

High-temperature memory in calcic amphiboles and constraints on compositional control of their $^{40}\text{Ar}/^{39}\text{Ar}$ ages

Wolfgang Siebel Geo Forschungs Zentrum Potsdam, Telegrafenberg, D-14473 Potsdam, Germany

Friedhelm Henjes-Kunst Federal Institute for Geosciences and Natural Resources, Stilleweg 2, D-30655 Hannover, Germany

Dieter Rhede Geo Forschungs Zentrum Potsdam, Telegrafenberg, D-14473 Potsdam, Germany

Analytical techniques

For Ar-Ar analysis, sample aliquots of about 30–250 mg (depending on K₂O concentrations and estimated ages) were wrapped in aluminum foil and tightly stacked into a quartz ampoule. An intralaboratory standard biotite (SN: 313.7 ± 0.9 Ma; 1σ error), which was used as a flux monitor, was placed in double aliquots on top and bottom of each stack and in single aliquots between each sample. The ampoules were evacuated, preheated at 150 °C, and sealed. The quartz ampoules were shielded from thermal neutrons by a 1-mm-thick Cd liner and then sealed into an aluminum container. Irradiation with fast neutrons was performed for 96 h in position I/44 of the 5 MW scientific reactor FRG I, Forschungszentrum GKSS, Geesthacht. Gas was extracted during 11 to 19 heating steps, each step with a constant temperature for 30 minutes, automatically controlled via a thermocouple. The last step was run at a maximum temperature of 1550 °C in order to ensure total degassing of the samples. The gas released at each step was trapped by cooled charcoal and then purified by a zirconium-aluminum getter heated to 800 °C for 3 min. The intensities at masses 40, 39, 37, and 36 were measured by peak-jumping on a VG MM 1200 mass spectrometer. The isotopic ratios calculated from the mass intensities were corrected for mass discrimination, total-system blank, and decay of ^{39}Ar and ^{37}Ar . Corrections for interfering isotopes were made with factors determined previously for irradiation at GKSS. The neutron flux gradient along the sample capsule was corrected by regression of the calibration factor $f = ({}^{40}\text{Ar}_{\text{rad}}/{}^{39}\text{Ar}_K)/({}^{40}\text{Ar}_{\text{rad}}/K)_{\text{conventional}}$ determined for the mica monitors. The error in the $^{40}\text{Ar}/{}^{39}\text{Ar}$ age was calculated by error propagation of uncertainties including those of the in-run statistical parameters, the errors derived for the blank contributions, the correction factors, and the flux-calibration factor. The IUGS-recommended constants (Steiger and Jäger, 1977) were used.

REFERENCES

- Steiger, R.H., and Jäger, E, 1977, Subcommission on geochronology: Convention on the use of decay constants in geo- and cosmochronology: Earth and Planetary Science Letters, v. 36, p. 359–362.

^{40}Ar - ^{39}Ar analytical data of hornblende R2b (200-112 μm)

| No. | T (°C) | R/(R+A) (%) | $^{40}\text{K}/^{40}\text{Ar}$ (10 ¹) | $^{36}\text{Ar}/^{40}\text{Ar}$ (10 ⁻⁴) | $^{37}\text{Ar}/^{39}\text{Ar}$ (10 ⁰) | $^{39}\text{Ar}/\text{Sum}^{39}\text{Ar}$ (%) | Date (Ma) |
|-------|-------------------------------|----------------|--|--|---|--|--------------|
| 0 | 600 | 71.53 | 3.792 | 9.635 | 3.535 | 2.53 | 298.6 |
| | +/-2s | 1.42 | 0.025 | 0.482 | 0.038 | | 5.9 |
| 1 | 700 | 85.50 | 6.182 | 4.908 | 1.888 | 5.03 | 223.6 |
| | +/-2s | 0.87 | 0.040 | 0.294 | 0.022 | | 2.6 |
| 2 | 800 | 94.72 | 7.143 | 1.787 | 1.120 | 5.50 | 214.9 |
| | +/-2s | 0.45 | 0.039 | 0.151 | 0.031 | | 1.5 |
| 3 | 900 | 93.93 | 5.694 | 2.055 | 5.395 | 7.71 | 263.7 |
| | +/-2s | 0.46 | 0.032 | 0.157 | 0.033 | | 1.9 |
| 4 | 940 | 94.45 | 4.785 | 1.877 | 13.218 | 10.31 | 311.3 |
| | +/-2s | 0.59 | 0.026 | 0.199 | 0.073 | | 2.4 |
| 5 | 960 | 96.11 | 4.748 | 1.316 | 4.813 | 9.72 | 318.5 |
| | +/-2s | 0.22 | 0.025 | 0.074 | 0.442 | | 1.7 |
| 6 | 980 | 96.89 | 4.780 | 1.052 | 4.125 | 12.40 | 318.9 |
| | +/-2s | 0.21 | 0.027 | 0.071 | 0.429 | | 1.8 |
| 7 | 1000 | 97.54 | 4.798 | 0.831 | 3.293 | 13.47 | 319.8 |
| | +/-2s | 0.15 | 0.025 | 0.052 | 0.354 | | 1.6 |
| 8 | 1020 | 97.84 | 4.812 | 0.732 | 3.269 | 9.45 | 319.8 |
| | +/-2s | 0.17 | 0.025 | 0.057 | 0.152 | | 1.6 |
| 9 | 1040 | 97.18 | 4.817 | 0.953 | 4.826 | 5.03 | 317.6 |
| | +/-2s | 0.43 | 0.027 | 0.146 | 0.231 | | 2.14 |
| 10 | 1070 | 95.42 | 4.622 | 1.550 | 10.410 | 4.01 | 324.4 |
| | +/-2s | 0.73 | 0.032 | 0.248 | 0.074 | | 3.1 |
| 11 | 1100 | 95.71 | 4.593 | 1.451 | 11.697 | 2.18 | 327.1 |
| | +/-2s | 0.85 | 0.036 | 0.286 | 0.094 | | 3.6 |
| 12 | 1130 | 94.90 | 4.341 | 1.725 | 11.229 | 2.74 | 341.7 |
| | +/-2s | 0.56 | 0.033 | 0.191 | 0.226 | | 3.0 |
| 13 | 1200 | 95.35 | 4.318 | 1.573 | 12.802 | 5.39 | 344.9 |
| | +/-2s | 0.41 | 0.031 | 0.138 | 0.093 | | 2.6 |
| 14 | 1300 | 94.66 | 4.298 | 1.807 | 18.609 | 3.55 | 344.1 |
| | +/-2s | 0.59 | 0.039 | 0.200 | 0.171 | | 3.5 |
| 15 | 1550 | 91.66 | 4.149 | 2.822 | 15.093 | 0.97 | 345.1 |
| | +/-2s | 1.46 | 0.064 | 0.571 | 0.248 | | 7.0 |
| TOTAL | | 94.80 | 4.878 | 1.759 | 6.478 | 100.00 | 306.9 |
| | +/-2s | 0.12 | 0.009 | 0.040 | 0.086 | | 0.7 |
| | +/-2s (intralaboratory error) | | | | | | 1.3 |

40Ar-39Ar analytical data of biotite R2b (315-250 µm)

| No. | T (°C) | R/(R+A) (%) | $^{40}\text{K}/^{40}\text{Ar}$ (10^1) | $^{36}\text{Ar}/^{40}\text{Ar}$ (10^{-5}) | $^{37}\text{Ar}/^{39}\text{Ar}$ (10^{-2}) | $^{39}\text{Ar}/\text{Sum}^{39}\text{Ar}$ (%) | Date (Ma) |
|-------|-------------------------------|----------------|--|--|--|--|--------------|
| 0 | 700 | 94.84 | 5.011 | 17.451 | 2.857 | 27.95 | 299.5 |
| | +/-2s | 0.11 | 0.027 | 0.363 | 2.422 | | 1.6 |
| 1 | 800 | 99.52 | 5.143 | 1.612 | 1.672 | 21.60 | 305.6 |
| | +/-2s | 0.09 | 0.026 | 0.296 | 3.877 | | 1.5 |
| 2 | 900 | 99.42 | 5.157 | 1.957 | 1.444 | 17.97 | 304.6 |
| | +/-2s | 0.09 | 0.027 | 0.305 | 2.866 | | 1.5 |
| 3 | 940 | 99.50 | 5.167 | 1.691 | -0.524 | 9.68 | 304.3 |
| | +/-2s | 0.26 | 0.027 | 0.871 | 6.417 | | 1.6 |
| 4 | 960 | 99.59 | 5.164 | 1.394 | 3.283 | 7.27 | 304.7 |
| | +/-2s | 0.20 | 0.027 | 0.663 | 3.590 | | 1.5 |
| 5 | 1005 | 99.54 | 5.149 | 1.563 | 0.456 | 3.96 | 305.4 |
| | +/-2s | 0.14 | 0.027 | 0.475 | 8.614 | | 1.6 |
| 6 | 1030 | 99.53 | 5.143 | 1.590 | 2.296 | 5.03 | 305.6 |
| | +/-2s | 0.24 | 0.028 | 0.798 | 6.172 | | 1.6 |
| 7 | 1050 | 99.59 | 5.128 | 1.388 | 5.848 | 4.24 | 306.6 |
| | +/-2s | 0.21 | 0.028 | 0.700 | 5.220 | | 1.6 |
| 8 | 1070 | 100.13 | 5.122 | -0.441 | 10.309 | 1.70 | 308.5 |
| | +/-2s | 0.48 | 0.035 | 1.623 | 15.346 | | 2.4 |
| 9 | 1090 | 100.28 | 5.166 | -0.931 | 8.007 | 0.52 | 306.5 |
| | +/-2s | 2.06 | 0.128 | 6.986 | 54.394 | | 9.1 |
| 10 | 1110 | 99.87 | 5.224 | 0.425 | 135.784 | 0.09 | 302.3 |
| | +/-2s | 10.79 | 0.299 | 36.364 | 280.212 | | 33.9 |
| TOTAL | | 98.19 | 5.111 | 6.119 | 2.333 | 100.00 | 303.6 |
| | +/-2s | 0.05 | 0.012 | 0.181 | 1.535 | | 0.7 |
| | +/-2s (intralaboratory error) | | | | | | 1.3 |

⁴⁰Ar-³⁹Ar analytical data of hornblende RL25 (200-112 µm)

| No. | T (°C) | R/(R+A) (%) | ⁴⁰ K/ ⁴⁰ Ar (10 ¹) | ³⁶ Ar/ ⁴⁰ Ar (10 ⁻⁴) | ³⁷ Ar/ ³⁹ Ar (10 ¹) | ³⁹ Ar/Sum ³⁹ Ar (%) | Date (Ma) |
|-------|---------------|-------------------------------|---|---|--|--|--------------|
| 0 | 600 +/-2s | 60.90 0.74 | 2.872 0.025 | 13.232 0.253 | 0.203 0.002 | 1.82 | 332.4 4.8 |
| 1 | 700 +/-2s | 79.02 0.28 | 4.497 0.027 | 7.101 0.098 | 0.104 0.001 | 4.29 | 279.6 2.0 |
| 2 | 800 +/-2s | 91.25 0.73 | 5.289 0.031 | 2.961 0.246 | 0.089 0.001 | 4.30 | 274.9 2.6 |
| 3 | 900 +/-2s | 91.55 0.24 | 4.730 0.026 | 2.858 0.081 | 0.385 0.002 | 6.21 | 305.7 1.8 |
| 4 | 940 +/-2s | 91.58 0.45 | 4.309 0.027 | 2.850 0.152 | 1.185 0.007 | 5.84 | 333.1 2.5 |
| 5 | 960 +/-2s | 92.42 0.12 | 4.355 0.023 | 2.565 0.042 | 1.471 0.008 | 10.05 | 332.6 1.8 |
| 6 | 980 +/-2s | 94.02 0.28 | 4.529 0.024 | 2.024 0.096 | 1.385 0.007 | 13.10 | 326.0 1.9 |
| 7 | 1000 +/-2s | 95.95 0.31 | 4.667 0.025 | 1.369 0.104 | 1.245 0.007 | 12.60 | 323.1 1.9 |
| 8 | 1020 +/-2s | 96.34 0.16 | 4.713 0.026 | 1.239 0.054 | 1.134 0.006 | 12.77 | 321.4 1.7 |
| 9 | 1040 +/-2s | 95.06 0.43 | 4.710 0.033 | 1.673 0.146 | 1.088 0.008 | 4.04 | 317.7 2.4 |
| 10 | 1070 +/-2s | 96.16 0.82 | 4.654 0.042 | 1.300 0.277 | 1.179 0.011 | 3.14 | 324.6 3.7 |
| 11 | 1100 +/-2s | 94.99 0.29 | 4.528 0.032 | 1.694 0.100 | 1.386 0.010 | 2.91 | 329.2 2.3 |
| 12 | 1130 +/-2s | 94.37 1.04 | 4.454 0.029 | 1.906 0.353 | 1.508 0.010 | 3.32 | 332.1 3.9 |
| 13 | 1200 +/-2s | 94.82 0.41 | 4.343 0.033 | 1.752 0.139 | 1.648 0.013 | 5.17 | 341.3 2.8 |
| 14 | 1300 +/-2s | 93.99 0.32 | 4.305 0.024 | 2.034 0.110 | 1.864 0.011 | 7.57 | 341.3 2.1 |
| 15 | 1550 +/-2s | 94.03 0.32 | 4.283 0.036 | 2.019 0.156 | 1.790 0.014 | 2.88 | 343.1 2.9 |
| TOTAL | | 92.58 +/-2s | 4.501 0.008 | 2.513 0.033 | 1.185 0.002 | 100.00 | 323.2 0.7 |
| | | +/-2s (intralaboratory error) | | | | | 1.3 |

^{40}Ar - ^{39}Ar analytical data of biotite RL25 (500-400 μm)

| No. | T (°C) | R/(R+A) (%) | $^{40}\text{K}/^{40}\text{Ar}$ (10^1) | $^{36}\text{Ar}/^{40}\text{Ar}$ (10^{-5}) | $^{37}\text{Ar}/^{39}\text{Ar}$ (10^{-2}) | $^{39}\text{Ar}/\text{Sum}^{39}\text{Ar}$ (%) | Date (Ma) |
|-------|-------------------------------|----------------|--|--|--|--|--------------|
| 0 | 500 | 37.54 | 3.187 | 211.357 | 76.921 | 0.35 | 192.1 |
| | +/-2s | 2.84 | 0.050 | 9.629 | 307.595 | | 14.2 |
| 1 | 550 | 74.37 | 4.395 | 86.739 | 2.688 | 1.10 | 270.0 |
| | +/-2s | 1.25 | 0.030 | 4.232 | 13.390 | | 4.6 |
| 2 | 580 | 84.86 | 4.745 | 51.227 | -12.763 | 1.85 | 284.2 |
| | +/-2s | 0.76 | 0.027 | 2.576 | 41.598 | | 2.9 |
| 3 | 600 | 92.94 | 4.755 | 23.890 | 0.001 | 2.44 | 308.5 |
| | +/-2s | 0.53 | 0.028 | 1.784 | 30.344 | | 2.4 |
| 4 | 620 | 96.53 | 4.885 | 11.738 | -4.240 | 3.49 | 311.6 |
| | +/-2s | 0.19 | 0.035 | 0.649 | 40.632 | | 2.2 |
| 5 | 650 | 96.05 | 4.820 | 13.352 | 2.095 | 5.65 | 314.0 |
| | +/-2s | 0.14 | 0.026 | 0.487 | 18.328 | | 1.6 |
| 6 | 680 | 99.21 | 4.947 | 2.672 | 1.629 | 9.08 | 315.9 |
| | +/-2s | 0.24 | 0.030 | 0.813 | 22.098 | | 1.9 |
| 7 | 720 | 99.51 | 4.943 | 1.659 | -1.927 | 10.75 | 316.9 |
| | +/-2s | 0.13 | 0.027 | 0.452 | 14.559 | | 1.7 |
| 8 | 780 | 99.67 | 4.946 | 1.109 | 0.001 | 8.41 | 317.2 |
| | +/-2s | 0.22 | 0.027 | 0.753 | 20.339 | | 1.7 |
| 9 | 830 | 99.58 | 4.937 | 1.406 | -4.221 | 5.71 | 317.6 |
| | +/-2s | 0.22 | 0.027 | 0.729 | 24.231 | | 1.7 |
| 10 | 870 | 99.58 | 4.942 | 1.414 | -5.987 | 4.03 | 317.2 |
| | +/-2s | 0.32 | 0.027 | 1.077 | 37.344 | | 1.9 |
| 11 | 910 | 99.61 | 4.956 | 1.309 | 5.503 | 8.76 | 316.5 |
| | +/-2s | 0.16 | 0.026 | 0.537 | 9.308 | | 1.6 |
| 12 | 940 | 99.94 | 4.961 | 0.197 | -1.479 | 4.08 | 317.2 |
| | +/-2s | 0.15 | 0.026 | 0.494 | 21.476 | | 1.6 |
| 13 | 970 | 99.81 | 4.963 | 0.646 | 0.756 | 7.98 | 316.7 |
| | +/-2s | 0.15 | 0.026 | 0.513 | 14.352 | | 1.6 |
| 14 | 1000 | 99.78 | 4.964 | 0.739 | -3.029 | 7.97 | 316.5 |
| | +/-2s | 0.13 | 0.026 | 0.430 | 16.256 | | 1.6 |
| 15 | 1040 | 99.72 | 4.951 | 0.937 | 0.610 | 9.89 | 317.1 |
| | +/-2s | 0.19 | 0.029 | 0.649 | 14.313 | | 1.8 |
| 16 | 1100 | 99.34 | 4.919 | 2.226 | -1.584 | 7.62 | 317.9 |
| | +/-2s | 0.21 | 0.033 | 0.701 | 11.897 | | 2.1 |
| 17 | 1550 | 100.43 | 4.983 | -1.439 | 28.175 | 0.86 | 317.3 |
| | +/-2s | 1.03 | 0.051 | 4.136 | 102.295 | | 4.2 |
| TOTAL | | 98.20 | 4.914 | 6.084 | -0.090 | 100.00 | 314.8 |
| | +/-2s | 0.06 | 0.009 | 0.200 | 5.192 | | 0.5 |
| | +/-2s (intralaboratory error) | | | | | | 1.2 |