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Uraninite in Variscan granitic pegmatites of the Tatric Unit (Western Carpathians, Slovakia): In-situ U–Th–Pb EDX dating

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Abstract: Uraninite occurs as rare accessory mineral in barren and beryl–columbite granitic pegmatites genetically related to Variscan S- and I-type granitic rocks from the Tatric Unit of the Western Carpathians, Slovakia. Uraninite forms individual euhedral crystals in alkali feldspars and muscovite (Prašivá Massif, Nízke Tatry Mountains) or more frequently numerous tiny (up to 15 µm in size) anhedral to subhedral inclusions in host metamict zircon (Žiar and Považský Inovec Mts.). Textural relationships indicate primary magmatic origin of individual euhedral uraninite crystals, but a subsolidus, secondary formation of uraninite inclusions in zircon by alteration processes, including metamictization and fluid-driven dissolution–reprecipitation of host zircon. The SEM-EDX based U–Th–Pb measurements and dating of uraninite were carried out. The primary magmatic uraninite, found in a pegmatite from the Prašivá Massif (Nízke Tatry Mts.), was dated at a weighted average age of 347±5 Ma. Systematically older single grain ages between 380–360 Ma were obtained for uraninite micro-inclusions in zircons in case of four pegmatites from the Považský Inovec Mts. In another pegmatite sample from that area, uraninite inclusions in zircon yielded systematically younger single grain ages between 336±9 Ma and 351±10 Ma (343±4 Ma in average). Uraninite from the Žiar Mts. pegmatite gave very consistent younger average age of 325±5 Ma. The non-conformity of uraninite ages in the Tatric West-Carpathian pegmatites is remarkable. The observed spread of ages would accord with recent geological models that propose a long-lived (at least two-stage) Variscan granitic–pegmatitic activity in the Tatric Unit during the Upper Devonian and the Lower Carboniferous. However, partial leaching and escaping of highly mobile U^{6+} from uraninite (especially from micrometer-sized inclusions in zircon) could be disturbed the U–Th–Pb system and gave seemingly higher age results. Conversely, partial Pb-loss could have yielded apparently younger uraninite ages. Consequently, the reliability and geological significance of the micro-uraninite ages are difficult to assess at present, and more geochronological data from other pegmatite minerals will be necessary to fully evaluate their significance.

Keywords: uraninite, zircon, pegmatite, U–Th–Pb dating, Variscan age, Tatric Unit, Western Carpathians

Introduction

Uraninite is an increasingly recognized accessory mineral for U–Th–Pb geochronological studies, with the restriction that it is less abundant in rocks compared to more common U–Th–Pb geochronometers such as zircon and monazite (Harlov et al. 2023, 2024). However, in the last years there have been more and more scanning electron microscope (SEM) studies which show that as an accessory micromineral, uraninite can be found in many rocks (Finger et al. 2017). Uraninite has an extremely high production rate of radiogenic Pb and, in general, a low common Pb content, due to incompatible incorporation of Pb in the uraninite structure. Moreover, Pb diffusion in uraninite is sluggish, such that post-crystallization Pb-loss effects in unaltered uraninite are relatively negligible (Waitzinger & Finger 2018). In this respect, uraninite could be considered almost as robust geochronometer as zircon or monazite, and thus highly suitable for U–Th–total Pb dating using the electron microprobe (EPMA) or scanning electron microscope–energy dispersive X-ray analysis (SEM–EDX). These methods allow for even very small uraninite grains and individual domains within larger grains to be targeted (Waitzinger & Finger 2018). However, it is worth mentioning that uraninite is highly prone to alteration upon interaction with hydrothermal fluids, which can compromise its suitability for geochronological studies if such alteration is not carefully assessed (e.g., Janeczek & Ewing 1992; Alexandre & Kyser 2005; Deditius et al. 2007; Yuan et al. 2019; Zhang et al. 2021; Harlov et al. 2024).

Geochronological dating of minerals in the West-Carpathian territory has a history of more than 60 years. At the beginning stood the K–Ar method (Kantor 1959), later followed the Rb–Sr method (e.g., Bagdasaryan et al. 1982) and classical (multigrain) TIMS U–Th–Pb zircon dating (e.g., Bibikova et

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al. 1988). The new Millennium brought the wide applications of in-situ zircon and monazite dating techniques, performed by SHRIMP, SIMS, LA-ICP-MS or EPMA (see reviews and data in Petrík & Kohút 1997; Petrík et al. 2001; Finger et al. 2003; Kohút et al. 2009; Broska et al. 2013; Kohút & Larionov 2021). Nowadays, geochronological research in the Western Carpathians is increasingly involving a broader range of minerals, including titanite (Uher et al. 2019), molybdenite (Kohút et al. 2024), and uraninite (Majzlan et al. 2020).

In this study, we present SEM–EDX-based measurements of U, Th, and Pb in accessory uraninite from seven samples of granitic pegmatites related to Variscan granitic massifs of the Nízke Tatry, Žiar and Považský Inovec Mountains of the Tatric Unit, the Western Carpathians (Slovakia). Besides of large individual crystals in one specimen, all other investigated samples represent tiny (several micrometer-sized) uraninite inclusions in metamict zircon. Our research represents the first attempt to date uraninite of this textural type by the in-situ U–Th–total Pb method. We discuss the geological significance of the obtained ages in the context of Variscan granite petrogenesis in the Tatric Unit versus a possible role of uraninite alteration and the resulting age disturbance.

Geological setting

The investigated uraninite occurs as rare accessory mineral in granitic pegmatites related to Variscan granitic rocks of the Paleozoic basement of the Tatric Unit, a significant part of the Western Carpathians (Fig. 1). The Western Carpathians form approximately W–E striking, northward-convex mountain range in the northernmost segment of the European Alpides. Paleozoic metamorphic and magmatic rocks form the oldest part of the Tatric Unit, incorporated into younger Alpine tectonic system of several allochthonous nappe sheets of Cretaceous age, which contain mainly Mesozoic sedimentary sequences. (Plašienka 2018).

Greenschist- to amphibolite-facies metamorphic rocks of the Tatric basement comprise mainly Ordovician to Devonian metapsammites to metapelites (phyllites, micaschists to paragneisses), alternating with metagranitic rocks (orthogneisses) and products of predominantly basic volcanism and plutonism (greenschists, amphibolites, metagabbros), and rare occurrences of metacarbonates, that have overcome several stages of regional and contact-thermal tectono-metamorphic overprints during Variscan (Upper Devonian to Carboniferous) orogenesis (e.g., Krist et al. 1992; Ivan et al. 2001; Putiš et al. 2008; Janák et al. 2022).

The metamorphic rocks of the Tatric Unit are intruded by widespread Variscan granitic rocks with Late Devonian to Early Carboniferous intrusion ages (see reviews by Petrík & Kohút 1997; Broska & Uher 2001; Petrík et al. 2001; Broska et al. 2013; Kohút 2014; Kohút & Larionov 2021). They form zoned plutons or composite massifs (intrusions within intrusions). The visual delineation of individual granite subtypes is often complicated due to the superimposed Alpine tectonics. The Tatric granitic rocks are mainly calc-alkaline, slightly metaluminous to peraluminous I-type (or I/S hybrid) biotite (±hornblende) tonalites and granodiorites, and less frequently S-type, muscovite-biotite granodiorites to (leuco)granites.

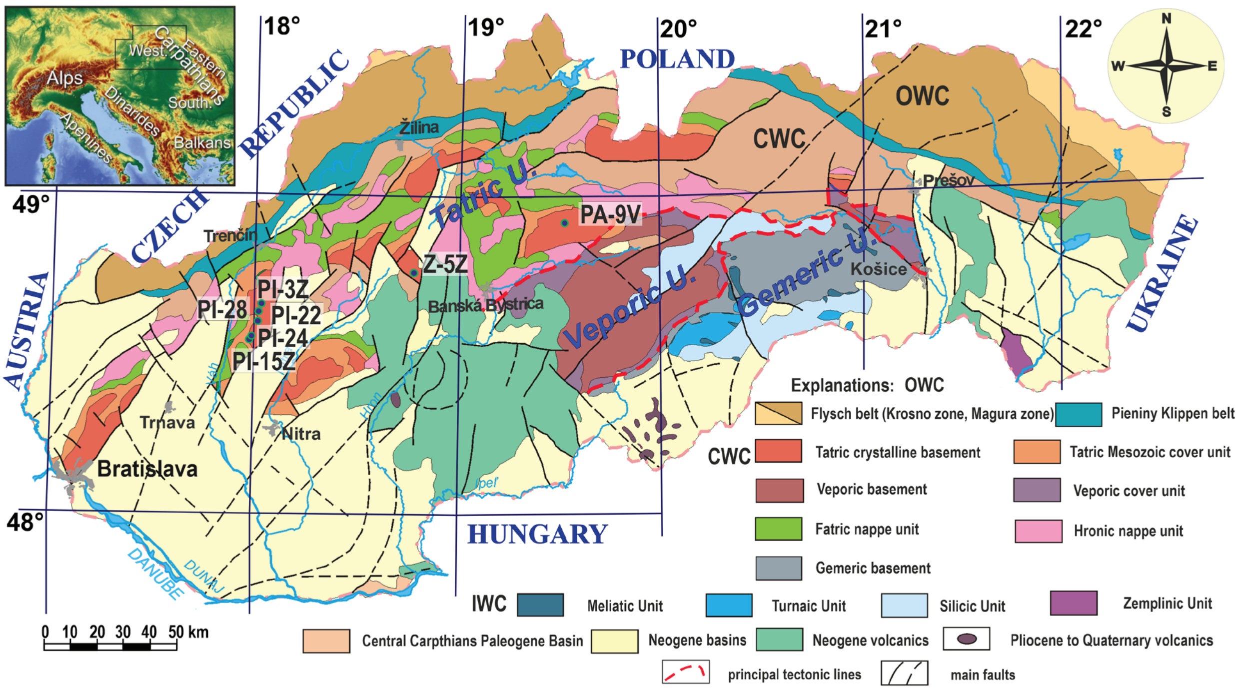

Fig. 1. Tectonic map showing the principal units in the Slovakian part of the Western Carpathians, simplified from Lexa et al. (2000), with the locations of studied samples indicated. Explanations: OWC – Outer Western Carpathians; CWC – Central Western Carpathians.

Granitic pegmatites are widespread in the Variscan granitic massifs of the Tatric Unit. The pegmatites form tabular bodies (dykes), less frequently lensoidal bodies, which intrude into the granitic rocks and, more rarely, the adjacent metamorphic rocks. The thickness of the pegmatites is usually from 0.5 to 2 m, locally up to 8 m (the Moravany nad Váhom, Striebornica Ridge pegmatite). The bodies attain a length of ca. 100 m; the contacts with adjacent rocks are usually sharp. The pegmatites commonly show internal, more or less well-developed zoning, generally with a fine-crystalline aplitic to coarse-crystalline quartz+K-feldspar (microcline)+albite+muscovite± biotite (annite) marginal zone, an intermediate zone with coarse-grained to blocky K-feldspar (±quartz, muscovite, or biotite), and a quartz core zone. Locally, irregular late, albite-rich, assemblages with dominant fine-grained saccharoidal or platy albite (cleavelandite variety) aggregates partially replace the older pegmatite units. Accessory minerals include mainly garnet (almandine–spessartine), less abundant metamict Hf-rich zircon and fluorapatite, rarely gahnite, monazite-(Ce), xenotime-(Y), uraninite, pyrite, ilmenite, native bismuth and other less frequent phases.

The most evolved granitic pegmatites of the Tatric Unit contain also rare-element accessory minerals of Be (beryl, phenakite and bertrandite), locally members of columbite, tapiolite, ixiolite, wodginite, microlite and pyrochlore groups, Nb–Ta-rich rutile, fersmite etc. (e.g., Uher et al. 1994, 1998b, 1998c, 2010, 2022, 2024; Uher & Broska 1995; Novák et al. 2000; Chudík et al. 2011). The mineral association and its chemical composition, as well as geochemical signatures of these most evolved granitic pegmatites of the Tatric Unit, enable classification into the lithium–caesium–tantalum (LCT) family, beryl type, and beryl–columbite subtype of the rare-element class (Černý & Ercit 2005).

Studied pegmatite rocks

Our study concerns accessory uraninite in three beryl–columbite granitic pegmatites from the Nízke Tatry, Žiar and Považský Inovec Mts., and three barren granitic pegmatites plus one pegmatitic leucogranite (without Be and Nb–Ta minerals) from the Považský Inovec Mts. The sampling points are indicated in Fig. 1, and the Appendix lists the sample points along with their locality names and precise geographic positions.

Prašivá Massif, Nízke Tatry Mts.

Sample PA-9V: The investigated granitic pegmatite forms a ca. 1 m thick dyke in the Prašivá-type granodiorite to granite. It is exposed in the abandoned Rakytová adit near the Augustín stibnite vein system in the Ľubeľská part of the Dúbrava antimony deposit (14 km SW of Liptovský Mikuláš town). The pegmatite dyke consists of coarse-grained crystalline (5 to 10 cm) K-feldspar–albite–quartz–muscovite to blocky K-feldspar (microcline) and graphic K-feldspar–quartz parts, and a quartz core. Accessory minerals comprise almandine–spessartine, zircon, fluorapatite, monazite-(Ce), pyrite, arsenopyrite, uraninite, rutile, columbite-(Mn) to tantalite-(Mn), and secondary stibiotantalite (Dávidová 1998; Uher 2000). Uraninite forms black crystals (up to 1 cm across) in association with blocky microcline, albite, and muscovite (Fig. 2).

Žiar Mts.

Sample Z-5Z: The investigated sample consists of fragments of granitic pegmatite material found on a forest ridge near Stredná Valley, approximately 600 m northeast of the Uhlisko gamekeeper's house and in the vicinity of Ráztočno and Jalovec villages, about 5 km north of Handlová town, in the southern part of the Žiar granitic massif. The pegmatite is situated within medium- to coarse-grained, banded, and augen orthogneisses of Ordovician age (486±11 Ma U–Pb zircon dating; Kohút 2013), which form the southern edge of the Variscan Žiar granitic massif. The thickness of the pegmatite body (dyke or lenticular in shape) exceeds 0.5 m, and it displays textural zoning, with aplitic, coarse-grained quartz–alkali feldspar–muscovite, graphic K-feldspar–quartz, blocky K-feldspar, and quartz core zones, locally with extensive albitization (saccharoidal albite-rich aplite). Accessory minerals of the pegmatite include zircon with uraninite inclusions, almandine to spessartine, fluorapatite, columbite-(Fe), columbite-(Mn), tantalite-(Fe), and secondary microlite-group minerals (Uher et al. 1994; Uher 2008).

Považský Inovec Mts.

Sample PI-15Z was collected from a large zoned pegmatite dyke (approximately 100 m long and up to 8 m wide) that intruded the Variscan muscovite-biotite granodiorites and

Fig. 2. Euhedral black uraninite crystal (6 mm in size) associated with muscovite and quartz in pegmatite from the Rakytová adit, Dúbrava antimony mine. Photo and sample courtesy of Martin Števko.

Paleozoic biotitic paragneisses, situated on the Striebornica ridge, about 2.4 km ESE of Moravany nad Váhom village and approximately 4 km east of Piešťany town. The pegmatite displays distinct zoning with graphic to blocky K-feldspar, coarse-grained quartz–K-feldspar–albite–muscovite, and quartz core zones, and is extensively albitized (saccharoidal albite aplite and cleavelandite domains). The dyke represents one of the most fractionated granitic pegmatite types known in the Western Carpathians; there is the common presence of beryl (crystals up to 15 cm across), secondary bertrandite, and Nb–Ta oxide minerals, including columbite-(Fe), columbite-(Mn), tantalite-(Fe), tantalite-(Mn), tapiolite-(Fe), and microlite-group members (e.g., Uher et al. 1994, 2010, 2022; Uher & Broska 1995; Novák et al. 2000). Other accessory minerals include Hf-rich zircon (up to 11 wt.% HfO_2) with uraninite inclusions (Uher & Černý 1998), almandine to spessartine, fluorapatite, monazite-(Ce), gahnite, cassiterite, pyrite, arsenopyrite, and sphalerite.

Sample PI-24 is a medium-grained, pegmatitic muscovite leucogranite from Moravany nad Váhom, Striebornica ridge, in close vicinity of the PI-15Z sample. This rock represents a small (probably dyke- or cupola-shaped) body of more evolved, leucocratic rock with transitional character between granites and pegmatites. In addition to quartz, plagioclase, K-feldspar, and muscovite, the rock contains accessory almandine, zircon, biotite, sillimanite, fluorapatite, monazite-(Ce), epidote, pyrite, sphalerite, and galena.

Other samples (PI-22, PI-3Z, and PI-32) represent relatively narrow (usually less than 1 m thick) and slightly zoned barren pegmatite dykes within host Paleozoic biotite paragneisses to micaschists (PI-22 and PI-28) or Variscan granitic rocks (PI-3Z).

Sample PI-22 is a coarse-grained quartz–K-feldspar–muscovite pegmatite dyke in migmatized biotite paragneiss xenoliths in adjacent granodiorites, from an abandoned quarry in Hradná Valley, approximately 3 km NW of Bojná village and about 10 km WNW of Topoľčany town. The pegmatite contains accessory almandine, sillimanite, epidote, zircon with uraninite inclusions, fluorapatite, monazite-(Ce), xenotime-(Y), rutile, and pyrite.

Sample PI-3Z is a coarse-grained quartz–K-feldspar–muscovite pegmatite dyke in leucocratic biotite–muscovite syenogranites, located on the N slope of Soľnisko Hill, approximately 4 km NW of Prašice village and 14 km NW of Topoľčany town. Accessory minerals comprise almandine, zircon with uraninite inclusions, fluorapatite, monazite-(Ce), xenotime-(Y), epidote, sillimanite, rutile, ilmenite, magnetite, pyrite, and arsenopyrite.

Sample PI-28 is a coarse-grained quartz–K-feldspar–muscovite–biotite pegmatite dyke in biotite paragneisses, located in a small valley W of Hrabový Hill, about 2 km NW of Podhradie village and approximately 15 km NW of Topoľčany town. Accessory minerals include almandine, zircon with uraninite inclusions, fluorapatite, monazite-(Ce), xenotime-(Y), anatase, brookite, ilmenite, magnetite, and pyrite.

Analytical methods

Uraninite has long been recognized as a suitable mineral for U–Th–total Pb dating using electron beam methods (e.g., Parslow et al. 1985; Bowles 1990; Cocherie & Legendre 2007; Votyakov et al. 2013; Waitzinger & Finger 2018). The uraninite grains analyzed in this study are mostly very small. Therefore, we applied the EDX-SEM-based low-voltage method of Waitzinger & Finger (2018), which offers the advantage of high spatial analytical resolution. Analyses were conducted on polished and carbon-coated heavy mineral mounts using a Zeiss ULTRAPLUS scanning electron microscope (SEM) equipped with a field emission (FE) cathode. Uniform electron beam conditions were maintained for quantitative analysis, with an accelerating voltage of 8 kV, a beam current of 2 nA, and a beam diameter of 100 nm. EDX analysis was performed with a large-area (50 mm²) silicon drift detector (X-MAX 50, Oxford Instruments). A counting time of 3 minutes resulted in an analytical precision of approximately ±0.4 wt.% (1σ) for U, while Pb could be analyzed at a detection limit of ~0.2 wt.%. The analytical uncertainty for Pb in uraninite is typically ~0.1 wt.%, corresponding to an error of about 18 Ma (1σ) for a single spot age. Thorium and other minor elements, (Si, Ca, Ce, and Y) can be analyzed at detection limits of 0.1–1.3 wt.%. Measurements on an uraninite reference material (Waitzinger & Finger 2018) were repeatedly carried out between sample measurements for quality control. The recommended age for this standard (91 Ma; TIMS age, Paar & Köppel 1978) was reproduced within error in every analysis. All analysis and evaluation methods were carried out according to Waitzinger & Finger (2018). To potentially rule out effects of post-crystalline alteration, short profiles with approximately 0.3 µm spacing were recorded wherever possible. A mean grain age was calculated with the Isoplot weighted average procedure when all profile measurements yielded consistent ages within analytical error (Ludwig 2012); in such cases, we assume the grains were unaffected by post-crystalline alteration. Most (about 90 %) of the measured grains passed this test.

Results

Uraninite textures and chemical composition

Investigated uraninite occurs in two principal forms: (1) large single crystals in alkali feldspar and muscovite, and (2) tiny inclusions in zircon. Single euhedral to subhedral uraninite crystals occur in the Dúbrava, Rakytová adit pegmatite, Nízke Tatry Mts. (PA-9V sample); they are mostly 100–200 µm in size, but occasionally even larger (Figs. 2, 3). The BSE images generally show homogeneous texture of uraninite, without compositional zonality (Fig. 3). However, EDX measurements revealed a few potentially altered domains in these crystals, with lower BSE signal corresponding to increased contents of Si, Ca, Sb and As, and a reduced

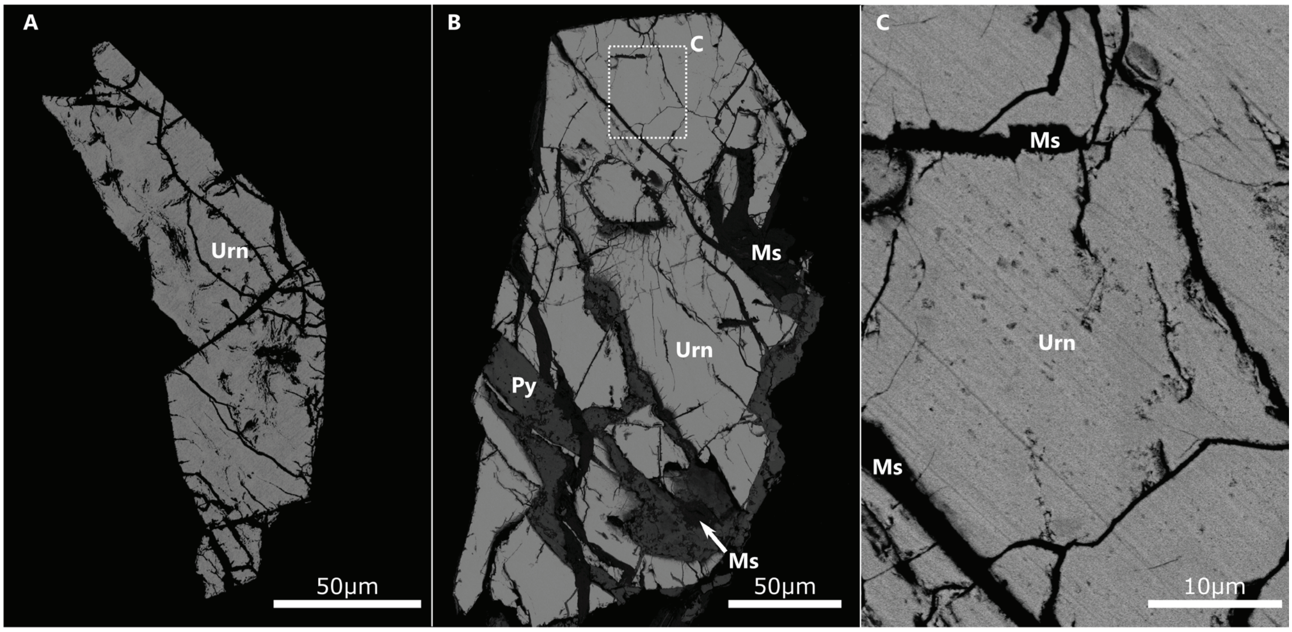

Fig. 3. Backscattered Electron (BSE) images of uraninite crystals from the Dúbrava, Rakytová adit pegmatite (Prašivá Massif, Nízke Tatry Mts., sample PA-9V) showing alteration along cracks (secondary mica and pyrite formation).

U concentration. These domains were excluded from age dating.

On the other hand, tiny uraninite inclusions (≤1 to 15 µm in size) form mostly anhedral to subhedral, rarely euhedral inclusions in metamict zircon, locally along with xenotime-(Y) inclusions or intergrowths (up to 120 µm), as well as muscovite and quartz inclusions (Fig. 4). The BSE images show that the uraninite inclusions are mostly concentrated in distinct alteration zones within the zircon, which likely had high primary U and Th contents. A thin inclusion-free rim is visible in Fig. 4A, relict concentric zoning is observed in Fig. 4D, and patchy zoning appears in Fig. 4F. Host zircon forms non-transparent, grey to brown, euhedral prismatic-dipyramidal crystals, usually 0.2 to 0.5 mm large, with nearly homogeneous to irregularly zonal texture, often porous with numerous anhedral micrometer-sized voids, corresponding with its metamict alteration.

The chemical composition of the investigated uraninite crystals and inclusions (Table 1) is dominated by uranium (89–96 wt.% UO_2). Subordinately, variable contents of thorium (0.0–4.5 wt.% ThO_2) are measured as well as regular and relatively uniform concentrations of lead (4.0–5.0 wt.% PbO). Contents of other elements are usually below detection limit; only Si and Y were detected in some analyses (up to 0.4 wt.% SiO_2, up to 1.1 wt.% Y_2O_3).

Age results

Fresh domains of large uraninite crystal from the Dúbrava, Rakytová pegmatite (Nízke Tatry Mts., sample PA-9V) yielded internally consistent single-point ages ranging from 373 to 335 Ma, which can be combined to a mean age of 347±5 Ma (Table 1).

Metamict zircon in the Ráztočno pegmatite (Žiar Mts., sample Z-5Z) contains numerous uraninite inclusions (Fig. 4A–C); however, most of them appear heterogeneous in BSE imaging and were therefore considered unsuitable for dating. We analyzed short profiles in four homogeneously looking uraninite inclusions (each 1–2 µm in size). The analyses yielded a consistent mean age of 325 ± 5 Ma (Table 1).

In three samples (PI-15Z, PI-24, and PI-3Z; Moravany nad Váhom and Prašice, Soľnisko Hill, the Považský Inovec Mts.), the uraninite inclusions yielded ages of approximately 370 Ma, which were excellently reproduced in profile measurements (Table 1). In contrast, somewhat younger uraninite ages of 359±10 Ma and 343±4 Ma were obtained from two other pegmatites from the Považský Inovec Mts. (Bojná, Hradná Valley, and Podhradie, Hrabový Hill pegmatites, samples PI-22 and PI-28).

Discussion

Textural relationships and composition of uraninite

Single large and nearly euhedral crystals of uraninite from the Dúbrava, Rakytová pegmatite (sample PA-9V) are associated with large K-feldspar and muscovite. Consequently, this uraninite can be interpreted as a primary magmatic mineral that precipitated from a highly fractionated pegmatite melt. On the contrary, textural relationships of the uraninite microinclusions in metamict zircon from the other investigated

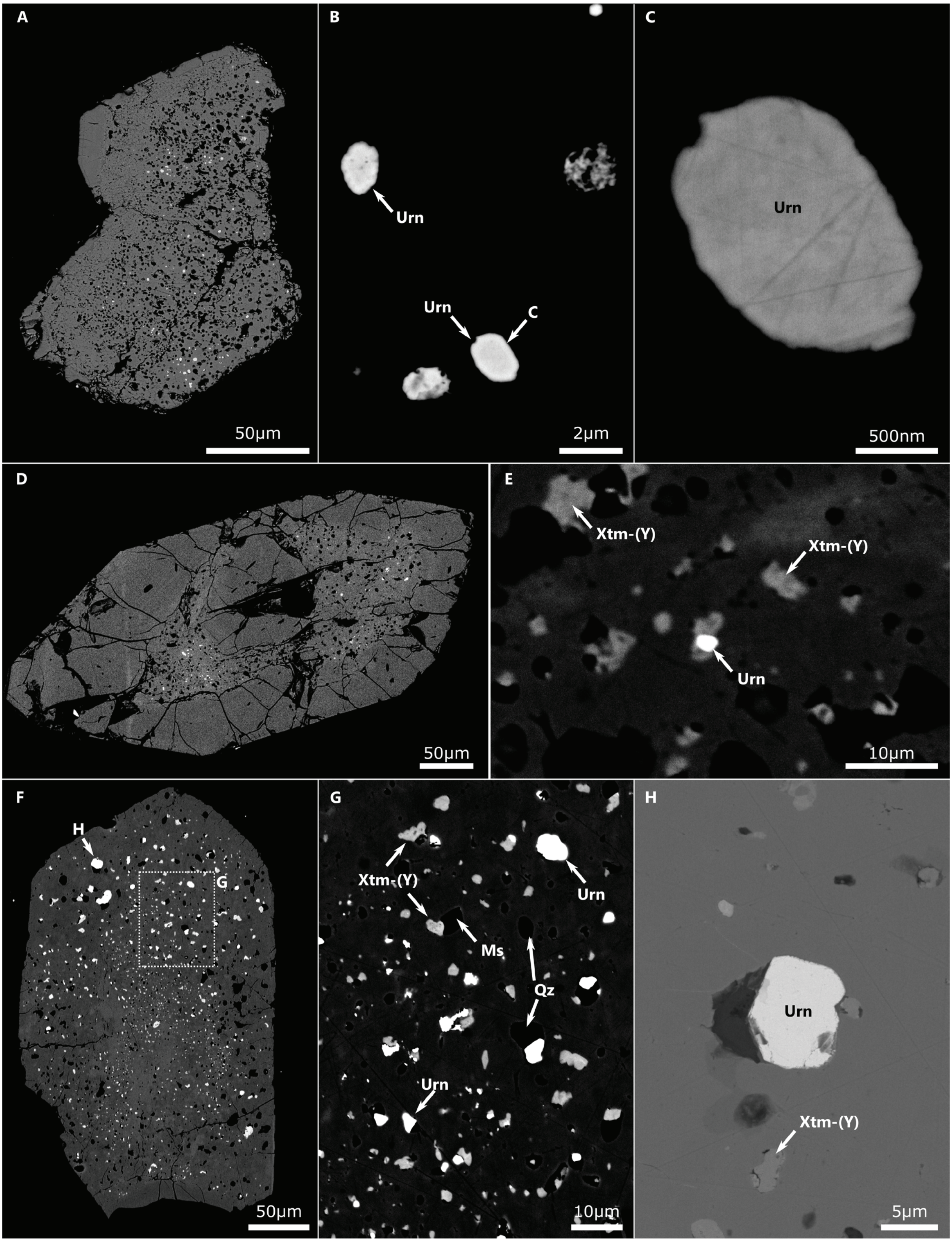

Fig. 4. Metamict zircons with numerous uraninite and locally xenotime-(Y) inclusions from pegmatite samples: **A–C**: Ráztočno pegmatite (Žiar Mts., sample Z-5Z); A — zircon with uraninite inclusions and an inclusion-free rim on the left side; B and C — uraninites (Urn) from A; **D, E**: Bojná, Hradná Valley pegmatite (Považský Inovec Mts., sample PI-22); D — showing a concentrically zoned zircon with uraninite inclusions; E — higher magnification of D revealing microcrystals of xenotime-(Y) (Xtm-Y)and uraninite (Urn); **F–H**: Prašice, Soľnisko Hill pegmatite (Považský Inovec Mts., sample PI-3Z); F — patchy zoned zircon with uraninite inclusions; G — higher magnification of rectangle in F showing inclusions of uraninite (Urn), xenotime-(Y) (Xtm-Y), muscovite (Ms), and quartz (Qz); H — bright crystal of uraninite in zircon from F.

Table 1: U, Th, and Pb contents, single-point and single-grain dates for uraninite microcrystals from Western Carpathian pegmatite samples. Note: < values under detection limit (0.1 wt.% for SiO_2 and 0.5 wt.% for Y_2O_3).

Sample	Locality ID	SiO_2 wt.%	ThO_2 wt.%	UO_2 wt.%	Y_2O_3 wt.%	PbO wt.%	Total wt.%	Age Ma	1σ error Ma
PA-9V	**Rakytová adit – NT Mts.**								
	Point 1	<	2.66	91.85	<	4.35	98.86	*342*	*18*
	Point 2	0.17	2.63	89.25	<	4.59	96.64	*371*	*18*
	Point 3	<	2.71	90.12	<	4.27	97.10	*343*	*18*
	Point 5	<	2.43	88.83	<	4.22	95.48	*344*	*18*
	Point 7	<	2.25	91.95	<	4.27	98.47	*336*	*18*
	Point 8	0.19	2.33	91.24	<	4.54	98.30	*360*	*18*
	Point 9	<	2.09	91.67	<	4.73	98.49	*373*	*18*
	Point 10	<	0.62	94.81	<	4.36	99.79	*335*	*17*
	Point 11	<	1.28	94.91	<	4.53	100.72	*347*	*17*
	Point 12	<	0.89	92.77	<	4.28	97.94	*336*	*18*
	Point 13	<	1.55	92.71	<	4.29	98.55	*336*	*18*
	Point 14	<	1.84	93.57	<	4.61	100.02	*357*	*18*
	Point 15	<	1.72	88.69	<	4.09	94.50	*335*	*19*
							Overall mean	***347***	***5***
Z-5Z	**Uhlisko, Ráztočno – Žiar Mts.**								
	Grain 1/1	0.29	0.49	94.18	<	4.09	99.05	*317*	*18*
	Grain 1/2	0.27	0.42	93.42	<	4.20	98.31	*328*	*18*
	Grain 1/3	0.24	0.19	92.81	<	4.22	97.46	*332*	*18*
	Grain 1/4	0.22	0.61	93.72	<	4.12	98.67	*321*	*18*
	Grain 1/5	0.19	0.68	94.26	<	4.11	99.24	*319*	*18*
							Mean	***323***	***8***
	Grain 2/1	0.20	0.00	94.86	<	4.17	99.23	*322*	*18*
	Grain 2/2	0.24	0.00	92.61	<	4.31	97.16	*340*	*18*
	Grain 2/3	0.20	0.00	91.23	<	4.00	95.43	*321*	*18*
							Mean	***328***	***11***
	Grain 3/1	0.27	0.00	94.57	<	4.20	99.04	*325*	*17*
	Grain 3/2	0.24	0.20	89.99	<	4.04	94.47	*328*	*18*
							Mean	***327***	***13***
	Grain 4/1	0.22	0.15	96.22	<	4.33	100.92	*329*	*18*
	Grain 4/2	0.24	0.48	93.74	<	4.13	98.59	*322*	*18*
	Grain 4/3	0.35	0.28	93.39	<	4.17	98.19	*326*	*18*
							Mean	***326***	***10***
							Overall mean	***325***	***5***
PI-15Z	**Striebornica Hill – PI Mts.**								
	Grain 1/1	<	4.35	89.83	<	4.66	98.84	*372*	*18*
	Grain 1/2	<	4.51	88.79	<	4.74	98.04	*382*	*17*
							Overall mean	***377***	***13***
PI-24	**Striebornica Hill – PI Mts.**								
	Grain 1/1	<	0.93	92.99	<	4.82	98.74	*376*	*18*
	Grain 1/2	<	0.52	96.17	<	4.85	101.54	*367*	*18*
	Grain 1/3	<	0.63	93.59	<	4.82	99.04	*374*	*18*
							Mean	***372***	***10***
	Grain 2/1	0.31	1.31	91.70	<	4.98	98.30	*393*	*19*
	Grain 2/2	<	0.93	93.18	<	4.58	98.69	*357*	*18*
	Grain 2/3	0.21	1.10	93.03	<	4.82	99.16	*375*	*18*
							Mean	***375***	***11***
	Grain 3/1	0.22	1.89	91.34	0.52	4.86	98.83	*383*	*19*
	Grain 3/2	0.24	0.82	95.70	<	4.75	101.51	*361*	*18*
							Mean	***372***	***13***
	Grain 4a/1	<	0.68	93.69	<	4.51	98.88	*351*	*18*
	Grain 4a/2	<	0.65	91.67	<	4.80	97.12	*380*	*19*
	Grain 4a/3	<	0.45	92.06	<	4.94	97.45	*389*	*19*
							Mean	***373***	***11***
	Grain 4b/1	0.25	0.52	92.06	<	4.66	97.49	*368*	*19*
	Grain 4b/2	0.23	0.64	93.40	<	4.82	99.09	*375*	*19*
	Grain 4b/3	0.25	0.32	93.24	<	5.04	98.85	*392*	*19*
	Grain 4b/4	<	0.59	95.02	<	4.97	100.58	*380*	*18*
	Grain 4b/5	<	0.59	95.02	<	4.97	100.58	*380*	*18*
							Mean	***379***	***9***

Table 1: continued

Sample	Locality ID	SiO_2 wt.%	ThO_2 wt.%	UO_2 wt.%	Y_2O_3 wt.%	PbO wt.%	Total wt.%	Age Ma	1σ error Ma
PI-24	**continued**								
	Grain 5a/1	<	0.57	93.37	<	4.88	98.82	*380*	*18*
	Grain 5a/2	<	1.00	94.41	<	4.88	100.29	*375*	*18*
	Grain 5a/3	<	0.25	94.67	<	4.88	99.80	*375*	*18*
							Mean	***377***	***11***
	Grain 5b/1	<	0.03	96.23	<	4.71	100.97	*357*	*18*
	Grain 5b/2	<	0.42	94.55	<	4.80	99.77	*370*	*18*
	Grain 5b/3	0.22	0.26	94.35	<	4.72	99.55	*364*	*18*
							Mean	***364***	***11***
							Overall mean	***373***	***4***
PI-22	**Hradná Valley – PI Mts.**								
	Grain 1	<	0.46	93.67	<	4.60	98.73	*358*	*17*
	Grain 2	<	1.48	93.93	1.12	4.73	101.26	*365*	*18*
	Grain 3	<	0.29	92.30	0.63	4.49	97.71	*354*	*18*
							Overall mean	***359***	***10***
PI-3Z	**Hrabový Hill – PI Mts.**								
	Grain 1/1	<	0.31	95.12	<	4.82	100.25	*369*	*18*
	Grain 1/2	<	0.72	94.95	<	4.92	100.59	*376*	*18*
	Grain 1/3	<	0.62	96.14	<	4.85	101.61	*367*	*17*
	Grain 1/4	<	0.83	95.86	<	4.98	101.67	*377*	*17*
							Mean	***372***	***9***
	Grain 1/5	<	0.05	92.53	<	4.51	97.09	*356*	*18*
	Grain 1/6	<	0.22	91.97	<	4.79	96.98	*379*	*18*
	Grain 1/7	<	0.34	93.32	<	4.87	98.53	*379*	*18*
							Mean	***371***	***11***
	Grain 2/1	0.27	0.39	95.36	<	4.78	100.80	*365*	*18*
	Grain 2/2	<	0.13	95.33	<	4.89	100.35	*374*	*18*
	Grain 2/3	<	0.16	94.86	<	4.64	99.66	*356*	*18*
							Mean	***365***	***10***
	Grain 3/1	<	0.04	94.71	<	4.75	99.50	*365*	*18*
	Grain 3/2	<	0.32	94.76	<	4.84	99.92	*371*	*18*
	Grain 3/3	0.18	0.35	93.95	<	4.69	99.17	*364*	*18*
	Grain 3/4	<	0.32	95.69	<	4.76	100.77	*362*	*18*
							Mean	***366***	***9***
							Overall mean	***369***	***5***
PI-28	**Solisko – PI MTS.**								
	Grain 1/1	<	0.36	95.94	<	4.51	100.81	*343*	*18*
	Grain 1/2	<	0.21	95.34	<	4.48	100.03	*343*	*18*
	Grain 1/3	<	0.14	96.02	<	4.43	100.59	*337*	*18*
	Grain 1/4	<	0.26	94.50	<	4.25	99.01	*328*	*18*
							Mean	***336***	***9***
	Grain 1/5	<	0.45	95.56	<	4.20	100.21	*321*	*18*
	Grain 1/6	<	0.28	95.68	<	4.72	100.68	*360*	*18*
	Grain 1/7	<	0.01	94.94	<	4.31	99.26	*332*	*18*
	Grain 1/8	<	0.73	94.58	<	4.36	99.67	*336*	*18*
							Mean	***343***	***9***
	Grain 1/9	<	0.64	95.18	<	4.43	100.25	*339*	*18*
	Grain 1/10	<	0.47	95.47	<	4.58	100.52	*350*	*18*
	Grain 1/11	<	0.58	95.35	<	4.59	100.52	*350*	*18*
							Mean	***346***	***10***
	Grain 2/1	<	0.41	95.68	<	4.64	100.73	*353*	*18*
	Grain 2/2	<	0.43	95.24	<	4.52	100.19	*346*	*18*
	Grain 2/3	<	0.14	95.82	<	4.66	100.62	*355*	*18*
							Mean	***351***	***10***
	Grain 3/1	<	0.11	93.92	<	4.44	98.47	*345*	*18*
	Grain 3/2	<	0.09	95.35	<	4.38	99.82	*336*	*18*
	Grain 3/3	<	0.33	95.52	<	4.69	100.54	*358*	*18*
							Mean	***346***	***10***
							Overall mean	***343***	***4***

samples indicate their secondary origin. Analogous micro-inclusions of U and Th minerals (uraninite, thorite, thorianite) in altered, metamict zircon have been previously described worldwide from several pegmatites or granites (e.g., Uher & Marschalko 1993; Uher et al. 1998a; Geisler et al. 2007; Tramm et al. 2021; Zhang et al. 2021), and also from the Tatric granitic pegmatites (Uher & Černý 1998). Based on micro-structural observations, these inclusions must be considered secondary minerals that formed later than the host zircon, probably due to an alteration-related release of U and Th from the magmatic zircon lattice. Zircon alteration was driven by the radioactive destruction of the zircon structure (metamictization) and a contemporary fluid-mediated metasomatism, which stimulated coupled dissolution-reprecipitation of the zircon and the growth of U–Th rich microinclusions (Geisler et al. 2007; Hetherington & Harlov 2008; Putnis et al. 2009; Harlov et al. 2024).

When assessing the crystal chemistry of uraninite, its complex and non-stoichiometric behaviour must be taken into account. Besides the dominant U^{4+}, a part of uranium commonly occurs in more oxidized U^{6+} valency state, and trivalent and divalent cations (mainly REE^{3+}, Ca^{2+}, and radiogenic Pb^{2+}), can enter the uraninite structure via heterovalent substitution mechanisms (Janeczek & Ewing 1992). Radiogenic lead, formed by radioactive decay of U^{4+} ($+Th^{4+}$) exists as divalent Pb^{2+} cation, entering the uraninite structure via possible (1) $U^{4+}+O^{2-}=Pb^{2+}+\square$ (vacancy) and (2) $2U^{4+}=U^{6+}+Pb^{2+}$ substitution mechanisms (Syverson et al. 2019). Moreover, we can assume also other mechanisms, such as (3) $U^{4+}+O^{2-}=(U^{6+}O_2)^{2+}+\square$, (4) $3U^{4+}=2U^{6+}+\square$, (5) $(U, Th)^{4+}+Ca^{2+}=2REE^{3+}$; or (6) $(U, Th)^{4+}+O^{2-}=REE^{3+}+(OH)^-$, (7) $3(U, Th)^{4+}+\square=4REE^{3+}$, (8) $3U^{4+}=U^{6+}+2REE^{3+}$, and (9) $2(U, Th)^{4+}=Ca^{2+}+U^{6+}$ (Ondrejka et al. 2016). In any case, a part of uranium is likely to exist in the U^{6+} or uranyl $(U^{6+}O_2)^{2+}$ form. The U^{6+} cation is generally soluble in aqueous solutions and therefore mobile even in low-temperature environments (e.g., Plášil 2014). The alteration of uraninite connected with loss or redistribution of uranium was documented from U-rich granites (Kempe 2003; Zhang et al. 2021). Consequently, partial escape of uranium, mostly in U^{6+} (uranyl) form, from altered uraninite could play an important role in disturbing the U–Th–Pb radioactive decay system, and it may seriously affect geochronological results.

Uraninite ages

It is imperative to compare the newly obtained uraninite dates with other geochronological data that are available for the investigated pegmatites and their host granite massifs (Table 2). The larger, probably magmatic uraninite crystals from the Dúbrava, Rakytová adit pegmatite (Prašivá Massif, Nízke Tatry Mts., PA-9V sample) show consistent ages around 350 Ma. The obtained mean age of 347±5 Ma is very close to the zircon ages of 353±3 Ma and 352±3 Ma published for the Prašivá main granite (Broska et al. 2013; Maraszewska et al. 2022). It is slightly younger than the Ar–Ar muscovite age of 358±5 Ma for the pegmatite from the same locality (Rakytová adit, Uher 2005). The uraninite ages are thus another confirmation that the main granite- and pegmatite-forming events in this massif took place in the early Carboniferous.

The interpretation of the measured ages for uraninite micro-inclusions in metamict zircon is not as unambiguous as for

Table 2: Currently available geochronological dates for Tatric Unit pegmatites and related granite massifs.

Mountains	Granitic body	Granite age	Method	Source	Pegmatite age	Method	Source
Malé Karpaty Mts.	Bratislava Massif	355±5 Ma	zircon U–Th–Pb	Kohút et al. (2009)	354.5±4.5 Ma	columbite U–Pb	Uher et al. (2024)
		355±18 Ma	monazite CHIME	Finger et al. (2003)	357±6 Ma	monazite CHIME	Uher et al. (2014)
		353 ± 2 Ma	monazite CHIME	Uher et al. (2014)	352±5 Ma	monazite CHIME	Uher et al. (2014)
					337.1±3.4 Ma	muscovite Ar–Ar	Uher (2005)
	Staré Mesto Massif	363.5±2.1 Ma	zircon U–Th–Pb	Kohút et al. (2025)	368.6±1.4 Ma	molybdenite Re–Os	Kohút et al. (2024)
		360±4.5 Ma	monazite CHIME	Kohút et al. (2025)			
Považský Inovec Mts.	Striebornica Massif	367±5 Ma	zircon U–Th–Pb	Kohút et al. (2009)	360±5.0 Ma	columbite U–Pb	Uher et al. (2024)
		364±17 Ma	monazite CHIME	Finger et al. (2003)			
Strážovské Vrchy Mts.	Suchý Massif	353±1.2 Ma	zircon U–Th–Pb	Broska et al. (in review)	352±8.5 Ma	columbite U–Pb	Uher et al. (2024)
		342±13 Ma	monazite CHIME	Finger et al. (2003)			
Žiar Mts.	Central granite type	348±4.3 Ma	zircon U–Th–Pb	Kohút et al. (2009)			
		348±22 Ma	monazite CHIME	Finger et al. (2003)			
		332±2 Ma	zircon rims U–Th–Pb	Kohút et al. (2013)			
Západné Tatry Mts.	Wolowiec Massif	365.1±2.4 Ma	zircon U–Th–Pb	Burda & Gaweda (2009)	350.5±1.2 Ma	molybdenite Re–Os	Mikulski et al. (2011)
Nízke Tatry Mts.	Prašivá type	352±3 Ma	zircon U–Th–Pb	Maraszewska et al. (2022)	352±3 Ma	molybdenite Re–Os	Majzlan et al. (2020)
		353±3 Ma	zircon U–Th–Pb	Broska et al. (2013)	358.1±4.7 Ma	muscovite Ar–Ar	Uher (2005)

the primary magmatic uraninite crystals. Textural relationships indicate a secondary origin of the uraninite inclusions due to post-solidification radiation damage and fluid-mediated dissolution–reprecipitation alteration processes in host zircon. However, these alteration processes probably took place immediately after the primary magmatic precipitation of zircon from pegmatite melt, and the obtained uraninite ages could be interpreted as close to the actual age of emplacement and magmatic solidification of the pegmatite samples.

In the case of the Považský Inovec Mts., pegmatite samples PI-15Z, PI-24, and PI-3Z provided unexpectedly high and, at the same time, relatively constant ages for the uraninite inclusions in zircon. Single grain ages here are consistently between 364±11 Ma and 379±9 Ma, and can be combined to statistically robust mean ages of 373±4 Ma for Moravany nad Váhom pegmatitic leucogranite (PI-24) and 369±5 Ma for Prašice, Soľnisko Hill pegmatite (PI-3Z). These ages are broadly similar to a SHRIMP zircon date of 367±5 Ma measured for the Považský Inovec main granite (Kohút & Larionov 2021). Monazite U–Th–total Pb chemical dating of adjacent Moravany nad Váhom granodiorite also gave a relatively high age of 364±17 Ma (Finger et al. 2003). Recent LA–ICP–MS U–Pb dating of columbite-tantalite from the Moravany nad Váhom, Striebornica rare-element pegmatite (sample identical to PI-15Z) yielded a concordant age of 360±5.0 Ma (Uher et al. 2024). The relatively large discrepancy between the uraninite and columbite ages of the pegmatites (≥10 Myr) is not yet clear, and it creates uncertainties about how to interpret the uraninite ages. One plausible explanation is a possible partial oxidation of uraninite microinclusions and escape of U in soluble and mobile $(U^{6+}O_2)^{2+}$ form, which leads to a decrease of U/Pb ratios and higher corresponding ages. An alternative interpretation could be some excess Pb in the uraninite, possibly inherited from parental zircon. Anyway, more precise geochronological studies on pegmatites and granites from the same area would now be needed to either confirm a late Devonian pegmatite age or to critically revisit the uraninite dates. Understandably, the U–Th–total Pb dating of uraninite micro-inclusions alone may evoke skepticism regarding the reliability and geological significance of such data, especially because the method is relatively new and too little is known about its possible pitfalls and misinterpretations.

It is noteworthy that secondary uraninite micrograins in the two other pegmatite samples from the Považský Inovec Mts. (PI-22 and PI-28) both yielded younger mean U–Th–Pb ages: 359±10 Ma and 343±5 Ma, respectively. These ages would be consistent with the formation of the Považský Inovec pegmatites at ca. 360 Ma, as proposed by Uher et al. (2024). Secondary uraninite formation in the zircons may have been somewhat delayed relative to the dated columbite (Table 2) and perhaps triggered by another thermal event at ca. 350–340 Ma.

Uraninite from the Ráztočno pegmatite (Žiar Mts., sample Z-5Z) gave relatively young ages around 330–325 Ma. Notably, these results broadly fit a SHRIMP age of 332±2 Ma published by Kohút et al. (2013) for zircon rims, whereas the main population of zircons in the Žiar granite massif defines a Concordia age of 351–348 Ma (Kohút & Larionov 2021; Broska et al. in review) consistent with a 348±22 Ma U–Th–total Pb monazite age (Finger et al. 2003). The uraninite data would thus be compatible with a mid-Carboniferous (Visean) formation age of the pegmatite. Alternatively, these apparently younger ages could also be interpreted as a result of uraninite hydrothermal alteration, in this case possibly connected with partial leaching and escaping of radiogenic lead. Partial loss of Pb, connected with cation diffusion, fluid-aided leaching, and other processes are documented in uraninite from various genetic settings (e.g., Janeczek & Ewing 1995; Kempe 2003; Alexandre & Kyser 2005; Yuan et al. 2019).

Conclusions

To summarize, our U–Th–total Pb EDX-based dating of accessory uraninite from Variscan granitic pegmatites of the Tatric Unit (Western Carpathians, Slovakia) provides new and important insights for a discussion about the age and the petrogenetic context of pegmatite formation in the Tatric Unit. For a long time, a Carboniferous to early Permian age has generally been assumed for these pegmatites, and this was also confirmed by early geochronological results (Uher 2005; Uher et al. 2014). However, according to the most recent geochronological studies (Kohút et al. 2024; Uher et al. 2024), it appears possible that an older, late Devonian pegmatite generation is also present in some areas of the Tatric Unit, e.g., in the Považský Inovec Mts. and in the Staré Mesto Massif, Malé Karpaty Mts. (Table 2). Note that granite ages are also relatively old in these massifs. Kohút & Larionov (2021), on the basis of SHRIMP zircon dating, have recently argued in favour of long-lived, at least two-stage granitic activity: (1) the older, Famennian–Tournaisian event (~365–350 Ma) related to subduction and initial collision, and (2) the younger Visean event (~350–330 Ma) associated with collisional and post-collisional melting in the Tatric Unit of the Western Carpathians. Our uraninite U–Th–total Pb dating results also gave at least two age intervals, including late Devonian (~380 to 360 Ma) ages for some leucogranite to pegmatite samples located in the Považský Inovec Mts., as well as early Carboniferous ages (~350–340 Ma) for the Nízke Tatry and Považský Inovec Mts. and even a mid-Carboniferous age (~325 Ma) for the Ráztočno pegmatite in Žiar Mts.

However, tiny uraninite inclusions in metamict zircon are of secondary origin and formed during post-magmatic alteration of magmatic zircon. Such uraninite could be susceptible to hydrothermal leaching and partial escaping of U^{6+} in uranyl form. Partial loss of radiogenic Pb can cause seemingly younger calculated ages of uraninite. Therefore, apparently too high or too low U–Th–total Pb uraninite ages could be a result of the above-mentioned alteration processes. Only future detailed mineralogical and geochemical studies of the granite–pegmatite systems and their accessory minerals can resolve these open issues.

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Appendix

List of sample points with geographic positions and locality names.

Sample №	locality	latitude	longitude
PA-9V	Rakytová adit – NT Mts.	N 48°58’46.68”	E 19°30’12.6”
Z-5Z	Uhlisko, Ráztočno – Žiar Mts.	N 48°47’20.76”	E 18°45’49.5”
PI-15Z	Striebornica Hill – PI Mts.	N 48°35’33.72”	E 17°53’50.1”
PI-24	Striebornica Hill – PI Mts.	N 48°35’38.82”	E 17°53’55.5”
PI-22	Hradná Valley – PI Mts.	N 48°36’37.45”	E 18°01’25.6”
PI-28	Hrabový Hill – PI Mts.	N 48°40’33.36”	E 18°01’41.0”
PI-3Z	Solisko – PI MTS.	N 48°40’48.86”	E 18°04’21.4”