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The chemical composition and dating of accessory zircon from granitic pegmatites in the north-eastern part of the Aduisky massif

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This work is made relevant by the necessity to improve chemical dating methods, when applied to high atomic and thorium zircons, for which isotopic methods cannot be used.
The purpose of the work is to study the chemical composition of the accessory zircon (cyrtolite) from granitic pegmatites in the north-eastern part of the Aduisky massif (in the Middle Urals) and determine how best to date it.
Methodology. The study comprised quantitative analysis of the chemical composition of the zircon by using a CAMECA SX 100 X-ray electron probe micro-analyser (with an electron beam diameter from 1 µm, BSE, SE, Cat, and determination of elements from beryllium to uranium). To measure the intensity of elements, we have selected the following analytical lines: Y Lα, Si Kα, Zr Lα, Hf Mα (analysing crystal TAP), U Mβ, Pb Mα, Ca Kα, Th Mα (analysing crystal PET), Yb Lα, Er Lα, Lu Lα (analysing crystal LiF). Calculation of the age of the zircon was carried out ася/cording to well-known, existing methods in addition to those developed by the authors.
Results. According to the microprobe analysis, the impurity content of ThO_2, UO_2 and PbO in the zircon varied significantly, within the ranges 0.13 to 2.69, 1.59 to 15.42 and 0.05 to 0.57 wt.%, respectively. The dating calculation was carried out for each mineral (in which the analysis took place). Their age was found to be between 280 and 219 Ma. At the same time, the weighted mean was 254 ± 6 Ma (with the Mean Square of Weighted Deviates being 0.17) and the isochron showed 255 ± 7 Ma. The values of the ages found for the zircon from the pegmatites "Mys-2" agree with the isotopic data. The period of formation of the Aduisky granite massif has been estimated to be between 291 ± 8.0 Ma and 256 ± 0.6 Ma (according to zircon and monazite dating, respectively) or within the range 255 to 241 Ma (according to mica dating).
Conclusion. We have studied the accessory zircon (cyrtolite) from granite pegmatites from the "Mys-2" vein, in the north-eastern part of the Aduisky massif. We have obtained the chemical composition and calculated the age to be 255 ± 7 Ma. Dating calculations show that veined pegmatites and host granites were formed almost simultaneously (at least, in this part of the Aduisky massif). This situation justifies microprobe dating of the U-Th zircon content because the minerals are usually in a metamict state and not suitable for accurate age determination.

Keywords: zircon, chemical dating, granite pegmatites, Aduisky granite massif, Middle Ural.

Introduction

Chemical dating of minerals is widely carried out [1, 2] and is based on the precise determination of the contents of radioactive elements (Th, U) and (not) radiogenic (total) Pb by X-ray microprobe analysis. Through the use of modern microprobe analysers and the thorough development of analytical procedures, it has become possible to quickly solve problems in the direct geochronological dating of accessory minerals in thin sections of rock. The X-ray microprobe analysis method can be used for chemical dating when the content of Th, U, Pb in these minerals is above 0.03 wt.% therefore, most of the work is devoted to monazites, although some relates to dating of uraninite and other radioactive minerals [2, 3]. There are only a few studies concerning the application of this method to zircon [2, 4, 5]. Due to the low contents of thorium, uranium and lead (Pb is often $n \times 0.001$ wt.%), zircon dating is performed using local isotope mass spectrometry with laser (LA-ICP-MS) or ion (SIMS) sampling. In practice, zircons with abnormally high concentrations of Th, U, Pb are found, particularly in alkaline and granitic pegmatites. In this case, isotope dating using a device with an ion probe is not applicable for technical reasonsand it is assumed that a high adulteration of radioactive and radiogenic elements makes the result of dating unreliable ([6] and others). It is not always possible to use mass spectrometry with laser sampling (which gives the average value) due to the large diameter and depth of the crater ($n \times 10$ µm). In our work, the microprobe analysis method was used for chemical dating of the zircon with abnormally high concentrations of U, Th, Pb, Hf, Y (i.e. we have determined the age of the cyrtolite).

Geology of the study area

In recent years, a number of new pegmatite veins have been discovered in the Aduisky granite massif [7]. A large number of them are located 6 to 7 km north of the village of Ozerny, south of Rezh. Pegmatites are located in the hills on the right bank of the river Rezh, which is situated on the north-eastern edge of the massif (1.5 km) [8]. There is a forest corridor at this location, associated with the power lines that cross the area. This site is rich in ceramic pegmatite veins, the largest of which were mined by tributors for feldspar for the ceramic industry at the beginning of the last century (1925–1927). Lump feldspar was mined from the upper, fractured parts of the veins to a depth of 2 to 3 m and in workings between 4 and 30 m long. The feldspar was transported by carts to the river and then further on to the station at Rezh. In total, about 1000 tons of feldspar were mined [9].

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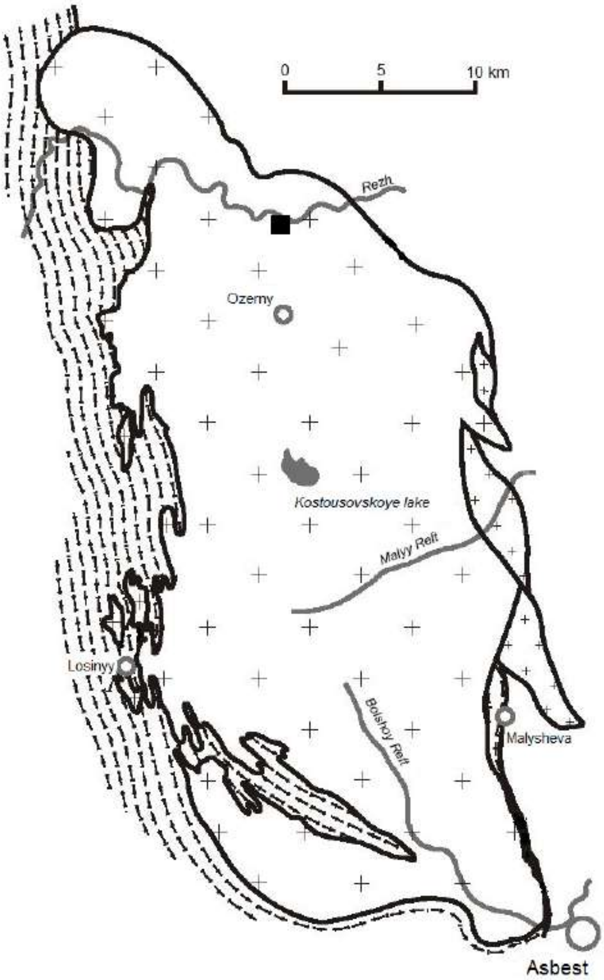


Figure 1. The lineaments of the Aduisky granite massif with marked settlements and water bodies (according to [11] with our simplifications). The filled square shows the location of the granite pegmatite "Mys-2".

Рисунок 1. Контуры Адуйского гранитного массива с вынесенными населенными пунктами и водоемами (дано по [11] с нашими упрощениями). Залитым квадратом показано расположение гранитного пегматита «Мыс-2».

One of the veins (known as "Mys-2" and located at GPS-fixing: 57°20'36,9'' N, 61°12'38,3'' E) was opened with a small digging pit of 10.0 × 1.0 × 1.5 m (Fig. 1). The pegmatite body lies in the fine-grained, slightly sheared, biotite granites and its strike is about 20°. The vein has a visual zonality because it contains graphic pegmatite with muscovite in its casing and a quartz core at its centre. The vein contains the following accessory minerals: garnet, apatite, brockite, columbite, zircon, ilmenite, magnetite, polycrase, titanite, allanite and epidote [8, 10].

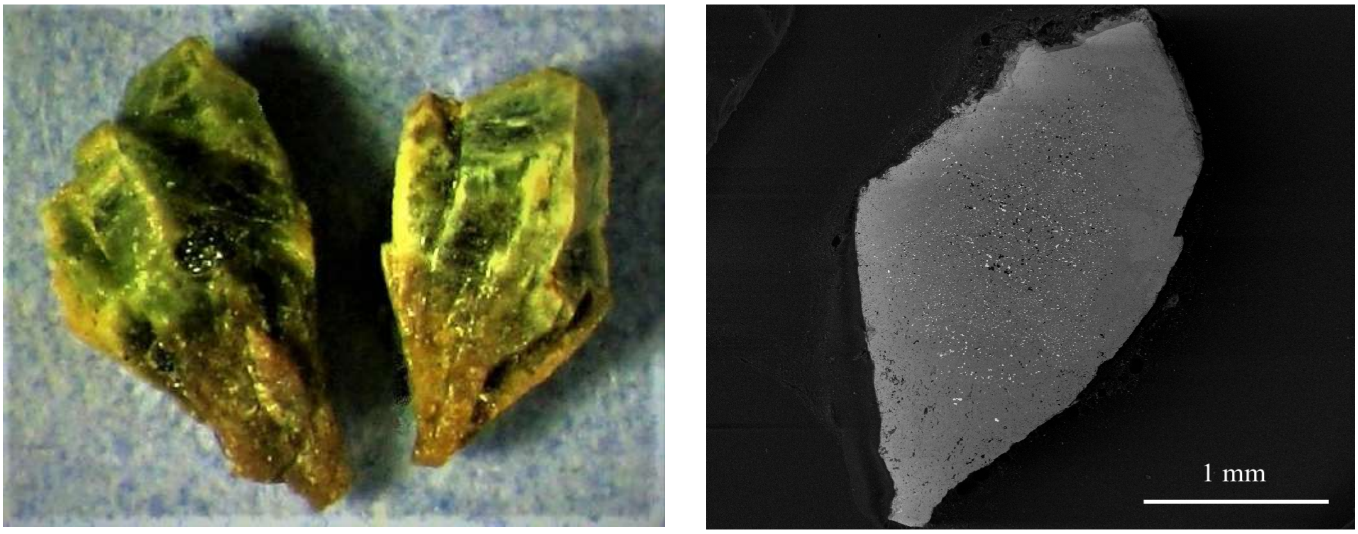
For the dating studies, relatively large but short, prismatic zircon (cyrtolite) crystals (up to 2.5 mm long) were selected from the block zone of the "Mys-2" vein. The mineral is characterised by a zoned colouration: light brown in the centre and dark green around the periphery of the crystals (Fig. 2, a). The green colour seems to be associated with the smallest inclusions of uraninite and thorite, which are found all over the zircon matrix. While preparing the specimens during the first grinding, the top part of the crystal was revealed (Fig. 2, b); the second grinding then opened up the deeper parts of the crystal. In addition, both polished surfaces were studied with respect to chemical dating to provide statistical data.

Study Methods

Quantitative analysis of the chemical composition of the zircon was carried out using a CAMECA SX 100 X-ray electron probe micro-analyser (electron beam diameter being from 1 μm, BSE, SE, Cat, determination of elements from beryllium to uranium). The optical field of view was 0.25 to 1.75 mm from the sample surface. The BSE image of the crystal shows weak, spotted heterogeneity due to different heavy element contents. Small inclusions of uranium and thorium phases (not more than 5 to 10 microns in size) are fixed in the zircon matrix. To measure the intensity of elements, we selected the following analytical lines: Y Lα, Si Kα, Zr Lα, Hf Mα (analysing crystal TAP), U Mβ, Pb Mα, Ca Kα, Th Mα (analysing crystal PET), and Yb Lα, Er Lα, Lu Lα (analysing crystal LiF). The calculation of the chemical age was carried out according to well-known methods [1, 2] in addition to those developed by the authors [3, 4].

Results and discussion

According to the quantitative microprobe analysis, the impurity content of ThO_2, UO_2, PbO in the zircon significantly varies within the ranges 0.13 to 2.69, 1.59 to 15.42 and 0.05 to 0.57 wt.% respectively (see Table). For each point of the crystal in which analysis was carried out, the age was calculated by the Montel method [1]; the range was found to be from 219 to 280 Ma. The weighted average is 254 ± 6 Ma and the Mean Square of Weighted Deviates = 0.17 (Fig. 3).



a b

Figure 2. Zircon crystals from granite pegmatite «Mys-2»; a – appearance, length of crystals up to 2.5 mm, b – crystal cut, BSE-image, CAMECA SX 100.
Рисунок 2. Кристаллы циркона из гранитного пегматита «Мыс-2»; а – внешний вид, длина кристаллов до 2,5 мм, б – срез кристалла в шашке, BSE-изображение, CAMECA SX 100.

Due to the wide range of uranium and lead oxides, we were able to form an isochron from a set of analytical points using the CHIME method [12] in the coordinates UO_2 * – PbO. Using its slope angle (m = 0.03456), we calculated the U * / Pb-age to be 255 ± 7 Ma (see Fig. 4). UO_2 * = (UO_2 + ThO_2^{eq}), where ThO_2^{eq} is the thorium content, converted to the equivalent uranium content and it is capable of producing the same amount of Pb during the lifetime of the system if U-Pb and Th-Pb-age values are equal. The resulting isochron passes through the origin of the coordinates; it indicates the absence of non-radiogenic (initial) lead in the matrix of the studied zircon, as well as the absence of any addition or subtraction of radioactive components in the process of crystal evolution. The analytical points are distributed throughout the isochron, which suggests that the age estimation is probably correct.

The chemical composition (in wt.%) of zircon from granite pegmatites in "Mys-2".
Химический состав (в мас. %) циркона из гранитных пегматитов Мыс-2.

No	1	2	3	4	5	6	7	8	9	10
ThO_2	0.03	0.17	0.13	0.73	0.71	0.82	0.92	1.05	2.65	0.63
UO_2	0.77	1.03	2.04	5.77	5.95	6.34	6.61	7.06	12.17	14.21
ZrO_2	57.19	59.19	57.73	54.55	54.06	53.95	53.14	52.38	45.87	44.32
HfO_2	5.35	6.36	6.16	5.88	5.76	6.30	6.21	6.18	4.67	5.40
SiO_2	32.53	31.76	31.38	30.81	30.63	30.86	30.67	30.44	28.68	32.38
Gd_2O_3	0.01	0.05	0.00	0.07	0.20	0.11	0.13	0.04	0.21	0.17
Dy_2O_3	0.13	0.24	0.08	0.09	0.14	0.20	0.04	0.01	0.22	0.18
Y_2O_3	0.44	0.86	0.70	0.98	1.13	0.97	1.00	1.04	1.63	1.54
PbO	0.00	0.00	0.00	0.13	0.11	0.15	0.14	0.15	0.38	0.53
FeO	0.15	0.09	0.08	0.05	0.13	0.03	0.02	0.01	0.06	0.13
In total	96.60	99.75	98.30	99.06	98.81	99.73	98.88	98.37	96.52	99.47

Note: the sample from 41 analyses with growth of uranium from 0.77 to 14.21 wt. %.

It is interesting to note that small inclusions of uraninite and uranium-containing thorite in the zircon matrix showed similar dating, within 253 to 251 Ma. This indirectly confirms the accuracy and correctness of the chemical dating of the cyrtolite crystal carried out by us.

The age values obtained for the zircon from the "Mys-2" pegmatites equate with isotopic data. Thus, the formation time of the Aduysky granite massif covers a wide interval and is estimated to have been from 291 ± 8 Ma (according to zircon dating [13]) to 256 ± 0.6 Ma (according to monazite dating [14]). A dating range of between 255 and 241 Ma was found according to dating of the micas [11]. At the same time, the accessory monazite was precisely dated in mesocratic granites within the vicinity of the village of Ozerny (mica is found in the intersecting pegmatites [11]); this fact indicates the same time interval of the vein formations and the granites enclosing them in the area of the Aduysky massif. Recently, one of the authors of this article obtained a similar and reasonably reliable Th-U-Pb dating for accessory monazites from pegmatite veins in the same vicinity (within Ozerny village); the date derived was 254 ± 15 Ma [15, 16].

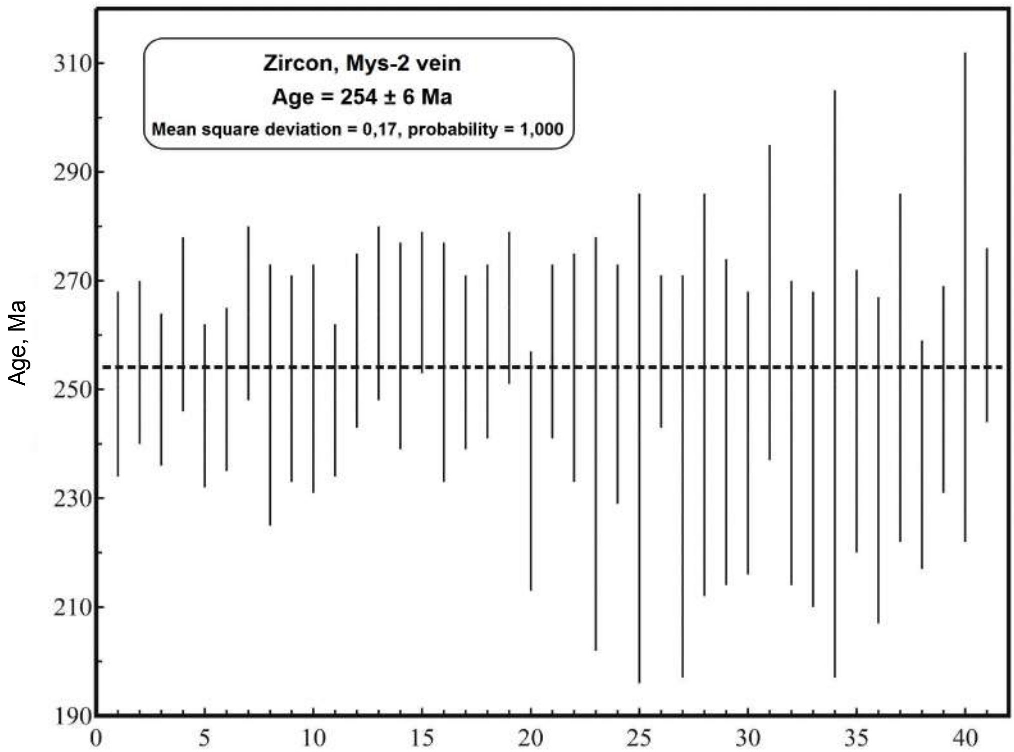


Figure 3. The weighted average Th-U-Pb-age of the zircon crystal according to microprobe analysis (41 definitions were made in total).
Рисунок 3. Средневзвешенный Th-U-Pb-возраст кристалла циркона по данным микрозондовых анализов (всего сделано 41 определение).

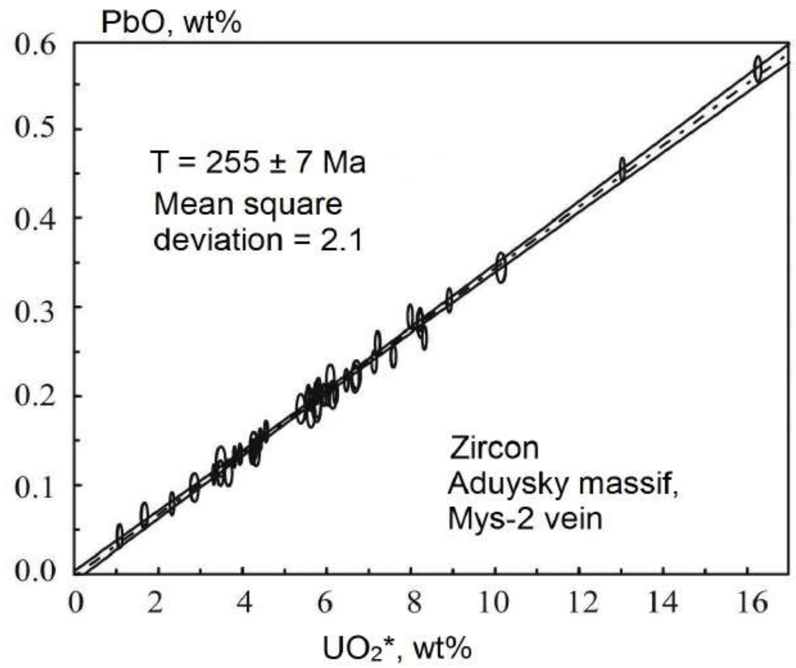


Figure 4. UO_2* – PbO data for the Aduiskiy zircon. Ellipses correspond to 1σ uncertainty values; dot-and-dash lines correspond to regression lines with two symmetric hyperbolas that fix errors.
Рисунок 4. UO_2*– PbO-данные для адуйского циркона. Эллипсы соответствуют значениям погрешности 1σ, штрих-пунктир – линии регрессии с двумя симметричными гиперболами, фиксирующими погрешности.

Conclusion

This work has studied the accessory zircon (cyrtolite) from granite pegmatites in the "Mys-2" vein, located in the north-eastern part of the Aduisky massif. We have obtained the chemical composition and calculated the age to be 255 ± 7 Ma. The calculated dating shows that veined pegmatites and their host granites have been formed almost simultaneously (at least, in this area of the Aduisky massif). In general, it can be added that the chemical age of zircons gives accurate results if the mineral contains significant concentrations of uranium, thorium and lead. This case is only one of a few successful examples of microprobe dating of zircon; it is usually seen in a metamict state and is unsuitable for correct age determination.

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Акцессорный циркон из гранитных пегматитов северо-восточной части Адуйского массива (химический состав и датирование)

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Актуальность работы обусловлена необходимостью совершенствования метода химического датирования в применении к высокоурановым и ториевым цирконам, которые невозможно датировать изотопными методами исследования.
Цель работы: исследование химического состава акцессорного циркона (циртолита) из гранитных пегматитов северо-восточной части Адуйского массива (Средний Урал) и определение его возраста.
Методология исследования: количественный анализ химического состава циркона выполнен на рентгеноспектральном электронно-зондовом микроанализаторе CAMECA SX 100 (диаметр пучка электронов от 1 мкм, режимы BSE, SE, Cat, определение элементов от бериллия до урана). Для измерения интенсивности элементов подобраны следующие аналитические линии: Y *Lα*, Si *Kα*, Zr *Lα*, Hf *Mα* (кристалл-анализатор TAP), U *Mβ*, Pb *Mα*, Ca *Kα*, Th *Mα* (кристалл-анализатор PET), Yb *Lα*, Er *Lα*, Lu *Lα* (кристалл-анализатор LiF). Расчет возраста проводился по известным методикам зарубежных авторов в дополнении с собственными наработками.
Результаты. По данным микрозондового анализа содержание примесей ThO_2, UO_2, PbO в цирконе сильно варьируют в пределах 0,13-2,69 1,59-15,42 и 0,05-0,57 мас. %, соответственно. Для каждой точки кристалла, в которой проводился анализ, выполнялся расчет возраста, разброс которого находится в интервале от 280 до 219 млн лет, при этом средневзвешенное значение возраста составляет 254 ± 6 млн лет (СКВО = 0,17), а изохрона показывает 255 ± 7 млн лет. Полученные значения возраста для циркона из пегматитов «Мыс-2» вполне хорошо сопоставимы и с изотопными данными. Время образования Адуйского гранитного массива оценивается в широком временном интервале от 291 ± 8 млн лет (по циркону) до 256 ± 0,6 млн лет (по монациту) и 255-241 млн лет (по слюдам).
Выводы. Нами изучен акцессорный циркон (циртолит) из гранитных пегматитов северо-восточной части Адуйского массива из жилы «Мыс-2». Для него был получен химический состав и рассчитан возраст 255 ± 7 млн лет. Рассчитанная датировка показывает, что жильные пегматиты и вмещающие их граниты формировались практически одновременно, по крайне мере, на данном участке Адуйского массива. Данный случай, это один из немногих удачных примеров микрозондового датирования U-Th-содержащего циркона, обычно он находится в метамиктном состоянии и непригоден для корректного определения возраста.

Ключевые слова: циркон, химическое датирование, гранитные пегматиты, Адуйский гранитный массив, Средний Урал.

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