# 41. <sup>40</sup>Ar-<sup>39</sup>Ar DATING OF ROCKS DRILLED AT SITES 458 AND 459 IN THE MARIANA FORE-ARC REGION DURING LEG 60<sup>1</sup>

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#### ABSTRACT

<sup>40</sup>Ar-<sup>39</sup>Ar dating of a high-MgO bronzite andesite from near the top of basement drilled at Site 458 shows the characteristic symptoms of artificially disturbed samples—i.e., an inverse staircase-type age spectrum, approximate linearity on an isochron plot, and concordance between total fusion age and isochron age. From conclusions based on other artificially disturbed samples (Ozima et al., 1979), we suggest that the reference isochron age (33.6 Ma) approximates the age of the sample.

A basalt from deeper in Hole 458 gives an isochron age of  $19.1 \pm 0.2$  Ma, which is slightly younger than the plateau age of  $21.4 \pm 1.0$  Ma. Both ages are, however, considerably younger than the age of fossils in the overlying sediments ( $30 \sim 34$  Ma). The age discrepancy may be explained if the  $^{40}$ Ar- $^{39}$ Ar age represents the age of secondary minerals, which formed later.

No useful age data were obtained from a basalt sample recovered from Hole 459B.

## SAMPLES

<sup>40</sup>Ar-<sup>39</sup>Ar dating has been carried out on two rocks from Site 458 and one rock from Site 459.

### Site 458

A total of 209 meters of pyroxene-plagioclase pillow lavas, including high-MgO bronzite andesite, which resembles boninite, and tholeiitic basalt, was cored. Dating was carried out on Samples 458-29-3, 3-8 cm (a bronzite andesite), and 458-40-2, 38-45 cm (a basalt). These samples are vesicular, aphyric, nearly glassy, and have high water contents ( $H_2O^+ = 2 \sim 3 \text{ wt.\%}$ , P. Fryer, 1979 personal communication). They show extensive alteration, in that the groundmass is altered to various secondary minerals, mainly clays. Sample 458-40-2, 38-45 cm is more aphyric with vesicles partially filled with zeolite. It comes from a highly fractured interval with a magnetic inclination opposite that of the sample from Core 28 (Bleil, this volume). Therefore it could represent an earlier eruption.

### Site 459

About 133 meters of basement basalt was drilled, all of it altered clinopyroxene-plagioclase basalt. <sup>40</sup>Ar-<sup>39</sup>Ar dating was applied to Sample 459-61-1, 133-138 cm.

### METHOD

Chunks of three samples (about 1.5 g each) were sealed in a quartz tube (9 mm dia.  $\times$  7 cm) with CaF<sub>2</sub>, K<sub>2</sub>SO<sub>4</sub>, and two standard samples (JG-1 biotite prepared by Japan Geological Survey; K<sub>2</sub>O = 7.64 wt.%, age t = 90.8 Ma). The quartz tube was then subjected to a neutron flux of about 10<sup>18</sup> nvt/cm<sup>2</sup> in a Japan material testing reactor. The heterogeneity in the neutron flux was estimated from the results on the standard samples to be generally less than 2%/cm along the quartz tube. The irradiated samples were heated with a high-frequency induction heater in which temperature was controlled by adjusting the output power with the aid of an optical pyrometer. Further experimental details on this technique are given elsewhere (Ozima et al., 1977).

### RESULTS

The experimental data are represented both in an age spectrum and an isochron plot (Figs. 1 and 2). The analytical data are given in Table 1. Table 1 also shows correction factors for interfering Ar isotopes, which were determined on monitored  $CaF_2$  and  $K_2SO_4$  irradiated with the sample. Age results are given in Table 2.

## Sample 458-29-3, 3-8 cm

This sample gives an inverse staircase-type age spectrum, i.e., younger apparent ages for the higher-temperature fractions (Fig. 1). The data do not form an isochron, but three temperature fractions (700, 800, 900°C), which constitute about 50% of the total <sup>39</sup>Ar released, are on a straight line. The straight line is close to a reference isochron of 33.6 Ma. The total fusion age, which is essentially similar to a conventional K-Ar age, is 31.8  $\pm$  0.7 Ma.

## Sample 458-40-2, 38-45 cm

In the age spectrum for this sample, an anomalously old age appears in the 600 °C fraction. However, the higher temperature fractions which constitute about 80% of the <sup>39</sup>Ar released give a plateau age of 21.4  $\pm$ 1.0 Ma in the age spectrum. All the experimental data but the lowest two fractions (<600 °C and 600 °C) fit reasonably well to an isochron, which gives an isochron age of 19.1  $\pm$  0.2 Ma with the intercept <sup>40</sup>Ar/<sup>36</sup>Ar of 322.1  $\pm$  18.7. The isochron and the plateau ages are nearly concordant but much younger than the total fusion age (40.5  $\pm$  1.2 Ma).

## Sample 459-61-1, 133-138 cm

Because of the large amount of air contamination (80  $\sim$  98%) in this sample, large uncertainties were intro-

<sup>&</sup>lt;sup>1</sup> Initial Reports of the Deep Sea Drilling Project, Volume 60.

Table 1. Analytical data for <sup>40</sup>Ar-<sup>39</sup>Ar analysis.

Temperature (°C)	40 <sub>Ar</sub> ∕36 <sub>Ar</sub> a (% error)	<sup>39</sup> Ar/ <sup>36</sup> Ar <sup>a</sup> (% error)	37 <sub>Ar/36Ar</sub> a (% error)	<sup>39</sup> Ar Fraction (%)	rb	Apparent Age (Ma) (one-sigma)
Sample 458-29-3, 3-8 cm		$J^{C} = 0.00366$	1			
< 600	477.2 (1.5)	17.9 (1.9)	9.4 (4.8)	12.0	0.60	$65.7 \pm 1.9$
600	602.7 (5.3)	42.5 (5.4)	45.3 (5.6)	13.5	0.98	$47.1 \pm 2.4$
700	597.1 (2.9)	82.5 (2.9)	274.8 (3.0)	15.5	0.93	$24.0 \pm 7.2$
800	926.7 (8.7)	143.9 (8.7)	716.6 (8.8)	16.1	0.99	$28.7 \pm 1.2$
900	1147.0 (1.4)	192.1 (1.4)	881.0 (13.8)	18.2	0.99	$29.0 \pm 1.4$
950	639.5 (1.7)	137.7 (1.7)	298.2 (16.8)	12.3	1.00	$16.4 \pm 2.4$
>1300	506.4 (6.5)	91.3 (6.6)	357.7 (6.6)	12.4	0.90	$15.2 \pm 1.4$
Sample 458-40-2, 38-45 cm		J = 0.003585				
< 600	303.7 (5.3)	2.3 (23.7)	3.8 (21.4)	1.3	0.04	$22.7 \pm 43.6$
600	680.9 (3.6)	20.9 (3.9)	38.1 (4.0)	19.9	0.79	$115.3 \pm 3.7$
700	530.6 (6.8)	71.6 (7.3)	298.0 (6.9)	10.9	0.93	$21.1 \pm 1.9$
800	535.3 (9.2)	68.9 (9.5)	345.6 (9.7)	7.5	0.90	$22.4 \pm 2.6$
850	464.3 (10.1)	42.0 (10.2)	357.1 (10.1)	11.5	0.95	$25.8 \pm 4.5$
950	535.2 (4.6)	73.0 (4.7)	602.6 (5.1)	11.6	0.93	$21.1 \pm 1.2$
>1300	1010.0 (5.6)	230.7 (5.6)	706.9 (5.6)	37.5	0.96	$19.9 \pm 0.5$
Sample 459-61-1, 133-138 cm		J = 0.003441	- Q. I			
< 600	301.3 (1.7)	3.0 (12.0)	2.7 (13.0)	6.1	0.08	$12.1 \pm 10.6$
600	371.8 (1.0)	6.3 (3.0)	16.6 (1.2)	31.0	0.34	$73.5 \pm 3.6$
750	352.3 (1.4)	11.2 (1.6)	93.4 (1.5)	18.2	0.25	$31.4 \pm 2.3$
850	317.3 (6.9)	10.0 (7.1)	115.3 (7.1)	7.1	0.59	$13.5 \pm 12.6$
900	351.2 (3.4)	15.0 (4.5)	440.8 (3.5)	12.7	0.51	$22.9 \pm 4.2$
1000	326.6 (5.9)	23.0 (6.0)	650.9 (6.0)	7.4	0.63	$8.4 \pm 4.7$
>1350	367.2 (12.1)	35.7 (12.1)	1865.0 (12.1)	17.4	0.81	$12.4 \pm 6.2$

<sup>a</sup> Correction factors used as  $({}^{39}\text{Ar}/{}^{37}\text{Ar})_{Ca} = 0.0007$ ,  $({}^{36}\text{Ar}/{}^{37}\text{Ar})_{Ca} = 0.00036$ , and  $({}^{40}\text{Ar}/{}^{39}\text{Ar})_{K} = 0.03$ . <sup>b</sup> Correlation coefficient (York, 1969). <sup>c</sup>  $J = [\text{exp(}k_f) - 1]/({}^{40}\text{Ar}/{}^{39}\text{Ar})_{S}$ ;  $r_{g}$  is age of a standard sample (JG1-biotite), S refers to a standard sample,  $\lambda$  is total decay constant of  ${}^{40}\text{K}$ .

Table 2. 40Ar-39Ar ages.

Sample	Total Fusion <sup>a</sup>	Plateau	Isochron		MSUMd
(interval in cm)	Age (Ma)	Age (Ma)	Age (Ma)	Intercept	(cutoff value)
458-29-3, 3-8	$31.8 \pm 0.7$		(33.6) <sup>b</sup>	(174,1)	: <u></u>
458-40-2, 38-45	$40.5 \pm 1.2$	$21.4 \pm 1.0^{\circ}$	$19.1 \pm 0.2^{\circ}$	322.1 ± 18.7	0.14 (3.97)
459-61-1, 133-138	$36.1 \pm 2.0$	-			_

 $\frac{a}{\lambda} = 5.543 \times 10^{-10} \text{ yr}^{-1}, \lambda_{e} = 0.581 \times 10^{-10} \text{ yr}^{-1}, \frac{40}{\text{ K/K}} = 0.0001167, \text{ total fusion age} = 1/\lambda \ln [1 + J(40\text{Ar}/39\text{Ar})\text{total}], (40\text{Ar}/39\text{Ar})\text{total}] = \sum_{i}^{D} f_{i} (40\text{Ar}/39\text{Ar})_{i}, f_{i} = 39\text{Ar fraction released}$ released.

released. b Isochron age was calculated from 700, 800 and 900°C fractions. <sup>c</sup> Plateau age and Isochron age were calculated from 700, 800, 850, 950, and >1300°C fractions. <sup>d</sup> MSUM = SUMS/n - 2. n = number of fractions (Brooks et al., 1972). Values in parentheses are cutoff values for the 95% significance level.

duced in the air Ar correction. The sample gives neither a plateau age nor an isochron. Since both the age spectrum and the isochron plot indicate heavy geological disturbances, we cannot derive any meaningful age information from it.

## DISCUSSION

The <sup>40</sup>Ar-<sup>39</sup>Ar systematics for Sample 458-29-3, 3-8 cm show the characteristic symptoms of artifically disturbed samples (Ozima et al., 1979)-that is, (1) an inverse staircase-type age spectrum, (2) approximate preservation of linearity in the isochron plot, (3) similarity between total fusion age and isochron age. Since Ozima et al. (1979) showed that the isochron age in such artificially disturbed samples is close to the original age in spite of the disturbances, we suggest that 33.6 Ma (reference isochron age) closely approximates the original age of this sample.

It also seems significant that the suggested age for this sample agrees well with the sediment record, in that the oldest fossil record in the overlying sediment is the middle Oligocene (30  $\sim$  34 Ma).

Experimental data for Sample 458-40-2, 38-45 cm, fit reasonably well to a straight line in the isochron plot; furthermore, all the fractions but those of  $< 600^{\circ}$ C and 600°C present a plateau in the age spectrum. The isochron and plateau ages are almost concordant but considerably younger than the total fusion age. The apparently high total fusion age is likely to be due to <sup>39</sup>Ar loss, which is characteristically reflected in the anomalously old apparent age in the 600 °C fraction in the age spectrum. In fact, Seideman (1978) found that for some altered and fine-grained deep sea basalts <sup>39</sup>Ar was lost during the irradiation from low temperature sites, showing anomalously old apparent age in the age spectrum.

The discrepancy between the fossil age (30 - 34 Ma)and the radiometric age ( $\sim 20$  Ma) may be understood in one of two ways: either (1) the radiometric age represents the time of the recrystallization or high-temperature metamorphism of the sample, which resets the <sup>40</sup>Ar-<sup>39</sup>Ar clock, or (2) the radiometric age dates essentially the high K-content secondary minerals which formed as seawater weathering products in the sample.

The first interpretation, however, seems rather improbable, since microscopic examination of the sample did not reveal recrystallization or high-temperature metamorphism of the sample. The second interpretation seems more likely. Hart and Staudigel (1978) reported a fairly nice Rb-Sr isochron for smectites separated from DSDP Leg 52 basalts, and they concluded that the formation of the smectites occurred in a very short time so that the Rb-Sr systematics did form a single isochron. Assuming that these conclusions are also applicable to the present sample, we suggest that the radiometric age indicates the age of the secondary minerals. The X.M.A. examination on the sample shows that potassium resides essentially in the groundmass or in the veinfilling secondary minerals such as zeolite. The large time gap between the fossil age and the radiometric age may suggest that the rock was buried soon after eruption under lava flows until the rock was exposed to alteration during later geological disturbances. Hence in the second interpretation, the radiometric age of 21.4 Ma or 19.1 Ma represents the time of such tectonic disturbances. The sample was obtained from a highly fractured interval. Natland and Mahoney (this volume) report that the alteration in this interval was nonoxidative, and overprinted an earlier stage of oxidative alteration. They suggest that the nonoxidative alteration occurred only after burial by sediments and was associated with faulting which produced the fracturing in the rocks. The data presented here support this interpretation.

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#### REFERENCES

- Brooks, C., Hart, S. R., and Wendt, I., 1972. Realistic use of twoerror regression treatments as applied to rubidium-strontium data. Reg. Geophys. Space Phys., 10:551-578.
- Hart, S. R., and Staudigel, H., 1978. Oceanic crust: Age of hydrothermal alteration. *Geophys. Res. Lett.*, 5:1009-1012. Ozima, M., Honda, M., and Saito, K., 1977. <sup>40</sup>Ar-<sup>39</sup>Ar ages of guyots
- in the eastern Pacific and discussion of their evolution. Geophys. J. Astr. Soc., 51:475-485.



Figure 1. Sample 458-29-3, 3-8 cm. Apparent age spectrum (left) and isochron plot (right). The values of  ${}^{40}\text{Ar}/{}^{36}\text{Ar}$ ,  ${}^{39}\text{Ar}/{}^{36}\text{Ar}$  are corrected for interfering Ar isotopes induced by Ca and K. Error is one-sigma.



Figure 2. Sample 458-40-2, 38-45 cm. Notations are the same as in Figure 1.

Ozima, M., Kaneoka, I., and Yanagisawa, M., 1979. Temperature and pressure effects on <sup>40</sup>Ar-<sup>39</sup>Ar systematics. *Earth Planet. Sci. Lett.*, 42:463-472. Seidemann, D., 1978. <sup>40</sup>Ar/<sup>39</sup>Ar studies of deep-sea igneous rocks. Geochim. Cosmochim. Acta, 42:1721-1734.

York, D., 1969. Least squares fitting of a straight line with correlated errors. *Earth Planet. Sci. Lett.*, 5:320-324.