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GEOCHEMISTRY

Immiscibility of Calcium Fluoride and Aluminosilicate Melts in Ongonite from the Ary-Bulak Intrusion, Eastern Transbaikal Region

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The addition of fluorides of Na, K, Ca, and other elements into silicate melts lowers the degree of their homogeneity owing to the formation of sybotaxic groups enriched in fluorine and cations-modifiers [1]. Many fluoride–silicate systems are characterized by a microheteregoneous structure in the hyperliquidus region related to immiscibility [2]. Under certain conditions, the evolution of natural magmatic systems gives rise to the formation of fluoride melts. Such examples are not numerous and mostly pertain to the effects of immiscibility in sodic fluoride melts (melts–brines) associated with peralkaline granitic magmas. The existence of calcium fluoride melts is confirmed by inclusions of fluoritic glass in mantle xenoliths captured by alkali basalt in New Zealand [3]. As was suggested previously, the residual glasses in ongonite from the Ary-Bulak intrusion were formed as a result of quenching of a calcium alumino-silicofluoride melt consisting of Ca, F, Al, and Si with an admixture of Na and K [4]. In this communication, we present new data on the active participation of calcium fluoride melts in crystallization of this rock.

Ongonite is a subvolcanic analogue of rare metal Li–F granite [4]. In contrast to substantially sodic ongonites from Mongolia, all ongonite varieties of the Ary-Bulak intrusion (31 samples, according to original and literature data) reveal the prevalence of K over Na. The intrusion is exposed as a domelike stock (0.8 km^2) among the Devonian volcanosedimentary rocks [4, 5]. The stock is composed largely of porphyritic ongonite. The aphyric rocks are known only in the marginal zone 50–100 m wide at the southwestern flank. Beyond this zone, porphyritic rocks with anomalously high Ca and F contents (Table 1, samples ARB-26, ARB-24, ARB-15) occur among common ongonites containing 0.2–0.7 wt % CaO. The high contents of CaO (7.8–18 wt %) and F (7.1–15.5 wt %) are inherent to all studied aphyric rocks. The Ca-rich porphyritic and aphyric rocks reveal a positive correlation between CaO and F (Fig. 1).

Ongonite porphyries contain up to 20 vol % phenocrysts of water-transparent sanidine, albite with K-feldspar rims, perfectly faced dipyramids of smoky quartz, and sporadic translucent topaz crystals with acicular wolframoixiolite inclusions. Occasional laths of dark mica (biotite–zinnwaldite series) contain 6–10 wt % F. The groundmass consists of uniformly distributed isometric quartz grains (20–30 μm) and tabular albite crystals (30–80 μm) rimmed with K-feldspar (5–20 μm). Aggregates of acicular topaz are identified in microporous spots of the groundmass, while microlites of quartz

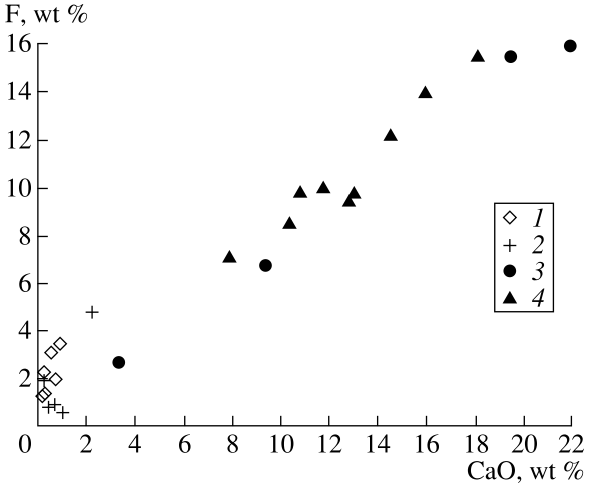

Fig. 1. F vs. CaO in rocks of the Ary-Bulak intrusion. (*1, 2*) Porphyritic ongonite (original and literature data, respectively); (*3*) porphyritic rock with “fluoritic” phase; (*4*) aphyric rocks with “fluoritic” phase and prosopite.

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Table 1. Chemical compositions of ongonites and their calcium variety in the Ary-Bulak intrusion, wt %

Component	1			2			3		
	ARB-28	ARB-23	ARB-20	ARB-26	ARB-24	ARB-15	ARB-4	ARB-8	ARB-19
SiO_2	72.79	71.48	67.83	70.02	62.60	50.63	57.24	56.25	51.99
TiO_2	0.02	<0.02	<0.02	0.05	<0.02	0.07	<0.02	<0.02	<0.02
Al_2O_3	15.69	15.53	17.83	14.93	14.20	12.97	16.29	16.52	17.81
Fe_2O_3	0.61	0.65	<0.10	0.60	<0.10	0.03	0.18	0.35	0.26
FeO	0.09	0.27	0.65	0.17	0.68	0.58	–	–	–
MnO	0.05	0.05	0.03	0.04	0.04	<0.03	0.03	<0.03	<0.03
MgO	0.03	0.31	0.79	0.03	0.09	0.18	0.14	0.19	0.34
CaO	0.19	0.75	0.93	3.31	9.26	19.38	13.01	11.69	14.46
Na_2O	3.79	3.85	2.80	3.57	3.15	1.11	0.38	0.56	0.53
K_2O	4.62	4.74	5.04	4.37	4.04	4.33	3.73	5.33	3.23
Li_2O	0.11	0.11	0.08	0.11	0.08	0.023	0.03	0.03	0.04
Rb_2O	0.22	0.23	0.39	0.19	0.16	0.30	0.31	0.37	0.26
Cs_2O	0.007	0.020	0.024	0.005	0.013	0.010	0.014	0.015	0.014
P_2O_5	0.03	0.02	0.03	0.02	0.05	0.14	0.06	0.06	0.05
F	1.22	2.00	3.50	2.70	6.80	15.50	9.85	10.00	12.25
$H_2O^{\pm}$	1.05	0.86	1.55	1.02	1.46	1.21	2.45	2.03	3.88
CO_2	–	–	0.05	–	0.22	–	0.17	0.22	0.11
Total	100.00	100.03	100.05	100.00	99.98	99.94	99.74	99.40	100.07
Ca-Fl, %	tr.	<1	<1.3	4.6	12.9	27.0	14.8–15.6	11.4–11.7	10.3–12.4
Pr, %	0	0	0	0	0	0	5.9–8.0	12.6–13.4	21.7–26.4

Note: (1) Porphyritic ongonite; (2) porphyritic rocks with “fluoritic” phase; (3) aphyric rocks with “fluoritic” phase and prosopite. Analyst G.A. Pogudina, Institute of Geochemistry, Siberian Division, Russian Academy of Sciences. The totals are given with correction for fluorine. Fe_2O_3 in samples ARB-4, ARB-8, and ARB-19 designates total iron as Fe_2O_3. Percentages of “fluoritic” phase (Ca–Fl) and prosopite (Pr) were calculated from mass balance. (–) Not analyzed; (tr.) traces.

contain inclusions of acicular topaz (<1–2 μm in cross section and 3–15 μm in length).

In Ca- and F-rich porphyritic ongonite, the interstices between minerals of the groundmass are filled with submicrometric “fluoritic” and “K-feldspathic” phases (Fig. 2a). In the areas free of visible inclusions (at the level of analytical resolution of one micrometer[1]), the fluoritic phase differs from fluorite crystals and always contains the following elements (wt %): 3–12 O, 0.5–3.3 Al, 0.2–1.5 Si, and up to 1.3 Sr, 0.5 Na, and 0.3 S. The composition of the K-feldspathic phase, as a rule, is close to potassium feldspar that rims albite, but however, is locally enriched in Ca (1.5–4.0 wt %). In this case, the ratio of the main components does not correspond to stoichiometry. In these rocks, albite phenocrysts rimmed with K-feldspar are replaced partly or completely with the fluoritic phase occasionally together with the K-feldspathic phase and a mineral that fits dickite or kaolinite $Al_2Si_2O_5(OH)_4$ in composition (Fig. 2a). In some samples, this mineral (further designated as dickite) occurs as submicrometric ingrowths in the fluoritic phase.

In aphyric rocks, smoky quartz and sanidine phenocrysts are scarce. In some samples, numerous large (up to 1 mm) segregations of Ca–Al fluoride—prosopite—have been detected (Figs. 2b, 2c). The XRD data corroborated identification of this mineral and indicated its characteristic peaks in powder diffraction patterns for all 10 studied samples of aphyric rocks. The large prosopite segregations are identified by characteristic sectorial wavy extinction. It is extremely difficult to identify this mineral in microgranular aggregates. The SEM examination has shown that prosopite is a rock-forming mineral of aphyric rocks. Its large (0.3–1.0 mm) segregations and intergrowths with sanidine or quartz phenocrysts often contain acicular topaz crystals (Figs. 2b, 2c) and quartz inclusions (20–50 μm). The fine-grained prosopite is dispersed rather uniformly throughout aphyric rocks. The mass balance has shown that the amount of prosopite in aphyric rocks may reach 26 wt % (Table 1). The composition of prosopite is

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[1] Analytical results presented in this paper were obtained with a LEO-1430 VP SEM equipped with an INCAEnergy 300 analyzer (accelerating voltage 20 kV, current 0.3–0.5 nA, beam diameter 1–10 μm, spectrum accumulation time 50 s); analyst N.S. Karmanov, Geological Institute, Ulan-Ude.

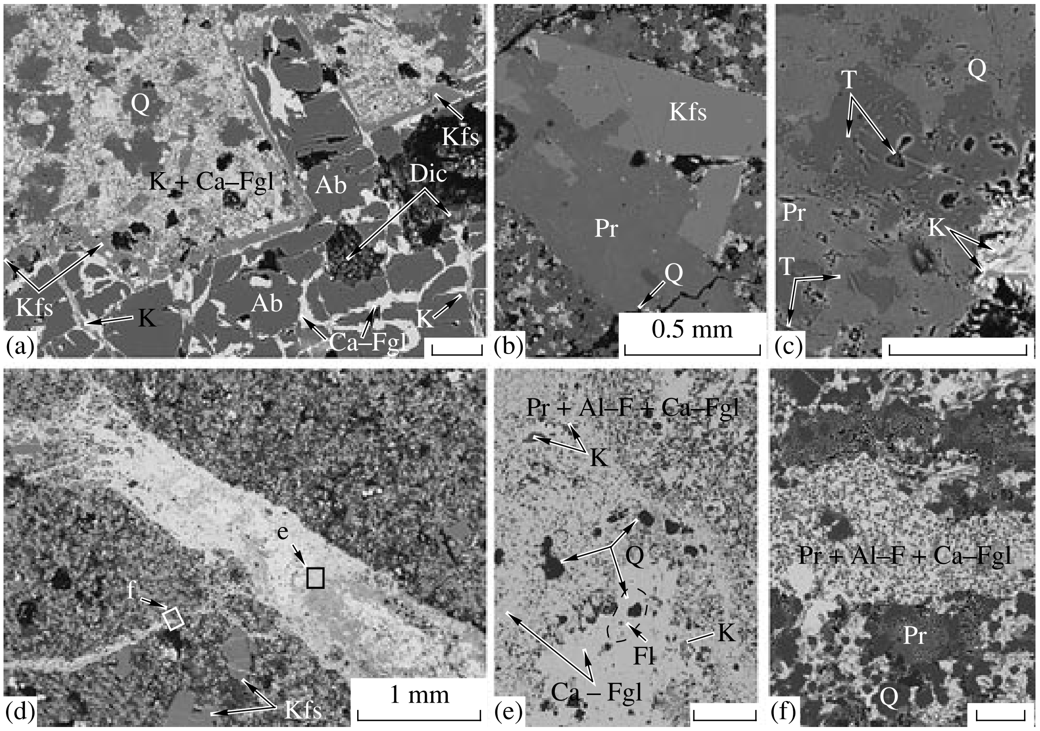

Fig. 2. (a) Porphyritic rock with "fluoritic" and "K-feldspathic" phases; (b, c) aphyric rock with prosopite; (d–f) pink and lilac veinlets in aphyric rocks. (Q) quartz; (Ab) albite; (Kfs) K-feldspar; (Dic) dickite; (Pr) prosopite; (Fl) fluorite; (T) topaz; (Al–F) calcium alumofluoride; (Ca–F gl) "fluoritic" phase; (K) "K-feldspathic" phase. Scale bar 50 µm, unless otherwise stated. BSE images.

close to $CaAl_2F_4(OH)_4$. It is characterized by insignificant variation and frequent presence of 0.5–1.3 wt % Sr. The clearly expressed growth zones in large prosopite grains show variation in the Sr content from 0.5 to 2.0 wt %. Previously, prosopite was not reported from ongonites.

As in calcic porphyritic ongonite, interstices between mineral aggregates of the groundmass in aphyric rocks are filled with submicrometric intergrowths of fluoritic and K-feldspathic phases. Sporadic sheath-shaped K-feldspar crystals filled with the fluoritic phase, occasionally in association with prosopite, dickite, and relicts of albite, occur in the groundmass. The fluoritic phase always contains an admixture of O, Al, and Si. The varieties with distinct fluidal structure and small (<1 cm) angular xenoliths of country mica schists are observed among aphyric rocks at the southwestern flank of the intrusion. Pinkish or lilac branching veinlets up to 1–5 mm thick (occasionally up to 1–2 cm) are developed here as well. Prosopite segregations together with fluoritic and K-feldspathic phases make up thin interlayers, which extend parallel to the fluidal banding and envelop schist xenoliths. The pink and lilac veinlets are composed of an aggregate of the fluoritic phase with numerous inclusions of prosopite and other Ca–Al fluorides, K-feldspathic phase, and quartz (Figs. 2d–2f). In proportions of F, Ca, and Al, Ca–Al fluorides correspond to gearksutite $CaAlF_4OH \cdot H_2O$ and carlhintzeite $Ca_2AlF_7 \cdot H_2O$ (both minerals are extremely unstable in the microprobe beam). Small fluorite segregations without O, Al, and Si occur sporadically in the groundmass of the fluoritic phase from lilac veinlets (Fig. 2e). The petrographic examination indicates that all rock-forming minerals (quartz, sanidine, albite, topaz, and prosopite) crystallized in the course of a common magmatic (subvolcanic) process in agreement with a genetic concept elaborated for the Ary-Bulak intrusion [4]. In line with this model, it may be suggested that intergrowths of fluoritic and K-feldspathic phases in interstices between minerals of the groundmass of porphyritic and aphyric rocks are products of partial crystallization (before and/or after quenching) of microemulsion of calcium fluoride and aluminosilicate melts.

It is probable that a part of the metastable calcium fluoride glass recrystallized to fluorite over a long time after solidification of the intrusion (142 Ma, after [6]). It is impossible to distinguish isotropic fluorite and recrystallized fluoritic glass by optic methods in thin sections. The XRD method used for this purpose allowed us to estimate the percentage of crystalline fluorite in the fluoritic phase (former glass). We compared the intensity of fluorite peak 220 in powder diffraction patterns of porphyritic and aphyric rocks with those of references mixtures of crystalline fluorite and low-Ca (0.3 wt % CaO) ongonite in the prescribed proportions (2.5, 5, 10, 20, and 30 wt % fluorite). In all cases, the CaF_2 content in rocks (C_{CaF_2}) calculated from the mass balance based on chemical compositions (under the condition that all Ca is bound in the fluoritic phase) was higher than the fluorite concentration ($C^{220}_{CaF_2}$) estimated

Table 2. Chemical compositions of silicate, fluoride glasses, fluorite in melt inclusions, and "fluoritic" phase in rocks, wt %

Component	1	2	Component	3	4	5	6	7	8	9
SiO_2	64.10	72.71	Si			0.23		0.73	0.75	0.34
Al_2O_3	18.65	13.19	Al		12.21	0.49	0–0.28	2.22	2.71	0.68
MgO	0.66		Mg	37.66		0.98		0–0.27		
CaO	4.29		Ca	0.44	1.20	45.36	49.14	43.68	41.91	48.84
Na_2O	2.89	5.38	Na		30.29	0–0.37	0–0.4		0.43	0–0.52
K_2O	5.13	4.69	K		0.82			0.15	0–0.36	0–0.32
F	7.59	6.69	F	61.90	54.35	49.07	50.87	47.63	44.82	46.71
Total	100.11	99.84	O			3.56		5.48	10.19	3.52
			S			0.27		0–0.12	0.14	
			Sr			0–0.3		0–0.41	0–0.62	0.37
			Total	100.0	98.87	100.10	100.01	99.89	100.95	100.45
N	9	19		2	8	5	2	5	5	6

Note: (1, 2) Aluminosilicate glasses in melt inclusions (MI): (1) MI shown in Fig. 3b with globules (glasses) of "fluoritic" and "sellaitic" phases; (2) MI with "cryolitic" phase; (3) "sellaitic" phase in MI shown in Fig. 3b; (4) "cryolitic" phase in MI 2; (5) MI filled with "fluoritic" phase; (6, 7) MI shown in Fig. 3c: (6) fluorite crystal, (7) "fluoritic" phase around fluorite crystal; (8, 9) fluoritic phase in porphyritic rock: (8) sample ARB-24, (9) sample ARB-15. Totals for analyses 1 and 2 are given with corrections for fluorine; gap denotes concentrations below the detection limit; *N* is number of analyses.

with the XRD method using the intensity of peak 220. Based upon these data, we calculated the content of fluorite in the fluoritic phase ($C^{220}_{CaF_2}/C_{CaF_2} \times 100$, %), which may be accepted conditionally as the degree of recrystallization of calcium fluoride glass. In the studied samples, this degree varies from 20 to 83%. In addition, it has been established that fluorite peak 220 in powder diffraction patterns widens to various extents in comparison with common fluorite. The greatest widening of this peak is typical of sample ARB-24 that contains up to 13% fluoritic phase at the least degree of its crystallinity (20%). The size of fluorite particles (regions of coherent dispersion) in calcium fluoride glass of this rock estimated from widening of peak 220 does not exceed 200 Å. In other samples, an increase in the degree of crystallinity of glass to 83% is accompanied by a reduced half-width of peak 220, which corresponds to an increase in the average size of fluorite particles to 800 Å. It is evident that the analysis of the fluoritic phase with a microprobe beam 1–10 μm in diameter yields an integral composition of calcium fluoride glass and fluorite microparticles. As has been shown above, the fluoritic phase contains appreciable amounts of O, Al, and Si, and, occasionally, Sr, S, Na, and K (Table 2, analyses 8 and 9). It is reasonable to suggest that incorporation of these elements (especially oxygen) in the fluorite structure results in change of the unit cell parameters. However, *d*-spacing of fluorite in all samples varies insignificantly (1.932 ± 0.001 Å) and corresponds to the reference value (1.932 Å). Thus, it may be suggested that admixtures concentrate largely in glass. Compensation of charge by entry of Al and Si into glass may be provided at the expense of partial replacement of F and Ca ($O^{2-} + Al^{3+} \rightarrow F^{-} + Ca^{2+}$). Thus, the XRD data give every reason to suppose that the fluoritic phase in Ca- and F-rich porphyritic and aphyric rocks is a mixture of calcium fluoride melt enriched in O, Al, Si, and other admixtures, on the one hand, and fluorite microparticles 200–800 Å, on the other hand, occurring in variable proportions.

Quartz and topaz phenocrysts in porphyritic rocks often contain primary melt inclusions (MI) and syngenetic gas-rich fluid inclusions (FI). We compared MI in quartz from common ongonite (sample ARB-23) and porphyritic ongonite with a great amount of fluoritic phase (sample ARB-24) taken at the southwestern flank of the intrusion. In both samples, quartz contains MI in the central and marginal zones of phenocrysts. Most melt inclusions are filled with a brownish residual glass and a deformed shrinkage bubble. In many cases, glass contains one or several crystalline phases (brown mica, K-feldspar, albite). The residual glass starts to melt at 450–500°C. Depending on the MI dimension, complete melting of glass is reached at 700–750°C in 1 or 2 h. Some small inclusions (3–5 μm) become homogeneous at that temperature. The long-term heating at ~ 800°C results in only partial melting of crystalline phases initially contained in MI. Complete melting is achieved after 30–35 hours of heating at 850–950°C. In many large MI (>50 μm), homogenization with dissolution of the gas bubble is not reached over 1 or 2 h at 1000–1050°C.

The residual and quenched glasses in most melt inclusions have aluminosilicate composition with a wide range of Si, Al, Na, K, and F contents and a Ca

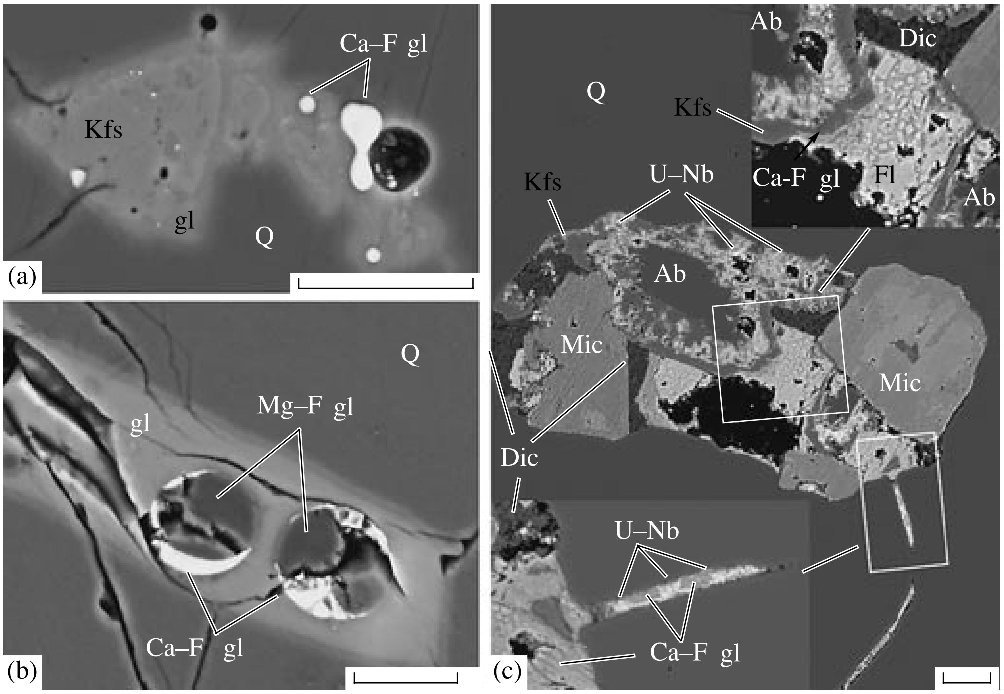

Fig. 3. Inclusions in quartz from (a, b) porphyritic ongonite (sample ARB-23 after heating to 900–950°C) and (c) from Ca- and F-rich porphyritic rock (sample ARB-24 without heating). (gl) Aluminosilicate glass; (Mic) mica; (Mg–F gl) “sellaitic” phase; (U–Nb) mineral with high U, Nb, Si, and Al contents. See Fig. 2 for other symbols. Scale bar 25 μm. BSE images.

content below 2 wt %. After heating to 900–950°C, MI filled with silicate glass containing rounded segregations (1–7 μm) of the fluoritic phase with typical O, Al, and Si admixtures were found in some quartz grains from sample ARB-23 of porphyritic ongonite (Fig. 3a). Silicate glass with an abnormally high Ca content (2.8–3.9 wt %, Table 2, analysis 1) fills a large MI. The glass includes globules (up to 20 μm in size) consisting of immiscible “fluoritic” and “sellaitic” phases (Fig. 3b). The sellaitic phase fits the stoichiometry of MgF_2 (Table 2, analysis 3), while the fluoritic phase contains admixtures of O, Al, Mg, and Si. In the heated quartz grains from calcic porphyritic ongonite (sample ARB-24), the rounded segregations of the fluoritic phase are clearly seen in MI (much more frequently than in MI from sample ARB-23). The silicate glass (Table 2, analysis 2) of such an MI includes spherical segregations (up to 50 μm) of a phase (or glass (?)) close to cryolite Na_3AlF_6 in composition with small Ca and K admixtures (Table 2, analysis 4).

Large quartz phenocrysts in ongonite are often crosscut by rounded, occasionally concentric fractures, which do not extend beyond the grain boundaries. Our data and those reported in [7, 8] suggest that the magmatic crystallization of quartz began at a temperature not lower than 750–650°C. Thus, the aforementioned fractures could have arisen as a result of $\beta \to \alpha$ transition in the process of phenocryst growth. According to the SEM data, such fractures in quartz phenocrysts from samples ARB-23 and ARB-24 are filled with a fluoritic phase occasionally containing flattened crystallites enriched in U, Nb, Si, and Al. In the late growth zone of a quartz grain in sample ARB-24, a silicate MI is associated with two inclusions. Inclusion 1 contains the fluoritic phase with microingrowths of dickite. Inclusion 2 contains the fluoritic phase, sheath-shaped K-feldspar crystals with albite relicts, zonal mica crystals on walls, and dickite in interstices between crystalline phases (Fig. 3c). The fluoritic phase with O, Al, Si, S, and Sr as admixtures (Table 2, analysis 7) contains numerous fluorite crystals with ideal stoichiometry without oxygen admixture (Table 2, analysis 6) and perfectly developed faces (Fig. 3c, inset).

The data on MI serve as direct evidence for capturing fluoride and aluminosilicate melts at the final stage of phenocryst growth. Fluorite in inclusions could have been formed both by its crystallization from a calcium fluoride melt and devitrification of glass. It is important to note that the calcium fluoride melt with inclusions of a mineral enriched in U, Nb, Si, and Al fills a rounded fracture related to inclusion 2. It is suggested that this fracture was formed as a result of $\beta \to \alpha$ transition of quartz during phenocryst growth. According to this interpretation, the calcium fluoride and residual aluminosilicate melts coexisted at a temperature below the polymorphic transition of quartz. Taking into account the previously obtained estimates of *PT* formation conditions of the Ary-Bulak intrusion (0.8–1.0 kbar at 650–700°C [7]), the temperature of this transition varies insignificantly with pressure and falls within an interval of 585–595°C.

It is known that the degree of immiscible liquids in fluoride–silicate melts changes from microscopic inclusions of one phase in another to their complete separation into two layers. The quenching of melts that are a fine emulsion with droplet dimensions varying from 0.*n* to *n* μm results in the formation of glasses with microemulsion texture (so-called speckled glasses) [1, 2, 9]. There is every reason to regard the submicrometer intergrowths of fluoritic and K-feldspathic phases in porphyritic and aphyric ongonites as products of partial quenching and crystallization of microemulsion that consisted of immiscible aluminosilicate and calcium fluoride melts. These melts coexisted with a fluid phase as indicated by syngenetic MI and FI in quartz and topaz and by the occurrence of water-bearing dickite in closed inclusions 1 and 2. Taking into account the F concentration in the fluid–magmatic system, it is reasonable to expect a considerable HF concentration in the fluid. We suggest that a combination of these factors gave rise to crystallization of rock-forming prosopite in aphyric rocks as a result of interaction of calcium fluoride melt, HF-bearing aqueous fluid, and earlier albite: $2NaAlSi_3O_8 + CaF_{2(melt)} + 4HF_{(fluid)} = CaAl_2F_4(OH)_4 + 6SiO_2 + 2NaF_{\uparrow(fluid)}$. At the postmagmatic stage, the high HF contents in the fluid facilitated autometasomatic replacement of relict albite with a dickite-type mineral: $2NaAlSi_3O_8 + 2HF_{(fluid)} + H_2O = Al_2Si_2O_5(OH)_4 + 4SiO_2 + 2NaF_{\uparrow(fluid)}$. Dickite also develops after calcium fluoride glass, consuming the O, Al, and Si contained therein. Formation of Ca- and F-rich rocks at the magmatic and postmagmatic stages led to partial binding of fluid by newly formed minerals (prosopite, dickite), gain of quartz, and removal of Na from the system.

The results of the study of melt inclusions show that the calcium fluoride and residual aluminosilicate melts coexisted at a temperature below the $\beta \rightarrow \alpha$ transition of quartz (595–585°C). The veinlets in aphyric rocks are filled with a fluoritic phase containing prosopite and hydrous Ca–Al fluoride microinclusions. This fact also testifies to the occurrence of the latest portions of Al-rich calcium fluoride melts in association with the fluid at a relatively low subsolidus temperature. Thus, Ca- and F-rich ongonites may be regarded as products of crystallization of immiscible aluminosilicate and calcium fluoride melts and their interaction with HF-bearing aqueous fluid.

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