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AGE AND COMPOSITION OF ZIRCONS FROM THE DEVONIAN PETRIVSKE KIMBERLITE PIPE OF THE AZOV DOMAIN, THE UKRAINIAN SHIELD

Results of a study of U-Pb and Hf isotope systematics and trace element concentrations in five zircon crystals separated from the Devonian Petrivske kimberlite are reported in the paper. Four zircons have yielded Paleoproterozoic and Archean ages, while one zircon grain gave a Devonian age of 383.6 ± 4.4 Ma (weighted mean $^{206}Pb/^{238}U$ age). The Precambrian zircons have been derived from terrigenous rocks of the Mykolaivka Suite that is cut by kimberlite, or directly from the Precambrian rock complexes that constitute continental crust in the East Azov. The Devonian zircon crystal has the U-Pb age that corresponds to the age of kimberlite emplacement. It is 14 m.y. younger than zircon megacrysts found in the Novolaspa kimberlite pipe in the same area. In addition, Petrivske zircon is richer in trace elements than its counterparts from the Novolaspa pipe. Petrivske and Novolaspa zircons crystallized from two different proto-kimberlite melts, whereas the process of kimberlite formation was very complex and possibly included several episodes of formation of proto-kimberlite melts, separated by extended (over 10 M.y.) periods of time.

Keywords: *kimberlite, zircon, Ukrainian Shield, U-Pb age, Hf isotopes, trace elements.*

Introduction. Kimberlite magmas, being undersaturated in silica, do not crystallize zircons. However, so-called zircon megacrysts have been found in some of the kimberlite bodies, although their origin and connection to their host kimberlites remains highly controversial [5, 8—10, 17, 19]. Zircon megaacrysts usually have alteration rims that indicate a lack of chemical equilibrium between zircons and their host kimberlite melts [9, 17, 21].

Zircons were found in Devonian kimberlite pipes and dykes that occur in the Eastern Azov area of the Ukrainian Shield, in a junction zone with the Paleozoic Donets Basin (Fig. 1). A detailed description of zircon megacrysts from the Novolaspa kimberlite pipe and dyke, and a discussion of their age and origin can be found in [17, 21]. According to the available data, zircon megacrysts from the Novolaspa pipe and dyke crystallized at 397.0 ± ± 1.0 Ma [17]. However, this age is 14 m.y. older than the age of kimberlite emplacement as defined by phlogopite and whole-rock Rb-Sr isochron (384.7 ± 3.9 and 383.3 ± 3.8 Ma for kimberlites of the Pivdenna and Novolaspa pipes, respectively; [23]). In this brief communication, we report results of geochronological and geochemical studies of zircons from kimberlites of the Petrivske pipe and compare them with the results obtained for zircon megacrysts from the Novolaspa kimberlite.

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Geological setting. Four kimberlite pipes (the Petrivske, Nadiya, Pivdenna, and Novolaspa ones) have been identified in the Eastern Azov area (Fig. 1). Their detailed description can be found in [21—23]. The stratigraphic age of the Azov kimberlites was defined as Frasnian (379-371 Ma). The Petrivske pipe occurs as a mushroom-shaped body with a surface exposure of 200 × 400 m. It cuts through sedimentary rocks of the Middle Devonian Mykolaivka Suite and is overlain by alkaline-mafic volcanic rocks of the Antonivka Suite (Middle-Late Devonian).

The Azov Domain of the Ukrainian Shield is composed predominantly of Archean rocks [1, 2, 6], whereas Paleoproterozoic rock complexes prevail in the eastern part of the Domain [15], which was intruded by the Devonian kimberlites. Archean (3130 to 2830 Ma) and Paleoproterozoic (2075 ± ± 9 Ma) detrital zircons were revealed in the Mykolaivka Suite sandstones, indicating a local source for these sediments [16].

Analytical methods. Zircons were analyzed by LA-ICP-MS: for U-Pb isotopes at the University of Tasmania, and for Hf isotopes and trace element concentrations at Curtin University, Western Australia. Zircon U-Pb geochronology was performed using an Agilent 7900 quadrupole ICP-MS coupled to a Coherent COMPex Pro 110 utilizing an ArF excimer laser, operating at a wavelength of 193 nm. Each analysis began with a 30-second analysis of the blank gas measurement followed by 30 seconds of acquisition time. Zircons were sampled on 30 μm spots using the laser at 5 Hz and approximately 2 J/cm^2 laser fluence. The detailed analytical method for U-Pb dating of zircon was outlined in [20].

Trace element and Hf isotope data were acquired at the John de Laeter Centre, Curtin University, using a LASS NPII + 7700 + SE RESOlution excimer laser operating at the following conditions: spot size 38 micron, laser frequency 10 Hz, and energy 3 j/cm^2. The Hf isotope composition was measured on a Nu Plasma II mass-spectrometer. Zircon crystals from the Mud Tank carbonatite were analyzed together with the samples to monitor the accuracy of the results. Zircons 91500, Plešovice, GJ-1, and R33 were also run as secondary reference standards. All reference material yielded ^{176}Hf/^{177}Hf ratios within an uncertainty of their respective reported values. Trace element data were collected simultaneously with Hf isotope data. Zircon standard GJ-1 was utilized as the primary reference material for concentration determination and to correct for instrument drift, using ^{91}Zr as the internal reference isotope and assuming 43.14% Zr in the unknowns. NIST SRM 612 was run as a secondary standard and yielded recommended values within 3% for all elements.

Results. Zircons found in kimberlites of the Petrivske pipe are quite big, reaching 200 μm in size. Most of the crystals, except crystal 5 in Figure 2, represent fragments of even bigger grains. However, all of them, except grain 3, do not fit the definition of zircon megacryst that usually occurs as rounded to semi-rounded crystals often covered by alteration rims and have either fine oscillatory banding or structureless interiors on CL images [5, 17, 19, 21].

Grain 1 has oscillatory zoning typical for igneous zircons; zircon 2 reveals a very bright in CL color which is not typical for kimberlite megacrysts; zircon 4 is represented by prismatic grain with well-developed zoning, and zircon 5 has a rounded appearance typical for detrital grains (Fig. 2). Only grain 3 has a dark appearance on the CL image and poorly developed oscillatory zoning. There are no alteration rims on any of the studied crystals.

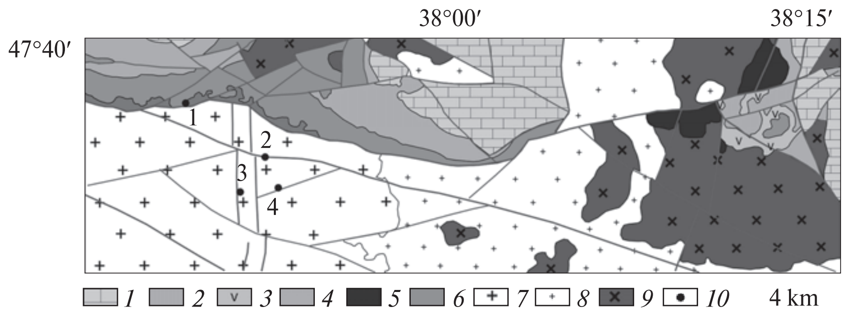

Fig. 1. Simplified geological map of the junction zone of the Donets folded basin and Azov Domain of the Ukrainian Shield. *1—6* — Paleozoic (*1* — Early Carboniferous, predominantly carbonate rocks; *2* — Late Devonian, predominantly terrigenous rocks with tuffite admixture; *3* — Late Devonian syenite and nepheline syenite; *4* — Middle-late Devonian, mafic-ultramafic volcanic rocks; *5* — Middle-late Devonian pyroxenites and ultramafic-alkaline rocks; *6* — Middle Devonian, mafic-ultramafic volcanic rocks with interlayers of terrigenous and carbonate rock); *7, 8* — Paleoproterozoic (*7* — Khlibodarivka complex (ca. 2040 Ma): granite, granosyenite, syenite, quartz monzonite; *8* — Anadol complex (ca. 2080 Ma): granite and migmatite); *9* — Archean: Maksymivka granodiorite association; *10* — kimberlite pipes (1 — Petrivske, 2 — Nadiya, 3 — Novolaspa, 4 — Pivdenna)

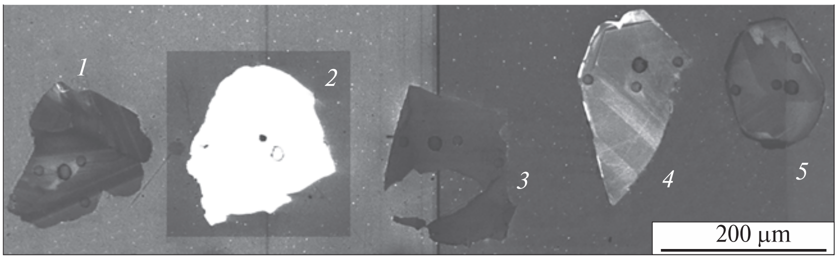

Fig. 2. CL images of zircons from kimberlites of the Petrivske pipe (*1—5* — explanation in the text)

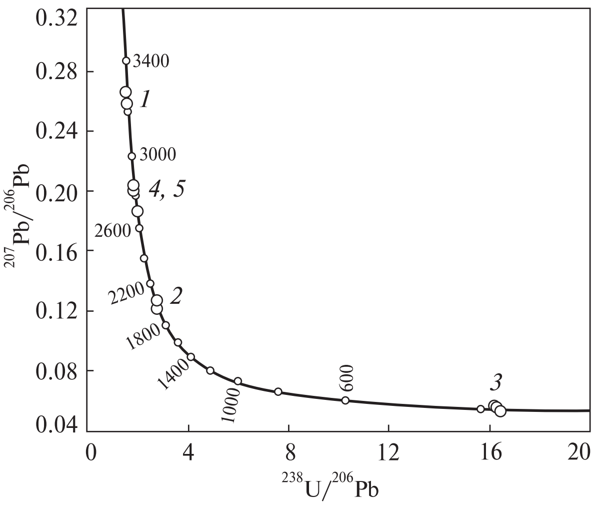

Fig. 3. Results of U-Pb dating of zircons from kimberlite of the Petrivske pipe. Zircon grain numbers are indicated

Four zircons separated from kimberlite of the Petrivske pipe yielded Paleoproterozoic (crystal 2) and Archean (crystals 1, 4 and 5) ages, while one zircon (crystal 3) gave a Devonian age (Fig. 3, Table 1). The oldest grain yielded a weighted mean $^{207}Pb/^{206}Pb$ age of 3249 ± 37 Ma, two grains yielded a weighted mean $^{207}Pb/^{206}Pb$ age of 2841 ± 9 Ma and one Paleoproterozoic grain crystallised at 2030 ± 37 Ma. The single Devonian crystal was dated at 383.6 ± 4.4 Ma (weighted mean $^{206}Pb/^{238}U$ age).

The initial Hf isotope compositions were calculated accordingly to their crystallization ages. A single Hf isotope analysis was carried out on the young (383.6 ± 4.4 Ma) zircon crystal and the calculated initial $^{176}Hf/^{177}Hf$ ratio is 0.28276, and εHf = 7.5. In terms of Hf isotope composition, it fits zircon megacrysts found in the Novolaspa kimberlite [17]. Archean zircons yielded nearly chondritic εHf values from 0 to 1, whereas Paleoproterozoic grains have εHf = –5.0 and –7.5 (Fig. 4, Table 1).

Archean and Proterozoic zircons found in the Petrivske kimberlite pipe differ markedly from the megacrystic zircons from the Novolaspa kimberlite [17] in terms of their chemical composition. They

Table 1. **Results of U-Pb dating of zircons from kimberlites of the Petrivske pipe**

Grain #	Position	Isotope ratios				Isotope ages, Ma ± 1 σ						Concentrations, ppm				
		$^{238}U/^{206}Pb$	± 1 σ	$^{207}Pb/^{206}Pb$	± 1 σ	$^{206}Pb/^{238}U$		$^{208}Pb/^{232}Th$		$^{207}Pb/^{206}Pb$		Pb	Th	U	Ti	Fe
1	*c*	1.5340	0.01	0.2581	0.0017	3236	27	3244	49	3235	11	29.7	64.0	47.7	3.3	0.0
1	*o*	1.5459	0.01	0.2573	0.0018	3216	30	3237	46	3230	11	27.1	55.6	44.0	2.4	5.2
1	*o*	1.4991	0.01	0.2655	0.0015	3295	27	3320	47	3280	9	40.6	85.1	63.8	5.0	2.6
2	*c*	2.7717	0.03	0.1273	0.0022	1986	24	1988	40	2061	30	7.7	19.9	23.0	5.2	253.3
2	*o*	2.7690	0.04	0.1259	0.0024	1988	25	2003	49	2042	34	7.0	17.2	21.2	6.9	61.7
2	*o*	2.7153	0.03	0.1221	0.0022	2021	25	2038	42	1987	32	7.4	21.6	21.0	4.4	19.6
3	*c*	16.4338	0.19	0.0540	0.0012	381	4	386	6	370	50	11.3	478.9	192.9	14.2	2.3
3	*o*	16.3411	0.16	0.0556	0.0011	383	4	384	5	434	43	13.8	650.7	237.1	15.2	11.8
3	*o*	16.1988	0.15	0.0565	0.0011	386	4	385	5	470	44	11.0	466.9	187.7	13.5	11.2
4	*c*	1.8453	0.02	0.2003	0.0014	2791	23	2784	43	2829	11	29.5	46.0	57.1	2.8	2.6
4	*o*	1.7873	0.01	0.2010	0.0014	2865	24	2867	48	2834	11	29.8	42.8	55.8	3.3	5.0
4	*o*	1.9629	0.02	0.1859	0.0015	2654	22	2631	40	2707	14	31.5	58.6	64.7	3.7	7.6
5	*c*	1.7954	0.01	0.2037	0.0011	2854	22	2863	39	2856	9	75.9	115.3	142.9	6.0	0.0
5	*o*	1.8013	0.02	0.2020	0.0011	2847	25	2836	43	2843	9	56.7	54.2	107.0	6.7	2.5
5	*o*	1.7968	0.01	0.2015	0.0011	2852	22	2852	40	2838	9	80.7	102.9	152.0	6.3	1.5

N o t e. *c* — central part of the crystal; *o* — outer part of the crystal.

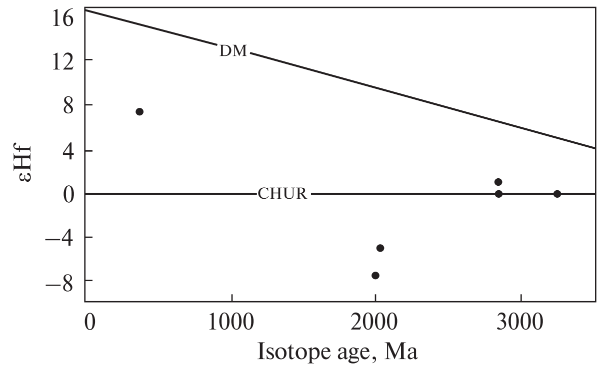

Fig. 4. Hf isotope composition in zircons from kimberlites of the Petrivske pipe

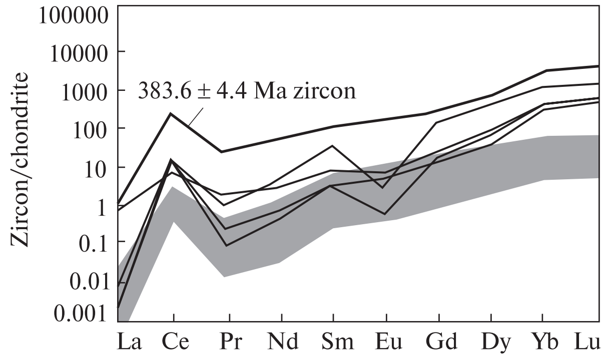

Fig. 5. Chondrite-normalized REE patterns in zircons from the Petrivske pipe. The shaded area indicates the composition of zircon megacrysts from the Novolaspa kimberlite [17]

Table 2. **Hf isotope composition of zircons from kimberlites of the Petrivske pipe**

Grain #	Age, Ma	$^{176}Lu/^{177}Hf$	$^{176}Yb/^{177}Hf$	$^{176}Hf/^{177}Hf \pm \sigma$	$^{176}Hf/^{177}Hf_T$	eHf_T	$\pm 2\sigma$
1	3249	0.000299	0.010910	0.280704 ± 18	0.280685	0.1	1.3
2	2030	0.000432	0.016550	0.281362 ± 19	0.281345	−5.0	1.3
3	384	0.003882	0.154320	0.282783 ± 28	0.282755	7.5	2.0
4	2841	0.000413	0.013552	0.281005 ± 25	0.280982	1.0	1.7
5	2841	0.000770	0.031770	0.280994 ± 18	0.280952	−0.1	1.3

Table 3. **Trace element composition of zircons from kimberlites of the Petrivske pipe, ppm**

Grain #	Ti	Y	Nb	La	Ce	Pr	Nd	Sm	Eu	Gd	Dy	Yb	Lu	Hf	Ta	Th	U
1	3.2	332	1.3	0.001	12.38	0.01	0.29	0.78	0.05	5.62	27.36	104.43	21.22	10504	0.39	34.89	28.76
2	4.7	396	3.0	0.270	6.72	0.24	2.08	1.75	0.58	7.54	33.53	106.60	21.23	7164	0.63	28.18	27.23
3	15.7	2890	11.9	0.439	224.91	3.31	32.43	25.56	13.64	83.36	251.89	707.81	135.43	5138	1.17	488.48	195.36
4	4.0	195	1.4	0.001	12.34	0.03	0.50	0.83	0.41	4.00	15.08	76.92	17.90	6356	0.49	46.84	63.46
5	5.9	1345	1.4	0.003	14.50	0.14	3.05	7.97	0.26	43.60	142.51	292.74	53.33	10259	0.52	102.16	130.11

have higher concentrations of P (28-352 ppm), Y (195-1345 ppm), REE (Ce = 6.7 to 14.5 ppm, Yb = = 76.9 to 292.7 ppm), Th (28—102 ppm) and U (27-130 ppm), whereas concentrations of Ti (2.8-5.2 ppm), Nb (1.3-3.0 ppm), Hf (0.64-1.05%) and Ta (0.4—0.6 ppm) are generally lower (Tables 2, 3). In contrast to the Devonian megacrysts, these zircons have more fractionated REE patterns (Fig. 5), and strong negative Eu/Eu* anomaly (0.04 to 0.7), but with even stronger positive Ce/Ce* anomalies (6 to 816).

As reported above, one zircon grain from the Petrivske pipe yielded a Devonian age of 383.6 ± ± 4.4 Ma, with an Hf isotope composition identical to zircons from the Novolaspa kimberlites. However, the Petrivske pipe zircon is significantly enriched in P, Nb, Th, U, Y and REE, but depleted in Ta and Hf.

Discussion. It is tempting to assume that studied Archean and Paleoproterozoic zircons were captured by emplacing kimberlites from the lower crust or lithospheric mantle. The ability of zircons to retain their old U-Pb isotope age at high mantle temperatures have been repeatedly discussed in the literature. For instance, [7, 13] suggested that the old ages will not survive the high temperatures, and the ages of mantle zircons found in kimberlites will correspond to the age of emplacement, i.e., to the cooling of zircons from the mantle temperature below the closing temperature in zircon. However, zircon ages much older than the kimberlite emplacement age have been reported in several studies [4, 10, 18] and explained by the low U and Th contents in kimberlitic zircon and a very slow diffusion rate for Pb [4, 9].

Zircons that yielded old ages in our study are different from their pristine mantle counterparts by their appearance and chemical composition. In fact, in terms of the chemistry, they fit zircons from syenites and felsic rocks [3]. Detrital zircons having similar ages were found in sandstones of the Mykolaivka Suite [16], which are cut by the Petrivske kimberlite pipe. Hence, these old zircons could be derived from the Devonian sediments (as indicated, for instance, by the rounded shape of the crystal 5), or directly from the Paleoproterozoic and Archean rocks that are widely distributed in the area [1, 2, 15].

The single Devonian zircon found in the Petrivske kimberlite generally suits the definition of kimberlite megacryst. However, in contrast to zircon megacrysts found in the Novolaspa kimberlite pipe and dyke [17], zircon from the Petrivske pipe is much richer in most of the trace elements. Moreover, it is ca. 14 M.y. younger than zircons from the Novolaspa kimberlite, and its age fits the age of kimberlite emplacement [23].

Zircons do not crystallize directly from the kimberlitic melt as it is too poor in SiO_2. Moreover, zircon megacryst captured by kimberlite melt, usually reacts with it, indicating the lack of chemical equilibrium [9, 12, 17, 21]. According to the proto-kimberlite melt model [9, 11, 14], the emplacement of kimberlite was preceded by the primitive melt or metasomatizing fluid. A so-called "low-Cr mineral suite", including zircon, crystallized from this primitive melt or fluid. Later, they were captured and transported by kimberlite magmas.

This model was applied to the zircon megacrysts found in the Novolaspa kimberlite [17]. These authors established that zircon megacrysts are ca. 14 M.y. older than the age of the host kimberlite, demonstrating the lack of direct affinity between them. However, in case of the Petrivske kimberlite, the single Devonian zircon is coeval to the kimberlite emplacement. The significant difference in the trace element composition suggests that Petrivske and Novolaspa zircon megacrysts crystallized from two different proto-kimberlite melts or fluids. Our data indicate that the process of kimberlite formation is very complex and may include several episodes of proto-kimberlite emplacement, separated by extended (over 10 M.y.) periods of time.

Conclusions. Zircon crystals separated from kimberlite of the Devonian Petrivske pipe (East Azov region of the Ukrainian Shield) have revealed wide variations in age, Hf isotope systematics and trace element composition. All of them, except one, do not fit the definition of zircon megacryst and have Paleoproterozoic to Archean ages. These zircons have possibly been derived from terrigenous rocks of the Mykolaivka Suite, which is cut by kimberlite, or directly from the Precambrian rock complexes that constitute continental crust in the East Azov.

The single Devonian zircon crystal has the U-Pb age that corresponds to the age of kimberlite emplacement, which is 14 m.y. younger than the age of zircon megacrysts found in the Novolaspa kimberlite pipe in the same area. In addition, Petrivske Devonian zircon is much richer in trace elements than zircon megacrysts from the Novolaspa pipe. Accordingly, we assume that Petrivske and Novolaspa zircon megacrysts crystallized from two different proto-kimberlite melts, whereas the process of kimberlite formation was very complex and included several episodes of proto-kimberlite emplacement, separated by long periods of time.

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ВІК ТА СКЛАД ЦИРКОНІВ З ДЕВОНСЬКОЇ КІМБЕРЛІТОВОЇ ТРУБКИ ПЕТРІВСЬКЕ, ПРИАЗОВ'Я, УКРАЇНСЬКИЙ ЩИТ

Викладено результати дослідження U-Pb і Hf ізотопних систем та визначення вмісту рідкісних елементів у п'яти кристалах циркону з девонської кімберлітової трубки Петрівське. Чотири кристали мають палеопротерозойський та архейський вік, тоді як один кристал має девонський вік в 383,6 ± 4,4 млн рр. (середньозважений $^{206}Pb/^{238}U$ вік). Циркони докембрійського віку походять з теригенних порід миколаївської світи, які січуться кімберлітами, або ж безпосередньо з докембрійських породних комплексів, які складають континентальну кору Східного Приазов'я. Кристал циркону девонського віку має U-Pb вік, який відповідає часу вкорінення кімберліту. Він на 14 млн рр. молодший, ніж мегакристи циркону, виявлені в Новоласпінській кімберлітовій трубці, розташованої неподалік. Окрім того, циркони з Петрівської трубки мають вищі концентрації рідкісних елементів, ніж циркони з Новоласпінської трубки. Циркони з цих двох трубок кристалізувалися з двох різних протокімберлітових розплавів, тоді як процес формування кімберлітів був дуже складним і, можливо, охоплював декілька епізодів утворення протокімберлітових розплавів, які відокремлені тривалими (більше 10 млн рр.) відтинками часу.

Ключові слова: кімберліт, циркон, Український щит, U-Pb вік, ізотопи Hf, рідкісні елементи.