(perhaps as deep as a few meters) below the sea floor.

Microspar in transition-zone rocks seems best interpreted as the result of precipitation (further cementation) from isotopically "light" formation waters that must have been depleted in oxygen-18, and perhaps mildly so in carbon-13, relative to Mississippian seawater. The microspar could have originated before dolomitization, in which case its increase in abundance with dolomite content is a net increase resulting from growth of dolomite chiefly at the expense of micrite; the microspar simply "survived" dolomitization better. Alternatively the microspar may postdate the dolomitization, reflecting perhaps more accessibility and "exposure" of unreplaced $CaCO_3$ to formation water in the more porous, permeable, dolomite-rich rocks. The second explanation seems more reasonable since it would not require freshwater diagenesis of $CaCO_3$ prior to marine or hypersaline-water dolomitization, as would the first hypothesis.

If these interpretations are correct, the δO^{18} differences between dolomite and calcite cannot have the same meaning as the 6 to 10 (*10*) or 4 to 7 per mille (*16*) differences estimated for dolomite and calcite coprecipitated at 25°C in isotopic equilibrium; nor can the dolomite be construed as "primary." The following summarized explanation seems more reasonable:

1) The dolomite formed from $CaCO_3$ and has since been quite stable with respect to δO^{18} and δC^{13}. It may have formed (i) solely from $CO_3^=$ provided by $CaCO_3$, without fractionation of O^{18}:O^{16}—a dolomitization process that is well supported (*10*); or (ii) in nonequilibrium from marine $CaCO_3$ in intrasediment seawater or derived brine, with oxygen from both the $CaCO_3$ and the water. I favor alternative (ii) because of the micrite interpretations suggested earlier and about to be summarized.

2) Before or after dolomitization (perhaps both) the original unreplaced lime mud became partly cemented by $CaCO_3$ precipitated by intrasediment seawater. The resultant partly lithified but still-permeable lime mudstone consisted mainly of micrite having an estimated δO^{18} between -1 and $+1$ per mille.

3) Unreplaced micrite in the more permeable dolomitic rocks later underwent marked changes in O^{18}:O^{16} and in C^{13}:C^{12} during a long and probably complex sequence of diagenetic events that began in Mississippian time. The net changes in calcite of these rocks were toward depletion in the "heavy" isotopes, probably accomplished by precipitation of $CaCO_3$ from formation waters containing less oxygen-18 and possibly less carbon-13 than seawater contained; the result was microspar.

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Argon-40: Excess in Submarine Pillow Basalts from Kilauea Volcano, Hawaii

Abstract. *Submarine pillow basalts from Kilauea Volcano contain excess radiogenic argon-40 and give anomalously high potassium-argon ages. Glassy rims of pillows show a systematic increase in radiogenic argon-40 with depth, and a pillow from a depth of 2590 meters shows a decrease in radiogenic argon-40 inward from the pillow rim. The data indicate that the amount of excess radiogenic argon-40 is a direct function of both hydrostatic pressure and rate of cooling, and that many submarine basalts are not suitable for potassium-argon dating.*

Potassium-argon ages of submarine basalts are now sought for information on the history and development of the ocean basins, especially on possible spreading of the sea floor (*1*). A fundamental assumption made in dating of volcanic rocks is that they contained no radiogenic Ar^{40} at the time they solidified. This assumption is generally true for terrestrial volcanic rocks (*2*) but has not been adequately tested for submarine lava flows.

At some time before solidification, lava flows must lose the radiogenic Ar^{40} that is continually generated by the decay of K^{40}, otherwise the K-Ar clock will not be reset to zero at the time of solidification. This loss of radiogenic Ar^{40} may take place (i) either partly or entirely during the melting leading to magma generation; (ii) while the liquid is rising to the surface or is stored in temporary reservoirs; or (iii) during eruption, as the pressure is released. The time of degassing is uncertain, but the existance of vesicles and lava fountains suggests that some gas is lost during and shortly after eruption. If release of pressure during eruption is important, the hydrostatic pressures at ocean depths may possibly impede loss of argon. Moreover, submarine pillow basalts are more rapidly quenched than subaerial flows—another condition that may prevent complete loss of argon.

To test the hypothesis that submarine flows contain no radiogenic Ar^{40} when they form, samples of Hawaiian pillow basalts were studied. They were chosen because (i) being no older than a few thousand years they should lack detectable radiogenic Ar^{40}; (ii) they enable study of the effect of ocean depth on rocks from the same source; and (iii) independent evidence suggests decrease in loss of volatiles with increase in ocean depth.

Samples were dredged from the crest of the submarine extension of the active east rift zone of Kilauea Volcano (Fig. 1); depths ranged from 550 to 5000 m. Presumably a lava flow erupted from the crest of the ridge would flow down one of the two flanks rather than directly down the crest; therefore samples from the crest probably come from close to their vent—not from flows erupted at a much shallower depth.

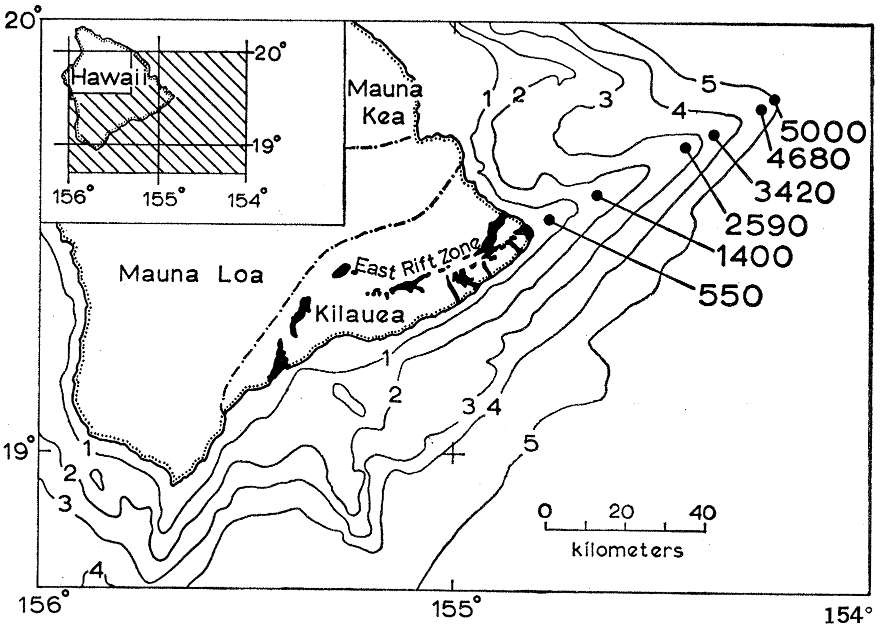

Fig. 1. Submarine topography adjacent to the Island of Hawaii, and sources of dredged samples. The number of each sample is its depth in meters. Dash-dot lines isolate individual volcanoes; black areas are flows of lava from Kilauea Volcano since 1750(?). Depth contours are in kilometers.

No other radiometric ages have been determined for submarine lavas from Kilauea Volcano. The following evidence shows that the pillows are young:

1) About half the surface of the subaerial rift zone is mantled by historic lava flows (Fig. 1). All exposed rocks of the rift zone belong to the Puna Volcanic Series, which is younger than Pahala Ash yielding radiocarbon dates of 10,000 and 17,000 years (*3*). If one assumes that the distribution of volcanic activity is similar on the submarine part of the same rift zone, the age of the exposed rocks is similar.

2) Basalt from the deepest canyon of the oldest volcano (Kohala) on the Island of Hawaii is younger than 0.8 million years (*4*). All 152 lava flows sampled on the Island of Hawaii, including those from the oldest exposed rocks from Kohala Volcano, show normal magnetic polarity (*5*); thus they are presumably younger than 700,000 years (*6*). Kohala Volcano is considerably older than Kilauea because it is deeply eroded with canyons deeper than 500 m; Kilauea is not eroded and has no surface drainage.

3) The pillows are remarkably fresh, with a layer of palagonite thinner than 15 μ and no detectable manganese oxide crust. Similar pillows dredged from the east rift zone of Mauna Kea Volcano (intermediate in age between Kohala and Kilauea), 30 km to the north, have palagonite rims 250 to 700 μ thick and manganese oxide crusts 400 to 1100 μ thick (*7*). These relations suggest that the Kilauea samples are younger by more than two orders of magnitude than those from Mauna Kea. Use of a rate of accumulation of manganese oxide of 3 mm/10^6 years (*8*) indicates that the Kilauea samples are younger than 1000 years.

Our samples were fragments of fresh basalt pillows or of basalt slabs 2- to 5-cm thick. The rocks are rather uniform olivine tholeiitic basalt (*9*), similar to tholeiites erupted subaerially on the same rift zone; they are all vitrophyric, and olivine is generally the most abundant phenocryst; clinopyroxene and plagioclase also occur as phenocrysts and are the dominant minerals of the groundmass.

Drastic quenching by seawater has caused a distinct change in texture from the rim to the interior of each sample. In sample 2590, glass systematically decreases from about 90 percent on the margin, where it is pale brown and transparent, to about 60 percent at 8-cm depth where it is dark brown and charged with fine, dusty, opaque inclusions. Plagioclase and pyroxene microlites increase in abundance and size with depth as they crystallize at the expense of glass.

The vesicularity of the pillows decreases systematically with ocean depth, because exsolution of dissolved gas was inhibited by water pressure at the eruptive vent (*9*). The outer glassy crust of

Table 1. Argon analyses of submarine pillow basalts from Kilauea. Percentages of K_2O are from Moore (*9*), except for sample 550 which was analyzed by flame photometry. The last two columns include estimates of analytical precision at 95 percent level of confidence.

Locality (No.) (*9*)	Sample		K_2O (%)	$Ar^{40}:Ar^{36}$	Ar^{40}_{rad} per gram ($\times 10^{-11}$ mole)	Apparent age ($\times 10^6$ yr)
	No. and depth (m)	Distance from pillow rim (cm)				
2	550	0–1	0.68	302.8	0.023 ± 0.023	0.22 ± 0.22
2	550	~ 18	.68	301.3	$.016 \pm .018$	$.16 \pm .18$
4	1400	0–1	.47	319.1	$.44 \pm .13$	6.3 ± 1.9
8	2590	0–1	.40	1022	$2.56 \pm .26$	42.9 ± 4.2
8	2590	1–2	.40	348.0	$1.97 \pm .30$	33.0 ± 5.0
8	2590	3.5–4.5	.40	314.5	$0.26 \pm .10$	4.3 ± 1.6
8	2590	7.5–8.5	.40	296.5	$< .09$	< 1.5
8	2590	10.5–12.0	.40	298.4	$< .057$	< 1.0
10	3420	0–1	.42	474.5	$.88 \pm .11$	14.1 ± 1.7
12	4680	0–1	.46	592.9	$2.08 \pm .23$	30.3 ± 3.3
13	5000	0–1	.33	402.0	$0.96 \pm .12$	19.5 ± 2.5

the pillows contains more than 10 percent vesicles to a depth of about 800 m; deeper, the vesicles become less abundant and smaller, practically disappearing at a depth of 4000 m where they are less than 0.1 mm in diameter.

The abundance of vesicles also changes systematically between the rim and interior of a single pillow. In sample 2590, vesicles increase from less than 0.5 percent on the rim to more than 2 percent toward the center. This change apparently reflects slower cooling toward the center. Slower cooling allows more time for gases to exsolve from the melt and promotes crystallization, thus causing further exsolution of gases from the diminishing volume of melt (glass).

Argon was determined with equipment and techniques described (*10*), except that an Ar^{38} tracer was not used. The $Ar^{40}:Ar^{36}$ ratio was measured for each sample, and the quantity of argon was determined from the ion-beam intensity, which was calibrated with a known quantity of Ar^{38}. This method introduces some uncertainty in the quantity of argon but it does improve detection of small departures of the ratio $Ar^{40}:Ar^{36}$ from the atmospheric value (*11*) of 295.5. The amount of Ar^{40} in a given sample is estimated to be within 10 percent at the 95 percent confidence level. The effect of correction for atmospheric Ar^{40} on the uncertainty in radiogenic Ar^{40} was estimated by a reported technique (*12*).

Repeated analyses of standard argon samples were made to determine the level at which real departures from the atmospheric $Ar^{40}:Ar^{36}$ value of 295.5 could be detected. The results indicate that a real difference can be claimed with 95 percent confidence if the ratio $Ar^{40}:Ar^{36}$ for a single analysis is greater than 300. As an additional basis for comparison, measurements were made on two historic lava flows from the subaerial part of the east rift of Kilauea. The 1750(?) and the 1955 flows gave $Ar^{40}:Ar^{36}$ values of 294.0 and 296.7, respectively, both indistinguishable from atmospheric argon.

The concentration of radiogenic Ar^{40} in the outer glassy crusts (*13*) of pillows from different depths clearly shows the effect of hydrostatic pressure (Table 1). Below 2000 m, the content of radiogenic Ar^{40} is variable but consistently large, leading to anomalously high K-Ar ages. At 1400 m there appears to be a considerable decrease, and at 550 m the radiogenic-Ar^{40} component is barely detectable. Samples from depths less than 550 m were unavailable, but the results suggest that the excess radiogenic Ar^{40} may possibly become negligible at shallower depths.

To test the effect of quenching, argon measurements were made on samples taken from various distances inward from the rim of pillow sample 2590. The results (Fig. 2) show that there is decrease in excess radiogenic Ar^{40} with distance inward from the pillow rim; the $Ar^{40}:Ar^{36}$ ratios of the innermost samples are indistinguishable from those of air argon. This decrease in excess radiogenic Ar^{40} inward appears to correlate with the observed mineralogical and textural changes which in turn reflect the decreased cooling rates toward the interior of the pillow (Fig. 2). Although modal amounts of minerals and glass change by less than a factor of two from the rim to the interior, the excess radiogenic Ar^{40} decreases by more than an order of magnitude. This fact indicates that the decrease in excess radiogenic Ar^{40} is probably due to cooling times and not to any particular petrologic property, such as amount of glass.

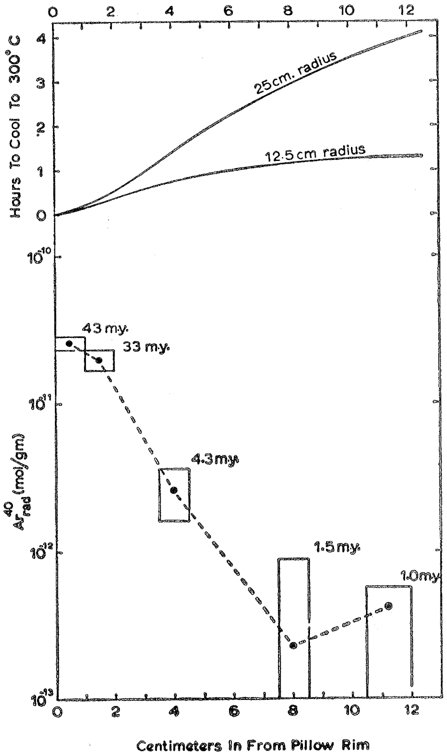

Fig. 2. Excess content of radiogenic Ar^{40} and apparent age as functions of distance inward from the rim of pillow sample 2590. Width of each box indicates thickness of analyzed sample; height of each box indicates 95 percent confidence limits. Top curves show the times required for spherical basalt pillows ($\kappa = 0.007$) to cool from 1200° to 300°C when one assumes a constant surface temperature of 0°C; *m.y.*, million years.

A sample 18 cm from the rim of pillow sample 550 also has detectable excess radiogenic Ar^{40}; the amount is roughly equivalent to that found in the rim. This finding indicates that excess Ar^{40} does not necessarily become negligible in pillow interiors.

It is clear that the hypothesis of zero radiogenic Ar^{40} at the time of solidification is not always valid for submarine volcanic rocks. The results of our experiments show that there can be a significant quantity of excess radiogenic Ar^{40} dissolved in submarine lava, and that the amount is influenced by hydrostatic pressure. Under conditions of rapid quenching, such as in the glassy crusts of pillows, excess Ar^{40} is trapped and at least partly retained. In the case of shallow eruptions, where the hydrostatic pressure is insufficient to inhibit vesiculation, much of the excess Ar^{40} escapes with other volatiles. However, even in samples from as shallow a depth as 550 m, the remaining excess Ar^{40} may be enough to cause large errors in K-Ar ages of young rocks or of rocks with very low contents of K_2O.

The retention of excess radiogenic Ar^{40} by submarine basalts also seems to be influenced by cooling time. Calculations have been made for the cooling of spherical pillows (Fig. 2). Such a model is an upper limit of quenching rate because the pillow margin (particularly the base) may rise substantially above 0°C, the pillows may be larger or more elongate than assumed, and heat of crystallization will supply additional heat to the system. The model indicates, however, that the excess Ar^{40} is lost from pillow interiors in a very short time. The mechanism of loss of excess radiogenic Ar^{40} is not known. However, the decrease in vesicularity with depth and toward pillow rims, and the corresponding increase in excess radiogenic Ar^{40}, indicate that exsolution from the basalt melt is important. It is also possible that some excess Ar^{40} may escape from pillow interiors by diffusion at high temperatures after the lava has solidified.

It may be that both rapid quenching and hydrostatic pressure are required to produce large excess Ar^{40} anomalies —the amount of excess Ar^{40} dissolved in the melt at the time of eruption, controlled by hydrostatic pressure, and the amount retained by the rock, related to cooling time. This suggests that the problem of excess radiogenic Ar^{40} may be less severe for coarse-grained rocks because they cool more slowly than fine-grained or glassy rocks.

Reliable potassium-argon ages probably will be obtained eventually from deep-sea samples, but criteria for recognition of reliable samples must first be developed. Meanwhile, ages from submarine volcanic rocks clearly cannot be used for testing of spreading of the sea floor without supporting evidence, such as determinations on two minerals from the same rock.

Much of the juvenile volatile content may still be present in volcanic rocks quenched on the ocean floor. This finding both supports earlier determinations that Hawaiian tholeiitic magma from depth contains H_2O at 0.45 percent by weight (*9*), and provides a new technique for study of volcanic gases—systematic analysis of the volatiles contained in the outer margins of fresh pillows collected from great depth.

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High-Resolution Electron Microscopy of Muscovite

Abstract. *Images of the (001) planes of muscovite were observed by electron microscopy after embedment of clay-size flakes in epoxy resin and sectioning by ultramicrotome. Dislocation or growth planes were indicated. Incomplete exchange of potassium for barium produced zones with 20-angstrom periodicity, suggesting regular interstratification.*

The interlamellar regions of phyllosilicate clays are the major sites of cation-exchange reactions occurring in the mineral fractions of soils and sediments. We have been concerned with determining the spatial arrangement of the individual molecular layers of weathered micas, and with relating this to cation selectivity (*1*). High-resolution electron microscopy can resolve lattice images of certain crystal planes. Since lattice images were first observed (*2*) in organic crystals containing heavy metal ions, they have been observed in other fine-particle materials with suitable crystal habit. Small crystals, lying horizontally on a microscopy substrate, must have the desired planes within 1 or 2 deg of the optical axis for successful imaging. Only recently have crystalline particles been oriented and microtomed for the purpose of seeing a particular set of lattice planes (see *3*).

In order to observe the lattice images of the (001) phyllosilicate planes associated with the interlamellar spacing, one must orient the flakes perpendicular to their normal position on a flat surface, and cut sections thin enough for the transmission of the electron beam. This was done as follows.

A layer of Araldite epoxy resin was cast on a glass microscope slide coated with a thin layer of silicone grease. After curing of the resin, a distilled-water suspension of mica particles was dried on the resin surface, and a second layer of resin was applied and cured. The cast "sandwich" was then removed from the slide and sawed into 1-mm cubes containing the sediment layer. The cubes were glued to larger blocks and mounted in the ultramicrotome. Thin sections approximately 300-μ square were cut at 90 deg to the sediment plane with a Leitz ultramicrotome using a diamond knife; they were estimated to be 300 to 600 Å in thickness as gauged by their reflected color (*4*).

Three samples all 0.2 to 2 μ in size were examined: (i) muscovite, (ii) barium-treated muscovite, and (iii) a weathered muscovite from a soil. Large flakes of a specimen muscovite from Amelia, Virginia, were broken apart in water with an Omni-Mixer, and the fraction between 0.2 and 2 μ was separated by centrifugation. An oriented sample of the fraction gave a sharp 10-Å x-ray diffraction peak as well as integral higher orders of this (001) reflection. Much of the potassium (76 percent) was exchanged from a portion of the fraction by repeated treatments with 0.1N $BaCl_2$ in an autoclave (*5*). The resultant sample gave a 12.1-Å (001) reflection. Higher orders are integral and indicative of a barium-dioctahedral vermiculite. In addition, interstratification of muscovite layers with vermiculite was confirmed by the asymmetry of the (001) peak.

X-ray diffraction also gave a 9.9-Å spacing for barium-saturated muscovite dried over P_2O_5. The resin-curing treatment or high vacuum in the electron microscope, or both, would remove the layer of water molecules associated with the 12.1-Å barium-vermiculite. The weathered muscovite was obtained by centrifugation of a sample of the B2 horizon of a Nason soil from Orange County, Virginia.

Electron micrographs (× 83,000) were taken on Eastman-P426 35-mm film with a Philips EM-200; they show parallel fringes that are lattice images of the (001) planes in the crystal and are formed by combination of the zero-order beam with a limited number of diffraction spectra from the periodic structure. The micrographs were made under bright-field conditions with a 50-μ back-focal-plane aperture; thus the image structure is formed from Bragg reflections of spacings greater than 4 Å.

The unweathered muscovite (Fig. 1A) shows fringes that are of uniform darkness when limited areas are considered. In contrast, in sections of the barium-treated sample (Fig. 1B) the dark fringes frequently alternate in intensity; this alternation may indicate zones where Ba has been substituted for K ions in alternate layers. Interstratification, indicated by x-ray diffraction, is apparently regular in these zones. Replace-