American Mineralogist, Volume 65, pages 306–312, 1980

Volatiles in Ti-rich amphibole megacrysts, southwest USA[1]

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Abstract

Ferro-kaersutite and kaersutite megacrysts (3.8 to 5.8 wt.% TiO_2) from peridotite-bearing alkalic basalt lavas and tuffs from near Hoover Dam, Peridot Mesa, Soda Springs, and Vulcan's Throne in Arizona, Salt Lake Crater in Hawaii, and Lunar Craters in Nevada were analyzed by electron microprobe and high-temperature mass spectrometry. During heating at 5°C/min all volatiles (H_2O, CO_2, F, Cl, S) were released between 950° and 1050°C under a vacuum of 1×10^{-8} torr. Total volatile loss ranged from 0.9 wt.% for high F-content amphiboles (0.225 wt.% F) to 1.9 wt.% for low F-content amphiboles (0.070 wt.% F). H_2O is the predominant volatile constituent of all amphiboles, varying inversely with F content, and ranges from 0.487 to 1.578 wt.%. S and Cl contents are low and show no apparent correlation in abundance with either H_2O or F. S ranges from 0.001 to 0.052 wt.%; Cl varies from 0.001 to 0.012 wt.%. F content varies proportionally with TiO_2 content. No other correlation between volatile and nonvolatile amphibole constituent was observed. CO_2 contents range from 0.095 to 0.337 wt.%. Rapid heating and quenching of amphibole in the mass spectrometer produced CO_2^+-ion spikes, which indicates that CO_2 is trapped in the amphibole as minute gas inclusions. The relatively low F/H_2O contents of Ti-rich amphiboles examined in this study indicate that F does not play a major role in extending their thermal stability.

Introduction

The presence or absence of a hydrous phase in the upper mantle is a fundamental point of models for magma genesis and tectonic activity. Numerous studies have investigated the limiting P–T–X factors controlling amphibole stability (Lambert and Wyllie, 1968; Hariya and Terada, 1973; Holloway, 1973; Merrill and Wyllie, 1975; Yagi *et al.*, 1975; Mysen and Boettcher, 1976; Popp *et al.*, 1977a,b; Allen and Boettcher, 1978). Amphibole stability is governed not only by the presence of volatiles, but also by the species present (Holloway, 1973; Gilbert and Briggs, 1974; Holloway and Ford, 1975; Allen and Boettcher, 1978). In particular, fluorine substitution for OH markedly increases the thermal stability of amphibole and results in a substantial increase in the melting interval (Holloway and Ford, 1975; see Fig. 1). Unfortunately, chemical analyses of amphiboles in the literature generally report only the nonvolatile constituents. In rare cases, fluorine is reported.

The purpose of this investigation is to provide quantitative data on the character and abundance of volatiles in Ti-rich amphiboles of probable mantle origin. Mantle-derived amphiboles may be present in alkalic basalts as poikilitic grains, veins, and interstitial grains within inclusions of chromium–spinel peridotites, and as megacrysts (Best, 1974). In the present study, only megacrysts were examined.

The origin of kaersutitic megacrysts is problematic. Cross and Holloway (1974) suggested that kaersutitic megacrysts from San Carlos, Arizona, crystallized from the host magma, basing this on the euhedral nature of the megacrysts and on the presence of apatite inclusions. Basu (1978) has shown that kaersutitic megacrysts from several localities, in-

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[1] Hawaii Institute of Geophysics Contribution No. 1012.

0003-004X/80/0304-0306$02.00

cluding Hoover Dam, Arizona, are genetically unrelated to their host basalts. $^{87}Sr/^{86}Sr$ ratios of the kaersutites are lower (0.70246 to 0.70311) than host basalts (0.70350 to 0.70420). Best (1974) documented a similarity in major-element composition of kaersutitic megacrysts and vein and poikilitic kaersutites in ultramafic xenoliths. In addition, a kaersutite from a vein in an ultramafic nodule from Dish Hill, California, has the same $^{87}Sr/^{86}Sr$ ratio as three kaersutite megacrysts from the same locality. This evidence suggests that kaersutitic megacrysts may represent disaggregated parts of kaersutitic veins in ultramafic rocks (Best, 1974; Basu, 1978; Wilshire *et al.*, in press).

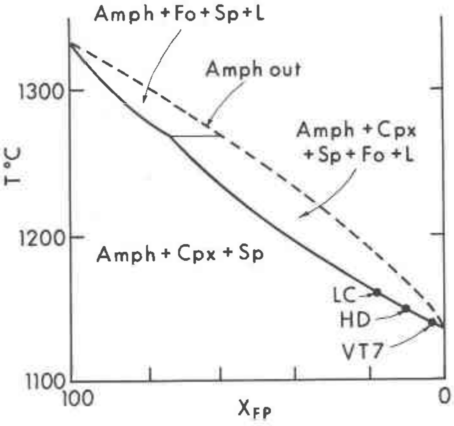

Fig. 1. Schematic *T-X* projection of the fluoro-pargasite–hydroxy-pargasite system at 15 kbar (from Holloway and Ford, 1975). X_{FP} is the mole fraction of fluoro-pargasite. Amphiboles from the present study are plotted to indicate their possible thermal stability limit: LC–Lunar Craters; HD–Hoover Dam; VT7–Vulcan's Throne 122377.

Samples

Samples from six localities in the southwest United States of America were used in this study.

Hoover Dam, Arizona

Nine miles south of Hoover Dam along U.S. Highway 93, camptonite dikes cut fanglomerates of probable Quaternary age (Campbell and Schenk, 1950). This locality is characterized by amphiboles of exceptional size (up to 15 cm in length) and embayed grains of andesine plagioclase, augite, and partially to completely altered olivines, plus rare 1–2 cm diameter xenoliths of spinel lherzolite.

Lunar Craters, Nevada

Lherzolite, wehrlite, dunite, pyroxenite, and gabbro xenoliths and amphibole, clinopyroxene, olivine, and anorthoclase megacrysts are common in this Pleistocene to Holocene age basanite flow from east-central Nevada (Trask, 1969; Holloway, 1978, written communication).

Peridot Mesa, Arizona

The Peridot Mesa samples are from the central cone of the San Carlos volcanic field. Dikes from San Carlos volcanic field intrude Plio-Pleistocene lake beds and conglomerates. The basanite flow from Peridot Mesa contains abundant ultramafic and mafic nodules, including spinel lherzolite, harzburgite, websterite, pyroxenite, wehrlite, and gabbro (Frey and Prinz, 1978). Also present are megacrysts of clinopyroxene, spinel, anorthoclase, and kaersutite which may be cognate (Wohletz and Patera, 1978).

Salt Lake Crater, Hawaii

This vent, part of the Honolulu Volcanic Series of Pleistocene to Recent age (Jackson and Wright, 1970), is known for its garnet-bearing ultramafic xenoliths in nephelinitic tuff (Beeson and Jackson, 1970). The nodule suite includes garnet websterite, garnet lherzolite, spinel lherzolite, wehrlite, harzburgite, and dunite (Jackson and Wright, 1970). Rare megacrysts of pyroxene, kaersutite, and dark mica are also present.

Soda Springs, Arizona

Megacrysts of kaersutite and anorthoclase are common in the iron-rich alkalic lava at this locality on the San Carlos Apache Indian Reservation. The basalt outcrop is small (~100 m in diameter), circular, and consists of fragmental, scoriaceous lava and volcanic bombs. The megacrysts are euhedral and contain small euhedral inclusions of kaersutite and apatite in anorthoclase and apatite in kaersutite, which may be the result of cotectic crystallization from the host magma (Cross and Holloway, 1974).

Vulcan's Throne, Arizona

Vulcan's Throne is a Holocene volcano located on the north rim of the Grand Canyon. A basanitic lava from a nearby vent, less than a few thousand years old, is the host for ultramafic inclusions and megacrysts. Spinel lherzolites are the predominant inclusion type; amphibole-bearing peridotites are rare (Best, 1975). Amphibole and clinopyroxene are common megacrysts; orthopyroxene and olivine megacrysts are rare (Best, 1970).

Experimental methods and results

High-temperature mass spectrometry

After crushing, elongate amphibole fragments (0.2–5 mm) were hand-picked with the aid of a binocular microscope. Unaltered fragments were immersed in 0.3 *N* HCl and washed in distilled water with an ultrasonic vibrator. Drying at 110°C completed sample preparations for degassing.

The volatile content and degassing behavior of all samples were obtained with a high-temperature mass spectrometric facility consisting of an effusion–vaporization source interfaced with a computer-controlled quadrupole mass spectrometer. Details of the Knudsen cell vaporization source and automated data acquisition system have been previously described (Muenow, 1973; Liu and Muenow, 1978). From this source a molecular beam of released volatiles is generated which is then directed through a movable shutterplate and into the ion source of the mass spectrometer. This configuration permits 100% transmission of volatiles from the sample to the ionizer. Use of the shutterplate allows distinction between molecular species emitted directly from the sample within the Knudsen cell and those desorbed or scattered from other surfaces of the instrument. The mass spectrometer is monitored by a PDP-11/45 minicomputer. Temperature is measured with a thermocouple secured within the base of the cell, and is also monitored by the computer.

The mass spectrometer was operated in a rapid-scan mode to obtain mass pyrograms. Detailed procedures of this technique have been previously described (Killingley and Muenow, 1975a; Delaney *et al.*, 1978). Samples (40–60 mg) were weighed and placed in a prebaked alumina cell liner and heated under a vacuum of 1×10^{-8} torr at a rate of approximately 5°/min to 1250°C. During the entire degassing period the mass spectrometer rapidly scans the spectrum, and ion-current signals for all mass peaks and temperatures are measured and stored on magnetic tape. Mass pyrograms were obtained as hard-copy computer plots of the intensities of parent molecular ions (or principal fragment ions) *vs.* temperature. System background contributions are determined by use of the shutterplate and from "blank" runs made before and after each sample study. When degassing of each sample is completed, the sample is cooled and reweighed. The sensitivity of the mass spectrometer was determined by calibration procedures based on the quantitative vaporization of a known amount of a standard (Grimley, 1967).

A mass pyrogram showing the typical gas-release behavior of the principal volatiles H_2O, CO_2, and F is given in Figure 2. Ion intensities, plotted along the vertical axis, are proportional to the partial pressures of each volatile, and the area under each curve is proportional to the total amount of each volatile released over the time–temperature interval. Each curve contains about 200 data points over a vaporization period of approximately 4 hours. As seen in Figure 2 release of volatiles from the amphiboles is confined to the narrow temperature range, 950°–1050°C. (The slowly rising baseline intensities are due to

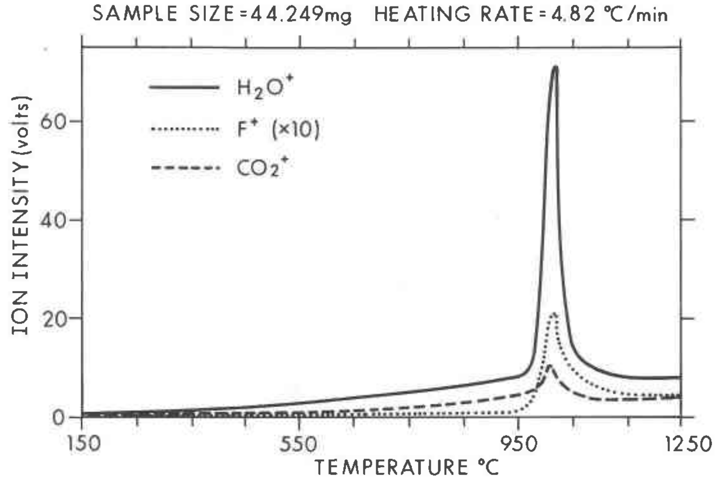

Fig. 2. Typical mass pyrogram showing gas release behavior of principal volatiles (H_2O, F, CO_2) in amphibole (Hoover Dam, NMNH 134924). Ion intensities and area under curves are proportional to partial pressures and total amount of volatile released, respectively.

small contributions from background gases within the instrument.) Two analyses were made for each sample and mass pyrograms for replicate samples were virtually identical. The volatile data are summarized in Table 1. Entries have been arranged from top to bottom in order of decreasing water (or increasing fluoride) concentrations. Total volatile loss ranges from 0.9 to 1.9 wt.%.

To determine whether the CO_2 detected in the amphiboles was present as minute fluid inclusions, the samples were rapidly heated to 900°C, just below the volatile release temperature and melting point, and then cooled quickly to induce differential thermal expansion and contraction of any fluid inclusion and the host amphibole. Previous studies (Killingley and Muenow, 1974, 1975b; Delaney *et al.,* 1978; Garcia *et al.,* 1979; Muenow *et al.,* 1979) on samples containing CO_2 fluid inclusions in phenocrysts in basalts have shown that on quenching samples rapidly heated to high temperature, CO_2 is released in bursts and observed as ion-current signal spikes (of mass 44, parent peak of CO_2) on the oscilloscope of the mass spectrometer and strip-chart pen recorder. In this study the monitored ion-current for mass 44 showed "spike"-type behavior at sample temperatures from 840° to 900°C (below the melting point) and on quenching to low temperatures. No other mass peaks showed this behavior.

Microprobe

Analyses were made with the automated UCLA ARL-EMX electron microprobe with natural and synthetic mineral standards. Both internal and external standards were used; the internal standard was a well-analyzed hornblende. Instrument operating conditions were usually 15 kV and 25 nA sample current. Each analysis is an average of six to eight points from a small area and three to six grains per sample. Core and rim analyses were made to check for zoning and overall homogeneity. Raw data were corrected for detector dead time, instrument current drift, and spectrometer background. Mineral analyses were then obtained by the corrections methods of Bence and Albee (1968) and Albee and Ray (1970). Accuracy is estimated to be 1–2% for major elements and 5–10% for minor elements, on the basis of replicate microprobe analyses compared with wet-chemical analyses. Results are given in Table 2.

Most of the amphiboles were essentially homogeneous, although several grains were strongly zoned.

Table 1. Amphibole volatile abundances determined by mass spectrometry (wt. %)

Sample	Total Volatile Loss	H_2O	CO_2*	F	Cl	S	F/H_2O (mole-ratio)
Vulcan's Throne (NMNH-122377)	1.73	1.578	0.107	0.070	0.007	0.018	0.05
Vulcan's Throne (NMNH-122378)	1.76	1.485	0.188	0.073	0.002	0.013	0.05
Salt Lake	1.87	1.467	0.307	0.085	0.010	0.001	0.06
Hoover Dam (NMNH-106434)	1.82	1.452	0.240	0.104	0.009	0.018	0.07
Soda Springs	1.39	1.180	0.095	0.093	0.012	0.019	0.08
Hoover Dam (NMNH-134924)	1.40	1.085	0.126	0.126	0.001	0.052	0.12
Peridot Mesa	1.26	1.003	0.141	0.115	0.002	0.018	0.12
Hoover Dam	1.57	0.993	0.337	0.189	0.006	0.020	0.18
Lunar Craters	0.93	0.487	0.209	0.225	0.009	0.001	0.44

Precision ± 10%.

*Principally gas inclusions. See text for discussion.

Table 2. Microprobe analyses of Ti-rich amphibole megacrysts

	Vulcan's Throne		Salt Lake	Soda Springs	Hoover Dam			Lunar Craters	Peridot Mesa
	122377	122378			1	134924	106434		
SiO_2	42.03	41.68	41.19	40.24	40.46	40.55	40.36	40.22	39.52
TiO_2	3.86	3.81	4.33	5.03	5.73	5.78	5.49	5.85	4.77
Al_2O_3	15.32	14.73	13.44	13.05	13.77	14.59	14.35	13.43	15.19
Cr_2O_3	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.00	0.1
FeO^t	7.68	8.65	11.81	15.45	8.98	8.63	8.69	11.67	12.62
MnO	0.07	0.05	0.15	0.29	0.12	0.07	0.08	0.15	0.1
MgO	14.99	13.89	11.09	9.48	13.00	13.74	13.58	11.29	10.84
CaO	10.30	10.24	10.77	10.54	11.32	11.03	10.93	10.86	10.42
Na_2O	2.83	2.99	2.98	2.98	2.51	2.53	2.47	2.74	3.09
K_2O	1.59	1.63	2.02	1.24	1.95	1.96	2.00	1.43	1.46
F	—	0.06	—	—	—	0.14	—	—	—
Total	98.68	97.74	97.79	98.33	97.85	99.03	97.96	97.64	98.11
					Cation Norm Based on 23 Oxygens				
Si	6.02	6.07	6.12	6.03	5.94	5.87	5.90	6.03	5.86
Al^{IV}	1.98	1.93	1.88	1.97	2.06	2.13	2.10	1.97	2.14
Al^{VI}	0.60	0.60	0.47	0.34	0.32	0.26	0.37	0.34	0.52
Ti	0.42	0.42	0.48	0.57	0.63	0.63	0.60	0.57	0.53
Fe	0.92	1.05	1.47	1.94	1.10	1.04	1.06	1.94	1.57
Mn	0.01	0.01	0.02	0.04	0.01	0.01	0.01	0.04	0.02
Mg	3.20	3.01	2.45	2.12	2.84	2.96	2.96	2.12	2.40
Ca	1.51	1.60	1.74	1.69	1.78	1.71	1.71	1.69	1.66
Na	0.79	0.84	0.86	0.87	0.71	0.71	0.70	0.87	0.89
K	0.29	0.30	0.38	0.24	0.37	0.63	0.37	0.24	0.28
F	—	0.03	—	—	—	0.06	—	—	—
Total	15.74	15.86	15.87	15.81	15.78	16.01	15.78	15.81	15.87

From core to rim Al, Fe, Na, Mn, and Ti decrease, Si, Ca, and Mg increase, and K remains nearly constant. This chemical variation is unlike that observed in amphiboles in ultramafic xenoliths, in which Ti, Fe, and K increase and Mg, Si, Na, and Cr decrease toward the rind of the xenolith (Wilshire *et al.*, 1971; Stewart and Boettcher, 1977).

Although only two F analyses were made by microprobe, they are very similar to the high-temperature mass spectrometer analyses (*i.e.*, probe 0.06 and 0.14 wt.%; mass spectrometer 0.073 and 0.126 wt.%).

Discussion

MacRae (1979) suggests that during ascent of hot magma a CO_2-rich vapor phase boils off and penetrates minerals, resulting in ubiquitous CO_2 fluid inclusions. Gutmann (1974) found numerous tubular voids in labradorite phenocrysts in a basaltic lava from northwest Sonora, Mexico. He interpreted them as fluid inclusions that nucleated as fluid bubbles on the faces of growing crystals. Tubicles of anorthoclase and calcite were reported in kaersutite megacrysts from the Kakanui Mineral Breccia of New Zealand. The calcite and anorthoclase were interpreted as secondary infillings in tubicles that originally formed as fluid inclusions (Wallace, 1977).

In this study, CO_2 was released from the amphiboles during heating at the same temperature as other volatiles (see Fig. 2) or as vapor "spikes" induced by rapid heating at 900°C and quenching of the amphiboles. We interpret the CO_2 spikes as evidence for minute CO_2 fluid inclusions rather than carbonate impurities. It is extremely unlikely that the structural site occupied by OH^- could accommodate CO_3^{2-}, because the size discrepancy between the two radicals is too great. Calcite occurs in fractures in some of the amphiboles; however, samples with visible calcite were carefully avoided. If calcite were present in the amphiboles, it would release CO_2 at a temperature much lower than the observed volatile release of the amphiboles.

The TiO_2 content of the amphiboles examined varies proportionally with the F content up to 0.12 wt.% F and 5.5 wt.% TiO_2 (Fig. 3). For amphiboles with F

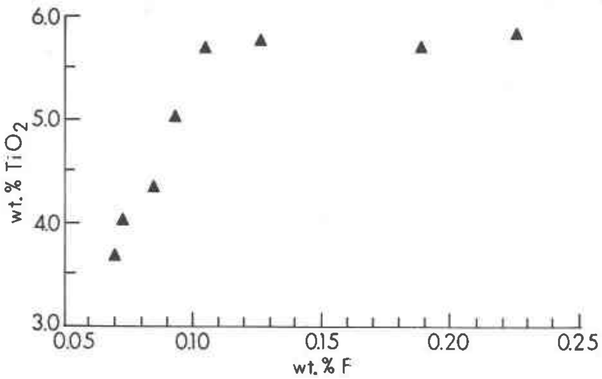

Fig. 3. Variation of TiO_2 and F contents in Ti-rich amphibole megacrysts.

contents greater than 0.12 wt.%, TiO_2 contents remain at values of approximately 5.5 wt.%. This trend of increasing F content with TiO_2 content is consistent with data on Ti-rich amphibole megacrysts in alkalic basalts reported by Boettcher and O'Neil (in press). Both TiO_2 (Merrill and Wyllie, 1975) and F (Holloway and Ford, 1975) extend the thermal stability range of amphiboles. No other trend is apparent between nonvolatile and volatile components of the amphiboles studied.

Substitution of F^- for OH^- in the amphibole structure increases the thermal and pressure stability of amphibole (Holloway and Ford, 1975). In the amphibole megacrysts examined, F is a minor constituent (2–10%) in the OH structural site, except in the sample from Lunar Craters (18%). With such a minor substitution of F for OH, the thermal stability of the amphiboles may be raised only approximately 5° to 10° (and up to 25°C for the Lunar Crater amphibole), if the work of Holloway and Ford (1975) on the pargasite–fluorpargasite system is applicable to the kaersutite–fluorkaersutite system (see Fig. 1). Thus, these results indicate that in naturally-occurring Ti-rich amphiboles, F does not play a major role in extending the stability of amphibole.

Acknowledgments

Samples for this study were generously provided by William G. Melson (from the National Museum of Natural History collection) and John R. Holloway. We thank the Earth and Space Sciences Department at UCLA for access to their microprobe, and Robert Jones for the F analyses determined by microprobe and several of the amphibole analyses. We are grateful to M. G. Best, D. H. Eggler, W. G. Ernst, J. R. Holloway, and John M. Sinton for their comments on an early draft of this manuscript. This work was sponsored by NSF grant EAR78-14776.

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Manuscript received, August 6, 1979;
accepted for publication, October 26, 1979.