

## 28. $^{40}\text{Ar}$ - $^{39}\text{Ar}$ GEOCHRONOLOGICAL STUDIES ON ROCKS DRILLED AT HOLES 462 AND 462A, DEEP SEA DRILLING PROJECT LEG 61<sup>1</sup>

M. Ozima, K. Saito,<sup>2</sup> and Y. Takigami,  
Geophysical Institute, University of Tokyo, Tokyo 113, Japan

### ABSTRACT

$^{40}\text{Ar}$ - $^{39}\text{Ar}$  step-heating dating was applied to a basalt from Hole 462 and to basalt and dolerite samples from Hole 462A. Only a basalt sample at Hole 462A yielded a reasonable isochron age,  $110 \pm 3$  million years. The radiometric age is consistent with the fossil record (Cenomanian) in the sediments, into which the basalt sill intruded. However, the age is much less than that of the oceanic basement as deduced from the magnetic anomaly (*M-26*).

### SAMPLES

Sample 462-60-1, 65-69 cm is a clinopyroxene-plagioclase phyric basalt containing some clays which are altered products of glasses. The sample is from a sill, next to glassy margins, which intruded into Cretaceous sediment (Cenomanian). Sample 462A-32-1, 46-49 cm is a clinopyroxene-plagioclase phyric basalt, and is lithologically quite similar to Sample 426-60-1, 65-69 cm; it is also from a sill, next to a glassy margin, which intruded into Cretaceous (Cenomanian) sediment. Glasses are partly altered to clays. Sample 462A-50-3, 130-134 cm is from a dolerite sill which intruded into sediments (Hauterivian). The degree of alteration here is the slightest among the three samples. The microscopic observations were made by Dr. H. Tokuyama, Ocean Research Institute, University of Tokyo.

### EXPERIMENTAL METHODS

Chunks of three samples (about 1 g each) were vacuum-sealed in a quartz tube (9 mm dia.  $\times$  7 cm) with  $\text{K}_2\text{SO}_4$  and two standard samples (JG1 granodiorite prepared by Geological Survey of Japan,  $t = 90.8$  m.y.,  $\text{K}_2\text{O} = 7.64\%$ ). The quartz tube was then subjected to a neutron flux of about  $5 \times 10^{17}$  in a Japan Material Testing Reactor (JMTR).

The correction factors for interfering neutron-induced Ar isotopes were determined on  $\text{K}_2\text{SO}_4$  and  $\text{CaF}_2$ ; the latter was irradiated in another quartz tube in the same irradiation run. The correction factors thus determined are  $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 0.117$ ,  $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 3.6 \times 10^{-4}$ , and  $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 7 \times 10^{-4}$ . These corrections were not significant, however, for the final age calculation. The heterogeneity in the neutron flux was estimated from the results on the two standard samples to be about 0.6%/cm along the length of the quartz tube. The irradiated samples were heated at seven temperature programs (500°C, 600°C, 700°C, 800°C, 900°C, 1000°C, and fusion) for 45 minutes. The temperature was controlled by adjusting the output power of the induction heater with the aid of an optical pyrometer. Further experimental details are given in Ozima et al. (1977).

### RESULTS

The experimental data are represented in both an age spectrum and an isochron plot (Figs. 1 and 2). The ana-

lytical data are given in Table 1, in which a total fusion age and an apparent K-Ar age for each temperature fraction are also shown. The age results are given in Table 2.

#### Sample 462-60-1, 65-69 cm

This gives a very ragged age spectrum, roughly of a staircase type, and no plateau age can be defined (Fig. 1). Except for the lowest-temperature fraction (500°C), however, other data points lie roughly on a line which is close to a reference isochron of 120 m.y. Although the considerable deviation of each data point from a line, and the ragged age spectrum, indicate that the sample suffered geological disturbances such as Ar-loss, the rough linear correlation in the isochron plot may be related to the age of the sample, as deduced from studies (Ozima et al., 1979) on  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  systematics of artificially disturbed samples. That the total fusion age is much less than the reference isochron age may indicate radiogenic- $^{40}\text{Ar}$  loss from the sample. We suggest 120 m.y. as a rough approximation of the age of the sample.

#### Sample 462A-32-1, 46-49 cm

Except for the 600°C fraction, which constitutes only 1.6% of the total  $^{39}\text{Ar}$  released, all other data points lie fairly well on a line (Fig. 2). The  $y$ -intercept of the line is 300, which is not significantly different from the atmospheric  $^{40}\text{Ar}/^{36}\text{Ar}$  ratio. The regression line was determined with the use of York's correlated error program (1969), and the statistical parameter MSUM (Brooks et al., 1972) was calculated to be 2.82. In the age spectrum, more than 80% of the total  $^{39}\text{Ar}$  released, though only for a single temperature fraction, gives an apparent K-Ar age of 110 m.y., which agrees well with the isochron age. The total fusion age (103.7 m.y.) is slightly less than the isochron age, suggesting a slight loss of the radiogenic  $^{40}\text{Ar}$  from the sample. From the linearity in the isochron plot and from the concordance between the isochron age and the major part of the apparent K-Ar age, we conclude that the sample is 110 m.y. old.

<sup>1</sup> Initial Reports of the Deep Sea Drilling Project, Volume 61.

<sup>2</sup> Present address: Department of Earth Sciences, Yamagata University, Yamagata-City, 990, Japan.

Table 1. Analytical data for age dating.

Temperature (°C)	$^{40}\text{Ar}^*/^{36}\text{Ar}^*$ (% error)	$^{39}\text{Ar}^*/^{36}\text{Ar}^*$ (% error)	$^{37}\text{Ar}/^{36}\text{Ar}$ (% error)	$^{39}\text{Ar}$ (% fraction)	Apparent Age (m.y.) (1 $\sigma$ )
462-60-1, 65-69 cm					
$J = 0.003666$					
500	553.1 (2.5)	10.9 (2.6)	7.37 (2.9)	21.5	150 (4.7)
600	511.0 (1.4)	15.1 (1.4)	15.4 (1.4)	29.6	92.2 (2.2)
700	334.4 (2.5)	10.6 (2.5)	102.1 (2.5)	18.7	24.1 (4.5)
800	399.3 (4.4)	12.5 (4.8)	180.2 (5.2)	7.0	53.9 (6.8)
900	537.2 (1.2)	18.5 (12.4)	178.6 (14.6)	3.8	84.5 (12.5)
1000	286.2 (6.8)	4.82 (6.8)	28.2 (6.8)	4.4	-12.8 (-28)
Fusion	363.4 (3.8)	12.6 (3.8)	407.7 (3.8)	14.9	35.2 (5.8)
Total fusion age = 76.5 $\pm$ 2.3 m.y.					
462A-32-1, 46-49 cm					
$J = 0.003502$					
500	294.3 (2.4)	0.007 (50.2)	0.16 (4.9)	0.19	-159 (-1488)
600	241.1 (1.1)	1.44 (1.9)	8.37 (1.2)	1.56	-256 (-16.8)
700	323.4 (0.5)	1.39 (1.4)	13.2 (0.6)	2.02	122.4 (7.0)
800	340.4 (2.3)	2.12 (2.6)	26.0 (2.3)	1.39	128.9 (19.0)
900	359.9 (1.8)	2.53 (2.2)	23.1 (2.0)	3.93	154.2 (12.7)
1000	410.2 (5.1)	7.83 (5.2)	35.5 (5.2)	4.39	90.2 (11.7)
Fusion	947.5 (1.4)	36.4 (1.6)	121.3 (1.5)	86.7	109.6 (1.8)
Total fusion age = 103.7 $\pm$ 11.2 m.y.					
462A-50-3, 130-134 cm					
$J = 0.003511$					
500	307.5 (2.7)	0.30 (1.6)	2.06 (4.8)	2.7	236 (150)
600	271.2 (2.4)	0.87 (5.9)	10.1 (2.9)	6.4	-186 (-58)
700	362.5 (1.3)	1.54 (4.8)	25.6 (1.4)	13.4	256 (18)
800	309.0 (2.4)	1.09 (4.0)	35.6 (2.5)	9.5	77.1 (40)
900	404.8 (2.9)	1.71 (4.3)	81.3 (2.9)	10.5	366 (28)
1000	299.0 (4.6)	2.43 (4.6)	107.1 (4.6)	14.8	8.9 (35)
Fusion	389.4 (7.3)	4.91 (9.2)	431.3 (9.6)	42.7	117 (27)
Total fusion age = 130.6 $\pm$ 15 m.y.					

Table 2. Final age dating results and associated data.

Sample (interval in cm)	Rock Type	Total Fusion Age <sup>a</sup> (m.y.)	Plateau Age (m.y.)	Isochron Age (MSUM) <sup>b</sup> (m.y.)
462-60-1, 65-69	Clinopyroxene-plagioclase phyric basalt	76.5 $\pm$ 2.3	—	120
462A-32-1, 46-49	Clinopyroxene-plagioclase phyric basalt	104 $\pm$ 11	110 <sup>c</sup>	110 $\pm$ 3 (2.86) except for 600°C fraction
462A-50-3, 130-134	Dolerite	130 $\pm$ 15	—	—

Note:  $\lambda = 5.543 \times 10^{-10} \text{ yr}^{-1}$ ,  $\lambda_e = 0.581 \times 10^{-10} \text{ yr}^{-1}$ ,  $^{40}\text{K}/\text{K} = 0.0001167$

<sup>a</sup> Total fusion age =  $\frac{1}{\lambda} \ln [1 + J(^{40}\text{Ar}/^{39}\text{Ar})_{\text{total}}]$ .  $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{total}} = \sum f_i (^{40}\text{Ar}/^{39}\text{Ar})_i$ .  $f_i = ^{39}\text{Ar}$  fraction released.

<sup>b</sup> MSUM = SUMS/ $N-2$ .  $N$  = number of fractions (Brooks et al., 1972).

<sup>c</sup> Only for the fusion fraction, which constitutes more than 80% of the  $^{39}\text{Ar}$  released.

### Sample 462A-50-3, 130-134 cm

This sample gave very scattered data points in the isochron plot and a very ragged age spectrum, neither of which provided any useful age information. The total fusion age is 131 m.y., the significance of which, however, is difficult to judge.

### DISCUSSION

The three samples we chose for the dating have the highest K-content among the rocks drilled at Site 462. According to the onboard XRF analyses,  $\text{K}_2\text{O}$  contents are 0.67% for Sample 462-60-1, 65-69 cm, 0.78% for Sample 462A-32-1, 46-49 cm, and 0.44% for Sample 462A-50-3, 130-134 cm; most of the cores have  $\text{K}_2\text{O}$  contents less than 0.1%. This suggests that the high K-content in the present samples may be due to some K-bearing alteration products, although microscopic examinations did not give any positive evidence of such K-bearing alteration products (Tokuyama, written communications, 1979). A preliminary EMP analysis

showed that K residues essentially along the grain boundaries. At present we cannot conclude whether the observed high K-content in the samples is due to some submicroscopic K-rich alteration products or is a primary characteristic.

According to the *Initial Core Descriptions*, Sample 462A-32-1, 46-49 cm is from a sill intruded into Cretaceous (Cenomanian) sediment. Hence, the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  age is in accordance with the fossil record. The concordance between the radiometric age and the fossil record is most easily explained by assuming that the radiometric age represents the time of the sill intrusion. It is also possible, however, that the radiometric age represents the formation age of some K-bearing alteration products, since formation of K-rich alteration products almost simultaneous with the emplacement of submarine volcanic rocks seems to be rather general (Hart and Staudigel, 1978). Whichever the case is, the radiometric age should give a good indication of the time of basalt intrusion. Hence, the radiometric age for Sample 462A-32-1, 46-49 cm clearly indicates the ex-

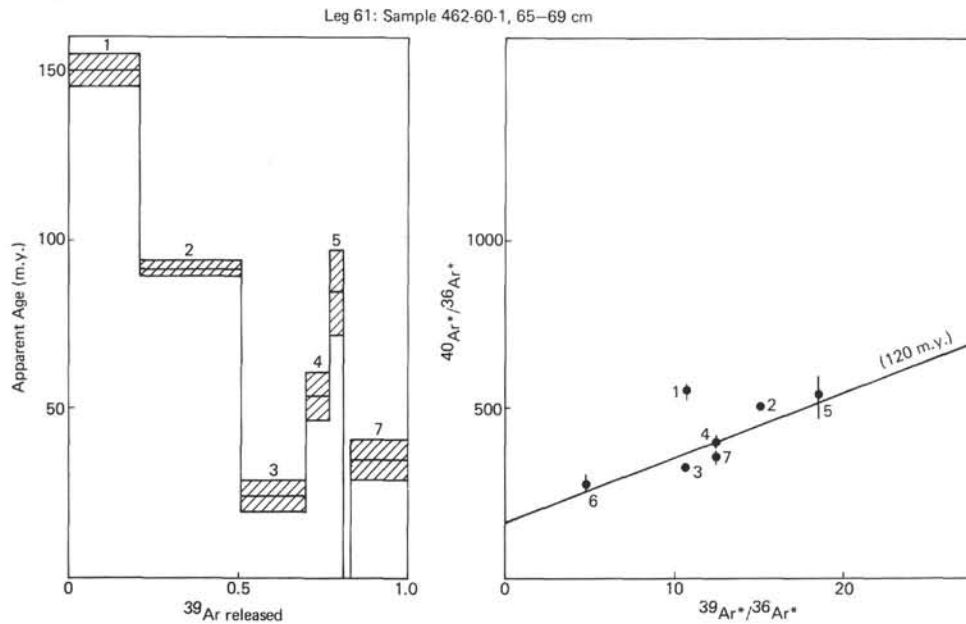


Figure 1. Apparent age spectrum (left) and isochron plot (right). Numerals 1, 2, 3, ... attached to each data point in the isochron plot correspond to temperatures 500°C, 600°C, 700°C, 800°C, 900°C, 1000°C, and fusion temperature, respectively. Asterisk in the isotopic ratios indicates the value corrected for interfering Ar isotopes induced from Ca and K by the neutron irradiation. Shadow for the apparent age indicates  $1\sigma$  error.

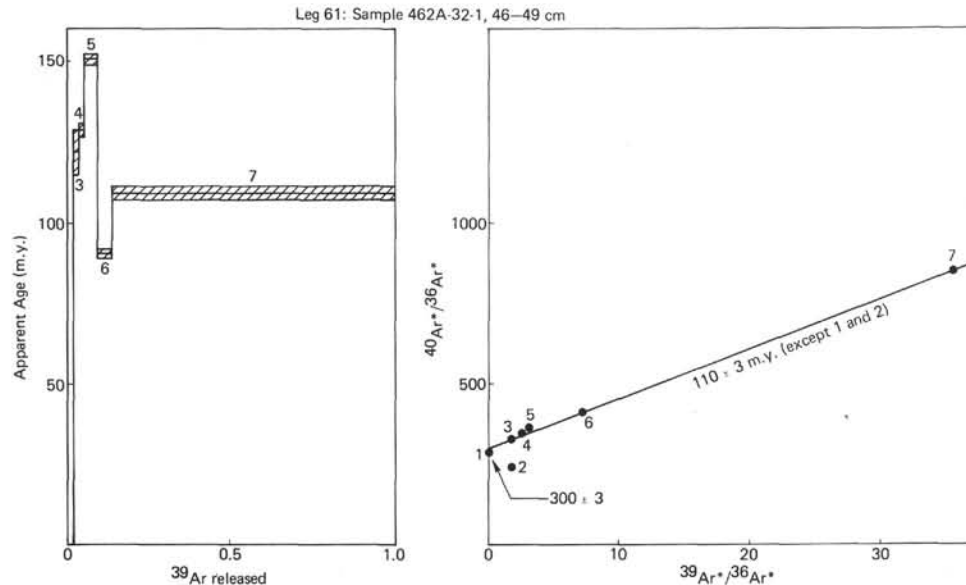


Figure 2. Notations are the same as in Figure 1. Error bars in the isochron plot indicate  $1\sigma$  (which was too small to be shown in Figure 1).

istence of much younger volcanism than in the oceanic basement, as deduced from the magnetic anomaly *M*-26 (~155 m.y.).

#### REFERENCES

- Brooks, C., Hart, S. R., and Wendt, I., 1972. Realistic use of two-error regression treatments as applied to rubidium-strontium data. *Rev. 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.  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  ages of guyots in the western Pacific and discussion of their evolution. *Geophys. J. R. Astr. Soc.*, 51:475-485.
- Ozima, M., Kaneoka, I., and Yanagisawa, M., 1979. Temperature and pressure effects on  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  systematics. *Earth Planet. Sci. Lett.*, 42:463-472.
- York, D., 1969. Least square fitting of a straight line with correlated errors. *Earth Planet. Sci. Lett.*, 5:320-324.