U-Pb (ID-TIMS) Geochronological Studies of High-Uranium Metamict Zircons: New Opportunities of Familiar Approaches

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Abstract—The possibility of the application of high-uranium self-irradiation-damaged metamict zircon for U-Pb geochronological studies (ID-TIMS) after preliminary high-temperature annealing and acid treatment was demonstrated using zircon from the Li–F granites of the Turga massif (Eastern Transbaikalia). Optimal conditions of high-temperature annealing and acid treatment of zircon with high self-irradiation α -dose can be selected to remove completely a metamict phase and preserve sufficient quantities of the mineral for U-Pb (ID-TIMS) studies.

Keywords: zircon, high-temperature annealing, acid treatment, U-Pb geochronological studies, Turga massif, Eastern Transbaikalia

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INTRODUCTION

Self-irradiation caused by the presence of uranium admixture and inclusions of uranium-bearing minerals usually causes the modification of crystal lattice of zircon and local amorphization. As known, the migration ability of radiogenic lead isotopes depends on the state of crystal lattice, and respectively, on the degree of irradiation damages of zircon structure (Holland and Kupl, 1950; Cherniak et al., 1991). The losses of radiogenic lead and uranium cause the disturbance of U/Pb isotope ratios in zircon and, respectively, the discordance of obtained age values. In addition, zircon with damaged structure can contain common lead admixture, which introduces additional errors and uncertainties in the assessment of its U-Pb age.

In some cases, a technique of preliminary high-temperature annealing (Mattinson, 2005) and acid treatment (chemical abrasion) (Makeev, 1981; Mattinson, 1994) are successfully used for U-Pb dating of radiation damaged zircons. The high-temperature annealing partially recovers the zircon crystallinity, while acid treatment provides removal of damaged domains as well as mineral and fluid inclusions. Chemical abrasion is usually applied to zircon with well preserved crystal structure. However, this approach has not yet been used for zircons with heavy self-irradiation damages (with self-irradiation α -dose of $D_\alpha > 2 \times 10^{18}$ α -decay/g, after (Zamyatin et al.,

2019)) (Mattinson, 2005; Huyskens et al., 2016; Widmann et al., 2019).

In this paper, the optimal conditions of high-temperature annealing and chemical abrasion of high-uranium zircon for its U-Pb dating (ID-TIMS) were determined using zircon from Li–F granites of the Turga massif (Eastern Transbaikalia) as an example. It is shown that the developed methodical approaches are efficient for dating of high-uranium metamict zircons with a high self-irradiation α -dose (D_α).

BRIEF GEOLOGY OF THE TURGA MASSIF

The Turga Li–F granite massif of the Kukulbey complex is located in the Turga River valley (Fig. 1), 350 km southeast of Chita, in the Turga–Kalangui ore zone with rare metal–gold–fluorite mineralization (*Ob'yasnitel'naya ...*, 2001). The massif is restricted to the core of brachyanticlinal fold made up of Lower Jurassic sediments of the Onon and Onon–Borzya groups represented by mudstone, siltstone, sandstone, and conglomerates. In the northwest, the massif is in contact with Lower Jurassic sedimentary rocks, Paleozoic limestone, and dolomite, as well as granitoids of the Undin granite–granodiorite complex (Troshin et al., 1983). The most part of the massif is occupied by the Li-siderophyllite granites of the first phase. The amazonite granites of the second phase compose individual stocks and dike swarms confined to the contact zone with monzonites of the Shakhtama Complex.

Fig. 1. Schematic geological map of the Turga massif (after Zaraisky et al., 2009).

(1) Undin granite–granodiorite complex (P_1); (2) Shakhrama monzodiorite–granodiorite–granite complex (J_{2-3}); (3–4) Kukulbey granite–leucogranite complex (J_3): (3) Li-siderophyllite granites of phase I of the Turga massif, (4) amazonite granites of phase II of the Turga massif; (5) terrigenous sediments of the Onon and Onon–Borzya groups (J_{1-2}): mudstone, siltstone, sandstone, conglomerate; (6) geochronological sampling localities and their numbers.

Geochronological studies were carried out for representative samples from both rock varieties of the Kukulbey complex: medium-grained equigranular granites with Li-siderophyllite and medium to fine-grained lithionite–amazonite–albite granites (Fig. 1). The granites of the Turga massif are subalkaline and ascribed to the peraluminous rare-metal granites, but have the elevated contents of high-field strength elements and contain apatitic accessory mineralization (Syrtsso et al., 2021).

METHODS

The composition of zircons from granites of the Turga massif was studied on a Hitachi S-3400N scanning electron microscope equipped with Oxford Instruments X-Max20, AzTec Energy 350 energy dispersive spectrometer at $U = 20$ kV, $I = 1.7$ nA, working distance of 10 mm, beam diameter 5 μ m, and acquisition time of 30 s (Centre for Geo-Environmental Research and Modelling (GEOMODEL), St Petersburg University Research Park). Raman spectra were recorded on an Horiba Jobin-Yvon LabRam HR 800 spectrometer equipped with a solid-state laser ($\lambda = 532.37$ nm, power of 100 mW) and on an Olympus BX 41 microscope equipped with $\times 10$ and $\times 50$ objectives (diameter of analyzed area of 2 μ m, acquisition time of 50–100 s, measurement error of 0.5 cm^{-1}) at the GEOMODEL Centre. The calibration was made using a 546.07-nm band of mercury lamp, which for used laser corresponds to a Raman shift of 471.26 cm^{-1} . Cathodoluminescence of the zircon was studied on a TESCAN VEGA3 scanning electron microscope

($U = 15$ kV, working distance of 12.5–13.0 mm) at the Institute of Precambrian Geology and Geochronology of the Russian Academy of Sciences, in St. Petersburg. Secondary electron (SE) images of zircon crystals were obtained on a HITACHI TM 3000 SEM (Resource Centre for Microscopy and Microanalysis, Research Park of the St. Petersburg State University).

The most transparent zircon crystals (40–300 grains) selected for U–Pb geochronological studies were subjected to high-temperature annealing in a SNOL E5CC muffle furnace in ceramic or quartz crucibles at 850 and 900°C for 48 hours (Mattinson, 2005) with subsequent acid treatment by 35% HF + 15% HNO₃ in proportions 5 : 1 for 2–6 h within a temperature range of 180–230°C. After the preliminary treatment, zircon was analyzed using standard technique (Krogh, 1973). Isotope measurements were carried out using ^{202}Pb – ^{235}U spike on a multicollector TRITON TI mass spectrometer in static and dynamic (using ion counters) regimes. The accuracy of U/Pb ratios and U and Pb contents was 0.5%. The blanks did not exceed 15 pg Pb and 1 pg U. Experimental data were processed using PbDAT and ISOPLOT softwares (Ludwig, 1991, 2003). Ages were calculated using uranium decay constants (Steiger and Jager, 1976). Corrections for common lead were introduced using Stacey–Kramers model (Stacey and Kramers, 1975).

RESULTS OF U-Pb GEOCHRONOLOGICAL STUDIES AND DISCUSSION

Zircon from phase I Li-siderophyllite granite of the Turga massif (sample Tu-0467/sb) occurs as euhedral

Fig. 2. Secondary electron (I–II), back-scattered electron (III), and cathodoluminescence (IV–VIII) images of zircon crystals (sample Tu-0467/sb) from Li-siderophyllite granite of phase I of the Turga massif. (VI, VII) and (VIII) images are zircon crystals after high-temperature annealing at 850°C and 900°C, respectively. Numerals show locations analyzed using Raman spectroscopy.

Fig. 3. Secondary electron (I–II), back-scattered electron (III–IV), and cathodoluminescence (V–XII) images of zircon crystals from amazonite granite of phase II of the Turga massif (sample Tu-832). (VII–IX) and (X–XII) images are zircon crystals after annealing at 850°C and 900°C, respectively. Numerals show locations studied with Raman spectroscopy.

translucent and opaque dark brown prismatic crystals, which are shaped by a combination of $\{111\}$ pyramid and $\{110\}$ prism (Fig. 2, I–II). The grains vary in size from 100–300 μm to 1 mm ($K_{\text{el}} = 1.7\text{--}3.0$) and show oscillatory zoning (Fig. 2; IV, VI–VIII). Central parts of the crystals frequently contain metamict domains (Fig. 2, III, V, VII; as well as see below Fig. 4, I, III, IV) enriched in uranium (up to 3–5 wt % UO_2) and thorium (up to 1–2 wt % ThO_2), and inclusions of U- and Th-bearing minerals (Ivanova et al., 2019). The zircon also contains melt and fluid inclusions. The latter are mainly restricted to the metamict zones of the crystals (Fig. 4; I, III, IV).

Zircon from phase II amazonite granite of the Turga massif (sample Tu-832) is represented by opaque and translucent white and brown euhedral crystals shaped by $\{100\}$ prism and $\{111\}$ bipyramid (Fig. 3, I–II). The crystal size varies from 50 to 200 μm ($K_{\text{el}} = 2.0\text{--}3.0$). Grains contain melt and numerous fluid inclusions (Fig. 5). Cathodoluminescence (Fig. 3) and Raman spectroscopic studies indicate its highly metamict state. The average ThO_2 content in zircon from the amazonite granite is 1–2 wt %, while UO_2 , 2–7 wt % (Ivanova et al., 2019).

Using formula from (Nasdala et al., 2001), the self-irradiation α -dose (D_α) for the studied zircons was calculated based on U and Th contents. The D_α value is $6.0 \times 10^{18}\text{--}1.1 \times 10^{19}$ α -decay/g for zircon from the Li-siderophyllite granite and $7.0 \times 10^{18}\text{--}2.3 \times 10^{19}$ α -decay/g for zircon from the amazonite granite. Such high D_α values indicate the intense development of irradiation-induced metamictization. For instance, at $D_\alpha > 6 \times 10^{18}$ α -decay/g, the content of amorphous phase in zircon is more than 80% (Zhang and Salje, 2001). The U-Pb geochronological studies of these zircons without preliminary treatment usually provide no reliable geochronological data. Therefore, experiments were carried out to determine the optimal conditions of high-temperature annealing and preliminary acid treatment of above described zircons, which would allow one to obtain reliable ages of high-uranium metamict zircons.

High-temperature annealing. The most transparent zircons collected for U-Pb geochronological studies from Li-siderophyllite and amazonite granites of the Turga massif were subjected to high-temperature annealing at 850°C and 900°C for 48 h. In compliance with (Mattinson, 2005), the temperature of 850°C was taken as the standard annealing temperature. According to literature data (Widmann et al., 2019), at temperature higher than 915°C the recrystallization of amorphous domains occurs. In order to improve the crystallinity of weakly damaged domains without recrystallization of highly damaged domains and as a result, to preserve more material after acid treatment, the temperature of some annealing experiments was increased up to 900°C (nos. 4, 7, 10 in Table 1). Visible

Fig. 4. Transmitted light images of zircon crystals from Li-siderophyllite granite of phase I of the Turga massif (sample Tu-0467/sb) prior to (I–II) and after annealing at 850°C (III–IV) and 900°C (V–VI). (MI) melt inclusions, (FI) fluid inclusions.

changes of zircon crystals occurred in the insignificant increase in their transparency, change of color (appearance of well expressed red tint), as well as a significant increase of cathodoluminescence (Fig. 2, VI–VIII; Fig. 3, VII–XII), which suggests the partial recovery of their crystallinity. This is confirmed by the Raman spectroscopic studies. The Raman band at $\sim 1008\text{ cm}^{-1}$ (Fig. 6) corresponding to the most structurally sensitive vibrational B_{1g} mode (ν_3) of silicate ion SiO_4 in zircon is considered as indicator (Nasdala et al., 2001, 2004). A shift of mode maximum could be caused by radiation distortion of zircon lattice, formation of solid solutions, and local stresses, while a strong widening of band ν_3 is typical of highly-damaged zircon (Shchapova et al., 2017, 2018).

High-temperature annealing of zircons from Li-siderophyllite and amazonite granites caused a well expressed increase of the Raman shift of ν_3 (SiO_4) band (toward 1008 cm^{-1} typical of “ideal” highly crystalline zircon) and a reduction in the band width (FWHM—full width at half maximum) (Fig. 7). This increase for zircon from Li-siderophyllite granite occurs on average from 991 ± 3 to $999 \pm 2\text{ cm}^{-1}$ (the FWHM varies from 27 ± 16 to $15 \pm 6\text{ cm}^{-1}$). The annealing temperature does not affect these parameters (Fig. 7a). Zircon from amazonite granite after annealing shows an increase of the Raman shift of ν_3 (SiO_4) band on average from 989 ± 3 to $996 \pm 2\text{ cm}^{-1}$ (the FWHM varies from 48 ± 24 to $19 \pm 4\text{ cm}^{-1}$). Thereby, after annealing at 900°C, the band of ν_3

Fig. 5. Transmitted light images of zircon crystals from amazonite granite of phase II of the Turga massif (sample Tu-832) prior to (I–III) and after annealing at 850°C (IV–VI) and 900°C (VII–IX). (MI) melt inclusions, (FI) fluid inclusions. Numerals show locations studied with Raman spectroscopy.

(SiO_4) mode in some cases is shifted closer to the “ideal” value of 1008 cm^{-1} (Fig. 7b), which may indicate that the elevated annealing temperature is more efficient for the recovery of crystallinity of metamict zircon.

Unfortunately, many of the studied zircon crystals lack band of ν_3 (SiO_4) mode in the Raman spectra (Fig. 6) due to the high irradiation damages, as well as fluorescence interference caused by the high contents of U, Th, and REE. In addition, all Raman spectra of

zircon from the Turga granites display bands in the area of $\sim 810\text{ cm}^{-1}$ (Fig. 6), which is not typical of this mineral and can be related to the $(\text{UO}_2)^{2+}$ in the lattice or the presence of torbernite microinclusions (Shchapova et al., 2017, 2018).

The described changes of zircon in response to the high-temperature annealing in general are consistent with data reported in (Nasdala et al., 1995, 1998, 2004; Widmann et al., 2019; Geisler et al., 2001, and others). Annealing at 850°C seems to be optimal for the stud-

Table 1. Results of U-Pb geochronological studies of zircons from granites of the Turga massif

No.	Size fraction (μm) and characteristics of zircon	U/Pb	Pb _c / Pb _t	Isotope ratios					Rho	Age, Ma		
				²⁰⁶ Pb/ ²⁰⁴ Pb	²⁰⁷ Pb/ ²⁰⁶ Pb ^a	²⁰⁸ Pb/ ²⁰⁶ Pb ^a	²⁰⁷ Pb/ ²³⁵ U	²⁰⁶ Pb/ ²³⁸ U		²⁰⁷ Pb/ ²³⁵ U	²⁰⁶ Pb/ ²³⁸ U	²⁰⁷ Pb/ ²⁰⁶ Pb
Li-siderophyllite granite of phase I of the Turga massif (sample Tu-0467/sb)												
1	100–300, 45 cryst., HTA = 850°C, ac.tr. = 2.0/220°C	42.3	0.13	382	0.0493 ± 1	0.0632 ± 1	0.1463 ± 3	0.0215 ± 1	0.62	139 ± 1	137 ± 1	163 ± 4
2	100–300, 300 cryst., HTA = 850°C, ac.tr. = 2.0/220°C	47.2	0.09	622	0.0492 ± 2	0.0737 ± 1	0.1485 ± 6	0.0219 ± 1	0.52	141 ± 1	140 ± 1	156 ± 8
3	100–300, 140 cryst., HTA = 850°C, ac.tr. = 2.0/230°C	45.2	0.03	1589	0.0491 ± 1	0.0671 ± 1	0.1518 ± 2	0.0224 ± 1	0.89	143 ± 1	143 ± 1	153 ± 2
4	100–300, 100 cryst., HTA = 900°C, ac.tr. = 2.0/230°C	32.3	0.32	135	0.0492 ± 2	0.0714 ± 1	0.1494 ± 7	0.0220 ± 1	0.46	140 ± 1	141 ± 1	157 ± 10
5	100–300, 180 cryst., HTA = 850°C, ac.tr. = 4.0/220°C	18.8	0.50	77	0.0505 ± 8	0.0916 ± 1	0.1869 ± 30	0.0268 ± 1	0.43	174 ± 3	171 ± 2	217 ± 35
Amazonite granite of phase II of the Turga massif (sample Tu-832)												
6	<75, 230 cryst., HTA = 850°C, ac.tr. = 4.0/180°C	46.5	0.04	1368	0.0498 ± 1	0.0176 ± 1	0.1553 ± 2	0.0226 ± 1	0.85	146 ± 1	144 ± 1	188 ± 2
7	<75, 40 cryst., HTA = 900°C, ac.tr. = 4.0/180°C	34.2	0.29	180	0.0501 ± 2	0.0155 ± 1	0.1568 ± 8	0.0227 ± 1	0.50	148 ± 1	145 ± 1	200 ± 11
8	<75, 180 cryst., HTA = 850°C, ac.tr. = 4.0/180°C	48.6	0.01	3546	0.0491 ± 1	0.0140 ± 1	0.1509 ± 2	0.0223 ± 1	0.89	143 ± 1	142 ± 1	154 ± 1
9	<75, 190 cryst., HTA = 850°C, ac.tr. = 2.0/220°C	33.0	0.16	330	0.0498 ± 3	0.0268 ± 1	0.1882 ± 13	0.0274 ± 1	0.50	175 ± 1	174 ± 1	186 ± 15
10	<75, 70 cryst., HTA = 900°C, ac.tr. = 6.0/180°C	15.4	0.73	44	0.0505 ± 8	0.0106 ± 1	0.1353 ± 24	0.0194 ± 1	0.36	129 ± 2	124 ± 1	219 ± 40

^a Isotope ratios corrected for blank and common lead; (Rho) correlation coefficient of $^{207}\text{Pb}/^{235}\text{U}$ vs $^{206}\text{Pb}/^{238}\text{U}$ errors; (Pb_c) common Pb; (Pb_t) total Pb; (HTA) high-temperature annealing of zircon; (ac.tr. = 2.0/220°C) acid treatment of zircon with given exposure (hours) and temperature. Errors (2 σ) refer to the last digits of corresponding ratios.

Fig. 6. Raman spectra of zircons from Li-siderophyllite (a–c) and amazonite (d–f) granites of the Turga massif. 55, 56, 17, 88(2)—prior to annealing; 30, 31, 32, 24(2), 38(2)—after annealing at 850°C; 47(2)—after annealing at 900°C. Numbers of spectra correspond to numbers of locations of zircon crystals studied with Raman spectroscopy (Figs. 2, 3, 5).

ied zircons. An increase of annealing temperature up to 900°C likely leads to the more complete recovery of the crystallinity of damaged zones and increase of their stability to leaching, which requires longer dura-

tion and likely higher temperature of acid treatment for the efficient removal of a metamict phase. An increase of annealing temperature up to 900°C is correlated with an increase of common lead fraction at

Fig. 7. Raman shift plotted against the FWHM (full width at half maximum) in cm^{-1} of the $\nu_3(\text{SiO}_4)$ Raman band of zircons from Li-siderophyllite (a) and amazonite (b) granites of the Turga massif based on Raman data prior to (field I) and after (field II) high-temperature annealing. (1) after annealing at 900°C , (2) after annealing at 850°C , (3) without annealing.

decreasing $^{206}\text{Pb}/^{204}\text{Pb}$ ratio in analyzed samples (nos. 4, 7, 10 in Table 1). This tendency can be evidences of the excess recrystallization of zones with losses of radiogenic lead.

Acid treatment. To study the efficiency of removal of a metamict phase of zircons during acid treatment, we conducted a series of experiments at different temperature and exposure duration. In most cases, only small fragments (5–20 μm) of zircon crystals have been preserved after acid treatment (Fig. 8).

Zircon from phase I Li-siderophyllite granite of the Turga massif was subjected to acid treatment at 220°C and 230°C for 2 and 4 hours (nos. 1–5, Table 1). A 2-h treatment presumably led to the removal of self-irradiation damaged zones and microinclusions of U- and Th-bearing minerals. Crystalline residues of zircon after 2-h treatment in general have the low content of common Pb (Table 1), while data points of their isotope composition plot on discordia with an upper intercept at 146 ± 4 Ma (MSWD = 0.067, at a zero lower intercept) (Fig. 9a). An increase of acid treatment duration up to 4 h (no. 5, Table 1) resulted in the practically complete dissolution of zircon crystals, and respectively, a sharp decrease of quantities of the min-

eral available for study and a significant increase in the U/Pb determination error.

Zircon from phase II amazonite granite of the Turga massif is characterized by the higher ability to the dissolution compared to the zircon from Li-siderophyllite granite, which is consistent with its higher self-irradiation α -dose. A preliminary acid treatment at 180°C for 4 hours is considered to be optimal for this zircon. As seen from Fig. 9b, the data points of isotope composition of zircon residues after treatment under these conditions (nos. 6, 7, 8; Table 1) are approximated by discordia with the lower intercept at 141 ± 1 Ma (MSWD = 0.014, lower intercept at 1575 ± 470 Ma).

The less destructive treatment of zircon from the amazonite granite provides preservation of not only finely crystalline residue, but also significant quantities of seemingly undisturbed or weakly fragmented zircons. An increase of temperature up to 220°C (no. 9, Table 1) or an increase of exposure duration up to 6 h (no. 10, Table 1) lead to a significant reduction of the crystalline residue and increase of measurement error of U/Pb ratios. At the same time, it is probable that an increase of annealing temperature up to 900°C may facilitate the partial recovery of damaged zones and increase of their resistivity to acid treatment (nos. 7 and 10, Table 1). Preliminary annealing at 850°C is inferred to be optimal for this zircon.

CONCLUSIONS

Our studies first demonstrated the principal opportunity of the use of high-uranium highly radiation damaged metamict zircon for U-Pb (ID-TIMS) geochronological studies. It is possible to determine the optimal conditions of high-temperature annealing and acid treatment of zircon with high self-irradiation α -dose to provide a practically complete removal of a metamict phase and to obtain sufficient quantities of zircon for U-Pb (ID-TIMS) dating. Such conditions for zircon with $D_\alpha \geq 2 \times 10^{18}$ α -decay/g are high-temperature annealing at 850°C and leaching in a mixture of 35% HF + 15% HNO_3 (5 : 1) at 180°C for 4 h.

Ages of granites of the Turga massif (146 ± 4 and 141 ± 1 Ma) obtained using a modified technique of chemical abrasion within error are consistent with the known age estimates for the rare-metal granites of the Kukulbey Complex: 142.1 ± 0.6 Ma, Rb-Sr method (Kostitsyn et al., 2004), 140.3 ± 2.6 and 140.6 ± 2.9 Ma, U-Pb zircon method (Abushkevich and Syritso, 2007). Thus, our study made it possible to specify available widely varying age data on the Li-F granites of the Turga massif: 133.8 ± 1.2 Ma, Rb-Sr method (Syritso et al., 2021); 143 ± 5 Ma, Rb-Sr method (Shergina, unpublished data); 152.7 ± 3.9 Ma, U-Pb (SIMS) zircon age (Udoratina et al., 2017).

Fig. 8. Images of zircon residues of phase I Li-siderophyllite granite of the Turga massif after 2 h acid treatment at 230°C.

Fig. 9. Concordia diagram for zircons from Li-siderophyllite (a) and amazonite (b) granites of the Turga massif. Point numbers correspond to the numbers in Table 1.

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