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SENTINEL GAP BASALT REACTED IN A TEMPERATURE GRADIENT

by

R. W. Charles and G. K. Bayhurst

ABSTRACT

Six basalt prisms were reacted in a controlled temperature gradient hydrothermal circulation system for two months. The prisms were centered at 72, 119, 161, 209, 270, and 310°C. Total pressure was 1/3 kbar. All prisms showed large weight loss: 5.5% to 14.9%. The matrix micropegmatite and natural nontronitic alteration reacted readily to clays at all temperatures. The first four prisms were coated with a calcium smectite, and the last two prisms were covered with discrete patches of potassium-rich phengite and alkali feldspar. The results indicated that clays may act as adsorbers of various ions.

I. INTRODUCTION

Basalt is being considered as a host rock for a radioactive waste repository.¹⁻⁴ One of the basalts under consideration is the Grande Ronde basalt of the Yakima Subgroup of the Columbia River Basin Group. Chemical reactions involving such basalt with groundwater are of interest to those working with any repository involving basalt. In the experiments presented here, a basalt sample from one of the flows of the Sentinel Bluff flows in a controlled temperature gradient circulation system under constant pressure in water that was initially distilled for a period of two months. The purpose of these experiments is to define some of the reaction relations between the fluid and basalt as well as the mass transport.

II. THE EXPERIMENT

Figure 1 exhibits the controlled temperature gradient system. Flow is clockwise. Details of the system's operation are given in Refs. 5 and 6. Six polished prisms of basalt (2.5 cm x 0.6 cm x 0.6 cm) were placed in a vessel in which a temperature gradient could be imposed at temperatures of 72, 119, 169, 209, 270, and 310°C with fluid flow from cool to hot. Total pressure was 1/3 kbar. All of the prisms were removed after 60 days of reaction time.

After 1, 2, 6, 14, and 30 days, the solution was sampled at the two points labeled with constant pressure and temperature samplers. The samplers are explained in Refs. 1-6. (A final sample, taken from the cooled fluid after 60 days, showed fractionation and is not included herein.) The fluid reached a steady state after 6 to 14 days. Solutions were analysed by plasma emission spectrometry. Rock prisms were examined using a scanning electron microscope (SEM) with a nondispersive detector, an electron microprobe, and an optical microscope. Thin sections were prepared for each rock prism, so interiors as well as surfaces could be examined.

Fig. 1. Controlled temperature gradient circulation system.

The aphanitic basalt shows a subophitic texture with intersertial nontronite and micrographic alkali feldspar plus quartz. The principle phases are augite, pigeonite, sodic labradorite, titaniferous magnetite, nontronite, and matrix. The matrix is a micropegmatite with a rhyolite norm. Normalized to Or-Ab-Q, the groundmass, while showing wide scatter, plots near the minimum melting point of granite (see Fig. 2). The normative concentration of the basalt is quartz-hypersthene. See Table I for details of phase composition, whole rock analyses, and norm.

III. RESULTS

A. Weight Loss

The basalt prisms showed rather high weight losses (Fig. 3 and Table II). The prism at 72°C showed a loss of 5.5% in two months of reaction time, and the prism at 310°C showed a loss of 14.9%. Included for comparison (in Fig. 3) is an experiment that was done under identical run conditions, except the rock was a granodiorite 9519, from the Fenton Hill HDR reservoir. At no time does the basalt show actual weight gains as does the granodiorite (at 72 and 119°C).

B. Solution Analyses

Solutions in contact with basalt seemed to reach a steady state at about 6 days (hot sample) and continue at this level for the remainder of the experiment (Table III, Fig. 4). Hot sample was extracted at 310°C; cold sample was extracted at room temperature. Initial maximums were observed (cold solution) for SiO₂, potassium, sodium, and possibly iron and calcium.

C. Morphological Changes

Scanning electron photomosaics of prisms in various stages of the reaction process are shown in the appendix (Figs. A-1 through A-8). Figure A-1 shows an unreacted rock prism. The darkest material is plagioclase and micropegmatite, and the gray material is pyroxene. Opaques are lightest. The only surface relief is some plucking caused by polishing.

Overgrowth morphology occurs in two types.

- (1) All pervasive clay at 72, 119, 161, and 209°C.
- (2) Discrete clay overgrowths plus feldspar at 270 and 310°C.

Fig. 2. Quartz-nephelene-kalsilite projection with "keyhole"-shaped plot of basalt matrix compositions near granite minimum melting.

Figure A-2 shows a portion of the rock prism reacted at 72°C. Much reaction has occurred. "Fracture" openings and voids represent dissolution of micropegmatite. Secondary precipitation consisting of botriodal platey aggregates is shown in Fig. A-3. The platey aggregates cover the rock surface more completely at 119°C (Fig. A-4). Cavities are in the matrix. Figure A-5 shows the same overgrowth, in this case at 161°C. The same overgrowth persists to 209°C (Fig. A-6).

Overgrowth morphology changes strikingly at 270°C (Fig. A-7). Two phases now cover the basalt surfaces. Clay of a different composition surrounds laths of recrystallized feldspar. Figure A-8 presents a portion of the 310°C prism showing similar morphology with possibly slightly better crystallization of the feldspars. Note in particular the euhedral crystals at 6 o'clock and 9 o'clock.

TABLE I
SENTINEL GAP BASALT WHOLE ROCK ANALYSIS, NORM, AND MINERALOGY

Wt % Oxides		Norm (%)	
SiO ₂	53.00	il	2.74
TiO ₂	1.93	ap	0.17
Al ₂ O ₃	14.80	or	7.00
Fe ₂ O ₃	2.00	ab	24.81
FeO	10.87	an	25.40
CaO	8.43	mt	2.13
MgO	4.59	di	13.80
Na ₂ O	2.70	hy	19.58
K ₂ O	1.15	Q	4.37
MnO	0.22		
P ₂ O ₅	0.31		
	100.00		

Augite	Mg _{0.88±.02}	Fe _{0.46±.03}	Ca _{0.70±.02}	Ti _{0.02±.01}	Al _{0.07±.01}	Si _{1.90±.01}	O ₆
Pigeonite	Mg _{0.82±.10}	Fe _{0.98±.07}	Ca _{0.26±.02}	Mn _{0.02±.01}	Al _{0.03±0.1}	Si _{1.92±.01}	O ₆
Plagioclase range	rim	Ab ₅₇	An ₄₀	Or ₃ to core	Ab ₄₀	An ₅₈	Or ₂
	average	Ab ₄₇	An ₅₁	Or ₃			
	Na _{0.47±.01}	Ca _{0.51±.02}	K _{0.03±.01}	Al _{1.47±.01}	Si _{2.49±.02}	O ₈	
Titaniferous magnetite	Mt ₅₅	Il ₄₅	+ (trace aluminum)				
nontronite	Ca _{0.32±.01}	Mg _{1.49±.03}	Fe _{3.40±.03}	Si _{7.31±.03}	Al _{1.20±.01}	O ₂₀	(OH) ₄
groundmass	Or _{29.0±1.4}	Ab _{29.4±4.9}	An _{3.1±.06}	Q _{38.5±4.4}

Fig. 3. Weight change of basalt prisms compared to weight change of granodiorite 9519.

TABLE II
BASALT PRISM WEIGHT LOSS

Prism	Weight		Change	
	Original (g)	Final (g)	(g)	(%)
1	3.0377	2.8708	-0.1669	- 5.5
2	3.0145	2.8373	-0.1722	- 5.9
3	3.0595	2.8445	-0.2150	- 7.0
4	3.0525	2.7980	-0.2545	- 8.3
5	3.0640	2.6438	-0.4202	-13.7
6	3.0172	2.5675	-0.4497	-14.9

TABLE III
ANALYSES OF SOLUTIONS IN CONTACT WITH BASALT^a

Duration (days)	SiO ₂		Potassium		Sodium		Iron		Aluminum		Calcium		Total	
	Hot	Cold	Hot	Cold	Hot	Cold	Hot	Cold	Hot	Cold	Hot	Cold	Hot	Cold
1	-	294	-	9.6	-	8.1	-	0.20	-	7.6	--	0.20	-	320
2	335	222	13.0	8.9	9.7	6.4	0.20	0.20	11.6	7.6	0.20	0.10	370	245
6	373	250	12.4	9.5	9.2	6.8	0.30	0.10	10.8	7.5	0.10	0.10	406	314
14	372	325	13.4	10.4	11.9	10.6	0.07	0.07	10.9	8.4	0.09	0.09	408	355
30	359	314	12.3	9.9	12.7	11.4	0.08	0.09	10.3	7.8	0.20	0.20	395	343

^aIn parts per million.

Fig. 4. Solution analyses.

D. Chemical Changes

Reactivity of the basalt varied from nonexistent to major. Table IV displays the reactivity of some of the primary phases. The analyses are from thin sections prepared from each of the six prisms. Plagioclase, augite, and pigeonite show little reaction.

Micropegmatite shows the most reactivity. Most important is the lack of free silica from norms calculated for the reacted micropegmatite (Fig. 5a). Normative anorthite shows a more or less steady increase (Fig. 5b), while normative albite steadily decreases (Fig. 5c). Normative Kspar may actually peak at about 270°C, becoming more reactive at 310°C (Fig. 5d). Cumulative norms are shown in Fig. 5e.

Nontronite also displays a gross change in composition from the original. Generally, silicon is leached. The resultant product has a more variable iron content than before and possibly higher magnesium. The Si/Al ratio for original nontronite is compared with reacted material in Fig. 6.

Surface analyses by electron microprobe and SEM yielded particularly poor analyses (Table V) from the standpoint of variability and poor totals. The rough surface and possibly variable composition contributed to this problem. A clay was identified on the 72, 119, 161, and 209°C prisms with the composition shown. Total magnesium + iron was near constant at about 5.5, and aluminum + silicon was near constant at about 8, although magnesium, iron, aluminum, and silicon varied about 0.5 atoms over the range of several dozen analyses. Composition and morphology indicate the material to be a smectite with magnetite inclusions. The Si/Al ratio is similar to the interior reacted nontronite mentioned above (Fig. 7).

A different clay, potassium bearing with a different Si/Al ratio than the previous clay, appears on the 270 and 310°C prisms. Also appearing is a phase with prismatic habit with alkali feldspar composition. Sodium and potassium contents for this second phase are variable, and Al/Si was almost 1/3.

IV. DISCUSSION

A. General

All of the primary (original) rock minerals reacted to some extent. Augite, pigeonite, and plagioclase showed clear surface recrystallization to clay. Analyses on the interior of each prism showed these phases to be resistant to

TABLE IV
SUMMARY OF PHASE COMPOSITIONS:
ANALYSES OF THIN-SECTIONED PRISMS (ATOMIC FORMULAS)

Plagioclase (8 ox)							
T (°C)	Na	Ca	K	Al	Si	Fe	
72	0.49±.03	0.53±.04	0.03±.01	1.46±.03	2.49±.04	0.03±.01	
119	0.44±.04	0.51±.06	0.03±.01	1.44±.07	2.51±0.7	0.03±.01	
161	0.45±.03	0.54±.03	0.02±.01	1.48±.03	2.49±.03	0.03±.01	
209	0.43±.01	0.56±.01	0.03±.01	1.50±.01	2.45±.01	0.03±.01	
270	0.43±.01	0.56±.01	0.02±.01	1.47±.01	2.47±.02	0.04±.01	
310	0.43±.03	0.57±.01	0.01±.01	1.50±.01	2.46±.01	0.04±.01	

Augite (6 ox)								
T (°C)	Na	Mg	Al	Si	Mn	Fe	Ca	Ti
72	0.02±.01	0.81±.02	0.07±.01	1.92±.01	0.01±.01	0.51±.02	0.67±.02	0.02±.01
119	--	0.84	0.08	1.90	0.01	0.48	0.70	0.02
161	0.02±.01	0.89±.03	0.07±.01	1.90±.01	0.01±.01	0.42±.03	0.70±.01	0.02±.01
209	--	0.79	0.07	1.92	0.01	0.60	0.60	0.02
270	0.02±.01	0.82±.03	0.08±.01	1.91±.01	0.01±.01	0.53±.02	0.65±.04	0.02±.01
310	0.02±.01	0.86±.02	0.07±.01	1.91±.01	--	0.48±.02	0.69±.01	0.03±.01

Pigeonite (6 ox)								
T (°C)	Mg	Al	Si	Mn	Fe	Ca	Ti	
72	0.94	0.03	1.93	--	0.87	0.21	--	
119	0.80	0.03	1.94	0.02	1.00	0.23	0.02	
161	0.80	0.07	1.89	0.02	0.97	0.27	0.01	
209	0.93	0.03	1.95	0.02	0.84	0.23	--	
270	0.90	0.03	1.93	0.02	0.87	0.25	0.02	
310	1.07	0.03	1.93	0.02	0.76	0.20	--	

TABLE IV (cont)

Matrix: Micropegmatite

Ab = albite, Or = Kspar, An = anorthite, Q = quartz

T (°C)	Ab	Or	An	Cl
Fresh	29	29	2	39
72	46	7	46	1
119	56	20	33	1
161	47	22	30	1
209	18	28	49	1
270	14	35	50	1
310	14	3	83	0

Nontronite (24 (O, OH))

T (°C)	Na	Ca	K	Mg	Fe ⁺²	Ti	Al	Si
72	0.03±.01	0.14±.05	0.02±.01	1.65±.14	4.87±.33	0.02±.01	3.19±.12	5.21±.15
119	0.13±.03	0.20±.03	0.12±.07	1.75±.15	4.93±.33	0.05±.03	3.09±.21	5.34±.21
161	0.23±.01	0.20±.01	0.26±.05	2.00±.18	4.22±.08	0.01±.01	2.62±.03	5.67±.11
209	0.11±.03	0.20±.05	0.05±.04	1.92±.06	4.53±.25	0.03±.01	3.24±.25	5.30±.30
270	0.22±.03	0.19±.06	0.62±.18	1.43±.06	4.71±.23	0.05±.03	2.63±.14	5.80±.02
310	0.22±.03	0.07±.04	0.12±.04	2.33±.17	4.97±.26	0.06±.02	2.92±.11	4.95±.20

alteration. Original nontronite and matrix rapidly recrystallized both at the surface and in the interior of the rock. Nontronite reacts at all temperatures with solution to form smectite similar in composition to that shown in Table V. Matrix, although rare at higher temperatures (270 and 310°C), becomes increasingly anorthitic and is almost completely devoid of free silica even at 72°C.

At temperatures of 72 to 209°C, the entire surface is covered by an aluminum silicate of calcium and magnesium with very variable iron content. SEM examinations suggest the material is a smectite with some enclosed magnetite.

Fig. 5a. Matrix norms.

Fig. 5b. Matrix norms.

Fig. 5c. Matrix norms.

Fig. 5d. Matrix norms.

Fig. 5e. Matrix norms.

Fig. 6. Si/Al for original nontronite and reacted nontronite.

TABLE V
SURFACE ANALYSES

T (°C)	Phase
72, 119, 161, 209	Clay approx. $\text{Ca}_{0.5}\text{Mg}_2\text{Fe}_{3.5}\text{AlSi}_5\text{Al}_3\text{O}_{20}(\text{OH})_4$ "Smeectite"
270, 310	Clay approx. $\text{KMg}_3\text{Fe}_{3.5}\text{Al}_2\text{Si}_6\text{O}_{20}(\text{OH})_4$ "Phengite" Feldspar approx. $\text{Na}_{0.5}\text{K}_{0.5}\text{AlSi}_3\text{O}_8$

Fig. 7. Si/Al for surface alterations.

At 270°C and 310°C, phengite (potassium-rich) clay plus magnetite and sanidine covers principally original plagioclase and pyroxene.

The clays are reasonable phases for the physical conditions of the experiment. Adularia, analcime, and silica presumably would be the stable phases instead of sanidine. Exsolution under these conditions would be quite sluggish. The anorthite matrix will eventually recrystallize to a calcium-zeolite.

Amorphous silica scale and a small amount of sanidine indicate depletion of silica and to some extent potassium and sodium from the original rock matrix.

B. Chemical Geothermometry

The Na-K-Ca, Na-K, and silica geothermometers were empirically developed from hydrothermal systems equilibrated with granitoid rock. Employing these techniques to basaltic systems is hazardous. In the present experiment because of the micropegmatite, we felt the Na-K-Ca geothermometer may have some application. As Table VI shows, this chemical geothermometer yields high temperatures. The silica chemical geothermometer will yield temperatures that are too low. More experimentation must be done to develop a suitable chemical geothermometer for basaltic rock.

C. Phase Relations

This section will attempt to define, in more detail, some of the phase relations occurring on the prisms. The original minerals and secondary minerals are approximated in Table VII. Because of the small amount of pigeonite, pyroxene is simplified as augite.

TABLE VI
CHEMICAL GEOTHERMOMETER TEMPERATURES BASED UPON THE Na-K-Ca GEOTHERMOMETER

<u>Duration</u> <u>(days)</u>	<u>Cold Solution</u> <u>(°C)</u>	<u>Hot Solution</u> <u>(°C)</u>
1	345	--
2	369	364
6	368	375
14	349	367
30	326	340

TABLE VII
COMPOSITIONAL APPROXIMATION USED IN THE SCHREINEMAKER
DEVELOPMENT BASED ON MICROPROBE AND SEM ANALYSES

Phase	Composition
auigite (A)	$\text{Ca}_{0.5}\text{Fe}_{0.5}\text{MgSi}_2\text{O}_6$
plagioclase (P)	$\text{Na}_{0.5}\text{Ca}_{0.5}\text{Al}_{1.5}\text{Si}_{2.5}\text{O}_8$
nontronite (N)	$\text{Ca}_{0.33}\text{Mg}_{1.5}\text{Fe}_{3.5}\text{Al}_{1.33}\text{Si}_{7.33}\text{O}_{20}(\text{OH})_4$
magnetite (M)	Fe_3O_4
phengite (F) (illite)	$\text{KMg}_3\text{Fe}_{3.5}\text{Al}_2\text{Si}_6\text{O}_{20}(\text{OH})_4$
smectite (S)	$\text{Ca}_{0.5}\text{Mg}_2\text{Fe}_{3.5}\text{Al}_4\text{Si}_5\text{O}_{20}(\text{OH})_4$

In this experiment, a rock displaying at least two mineral assemblages out of thermodynamic equilibrium reacts with a fluid initially out of equilibrium with both assemblages. Both rock and fluid change in bulk composition as the experiment proceeds. After about two weeks, a steady state is reached and continues for the duration of the experiment. At this point, there is a transient equilibrium reflected by relatively constant fluid composition causing precipitation of secondary phases uniform in composition (with the exception of iron, which is thought to be due to magnetite crystals included in secondary clay) within a given prism, and from prism to prism with respect to major element content. These reactions are not reversible, but the secondary phases are reasonable for the intensive parameters of the experiment. These reactions may be treated as infiltration metasomatism after the method of Korzhinskii.⁷

The minerals are part of the system $\text{K}_2\text{O}-\text{Na}_2\text{O}-\text{CaO}-\text{MgO}-\text{FeO}-\text{Fe}_2\text{O}_3-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$. K_2O , Na_2O , O_2 , SiO_2 , Al_2O_3 , and H_2O are mobile components, whereas CaO , MgO , and FeO are the inert components. Mobile and inert components may be chosen in any reasonable manner. Because of the relatively low solution concentration of calcium, magnesium, and iron, these elements are chosen as inert. Additional elements will result in a more complex chemographic net but will still contain this subnet. Mobile phases are quartz, water, sanidine (actually adularia plus albite) oxygen, and complexes containing K^+ , Na^+ , and Al^+ . The entire chemographic system consists of three components and six phases (Table VII) and, thus, has one negative degree of freedom. No compositional degeneracies exist for this system. All 6 invariant assemblages, 15 univariant assemblages, and 20 divariant assemblages exist.

A connected net for this system is shown in Fig. 8. All divariant fields are labeled. Important divariant assemblages are smectite bearing, phengite bearing, and non-hydrous phase bearing (high temperature). Some observations limit the pertinent assemblages with respect to this experiment.

- (1) Magnetite appears to be present with smectite and phengite.
- (2) Smectite and phengite do not appear together.
- (3) Nontronite never appears in the reacted rock.
- (4) Augite is metastable at low temperature.

These observations lead to a low-temperature assemblage of magnetite, plagioclase, and smectite; a moderate-temperature assemblage of magnetite, plagioclase, and phengite; and a high-temperature assemblage of magnetite, plagioclase, and augite.

The two possible nets oriented in $\mu_{\text{H}_2\text{O}} - \mu_{\text{SiO}_2}$ space are shown in Figs. 9 and 10. Only the important phase assemblages are labeled in these figures. One possible reaction path is labeled. This is the only region on the two diagrams that presents the proper sequence of reaction from more to less hydrous phases with relatively constant silica content.

V. CONCLUSIONS

Sentinel Gap basalt reacts readily at low temperature with water to produce an assemblage including clay minerals throughout the range of experimentation. These minerals may be useful as adsorbers for material implanted in the basalt. Sorption capabilities should be investigated with specific ions in mind.

Fig. 8. Connected net and compositional polyhedron.

Fig. 9. Net oriented in $\mu_{\text{H}_2\text{O}} - \mu_{\text{SiO}_2}$ space with [S] to [F] homotypical metastable.

Fig. 10. Net oriented in $\mu_{\text{H}_2\text{O}} - \mu_{\text{SiO}_2}$ space with [S] to [F] homotypical stable.

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APPENDIX

PHOTOMOSAICS OF BASALT

The following figures are scanning electron photomosaics of basalt prisms in various stages of the reaction process.

Fig. A-1. SEM photomosaic of a portion of a polished unreacted basalt prism.

Fig. A-2. SEM photomosaic of a portion of a prism reacted at 72°C.

72 ° C

72 ° C

Fig. A-3. Secondary placey material grown at 72°C.

Fig. A-4. Higher magnification of the platey aggregates showing more complete coverage of the rock surface reacted at 119°C.

Fig. A-5. SEM photomosaic of a portion of a prism reacted at 161°C.

Fig. A-6. SEM photomosaic of a portion of a prism reacted at 209°C.

Fig. A-7. SEM photomosaic of a portion of a prism reacted at 270°C showing strikingly different overgrowth morphology.

Fig. A-8. SEM photomosaic of a portion of a prism reacted at 310°C showing slightly better crystallization of the feldspars.

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