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ELECTRON MICROPROBE EVALUATION OF TERRESTRIAL BASALTS FOR WHOLE-ROCK K-Ar DATING*

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Four basalt samples for whole-rock K-Ar dating were analyzed with an electron microprobe to locate potassium concentrations. Highest concentrations of potassium were found in those mineral phases which were the last to crystallize. The two reliable samples had potassium concentrated in fine-grained interstitial feldspar and along grain boundaries of earlier formed plagioclase crystals. The two unreliable samples had potassium concentrated in the glassy matrix, demonstrating the ineffectiveness of basaltic glass as a retainer of radiogenic argon. In selecting basalt samples for whole-rock K-Ar dating, particular emphasis should be placed on determining the nature and condition of the fine-grained interstitial phases.

1. Introduction

The usefulness of the whole-rock K-Ar technique for dating mafic volcanic rocks, particularly basalt, has been well established (see, for example [1–4]). Critical to the success of this technique are the petrographic criteria used to select samples that will give meaningful calculated ages. Although these criteria have been discussed briefly by several authors [1, 5–9] and their validity confirmed by successful use, they are largely decuded from petrologic considerations and have not been independently investigated to any significant degree.

We have used an electron microprobe to determine the location of potassium in four basalts whose reliability for whole-rock K-Ar dating was not only apparent upon petrographic examination but could be established independently. Two of these basalts give reliable whole-rock ages and two do not. Our findings reaffirm the validity of the normal selection criteria and demonstrate that particular emphasis should be placed on determining the nature and condition of the fine-grained interstitial phases when assessing a basalt for whole-rock dating.

Standard petrographic thin sections, 1″ in diameter and ground to a high surface polish, were used for this study. Areas of each section representative of the whole rock were selected, photographed, and analyzed with an Applied Research Laboratories electron microprobe operated at 15 kV accelerating voltage. X-ray scanning images of the potassium K_α radiation were obtained using an ADP diffracting crystal and oscilloscope-mounted polaroid camera. Sample current and calcium K_α scanning images were also made and photographed to assist in mineral identification and location. The K-Ar age measurement for sample 509-69-7 was made using flame photometry with a lithium internal standard for potassium, isotope dilution for argon, and techniques summarized by Dalrymple and Lanphere [9].

2. Summary of petrographic criteria

In principle, a rock that is composed entirely of phases that are good K-Ar geochronometers should itself be a good geochronometer. Practical application of this principle requires that samples for whole-rock dating be sufficiently coarse grained that every com-

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ponent can be identified. For this reason alone, basalts whose groundmass is clouded and obscured by cryptocrystalline opaque Fe-Ti oxides are usually considered unsuitable for whole-rock dating. Ideally, every phase in a prospective sample should be free of alteration, completely retentive of radiogenic argon, and primary, i.e., originating at the time the rock crystallized.

The critical question is, where is the potassium, and is it in components that will have retained the argon that has been generated since the rock crystallized? From petrologic considerations, most of the potassium should be found in the last phases to solidify, and the few observations made with an electron microprobe confirm this [5, 9], except in the case of some Hawaiian tholeiites with unusually low ($\approx 0.1\%$) K_2O contents [8]. The fine-grained interstitial phases are the ones most likely to contain the potassium and argon and are therefore the phases whose freshness and argon retention properties are most likely to influence the reliability of the age measurement. In most basalts, these critical phases are fine-grained feldspar and/or glass.

Alteration probably is one of the most common bases for rejecting a basalt for dating. The problems with alteration are threefold: (1) alteration may occur after initial crystallization of the rock, and the altering process may release all or part of the radiogenic argon that has accumulated; (2) the argon retention properties of alteration products are largely unknown; and (3) the alteration products often are not primary and, even if retentive, will not record the time of formation of the rock. The degree to which alteration will affect the apparent K-Ar age of a rock is a direct function of the amount of potassium that that phase contains relative to the rock as a whole. Alteration of a mineral such as olivine and possibly pyroxene, which usually contain a negligible amount of K_2O compared with the total rock, will not adversely affect the whole-rock K-Ar age [2, 3, 6]. Studies by Webb and McDougall [10] and McDougall et al. [2] have suggested that some alteration of interstitial material may not result in argon leakage, but with the exception of minerals with negligible K_2O content, it is not now possible to determine the amount or type of alteration that is tolerable. Such samples probably should be avoided whenever possible, for it is better to reject a suitable sample than to date an unsuitable one.

With few exceptions, argon retention properties are known for only the most common minerals. A potassium-bearing phase that is widely found in many mafic volcanic rocks is glass, and the ability of basaltic glass to retain argon quantitatively is largely unknown. For this reason, basalt which contains glass is usually considered unsuitable for dating. Some basalts that contain small amounts of fresh glass seem to give reliable ages [2, 10], but there are numerous examples of basaltic glasses that do not [9]. A method for evaluating the retentivity of glasses and other volcanic rocks by determining the amount of hydration recently described by Kaneoka [11] indicates that $H_2O(+)$ contents greater than 1% may be accompanied by loss of radiogenic argon. In general, however, it is difficult to distinguish between retentive and nonretentive glasses or to say what percentage of glass can be tolerated; samples that contain glass are therefore not normally used for whole-rock dating. Devitrified glass also is considered unsuitable for the reasons given for the unsuitability of alteration products. Other materials of uncertain argon retentivity that may be found in basalt are zeolites, most feldspathoids, clay minerals, and various forms of secondary mineralization.

For obvious reasons, potassium-bearing minerals that did not form at the same time as the rock in which they occur are undesirable in a sample used for whole-rock dating. In this category are vein or vesicle fillings, xenoliths or xenocrysts, and many alteration or devitrification products. Most such materials would tend to make the apparent K-Ar age younger than the true age, but xenoliths and xenocrysts can have the opposite effect because they are commonly a source of inherited (by contamination) ^{40}Ar.

3. Results

The geologic settings of the four samples studied have been described in previous publications, as have most of the age data, summarized in table 1.

Sample 509-62-5 is from an olivine basalt flow that caps the Benton Range in Mono County, California [12]; its K-Ar age has been studied intensively [13]. K-Ar age measurements on seven samples collected over a distance of 3 km gave an age of 3.32 ± 0.07 my (standard deviation) despite differences of as great as 20% in K_2O content. Primary biotite formed as a vesicle lining during the late stages of crystallization was

Table 1
Apparent and control ages of samples studied.

Sample no.	Material analyzed	Apparent K-Ar age* (10^6 yr)	Control age (10^6 yr)	Ref.
		Reliable		
509-62-5	whole-rock	3.4 ± 0.1	3.3 ± 0.1	[13], [14]
A-11	whole-rock	4.88 ± 0.2	4.83 ± 0.1	[4]
		Unreliable		
509-64-2	whole-rock	11.6 ± 0.6	>22.2 ± 0.4	[7]
509-69-7	crushed basalt matrix	**1.6 ± 0.1	7.4 ± 0.2	[7]

* The ± figures are standard deviations of precision. $\lambda_\epsilon = 0.585 \times 10^{-10}$ yr^{-1}, $\lambda_\beta = 4.72 \times 10^{-10}$ yr^{-1}, $^{40}K/K_{total} = 1.19 \times 10^{-4}$.
** % K_2O = 2.69, 2.67; $^{40}Ar_{rad}/g = 6.15 \times 10^{-12}$ mol; $^{40}Ar_{rad} \times 100/^{40}Ar_{total} = 61.7$.

concentrated from one sample; it gave an age of 3.17 ± 0.30 my. Twelve age measurements on the hand sample from which the microprobe section was prepared have a mean of 3.42 ± 0.06 my and a recent measurement by the $^{40}Ar/^{39}Ar$ technique gave 3.36 ± 0.48 my [14]. This rock is a massive, nonvesicular, holocrystalline porphyritic olivine basalt containing phenocrysts of olivine and pyroxene and an intergranular groundmass of feldspar, olivine, pyroxene, and opaque Fe-Ti oxides. There is no trace of any alteration or secondary mineralization, and all grain boundaries are well defined and free of cryptocrystalline material. The potassium in this rock (fig. 1a) is concentrated in an interstitial feldspar, probably anorthoclase or sanidine, that constitutes 20% or less of the rock by volume. There is no detectable K_2O in the plagioclase phenocrysts. Clearly, the suitability of this rock for whole-rock K-Ar dating is due almost entirely to the fresh condition of this interstitial feldspar and very little to the condition or nature of the major mineral phases.

Sample A-11 is from one of four superposed flows on Nunivak Island, Alaska, that record a reversed to normal polarity transition of the earth's magnetic field [4]. This sample, the uppermost of two reversely magnetized lavas, gives a whole-rock age of 4.88 ± 0.2 my. It is directly overlain by two normally magnetized lavas, the lower of which gives a whole-rock age of 4.83 ± 0.1 my. Ages of 4.88 ± 0.2 my (normal polarity) and 4.84 ± 0.2 my (reversed polarity) were obtained on two nearby flows that record the same polarity transition. This rock is massive, coarse grained, holocrystalline, porphyritic olivine basalt with phenocrysts of olivine and plagioclase set in an intergranular groundmass of plagioclase laths, pyroxene, olivine, and opaque Fe-Ti oxides. Some olivine phenocrysts show rims of iddingsite; otherwise the rock is completely unaltered. All grain boundaries are well defined and completely free of cryptocrystalline material. Although individual plagioclase grains contain some potassium (fig. 1b), most is in the rims of groundmass feldspar laths, along the boundaries of plagioclase phenocrysts, and, in late-stage, irregular patches of interstitial potassium feldspar. As with the previous sample 509-62-5, the potassium in this rock is concentrated largely in high-potassium feldspar that occupies a volume of less than 10% of the rock; its suitability for whole-rock dating is mainly a function of the condition of this interstitial feldspar rather than the major mineral phases.

Sample 509-64-2 is from a basalt flow in the type section of the Lovejoy Basalt, Sierra Nevada, California [15]. The age of the Lovejoy is limited by a K-Ar age of 22.2 ± 0.4 my on sanidine from a rhyolite tuff that overlies Lovejoy basalts at the Lovejoy type section and a date of 23.8 ± 0.6 my on plagioclase from a dacite vitric tuff that occurs about 30 m below Lovejoy basalt flows at Oroville Table Mountain [7].

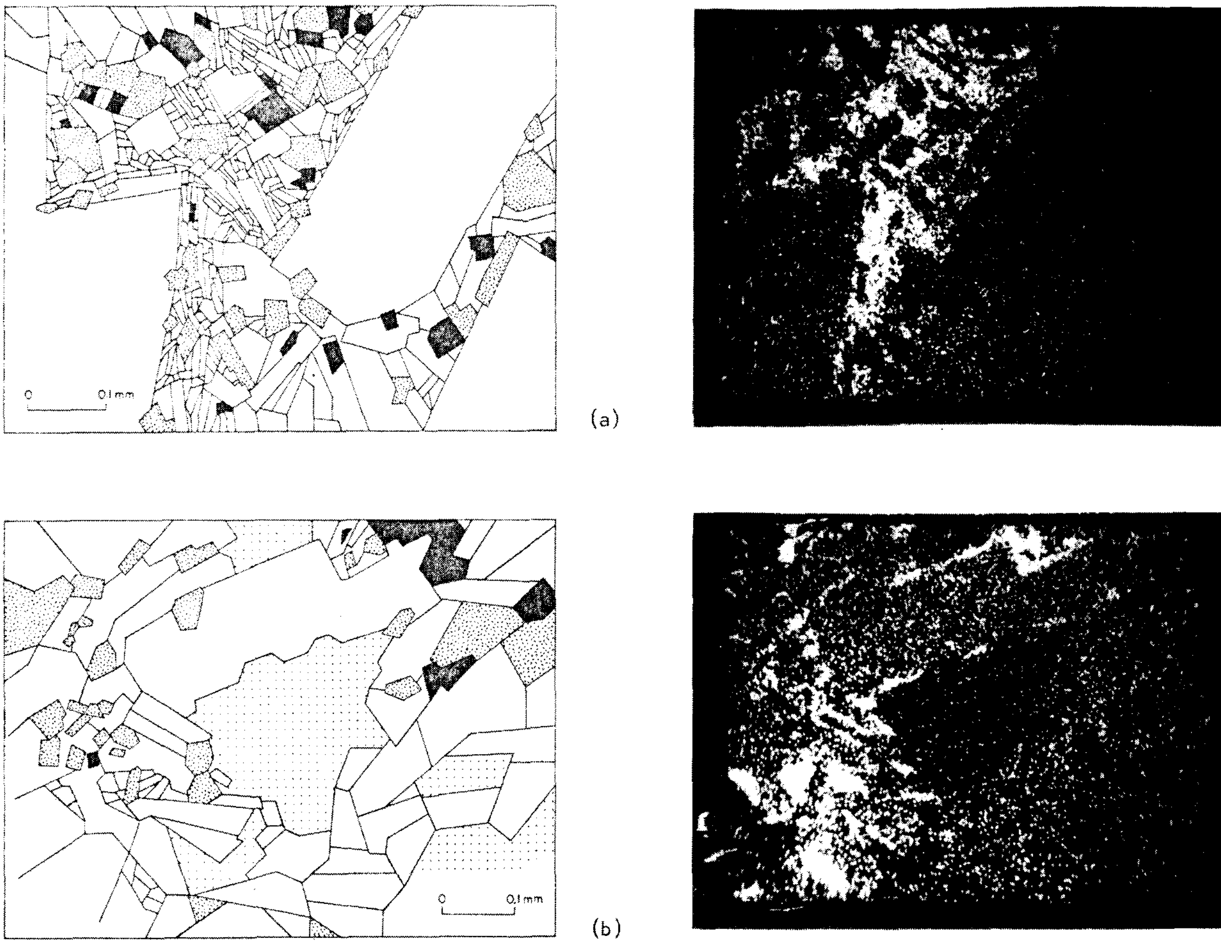

Fig. 1. Thin section sketches (left) and electron microprobe potassium K_α scanning images (right) showing the distribution of potassium in two samples considered reliable for whole-rock K-Ar dating. Dark grains, magnetite; heavily stippled pattern, olivine and pyroxene; clear portions, plagioclase laths; lightly stippled pattern, cavities in the rock caused by diktytaxitic texture. (a) Sample 509-62-5; (b) Sample A-11. The scanning images show the potassium is concentrated in the interstitial feldspar and along boundaries of plagioclase phenocrysts.

The whole-rock age of 11.6 ± 0.6 my on sample 509-64-2 is clearly too young. This rock is a dense, nonvesicular, medium-grained basalt with hyalopilitic texture and few phenocrysts of olivine and plagioclase. The groundmass is composed of plagioclase laths, olivine, and pyroxene in a matrix of about 30% clear brown glass. The feldspar is fresh and euhedral, and the groundmass mafic minerals are unaltered; olivine phenocrysts are partly altered. The glass is mostly fresh, transparent, and isotropic, but about 15% is altered to a yellowish birefringent material. The potassium (fig. 2a) appears to be irregularly distributed in feldspar phenocrysts and small feldspar laths and throughout the glass and altered glass matrix. We estimate that more than half of the K_2O occurs in the glass and altered glass, which probably accounts for the anomalously low age for this rock.

Sample 509-69-7 is from the lower of two hypersthene basalt flows capping Boreal Ridge near Donner Pass in the Sierra Nevada, California [16]. Plagioclase from this flow gives a K-Ar age of 7.4 ± 0.2 my [7], whereas the glassy matrix with phenocrysts removed gives an age of only 1.6 ± 0.1 my. Because the matrix is about 75% of the rock, a whole-rock age on

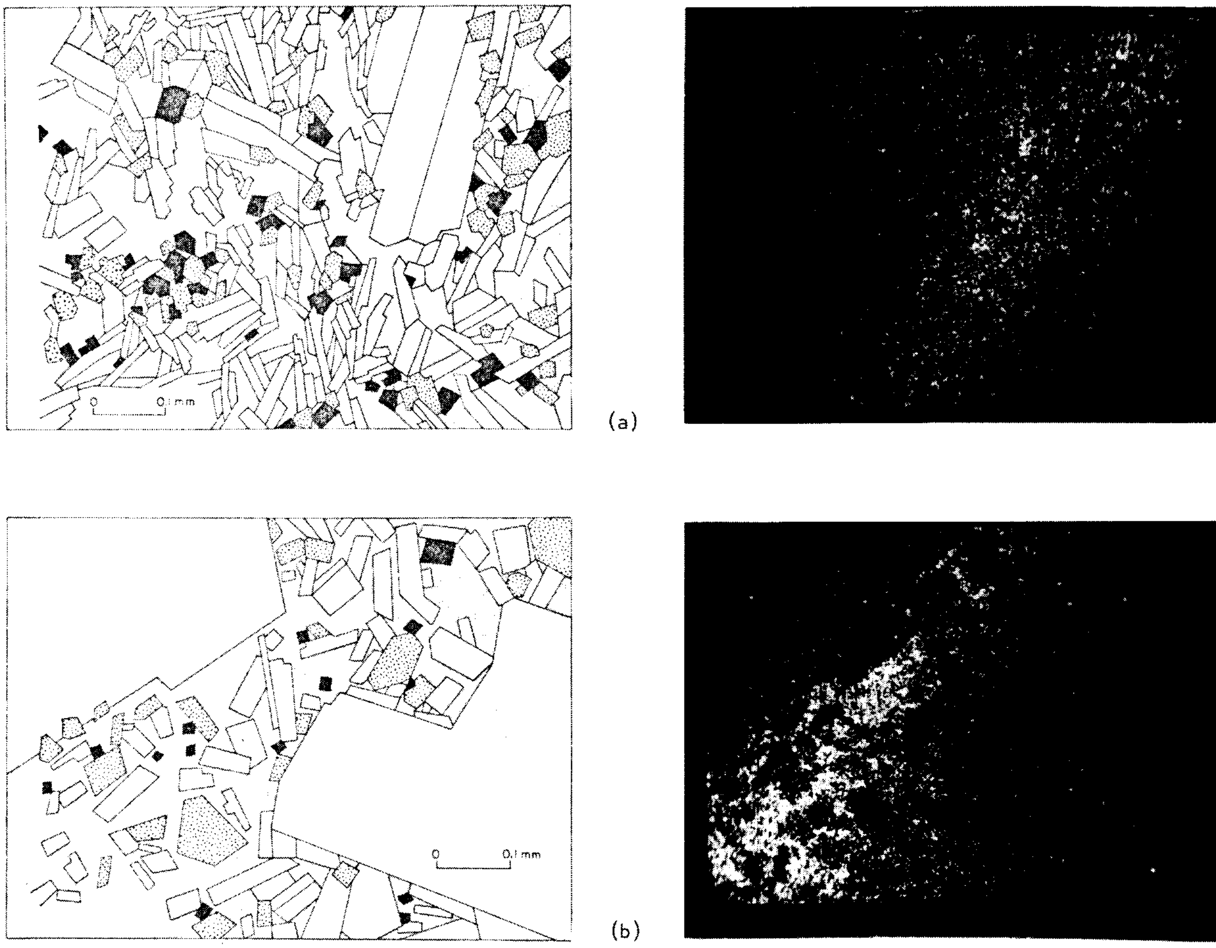

Fig. 2. Thin section sketches (left) and electron microprobe potassium K_α scanning images (right) showing the distribution of potassium in two samples considered unreliable for whole-rock K-Ar dating. Symbols as for fig. 1. (a) Sample 509-64-2; light shading = glass and altered glass. (b) Sample 509-69-7; light shading = fresh glass. The scanning images show the potassium is mainly concentrated in the glassy matrix.

this flow would clearly be too low. This rock is a massive, nonvesicular, porphyritic basalt with hyalopilitic texture. Phenocrysts of plagioclase and olivine are set in a groundmass of plagioclase laths, pyroxene, olivine, and about 30% fresh brown glass. There is no trace of alteration of any of the phases, and the glass is not devitrified. The potassium (fig. 2b) is concentrated almost entirely in the glass, and there is little or no detectable K_2O in the feldspar. The glass in this rock, though completely unaltered, has not retained its radiogenic ^{40}Ar.

4. Conclusions

Potassium in the basalts examined is highly concentrated in the last phases to crystallize, as expected from geochemical considerations and as found in published studies on results with the electron microprobe [5, 9]. Potassium in the two basalts that give reliable ages is in well crystallized and fresh interstitial potassium feldspar, probably anorthoclase or sanidine. The two unreliable basalts have K_2O concentrated mostly in interstitial glass, which has not retained radiogenic ^{40}Ar. Similar potassium concentrations have been found in the lunar basalts and some radiogenic ^{40}Ar

loss is indicated from the interstitial phases in these rocks [17, 18]. These results demonstrate the validity of the petrographic criteria generally used for selecting mafic volcanic rocks for whole-rock K-Ar dating [1, 5–9] and summarized above.

In assessing a rock for K-Ar dating, particular attention should be given to the identity and condition of the interstitial phases, particularly the material along the boundaries of, and between, groundmass feldspar grains. The age information in most basalts is stored largely in these areas. More detailed studies may eventually permit whole-rock K-Ar dating techniques to be reliably extended to partially altered rocks and rocks containing glass. With the present state of knowledge, we recommend rejecting such rocks unless the reliability of the ages can be checked independently.

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