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Reappraisal of the applicability of MK-1 apatite as a reference standard for (U—Th)/He geochronology

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ABSTRACT

The (U—Th)/He dating technique is widely used in studying the shallow-crustal geological processes including exhumation of orogenic belts, surface weathering, fault motion, topographic evolution. A reference material with homogeneous composition and highly reproducible (U—Th)/He dates is crucial to get accurate and precise date results for the unknown samples. As the most commonly used reference material, the Durango apatite has its limitations such as heterogeneous distribution of parent isotopes and large date dispersion for different fragments. In order to overcome the shortcomings of Durango apatite and meet the increasing demand for reference material, we developed a new reference material, MK-1 apatite, which was collected from the Mogok metamorphic belt (MMB) of Myanmar, for the (U—Th)/He community. In this study, we presented dating results of 191 apatite fragments in six different laboratories, which showed highly reproducible dates and Th/U ratios, with an average date of 18.01 ± 0.37 Ma (sd.) and Th/U ratio of 0.72 ± 0.06 (sd.) respectively. Combined with textural and compositional results in the previous study (Wu et al., 2019), we concluded that he MK-1 apatite could serve as a good new reference material for (U—Th)/He geochronology. We use the weighted mean dates of 17.99 ± 0.02 Ma that obtained from six different laboratories as the intercalibrated dates for the new reference material.

1. Introduction

The distinctive crystallographic structure and composition of apatite make it widely used in numerous research fields (Hughes and Rakovan, 2015). From the geological perspective, apatite is the most ubiquitous rock-forming mineral that residing in all kind of crustal rocks. In apatite structures, extensive substitution between trace elements and calcium makes it a good carrier for radioactive isotopes such as uranium and thorium. This characteristic of apatite forms the foundation of geochronological researches of apatite including U—Pb, fission track and (U—Th)/He techniques (Chew and Spikings, 2015, David et al., 2019; Malusà and Fitzgerald, 2020).

The (U—Th)/He dating technique, which uses the decay of nuclides, ^{238}U, ^{235}U, ^{232}Th, and ^{147}Sm, and the accumulation of their daughter isotope, ^{4}He, to date minerals, is widely used in studies of shallow-crustal geological processes (Ehlers and Farley, 2003). (U—Th)/He

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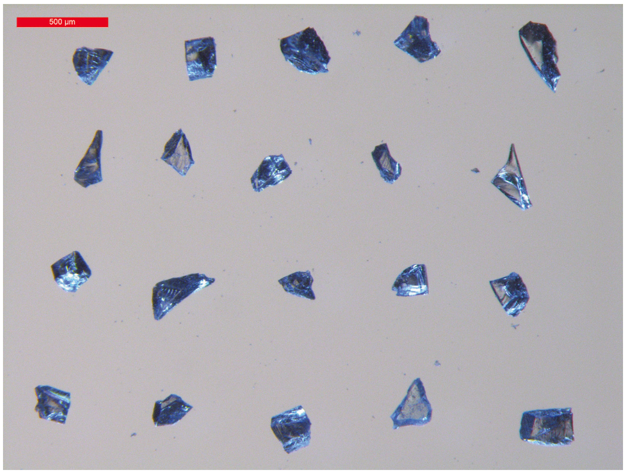

Fig. 1. Typical fragments of the MK-1 apatite used for (U—Th)/He geochronology.

geochronology is an absolute dating method, meaning that the results do not depend upon a reference material. However, because date reference materials are used to inspect experimental procedures, it is still very important to get precise and accurate geological dates. At present, the most commonly used apatite reference material used by both the (U—Th)/He and fission track communities is the Durango apatite (McDowell et al., 2005). However, this gem-quality apatite is known to exhibit large (U—Th)/He date dispersion between individual fragments partially due to the heterogeneous distribution of parent isotopes revealed by laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) analysis (Boyce and Hodges, 2005). To overcome the limitations of Durango apatite as a reference material and meet increasing demand for more homogenous reference materials, Wu et al. developed a new potential reference standard: MK-1 gem-quality apatite from the Mogok metamorphic belt in Myanmar (Wu et al., 2019). As a reference material for (U—Th)/He geochronology, it should meet at least three criteria: (1) good (U—Th)/He date reproducibility, (2) homogeneous both in structure and chemistry (such as uranium and thorium distribution), and (3) gem-quality crystals that have no inclusions and avoiding uncertainties resulted from alpha-ejection correction (Farley et al., 1996). Detailed structural, chemical and geochronological studies (Wu et al., 2019) suggest that the MK-1 apatite satisfied all the three criteria mentioned above. In this study, we summarized (U—Th)/He date results in six different laboratories and reemphasized that the MK-1 apatite is a good new reference material for (U—Th)/He geochronology.

2. Sample description and analytical method

The MK-1 apatite, which was collected from the Mogok metamorphic belt (MMB) of Myanmar, is a centimeter-scale gem-quality megacryst developed in the ruby-bearing marble (Fig. 1 of Wu et al., 2019). This crystal of MK-1 is quite transparent with blue color. Detailed geological background of the MMB and the sample was described in Wu et al. (2019).

Apatite (U—Th)/He results were produced in six different laboratories: $^{40}Ar/^{39}Ar$ and (U—Th)/He geochronology laboratory, Institute of Geology and Geophysics, Chinese Academy of Sciences (IGGCAS), John de Laeter Research Centre of Curtin University (CU), LA-ICP-MS laboratory of Institute of Geochemistry, Chinese Academy of Sciences (IGCAS), Isotopic laboratory of Institute of Geology, Chinese Academy of Geological Sciences (IGCAGS), Neotectonics Geochronology laboratory of Institute of Geology, China Earthquake Administration (IGCEA), (U—Th)/He laboratory at the National Institute of Natural Hazards, Ministry of Emergency Management of China (NINH). In order to avoid the uncertainty caused by alpha-ejection effect (Farley et al., 1996), the inner part of the MK-1 was crushed to small fragments of 200–300 μm in long dimension (Fig. 1) using agate mortar. Each fragment was wrapped in a 1 mm long × 1 mm diameter platinum (Pt) capsule and loaded into a drilled oxygen-free copper disk for helium extraction using fully automatic helium extraction line named Alphachron MK II (Australian Scientific Instrument Pty Limited). After helium extraction, Pt-wrapped grains were transferred to Savillex PFA vials and spiked with $^{230}Th—^{235}U$ solution with known concentration. All the spiked solutions were measured on inductively coupled plasma mass spectrometry (ICP-MS). (U—Th)/He dates were calculated by IsoplotR (Vermeesch, 2018). Detailed analytical procedure was described in Wu et al., 2019.

In order to access the effect of the ^{147}Sm derived ^{4}He to the measured dates, laser ablation ICP-MS trace elements analysis were performed on a Thermo Fisher X Series-II ICP-MS equipped with an Resonetics M50LR 193 nm laser ablation system at IGGCAS on five randomly selected fragments of MK-1 apatite. The analytical conditions involved an ablation pit of 33 μm diameter, an ablation time of 15 s, a repetition rate of 7 Hz and a laser beam energy density of 3.5 J/cm^2. NIST 612 standard glass was used as internal reference material for concentration calculation. Durango (DUR), Madagascar (MAD) and McClure Mountain apatites (MMAP) were used as external references to monitor the external uncertainties.

3. Results

(U—Th)/He date results of one hundred and ninety-one MK-1 fragments were shown in Table S1 and summarized in Table 1, Fig. 2. LA-ICP-MS trace element results were shown in Table S2.

One hundred and twelve fragments (including twenty-one fragments from Wu et al., 2019) obtained by IGGCAS got (U—Th)/He dates between 16.97 ± 0.32 Ma and 18.50 ± 0.35 Ma. The population forms a normal distribution with the weighted mean of 18.01 ± 0.03 Ma (Table S1, Fig. 2b). The Th/U ratios range between 0.61 and 0.80, with an average value of 0.71 ± 0.03.Seventy-nine fragments from other five laboratories got weighted mean dates and Th/U ratios of 18.06 ± 0.26 Ma and 0.69 ± 0.01 (Curtin University, $N = 9$, Wu et al., 2019), 17.88 ± 0.07 Ma and 0.74 ± 0.05 (Institute of Geochemistry, Chinese Academy of Sciences, $N = 23$), 18.09 ± 0.15 Ma and 0.71 ± 0.02 (Institute of Geology, Chinese Academy of Geological Sciences, $N = 7$), 17.88 ± 0.06 Ma and 0.73 ± 0.02 (Institute of Geology, China Earthquake Administration, $N = 21$), 18.12 ± 0.07 Ma and 0.81 ± 0.10 (National Institute of Natural Hazards, Ministry of Emergency Management of China, $N = 19$), respectively, which were indistinguishable within error from each other (Table 1).

All 191 fragments (including 161 newly obtained fragments and 30 fragments from Wu et al., 2019) yielded (U—Th)/He dates ranging from 16.97 ± 0.32 Ma to 18.92 ± 0.32 Ma, with a weighted mean date of 17.99 ± 0.02 Ma ($N = 191$, MSWD = 1.41) (Fig. 2a, Table 1, Table S1). The Th/U ratios for these fragments ranged from 0.61 to 0.89 with an average value of 0.71 ± 0.04 (Table S1).

The isochron date can be used to detect "parentless helium" (e.g., helium in inclusions), as proposed by Vermeesch, 2010. All the data from each laboratory were plotted on the [He]-[P] ([P] represents the present-day helium production rate, which can be determined by the concentration and decay constant of the parent isotopes) correlation diagram, and all the points defined a straight line (Fig. 3). We calculated the isochron dates of 18.08 ± 0.04 Ma, 17.86 ± 0.16 Ma, 18.19 ± 0.55 Ma, 17.87 ± 0.51 Ma, 17.64 ± 0.11 Ma, 18.09 ± 0.12 Ma respectively in the six laboratories (Fig. 3), which were indistinguishable within uncertainty from the arithmetic average and weighted mean in each laboratory (Table 1). The intercept values on the vertical axis ranged from −8.5e-17 to 2.4e-15 mol, which were indistinguishable from zero, suggested that there is no "parentless helium" in these fragments. ^{147}Sm-derived ^{4}He could contribute to the total amount of ^{4}He released from an apatite grain and could cause overestimated (U—Th)/He dates

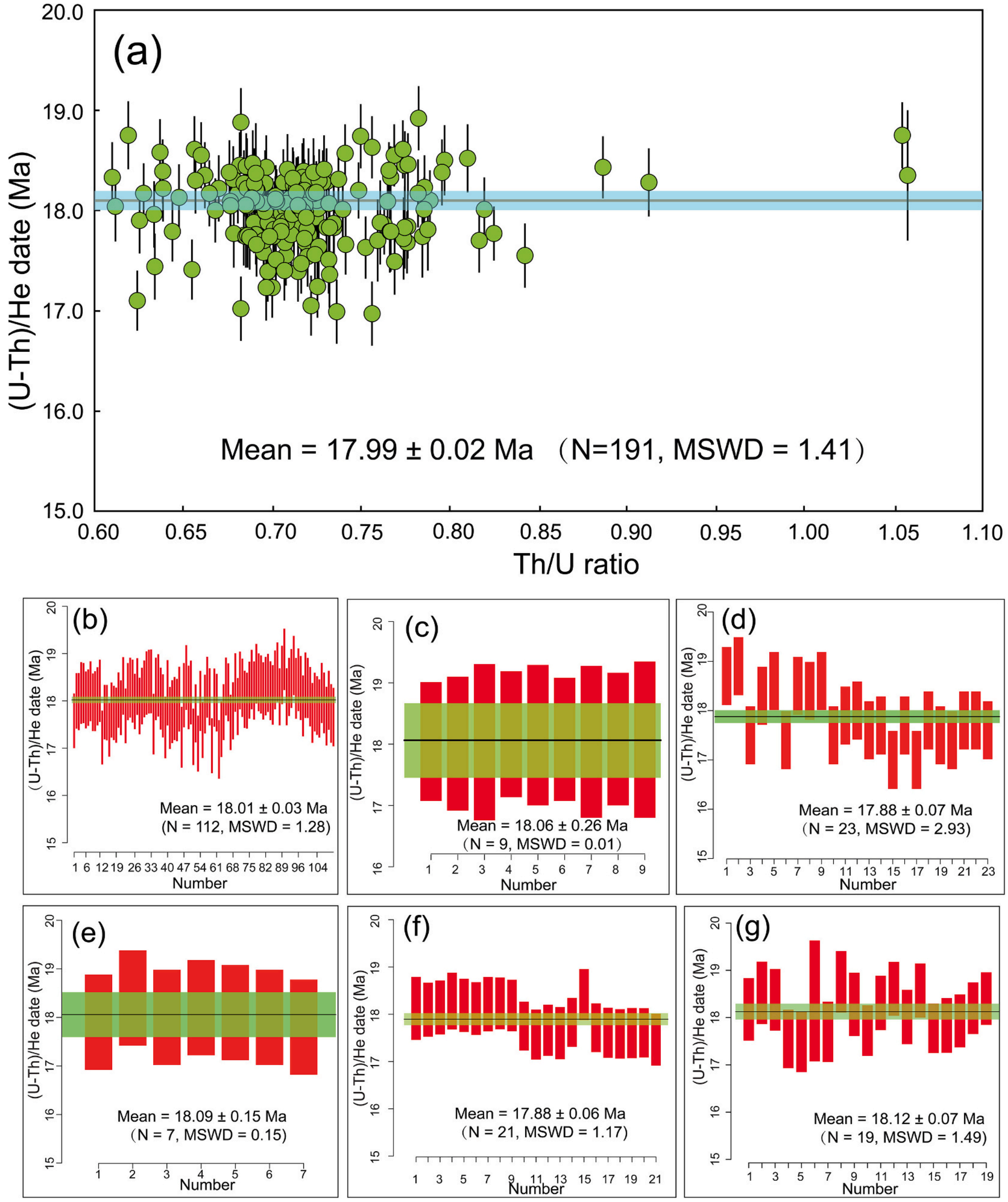

Fig. 2. (a) (U—Th)/He date vs. Th/U ratio for all 191 fragments of MK-1 apatite in six different laboratories; (b) weighted mean date for 112 fragments of MK-1 at IGGCAS (including 21 fragments from Wu et al., 2019); (c) weighted mean date for 9 fragments of MK-1 at CU (Wu et al., 2019); (d) weighted mean date for 23 fragments of MK-1 at IGCAS; (d) weighted mean date for 7 fragments of MK-1 at IGCAGS; (f) weighted mean date for 21 fragments of MK-1 at IGEAS; (g) weighted mean date for 19 fragments of MK-1 at NINH.

(Fitzgerald et al., 2006). This effect is more obvious when apatite contains low uranium and thorium concentrations. However, for MK-1 apatite, LA-ICP-MS analyzing results showed that the samarium concentration ranged between 3.6 and 11.1 μg/g (Fig. 4, Table S2), which could only cause 0.02% underestimation for the ^{4}He production and the effect of ^{147}Sm-derived ^{4}He could be negligible.

4. Discussion

4.1. Homogeneity and date reproducibility

The most important requirement for a good (U—Th)/He dating reference material is its chemical homogeneity. Chemical variation (such as uranium and thorium) revealed by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) analysis could partially lead to date dispersion that far exceeds analytical uncertainty, which was verified in most widely used Durango apatite (Boyce and Hodges, 2005). Chemical inhomogeneity (such as zoning, fluid and/or gas inclusions) could also compromise the parent/daughter isotope distributions and therefore the (U—Th)/He dates (Danišík et al., 2017). The MK-1 apatite was proved to be chemically homogeneous by detailed microanalysis (Wu et al., 2019) at single fragment level and between different fragments.

Arithmetic mean dates of 18.02 ± 0.36 Ma (2.00%, $N = 112$), 18.05 ± 0.09 Ma (0.49%, $N = 9$), 17.88 ± 0.54 Ma (3.02%, $N = 23$), 18.05 ± 0.19 Ma (1.05%, $N = 7$), 17.90 ± 0.30 Ma (1.68%, $N = 21$), 18.13 ± 0.38 Ma (2.10%, $N = 19$) were obtained respectively from six different laboratories, which were all in agreement within uncertainty with the

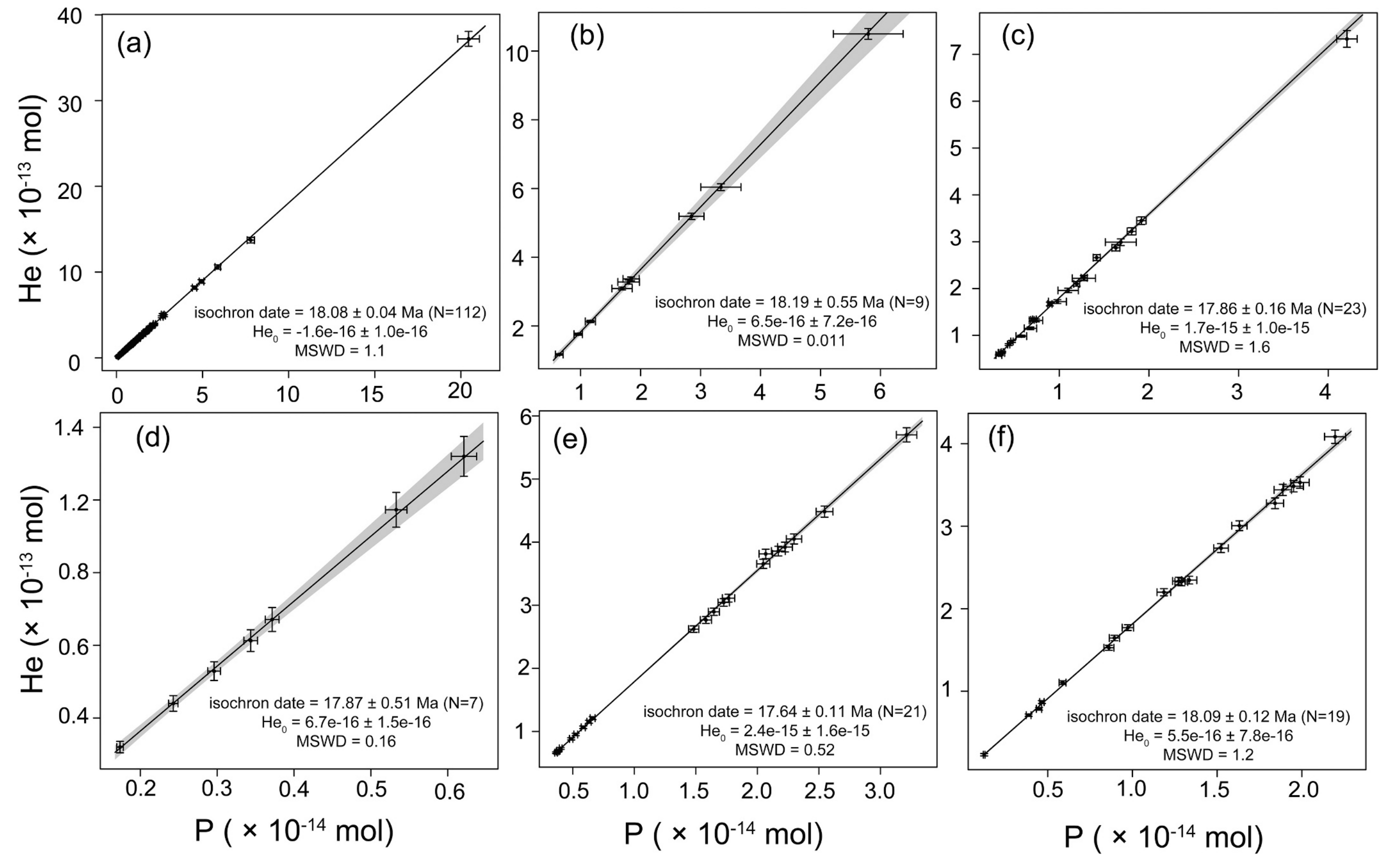

Fig. 3. Isochron date results of MK-1 apatite fragments at (a) IGGCAS; (b) CU; (c) IGCAS; (d) IGCAGS; (e) IGCEA; (f) NINH.

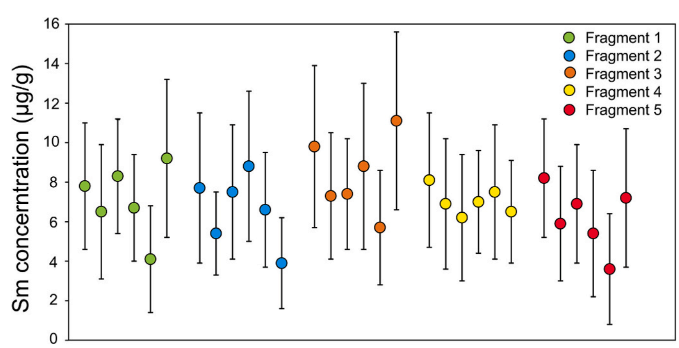

Fig. 4. LA-ICP-MS analyzing results for Sm concentration of five MK-1 apatite fragments.

published date of 18.0 ± 0.2 Ma (Wu et al., 2019). These results show that the MK-1 apatite fragments could get highly reproducible dates both intra-laboratory and between different laboratories, which meets the most important criteria for a potential (U—Th)/He dating reference material.

4.2. Calibrated date for MK-1 apatite

Previous research (Wu et al., 2019) gave a mean (U—Th)/He date of 18.0 ± 0.2 Ma for thirty randomly selected fragments of MK-1 apatite in two laboratories. In this study, to better constrain the accurate date and make intra-lab comparison, we analyzed one hundred and ninety-one fragments (including thirty fragments from Wu et al., 2019, Table S1) in six different laboratories (Table S1, Figs. 2, 3, 4). We got weighted mean dates of 18.01 ± 0.03 Ma, 18.06 ± 0.26 Ma, 17.88 ± 0.07 Ma, 18.09 ± 0.15 Ma, 17.78 ± 0.06 Ma, and 18.12 ± 0.07 Ma in six different laboratories respectively (Fig. 2; Table 1). The dates of 17.88 ± 0.07 Ma and 17.78 ± 0.06 Ma were slightly younger than the other mean dates probably be due chemical variations of the uranium and thorium distribution (Table S2). The weighted mean date of all these six laboratory mean dates was summarized together (Fig. 5) and yielded a 17.99 ± 0.02 Ma ($N = 6$, MSWD = 2.05), which was indistinguishable from the

Table 1
(U—Th)/He date summary in six different laboratories, detail results for each fragment see the Supplementary Table S1.

Laboratory	N[c]	Arithmetic mean date (Ma)	1S.D. (Ma)	Weighted Mean date (Ma)	± 1σ (Ma)	MSWD	Isochron Date (Ma)	± 1σ (Ma)	MSWD	Intercept value (mol)	± 1σ (mol)
IGGCAS[a]	112	18.02	0.36	18.01	0.03	1.28	18.08	0.04	1.10	-1.6E-16	1.0E-16
CU[b]	9	18.05	0.09	18.06	0.26	0.01	17.86	0.16	1.60	1.7E-15	1.0E-15
ICCAS	23	17.88	0.54	17.88	0.07	2.93	18.19	0.55	0.01	6.5E-16	7.2E-16
IGCAGS	7	18.05	0.19	18.09	0.15	0.15	17.87	0.51	0.16	6.7E-16	1.5E-16
IGDEA	21	17.90	0.30	17.88	0.06	1.17	17.64	0.11	0.52	2.4E-15	1.7E-15
NINH	19	18.13	0.38	18.12	0.07	1.49	18.09	0.12	1.20	5.5E-16	7.8E-16
All	191	18.01	0.37	17.99	0.02	1.41	18.06	0.03	1.10	−8.5E-17	9.8E-17

Notes: [a]twenty-one fragment date results are from Wu et al., 2019; [b]nine fragment date results are from Wu et al., 2019; [c]numuber of dated fragments.

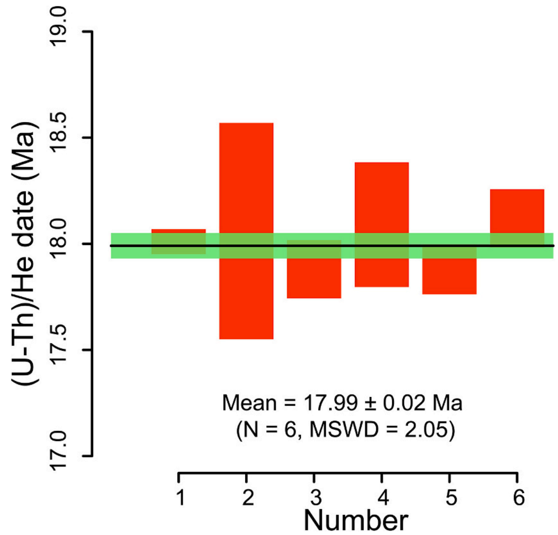

Fig. 5. Mean date of six weighted mean dates from different laboratories as the calibrated date for MK-1 apatite reference material.

weighted mean of all 191 fragments (Fig. 2, Table 1). We used this date as the calibrated date for the new apatite reference material, which was in good agreement with the published date (Wu et al., 2019).

4.3. Significance of the new apatite reference material

In recent years, to fulfill demand for reference material, new apatite and zircon (U—Th)/He reference materials were developed by different laboratories (Kraml et al., 2006; Li et al., 2017; Tian et al., 2017; Wu et al., 2019; Yu et al., 2020). In (U—Th)/He and fission track community, the Durango apatite is the most commonly used reference material. However, as a geochronological reference material, the Durango apatite has its limitations such as heterogeneity of the parent nuclides and over-dispersed (U—Th)/He dates far beyond the analytical uncertainty (Boyce and Hodges, 2005). To compare the date reproducibility of MK-1 apatite with the extensively used Durango apatite, we randomly selected 53 (U—Th)/He dates of Durango fragments analyzed in IGGCAS helium laboratory (Table S2, Fig. 6). 53 Durango apatite fragments yielded identical weighted mean and central dates (Vermeesch, 2018) 31.42 ± 0.31 Ma ($N = 53$, MSWD = 14.8) with a dispersion of 7.0%. We selected 53 fragments from our 191 dates and made a new population. This population got weighted mean date of 18.18 ± 0.05 Ma (N = 53, MSWD = 0.86), with a dispersion of 0.35% (Fig. 6a, c), which is much less scattered than the Durango date distribution (Fig. 6). This result suggests that the MK-1 apatite is more reproducible and less dispersed than the Durango apatite as a reference material.

1. Due to the low closure temperature of helium in U,Th-bearing minerals, (U—Th)/He became an important tool in studying the shallow crustal geological processes such as exhumation of orogenic belts, surface weathering, fault motion, topographic evolution and so on. However, apatite from slowly cooled geological terranes often

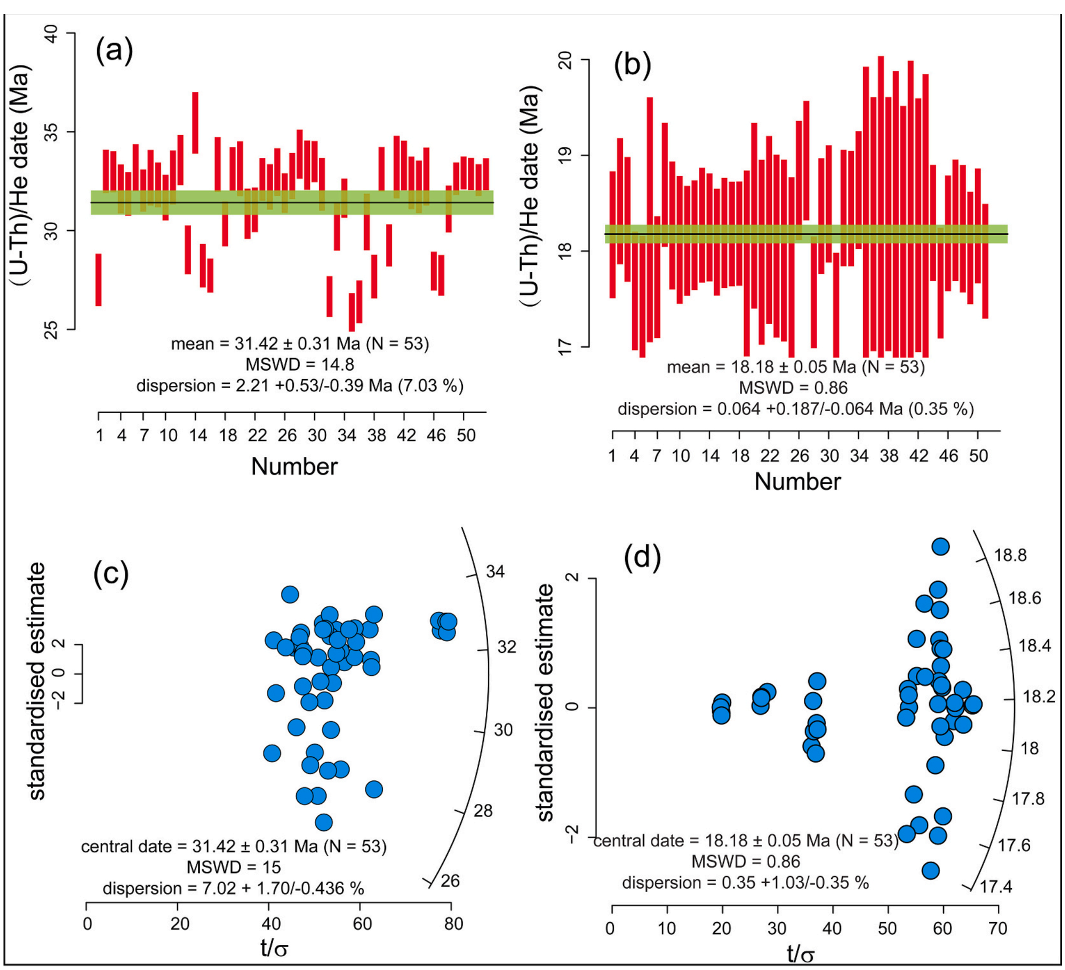

Fig. 6. (a, b) Weighted mean dates of 53 Durango and MK-1 fragments; (c, d) ratio plots of 53 Durango and MK-1 fragments.

yielded over dispersed dates much larger than the analytical uncertainty (Fitzgerald et al., 2006). Therefore, a highly reproducible reference material is crucial to get credible date results for the unknown samples by minimizing uncertainty caused by inhomogeneity of the reference material. Multidisciplinary study suggested that the MK-1 apatite was homogeneous in structure, chemical composition, and (U—Th)/He dates (Wu et al., 2019), which was superior to extensively utilized Durango apatite as a (U—Th)/He reference standard. Consistent (U—Th)/He dates reported in multiple laboratories reconfirmed that the MK-1 apatite could be served as a worldwide reference material for the (U—Th)/He community. The MK-1 megacrystal was 10 mm in long dimension (Wu et al., 2019, Fig. 1). Tens of thousands of 100 μm sized fragments was acquired after crushing and sieving. Anyone who is interested in calibrating this reference material can get one aliquot of dozens of fragments to start by contact the corresponding author of this paper. Although the amount of Mk-1 is somewhat limited at the date of publication, but we are obtaining more material from the field and more crystals will be available soon.

5. Conclusion

In this study, we reported 191 (U—Th)/He dates (including 30 previous published dates and 161 newly obtained dates) of MK-1 apatite fragments yielded from six different laboratories. All the dates were consistent with the published data. Moreover, statistical analysis of dates in each separate laboratory suggested that the MK-1 apatite could get highly reproducible (U—Th)/He dates in different laboratories. Weighted mean date of mean dates from all six laboratories of 17.99 ± 0.02 Ma ($N = 6$, MSWD = 2.05) was adopted as the calibrated date for the new apatite reference material. Comparison between MK-1 and Durango apatites suggested that the MK-1 apatite could get more reproducible and less dispersed dates than Durango apatite. Combined with previous chemical observations, we concluded that the MK-1 apatite could serve as a good new reference material for (U—Th)/He geochronology.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemgeo.2021.120255.

Declaration of Competing Interest

The authors declare that they have no conflict of interest.

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