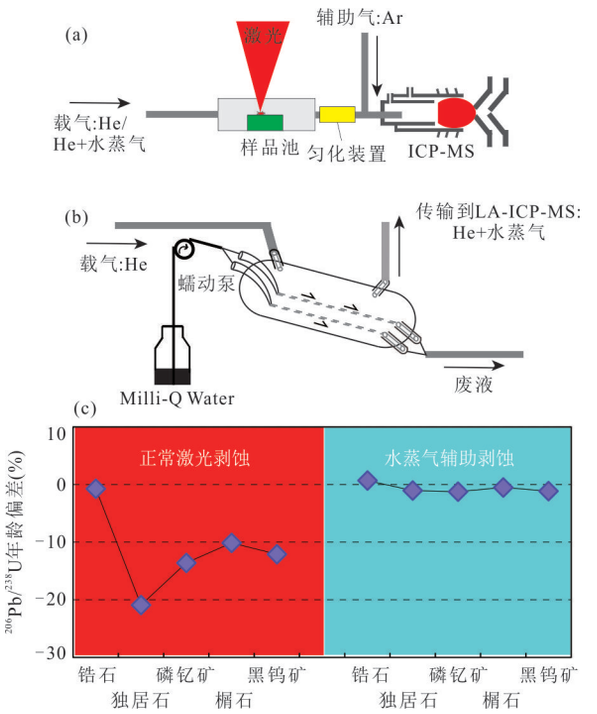

图4 正常剥蚀和水蒸气辅助剥蚀时，以锆石为外标LA-ICP-MS分析不同副矿物(锆石、榍石、独居石和磷钇矿)的U-Pb年龄结果

Fig. 4 The obtained U-Pb ages of different accessory minerals calibrated with zircon in normal ablation mode and water vapor-assisted ablation mode

a.剥蚀池中加入水蒸气；b.水蒸气稳定引入装置；c.不同分析模式获得的年龄结果

如何减小或消除不同副矿物间U-Th-Pb定年过程中的基体效应，实现各类副矿物U-Th-Pb年龄分析时的非基体匹配校正，一直是副矿物U-Th-Pb年代学研究的热点与难点.已有研究表明，通过优化激光剥蚀参数可在一定程度上减小不同矿物间U-Th-Pb年龄分析的基体效应.如通过激光线扫描剥蚀，以锆石为外标分别成功地获得了榍石(Storey *et al.*, 2007)、磷灰石(Chew *et al.*, 2011)和金红石(Hou *et al.*, 2020)的U-Th-Pb年龄，但该方法严重制约了LA-ICP-MS技术的高空间分辨能力；通过优化仪器条件，McFarlane (2016)使用玻璃标样NIST 610做外标，对褐帘石进行了U-Th-Pb年龄分析.采用数学模型对外标锆石和待测样品褐帘石分别进行“downhole”分馏校正后也获得了准确的褐帘石U-Th-Pb年龄(Burn *et al.*, 2017).Luo *et al.* (2018a, 2020)提出水蒸气辅助激光剥蚀技术(图4)，通过向激光样品池内引入适量水蒸气，可显著降低不同副矿物间的Pb/U分馏差异，使不同副矿物间U-Th-Pb年龄分析的基体效应从10%～24%减小到1%～2%，成功实现以锆石或NIST玻璃为外标校正分析榍石、独居石、磷钇矿、氟碳铈矿以及黑钨矿等副矿物的U-Th-Pb年龄(Luo *et al.*, 2018a, 2019, 2020, 2021a).通过引入水蒸气可使U、Th、Pb元素信号灵敏度增加约2倍，有助于提高LA-ICP-MS副矿物U-Th-Pb定年分析的空间分辨率.该水蒸气辅助激光剥蚀技术简单有效，有利于进一步拓宽LA-ICP-MS副矿物U-Th-Pb方法在地质学中的广泛应用.但当激光产生的剥蚀坑纵横比较大(即小束斑、高频率剥蚀)时，由于不同副矿物间元素分馏效应显著(Mank and Mason, 1999)，即使采用水蒸气辅助法也无法完全消除不同副矿物间的基体效应(Luo *et al.*, 2018b, 2020).

研究表明同种矿物间基体性质的差异也会造成U-Pb定年结果的显著系统偏差.如研究发现LA-ICP-MS法分析锆石标准样品时，不同标准样品互相校正时获得的分析结果与其TIMS年龄相比约3%～4%的系统偏差(Black *et al.*, 2003a, 2003b, 2004; Klötzli *et al.*, 2009; Košler *et al.*, 2013).造成该系统偏差的理论机制尚未完全清楚，可能与锆石样品的晶体化学组成(如微量元素含量)(Black *et al.*, 2004)、晶体颜色(Kooijman *et al.*, 2012)、放射性损伤程度(Allen and Campbell, 2012; Steely *et al.*, 2014)等有关.锆石晶体的物理化学性质差异导致激光样品相互作用过程中剥蚀速率不同最终引起分析结果的系统偏差(Marillo-Sialer *et al.*, 2014).通过对锆石样品进行加热退火或化学刻蚀处理，可以有效降低或消除不同锆石间的基体效应，提高其U-Pb年龄分析结果的

准确性(Allen and Campbell, 2012; Crowley *et al.*, 2014; von Quadt *et al.*, 2014),但锆石标准样品和待测样品的加热退火温度需超过950°以上,以完全消除不同程度蜕晶化样品对分析结果的影响(Marillo-Sialer *et al.*, 2016).而加热处理过程较为繁琐且不适用于所有样品(如薄片中的锆石),因此有学者提出使用线性回归模型直接校正不同锆石基体间α衰变剂量差异引起的年龄系统偏差(Sliwinski *et al.*, 2017),以获得准确的分析结果.

在今后的研究中,需要加深对元素分馏和基体效应机理的研究和探讨,进一步减小或消除其对副矿物U-Th-Pb年龄分析结果的影响,以提升U-Th-Pb年龄结果的准确性和精密度(Schaltegger *et al.*, 2015),再结合统一的数据处理规范和分析流程(Horstwood *et al.*, 2016),最终提高不同LA-ICP-MS分析实验室间U-Th-Pb年龄结果的重现性(Li *et al.*, 2015a).

3　标准样品研发

由于LA-ICP-MS分析过程中存在元素分馏和基体效应,采用基体匹配的矿物标样进行校正是获得准确数据结果最直接、最可靠的手段.尽管非基体匹配校正策略在一定程度上减小或消除了不同副矿物间U-Th-Pb年龄分析时的基体效应,获得了可满足实际应用需求的分析结果,但在实际测试流程中选择与待测样品基体匹配的矿物标样作为质量监控仍是必不可少的.因此,基体匹配的高质量矿物标样是开展含U副矿物微区原位U-Th-Pb年代学分析的基础和前提.但可用于副矿物U-Th-Pb年代学分析的高质量矿物标样往往极度匮乏,学者们对此开展了大量的研究,也取得了较丰硕的研究成果.不同的常用副矿物相均有相应基体匹配的U-Th-Pb定年标准样品报道,如锆石标样91500(Wiedenbeck *et al.*, 1995)、GJ-1(Jackson *et al.*, 2004)、Plešovice(Sláma *et al.*, 2008)等;榍石标样Khan(Heaman, 2009)、BLR-1(Aleinikoff *et al.*, 2007)、OLT-1(Kennedy *et al.*, 2010)等;独居石标样44069(Aleinikoff *et al.*, 2006)等;磷灰石标样MAD(Thomson *et al.*, 2012)、Durango(Chew *et al.*, 2011)等;褐帘石标样BONA(von Blackenburg, 1992)、SISS(von Blackenburg, 1992)、CAP(Barth *et al.*, 1994)、AVC(Barth *et al.*, 1994)、Tara(Gregory *et al.*, 2007)等;磷钇矿标样MG-1和BS-1(Fletcher *et al.*,2004)等;石榴石U-Pb年龄分析标样Willsboro、Mali(Seman *et al.*, 2017)等;碳酸盐标样WC1(Roberts *et al.*, 2017)等.详细的锆石、斜锆石、榍石、独居石、磷钇矿、磷灰石、褐帘石、金红石、锡石、氟碳铈矿、石榴石、黑钨矿和碳酸盐等副矿物标样信息及参考文献如附表1所示.

近年来中国学者在副矿物U-Th-Pb标样研发上取得了显著成果,如锆石标样Pengla(Li *et al.*, 2010)、Qinghu(Li *et al.*, 2013)、KV01(Wei *et al.*, 2020)、SA01(Huang *et al.*, 2020)、SA02(Huang *et al.*, 2021)、Jilin(Luo *et al.*, 2021b)、Tanz(Hu *et al.*, 2021)等.其中,Tanz锆石巨晶共计重约9.06 kg,最大单颗粒重366 g,是目前世界上最大的锆石巨晶标样(Hu *et al.*, 2021).榍石标样Ontario(Ma *et al.*, 2019)、T3(Ma *et al.*, 2019)、YQ82(Ma *et al.*, 2019; Ling *et al.*, 2022a, 2022b)等.独居石标样RW-1(Ling *et al.*, 2017),褐帘石标样A007、A011、A012(Yang *et al.*, 2022a),锡石标样RG-114、BB#7、19GX(Yang *et al.*, 2022b),金红石标样RMJG(Zhang *et al.*, 2019),氟碳铈矿K9(Yang *et al.*, 2014a; Li *et al.*, 2020),石榴石WS20(Yang *et al.*, 2018)和PL57(Li *et al.*, 2022)以及黑钨矿MTM(Luo *et al.*, 2019)和YGX-2113、Panasqueira、Cornwall、YGX-2107、SHM(Yang *et al.*, 2020)等.这些标准样品部分已在国内外实验室得到了广泛使用,说明我国学者在副矿物U-Th-Pb定年研究方面做出了重大贡献.尽管已有众多常见副矿物U-Th-Pb分析的标准样品,但大部分标样存在U、Th元素含量低、含矿物包裹体或普通铅含量高等缺点.并且,值得注意的是大部分样品储量严重受限,多数为重量较小的单晶颗粒,不利于该矿物标样的长期使用和全球多家实验室间的数据对比.近年来国内外相继建立了大量的微区分析实验室,对副矿物U-Th-Pb年代学分析标准样品的需要也日益增加.因此,亟需进一步开展储量丰富、高质量的副矿物标准样品研发工作,以满足国内外副矿物U-Th-Pb年代学微区分析实验室高质量科研需求.此外,我国用于副矿物U-Pb年龄微区分析的有证标准物质极少,仅有中科院地质与地球物理研究所研制的一个锆石铀铅年龄微区分析标准物质.因此,加强副矿物U-Th-Pb年龄微区分析国家标准物质的研发也是将来重要的研究课题.

4 普通铅校正问题

副矿物U-Th-Pb定年分析需要准确测定放射性成因子体和母体同位素组成,并根据放射性衰变理论进行年龄计算.而在实际分析中需要考虑非放射性成因子体Pb同位素的影响,我们通常将所有非放射性成因铅统称为普通铅,主要包括在副矿物初始结晶过程中混入矿物晶格的初始铅以及分析过程中带来的污染等(Andersen, 2002).在U-Th-Pb衰变体系中,^{206}Pb、^{207}Pb和^{208}Pb分别由^{238}U、^{235}U和^{232}Th放射性衰变形成,^{204}Pb属于非放射性成因同位素.如以^{238}U-^{206}Pb体系为例,若所测矿物中存在普通铅,则:

$$^{206}Pb_m = {}^{206}Pb_c + {}^{206}Pb^* = {}^{206}Pb_c + {}^{238}U(e^{\lambda_{238}t}-1), \quad (1)$$

式中$^{206}Pb_m$为质谱测试的总^{206}Pb信号,$^{206}Pb_c$为普通铅组成,$^{206}Pb^*$为放射性成因Pb,λ_{238}为^{238}U的衰变常数.由等式(1)计算的$^{206}Pb/^{238}U$年龄为:

$$t_{206} = \frac{1}{\lambda_{238}}\ln\left(\frac{^{206}Pb_c + {}^{206}Pb^*}{^{238}U}+1\right), \quad (2)$$

由等式(2)可知,由于普通铅的混入会造成年龄测试值高于其真实值.因此,在开展副矿物U-Th-Pb年龄测试过程中需对矿物中所含普通铅进行校正.

普通铅校正即为通过测定样品中普通铅组成,计算比例对其进行扣除.最简单直接的方法是通过测定非放射性成因^{204}Pb组成,再对矿物中普通铅直接校正扣除.该方法的要求是准确测定^{204}Pb信号,在ID-TIMS和SIMS分析中常用此方法进行普通铅校正.但对于LA-ICP-MS分析,^{204}Pb方法的使用受到诸多限制,主要原因为:(1)LA-ICP-MS所使用气体中Hg背景含量较高,^{204}Hg对^{204}Pb的准确测定产生同质异位素的干扰;(2)^{204}Pb丰度较低,质谱(尤其是四极杆)难以对其准确测定.为准确测定副矿物中的^{204}Pb组成,LA-ICP-MS实验室常通过除汞装置(Hirata *et al.*, 2005; Hu *et al.*, 2015)或ICP-MS/MS质量筛选(Gilbert and Glorie, 2020; Xiang *et al.*, 2021)以降低或消除^{204}Hg的测试干扰.

除^{204}Pb法外,常用的还有^{207}Pb法以及^{208}Pb法,以下将对该3种校正方法基本原理进行简要阐述:

首先我们定义:$f_{206} = {}^{206}Pb_c / {}^{206}Pb_m$, (3)

f_{206}为普通铅$^{206}Pb_c$占所有$^{206}Pb_m$的比例,则放射性成因$^{206}Pb^*/{}^{238}U$可由下式计算:$^{206}Pb^*/{}^{238}U = (1-f_{206})\times({}^{206}Pb/{}^{238}U)_m$, (4)

其中:$(^{206}Pb/^{238}U)_m$为测试所得$^{206}Pb/^{238}U$比值.

(1) ^{204}Pb法校正

如上所述,^{204}Pb法校正的前提是准确测试^{204}Pb的组成,测量时需准确扣除Hg干扰,干扰扣除方程如下所示:

$$^{204}Pb = ({}^{204}Pb + {}^{204}Hg)_m - \left(\frac{^{204}Hg}{^{202}Hg}\right)\times {}^{202}Hg_m, \quad (5)$$

由于^{204}Pb为非放射性成因,测试所得^{204}Pb即全为普通铅.则等式(3)可变化为

$$f_{206} = \frac{^{206}Pb_c}{^{206}Pb_m} = \frac{\frac{^{206}Pb_c}{^{204}Pb_m}}{\frac{^{206}Pb_m}{^{204}Pb_m}} = \frac{\frac{^{206}Pb_c}{^{204}Pb_c}}{\frac{^{206}Pb_m}{^{204}Pb_m}} = \left(\frac{^{206}Pb}{^{204}Pb}\right)_c / \left(\frac{^{206}Pb}{^{204}Pb}\right)_m, \quad (6)$$

其中$(^{206}Pb/^{204}Pb)_c$可采用与待测副矿物同源的低U矿物(如斜长石、方铅矿等)铅同位素组成,或通过3D Tera-Wasserburg图解计算获得(Andersen, 2002; Krestianinov *et al.*, 2021),或由地球铅同位素演化模型(Stacey and Kramers, 1975)获得;$(^{206}Pb/^{204}Pb)_m$为测试所得.

(2) ^{207}Pb法校正

该方法为通过测试$^{207}Pb/^{206}Pb$比值计算普通铅组成,由等式(3)可得如下等式:

$$\frac{^{207}Pb^*}{^{206}Pb^*} = \frac{({}^{207}Pb/{}^{206}Pb)_m - f_{206}\times({}^{207}Pb/{}^{206}Pb)_c}{1-f_{206}}, \quad (7)$$

由等式(7)变换后可得普通铅比例计算公式如下:

$$f_{206} = \frac{({}^{207}Pb/{}^{206}Pb)_m - ({}^{207}Pb^*/{}^{206}Pb^*)}{({}^{207}Pb/{}^{206}Pb)_c - ({}^{207}Pb^*/{}^{206}Pb^*)}, \quad (8)$$

其中$({}^{207}Pb/{}^{206}Pb)_m$为测试的$^{207}Pb/^{206}Pb$比值;$({}^{207}Pb/{}^{206}Pb)_c$为初始$^{207}Pb/^{206}Pb$比值,可采用与待测副矿物同源的低U矿物(如斜长石、方铅矿等)铅同位素组成,或者由地球铅同位素演化模型(Stacey and Kramers, 1975)获得;$({}^{207}Pb^*/{}^{206}Pb^*)$为放射性Pb同位素比值,可由$^{235}U/^{238}U$比值及样品年龄估算获得,具体计算公式如下:

$$\frac{^{207}Pb^*}{^{206}Pb^*} = \frac{^{235}U}{^{238}U}\times\frac{e^{\lambda_{235}\times t}-1}{e^{\lambda_{238}\times t}-1}. \quad (9)$$

(3) ^{208}Pb法校正

该方法计算公式为:

$$f_{206}=\frac{(^{208}Pb/^{206}Pb)_m-(^{208}Pb^*/^{206}Pb^*)}{(^{208}Pb/^{206}Pb)_c-(^{208}Pb^*/^{206}Pb^*)},\quad(10)$$

其中$(^{208}Pb/^{206}Pb)_m$为测试所得$^{208}Pb/^{206}Pb$比值;$(^{208}Pb/^{206}Pb)_c$为初始$^{208}Pb/^{206}Pb$比值,同样可由与待测副矿物同源的低U矿物(如斜长石、方铅矿等)铅同位素组成获得,或由地球铅同位素演化模型(Stacey and Kramers, 1975)获得;$(^{208}Pb^*/^{206}Pb^*)$为放射性Pb同位素比值,可由样品$^{232}Th/^{238}U$比值及年龄估算获得,具体计算公式如下:

$$\frac{^{208}Pb^*}{^{206}Pb^*}=\frac{^{232}Th}{^{238}U}\times\frac{e^{\lambda_{232}\times t}-1}{e^{\lambda_{238}\times t}-1},\quad(11)$$

^{208}Pb法在放射性成因^{208}Pb较低时才能获得准确的校正结果,因此只适用于Th/U比值低的副矿物.若样品中不含Th,则该方法与^{204}Pb法类似.

需要指出的是,^{207}Pb和^{208}Pb校正法分别假定样品的$^{206}Pb/^{238}U$和$^{207}Pb/^{235}U$或$^{208}Pb/^{232}Th$为谐和年龄.如果含普通铅样品存在铅丢失,则不能直接采用^{207}Pb和^{208}Pb法进行普通铅校正,此时可使用Andersen提出的3D谐和图解进行校正,根据3D图中的几何原理进行计算,详细计算公式可见Andersen(2002).

在进行含U副矿物U-Th-Pb年龄分析时,不仅需要考虑待测样品中普通铅组成对其分析结果的影响,同时也需要考虑标准样品中普通铅组成对分析结果的影响.当所使用的副矿物标样也含有普通铅时,需先对标样进行校正,再进行元素分馏及仪器漂移校正.对标样的普通铅校正需在背景信号扣除之后,再直接对瞬时信号进行校正.具体校正公式如下:

$$^{206}Pb_r=^{206}Pb_m\times(1-f_{206}),\quad(12)$$

$$^{207}Pb_r=^{207}Pb_m\times(1-f_{207}),\quad(13)$$

其中$^{206}Pb_r$为普通铅校正后的瞬时^{206}Pb信号,$^{206}Pb_m$为测试的瞬时^{206}Pb信号,f_{206}为普通铅$^{206}Pb_c$占所有$^{206}Pb_m$的比例;同理,$^{207}Pb_r$为普通铅校正后的瞬时^{207}Pb信号,$^{207}Pb_m$为测试的瞬时^{207}Pb信号,f_{207}为普通铅$^{207}Pb_c$占所有$^{207}Pb_m$的比例.

若^{204}Pb信号可准确测定,则可采用^{204}Pb法进行校正.由式(6)可知,等式(12)、(13)可变换为:

$$^{206}Pb_r=^{206}Pb_m\times\left[1-(^{206}Pb/^{204}Pb)_c/(^{206}Pb/^{204}Pb)_m\right],\quad(14)$$

$$^{207}Pb_r=^{207}Pb_m\times\left[1-(^{207}Pb/^{204}Pb)_c/(^{207}Pb/^{204}Pb)_m\right],\quad(15)$$

其中$^{206}Pb_r$为普通铅校正后的瞬时^{206}Pb信号,$^{206}Pb_m$为测试的瞬时^{206}Pb信号,$(^{206}Pb/^{204}Pb)_c$为初始普通铅$^{206}Pb/^{204}Pb$比值,$(^{206}Pb/^{204}Pb)_m$为测试$^{206}Pb/^{204}Pb$比值;$^{207}Pb_r$为普通铅校正后的瞬时^{207}Pb信号,$^{207}Pb_m$为测试的瞬时^{207}Pb信号,$(^{207}Pb/^{204}Pb)_c$为初始普通铅$^{207}Pb/^{204}Pb$比值,$(^{207}Pb/^{204}Pb)_m$为测试的$^{207}Pb/^{204}Pb$比值.

若采用^{207}Pb法进行校正,则f_{206}可采用公式(8)进行计算,校正后的^{206}Pb和^{207}Pb信号分别为:

$$^{206}Pb_r=^{206}Pb_m\times(1-f_{206}),\quad(12)$$

$$^{207}Pb_r=^{207}Pb_m-^{206}Pb_m\times(^{207}Pb/^{206}Pb)_c\times f_{206},\quad(16)$$

其中$^{206}Pb_r$为普通铅校正后的瞬时^{206}Pb信号,$^{206}Pb_m$为测试的瞬时^{206}Pb信号,f_{206}为普通铅$^{206}Pb_c$占所有$^{206}Pb_m$的比例;$^{207}Pb_r$为普通铅校正后的瞬时^{207}Pb信号,$^{207}Pb_m$为测试的瞬时^{207}Pb信号,$(^{207}Pb/^{206}Pb)_c$为初始$^{207}Pb/^{206}Pb$比值.

需要指出的是,对于普通铅含量较高的副矿物标准样品,即使标样进行普通铅校正,仍会存在过校正或校正不足的情况,从而对待测样品的校正年龄和初始铅同位素组成造成影响.因此高质量、低普通铅或不含普通铅的矿物标样研发十分必要.

当标准样品和待分析样品均含普通铅时,采用^{207}Pb法普通铅校正以及分馏校正的示意图解如图5所示.A、B分别为同一样品中含不同普通铅比例的两点,由这些含不同普通铅的样品点构筑的回归线(蓝色实线)与y轴的交点Y为样品所含初始铅同位素组成,与谐和线下交点为经普通铅校正后的$^{206}Pb/^{238}U$年龄,与x轴下交点为X_T.在实际测试过程中,由于存在Pb/U元素分馏和仪器漂移,因此需对测试所得Pb/U比值进行校正;而$^{207}Pb/^{206}Pb$同位素分馏较小,相对于Pb/U分馏可忽略不计.假定A_1、A_2分别为样品点A、B的实际测试点,F_1、F_2分别为其分馏校正因子,则A_1、B_1也会分布于同一不一致线上(图中虚线),与x轴下交点为X_M,$F_1=A_1A_2/AA_2$;$F_2=B_1B_2/BB_2$.而A_1、B_1分别为相同仪器条件下测试的同一样品,其分馏行为一致,即$F_1=F_2$.如图所示,由相似三角形原理:

$$\frac{A_1A_2}{X_MO}=\frac{YA_2}{YO},\quad(17)$$

$$\frac{AA_2}{X_TO}=\frac{YA_2}{YO},\quad(18)$$

由等式(17)(18)可得:$\frac{A_1A_2}{AA_2}=\frac{X_MO}{X_TO}$,　(19)

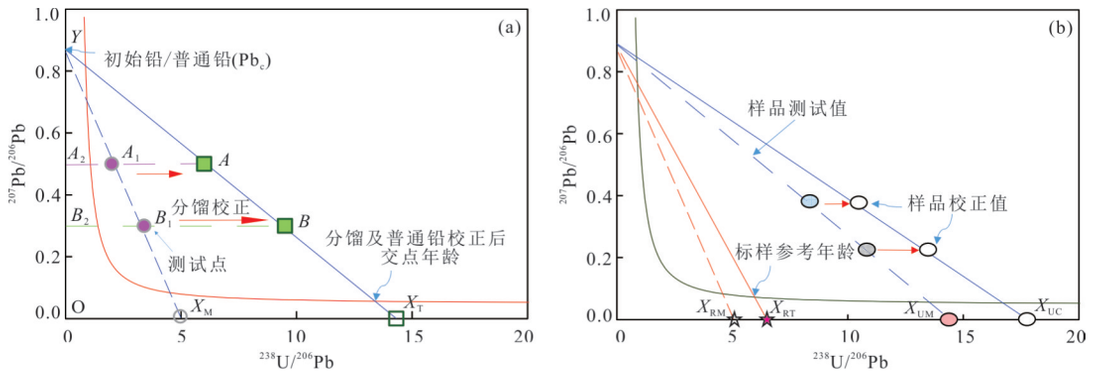

图 5 含普通铅标准样品和待测样品 ^{207}Pb 法普通铅校正及分馏校正图解

Fig. 5 The calibration procedure of common lead and U-Pb elemental fractionation

即：$F_1=\dfrac{X_MO}{X_TO}$, (20)

同理可得：$F_2=\dfrac{X_MO}{X_TO}$, (21)

等式(20)(21)表明采用含普通铅的标准样品作外标分析含普通铅样品时，可在 Tera-Wasserburg 图解中分别计算经过标样不同测试点与 x 轴的下交点 X_M，以及经过其推荐值与 x 轴的下交点 X_T，两者比值即为 Pb/U 分馏校正因子．再用此因子校正待测样品中各测试点的分馏行为．经过校正后的各分析点构筑的不一致线与 Tera-Wasserburg 图解中谐和线交点即为待测样品经元素分馏和普通铅校正后的准确 U-Pb 年龄(图 5b)．该方法现广泛应用于方解石、黑钨矿、白钨矿等含普通铅副矿物 U-Pb 年龄的准确分析(Roberts *et al.*, 2017; Tang *et al.*, 2020, 2022; Carr *et al.*, 2021; Wu *et al.*, 2022)．

对于含普通铅的低 U 副矿物，可在 U-Th-Pb 年龄测试前开展样品的面扫描分析(Drost *et al.*, 2018; Hoareau *et al.*, 2021)，进而选择 U 含量较高和 Pb/U 比值变化大的区域进行分析，以在谐和图解上成功构筑不一致线(Drost *et al.*, 2018; Hoareau *et al.*, 2021)，或采用贝叶斯回归校正(Davis and Rochín-Bañaga, 2021; Rochín-Bañaga *et al.*, 2021)，以提高此类副矿物 U-Pb 定年分析成功率．

5 高空间分辨率和高效率分析

含 U 副矿物大多具有复杂的内部结构(如继承核、增生边等)或矿物颗粒细小，因此需要开展 U-Th-Pb 年龄的高空间分辨率分析．一般情况下，对于 U 含量约几百 ppm 的副矿物(如锆石)，LA-ICP-MS 进行 U-Th-Pb 年龄分析的剥蚀束斑为 25～35 μm；对富含 U、Th 的副矿物，如独居石、晶质铀矿等，剥蚀束斑可低至 5～10 μm(Paquette and Tiepolo, 2007; Zong *et al.*, 2015)；对金红石等 U 含量较低(几十 ppm)的副矿物，分析束斑一般为 50 μm 以上(Zack *et al.*, 2011)．在纵向空间分辨率上，以平均剥蚀速率 0.1 μm/pulse 计算，激光剥蚀坑深度约为 10～40 μm．随着仪器灵敏度的提高和分析策略的改进，学者们也开展了很多 LA-ICP-MS 副矿物 U-Th-Pb 年代学的高空间分辨率分析．如使用多接收等离子体质谱实现锆石 5～15 μm 空间分辨率下的准确分析(Johnston *et al.*, 2009; Hattori *et al.*, 2017; Xie *et al.*, 2017; Lin *et al.*, 2021)和斜锆石 10 μm 剥蚀束斑条件下的 U-Pb 年龄分析(Ibanez-Mejia *et al.*, 2014)．也有研究者采用单接收磁质谱仪，开展 5～20 μm 剥蚀条件下的锆石 U-Pb 年龄高空间分辨率准确分析(Mukherjee *et al.*, 2019; Wu *et al.*, 2020)．因此，通过使用高灵敏度质谱仪，LA-ICP-MS 锆石 U-Pb 年龄分析横向空间分辨率可达 5 μm．在纵向空间分辨率方面，通过采用低频率剥蚀(Hu *et al.*, 2012; Corbett *et al.*, 2020)或单脉冲(Cottle *et al.*, 2009; Kelly *et al.*, 2014)分析可实现 LA-ICP-MS 锆石 U-Pb 年龄的高空间分辨率深度剥蚀，纵向空间分辨率可达 0.1 μm(Iwano *et al.*, 2021)．采用单脉冲剥蚀获取的每个分析点信号为短暂瞬态信号，对此类瞬态信号数据处理时可采用全计数积分(Cottle *et al.*, 2009; Johnston *et al.*, 2009)、线性回归计算 Pb/U 同位素斜率(Fietzke *et al.*, 2008; Feng *et al.*, 2022)和计算平均值(Iwano *et al.*, 2021)等方法计算其同位素比值和不确定度．使用多接收杯等离子体质谱分析时，通常采用法拉

第杯接收U、Th信号,而离子计数器接收Pb同位素信号,不同检测器对U、Th、Pb信号的响应时间也是影响瞬态信号分析结果的重要因素(Cottle *et al.*, 2009; Johnston *et al.*, 2009; Claverie *et al.*, 2016; Craig *et al.*, 2020).采用这种高空间分辨率的分析方式,学者们对变质锆石的增生年龄(Viete *et al.*, 2015)和榍石的变质演化历史(Stearns *et al.*, 2016)等复杂地质年龄进行了准确分辨.今后可结合仪器信号增敏研究、优化剥蚀参数、改进气溶胶传输系统,进一步提升LA-ICP-MS副矿物U-Th-Pb年代学分析的微区空间分辨能力.

碎屑锆石年代学广泛应用于地壳演化、沉积物起源及古气候重建等地质作用研究(Schoene, 2014;Zhang *et al.*, 2022),激光剥蚀等离子体质谱由于其样品分析速度快等优势,常被应用于碎屑副矿物U-Th-Pb年代学分析(Pettit *et al.*, 2019;Sylvester *et al.*, 2022).为准确统计刻画碎屑锆石的起源,其样品分析点数量往往很大(Vermeesch, 2004; Pettit *et al.*, 2019).而当前LA-ICP-MS常规锆石U-Pb年龄单点分析时间约为2分钟,碎屑锆石年代学研究对激光剥蚀等离子体技术的分析效率提出了更高要求.上述单脉冲分析技术(Cottle *et al.*, 2009, 2012)通过在单颗锆石表面单次脉冲剥蚀获得可靠年龄即是一种高效率分析模式,可实现约100点/h的U-Pb年龄分析.还有学者采用激光高频剥蚀实现每小时约250~300点的分析(Chew *et al.*, 2019;Feng *et al.*, 2022),Sundell *et al.* (2021)通过优化软件算法,理论上可实现的最高分析效率约为1 200点/h,其单点分析耗时仅3 s.但获取速度的提升会对年龄结果的准确度和精度造成一定影响,在高效率分析时获得的平均年龄偏差达1.1%,随机误差可达1.3%(1*s*)(Sundell *et al.*, 2021).U-Th-Pb年龄快速分析技术极大提高了激光剥蚀等离子体质谱技术碎屑锆石年代学研究的分析效率,有助于增强碎屑副矿物U-Th-Pb年代学的广泛应用.

6　结论与展望

近几十年来,随着激光剥蚀电感耦合等离子体质谱仪器的发展和分析技术的提高,LA-ICP-MS副矿物U-Th-Pb定年分析取得了长足的进步和发展.在元素分馏和基体效应抑制策略、基体匹配矿物标准样品研发、普通铅校正方案、高空间分辨率及高效率分析等方面均取得了系列重要进展.但人们对高分析性能的追求是永无止境的,如何进一步减小或消除元素分馏和基体效应等对分析结果的影响,以进一步提高副矿物U-Th-Pb定年结果的准确性、精密度和空间分辨能力仍是需要努力的重要方向.

尽管当前已报道众多用于不同副矿物相U-Th-Pb年龄及同位素分析的标准样品,但高质量的副矿物U-Th-Pb年龄微区分析标样仍然非常欠缺.特别是近年来大量微区分析实验室的相继建立,使高质量副矿物U-Th-Pb定年分析标样供需矛盾日益突出.因此持续开展不同种类储量丰富的高质量标准样品研发是副矿物U-Th-Pb定年分析研究的重要内容.

近年来,由于激光剥蚀等离子体质谱的高灵敏度及可快速面扫描等优势,建立了大量的富含普通铅、低U副矿物的U-Th-Pb定年新方法,为厘定重要地质事件的时限提供了新的约束手段,展现出广阔的地质应用前景.如何更进一步提升仪器灵敏度,开发更多低U副矿物的高空间分辨率U-Th-Pb年龄分析新方法,也是未来LA-ICP-MS副矿物年龄分析的研究重点.

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附表见本刊官网(http://www.earth-science.net).

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