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New data on minerals №52, iss.2 (2018), pp. 51 - 58

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Cristobalite in extrusive rocks of Bezymianny volcano

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Abstract
We present the first data on the systematic study of compositional and morphological variability of cristobalite in extrusive rocks of Bezymianny volcano (Kamchatka). Andesites and dacites of all seven studied extrusive domes contain cristobalite, which content reaches up to 6 vol.%, and thus may be considered as rock-forming mineral. We distinguish 4 different morphological types of cristobalite – 1) isometric grains surrounded by pores with the characteristic fish-scale cracking; 2) the “prismatic” cristobalite grains in clusters; 3) “pea”-type grains in the matrix glass; 4) “feathery”-type crystals. There were not revealed any clear dependencies between the morphological type of cristobalite, its composition and the composition of host extrusive rocks. Content of minor elements in cristobalite (mainly Al and Na) is up 10 wt.% on an oxide basis. The main mechanism of their isomorphic substitution is $Si^{4+} \rightarrow Al^{3+} + (Na^+, K^+)$, which can reflect the existence of a solid solution of cristobalite with isostructural to it carnegieite end-member. The entry of Ti (up to 0.27 wt.% of TiO_2), Fe (up to 0.43 wt.% of FeO) and Ca (up to 0.15 wt.% of CaO) into the cristobalite structure is described. Cristobalite from extrusive rocks of Bezymianny volcano has the widest range of composition in comparison with all previously published analyses of this mineral.

Introduction

For the first time cristobalite was described by Gerhard vom Rath (1887) in andesites of Cerro San Cristobal (Mexico) as intergrows with tridymite, established as a distinct mineral species in 1890 by Ernest-François Mallard and was studied in detail by Clarence Fenner in 1913 [Rogers, 1928].

Cristobalite has been described for many silicic and intermediate volcanic rocks [Horwell et.al., 2013; Larsen et.al., 1936; Martel et.al., 2000; Ohashi, 1936]. The relevance of cristobalite study is linked to the discovered carcinogenicity of this mineral [Erdmann et al., 2014; Smith, 1998]. Cristobalite grains in the extrusive rocks are associated with pores, which is interpreted as the result of its precipitation from gas phase during the eruption [Horwell et.al., 2013]. Cristobalite is found in different morphological varieties and characterized by a wide range of impurities, first of all, Al and Na [for example, Horwell et al., 2012]. The abundance of cristobalite in the extrusive domes, coupled with the diversity of its morphology and composition, make it a potential indicator of extrusive eruptions.

Despite the fact that cristobalite is a high-temperature modification of SiO_2, it may form during the devitrification of silicic glasses [Swanson et al., 1989], in diatomites [Ibrahim, Selim, 2012] and in opals [Smith, 1998]. In decompression experiments with rhyolite melts, cristobalite is present as an equilibrium phase at 850 °C and < 75 MPa [Martel et al., 2012]. “Platy” cristobalite in the groundmass formed along with the laths of plagioclase at the same temperature and 5-10 MPa in the fluid-rich environment [Martel, 2012].

Bezymianny is an active island-arc andesitic volcano in the central part of Klyuchevskoy group of volcanoes. Extrusive dome Novy (new) is currently located in the central part of the volcano. 11 old extrusive domes nest on the southern and south-western flanks of Bezymianny [Braitseva et al., 1990, Almeev et al., 2013], (Fig. 1).

The age of modern Bezymianny volcano edifice is ~4700 years, while around 11000-7000 years ago Pra-Bezymianny volcano was active in this area [Braitseva et al., 1995]. Extrusion of old domes took place in several stages. At the first one (Late Pleistocene), extrusive domes on the southern slope of Kamen volcano were formed prior to Pra-Bezymianny formation. It is customary to distinguish two groups of Late Pleistocene domes – early: Gladkii, Pravilnyi, Raschlenionny and late: Plotina, Stupenchaty, Dvuglavy, Razlaty, Kulich, which were formed as a result of subglacial eruptions. The second stage of extrusive domes growth took place in Holocene and was synchronous with the formation of modern volcano edifice. At this stage Expeditsii, Lokhmaty, Extrusivny Greben, and Treugolny Zub domes were formed [Braitseva et al., 1990, Almeev et al., 2013]. The youngest dome is Novy started to grow within the summit caldera of Bezymianny after the eruption of 1956.

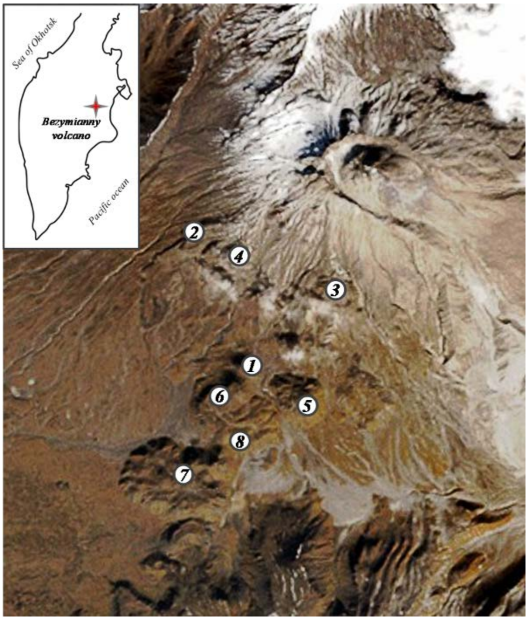

Fig. 1. The location of sampled extrusive domes of Bezymianny volcano. 1 – Gladkii , 2 – Treugolny Zub, 3 – Lokhmaty, 4 – Extrusivny Greben, 5 – Expeditsii, 6 – Dvuglavy, 7 – Plotina, 8 – Stupenchaty.

The results of the study of morphology and composition of cristobalite from andesites and dacites from extrusive domes of Bezymianny volcano are presented in this work for the first time. There will be shown that cristobalite is widespread in all extrusive rocks of the volcano.

Materials and methods

We studied 7 samples of extrusive domes of Bezymianny volcano: SK 09-16 – Expeditsii, SK-13-09 – Gladkii, SK-13-11 – Extrusivny Greben, SK-13-14 – Treugolny Zub, SK-13-10 – Dvuglavy, PK-13-24 – Lokhmaty, SK-09-14 – Stupenchaty. Data on coordinates of sampling are listed in Table 1.

Quantitative analyses of phases composition were conducted using scanning electron microscope "JEOL JSM-6480LV" with tungsten thermionic cathode and energy dispersive spectrometer XMaxN (Oxford Instruments) with 50 μm Li-Si semiconductor detector (129 eV resolution on Kα Mn) in Department of Geology of MSU. Electronic images are obtained in the mode of back-scattered electron detection at accelerating voltage of 20 kV and 10 nA beam current. Analyses were performed at 20 kV and 0.7 ± 0.01 nA. Such beam current for analyses was chosen to minimize the effect of cation migration under the influence of electron probe. In order to provide high counting rate and optimal dead time detector was set closer to the sample. The livetime of the spectrum accumulation was 100 seconds. Optimization of spectral lines profiles of measured elements was done using pure metals and oxides as standards. An iterative correction for the average atomic number, absorption, and secondary fluorescence was introduced automatically following a procedure of XPP-correction ("INCA", version 17a, Oxford Instrument). Analytical conditions were chosen so that the relative measurements errors (analysis reproducibility) did not exceed 1.5 rel.%. Standardization procedures provided the absolute error of the measurement in the range of 1-1.5 rel.%.

The identification of the silica phase was carried out using micro-Raman spectrometer XPloRA (Horiba Scientific) with 532 nm excitation wavelength. There were obtained spectra in the range of 100-4000 cm^{-1} with a spectral resolution around 1 cm^{-1} (diffraction grating 1800T), horizontal polarization and fully open aperture.

Quantitative estimations of phase proportions in studied samples were carried out using X-ray phase analysis with a pure Al_2O_3 as an internal standard. Automatic X-ray diffractometer DRON-3M (Laboratory of Lithology and Marine Geology, MSU) was used for these analyses, the analytical conditions are 30 kV and 20 mA.

Table 1. Studied samples and their sampling coordinates

Sample	SK-09-16 Expeditsii	SK-13-09 Gladkii	SK-13-11 Extrusivny Greben	SK-13-14 Treugolny Zub	SK-13-10 Dvuglavy	PK-13-24 Lokhmaty	SK-09-14 Stupenchaty
Latitude	N55°55′50.09″	N55°57′10″	N55°57′50.3″	N55°57′50.3″	N55°57′29.1″	N55°56′48.9″	N55°55′46″
Longitude	E160°34′36″	E160°33′35.9″	E160°33′58.3″	E160°33′58.3″	E160°33′35.9″	E160°35′25.9″	E160°33′43″

For the X-ray phase analysis, the rocks were powdered. The corundum powder (Al_2O_3) was added to the measured samples in a mass ratio of 1:1. Thereafter the sample was thoroughly mechanically homogenized and spread in a thin layer to the cuvette. The range of measurement angles is 10-80 2θ in increments of 0.05 2θ. The phase determination was carried out using software MATCH! 1.11. Spectra of minerals were compared with spectra from PDF-2 (2003) database. Quantitative determination of the phase proportions was carried out using the method of corundum numbers based on the comparison of phase of interest and corundum maximum intensity. For these calculations equation (1) was used, where Wi – weight % of the phase of interest, W_{Al2O3} – weight % of corundum in the mixture, K_i^{Al2O3} – corundum number of the phase of interest, I_i^S – maximum intensity of the phase of interest, I_s^{Al2O3} – maximum intensity of the corundum. Corundum numbers of minerals were taken from the PDF-2 database (2003).

$$\frac{w_i}{w_{Al2O3}} = \frac{1}{K_i^{Al2O3}} \frac{I_S^i}{I_S^{Al2O3}} \qquad (1)$$

For calibration and verification of this semi-quantitative method of X-ray phase analysis, the content of cristobalite in our samples was also determined by BSE image processing using the ImageJ graphics editor.

Petrography of studied rocks from extrusive domes

Rocks of Bezymianny extrusive domes form a continuous calc-alkaline trend corresponding to the rocks of 1956-2012 eruptions of the volcano. SiO_2 content in these rocks varies from 57.3 to 65.7 wt.% at the range of total alkalis 4.7-5.8 wt.% [Almeev et al., 2013] (Fig. 2). Domes with more basic composition have wider variations of SiO_2 and total alkalis than domes with more acidic composition. The rocks of Stupenchaty, Plotina, Dvuglavy, Expeditsii, and Extrusivny Greben fall in the field of andesites, while the rocks of Gladkii, Treugolny Zub and Lokhmaty – in the field of dacites.

The rocks have porphyry texture with medium to largely sized phenocrysts, finely porous, have light gray shades. Large phenocrysts of plagioclase with complex zoning and elongated phenocrysts of hornblende can be found in all of the studied samples with an exception of Dvuglavy and Stupenchaty domes, which have aphyric texture. Breakdown hornblende and clinopyroxene phenocrysts are present in Plotina dome in a small amount. Orthopyroxene can be found in the of Gladkii dome along with plagioclase and hornblende.

Ground mass of the studied rocks consists of plagioclase, hornblende, cristobalite, and volcanic glass. Pores are usually separated from each other, but in rocks of Lokhmaty and Plotina domes, they form channels. Cristobalite crystals, as well as associated feathered feldspars, are confined to these pores and channels (Fig. 3).

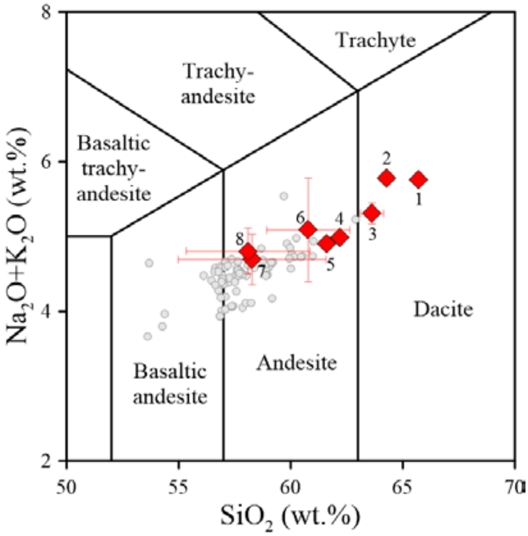

Fig. 2. The rocks composition of Bezymianny volcano following [Almeev et al., 2013]. Grey circles – rocks of modern eruptive cycle (1956-2012), red diamonds – composition of extrusive domes. Error bars of red diamonds are characterizing the range of rocks composition for each dome. Numbers are corresponding to the extrusive domes on the figure 1.

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Cristobalite morphology

Four morphological types of cristobalite can be distinguished: 1) crystals with the "fish-scale" cracking 2) the "prismatic" cristobalite; 3) "bead-type" rounded crystals without cracking; 4) "feathery" cristobalite. The size of "fish-scale"-type crystals varies in wide range: for some samples, it does not exceed 50 µm, for others can be found crystals up to 300 µm. The length of "prismatic" crystals does not exceed 100 µm, "bead-type" – 30-40 µm, the size of "feathery" cristobalite – first tens of microns. "Feathery" crystals are characterized by almost complete lack of cracking and isometric shape with no visible crystal faces.

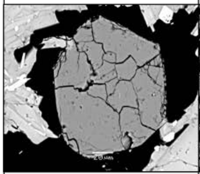
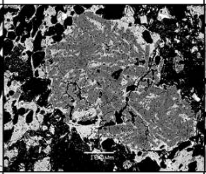
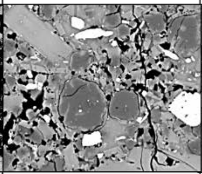
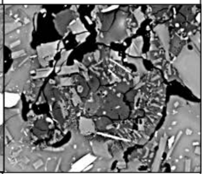
	«Fish-scale» cristobalite	Prismatic cristobalite	Bead-type cristobalite	Feathery cristobalite
				
Gladkii	+	+		
Dvuglavy	+		+	
Lokhmaty	+		+	
Plotina	+	+		+
Stupenchaty	+			
Treugolny Zub	+			+
Expeditsii	+		+	+
Extrusivny Greben	+		+	

Fig. 3. Prevalence of various types of cristobalite in extrusive domes.

«Fish-scale» cristobalite (Fig. 3) is found in all of the studied samples and it is a common morphological type for other described in literature extrusive domes, for example, it was described for Soufrière Hills (Montserrat, British Overseas Territories) [Horwell et al.,2013], Colima (Mexico), Mount St. Helens (USA), Unzen (Japan), Santiaguito (Guatemala), Merapi (Indonesia) [Damby, 2012] and Obsidian dome (USA) [Swanson et al., 1989]. «Fish-scale» cristobalite is always confined to the pore space, it is forming on the walls of the pores and channels, also it periodically fills them entirely. In some cases, it may be bound by a glassy porous matrix as, for example, in the rocks of Gladkii dome.

"Prismatic" cristobalite is commonly associate with «Fish-scale» crystals and confined to the walls of the pores. It can form large aggregates consisting of small twinned lamellar crystals, which filling the pore space. These aggregates are frequently surrounded by a rim of volcanic glass. "Prismatic" crystals of cristobalite have been found only in the rocks of Plotina and Gladkii domes. They have been also described in the work of Horwell with coauthors [2013] and common in all volcanic rocks described in the Ph.D. thesis of David Damby [2012].

"Bead-type" cristobalite is common for Dvuglavy, Expeditsii, Extrusivny Greben, and Lokhmaty domes. It can be found in the pore space, but most often it is in the groundmass as individual small crystals surrounded by volcanic glass.

"Feathery" cristobalite is also found only in rocks of Plotina, Treugolny Zub and Expeditsii domes. Earlier it was described in the rocks of Soufrière Hills [Horwell et al.,2013], Colima, Mount St. Helens, Santiaguito and Merapi [Damby, 2012]. It is usually found in aggregates with feldspar and quartz in volcanic glass and also in the rims of large cristobalite crystals.

Raman spectroscopy

Raman spectra were obtained for the cristobalite from rocks of Lokhmaty, Treugolny Zub, Extrusivny Greben, Expeditsii, Dvuglavy, and Stupenchaty extrusive domes. Crystals from rocks of Gladkii dome were not large enough to collect good quality spectra.

Raman spectra were compared with the reference cristobalite, quartz and tridymite spectra from RRUFF database and shown of figure 4. Two pronounced bands on 225 and 415 cm^{-1} present in all collected spectra correspond to the α-cristobalite [for example, Liang et al., 2006].

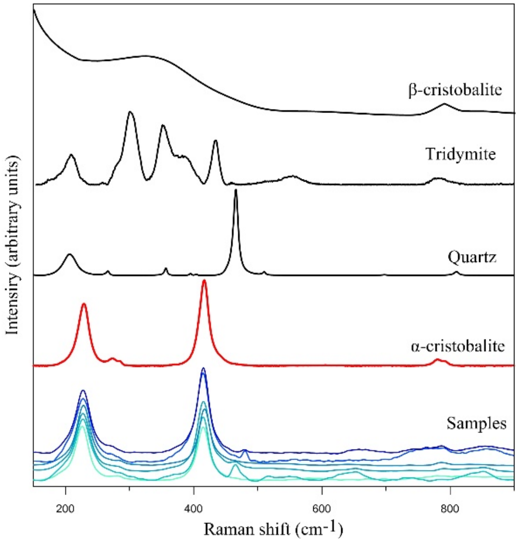


Fig. 4. Raman spectra of SiO_2 phases from the rocks of Bezymianny volcano extrusive domes. Reference spectra of SiO_2 polymorphic modifications are plotted following RRUFF database.

Chemical composition of cristobalite

The cristobalite from the studied samples is characterized by the contents of SiO_2 from 90.2 to 99.9 wt.%, Al_2O_3 from 0.15 to 5.91 wt.%, Na_2O from 0 to 2.69 wt.%, K_2O from 0 to 1.24 wt.% (see Table 2 and electronic supplementary). Also, it contains TiO_2, FeO_t, CaO within the first tenths of wt.%.

Cristobalite composition is nearly the same within the rocks of each extrusive dome and has significant differences between different domes: each dome is characterized by a smaller dispersion of cristobalite composition compared with the difference between different extrusive domes (Fig. 5).

Table 2. Cristobalite analyses for the different extrusive domes of Bezymianny volcano.

Sample №	Extrusive dome	SiO_2	TiO_2	Al_2O_3	FeO	CaO	Na_2O	K_2O	Total
SK-13-11	Extrusivny Greben	96.91	0.07	1.69	b.d.l.	0.08	0.69	0.22	99.66
SK-13-11	Extrusivny Greben	95.61	0.10	1.54	0.13	b.d.l.	0.72	b.d.l.	98.10
SK-13-14	Treugolny Zub	95.38	0.20	1.89	b.d.l.	b.d.l.	0.62	0.11	98.20
SK-13-14	Treugolny Zub	92.86	0.16	3.48	0.22	0.13	1.12	0.59	98.56
SK-13-10	Dvuglavy	94.77	0.15	4.50	0.14	0.08	2.75	b.d.l.	102.39
SK-13-10	Dvuglavy	94.90	0.20	4.35	0.13	b.d.l.	2.51	b.d.l.	102.09
SK-09-16	Expeditsii	93.70	0.17	5.90	0.10	0.11	2.56	0.27	102.81
SK-09-16	Expeditsii	94.92	0.12	4.80	0.14	0.10	2.24	0.22	102.54
PK-13-24	Lokhmaty	91.98	0.17	3.91	0.25	0.14	1.50	0.53	98.48
PK-13-24	Lokhmaty	91.10	0.21	4.95	0.34	0.14	1.74	0.81	99.29
SK-09-14	Stupenchaty	96.88	0.10	1.19	b.d.l.	0.12	0.73	b.d.l.	99.02
SK-09-14	Stupenchaty	96.84	0.11	2.10	b.d.l.	0.11	1.12	b.d.l.	100.28
SK-13-09	Gladkii	99.22	0.14	0.53	b.d.l.	b.d.l.	0.12	0.07	100.15
SK-13-09	Gladkii	99.32	0.09	0.54	0.12	b.d.l.	0.10	0.08	100.31

Notes:
* There are given two compositions of cristobalite with the maximum amount of impurities for rocks of each studied dome.

Content of cristobalite in extrusive rocks

The content of cristobalite in rocks of extrusive domes was identified on the base of powder diffraction data. Data are listed in table 3.

The contents of rock-forming minerals obtained by the quantitative X-ray phase analyses generally correspond to the optical petrographic observations. Cristobalite content in studied rocks varies in the range from 3 to 6 vol.%.

Table 3. The content of crystal phases in rocks of studied extrusive domes (wt.%) obtained by the method of quantitative X-ray phase analyses.

Extrusive dome	Wt.%			
	Plagioclase	Hornblende	Pyroxenes	Cristobalite
Extrusivny Greben	68	18	8	6
Stupenchaty	70	6	19	4
Expeditsii	53	35	6	6
Gladkii	45	43	9	3
Treugolny Zub	65	22	8	6
Lokhmaty	42	37	15	6

Discussion

The composition and content of cristobalite in extrusive rocks do not depend on the whole-rock composition, the composition of rock-forming minerals, and morphological type. These facts indicate that process of cristobalite formation is most likely controlled by the character of magma extrusion: the rate of ascent, porosity, and the gas phase composition.

Two of the four morphological types of cristobalite ("fish-scale" and "bead-type") have an isometric shape and could have been crystallized in cubic crystal system. Using the Raman spectroscopy, however, there was identified the only low-temperature tetragonal modification of cristobalite (α-cristobalite). High-temperature cubic cristobalite modification perhaps does not survive during relatively slow cooling of extrusive domes and become tetragonal. Such transitions are known for the leucite from subvolcanic rocks [Palmer et al., 1998].

The entry of impurities is linked mainly with the isomorphic substitutions. The main scheme of the substitutions is $Si^{4+} \rightarrow Al^{3+} + (Na^+, K^+)$ (these elements are the main impurities in cristobalite, Fig. 5). At the same time, the Si deficiency for the cristobalite from Lokhmaty and Expeditsii extrusive domes can't be compensated by the entry of alkalis and total other impurities either. This feature most likely linked to the partial loss of Na during the analyses under the influence of electron beam, caused by high Na content and small size of crystals, which does not allow analyze by defocused beam. Possible entry of water in the cristobalite structure is not supported by Raman spectroscopy (band in the range of 3200-3600 cm^{-1} are absent).

It is noteworthy that there is a correlation between the total content of alkalis with contents of Ti(IV) и Fe(III). The contents of Ti and Fe are also increasing with the increase of other impurities contents.

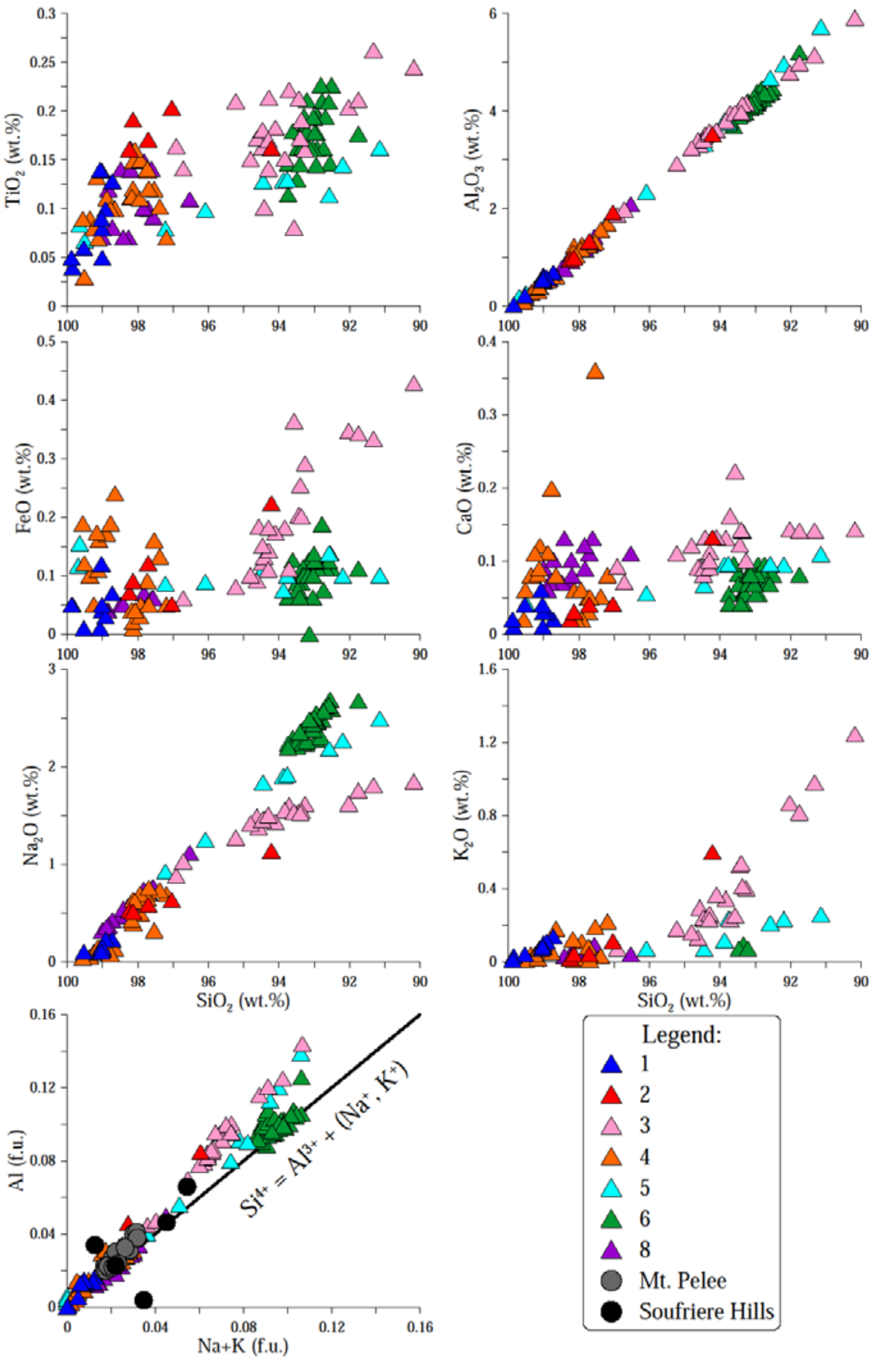


Fig. 5. Chemical composition of cristobalite from extrusive domes of Bezymianny volcano. The number are marking different domes and corresponding to the figure 1. The composition of cristobalite from Mt. Pelée and Soufrière Hills volcanoes are plotted for the comparison [Horwell et al., 2013]

We suggest that these isomorphic substitutions can be viewed as a limited mixing of cristobalite and isostructural to it carnegieite end-member. In this case, the solubility of the carnegieite end-member in cristobalite may be dependent on the crystallization temperature. This assumption explains the lack of impurities in low-temperature cristobalite, which is formed during the devitrification of acidic glasses and in sedimentary processes.

Cristobalite crystallizes preferentially from the gas phase, which filtered through cracks and pore space of extrusion domes [Horwell et al., 2010, 2013, 2014]. Small amounts of cristobalite in the rock are usually explained by the melt degassing and extraction of silica into the bubbles by the halogens in situ [Schipper et al., 2017]. There are up to 6 vol.% of cristobalite in the extrusive rocks of Bezymianny volcano and it is unlikely to crystallize without the fluid-related influx of silica in the rocks. The lack of zoning in the glass around cristobalite-rich pores is also an argument against redistribution of components in situ. At the same time, a fluid-related influx of silica has to increase its content in the rocks. Thus, the resulting whole-rock composition will not reflect the composition of the magma and it should be taken into account when interpreting data and modeling crystallization processes. We estimated the effect of cristobalite crystallization on the whole-rock composition using mass-balance calculations. As the parameters for this calculation we used cristobalite content in the rocks of extrusive domes (Table 3) and its average composition (electronic supplementary table). The whole-rock composition without cristobalite should have noticeably lower SiO_2 content and original magma composition without cristobalite for the part of samples should have been correspond to basaltic andesite field (for Stupenchaty dome) on the TAS classification chart (Fig. 6).

Conclusions

Cristobalite is found in all studied extrusive domes of Bezymianny volcano. It content varies from 3 to 6 vol.%, i.e. for extrusive rocks, it should be regarded as secondary or even rock-forming mineral.

Cristobalite of studied rocks contains 0.15–5.91 wt.% of Al_2O_3, up to 2.69 wt.% of Na_2O, up to 1.24 wt.% of K_2O and the first tenth percent of TiO_2, FeO and CaO. Predominate reaction of isomorphic substitution is $Si^{4+} \rightarrow Al^{3+} + (Na^+, K^+)$, which can reflect the existence of a solid solution of cristobalite with isostructural carnegieite. It can be proved by the methods of single-crystal X-ray diffraction and measurements of optical and physical properties depending on the content of aluminum and alkalis. In this case, a regular change in the unit cell parameters and refractive index should be observed, which would unequivocally confirm the assumption about the existence of the solid solution.

Cristobalite crystallizes only from the gaseous phase during the growing of extrusive domes. Its content in the extrusive domes of Bezymianny volcano reach 6 vol. %. Such amount of cristobalite can increase bulk silica content by up to 0.5-1 wt.%. It should be taken into account during the analyses, interpretation of extrusive domes whole-rock composition and it also plays an important role in the classification of rocks.

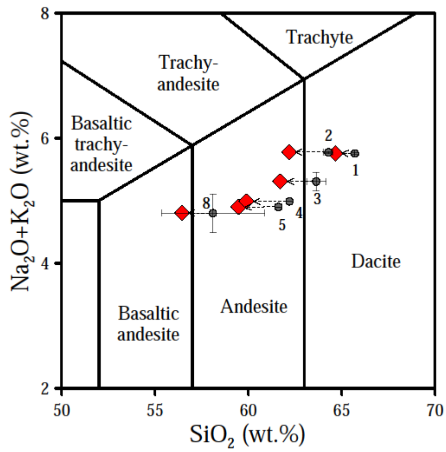


Fig. 6. Whole-rock composition of the Bezymianny volcano extrusive domes on the TAS classification chart. Gray circles indicate the compositions from the work of Almeev et al (2013), red diamonds – the same compositions recalculated without cristobalite. Numbers are corresponding to the extrusive domes on the figure 1.

Authors are grateful to L.A. Pautov for the valuable comments on the manuscript. This work was done with a support of RFBR grant № 16-35-60113 and using equipment purchased at the expenses of the Moscow University Development Program.

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