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^{40}Ar-^{39}Ar studies of whole rock nakhlites: Evidence for the timing of formation and aqueous alteration on Mars

Timothy D. SWINDLE* and Eric K. OLSON

Lunar and Planetary Laboratory, University of Arizona, Tucson, Arizona 85721–0092, USA
*Corresponding author. E-mail: tswindle@u.arizona.edu

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Abstract–20–25 mg whole rock samples of the nakhlites Lafayette and Nakhla have been analyzed via the ^{40}Ar-^{39}Ar technique, in part to verify their formation ages, but primarily, in an attempt to determine the timing of aqueous alteration in these martian meteorites. As in previous studies, plateaus in apparent age are observed at about 1300 Ma (1322 ± 10 for Lafayette, 1332 ± 10 and 1323 ± 11 for Nakhla), presumably corresponding to crystallization ages. The plateaus are not entirely flat, perhaps reflecting the effects of recoil during creation of ^{39}Ar in the nuclear irradiation. The first 5–20% of the K-derived Ar released from all three samples give apparent ages <1300 Ma. Coupled with the fact that chronometric isotopic studies of nakhlites typically show some disturbance, we believe the low temperature pattern represents more recent (than 1300 Ma) formation of martian aqueous alteration products such as iddingsite. No low temperature plateaus are observed. This is consistent with petrographic evidence for multiple formation events, although the lack of low temperature plateaus is far from conclusive. On the other hand, if there was a single time of alteration, we believe that it will be difficult, if not impossible, to determine it using the K-Ar system.

INTRODUCTION

For more than a century, the topic of liquid water on Mars (e.g., Lowell 1895; Malin and Edgett 2000) has been a major part of the intense scientific interest in the fourth planet from the Sun. Among the martian meteorites, the most-studied evidence for alteration by liquid water is within the nakhlites (e.g., Bridges et al. 2001). Bunch and Reid (1975) first noted "iddingsite" within Lafayette, and Ashworth and Hutchison (1975) soon identified aqueous alteration products in Nakhla. Later authors showed that the "iddingsite" is a mixture of smectite clays, iron oxides, and ferrihydrides and that petrographic relationships verify that the iddingsite is martian not terrestrial (Treiman et al. 1993). But when did that alteration occur?

Nyquist et al. (2001) reviewed all geochronological studies of martian meteorites as of mid-2000. They point out that nakhlites seem to have relatively simple histories in all systems in which they have been studied (Rb-Sr, Sm-Nd, U-Th-Pb, and K-Ar), indicating crystallization at ~1300 Ma. Their best estimates of the formation ages are 1270 ± 10 for Nakhla and 1320 ± 20 for Lafayette, nearly, but not quite, contemporaneous.

However, virtually every study has shown at least some minor complications, starting with the fact that the first two Rb-Sr studies (Gale et al. 1975; Papanastassiou and Wasserburg 1974) both produced linear arrays, but each had some data points not on the array, and the two studies gave slightly different results. Part of the discrepancy could be the result of sample heterogeneity of the gram-sized samples (Nyquist et al. 2001), but one of the first studies of nakhlite alteration products (Ashworth and Hutchison 1975) already suggested the possibility that the iddingsite could be the cause of some of the problems. Chen and Wasserburg (1986) noted "relatively recent" Pb loss in Nakhla. Although they did not attribute it to the formation of iddingsite, Nakhla has had a relatively benign existence otherwise. It is worth noting here that, for iddingsite formation to affect any of these systems, the alteration would have to postdate crystallization by a significant amount of time, since evidence for any disturbance very shortly after formation would have been smoothed over by the intervening isotopic evolution.

The most detailed study of the ages of alteration products in Lafayette was by Swindle et al. (2000), who separated 1–23 mg samples of iddingsite. They determined K-Ar ages, finding the K and ^{40}Ar abundances of each sample and calculating an age based on the decay of ^{40}K to ^{40}Ar (1280 Ma half-life). They obtained ages ranging from 0 to nearly 700 Ma, the latter agreeing (Table 1) with a two-point Rb-Sr "isochron" from Lafayette acid leachates attributed to the

Table 1. Alteration ages in nakhlites.

Sample	Technique	Type of sample	Age (Ma)	Reference
Lafayette	K-Ar	Iddingsite	0–679 ± 91	Swindle et al. (2000)
Lafayette	Rb-Sr 2-point "isochron"	Iddingsite-rich HCl leachate	679 ± 66	Shih et al. (1998)
Governador Valadares	^{40}Ar-^{39}Ar 600 °C extraction	Whole rock	880 ± 32	Bogard and Husain (1977)

weathering products (Shih et al. 1998). A low temperature extraction from a ^{40}Ar-^{39}Ar study of the nakhlite Governador Valadares (Bogard and Husain 1977) gave a similar apparent age, although it could also represent partial loss of ^{40}Ar from some other phase.

Normally, the ^{40}Ar-^{39}Ar technique is preferred to the K-Ar technique, but Swindle et al. (2000) used the latter in studying the Lafayette iddingsite. The difference between a K-Ar and a ^{40}Ar-^{39}Ar experiment is that, in the ^{40}Ar-^{39}Ar experiment, the sample is irradiated with neutrons. This converts a fraction of the ^{39}K in the sample into ^{39}Ar, which then serves as a proxy for the elemental abundance of K. A step-wise heating experiment can then determine the apparent age of a series of steps, potentially making it possible to detect different ages from different minerals or to detect the effects of diffusion from a mineral (McDougall and Harrison 1999; Turner and Cadogan 1974). However, in fine-grained samples, the recoil of ^{39}Ar that occurs as it is produced in the reactor can be sufficient to eject many of the ^{39}Ar atoms. This commonly observed effect (e.g., McDougall and Harrison 1999; Turner and Cadogan 1974) can lead to erroneously high apparent ages in the sites from which recoil occurs and to erroneously low apparent ages in the sites into which the ^{39}Ar recoils (radiogenic ^{40}Ar does not experience enough recoil in its production for this to be a problem). Swindle et al. (2000) performed a preliminary ^{40}Ar-^{39}Ar study on a few iddingsite samples, which were encapsulated in evacuated vials (cf., Foland et al. 1992; Smith et al. 1993), and demonstrated that a significant amount of recoil does occur from the fine-grained alteration products. This eventually led Swindle et al. (2000) to perform a K-Ar experiment, which does not use ^{39}Ar produced by irradiation to determine K contents, instead of a ^{40}Ar-^{39}Ar experiment. Another advantage to the K-Ar technique was that, by not having to crack vials in vacuum and then analyze friable samples that remained in different places in the vial, Swindle et al. (2000) were able to minimize mechanical loss of sample.

However, there were some troubling aspects of the K-Ar experiment on Lafayette iddingsite (Swindle et al. 2000). First, they found a range of ages rather than a single age. Although the highest age was in rough agreement with two other determinations, neither of those would be considered convincing. Second, the K-Ar study did not agree well with an earlier ^{40}Ar-^{39}Ar study of Lafayette by Podosek (1973). In that study, a whole rock sample of Lafayette was interpreted as giving a simple result with apparent ages of roughly 1300 Ma for virtually all of the temperature extractions. Swindle et al. (2000) noted that, given the amount of potassium in the iddingsite and the amount in the entire meteorite, the later formation age(s) of the iddingsite should have been apparent in the whole rock study, as low apparent ages at the low temperatures where the iddingsite would be expected to degas, as low apparent ages in low-K extractions in which the effects of recoil would be expected to be seen, or both.

Although the normal pattern is to find a whole rock effect and then to look in mineral separates, we have done the reverse. In the present study, we analyzed one whole rock sample of Lafayette and two whole rock samples of the type nakhlite, Nakhla, all samples roughly three orders of magnitude larger than the iddingsite separates of Swindle et al. (2000). Nakhla also contains aqueous alteration products (Ashworth and Hutchison 1975; Gooding et al. 1991), though they are not as prevalent in thin section as are those in Lafayette (Treiman et al. 1993). Podosek (1973) actually observed a more disturbed Ar-Ar apparent age spectrum for Nakhla (Fig. 1), but most of the disturbance was at the high temperature end. Our primary goal was to use about three times as many temperature steps as Podosek (1973) to look for evidence of aqueous alteration products in low temperature extractions from both meteorites. In addition, we hoped that we might see a "plateau"—several extractions with the same age—in the low temperature data if much or all of the alteration occurred at a single time. Of course, recoil could destroy any plateau that might exist, but in a whole rock sample, the recoil should be evident as low apparent ages in low-K (presumably high temperature) extractions. However, if the amount of recoil loss is related to the ratio of external to internal surfaces in a fine-grained sample (Dong et al. 1995), by minimizing the area of external surface, we hoped to minimize the amount of recoil loss. This is similar to the approach of irradiating clay-rich thin sections, which has been used successfully (Dong et al. 1997a).

There is one other possibility that we can explore. The high temperature drop in apparent ages in the Nakhla sample of Podosek (1973) is accompanied by an increase in the amount of Cl-derived ^{38}Ar released (Gilmour et al. 1998). Gilmour et al. (1998) suggested that the high temperature releases might contain evidence for aqueous alteration products. Although their group found no evidence to support this in a study of various whole rock, etched, and mineral separate samples from Nakhla (Burgess et al. 2000; Gilmour et al. 1998), that does not mean we would not see it in a sample from a separate piece of Nakhla or in a sample from the more iddingsite-rich Lafayette.

Finally, the present study provides another set of data about the crystallization ages of Nakhla and Lafayette. In

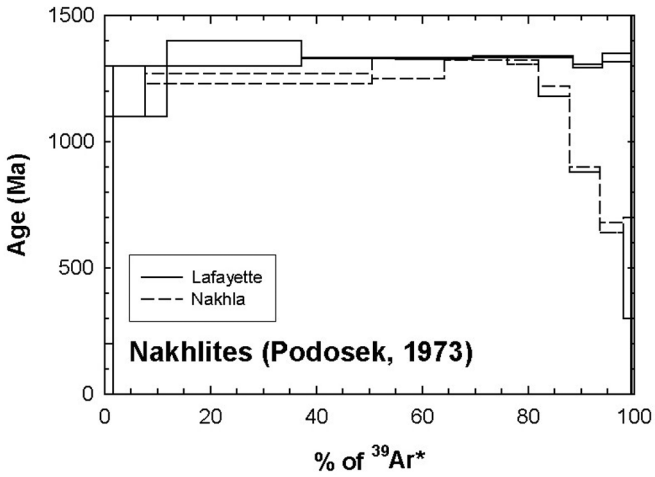

Fig. 1. Plot of apparent ^{40}Ar-^{39}Ar age versus the fraction of K-derived ^{39}Ar released (plateau plots) for whole rock nakhlites analyzed by Podosek (1973).

assigning "best" ages, Nyquist et al. (2001) used the fact that most radiometric ages of Lafayette are older than most radiometric ages of Nakhla and assigned an older age to Lafayette. Using the same technique at the same time on both provides a good test of whether there are real differences.

Mineralogy and Mass Balance

Before discussing the present experiment in detail, it is worthwhile to briefly review the relevant mineralogy of nakhlites. The mineralogy has been discussed in detail elsewhere (Berkley et al. 1980; Bunch and Reid 1975; Gooding et al. 1991; Treiman 1986, 1993; Treiman et al. 1993). We will use the results from these studies, along with whole rock elemental abundances from the tabulation of Lodders (1998), to discuss mass-balance constraints on the three elements (other than Ar) that can be measured in our experiment, K, Ca, and Cl.

Both Nakhla and Lafayette consist primarily (~80%) of a high-Ca pyroxene with 18–19% CaO. This pyroxene is the primary host of Ca in the nakhlites, with enough to account for virtually all the Ca in whole rock analyses. The nakhlites also contain 5–10% olivine, but this should contribute a few percent or less of the K and less than 1% of the total Ca. There is some feldspar, including ~1% K-feldspar (Bunch and Reid 1975), which is presumably the primary source of K. The plagioclase could also contain up to a few percent of the rocks' Ca. Also, some authors have noted the presence of minor amounts of chlorapatite (Berkley et al. 1980; Bunch and Reid 1975), which could be an important host for Cl.

There is, of course, one other minor to trace constituent that is far more important to this experiment than the chlorapatite—the iddingsite discussed above. The iddingsite constitutes as much as a few percent of the rock in Lafayette and contains between 0.3 and 0.8 wt% K (Treiman and Lindstrom 1997). Since whole rock samples of Nakhla and Lafayette have ~0.1 wt% K, the iddingsite could provide several percent of the total K budget. However, the iddingsite is not uniformly distributed. In the first study that identified it, Bunch and Reid (1975) pointed out that iddingsite seemed to be completely absent in some thin sections. In addition, it is apparently more abundant, in general, in Lafayette than in Nakhla (Gooding et al. 1991; Treiman et al. 1993).

PROCEDURES

We analyzed three whole rock nakhlite chips, one from Lafayette and two from Nakhla, each with a mass of 21 to 26 mg (Tables A1–A3). To keep handling to a minimum, samples were wrapped in high-purity tin foil without any crushing, mineral separation, or other sample preparation. They were irradiated at the University of Michigan reactor for 575 hr at the L-67 position. The irradiation was longer than was necessary for the nakhlite analyses, since the irradiation was designed primarily for small (<50 μg) clasts from lunar meteorites (Cohen et al. 2002). The J-factor, determined from analyses of MMhb-1 hornblende, was 8.55×10^{-2}. Different samples of MMhb-1 gave results varying by approximately 1%. Although some of this could be from flux variations related to location within the package, we conservatively took the same J-factor for all samples with a 1% uncertainty. Ultimately, this turns out to be the dominant statistical uncertainty for most extractions, but as will be seen below, the difficulty in interpreting the apparent age spectra does not come from a lack of statistical precision. Samples of CaF_2 were included in the package to monitor interfering reactions from Ca, but the CaF_2 results were inconsistent from sample to sample, and the ratios of ^{37}Ar to other isotopes were frequently lower than we or others have measured in the past for the reactor used, so we suspect the samples were somehow contaminated. However, the pyroxene in nakhlites is so Ca-rich that some of the high temperature extractions in this experiment set very good limits on Ca interference, as will be described below.

Samples were analyzed in the University of Arizona noble gas mass spectrometry laboratory. The extraction line was first baked to ~250 °C overnight, while the samples were held at ~160 °C. For analysis, heating was done in a double-vacuum system, with a Ta crucible inside a W element that was resistance-heated, with its temperature controlled to within approximately 1 °C. One of the primary goals of the experiment was to study the argon that came off at the low temperatures at which we would expect clay and other alteration products to degas, so heating steps began at 250 °C, just above the melting point of the tin foil in which the samples were irradiated, and typically increased by 25 °C at the low temperatures.

Blank corrections were based on an average of blanks run before, during, and after the analyses of the samples. "Cold"

blanks and blanks with the furnace heated differed by less than a factor of two in ^{40}Ar (all roughly 10^{-10} cm^3 STP). Data were then corrected for radioactive decay of ^{37}Ar and ^{39}Ar.

At this point, we needed to correct for interfering reactions produced by Ca. Normally, even if our Ca monitors were not consistent, we could calculate $(^{39}Ar/^{37}Ar)_{Ca}$, the critical ratio, based on previous experiments in the reactor. However, our samples were irradiated in several installments (to accommodate reactor scheduling), so calculation becomes more difficult. Instead, we did a preliminary analysis of the data. In each sample, the highest temperature step with detectable ^{39}Ar (at 1200 °C or 1250 °C) had a ^{39}Ar/^{37}Ar ratio of 7.5×10^{-4} to 8×10^{-4}, which sets an upper limit on the $(^{39}Ar/^{37}Ar)_{Ca}$ ratio. However, we can set better limits by making some assumptions based on the pattern of apparent ages. In all three samples, the 1200 °C or 1250 °C step plus the previous step account for >90% of the $^{37}Ar_{Ca}$ in the sample. We assume this is from pyroxene (see above). Analyses of pure pyroxene separates (Burgess et al. 2000; Gilmour et al. 1998) give ages of approximately 1300 Ma, but in our experiments, the apparent ages are dropping at the temperatures just below that of maximum $^{37}Ar_{Ca}$ release, presumably the result of recoil. If we assume that the highest temperature step with measurable ^{39}Ar does not have an apparent age greater than the plateau at about 1300 Ma, the upper limit becomes 6.0×10^{-4}, which is close to the expected value for the reactor we used (McDougall and Harrison 1999). We somewhat arbitrarily assumed that the apparent age of the highest-Ca extraction from Lafayette should have been roughly 1000 Ma. This is in the range of the 1000 °C and 1100 °C extractions from Nakhla, which show significant recoil effects, but some of which have much higher measured (^{39}Ar/^{37}Ar) ratios and, hence, better-determined apparent ages. In this fashion, we determined a $(^{39}Ar/^{37}Ar)_{Ca}$ ratio of $(5.3 \pm 0.7) \times 10^{-4}$. Using a different lower limit would produce a noticeable change in the results for only the one or two highest-Ca extractions from each sample (for which the apparent age is poorly defined anyway) and would not change any of our conclusions.

In addition, there is Ar produced by cosmic ray spallation of Ca. To determine the isotopic composition of this Ar, we used the highest-Ca extractions. For Nakhla, the average for the two samples gave $(^{36}Ar/^{37}Ar)_{Ca} = 0.000676 \pm 0.000008$ and $(^{38}Ar/^{37}Ar)_{Ca} = 0.001016 \pm 0.000020$ for a ^{38}Ar/^{36}Ar ratio of 1.50. Although the ^{38}Ar/^{36}Ar is consistent with Ca spallation, there is probably also some ^{36}Ar produced from Ca in the reactor (McDougall and Harrison 1999) and some ^{38}Ar produced from Cl. However, the $(^{36}Ar/^{37}Ar)_{Ca}$ from the reactor is expected to be several times smaller than the ratio we measured, so we assumed that the ^{38}Ar was entirely Ca spallation. For Lafayette, the highest-Ca extraction gave ratios of 0.000597 ± 0.000019 and 0.001103 ± 0.000035, which would lead to a ^{38}Ar/^{36}Ar ratio of 1.85. We took the measured $(^{36}Ar/^{37}Ar)_{Ca}$ value and multiplied it by 1.50 for ^{38}Ar on the assumption that the excess ^{38}Ar was from neutron capture on ^{37}Cl. The 15% difference in $(^{36}Ar/^{37}Ar)_{Ca}$ between the two meteorites is surprising, since that ratio should be proportional to the cosmic ratio exposure age and the two nakhlites are believed to have the same exposure age (e.g., Nyquist et al. 2001). We then corrected the data for the Ca interference and Ca spallation, based on the ^{37}Ar amounts. Next, we made a minor correction for reactor-produced K interferences at ^{40}Ar, using typical values for this reactor (McDougall and Harrison 1999), along with K spallation (assumed to be 1% of the total spallation). This correction has little effect, systematically lowering all ages by ~1 Ma for 1300 Ma ages, considerably less than the quoted 1σ uncertainties in every case. Finally, neutron capture on ^{35}Cl produces ^{36}Cl, the half-life (3×10^5 years) of which is short enough that a measurable amount can decay to ^{36}Ar within a year of irradiation. We used the measured ^{38}Ar to correct for Cl-produced ^{36}Ar (Foland et al. 1993), assuming a 10% uncertainty in the ^{36}Ar/^{38}Ar ratio.

At this point, there is still some ^{36}Ar left, particularly in the lower temperature steps, which could be a sign of trapped Ar. At the very lowest temperatures, the ^{40}Ar/^{36}Ar is usually very similar to the terrestrial atmospheric value, suggesting terrestrial contamination. We assumed that all ^{36}Ar at this stage was terrestrial contamination and subtracted appropriate amounts of ^{40}Ar and ^{38}Ar out (see discussion below). In most steps, the amount of ^{36}Ar before this correction is within 1–2σ of zero, but for the sake of completeness, we also corrected for the remaining ^{36}Ar in all temperature steps. This turns out to be a major source of statistical uncertainty in the apparent ages calculated for many extractions. This also drives some of the high temperature apparent ages to zero, where the amount of ^{40}Ar after blank corrections is very small. Although we suspect these high temperature ages may simply represent imperfect blank corrections, rather than incorporated air, we report the apparent ages in Tables A1–A3 in this fashion anyway for the sake of consistency (our conclusions are not affected).

RESULTS

Isotopic ratios (corrected for blanks and radioactive decay but not for Ca, K, or Cl interference or trapped argon) and calculated apparent ages (with all those corrections made) are given in Tables A1–A3 and displayed in Fig. 2. For each sample, most of the gas is part of an apparent plateau. Details of these apparent plateaus are given in Table 2. None of the calculated ages include the uncertainty in the J-factor or the age of the flux monitor, which amount to approximately 9 Ma for a 1300 Ma age.

The Lafayette data (Fig. 2a) display a relatively good plateau over nearly 80% of the ^{39}Ar release. The flattest portion of the plateau is from 625 °C to 825 °C (Table 2), giving an age of 1322 ± 10 Ma over 57% of the ^{39}Ar release.

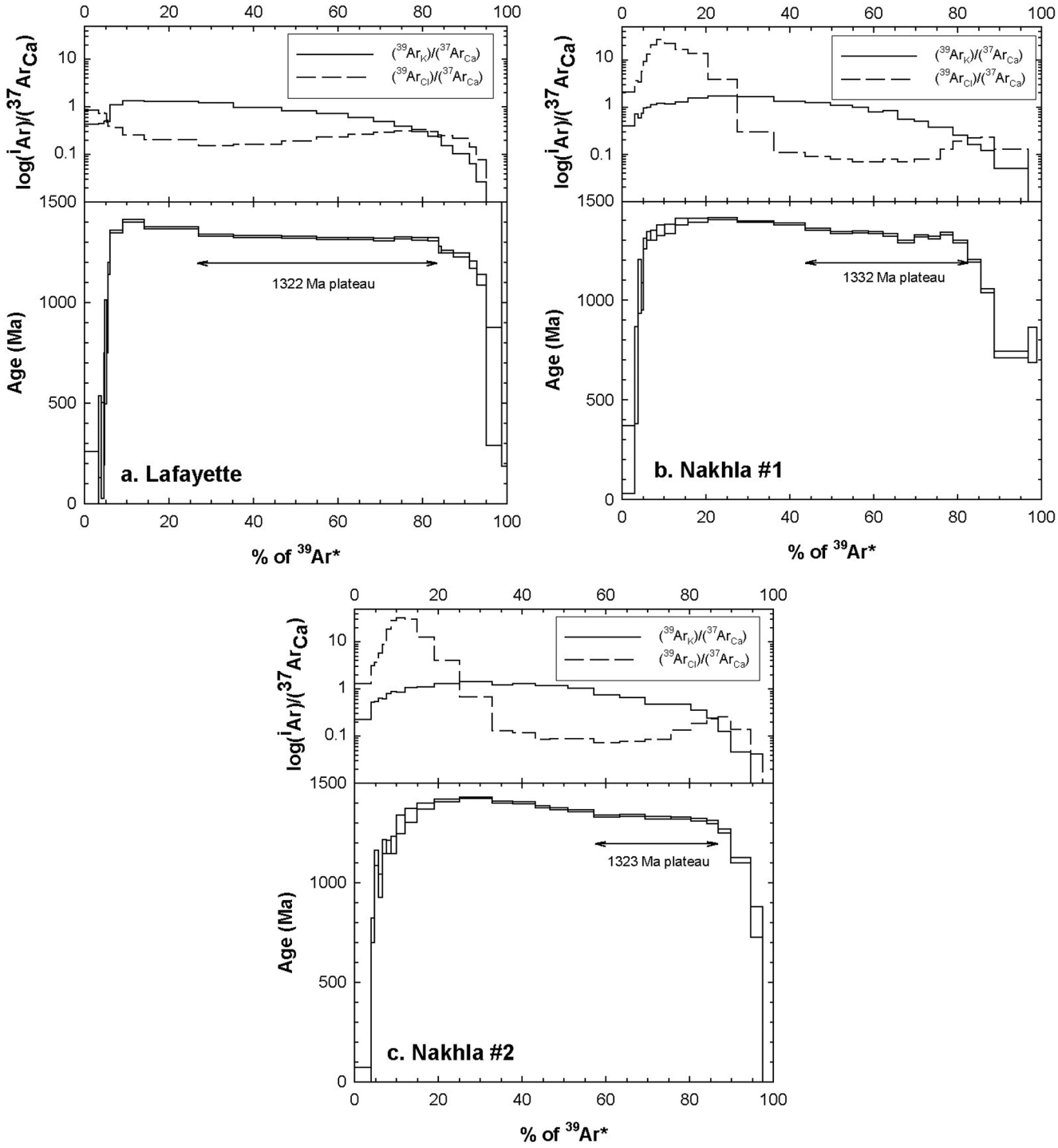

Fig. 2. ^{40}Ar-^{39}Ar plateau plots and plots of relative K/Ca ratios for whole rock nakhlites. The K/Ca plots are given in terms of the ratio of K-derived ^{39}Ar to Ca-derived ^{37}Ar rather than converting to elemental abundances because the irradiation history makes it difficult to accurately make the conversion for Ca abundance. The arrows span the steps included in plateau age calculations (Table 2).

Including the three previous steps, which are not quite in line, changes the plateau age to 1337 Ma. The start of the plateau clearly corresponds to a distinct phase (presumably plagioclase), as shown by the sudden increase in the K/Ca ratio. The end of the plateau is not obvious in the K/Ca plot, so it may represent a range of plagioclase compositions or a mixture with some of the high-Ca pyroxene. The Nakhla data (Figs. 2b and 2c) are quite similar to the Lafayette data with a relatively good plateau in the center of the spectrum and lower apparent ages at each end.

Table 2. Nakhlite ^{40}Ar-^{39}Ar plateau ages.[a]

Sample	Type	Temp. (°C)	% ^{39}Ar	Age (Ma)
Lafayette	Plateau	625–825	57	1322 ± 10
	High-T	500–1250	94	1295 ± 36
	All	250–1250	100	1242 ± 38
Nakhla #1	Plateau	575–750	35	1332 ± 10
	High-T	350–1250	94	1263 ± 29
	All	250–1250	100	1231 ± 29
Nakhla #2	Plateau	625–750	30	1323 ± 11
	High-T	450–1200	90	1304 ± 38
	All	250–1200	100	1252 ± 36

[a]The ages are calculated as the apparent age of the sum of all gas released in the steps listed. "Plateau" is the best plateau, "High-T" is the sum of all gas excluding the lowest temperatures steps with apparent ages <1000 Ma, and "All" includes all gas except the very highest temperature blank-dominated extractions. For extractions where the fully corrected ^{40}Ar/^{39}Ar ratio is less than 0, it has been set to zero, but the 1σ uncertainty has been included in calculations of uncertainties.

In addition, there is a falloff of apparent age at the highest temperatures, the signature of recoil, for all samples. However, this is not coupled with high apparent ages at low temperatures, unless it is the gradual trend of decreasing age with increasing temperature seen at intermediate temperatures in all three samples.

We are most interested, though, in the lowest temperature extractions, where we would expect to see evidence of the alteration products. The first 5–15% of all three samples (our Lafayette sample is shown with expanded scale in Fig. 3) clearly show evidence for ages much lower than 1300 Ma, most of them lower than 900 Ma. In addition, the K/Ca plots show these are associated with a different mineral than the plateau, particularly in Lafayette. However, there is no evidence for a low temperature plateau in any of the samples with the possible exception of three steps at ~1180 Ma in Nakhla #2.

An alternative interpretation comes from an analysis of a three-isotope plot such as Fig. 4. In this plot, for which the data have been corrected for all interferences and spallation but not for any atmospheric contamination, the low temperature data for each of the three samples are roughly collinear and come close to being mixtures of terrestrial atmosphere (on the y-axis) and a 1300 Ma-old component (on the x-axis), requiring no young apparent ages at all. This is plausible if either iddingsite formation occurred very shortly after formation or the samples analyzed happened to contain no iddingsite.

In detail, however, this interpretation is less than satisfying. First, the data are not truly collinear. In no case is it possible to fit a line to any subset of the low temperature data with $\sqrt{[\chi^2/(N-2)]} < 2.5$ (the value is very close to 1 for a good least-squares fit). Second, the implied trapped ^{40}Ar/^{36}Ar (the inverse of the y-intercept) is 272 ± 2 and 248 ± 3 for the best fits for the two Nakhla samples, many σ lower than the 295.5 value of the terrestrial atmosphere. While the trapped ^{40}Ar/^{36}Ar for Lafayette is closer to atmosphere, 287 ± 1, the x-intercept implies an age of only 762 ± 78 Ma, significantly lower than 1300 Ma (the two Nakhla samples give implied ages of 1323 ± 12 Ma and 1270 ± 16 Ma). So, while we cannot rule out this interpretation in detail, we think it more likely that there is a significant amount of terrestrial contamination at the low temperatures combined with apparent ages that increase steadily from near zero, a situation that would also give the observed results. In agreement with this, in each of the three samples, the lowest temperature (250 °C) step contains a significant amount of ^{39}Ar (>3% of the total) and has ^{40}Ar/^{36}Ar between 293 ± 3 and 299 ± 3, consistent to within 1σ with atmospheric contamination and a zero age.

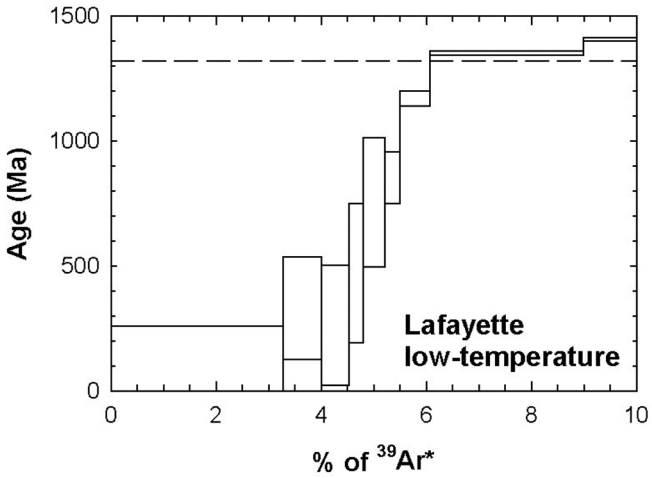

Fig. 3. Plot of apparent ^{40}Ar-^{39}Ar ages for low temperature extractions from Lafayette. There are apparent ages much less than 1300 Ma (reference line), but there is clearly no plateau.

DISCUSSION

The plateau age of Lafayette, 1322 ± 10 Ma, is in excellent agreement with the Ar-Ar plateau age (Podosek 1973) of 1330 ± 30 Ma and the preferred age, based on all radiometric studies, of 1320 ± 20 Ma (Nyquist et al. 2001). The Nakhla samples give plateaus within 1σ of each other and of our Lafayette sample, although they are somewhat higher

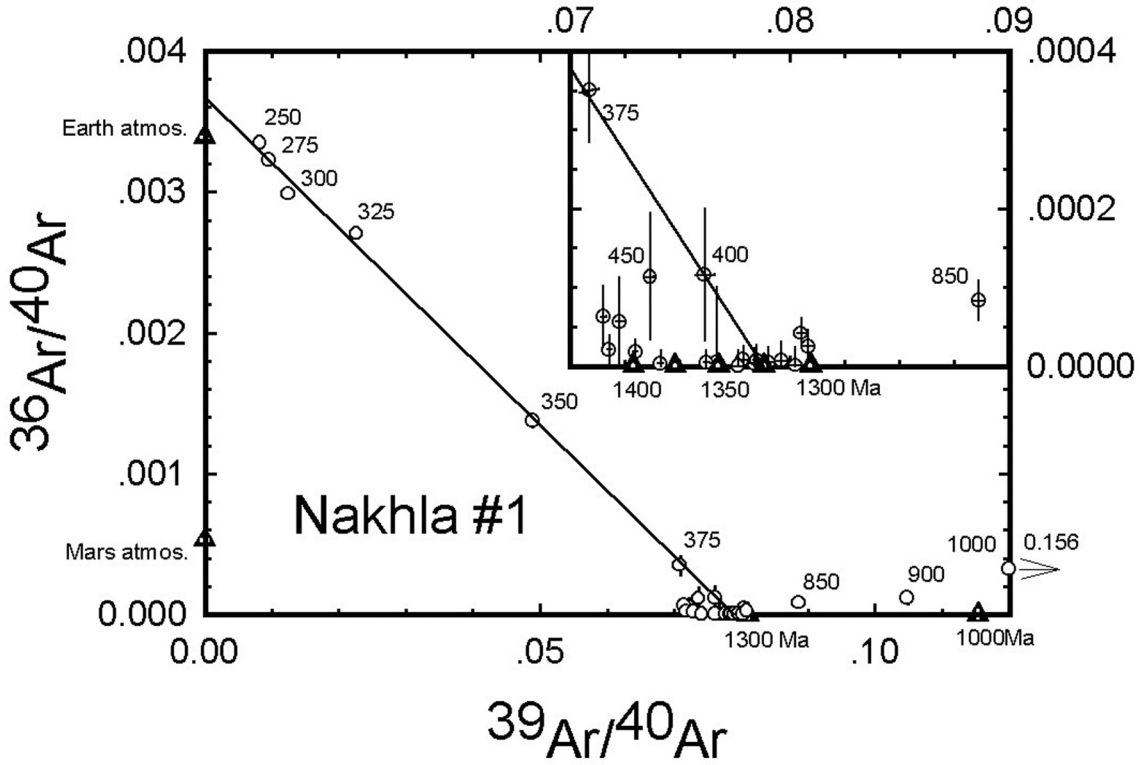

Fig. 4. Three-isotope plot of ^{36}Ar/^{40}Ar versus ^{39}Ar/^{40}Ar for Nakhla #1 (the other two samples give very similar results, although the low temperature data are less collinear). Plausible tie points are given by solid triangles at the compositions of the terrestrial and martian atmospheres (along the y-axis) and at ages of 1000 Ma and 1300 Ma (along the x-axis) for the main plot, and at 25 Ma intervals for the inset. Error bars not shown are smaller than the symbols. The numbers next to symbols are extraction temperatures in °C. The line on the main plot is a least-squares fit to low temperature data (250–400 °C), while that on the inset is a line from the terrestrial atmospheric composition (the correction used for generating Figs. 2 and 3). Data above 1000 °C, which have large error bars, are not plotted. Although terrestrial contamination of a 1300 Ma-old sample appears to be plausible, detailed considerations suggest that terrestrial contamination of a sample that has increasing age with increasing temperature is more likely (see text).

than the Nyquist et al. (2001) preferred age of 1270 ± 10 Ma. The Nakhla samples of Gilmour et al. (1998) and Burgess et al. (2000) give comparable Ar-Ar ages. The earlier Ar-Ar study (Podosek 1973) yielded ages of ~1300 Ma, but the apparent age spectrum was difficult to interpret in detail.

Although the spectra are less complicated than in the study of Podosek (1973), the Nakhla "plateaus" in our study are still somewhat troubling. We have chosen relatively flat portions of the apparent age spectra for Table 2, but in fact, the apparent ages steadily rise to >1400 Ma at 525 °C then steadily decrease in each sample.

One possibility is that we have performed an inappropriate correction for trapped Ar, since we merely assumed terrestrial composition. The nakhlites, especially Nakhla, did incorporate some martian Kr and Xe by some unknown mechanism (e.g., Bart et al. 2001; Gilmour et al. 1999, 2000; Mathew and Marti 2002; Ott 1988; Ott and Begemann 1985; Swindle et al. 2000). However, there is little ^{36}Ar (other than that from spallation) in the extractions making up the apparent plateaus, and if only the intermediate temperature data are considered on a three-isotope plot (Fig. 4), the data are certainly not collinear. They scatter along the x-axis, so there is no trapped ^{40}Ar/^{36}Ar ratio that would make the plateau significantly flatter. In particular, making a correction assuming trapped martian atmospheric Ar (^{40}Ar/^{36}Ar ~1900; Garrison and Bogard 1998) would overcorrect at the lowest temperatures and would not make any difference at many of the intermediate temperatures, although it would produce an apparent age spectrum even less flat than the one shown.

Another possibility is recoil, which was the preferred interpretation of Bogard and Garrison (2003) for patterns of steadily decreasing age with increasing extraction temperature in eucrites (e.g., their Figs. 1a and 4c). We definitely believe we are seeing the effects of recoil (McDougall and Harrison 1999; Turner and Cadogan 1974), which generally are manifested as high apparent ages at low extraction temperatures and low apparent ages at high extraction temperatures. The high temperature extractions do show a decrease in apparent ages, and the Lafayette sample shows three extractions just before what we have defined as the plateau with distinctly elevated apparent ages. There is enough "extra" ^{39}Ar in the last steps of the Nakhla samples that we could produce a flat plateau by arbitrarily

redistributing it throughout all the extractions. As an exercise, we have calculated the apparent ages we would obtain by summing all of the extractions except the lowest temperature extractions, and we find that the results ("High-T" in Table 2) are within 1σ of our chosen plateau in two of the three cases. The uncertainties are large, however, because the highest temperature steps have large uncertainties because of the correction for Ca-derived ^{39}Ar.

We know that there is recoil from the alteration products (Swindle et al. 2000), which include fine-grained clay. An obvious recoil recipient would be the high-Ca pyroxene, which has an extremely low K/Ca ratio, so that recoil could contribute a significant fraction of the ^{39}Ar. Since the alteration products are associated with olivine, which is also low-K and refractory, olivine is also a candidate for the recoil recipient. If the recoil does come from phases degassing at the lowest temperatures, that means that the true ages in the low temperature steps are even lower than they appear to be, and the steadily increasing ages up to 525 °C could be fortuitous.

Earlier, we mentioned one other possibility for the decreasing apparent ages at high temperatures, although we now think it unlikely. That possibility is the presence of younger Cl-rich phases that degas at higher temperatures (Burgess et al. 2000; Gilmour et al. 1998). As seen in Fig. 2, there is Cl-derived ^{38}Ar released at a higher temperature. However, the nakhlites contain chlorapatite (Berkley et al. 1980; Bunch and Reid 1975), which is a more likely source of Cl-derived ^{38}Ar at higher temperatures and is not related to the alteration.

There is still a mass-balance problem—the average K abundance measured in iddingsite by Swindle et al. (2000) means that iddingsite could account for 15–30% of the Lafayette whole rock K abundance measured by Podosek (1973). Our data, like Podosek's, suggest that <10% of the K is in low temperature, low age sites that we would expect for iddingsite. We suspect that the disagreement means that the iddingsite samples analyzed by Swindle et al. (2000), which were the largest that could be identified, were not typical in their elemental abundances. "Iddingsite" is not a single mineral and, in fact, consists of multiple, chemically distinct layers with K-rich material forming near the end of the sequence (Vicenzi and Eiler 1998; Vicenzi and Heaney 2000). It is also inhomogeneously distributed, as evidenced by the fact that some Nakhla whole rock samples show no disturbance of the Ar-Ar systematics at low ages (Burgess et al. 2000; Gilmour et al. 1998), but others (Podosek 1973; this study) do.

We stress that in the Ar-Ar data for all three of our whole rock samples, there is distinct evidence for a low temperature disturbance, with the evidence strongest at the lowest temperatures. We believe that iddingsite formation at some time much later than crystallization is a more likely cause for this disturbance than variable amounts of loss of radiogenic ^{40}Ar over time. Although much of the K in the iddingsite is probably in K-rich clays (Vicenzi and Heaney 1999), and some terrestrial clays are apparently leaky to radiogenic Ar (Dong et al. 1997b, and references therein), it has also been shown that, in terrestrial settings, some clays can retain radiogenic Ar for at least 400–700 Ma (e.g., Dong et al. 1997b; Hurley et al. 1961). Since the nakhlite environment on Mars was presumably much cooler than the terrestrial environments that have been studied (as well as less chemically active), this would enhance Ar retention on Mars. Hence, we see no reason a priori to think that clays would have to have been degassed during their time on Mars. Furthermore, even if Ar were lost with time under martian conditions, that would not explain the disturbances in the Rb-Sr system. The other obvious time when Ar loss and disturbance of the Rb-Sr system could have occurred was during the shock event that launched the rocks on their way to becoming meteorites, but the nakhlites are the least-shocked of the martian meteorites and apparently suffered no more than about a 100 °C peak temperature increase because of shock (Nyquist et al. 2001; Table 1). Hence, while we certainly cannot rule out diffusive loss of radiogenic Ar, the most severe thermal or chemical event these meteorites suffered appears to have been the infiltration with liquid water that led to iddingsite formation, the very event we are trying to date.

The other important point is that there is no evidence that iddingsite formation happened at a single time. Of course, absence of evidence for a single formation event is not the same as evidence of absence of a single event. In this case, the evidence for disturbances in other systems does not preclude the occurrence of a single event followed by loss of variable amounts of radiogenic ^{40}Ar from different samples. However, the petrographic evidence for multiple layers representing multiple events (Vicenzi and Heaney 2000), coupled with the evidence for the present-day presence of water (although probably frozen) near the surface of Mars in many locations (Mitrofanov et al. 2003; Tokar et al. 2003), means that multiple ages are a reasonable possibility. The best constraints probably still come from the K-Ar study of Swindle et al. (2000), who had some iddingsite chips with firm minimum ages of roughly 600 Ma. Hence, some iddingsite formed at least that early. A model-dependent upper limit could be derived from the Rb-Sr data.

CONCLUSIONS

Whole rock ^{40}Ar-^{39}Ar analyses of the nakhlites Lafayette and Nakhla are consistent with each other. More importantly, despite some apparent paradoxes in comparing results from K-Ar studies of physically separated samples of alteration products from Lafayette (Swindle et al. 2000) with earlier whole rock ^{40}Ar-^{39}Ar studies of the same meteorite (Podosek 1973), it appears that the K-Ar systematics of Lafayette and Nakhla are basically consistent from experiment to experiment and are also consistent with results from whole

rock studies of the nakhlites (Bogard and Husain 1977; Podosek 1973) and recent ^{40}Ar-^{39}Ar studies of mineral separates, etches, and whole rock chips from Nakhla (Burgess et al. 2000; Gilmour et al. 1998).

The most obvious feature is that all nakhlites have plateaus in their apparent age spectra at 1320–1335 Ma with no evidence for distinct crystallization ages. On closer inspection, the plateaus for Nakhla and Lafayette in this study are not quite flat, sloping downward from ~1400 Ma to ~1300 Ma (the details vary from sample to sample). This could reflect either recoil or some uncorrected non-radiogenic ^{40}Ar at low temperatures. Although neither of those explanations works well in detail, we believe that recoil is more likely.

A second shared feature in many of the samples (including ours) is a drop-off in apparent age at high temperatures, presumably a result of recoil of ^{39}Ar from fine-grained high-K sites into low-K high temperature mineral sites. The high-K source of the recoiling ^{39}Ar is not obvious in the Ar-Ar data, since there are no anomalously high apparent ages at low temperatures, but it may be the sloping plateau.

For the purposes of this experiment, the most important feature is the low apparent ages at low temperatures. Although there may be some effects of recoil present, that would mean that the true ages are even younger than the apparent ages we measured. We believe that the young ages represent the formation ages of alteration products rather than partial loss of radiogenic Ar because there is no thermal event in the meteorites' histories that would seem likely to cause partial or complete degassing, and other chronometric systems also have disturbances indicative of post-formation loss. However, we cannot rule out loss of radiogenic Ar from grain surfaces. There are no low temperature plateaus, although the clear presence of terrestrial atmospheric contamination in all three samples (as indicated by three-isotope plots) makes the data difficult to interpret. Petrographic evidence suggests the formation of multiple generations of alteration products. Combining the present high (temperature) resolution whole rock study with recent results on mineral separates and separates of the alteration products (Burgess et al. 2000; Gilmour et al. 1998; Swindle et al. 2000), we believe that it will be difficult, if not impossible, to define a single time of alteration from the K-Ar system, even if such a single time exists.

Editorial Handling—Dr. Marc Caffee

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APPENDIX

Table A1. Lafayette (23.83 mg).[a]

Temp.	^{40}Ar	$^{40}Ar = 100$				Age
(°C)	(10^{-8} cm^3 STP)	^{36}Ar	^{37}Ar	^{38}Ar	^{39}Ar	(Ma)
250	505	0.3425 (33)	0.85 (6)	0.7216 (48)	0.3629 (15)	0 (259)
275	30	0.3309 (27)	3.03 (16)	2.229 (7)	1.373 (6)	331 (204)
300	23	0.3338 (26)	2.77 (18)	2.160 (9)	1.254 (7)	262 (239)
325	16	0.3298 (26)	1.81 (27)	1.409 (6)	0.9101 (23)	470 (279)
350	27	0.3230 (27)	1.62 (32)	1.184 (8)	0.8411 (32)	753 (258)
375	7	0.2880 (54)	4.9 (6)	2.537 (16)	2.320 (13)	852 (103)
400	4	0.091 (8)	14.6 (6)	5.73 (6)	7.25 (6)	1168 (30)
500	23	0.0273 (14)	6.40 (27)	2.388 (6)	7.221 (23)	1351 (7)
550	41	0.0166 (11)	5.23 (15)	1.3753 (37)	7.023 (10)	1405 (6)
600	99	0.0148 (7)	5.63 (13)	1.1581 (34)	7.318 (12)	1371 (5)
625	60	0.0112 (10)	6.28 (27)	0.9602 (48)	7.700 (8)	1334 (5)
650	82	0.0095 (5)	8.14 (13)	1.3643 (25)	7.833 (9)	1327 (5)
675	58	0.0108 (6)	9.36 (12)	1.838 (6)	7.862 (14)	1323 (5)
700	54	0.0134 (7)	10.9 (7)	2.605 (6)	7.882 (8)	1317 (5)
725	42	0.0149 (8)	13.3 (6)	3.529 (12)	7.892 (13)	1317 (6)

Table A1. Lafayette (23.83 mg).[a] *Continued.*

Temp. (°C)	^{40}Ar (10^{-8} cm^3 STP)	^{40}Ar = 100 ^{36}Ar	^{37}Ar	^{38}Ar	^{39}Ar	Age (Ma)
750	35	0.0208 (7)	16.11 (32)	4.813 (19)	7.859 (21)	1311 (6)
775	30	0.0200 (8)	20.2 (8)	6.302 (15)	7.880 (15)	1320 (6)
800	27	0.0273 (12)	25.85 (33)	8.541 (16)	7.859 (14)	1316 (6)
825	17	0.0286 (19)	32.9 (1.7)	9.921 (37)	7.976 (25)	1313 (8)
850	5	0.0479 (43)	33.4 (1.6)	8.64 (7)	7.93 (6)	1263 (17)
850 (R[b])	20	0.0583 (11)	52.1 (1.4)	12.960 (34)	8.126 (21)	1251 (8)
900	27	0.0782 (15)	79.8 (1.9)	17.762 (32)	8.263 (13)	1235 (9)
950	5	0.1219 (34)	129.4 (1.2)	18.92 (18)	8.40 (7)	1186 (18)
1050	7	0.2591 (41)	324.8 (4.2)	25.24 (23)	8.76 (8)	1111 (27)
1150	12	2.360 (14)	3580 (33)	16.74 (10)	10.25 (6)	582 (294)
1250	3	29.2 (6)	48906 (1165)	53.9 (1.2)	37.4 (8)	1052 (868)
1350	0.8	1.55 (9)	1115 (92)	2.17 (18)	1.14 (6)	–
1550	0.6	0.540 (32)	129 (13)	0.387 (38)	0.177 (31)	–

[a]Isotopic ratios corrected for blanks and decay (to date irradiation concluded) only. The apparent age was calculated as discussed in text, including corrections for interferences caused by Ca, K, and Cl in the reactor, Ca and K spallation, and terrestrial atmospheric contamination. Uncertainties in the absolute amount of Ar, based on calibrations, are 5% or less. The numbers in parentheses represent 1σ uncertainty in final digit(s) of isotopic ratios and apparent ages. When the fully corrected ^{40}Ar/^{39}Ar ratio is more than 1σ less than 0, the age is given as 0 without uncertainty. Extractions at temperatures above 1250 °C are only slightly above blank levels for most isotopes, so no apparent ages are calculated. The uncertainty in apparent ages does not include uncertainty in J factor, 9 Ma for a 1300–1400 Ma age, or uncertainties in K decay constants and isotopic abundances (Bogard and Garrison 2000; Min et al. 2000), which could lead to a comparable uncertainty.

[b]We suspected that the furnace might not have reached the target temperature on the original 850 °C step, so a re-extraction was performed.

Table A2. Nakhla #1 (21.06 mg).[a]

Temp. (°C)	^{40}Ar (10^{-8} cm^3 STP)	^{40}Ar = 100 ^{36}Ar	^{37}Ar	^{38}Ar	^{39}Ar	Age (Ma)
250	341	0.3385 (25)	2.07 (29)	4.391 (18)	0.8243 (41)	199 (170)
275	77	0.3265 (30)	1.32 (13)	4.845 (10)	0.9584 (18)	623 (244)
300	53	0.3042 (25)	2.09 (17)	7.258 (11)	1.2445 (37)	1069 (134)
325	19	0.2829 (18)	3.11 (46)	18.06 (7)	2.265 (8)	1018 (70)
350	14	0.1683 (32)	5.1 (6)	47.03 (26)	4.886 (24)	1283 (26)
375	13	0.0939 (32)	7.1 (1.0)	94.50 (53)	7.076 (44)	1322 (21)
400	16	0.0967 (22)	6.7 (6)	142.0 (6)	7.598 (42)	1325 (25)
425	22	0.1007 (15)	6.3 (6)	170.0 (6)	7.651 (25)	1350 (27)
450	33	0.0955 (11)	6.35 (27)	140.72 (31)	7.350 (15)	1357 (24)
475	39	0.0641 (13)	5.6 (6)	94.99 (22)	7.210 (16)	1392 (16)
500	60	0.0462 (9)	4.57 (34)	62.44 (9)	7.137 (11)	1400 (11)
525	87	0.0163 (6)	4.15 (25)	16.130 (26)	7.163 (8)	1409 (5)
550	110	0.0081 (4)	4.40 (19)	1.3223 (39)	7.283 (7)	1394 (5)
562	91	0.0070 (6)	5.6 (6)	0.6529 (28)	7.397 (10)	1383 (5)
575	73	0.0075 (5)	6.16 (30)	0.5465 (18)	7.604 (11)	1356 (5)
587	61	0.0068 (7)	7.07 (28)	0.5446 (41)	7.772 (16)	1339 (5)
600	45	0.0073 (10)	7.9 (6)	0.5699 (44)	7.749 (17)	1341 (6)
612	42	0.0065 (11)	9.88 (42)	0.6643 (37)	7.826 (21)	1338 (6)
625	39	0.0098 (10)	9.26 (42)	0.752 (6)	7.835 (17)	1327 (6)
650	45	0.0167 (9)	14.67 (27)	0.991 (5)	8.038 (18)	1294 (6)
675	39	0.0135 (10)	15.40 (27)	1.280 (6)	7.891 (19)	1321 (6)
700	33	0.0173 (15)	21.0 (1.3)	1.745 (8)	7.953 (20)	1314 (7)
750	34	0.0075 (14)	21.0 (1.3)	2.853 (20)	8.015 (24)	1335 (7)
800	41	0.0279 (11)	31.6 (5)	6.077 (13)	8.081 (20)	1295 (6)
850	32	0.0507 (11)	54.03 (16)	12.100 (34)	8.865 (25)	1196 (7)
900	28	0.0799 (17)	88.9 (2.2)	20.43 (5)	10.497 (34)	1048 (10)
1000	47	0.2473 (27)	311.2 (1.0)	42.13 (8)	15.653 (25)	727 (15)
1100	19	3.031 (15)	4345 (29)	26.16 (12)	12.092 (45)	0
1250	7	35.97 (37)	53037 (610)	54.9 (6)	42.14 (44)	0
1350	0.2	0.72 (28)	272 (157)	1.5 (1.2)	0.81 (40)	–
1550	0.8	0.400 (34)	10 (15)	0.206 (23)	0.134 (37)	–

[a]See footnote (a) of Table A1.

Table A3. Nakhla #2 (25.95 mg).[a]

Temp.	^{40}Ar	^{40}Ar = 100				Age
(°C)	(10^{-8} cm^3 STP)	^{36}Ar	^{37}Ar	^{38}Ar	^{39}Ar	(Ma)
250	194	0.3504 (30)	6.7 (7)	8.970 (39)	1.5160 (41)	0 (73)
275	20	0.2886 (24)	5.9 (9)	18.59 (6)	3.107 (11)	761 (61)
300	18	0.2379 (25)	6.2 (7)	22.89 (7)	3.445 (12)	1124 (39)
325	25	0.2779 (21)	4.28 (7)	24.81 (7)	2.699 (8)	984 (59)
350	15	0.2006 (49)	8.0 (6)	70.42 (26)	4.999 (18)	1181 (35)
375	9	0.1586 (23)	10.1 (1.1)	189.1 (1.2)	8.03 (5)	1177 (33)
400	13	0.1919 (32)	9.2 (8)	262.9 (1.2)	8.098 (36)	1189 (44)
425	19	0.1871 (31)	9.2 (6)	299.7 (9)	7.827 (25)	1292 (48)
450	29	0.1358 (14)	7.04 (49)	211.60 (47)	7.492 (20)	1336 (34)
475	42	0.0627 (16)	6.33 (40)	80.85 (13)	7.148 (11)	1383 (15)
500	62	0.0235 (9)	5.31 (33)	21.365 (30)	7.059 (14)	1412 (6)
525	82	0.01037 (41)	4.88 (10)	3.390 (7)	7.034 (9)	1425 (5)
537	50	0.0094 (6)	5.8 (7)	0.7811 (49)	7.206 (13)	1402 (5)
551	54	0.0099 (10)	5.50 (41)	0.6602 (20)	7.193 (12)	1401 (5)
562	36	0.0098 (14)	6.21 (46)	0.5400 (22)	7.368 (14)	1379 (6)
575	41	0.0096 (10)	6.25 (29)	0.5629 (19)	7.442 (12)	1371 (5)
600	60	0.0092 (8)	7.3 (5)	0.6509 (39)	7.557 (12)	1359 (5)
625	60	0.0138 (7)	10.07 (47)	0.7474 (34)	7.694 (12)	1334 (5)
650	57	0.0124 (7)	11.85 (27)	0.923 (6)	7.746 (11)	1335 (5)
675	59	0.0207 (8)	15.94 (22)	1.3658 (31)	7.698 (8)	1325 (5)
700	44	0.0163 (9)	16.6 (6)	2.243 (9)	7.830 (10)	1324 (6)
725	35	0.0230 (12)	21.9 (2.0)	4.072 (8)	7.859 (16)	1314 (7)
750	26	0.0355 (21)	32.57 (40)	7.595 (23)	7.852 (25)	1304 (8)
800	272	0.0671 (14)	62.4 (3.2)	16.085 (38)	8.035 (17)	1259 (10)
900	41	0.1776 (7)	185.8 (2.4)	26.43 (5)	8.672 (13)	1110 (13)
1000	20	0.7622 (44)	1047 (28)	45.06 (14)	10.887 (32)	801 (76)
1100	9	5.518 (39)	7845 (50)	25.10 (17)	15.54 (10)	0
1200	7	36.70 (31)	54371 (452)	54.23 (46)	41.92 (35)	0
1300	0.2	0.48 (14)	1840 (680)	2.9 (1.1)	1.38 (33)	–
1550	0.5	0.24 (9)	77 (17)	0.252 (31)	0.16 (7)	–

[a]See footnote (a) of Table A1. Also, note that 1300 °C and 1550 °C extractions were only slightly above blank level for most isotopes, so an apparent age is not calculated.