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# CRYSTALLIZATION OF GRANITIC GLASSES AT 700°C AND 1 KBAR

By

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### Abstract

Four granitic glasses ranging in composition from quartz diorite to granite were crystallized in the presence of excess water on the HM, NNO, QFM and MW buffers at 700°C in temperature and 1 kbar in total pressure. Quartz and plagioclase were formed in all runs, but crystallizations of alkali feldspar, biotite and clinoamphibole were restricted by their bulk composition. Amounts of ferromagnesian constituents were affected at oxygen fugacities above the NNO buffer. The results suggest that phase equilibria of granitic rocks are explained by an  $(Al_2O_3 - Na_2O - K_2O) - (Na_2O + CaO) - K_2O - (Fe_2O_8 + FeO + MnO + MgO)$  tetrahedron.

### Introduction

A number of experimental studies have been reported on the "Granite System" (e.g., TUTTLE and BOWEN, 1958; LUTH *et al.*, 1964), the "Granodiorite System" (e.g., VON PLATEN, 1965; WHITNEY, 1975) and natural rock-water systems (e.g., PIWINSKII and WYLLIE, 1968, 1970; ROBERTSON and WYLLIE, 1971a, b). However, relationships between salic and femic constituents have not been given in many cases of these studies. Crystallization of granitic glasses under various experimental conditions will provide the relationships during formation of the granites.

Many Tertiary granitic intrusive bodies ranging in composition from quartz diorite to granite occur in Kagoshima Prefecture, Japan. The present paper gives the results on crystallization of glasses made by fusion of four granitic rocks from the intrusive bodies.

# **Description of Granitic Rocks**

A quartz diorite, two granodiorites and an aplitic granite were prepared for this study from the Tertiary granitic intrusive bodies in Kagoshima Prefecture, Japan.

The quartz diorite SK01 was collected from the Teuchi-type (newly named) of the Shimokoshikijima granodiorite body cropping out in the Shimokoshikijima Island located in the northwestern part of the Prefecture. It is a dark gray-colored and medium-grained rock with granular texture, and mainly composed of plagioclase, quartz,

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potassium feldspar, biotite and hornblende.

The granodiorite OS03 was collected from the Uchinoura-type of the Ōsumi granodiorite body exposing in the eastern and southern parts of the Ōsumi Peninsula. It is a light gray-colored and medium-grained rock with granular texture, and mainly composed of plagioclase, quartz, potassium feldspar, biotite and a small amount of hornblende.

The granodiorite TK01 was collected from the Shinkoji-type of the Takakumayama granite body cropping out in the central part of the Peninsula. It is a light gray-colored and medium-grained rock with granular texture, and mainly composed of plagioclase, quartz, potassium feldspar and biotite.

The aplitic granite TK09 was collected from the Sarugajo-type of the same body. It is a light-colored and fine-grained rock with semiporphyritic texture, and composed of quartz, potassium feldspar, plagioclase, biotite and small amounts of garnet, muscovite and tourmaline.

Chemical analyses and CIPW norms of these granitic rocks are presented in Table 1. Compositions were determined by a combination of the 'standard' and 'ion exchange resin and chelate titration' (OKI *et al.*, 1962) methods. The differentiation indices (D.I.: THORNTON and TUTTLE, 1960), the sum of normative quartz, orthoclase and albite, vary from 58 in the quartz diorite to 91 in the aplitic granite. Normative diopside was calculated only in the quartz diorite; normative corundum was calculated in the other rocks. Figure 1 shows composition of the granitic rocks plotted in the normative quartz-total plagioclase-orthoclase system. In Figure 1, the granitic rocks except for the quartz diorite are closely plotted along minimum melting compositions at pressures below 5 kbars in the NaAlSi<sub>3</sub>O<sub>8</sub>-KAlSi<sub>3</sub>O<sub>8</sub>-SiO<sub>2</sub>-H<sub>2</sub>O system (TUTTLE and BOWEN, 1958; LUTH *et al.*, 1964).

# **Experimental Methods**

# 1. Starting Materials

Powdered rock samples were melted in Pt crucible at 1,300°C and one atmospheric



Fig. 1. Composition of granitic rocks plotted on the normative quartz-plagioclase (albite + anorthite)-orthoclase diagram. Pluses represent minimum melting composition in the NaAlSi<sub>3</sub>O<sub>8</sub>-KAlSi<sub>3</sub>O<sub>8</sub>-SiO<sub>2</sub>-H<sub>2</sub>O system (TUT-TLE and BOWEN, 1958; LUTH *et al.*, 1964).

Nos.	SK01	OS03	TK01	TK09
SiO <sub>2</sub>	63.60	67.23	71.20	75.82
TiO <sub>2</sub>	0.42	0.64	0. 30	0.09
$Al_2O_3$	16.66	15.52	14.22	13.94
$\mathrm{Fe_2O_3}$	0.91	0.60	1.01	0.19
FeO	4.04	4.40	1.47	0.48
MnO	0.23	nd.	0.07	tr.
MgO	3.00	1.31	0.97	0.25
CaO	5.44	2.37	2.40	0. 74
$Na_2O$	3.50	3.70	2.88	3.30
K <sub>2</sub> O	1.60	3. 23	3. 70	4.10
$H_2O+$	0.94	0.62	1.14	0.32
$H_2O-$	0. 10	0.22	0.26	0.24
$P_2O_5$	0.04	0.30	0.08	0.03
Total	100. 48	100. 14	99. 70	99.50
Q	18.6	24.2	33. 2	38.8
Ör	9.5	19.1	21.9	24.2
Ab	29.6	31.3	24.4	27.9
An	25.0	9.8	11.4	3.5
С		2.3	1.3	2.8
Hy(En)	7.1	3.3	2.4	0.6
Hy(Fs)	6.1	6.5	1.5	0.6
Di(Wo)	0.7			
Di(En)	0.4			-
Di(Fs)	0.3		-	
Mt	1.3	0.9	1.5	0.3
I1	0.8	1.2	0.6	0.2
Ар	0.1	0.7	0.2	0.1
D.I.	57.7	74.6	79.5	90. 9

Table 1. Chemical analyses and CIPW norms of granitic rocks

SK01: Hornblende-biotite quartz diorite of the Teuchi-type of the Shimokoshikijima granodiorite. Analysis by M. KAWAKAMI (1973).

OS03: Hornblende-bearing biotite granodiorite of the Uchinoura-type of the  $\bar{O}$ sumi granodiorite. New analysis.

TK01: Biotite granodiorite of the Shinkoji-type of the Takakumayama granite. Analysis by M. YAMAMOTO (1975).

TK09: Biotite aplitic granite of the Sarugajo-type of the Takakumayama granite. Analysis by J. OGURA (1970).

pressure and quenched by simply removing the crucible from furnace into water. Because granitic rock has high viscosity, the quenching was carefully repeated three times. Crystalline phases in granitic glasses were checked with the X-ray powder diffraction method (Figs. 3–6). Only the granodiorite OS03 was completely melted. Quartz was found in X-ray patterns for the other glasses. Cristobalite was also found in the glass TK09. Presence of quartz and cristobalite is not significant for this study, since granitic rock is silica-oversaturated.

Figure 2 shows relation between refractive indices and the differentiation indices of the granitic glasses. The refractive indices were measured by the immersion method, and range from 1.547 in the glass SK01 to 1.490 in TK09. In Figure 2,

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Fig. 2. Relation between refractive indices (n) and differentiation indices (DI) of granitic glasses.

solid line represents a curve obtained by the least squares method. The refractive indices vary linearly with the differentiation ones, although some glasses contain one or two of silica minerals.

# 2. Experimental Procedures

Experiments on crystallization of granitic glasses were all performed in standard 'cold seal' pressure vessels (TUTTLE, 1949). The fluid pressure, always equals to the total pressure in runs, was maintained within 20 bars of 1 kbar for period up to thirteen days. The temperature was measured with chromel-alumel thermocouples and did not deviate more than  $\pm 5^{\circ}$ C from 700°C during a run. Oxygen fugacity was controlled by using the buffer technique devised by EUGSTER and WONES (1962). The hematite-magnetite (HM), nickel-nickel oxide (NNO), quartz-fayalite-magnetite (QFM) and magnetite-wüstite (MW) buffers were used for controls of oxygen fugacity.

The charge of the starting material plus water was put in an inner Pt capsule and sealed by electric welding. Proportion of water added to the starting material was 1:10 by weight. This capsule, surrounded by a suitable buffer plus excess water, was enclosed in an outer Au capsule and sealed by gas torch welding. Quenching was achieved by simply removing pressure vessel from furnace and the vessel was cooled down to room temperature.

Run products were removed from charge containers, dried at  $60^{\circ}$ C in air and subsequently examined by means of only an X-ray diffractometer employing CuKa radiation ( $\lambda = 1.54178$  Å) ranging in  $2\theta$  from 5° to 60°. X-ray powder diffraction patterns for the products are shown in Figs. 3 to 6.

# **Experimental Results**

Experimental data for the four granitic glasses are presented in Table 2. Solid phases obtained are quartz, plagioclase, alkali feldspar, mica, amphibole, hematite and magnetite. Quartz, plagioclase and magnetite were formed in all runs, but crystallizations of alkali feldspar, mica, amphibole and hematite were restricted.

Figure 7 shows relation between  $2\theta$  values of 060 and  $\overline{2}04$  reflections of alkali





- 1. Natural rock.
- 2. Glass.
- 3. Run product on the HM buffer.
- 4. Run product on the NNO buffer.
- 5. Run product on the QFM buffer.
- 6. Run product on the MW buffer.



Fig. 4. X-ray powder diffraction patterns for run products from the glass OS03 at 700°C and 1 kbar.

Numbers correspond to those in Fig. 3.



Fig. 5. X-ray powder diffraction patterns for run products from the glass TK01 at 700°C and 1 kbar. Numbers correspond to those in Fig. 3.

Fig. 6. X-ray powder diffraction patterns for run products from the glass TK09 at 700°C and 1 kbar. Numbers correspond to those in Fig. 3.



Nos.	Buffer	Time (days)	Phases
SK 01	HM	13	Pl, Qz, Bt, Hb, Mt, Hm
OS 03	$\mathbf{H}\mathbf{M}$	10	Pl, Qz, Af, Bt, Mt, Hm
TK01	$\mathbf{H}\mathbf{M}$	13	Pl, Qz, Af, Bt, Mt, Hm
TK 09	$\mathbf{H}\mathbf{M}$	10	Pl, Qz, Af, Mt, Hm
SK 01	NNO	10	Pl, Qz, Bt, Hb, Mt
OS 03	NNO	10	Pl, Qz, Af, Bt, Mt
TK 01	NNO	10	Pl, Qz, Af, Bt, Mt
TK 09	NNO	10	Pl, Qz, Af, Bt, Mt
SK 01	QFM	10	Pl, Qz, Bt, Hb, Mt
OS 03	QFM	10	Pl, Qz, Af, Bt, Mt
TK 01	QFM	10	Pl, Qz, Af, Bt, Mt
TK 09	QFM	10	Pl, Qz, Af, Bt, Mt
SK 01	MW	10	Pl, Qz, Bt, Hb, Mt
TK01	$\mathbf{MW}$	10	Pl, Qz, Af, Bt, Mt

Table 2. Experimental results on granitic glasses at 700°C and 1 kbar

Abbreviations: Pl, plagioclase; Qz, quartz; Af, alkali feldspar; Bt, biotite; Hb, clinoamphibole; Mt, magnetite; Hm, hematite.

Fig. 7. Plots of alkali feldspars on the  $2\theta$ (060)- $2\theta$ (204) diagram simplified from WRIGHT (1968). Curves A, B and C represent maximum microcline-low albite series, alkali-exchanged orthoclase and high sanidine-high albite series, respectively.



feldspars. The  $2\theta$  values were determined by their peaks of CuK $a_1$  radiation ( $\lambda = 1.54050$  Å) scanned at a rate of  $1/4^{\circ} 2\theta$  per min. on chart scale of  $1/8^{\circ} 2\theta$  per cm. In Figure 7, the synthesized alkali feldspars are plotted near the orthoclase side of the 'high sanidine-high albite' solid solution series of ORVILLE (1967), indicating that those are essentially of the sanidine variety. Sanidine was formed in runs for granitic glasses except for the glass SK01. It was always accompanied with mica, but did not coexist with amphibole.

Amounts of mica are inversely proportional to those of iron oxides, as seen from a comparison of their X-ray intensities in Figs. 3–6. This suggests that synthesized mica will be essentially of biotite on the phlogopite-annite join. Biotite was formed in

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almost all runs, but did not crystallize in the run of the glass TK09 on the HM buffer. According to the experimental work on the join by WONES and EUGSTER (1965), magnesian biotite is stable at a relatively higher oxidation state as compared to iron one. The aplitic granite is a magnesium-poor rock (Table 1). Therefore, it is not seen whether biotite could not be detected by the X-ray pattern since its synthesized amount was very small, or magnesium ion was contained in iron oxides as a magnesioferrite molecule.

 $2\theta$  values and X-ray intensities of amphibole in Fig. 3 indicate that it is clinoamphibole. Clinoamphibole was formed only in runs of the glass SK01. The fact that normative diopside is calculated in the quartz diorite suggests that synthesized clinoamphibole will be essentially of the calcic variety. According to experimental studies of calcic amphiboles, magnesian end members such as tremolite and pargasite (Boyd, 1959) are stable at 700°C and 1 kbar. It is, therefore, impossible to establish the solid solution series to which clinoamphibole obtained belongs.

Hematite was formed only in runs on the HM buffer. Magnetite was formed in all runs. Wüstite could not be detected in all X-ray patterns. Although titanium ion may be contained in iron oxides as an ilmenite molecule, this was not observed in detail.

# Discussion

# 1. Coexisting Feldspars

It is considered that coexisting feldspars are structurally the high temperature forms. Compositional change of the feldspars was observed in their X-ray patterns.

In Figures 3 to 6,  $2\theta$  values of  $\overline{2}01$  reflection of sanidines increase with increasing of the differentiation indices of the bulk composition. According to ORVILLE (1967), this indicates that KAlSi<sub>3</sub>O<sub>8</sub> contents decrease with increasing of the indices. The values could not be accurately determined by the X-ray powder diffraction method except for sanidines from runs of the glass TK09, since peaks of the  $\overline{2}01$  reflection of sanidine and 110 one of quartz overlapped each other. The  $2\theta$  values of some sanidines are presented in Table 3. In Table 3, the values are fairly uniform in sanidines from runs of the same glass at various oxygen fugacities.

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Nos.	HM (Or%)**	NNO (Or%)**	QFM (Or%)**	MW (Or%)**
OS 03 TK01 TK09	21. 11 (86) 21. 13 (84) 21. 32 (67)	nd. 21. 11 (86) 21. 31 (67)	21. 12 (85) 21. 16 (82) 21. 32 (66)	 21. 13(84) 

Table 3.  $2\theta$  values of  $\overline{2}01$  reflection of alkali feldspars\*

\* Ni-filtered CuKa radiation ( $\lambda = 1.54178$  Å). Silicon external standard. 1° and 0.3 mm slits.  $1/4^{\circ}$  20 per min. scan speed.

\*\* Or% estimated from  $2\theta(\bar{2}01)$  data of 'high sanidine-high albite' solid solution series of ORVILLE (1967).

sanidine will not be affected by oxygen fugacity.

 $2\theta$  values of  $\overline{2}01$  reflection of plagioclases are presented in Table 4. The values decrease with increasing of the differentiation indices of the bulk composition, and KAlSi<sub>3</sub>O<sub>8</sub> contents increase. On the other hand, the values are fairly uniform in plagioclases from runs of the same glass at various oxygen fugacities. This suggests that composition of plagioclase will not be also affected by oxygen fugacity, as well as sanidine. Such changes in the KAlSi<sub>3</sub>O<sub>8</sub> content of sanidine and plagioclase are similar to those in the "Granodiorite System" experimentally studied by WHITNEY (1975).

Nos.	HM	NNO	QFM	MW
SK 01	22. 04	22.04	22.06	22.06
OS 03	21. 99	22.00	22.02	
TK 01	21. 97	21. 96	<b>21.</b> 99	22.00
TK 09	21. 95	21. 97	21. 97	

Table 4.  $2\theta$  values of  $\overline{2}01$  reflection of plagioclases\*

\* See footnote, Table 3.

# 2. Ferromagnesian Constituents

Figure 8 shows changes in ratio of X-ray intensities for 001 reflection of biotites from runs of the glasses SK01 and TK01 as a function of oxygen fugacity. Ratios were calculated on the basis of their relative intensities of biotites on the HM buffer. In Figure 8, the ratios are smallest in the biotites on the HM buffer, and fairly uniform in those on the other buffers. The ratios in amount of biotites at oxygen fugacities above the NNO buffer are similar to weight proportion of synthetic ones calculated

Fig. 8. Changes in ratio of X-ray intensities for 001 reflection of biotites and 110 one of clinoamphiboles with oxygen fugacity. Ratios were calculated on the basis of their relative intensities for run products on the HM buffer. Open and solid circles represent biotites and clinoamphiboles, respectively. Dotted curve represents change in weight proportion of synthetic biotites calculated from experimental data at 700°C and 1,035 bars by WONES and EUGSTER (1965).



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from experimental data at 700°C and 1,035 bars by WONES and EUGSTER (1965). According to the data, composition of the biotites at divariant points, biotite-sanidine-magnetite, in the phlogopite-annite join is  $Phl_{62}Ann_{23}$  on the HM buffer,  $Phl_{31}Ann_{62}$  on NNO and  $Phl_{11}$  on QFM. It is suggested, therefore, that composition of biotites obtained will vary from about  $Phl_{62}Ann_{23}$  on the HM buffer to about  $Phl_{31}Ann_{62}$  on NNO, and that on the other buffers will be affected by the bulk composition.

Clinoamphibole was formed only in runs of the glass SK01. Change in ratio of X-ray intensities for 110 reflection of clinoamphibole is shown as a function of oxygen fugacity in Fig. 8, as well as biotites. The change of clinoamphibole is similar to the changes of biotites except for the MW buffer. In the temperature-oxygen fugacity diagram, stability curve of ferropargasite experimentally determined by ERNST (1966) is similar in shape to that of annite by EUGSTER and WONES (1962). As mentioned above, magnesian end members such as tremolite and pargasite (BOYD, 1959) are stable at 700°C and 1 kbar. It is estimated, therefore, that stability relation of clinoamphibole obtained will be similar to that on the phlogopite-annite join experimentally determined by WONES and EUGSTER (1965).

# 3. Phase Equilibria of Granitic Rocks

In this study, sanidine was not formed in runs of the glass SK01, but biotite was formed. This suggests that potassium ions are originally used to crystallize biotite and sanidine is formed from the residual ions. On the other hand, clinoamphibole is not formed in runs of the glasses OS03, TK01 and TK09, but plagioclase was formed. This also suggests that calcium ions are originally used to crystallize plagioclase and clinoamphibole is formed from the residual ions. Sanidine and clinoamphibole did not coexist in all runs, indicating that presence or absence of these minerals are affected by an amount of potassium ion.

Figure 9 shows composition of the four granitic glasses plotted in an  $(Al_2O_3-Na_2O-CaO)-(Na_2O+CaO)-K_2O-(Fe_2O_3+FeO+MnO+MgO)$  tetrahedron by mol. per cent. In Figure 9, solid lines represent mineral assemblages of run products. Clinoamphibole is indicated by the tremolite-ferrotremolite solid solution series. In the basal plane,  $(Na_2O+CaO)-K_2O-(Fe_2O_3+FeO+MnO+MgO)$  system, the glass SK01 is plotted in the albite-biotite-tremolite triangle, and the other glasses are plotted in the albite-sanidine-biotite one. As seen from broken lines in Fig. 9, plot of the glass SK01 is located in the plagioclase-biotite-tremolite volume, and plots of the other glasses are located in the plagioclase-biotite plane dividing into the two volumes is important for explanation of phase equilibria such as presence or absence of sanidine and clinoamphibole.

# 4. Genetical Considerations of Granitic Rocks

As mentioned above, this experimental data indicate that sanidine and clinoam-



Fig. 9. Composition of granitic glasses plotted in the  $(Al_2O_3 - Na_2O - K_2O) - (Na_2O + CaO) - K_2O - (Fe_2O_3 + FeO + MnO + MgO)$  tetrahedron by mol. per cent. Open and solid circles represent plots in the tetrahedron and those projected on the basal  $(Na_2O + CaO) - K_2O - (Fe_2O_3 + FeO + MnO + MgO)$  plane, respectively. Hatched wall represents plagioclase-biotite plane. Numbers along plagioclase solid solution are anorthite contents. Abbreviations: An, anorthite; Ab, albite; Sa, sanidine; Bt, biotite; Tr, tremolite; Hm, hematite; Mt, magnetite.

phibole do not coexist at an equilibrium state. However, the quartz diorite contains potassium feldspar, and the granodiorite OS03 contains a small amount of hornblende. It may be suggested, therefore, that these rocks would not have been equilibrated during formation of these granitic intrusive bodies, and that fractional crystallization would have taken place. On the other hand, the mineral assemblages of the granodiorite TK01 and the aplitic granite are agreement with those of their run products. This suggests that equilibria of these rocks would have been attained or nearly attained during formation of the granite body.

It was shown in Fig. 8 that composition of biotite is possible to change in runs at oxygen fugacities above the NNO buffer. As reported by YAMAMOTO and  $\bar{O}BA$  (1974), it has been known that composition of biotites changes with that of their host granitic rocks. It is suggested, therefore, that biotites in the granitic rocks would have been formed at oxygen fugacities above the NNO buffer.

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