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# Fluorite and related fluids in the Karkonosze granitoid pluton, SW Poland

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## ABSTRACT:

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Fluorite mineralization was studied in the Variscan granitoid Karkonosze pluton in the northern part of the Bohemian massif (Lower Silesia, Poland). Fluid inclusions in fluorite and quartz were investigated by the following methods: heating and freezing on an immersion microscope stage, spectrophotometric and electron probe analysis, calcination and water leachate. The parent fluids of fluorite were of the Na-Ca-Cl type with a low $CO_2$ content. The fluoride ions had sources in the pluton and in its host rocks. Fluid inclusion observations provide evidence of various post-formation alteration. such as refilling, partition, cracking, migration, expulsion or vacuole modification from irregular to cubic habit. A final model of fluorite origin and parent fluid evolution is presented.

**Key words:** Karkonosze pluton; Variscan granitoid; Pegmatite; Fluorite; Fluid inclusions; Inclusion alterations; Post-magmatic fluids.

## INTRODUCTION

Since ancient times fluorite has attracted attention as a precious stone; in the Roman Empire it was called ‘myrrhites’ (Plinius 1582, p. 676). In the Middle Ages it was most probably considered to be a soft variety of amethyst (Albertus Magnus 1518, pp. 49, 50, 1967 p. 74). In the 10th-11th centuries also Al-Beruni (1989, p. 167) included fluorite to amethyst (Arabic *jamast*). However, in the 15th century its use as a flux in technological processes, occurrence and mining methods were described (Agricola 1530, p. 132: ‘*lapides ignis liquescentes*’, i.e., stones liquefied by fire). It was the target of prospecting and of studies of the fluorite occurrences in Lower Silesia (now SW Poland) as well. Early information was published in the last year of the 16th century (Schvvenckfelt 1600, p. 379), and further research was summarized c. 200 years later (Kapf 1790; Kaluza 1818). In the Variscan Karkonosze pluton in Lower Silesia subsequent fluorite studies were small parts of other investigations (Klockmann 1882, pp. 380, 397; Müller 1889, p. 26; Gajda 1960; Klomínský 2018, p. 118). One article noted the absence of fluorite in pegmatite, whereas it was expected there (Rose 1842, p. 153). Concise information on fluorite in the pluton may be found in some mineralogical publications referring to this intrusive body that collected the earlier data (Traube 1888, pp. 87–91; Schneider 1894; Lis and Sylwestrzak 1986, p. 210; Sachanbiński 2005, pp. 173, 174; Kozłowski and Sachanbiński 2007, p. 164; Knapik *et al.* 2013, pp. 37, 38; Gadas *et al.* 2015, p. 75).

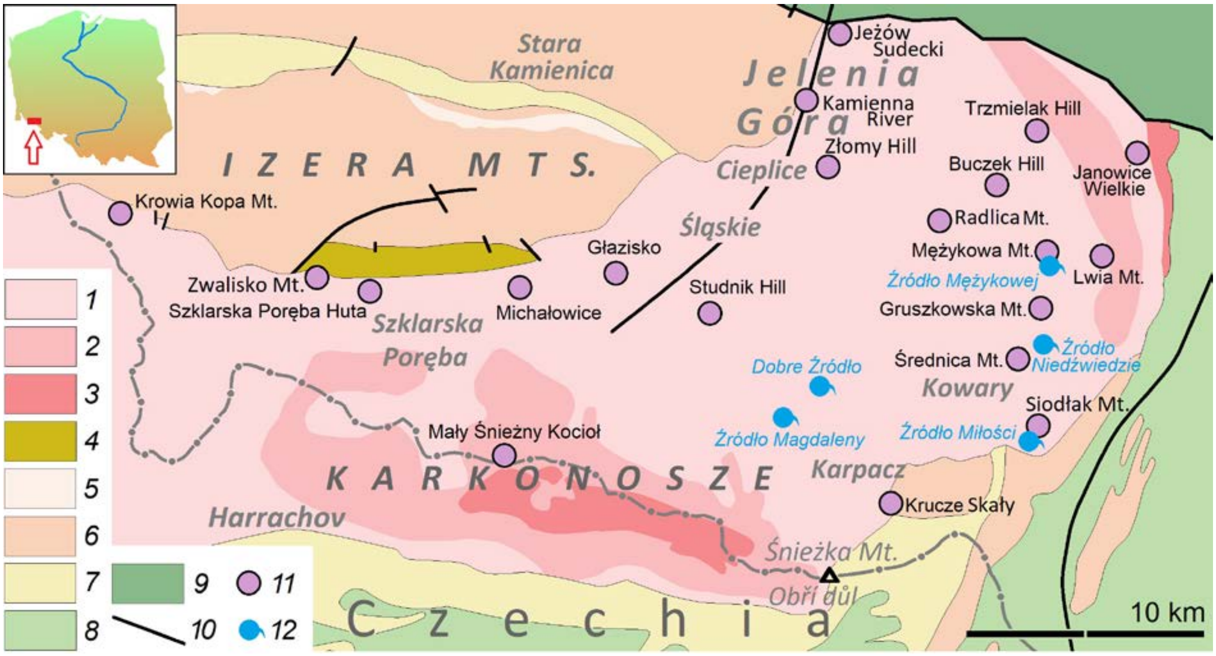
This article presents a mineralogical investigation of fluorite specimens from 20 pegmatitic occurrences in the Polish part of the Karkonosze Variscan granitoid outcrops, in part mentioned in the references cited above, but most of them found at new sites. The characteristic features of the parageneses and fluorite specimens are given, but the main attention is drawn to the crystallization conditions of this mineral and to its parent fluids. The fluid inclusion method was used in this investigation. Moreover, the data on fluorine content in post-magmatic fluids were obtained from secondary inclusions in the magmatic rock-forming quartz of the 228 granitoid samples from the whole outcrop area of the pluton, the Czech part included. The authors' and published reference analytical results of the fluorine concentrations in current thermal waters in the Polish part of the pluton were also used in the final discussion. The important problem of the secondary changes of the trapped fluid inclusions in fluorite is considered on examples found in the investigated specimens.

## GEOLOGICAL SETTING

The Lower Silesia area is geologically the prevalent part of the NE domain of the Variscan Bohemian massif (Mazur *et al.* 2018). The area formed by multistage accretion which started in the Middle Devonian and lasted till the late Carboniferous. The domain includes Neoproterozoic fragments of the Gondwana continent, lower Paleozoic granitoid intrusions metamorphosed to gneiss complexes, middle Paleozoic sediments of extensional continental margins and volcanic-sedimentary basin sequences with ophiolite series, upper Paleozoic (Carboniferous, Variscan) granitoid plutons, and intramontane basins (Mazur *et al.* 2007). The accretion-collision processes, later intrusive events and a network of faults that appeared in large part during the Alpine Orogeny, caused the blocky pattern of rock occurrence in Lower Silesia (Quenardel *et al.* 1988; Mazur *et al.* 2010).

The Karkonosze pluton is large (c. 70 km W-E, 8 to 20 km N-S); there is a good possibility of sampling, accessible contacts with the envelope rocks and the occurrence of fluorite in what is broadly understood as the exocontact zone. It was recognized as a typical example of a granite body almost 250 years ago (Gerhard 1781, p. 47). In the eastern part of the pluton, the following varieties of granitoid were distinguished: a porphyritic one with several-centimeter long orthoclase porphyrocrysts in a fine-grained quartz-feldspar-mica groundmass, a medium-grained equigranular one with similar mineral compositions, and a very fine-grained one with abundant albite, apparently aplitic (Rose 1842). Pegmatites (i.e., German *Ganggranit* – vein granite) were also identified and described (Müller 1889). Later new granite names were proposed: porphyritic – central, equigranular – ridge and fine-grained – granophyric granite (Borkowska 1966). According to the QAPF classification, the central and crest varieties consist of monzogranite and granodiorite, but the granophyric type is monzogranite exclusively (Text-fig. 1; cf. Krenz *et al.* 2001). The mineral composition is typical of such rocks: potassium feldspars (in the first variety as porphyrocrysts up to 10 cm long, frequently with zoning and Na plagioclase outer rims), Na-rich plagioclase, quartz, biotite, rare muscovite and hornblende (the latter in the first and third varieties). Melt inclusion studies in magmatic quartz (Kozłowski 2007) have indicated that the parent melt composition varied from tonalitic to granitic, the water content in the melt changed from 9 to 4 wt% and the temperature of crystallization of the earliest minerals ranged from c. 990 to c. 840ºC. It is noteworthy that these parameters refer to the beginning of crystallization from magma in this pluton and the high temperature of the intruding melt is also suggested by the thermal conditions of the formation of the northern contact calc-silicate rocks at Izerskie Garby (Fila-Wójcicka 2000a, b). The review of the considerations limiting the acid magma intruding conditions, thermal ones inclusively (e.g., Weinberg and Hasalová 2015a), have caused serious discussions (e.g., Clemens and Stevens 2015; Weinberg and Hasalová 2015b), however, they exceed the subject of this article. The tectonic and petrographic data from the Czech part of the pluton are comparable to those of the Polish part (Klomínský 1969, 2018; Chaloupský *et al.* 1989; Klomínský *et al.* 2010).

Multiphase intrusion of the Karkonosze granitoid magma was initially suggested (Cloos 1924), and confirmed by detailed studies (Žák and Klomínský 2007; Žák *et al.* 2013, 2014). The possibility emerged that the pluton formed by mixing (or mingling) of felsic and mafic magmas (Słaby and Martin 2008). U-Pb isotope measurements in zircon for the main granitoid species from the Czech part of the pluton yielded age determinations from 320 to 315 Ma (Žák *et al.* 2013). The same method applied to two granitoids from the Polish part of the pluton, equigranular and porphyritic ones, indicated their formation age of 320–312 Ma and evidenced the short formation time of the multiphase intrusion (Kryza *et al.* 2014a, b; Mikulski *et al.* 2020). At outcrops one may recognize distinct compositional, structural and textural changes of the rock over various, even short, distances. Thus, the best documented studies and the authors' field observations suggest that the magma could have formed by melting of a complex series of parent rocks and that homogenization of the melts was limited. This could have influenced variations in



Text-fig. 1. Eastern part of the Karkonosze Variscan pluton and its metamorphic envelope: *1* – porphyritic monzogranite and granodiorite; *2* – medium-grained biotite monzogranite; *3* – fine-grained monzogranite; *4* – hornfels; *5* – leucogranite; *6* – gneiss; *7* – chlorite-mica schists; *8* – mafic metamagmatites and schists; *9* – mainly mafic metamagmatic rocks; *10* – faults; *11* – fluorite sampling locations; *12* – springs. Geological background after Krenz *et al.* (2001), modified. Insert – location in Poland.

the distribution of elements, accessory minerals and the post-magmatic mineralization in the pluton rocks.

The exocontact zone in the metamorphic cover of the Karkonosze pluton consists of three parts, being metamorphic complexes: northern – Izera region (Oberc 1961), eastern – Rudawy Janowickie (Teisseyre 1971), and Southern Karkonosze (Chaloupský *et al.* 1989). The northern terrain adjacent to the granite, i.e., the Izera Mts, was early described as built of gneiss including zones of mica-chlorite schist with garnet (Kapf 1790; Buch 1798, 1802). Mica and hornblende schists occur in the eastern cover of the Karkonosze granite, i.e., Rudawy Janowickie Mts (Buch 1798, 1802). Soon after, exhaustive descriptions of granite-gneiss, gneiss, hornfels, hornblende schist and mica schist of the Karkonosze pluton cover were published (Raumer 1813).

New research showed that the northern envelope orthogneiss and granite-gneiss formed 515–480 Ma ago mostly from an S-type granite protolith (Kozłowska-Koch 1960, 1965; Smulikowski 1972; Borkowska *et al.* 1980; Żaba 1982, 1984; Żelaźniewicz 2003; Żelaźniewicz *et al.* 2003). The cover rock series includes also four latitudinal metapelite schist zones within the gneiss area. Three northern zones consist mostly of muscovite-biotite-chlorite-quartz schists with garnet, albite, chloritoid, margarite, etc., metamorphosed under greenschist facies conditions (Kozłowski K. 1973, 1974; Szałamacha and Szałamacha 1974; Szałamacha 1975; Makała 1994, 1996). The southern schist zone was altered by the thermal influence of the Karkonosze pluton to cordierite-andalusite-biotite-plagioclase-garnet hornfels (Żaba 1979; Kanasiewicz 1984; Fila-Wójcicka 2004). Close to the schist-gneiss borders metasomatites developed, namely leucogranite (Smulikowski 1958; Pawłowska 1967; Kozłowski K. 1974) and topaz, tourmaline and muscovite varieties of greisen (Karwowski 1973; Karwowski and Kozłowski 2002).

The eastern envelope metamorphic rocks form a series of overfolds or a 'pile of nappes' (Mazur 2003, 2005). The direct exocontact zone was formed by granite-gneiss and augen gneiss, moreover also by hornblende schists, cordierite hornfels, andalusite-mica schist with lenses of diopside-rich calc-silicate rock, graphite schist and dolomitic marble; it also includes diopside-bearing amphibolite and quartzite schists. Further eastwards occur mica, chlorite, amphibolite, etc., schists (Berg 1903, 1940a, b). The envelope rocks formed from the late Proterozoic to the

Silurian or maybe the Early Devonian; the maximum temperature of metamorphism exceeded 500ºC, the highest pressure varied for different metamorphic paths from c. 5 to c. 10 kbar (Mazur 2003).

The southern envelope is similar to its eastern part; however, the tectonic pattern of the cover is less complicated here. The gneiss-schist complex extends there, but low metamorphism phyllite with quartzite and muscovite schist with calc-silicate rocks, calcite and dolomitic marbles and greenstone prevail. The protoliths formed most probably from the early Proterozoic to the Silurian (Plamínek 1978; Chaloupský *et al.* 1989; Tásler 2016).

## SAMPLES AND THEIR OCCURRENCE

Pegmatites, the parent bodies of the investigated fluorite samples, are quite common in the Karkonosze pluton and occur mostly in the porphyritic granitoid. Their size varies from a cubic dm to c. 100 m$^3$ and their shape may be close to isometric, but also ellipsoid, lenticular, irregular and vein type. Potassium feldspar, oligoclase, albite, quartz, biotite and muscovite are the main components, however, numerous subordinate and accessory minerals are present. The texture of the pegmatites is usually typical, with concentric or parallel-symmetric zoning and common central miarole (Matyszczak 2018). In a general sense pegmatites in granitoids are believed to form from the remnant magmatic melt (Fersman 1940, pp. 23–32; London 1992; Thomas *et al.* 2006; Simmons 2008), but indices of their development by recrystallization were recognized early (Schaller 1925, 1926). Some Karkonosze pegmatites also show evidence that they formed by the post-magmatic accretionary recrystallization of aplite (Kozłowski 1978, 2002).

Karkonosze pegmatites are considered essentially as belonging to the niobium-yttrium-fluorine (NYF) family *sensu* Černý and Ercit (2005, see also Černý *et al.* 2012; Pieczka *et al.* 2015; Evans *et al.* 2018; Matyszczak 2018). Most may be ascribed as close to the euxenite, gadolinite, and allanite-monazite type of rare elements – rare earth elements subclass. Moreover, the gadolinite-fergusonite type of miarolitic rare earth elements subclass may be distinguished (Evans *et al.* 2018). Nevertheless, an aspect of this classification of the Karkonosze pegmatites should be explained. They have low contents of fluorine, one of the main components of the NYF family, thus they are not of the *classical* NYF type. However, as has been shown (Hanson 2016), the acronym of a family can be sometimes misleading and in such cases the geotectonic discrimination of the parent granitoid may be a useful tool in suggesting the possible family of the pegmatites. The Karkonosze granitoid is considered to be of post-collision type (Mikulski 2007) and hence it generally hosts pegmatites of the NYF type as broadly defined (Hanson 2016), despite their depletion in fluorine.

Twenty localities with fluorite in pegmatites were found by the authors in the Karkonosze pluton (Text-fig. 1) in the years 1968–2006. All occurred in the porphyritic granitoid, excepting two – one in the pegmatite in medium-grained biotite monzogranite in Mały Śnieżny Kocioł and the other in granite-gneiss at Krucze Skały. Five were found in areas already known as fluorite occurrences (Klockmann 1882; Müller 1889; Gajda 1960; Klomínský 2018). The outcrops, pegmatites and mineral associations are briefly described below.

**Buczek Hill** (15º50'19"E, 50º52'11"N), outcrop in the rock at the hill summit, a lentiform, 62×17 cm pegmatite, almost the whole lens filled by potassium feldspar, albite and gray quartz (partly granophyric ones). A c. 5 cm void was present between quartz crystals with two (2.1 and 3.8 mm) violet fluorite octahedra and small crystals of calcite, chlorite, natrolite and chabasite.

**Głazisko** (15º37'38"E, 50º49'55"N), outcrop in the rock close to the left bank of Wrzosówka creek; in the creek bed fluorite in pegmatite was known earlier as well (Kozłowski and Sachanbiński 2007). The pegmatite at Głazisko was orbicular, 18 cm in diameter, filled by feldspars and quartz with c. 1 cm space in the middle, where subhedral (cube and octahedron faces) colorless fluorite grains, 1–2 mm in size, and natrolite occurred.

**Gruszkowska Mt.** (15º52'04"E, 50º49'44"N), outcrop in the rocks to the west of the summit, vein-type pegmatite, 20–34 cm thick, with asymmetric layering (upper side almost without granophyre), completely filled by potassium feldspar and quartz with a tiny veinlet (3.4 cm long, 1–1.7 mm thick) of pale-violet fluorite with discernible subhedral cubic crystals.

**Janowice Wielkie** (15º55'33"E, 50º52'55"N), granitoid wall on the left side of Bóbr River, a group of four pegmatite nests of irregular shapes and dimensions at 11–17 cm, filled with feldspars and in part transparent quartz. One quartz crystal included a violet-colored, almost perfect 4.2 mm cube of fluorite.

**Jeżów Sudecki** (15º44'50"E, 50º55'31"N), granitoid block in the Szumiąca creek bed with an irregular pegmatite up to 24 cm in size. In the central quartz part, a subhedral green fluorite grain (3.9 mm in size) with faces of octahedron was found.

Text-fig. 2. Euhedral fluorite crystals from the Karkonosze pluton pegmatites: Krowia Kopa Mt.: 1 – cube, 2 – tetrahexahedron, 3 – dodecahedron, 4 – octahedron; Siodłak Mt.: 1 – cube, 2 – hexoctahedron; Krucze Skały: 1 – cube, 2 – octahedron, 3 – trisoctahedron, 4 – dodecahedron; Zwalisko Mt.: 1 – cube, 2 – tetrahexahedron, 3 – trapezohedron, 4 – dodecahedron; exact drawings of real crystals and true colors presented. Scale bars 1 mm.

**Kamienna River** (15º43’49”E, 50º53’56”N), outcrop in the area of Jelenia Góra city, *in situ* granitoid in the left bank of the river bed. Elongated ellipsoid (41×26 cm in size) with typical main minerals and transparent quartz in the center; natrolite, chabasite, thomsonite and two pale-violet fluorite octahedra (2.3 and 1.7 mm in size) were found between the youngest parts of the quartz crystals.

**Krowia Kopa Mt.** (15º20’30”E, 50º51’55”N), outcrop on the southern slope of the mountain, almost spherical pegmatite, 23–27 cm in size, with typical arrangement of the main minerals. The central void contained lepidolite*, epidote, pyrite, chalcopyrite, calcite, natrolite and a 6.6 mm in size, yellow euhedral fluorite crystal with faces of cube, tetrahexahedron, dodecahedron and octahedron (Text-fig. 2).

**Krucze Skały** (15º46’08”E, 50º46’07”N), a rock group in the town of Karpacz, included to this study as an exception. The rocks are Izera-type granite-gneiss and occur close to the contact with the porphyritic granitoid. A large pegmatite group formed within the granite-gneiss, possibly due to influence of the Karkonosze pluton. The sampled pegmatite was of irregular shape and linked with others, exploited in the past. This pegmatite on feldspars and quartz and in the youngest parts of them contained lepidolite*, dumortierite, epidote, chlorite, natrolite, thomsonite, calcite and violet, euhedral, 4.3 mm large crystal of fluorite with faces of cube, octahedron, trisoctahedron and dodecahedron (Text-fig. 2).

**Lwia Mt.** (15º54’28”E, 50º50’29”N), an abandoned quarry, situated c. 420 m southward from the mountain summit. The pegmatite occurred in aplite and it was a 2.3 m long and up to 34 cm thick vein, diminishing toward its ends. It had thin granophyric zones at the edges and in the central part a feldspar-quartz-muscovite filling with several, 5–7 cm long and up to 2 cm wide voids. Each contained epidote, calcite, chlorite; moreover, two of them – chabasite and one, 3.9 mm large, cubic crystal of pale green fluorite.

**Mały Śnieżny Kocioł** (15º33’09”E, 50º46’56”N), outcrop in the western wall of this Pleistocene glacier cirque in the Karkonosze Mts main ridge. Medium-grained biotite monzogranite is the parent rock of the pegmatite and it is also the wall rock of a volcanic vent and breccia zone of an Oligocene basaltoid (olivine basalt with a shift to basanite due to alkalis from contamination by the granitoid (Charpentier 1804, pp. 63–72; Daubuisson 1814, p. 235; Singer 1820; Mosch 1821, p. 217; Cloos and Korn 1934; Birkenmajer 1967; Białowolska 2000; Bakun-Czubarow and Białowolska 2002; Zagożdżon and Zagożdżon 2006). Approximately 170 m southward from the vent zone a pegmatite of irregular shape reaching 48 cm in size was found. It was massive without voids and with translucent quartz in the central part which had solid inclusions of epidote, pyrite and fluorite; the latter formed pale violet subhedral cubic crystals of dimensions from 1.2 to 3.7 mm.

**Mężykowa Mt.** (15º52’12”E, 50º50’41”N), an abandoned quarry, isometric pegmatite, 74 cm in size, with thick granophyric zone, 10–15 cm long subhedral potassium feldspar and quartz crystals with accessory hematite, rutile, allanite, epidote, muscovite and chlorite in between. Fluorite was found only in the allanite alteration products as very pale violet cubes up to 0.2 mm of the edges (Text-fig. 3).

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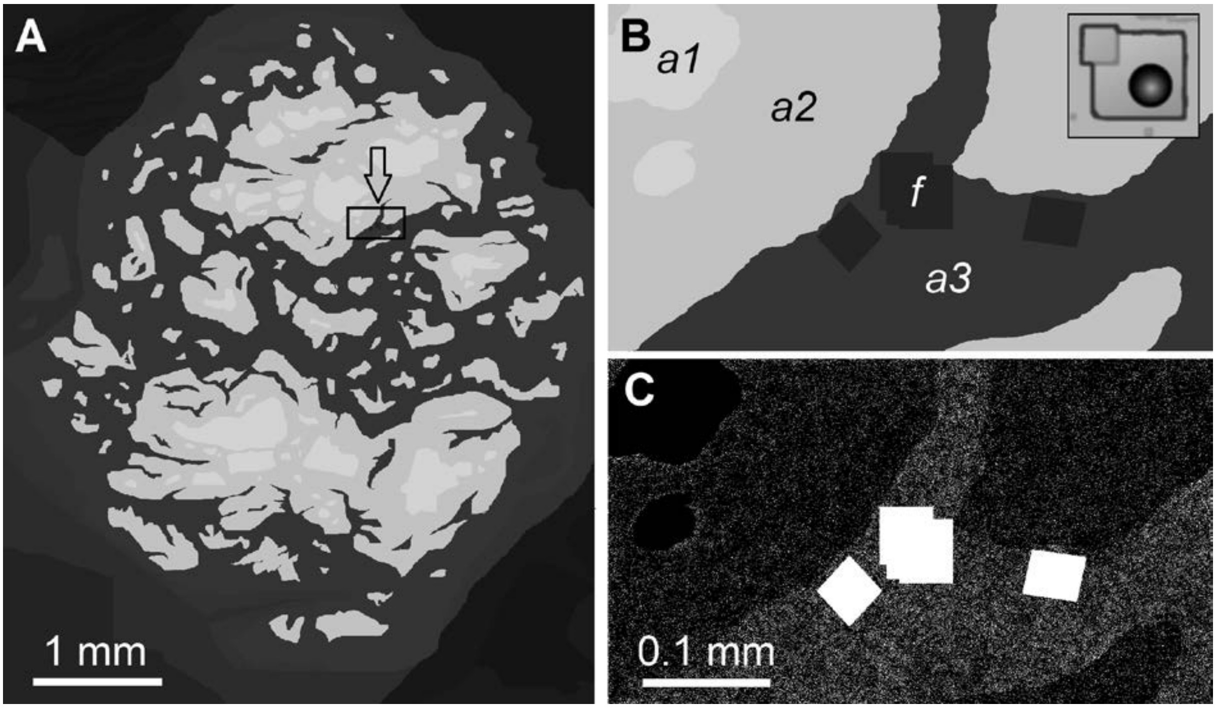
* Lepidolite composition from the pegmatite at Krowia Kopa Mt.: $SiO_2$ 43.19, $Al_2O_3$ 26.32, MnO 2.10, FeO 1.42, $Li_2O$ 6.57, $Na_2O$ 0.46, $K_2O$ 10.82, F 6.85, OH 2.18, Σ 99.91 wt% → $(K_{0.93}Na_{0.06})_{\Sigma 0.99}(Li_{1.78}Al_{1.02}Mn_{0.12}Fe_{0.08})_{\Sigma 3.00}(Al_{1.07}Si_{2.91})_{\Sigma 3.98}[F_{1.46}(OH)_{0.52}]_{\Sigma 1.98}$; authors’ EPMA determinations, for the Li analytical method see Takahashi *et al.* (2016), OH determined by the IR spectrometry. The compositions of lepidolite from the Krucze Skały and Średnica Mt. (see below) samples were similar.



Text-fig. 3. Allanite with fluorite. A – allanite grain mainly altered and metamictised, BSE image; arrow indicates the detail shown in B – relics of fresh allanite (*a1*), poorly altered (*a2*) and strongly altered (*a3*) one with crystals of fluorite (*f*), BSE image; C – same field as in B, fluorine *Kα* X-ray scan picture. Insert – optic microscope image of fluid inclusion (size 9 µm) in the largest fluorite crystal; the square in the left upper corner of the inclusion is a detail of its shape. Abandoned quarry, Mężykowa Mt.

**Michałowice** (15º34'30"E, 50º50'15"N), a working quarry when samples were collected, now abandoned. The walls show several pegmatites, usually of irregular shape and of various sizes; the sampled one was c. 1 m long with typical contents and numerous accessory minerals (Kozłowski and Dzierżanowski 2007; Kozłowski 2011; Mochnacka *et al.* 2015; Kozłowski and Matyszczak 2018, pp. 92–95), fluorite inclusive (Gajda 1960, p. 566). Small crusts of subhedral cubes (up to 8 mm in size) of violet fluorite were found on quartz. Fluid inclusions in fluorite from this quarry were already investigated (Karwowski *et al.* 1983).

**Radlica Mt.** (15º48'31"E, 50º52'02"N), rocks on the eastern slope close to the mountain summit, small (31 cm) almost spherical pegmatite with usual zoning of the filling. In the central void on quartz, albite and potassium feldspar there were small crystals of epidote, calcite, natrolite and chabasite; among them a 3.1 mm cube of blue fluorite was found.

**Siodłak Mt.** (15º52'00"E, 50º47'27"N), NW wall of an abandoned quarry on the western slope of the mountain, lentiform pegmatite, 65 cm long and up to 18 cm thick, in an aplite vein. This pegmatite had a thin and discontinuous granophyric rim and filling mainly by potassium feldspar with twice lower amount of quartz. In small voids accessory epidote, muscovite, calcite, chabasite and thomsonite were found, as well as two crystals of green fluorite (1.8 and 3.9 mm in size) with faces of cube and hexoctahedron (Text-fig. 2).

**Studnik Hill** (15º40'11"E, 50º49'32"N), exposure located c. 100 m to the southwest from the hill summit; in a rock covered by soil a pegmatite 47 cm in size and irregular shape was found. It contained a thin rim of granophyric intergrowths and typical feldspar-quartz filling. The central void (c. 16 cm dia.) was not visible, it was opened by crushing the rock. Accessory minerals included epidote, allanite, rutile, chlorite, calcite, natrolite, lévyine and chabasite; groups of violet cubes (1.8–4.1 mm in size) of fluorite occurred in this assemblage; few of its crystals were included in the late transparent quartz. Close to this pegmatite (at a distance of 100–240 m) three blocks (23–41 cm in size) of basalt with TAS parameters (Le Maître *et al.* 2002) $Na_2O + K_2O = 2.87$, $SiO_2$ 47.66 wt% (authors' data) were found. No parent basalt body could be traced there, but it is possible that they had come from a dike or more probably a sill now largely or completely eroded. This basalt blocks occurrence was known in the past (Berg 1921, pp. 38, 39, 1940c).

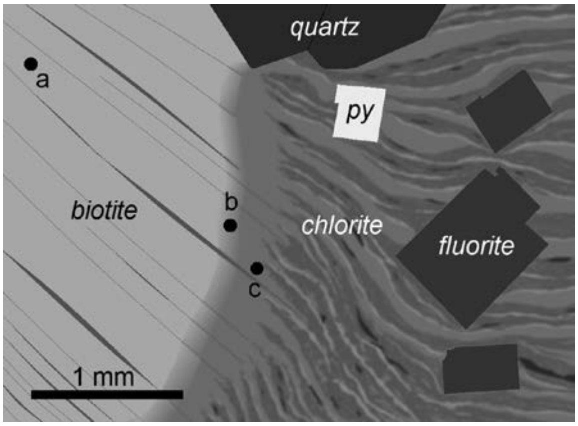
**Szklarska Poręba Huta** (15º29’36”E, 50º49’36”N), rock above the western wall edge of the quarry; half-elliptic pegmatite bordering on an aplite vein in granite with a granophyric zone only at the contact with aplite. The main minerals are: potassium feldspar, oligoclase, albite, quartz and biotite; the accessory minerals include: epidote, pyrite, tourmaline and violet fluorite (few cubes 2.9–4.6 mm in size).

**Średnica Mt.** (15º51’27”E, 48º52’47”N), eastern slope of the mountain, c. 200 m from the summit; typical ellipsoid pegmatite nest, 37 cm long, with lepidolite, epidote, chlorite, calcite and natrolite; two colorless fluorite cubes (3.6 and 4.4 mm in size) found between the rock crystals.

**Trzmielak Hill** (15º51’38”E, 50º53’11”N), southern slope of the hill, c. 50 m from the summit, edge of the railway excavation. Pegmatite (63 cm long) was composed of two irregular parts surrounded and separated by granophyric intergrowths. One part was completely filled by potassium feldspar, quartz, minor oligoclase and albite, and the second, larger one, had a central void, where aggregates of tiny grains of epidote, calcite and natrolite were found on pale smoky quartz and feldspars. A cubic, 4.7 mm in size, crystal of pale violet fluorite was included partly in quartz and its upper part was overgrown by calcite crust.

**Złomy Hill** (15º44’19”E, 50º52’51”N), quarry at the southern slope of the hill; pegmatite (58 cm in size) of irregular shape found next to biotite- and hornblende-rich schlieren, partly included into the pegmatite. Spots of granophyric intergrowths and feldspar-quartz aggregates occurred between the schlieren fragments. In these fragments biotite altered to chlorite and in the latter crystallized euhedral pyrite and yellowish fluorite cubes, up to 1 mm in size (Text-fig. 4).

**Zwalisko Mt.** (15º27’08”E, 50º50’54”N), southern slope of the mountain near the contact with hornfels; 71 cm long and to 34 cm thick lenticular pegmatite with a typical filling (feldspars, biotite, quartz) with accessory schörl (needles), epidote, allanite, topaz and euhedral blue fluorite crystal, 5.1 mm in size, with forms: cube, tetrahexahedron, trapezohedron and dodecahedron (Text-fig. 2).



Text-fig. 4. Fluorite in post-biotite chlorite, py – pyrite, BSE image. Pegmatite in porphyritic monzogranite with biotite- and hornblende-rich schlieren; fluorine content in biotite, wt%: a – 0.81, b – 0.38, c – 0.06, in chlorite – below detection limit. Złomy Hill.

Moreover, 228 samples of magmatic quartz from the whole outcrop area of the pluton (Czech and Polish parts) were analyzed for fluorine content in secondary inclusion fluids. In addition to the above material, data on the fluoride ion content in water from 5 springs in the Karkonosze granitoid were obtained (Text-fig. 1): Źródło Niedźwiedzie (Bear Spring; 15º52’01”E, 50º49’03”N) on the northwestern foot of Wilczysko Mt., Dobre Źródło (Good Spring; 15º43’46”E, 50º48’04”N) on the western slope of Grabowiec Mt., Źródło Miłości (Love Spring; 15º52’00”E, 50º47’26”N) in an abandoned quarry on Siodłak Mt., Źródło Magdaleny (Magdalena Spring; 15º43’46”E, 50º47’42”N) on the northern slope of Czoło Mt. and Źródło Mężykowej (Mężykowa Mt. Spring; 15º52’12”E, 50º50’41”N) in an abandoned quarry (the spring terminated in late 1970s).

## METHODS

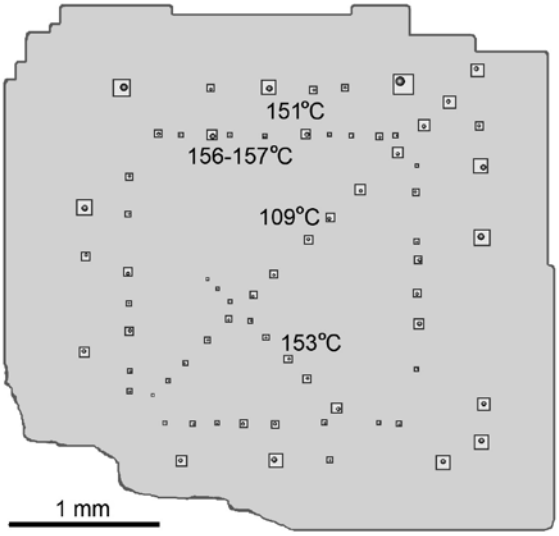
Fluorite preparations for the inclusion studies were 0.1–0.5 mm thick polished plates parallel to the cubic or octahedral faces of the mineral. Fluid inclusions were investigated mainly in polarized light, oriented perpendicularly or obliquely to the preparation, or both jointly, to find the traces of the fluid inclusion migration in crystals. Homogenization (Th) and last ice crystal melting (Tm) temperatures were measured in the usual way (Roedder 1984, pp. 181–219), but with the use of a special immersion heating-freezing stage (Karwowski *et al.* 1979). Silicone oil (boiling temperature 315ºC) was the immersion fluid for heating and ethanol (melting temperature -114.1ºC) for freezing. Accuracy of estimation for Th was ±0.5ºC and for Tm ±0.1ºC. Interpretation of the freezing measurements, i.e., determinations of the main components and total salt concentrations (ΣS) were made on the basis of physical-chemical calculations (Crawford 1981; Kozłowski 1984). Single inclusions were opened either by a microscope crushing stage (Roedder 1984, pp. 212–219) or a microscope hardness tester with diamond indenter. Solution flew out from an inclusion on the preparation surface and after water evaporation elements were determined in the precipitate. Abbreviations for the fluid inclusion description were used according to Roedder's (1984, p. 198) suggestions. Analyses of chemical composition, BSE and X-ray scan images were made by ARL-SEMQ and Cameca SX100 electron probe microanalysers. The preparations were gold-covered (for C determination), reference substances: orthoclase, dolomite, MgO, pyrite and standard spectrometer crystals were used, electron beam accelerating voltage 7–20 keV, beam current 8–12 nA, beam spot diameter 3–8 µm, count time 4–12 sec., radiation peaks Al*Kα*, F*Kα*, Fe*Kα*, K*Kα*, Mg*Kα*, S*Kα*; for carbon voltage 10 keV, beam current 300 to 400 nA, C*Kα* (Robaut *et al.* 2006). Granitoid samples for determinations of fluorine in post-magmatic fluid inclusions were crushed, quartz was separated, cleaned by washing, dried and ground to powder. Leachates from powdered quartz were made by use of warm triple distilled water and then analyzed for $F^-$ ion contents by the spectrophotometric zircon-eriochrome-cyanine and zircon-alizarine S methods (Marczenko 1968, pp. 267–279). Water amount in the inclusions was determined by the calcination method and then formal average concentration of fluorine in the fluid was calculated (Karwowski and Kozłowski 1971). Fluoride ion contents in spring waters of the pluton area were determined by the same spectrophotometric method. The ARL-SEMQ electron probe analyses were made at the University of Tübingen by AK and fluid inclusion studies, classic chemical analyses and Cameca SX100 electron probe determinations in the laboratories of the Faculty of Geology, University of Warsaw.

## RESULTS

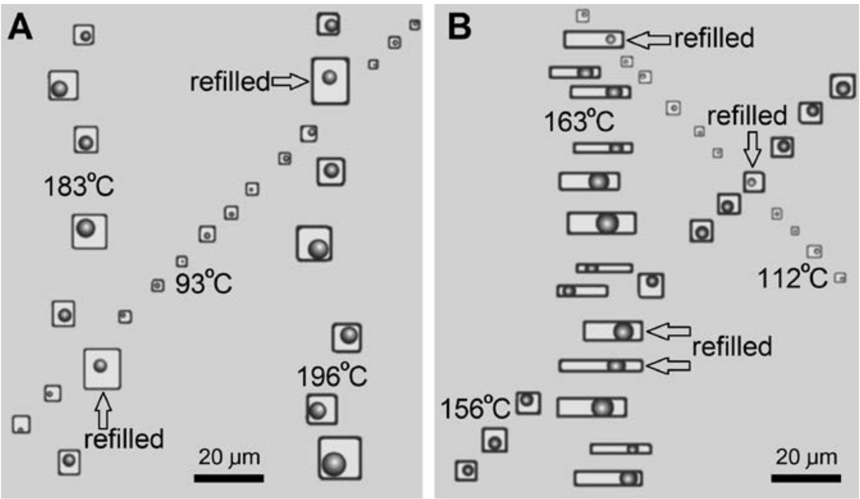
### Primary and secondary fluid inclusions

The studied fluorite crystals contained innumerous fluid inclusions; in many specimens only a few were found. The size of the inclusions was up to 20 µm but commonly achieved only 10 µm; larger ones were exceptions. Approximately more than 80% of the inclusions were of primary type, arranged distinctly in crystal growth zones. Secondary inclusions in healed cracks agreeing with the octahedral cleavage of fluorite occurred much more rarely and the crack planes were small, usually not cutting the whole crystal (Text-fig. 5). Several healed fissures were observed inside the fluorite grains, not contacting with their surfaces, as it could distinctly be seen in the grain in immersion liquid before making the preparation plates. This means that the fissure was healed before the outer zones of the crystal formed, i.e., the secondary inclusions in this case are older than the primary ones in the younger growth zones. Such secondary inclusions were named primary-secondary or pseudosecondary and the criterion for their identification was the outer zone of the crystal, precipitated from the same solution that healed the fissure (Ermakov 1971). Here is the first problem of 'the same solution', because it happened not rarely that the outer zone started to crystallize from a solution of another temperature, etc., than the healing fluid. Moreover, a preparation could be cut in such a way that it omitted the younger zone which not necessarily covered the whole crystal. This doubt was resolved by calling the secondary inclusions of high Th 'pseudosecondary' ones. None of the above approaches has a robust basis; such inclusions are simply secondary of early generation (Kalyuzhnyi 1960, pp. 8–19).

Both primary and secondary inclusions usually have a euhedral cubic habit and a two-phase (liquid



Text-fig. 5. Fluorite crystal with two distinct zones of primary inclusions (older Th 156-157ºC and younger 151ºC) and two generations of secondary inclusions (older Th 151ºC and younger 109ºC); note that the cracks that caused formation of the secondary inclusions do not cut the whole crystal; moreover, the older generation formed during crystal growth and the younger one – when the growth finished. The inclusion sizes are not to the scale of the crystal. Środnica Mt.



Text-fig. 6. Refilling of fluid inclusions in fluorite. A – two zones of primary inclusions (Th 196ºC and 183ºC) with two inclusions opened by fracture and refilled by a cooler fluid (Th 93ºC); Trzmielak Hill. B – one zone of primary inclusions (Th 163ºC) and two generations of the secondary inclusions (Th 156ºC and 112ºC), two primary inclusions refilled by the 'older secondary fluid' and one by the 'younger secondary fluid'. The formation sequence of the secondary inclusion generations is indicated by refilling of an older inclusion by the 'younger secondary fluid'; Szklarska Poręba Huta quarry.

> gas by volume) filling. This habit suggests rather slow crystallization of fluorite, thus not a rapid temperature drop. Fluorite crystallized mainly as one of the last components in the druses either on the surfaces of older minerals or only in part inside the surrounding ones. This caused rare formation of fissures and thus infrequent secondary inclusions; a good example was found in fluorite from Średnica Mt. (Text-fig. 5).

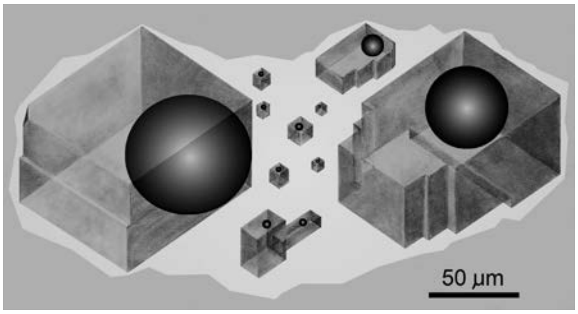
## Refilling of fluid inclusions

If an opening fissure meets a fluid inclusion (primary or secondary), it removes the original filling (solution) and replaces it by a new one (Kalyuzhnyi 1982, pp. 46–52), the same that fills the secondary inclusions forming in the healing crack.

The emptied inclusion vacuole usually has the original shape; thus comparing this feature and Th values it is easy to establish the sequence of inclusion formation, especially if the succession of secondary inclusion generations is not connected with consequent temperature decrease. On the other hand, if the distance between inclusions, especially the secondary ones, is large, a misinterpretation is possible that the refilled inclusion and the neighboring non-refilled ones point to inhomogeneity of the parent fluid. Two good examples of inclusion refilling were found in the Karkonosze pegmatites from Trzmielak Hill and Szklarska Poręba Huta (Text-fig. 6A and B).

## Recrystallization of host mineral around inclusion

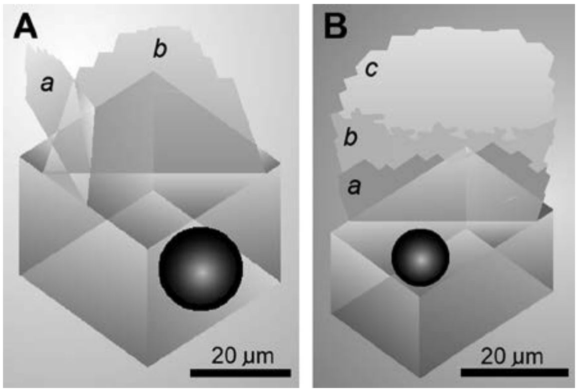
One of the fluorite crystals from Michałowice quarry included a group of fluid inclusions with variable liquid to gas proportions (Text-fig. 7) bordering on inclusions with stable proportions. This was originally a shapeless primary inclusion that probably formed when fluorite crystallized quickly due to local temperature decrease in an opening fissure. After fissure closing or filling e.g., by fluid, the temperature gradually reached the previous or almost previous value and very slow cooling of the whole rock continued. The initial habit of the inclusion vac-



Text-fig. 7. Fluid inclusion in fluorite, originally of abnormal shape and due to recrystallization divided into several daughter inclusions with vacuole faces of cube. The initial shape is outlined by the light (pale violet) part in distinctly not recrystallized darker fluorite. Note the different volume proportions of gas bubble and liquid phase in various daughter inclusions. Michałowice quarry.

uole was unstable and it began to change towards the minimum free surface energy habit, i.e., a cubic form for fluorite. However, this process is commonly connected with fragmentation of an elongated inclusion into several parts (Lemmlein 1951).

Finally, a group of inclusions was formed with different liquid to gas ratios. After the separation started, quite soon on cooling a gas bubble appeared and enlarged. The left daughter inclusion (Text-fig. 7) preserved the earliest gas bubble of the whole, undivided parent inclusion; the bubble enlarged during cooling. Other daughter inclusions have phase relationships depending on the intermediate stages of dividing and on closing or not gas bubbles in the vacuoles. The small inclusions yield Th indicating the last phase of separation. In cursory reviews such an inclusion group may give the impression of a trace of an inhomogeneous fluid.



Text-fig. 8. Cracked inclusions in fluorite crystals, heated by basaltoid melt from an adjacent volcanic vent, the cracks agree with the octahedral fluorite cleavage. A – two crossing healed cracks *a* and *b* with many fine secondary inclusions, B – one crack in part *a* not healed, in part *b* healed by fluorite almost without secondary inclusions and in part *c* healed with numerous minute secondary inclusions; individual inclusions in the healed fractures not shown in the drawing. Pegmatite in Mały Śnieżny Kocioł.

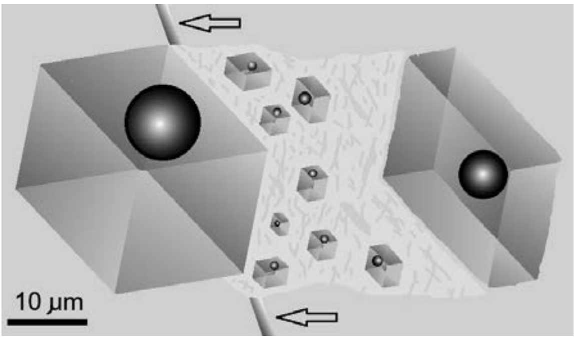
### Thermally cracked inclusions

Fluorite in the pegmatite in Mały Śnieżny Kocioł was close to a volcanic vent which caused a distinct temperature increase in its surroundings. Fluid inclusions in fluorite were strongly overheated and this was a cause of serious changes. The increasing pressure of the inclusion fillings caused cracks along the octahedral cleavage planes (Text-fig. 8). The fissures were filled by the hot solution from the inclusion, which dissolved some fluorite from the vacuole and fissure walls.

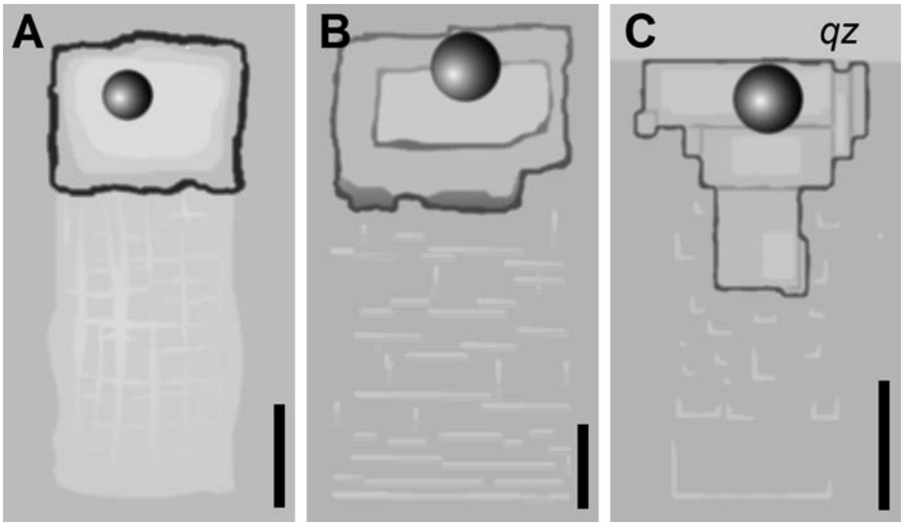
On gradual cooling the fissures were healed with the formation of numerous tiny secondary inclusions (Text-fig. 8A); such a response of fluid inclusions to overheating was observed in experiments (Lemmlein and Kliya 1954). But another cracked inclusion (Text-fig. 8B) displayed a different pattern of fissure healing. This inclusion got one crack and only the part of the fissure most distant from the inclusion (zone *c*) was typically healed with the origin of very small secondary inclusions. Closer to the overheated inclusion, zone *b* is filled by fluorite of distinct dendritic growth with few minute secondary inclusions. Fissure zone *c* closest to the parent inclusion remained unhealed. Probably the crack fissure was relatively thick and the healing, initially quick (zone *c*), later became slower and this caused full filling of the second zone *b*. However, fluorite dissolved in the inclusion fluid was used up and the temperature was already too low to cause recrystallization, and thus zone *a* remained unhealed. In this fluorite only inclusions larger than 9–10 µm cracked; the smaller ones on heating had internal stress increase insufficient to crack. Thus Th measurements were made for primary inclusions 3–6 µm in size.

### Migration of inclusions

Fluid inclusion migration in crystals caused by a thermal gradient was investigated in experiments with sodium nitrate, halite and quartz (Lemmlein 1952; Roedder and Belkin 1980; Roedder 1982; Bakker 1992). Examples of natural migration of fluid inclusions in fluorite crystals due to temperature differences were found at Studnik Hill. This is also a premise of basalt dike or sill presence (see above).



Text-fig. 9. Fluid inclusion divided into ten daughter inclusions during inclusion migration (to the right). Large (left) part of the original inclusion was blocked by a zone of structural defects in the fluorite crystal (arrows). Traces of migration visible between two marginal inclusions. Studnik Hill.



Text-fig. 10. Fluid inclusions after migration (upwards in the figure) in a fluorite crystal. The migration was probably relatively quick and resulted in the imperfect shape of the inclusions. A – migration with more or less constant speed, B – migration with variable speed, C – inclusion that reached the border of fluorite with quartz (*qz*), its shape suggests joining of two or more inclusions. Zones of various shades inside the inclusions are details of the inclusion faces, visible in oblique light. Scale bars 10 µm. Studnik Hill.

The schemes of migration are sometimes complicated by the mineral host, e.g., due to a zone of structural defects that extended in the heated crystal. In this case part of the migrating inclusion was stopped in (or almost in) the starting position and another ‘free’ part moved, leaving behind a number of small inclusions (Text-fig. 9). The migration path is marked by slightly paler color and traces, probably caused by minute structure imperfections. These traces may be visible in oblique polarized light. All inclusions shown in this image have Th 136ºC; thus the parent inclusion partition and migration of the daughters should have taken place when fillings of all inclusions were homogeneous.

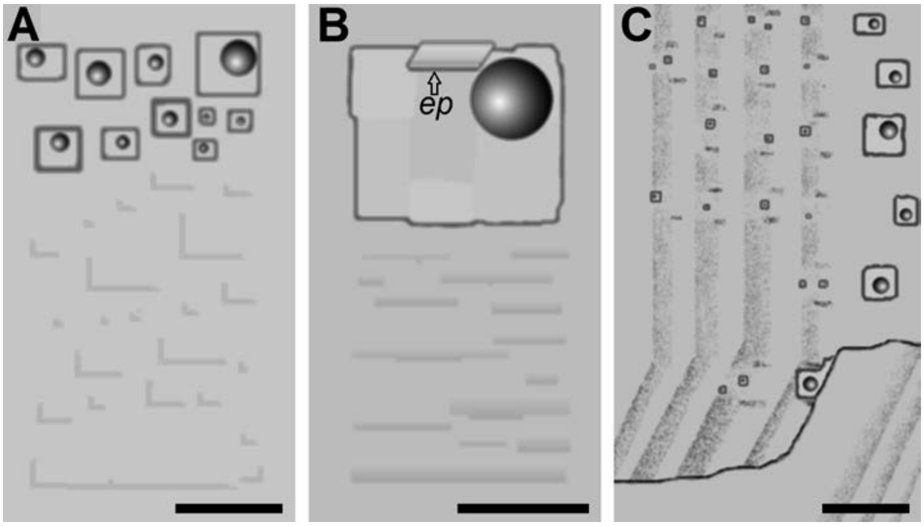
An almost ideal habit of the vacuoles of migrating inclusions is infrequent and may be a result of low speed of the movement. More frequently the inclusion vacuole has faces of complex shape (Text-fig. 10). The deformation is smallest if migration was continuous through a crystal with minute structure disturbances (Text-fig. 10A). But frequently the movement was more complicated and this influenced the final habit of the vacuole (Text-fig. 10B). When an inclusion in its movement met quartz or feldspar crystals, it stopped and as a result the next inclusions joined the first one (Text-fig. 10C).

Fragmentation of the inclusion vacuole during migration is also possible (Text-fig. 11A). The evidence that a single inclusion was at the start place may be seen as the darker gray zone that appears in oblique polarized light. Such traces above the start line indicate splitting of the inclusion. This process might have been caused by a blocky framework of the crystal.

Migrating inclusions that reached a solid inclusion may cause problems in the interpretation (Text-fig. 11B). They may be incorrectly classified as originally three-phase ones with a trapped or even daughter mineral. The illustrated inclusion stopped at the solid inclusion of epidote, but the solid phase may be completely included in the moving vacuole. Thus search for the traces of migration is very useful in this case.

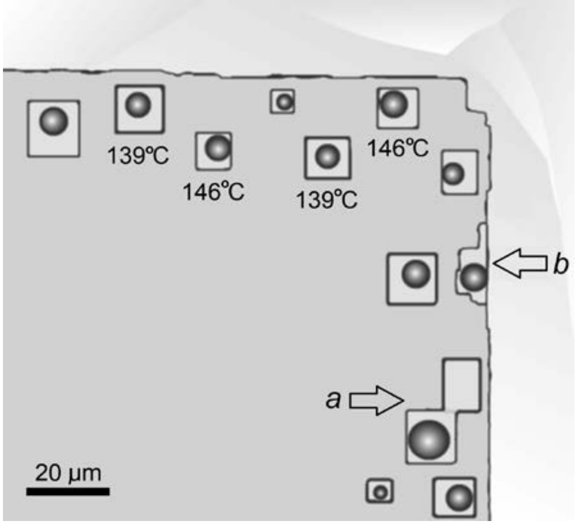
A very special example of migration of an inclusion group was found in another fluorite crystal (Text-fig. 11C). This specimen had almost regular zones of tiny solid inclusions of chlorite in the outer part of fluorite and fluid inclusions in the inner part. The moving inclusions left chlorite-free paths and ‘pushed off’ aside this mineral by recrystallization of the parent fluorite. Moreover, they left small portions of the fluid filling as tiny, 1–2 µm inclusions in the track that they went along. Probably ‘omitted’ grains of chlorite caused the formation of these very small vacuoles. One migrating inclusion met the host fluorite face and could not pass it, probably due to a different orientation of the neighboring fluorite grain.

A peculiar pattern of inclusion migration occurred in a fluorite crystal overgrown by quartz in a pegmatite at Janowice Wielkie. The crystal contained inclusions only in its outer zone, with a thickness



Text-fig. 11. Fluid inclusions after migration (A, B upwards, C to the right) in a fluorite crystal. A – migration with one inclusion dividing into several daughters probably due to structural defects of the fluorite crystal, but without leaving any inclusion at the start position. B – inclusion that during migration met a solid inclusion of epidote (*ep*), which may be incorrectly interpreted as a grain trapped in the inclusion during its formation. C – migration of fluid inclusions through zones of chlorite trapped in the fluorite during its crystallization. Migration 'traces', i.e., minute fluid inclusions were left and chlorite was removed from the movement paths. Note the change of the inclusion vacuole habit at the border of two fluorite grains. Scale bars 10 µm. Studnik Hill.

c. 1% of the host crystal size. The Th values of the inclusions were between 146ºC and 139ºC, but any sequence in the Th change could be found (Text-fig. 12). The picture shows only an example of positions of the lowest and highest Th, but intermediate results occurred as well, all in chaotic distribution.



Text-fig. 12. Part of fluorite grain in quartz with fluid inclusions gathered in the crystal outer zone as a result of crystal 'cleaning'. The inclusions migrated from various growth zones, as evidenced by different groups of Th in the same crystal volume. Some inclusions 'collided' with one another (*a*) or with the surrounding quartz (*b*). Janowice Wielkie.

Several inclusions had apparently joint vacuoles (*a* in Text-fig. 12) or were flattened on quartz at its border with fluorite (*b* in Text-fig. 12). These features tentatively could be explained by migration of fluid inclusions due to a thermal gradient. However, the same inclusion pattern was along the whole borders of the crystal, thus this reason for inclusion migration is not correct, all the more any migration traces, like the described above, were not found.

The proposed explanation is that the host fluorite was at an adequately high temperature and for a long enough time to recrystallize with cleaning most its volume, i.e., removing physical admixtures and structural defects. Thus fluid inclusions were pushed toward the fluorite borders and mixed, so they lost the genetic sequence in crystal space. The central part of the crystal was blank and homogeneous. However, the cleaning was not perfect, maybe due to the time not being long enough for expulsion of all fluid inclusions. Hence, the migration took place with complete recrystallization of almost the whole crystal, but it stopped at the final stage.

## Homogenization temperature and inclusion fluid composition

Not all inclusions in the fluorite crystals were suitable for the heating and freezing runs. In addition to the above indicated inclusion alterations, the perfect cleavage of this mineral resulted not rarely in inclusion decrepitation on heating and even on freezing. Nevertheless, the results obtained (Table 1) are representative for the conditions of fluorite crystallization in the investigated pegmatites of the Karkonosze pluton.

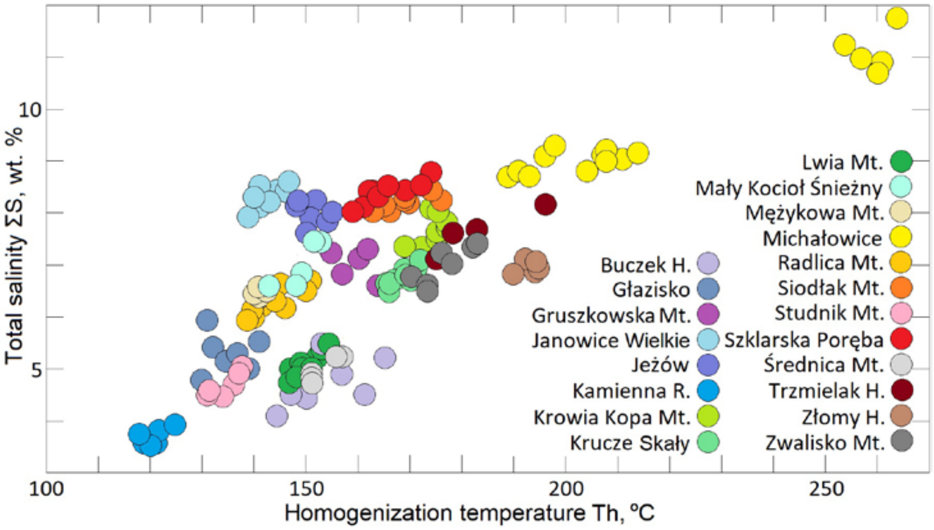
Th values for primary inclusions are in the temperature ranges 118–264ºC for all the investigated pegmatitic samples of fluorite (Table 1), but mostly they are lower (118–214ºC). The changes of inclusions Th in individual outcrops are on a small scale, the difference between the highest and lowest Th is from a few to 75ºC (Text-fig. 13). The latter concerns the considerable fluorite occurrence in Michałowice quarry with two distinct ‘sub-ranges’ of Th: 189–214ºC and 254–264ºC. If one omits it, the largest range is 21ºC (Trzmielak Hill). This indicates that fluorite crystallization was generally a short late episode in mineral crystallization in the pegmatites; however, the mineral gives significant information on the fluorine geochemical behavior in the Karkonosze pluton (see Discussion).

All primary inclusions in fluorite homogenized in the liquid phase. Also other minerals paragenetic with fluorite, namely quartz (rock crystal and pale smoky varieties), calcite and epidote contained inclusions of liquid; those homogenizing in a gas phase were not found. Moreover, secondary inclusions in fluorite were of the same kind; their Th values ranged from 89 to 161ºC.

The total salinity of the inclusion fluids in fluorite was low to moderate, from 3.5 to 11.8 wt%, and each sample had a very narrow range of salinity; only for fluorite from Michałowice it was more variable (Table 1). A positive correlation of homogenization temperature and total salinity is generally distinct, but inclusions from individual fluorite specimens may rather occupy compact fields than to be arranged along a line (Text-fig. 13). Sodium chloride and calcium chloride were the main salts dissolved in

| Fluorite occurrence | *N* | Th °C | ΣS wt. % | NaCl wt. % | $CaCl_2$ wt. % |
|---|---|---|---|---|---|
| Buczek Hill | 7 | 144–161 | 4.1–5.5 | 2.0–2.5 | 2.0–3.3 |
| Głazisko | 7 | 130–141 | 4.8–5.9 | 2.0–2.4 | 2.9–3.6 |
| Gruszkowska Mt. | 5 | 155–164 | 6.6–7.3 | 3.2–4.0 | 3.0–3.6 |
| Janowice Wielkie | 5 | 139–146 | 7.9–8.5 | 3.9–4.2 | 3.9–4.5 |
| Jeżów Sudecki | 7 | 148–155 | 7.6–8.2 | 3.5–3.9 | 4.0–4.5 |
| Kamienna River | 6 | 118–125 | 3.5–3.9 | 1.7–1.9 | 1.7–2.1 |
| Krowia Kopa Mt. | 7 | 169–177 | 7.3–8.1 | 3.4–3.9 | 3.7–4.2 |
| Krucze Skały | 9 | 165–172 | 6.5–7.1 | 2.9–3.6 | 3.2–3.8 |
| Lwia Mt. | 9 | 147–154 | 4.8–5.5 | 2.2–2.8 | 2.4–2.9 |
| Mały Śnieżny Kocioł | 5 | 143-153 | 6.6–7.4 | 3.1–3.7 | 3.4–3.8 |
| Mężykowa Mt. | 4 | 140-141 | 6.3–6.4 | 2.9–3.1 | 3.3–3.4 |
| Michałowice | 16 | 189–264 | 8.7–11.8 | 5.4–7.8 | 3.1–4.0 |
| Radlica Mt. | 9 | 139–151 | 5.9–6.7 | 2.5–3.3 | 2.2–3.7 |
| Siodłak Mt. | 8 | 163–176 | 8.0–8.4 | 4.1–4.6 | 3.6–4.0 |
| Studnik Hill | 6 | 131–137 | 4.5–5.0 | 2.1–2.4 | 2.3–2.6 |
| Szklarska Poręba Huta | 8 | 159–172 | 8.0–8.8 | 3.1–4.0 | 4.4-5.0 |
| Średnica Mt. | 6 | 151–157 | 5.0–5.2 | 2.8–2.9 | 2.1–2.3 |
| Trzmielak Hill | 4 | 175–196 | 7.1–8.2 | 3.3–4.0 | 3.8–4.3 |
| Złomy Hill | 5 | 191–194 | 6.8–7.1 | 3.7–4.1 | 2.8–3.1 |
| Zwalisko Mt. | 7 | 173–185 | 6.5–7.4 | 3.1–3.5 | 3.4–4.0 |

Table 1. Ranges of homogenization temperature (Th), total salinity (ΣS) and concentrations of NaCl and $CaCl_2$ in primary inclusion fluids in fluorite; *N* – number of inclusions.



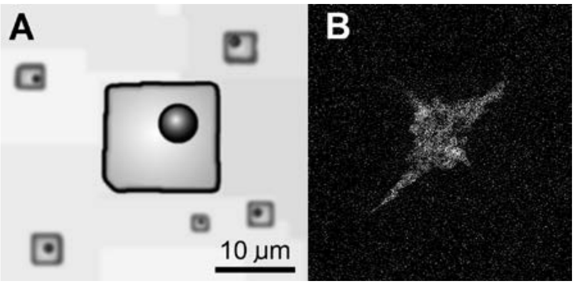
Text-fig. 13. Plot of homogenization temperature *vs*. total salinity of primary fluid inclusions in fluorite from the Karkonosze pluton pegmatites; each circle marks one Th value, see Table 1.

inclusion solutions (Table 1). This feature concerns both primary and secondary inclusions. The NaCl concentration varies from 2.0 to 7.8 wt% and that of $CaCl_2$ from 1.7 to 5.0 wt%. In inclusions of some samples the NaCl concentration is higher, but in others $CaCl_2$ prevails. This kind of hydrothermal solution was recognized earlier as typical of the late stage of post-magmatic mineralization in the Karkonosze pluton (Kozłowski and Marcinowska 2007).

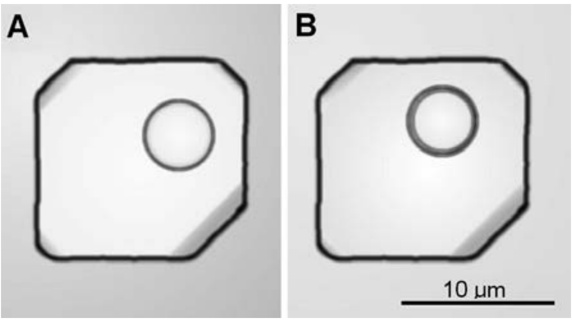
Several other elements were found in inclusion fluids released from vacuoles by indentation. These determinations were made in one or two inclusions in each sample. Magnesium (Text-fig. 14) and potassium occurred in all tested inclusions. Iron in 7 samples was below detection limit or doubtful, thus Fe ions were found in the fluid in inclusions of 13 samples. Aluminum in 4 samples was not detectable and in 7 it probably occurred, consequently its presence was sure in only 11 samples. Sulfur in 3 samples could not be detected and in 4 other it was very close to the detection limit; hence in 13 samples it was a detectable component. Carbon was present in inclusions in all samples (X-ray C*Kα* scan images) probably as the carbonate or bicarbonate ion, but the separate phase of liquid $CO_2$ was found in 4 inclusions (samples: Krowia Kopa, Krucze Skały and Zwalisko) after preparations cooling to +2°C (Text-fig. 15). The roughly estimated concentrations of the above named elements are in the range of a few tenths of percent by weight.

For a better understanding of fluorine behavior in post-magmatic fluids its content was determined in secondary fluid inclusions in magmatic quartz of the Karkonosze granitoids. Fluorine presence was checked by the F*Kα* X-ray scan image (Text-fig. 16). The fluorine concentration in inclusion solution is an average value of all generations in a given sample. The data obtained were divided into four classes: <0.005 (i.e., below the analytical determination limit), 0.005–0.009, 0.010–0.049 and 0.05–0.10 wt%. More precise values are not useful for interpretation, because one quartz sample contained up to five generations of secondary fluid inclusions. Thus the fluorine content is treated as a local geochemical feature and the data are not listed in a table but are shown on the geological map (Text-fig. 17) and in the sample amount *vs*. F content classes plot (Text-fig. 18).

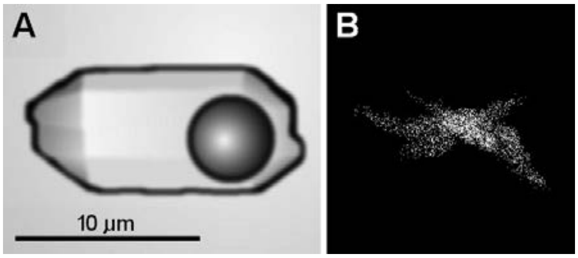
In the majority of the 228 investigated quartz samples, the inclusion fluids contained fluorine below the analytical determination limit (<0.005 wt%). Only in 48 samples, i.e., in 21%, higher concentrations were found. They fell in the ranges of 0.005–0.10 wt%, most frequently close to the lower limit (Text-fig. 18).



Text-fig. 14. Fluid inclusion in fluorite. A – optical microscope image, B – same field, inclusion was opened by point indentation and inclusion solution leaked and dried on the preparation surface; magnesium *Kα* X-ray scan picture of the precipitate, the Mg-rich area outline refers to the octahedral cleavage of fluorite. Note the scarce presence of Mg in host fluorite. Radlica Mt.

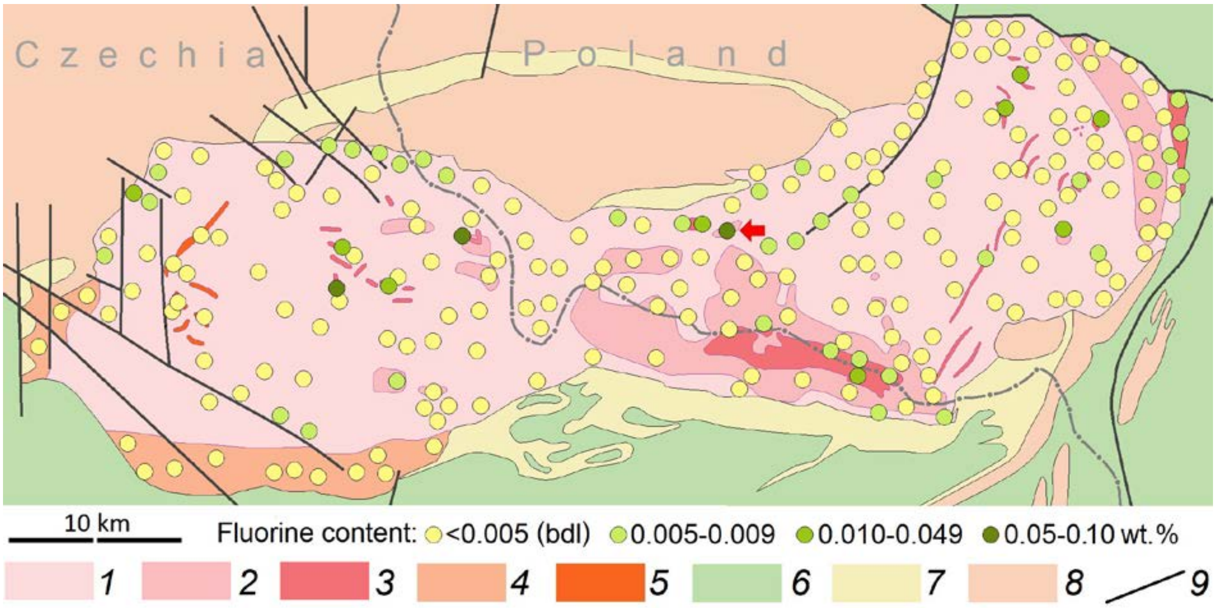


Text-fig. 15. Inclusion with $CO_2$ in fluorite at room temperature (A) and cooled to +2°C (B), thin rim of liquid carbon dioxide around gas bubble is visible. Zwalisko Mt.

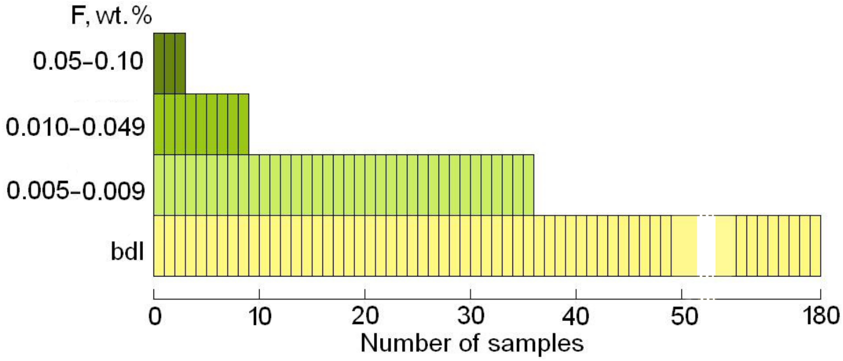


Text-fig. 16. Secondary gas-liquid inclusion in a grain of magmatic quartz in porphyritic monzogranite. A – optic microscope image, B – same field, inclusion was opened by point indentation and inclusion solution leaked and dried on the preparation surface; fluorine *Kα* X-ray scan picture of the precipitate, the outline of the fluorine-present area refers to the irregular fracture of quartz. Michałowice quarry (see map in Text-fig. 17).

The highest fluorine contents in these inclusions were found in samples taken close to the pluton borders. Other zones with fluorine-rich post-magmatic fluids extended along certain faults and close to the contact zones of different varieties of the pluton granitoids, also those forming vein-type intragranitic



Text-fig. 17. Fluorine contents in secondary fluid inclusions in rock-forming quartz of the whole outcrop area of the Karkonosze granitoids; *1* – porphyritic monzogranite and granodiorite; *2* – medium-grained biotite monzogranite; *3* – fine-grained monzogranite; *4* – biotite-muscovite monzogranite; *5* – biotite-amphibole granodiorite; *6* – mafic metamagmatites and schists; *7* – chlorite-mica schists; *8* – gneiss; *9* – faults; bdl – below analytical determination limit; arrow points to Michałowice quarry, where a granitoid sample was taken to check fluorine presence in a single secondary fluid inclusion in magmatic quartz (Text-fig. 16). Geological background after Krenz *et al.* (2001), changed.



Text-fig. 18. Number of samples in fluorine concentration classes for secondary inclusions fluids in magmatic quartz of the Karkonosze granitoids, see Text-fig. 17; bdl – below analytical determination limit of 0.005 wt%.

bodies (Text-fig. 17). This may be explained by the accumulation of fluorine along the discontinuities in the pluton. A possible inflow of fluorine-enriched fluids from the envelope rocks of the pluton may be another interpretation.

Fluorine is a minor element in parental magmas of granitoids and is either bound in minerals, mainly in granitoid biotite, or becomes a component of post-magmatic fluids. Biotite from the granitoid of the Michałowice quarry in the Polish part of the pluton was described as containing only 0.02 wt% fluorine (Borkowska 1966, p. 97); this value recalculated to the concentration in magmatic melt gave 0.0012 wt% F. However, we have obtained different data (Table 2).

Thus, the fluorine content in the melt probably varied roughly from 0.04 to 0.08 wt% (400–800 ppm) if account is taken of the amount of this element only in biotite, because the presence of other F-containing minerals (e.g., apatite) in the Karkonosze granitoids is low and may be neglected in this case. The above values for the Karkonosze granitoids, when compared with the data for other rocks of this type

| Granitoid sample occurrence | Biotite content vol. % | F in biotite wt. % | F in granitoid* wt. % |
|---|---|---|---|
| Głazisko | 5.8 | 0.54 | 0.038 |
| Janowice Wielkie | 8.3 | 0.77 | 0.079 |
| Jeżów Sudecki | 6.9 | 0.49 | 0.041 |
| Mężykowa Mt. | 5.5 | 0.62 | 0.041 |
| Michałowice quarry | 7.1 | 0.76 | 0.065 |
| Siodłak Mt. | 6.7 | 0.68 | 0.055 |
| Złomy Hill | 6.3 | 0.85 | 0.064 |
| Zwalisko Mt. | 8.2 | 0.83 | 0.082 |

Table 2. Biotite contents in the Polish part of the Karkonosze granitoid and calculated fluorine concentrations in the parent melt (personal data). * accepted as approximate content in parent melt.

(Bailey 1977), suggest that F was present in the melt of the Karkonosze intrusion in rather moderate concentrations, in the ranges determined for the I-type granitoids of mean value 540 ppm (Wang *et al.* 2018). Nevertheless, the melt could be a source of fluorine in post-magmatic fluids as well.

## DISCUSSION

Granitoids of the Czech part of the Karkonosze pluton have 6.6–10.1 vol% biotite, containing 0.31–2.19 wt% fluorine (Klominský 1969, pp. 22–25). This suggests concentrations of fluorine in the magmatic melt of 0.05–0.09 wt%. However, biotite with the highest fluorine content occurs in biotite-muscovite monzogranite and biotite-amphibole granodiorite, but these granitoid varieties are absent in the Polish part of the pluton (Text-fig. 17). The fluorine amount in biotite from other granitoid varieties is similar to that from the Polish part of the massif. Also fluorine concentrations in the early post-magmatic fluids display a very similar pattern for the whole pluton and low values strongly prevail. This results in rare and scarce occurrences of fluorite (and other fluorine-containing post-magmatic minerals) within the Karkonosze granitoids. Any even moderate-scale accumulation of such minerals has not yet been found in these rocks.

On the other hand, fluorine minerals occur in the rocks of the direct to relatively distant envelope of the pluton (Fiedler 1863, pp. 80, 81; Schneider 1894; Chojecka 1980). In the southern exocontact zone two deposits with fluorite mineralization were exploited: Obří Důl (Šita and Bradna 1967; Tásler 2012) and Harrachov (Šita and Bradna 1967; Reichmann 1968, 1982; Sejkora and Řídkošil 1997). Also the eastern exocontact rocks contain vein-type fluorite mineralization, e.g., at Kowary, Miedzianka or Ciechanowice (Kapf 1790; Kaluza 1818; Schweitzer 1846, p. 56; Effnert 1882; Berg 1903, pp. 62, 68). Fluorine minerals are peculiarly frequent in the northern envelope of the pluton. Fluorite occurs in metamorphic rocks in a vein at Jeżów Sudecki (Szałamacha 1976; Sroga *et al.* 2018), in skarns at Garby Izerskie in Stanisław quarry (Kozłowski 1978; Fila-Wójcicka 2000a, b), and farther to the north in gneiss, leucogranite or greisen as metasomatites, nests and veinlets (Kozłowski *et al.* 1996, 1997a, b; Ilnicki *et al.* 1997; Mystkowska-Mazur 2000). Topaz, tourmaline and muscovite greisens formed a latitudinal zone, c. 11 km north of the pluton contact (Budkiewicz 1949; Kozłowska 1956; Pawłowska 1966; Karwowski 1973; Karwowski and Kozłowski 2002; Klomínský 2018, pp. 107–111).

Hence, the poor in fluorine Karkonosze pluton is surrounded by rock complexes with mineralization zones relatively abundant in this element. In the Karkonosze pluton the formation of granitoids left a very low fluorine concentration in the post-magmatic fluids (Text-fig. 17). In many samples of magmatic quartz, fluorine both detectable or not detectable in leachates from secondary inclusions, was recognized only in very low amounts in single opened inclusions (Text-fig. 16). This confirms that biotite consumed most of this element from the parent melt. But this mineral may be considered as a source of 'magmatic' fluorine released to hydrothermal fluids during its alteration to chlorite. This process developed probably as a late one at relatively low temperature, as suggested by the sample from Złomy Hill (Text-fig. 4). In this sample magmatic biotite had 0.81 wt% F and progressive alteration caused a lower F content (0.38, next 0.06 wt%) finally to F-free chlorite. The released fluorine formed fluorite crystals with fluid inclusions of Th 191–194ºC (Table 1). This is also an indication of the temperature of the origin of post-biotite chlorite in this paragenesis.

Intruding magma caused heating of the envelope rocks, and dissolution and migration of some of their components. During crystallization of magmatic minerals the disappearing melt left water, originally dissolved in it, as a post-magmatic solution, in part migrating into the envelope rocks. A model was proposed for the northern envelope of the pluton, in which the fluids coming from the crystallizing magma entered the rocks of the metamorphic envelope and mixed in various proportions with the local fluids, becoming parent solutions of various minerals, fluorine-bearing ones like fluorite, muscovite and topaz inclusively (Wiszniewska *et al.* 1998). But on cooling of the intrusion (and due to tectonic phenomena) fractures developed in the young pluton, especially at c. 600ºC after the high-to-low temperature phase transition of quartz. This change causes instant decrease of linear dimensions of the quartz grains by 0.45%. In granitoids containing c. 30 vol% of this mineral (Borkowska 1966; Klominský 1969) the mechanical tension is significant, resulting in rock cracking and sucking of fluids into fractures from intergranular films in the granitoid, but also from rocks of the envelope. The network of cracks and faults in the Karkonosze pluton is fairly dense (Klominský 1969, pl. 22) and fluorite occurrence locations (Text-fig. 1) as well as places of increased F contents in early fluids (Text-fig. 17) suggest that some fluorine inflow from the envelope area is very probable. Later fluids that formed quartz veins in the Karkonosze pluton were also fluorine-poor; low fluorine contents were typical of the parental fluids of quartz veins in the Izera gneiss as well. High fluorine contents were found in the fluid inclusions in vein quartz from metasomatites of the Izera area (Kozłowski 1978).

Calcium is the second element necessary for fluorite crystallization. It could be a minor component of early post-magmatic fluid or came by solution inflow from the envelope rocks. Moreover, the quite common in the pluton alteration of oligoclase or andesine to albite by diffusive exchange of Ca for Na (Nowakowski 1976; Nowakowski and Kozłowski 1981) in the plagioclase structure added a significant amount of calcium to the fluid. Fluorine from hydrothermal fluids was also active in the decomposition of calcium minerals, e.g., allanite, especially in connection with its metamictisation. A good example was found in pegmatite at Mężykowa Mt. (Text-fig. 3) with fluorite crystals in metamictised allanite; fluid inclusions in this fluorite had Th 140–141ºC (Table 1).

Sodium was another main cation of the hydrothermal fluids, with concentrations roughly similar to those of calcium (Table 1). Potassium, magnesium, aluminum, iron and sulfur were commonly detected, but their amounts were rather low. The chloride anion was the main one, but lower concentrations of carbonate or bicarbonate anions were present in all checked inclusions. However, carbon dioxide as a separate fluid phase was detected rarely and only at low temperatures (Text-fig. 15). A group of mineral water springs of the Na-Cl type with abundant $CO_2$ was recently found in the Czech part of the Karkonosze pluton near Albrechtice (Goliáš *et al.* 2014); it confirms the possible presence of this solution in the granitoids. Generally, the present-day mineral waters cannot be directly compared with the past hydrotherms; nevertheless they occur in a very similar environment.

Temperatures of the present mineral waters are distinctly higher in the eastern (Polish) part of the Karkonosze pluton than in the western (Czech) part (Klominský 2006). They have been known as therapeutic remedies for hundreds of years (Schwenckfeldt 1607; Zimmermann 1786). Their temperature is from a few degrees to 87.5ºC (Fistek and Fistek 2005). Though the composition of the dissolved components is variable, all the analyses known to the authors indicate the presence of the fluoride anion, e.g., in Cieplice Śląskie at c. 3.5–13 mg/L, and in the Karpniki area at 11.4–16.0 mg/L (Falkiewicz and Starzewska 1975, p. 44; Fistek and Fistek 2005; Liber-Makowska and Łukaczyński 2016). The authors' determinations of fluoride ion concentrations in waters from springs in the Karkonosze granitoid were as follows: Źródło Niedźwiedzie 6.1 mg/L, Dobre Źródło 3.0–12.1 mg/L, Źródło Miłości 4.2–11.9 mg/L, Źródło Magdaleny 8.4 mg/L, and Źródło Mężykowej 5.5–12.3 mg/L. The Karkonosze springs are considered as descension-ascension type connected with the uppermost Karkonosze ridge (Januszewski and Koszarski 1979, pp. 171, 172). Thus water from rain or snow flowing down by fissures in the granitoid extracts fluorine from minerals, probably from biotite, repeating in some sense the earlier hydrothermal phenomena.

Fluid inclusions in the investigated fluorite specimens had peculiar features. They usually were not abundant in the host crystals and specific early secondary inclusions were found (Text-fig. 5). Without careful observations of the whole grain it was possible to misinterpret such inclusions as formed from an inhomogeneous fluid. Also refilling of inclusions of various generations was found (Text-fig. 6); in this case the same erroneous (or another) interpretation is possible. Originally large inclusions, especially with elongated and irregular vacuole shapes, may change their habit to cubic by recrystallization of the host fluorite. This is usually connected to the partition of the large inclusion to the daughters with various liquid to gas proportions (Text-fig. 7); thus a mistake in genetic interpretation is possible.

Fluid inclusions in fluorite from the Karkonosze pluton gave only homogenization temperatures, which for primary inclusions were in the range of 118–264ºC (Table 1). Unfortunately, determination of the pressure of the fluids was not possible. Liquid carbon dioxide was found in very low amounts and only at low temperatures (Text-fig. 15). On the basis of general conclusions from investigations of the hydrothermal environment in the pluton (Kozłowski and Marcinowska 2007) the pressure may be inferred to be c. 0.6 kbar. The probable correction to Th determinations should be 40–50ºC.

## CONCLUSIONS

The parental magma of the Karkonosze granitoid intrusion was fluorine poor and in the rocks most of this element concentrated in biotite. The early post-magmatic fluid, preserved in secondary inclusions in magmatic quartz has low to very low concentrations of fluorine.

Rock complexes of the metamorphic envelope include fluorite veins and metasomatites, which were in part sources of F-containing fluids that migrated into the pluton. These fluids and decomposed parts of biotite supplied fluorine for fluorite crystallization.

Parental solutions of fluorite were of epi- to late mesothermal stage with the composition of Na-Ca-Cl type and low although common admixtures of K, Mg, Fe, Al, S and $CO_2$. Temperature of fluorite formation was not lower than 118–264°C in various outcrops, as indicated by Th values of fluid inclusions.

Part of the fluid inclusions in the investigated fluorite is a good example of several types of post-formation changes, such as inclusion refilling, partition, cracking, migration, expulsion or vacuole modification from irregular to cubic habit. Inclusions in fluorite in two outcrops were affected by high temperature of a basaltoid vent and sill that intruded the granitoid. The inclusion alterations require thorough observations and very careful interpretation during the investigations.

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