# <sup>39</sup>Ar-<sup>40</sup>Ar Dating of Basalts and Rock Breccias From Apollo 17 and the Malvern Achondrite

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The principles and the potential of the <sup>30</sup>Ar-<sup>40</sup>Ar dating technique are illustrated by means of results obtained for 12 Apollo 17 rocks. Emphasis is given to methodical problems and the geological interpretation of lunar rock ages. Often it is ambigious to associate a given lunar breccia with a certain formation, or a formation with a basin. In addition, large-scale events on the Moon have not necessarily reset radiometric clocks completely. One rock fragment has a well-defined plateau age of 4.28 b.y., but the ages of two Apollo 17 breccias define an upper limit for the formation age of the Serenitatis basin at 4.05 b.y. Ages derived from five mare basalts indicate cessation of mare volcanism at Taurus-Littrow  $\sim$  3.78 b.y. ago. Ca-<sup>37</sup>Ar exposure ages show that Camelot Crater was formed by an impact  $\sim$  95 m.y. ago.

After a short summary of the lunar timetable as it stands at the end of the Apollo program, we report about <sup>®</sup>Ar-<sup>40</sup>Ar and rare gas studies on the Malvern meteorite. This achondrite resembles lunar highland breccias in texture as well as in rare-gas patterns. It was strongly annealed at some time between 3.4 and 3.8 b.y. ago. The results indicate that very similar processes have occurred on the Moon and on achondritic parent bodies at comparable times, leading to impact breccias with strikingly similar features, including the retention of rare-gas isotopes from various sources.

A major goal in exploring the history of the solar system is to establish an absolute time scale for the evolution of planetary objects such as the Moon or meteorites and their parent bodies. This implies the application of various dating techniques to physically accessible materials. In principle, ages of ancient rocks can be determined by the Rb-Sr, U-Pb, or K-Ar methods. Difficulties arose in applying these methods to lunar samples. The major shortcomings of the three methods turned out to be very low Rb concentrations in many lunar rock types, the extreme volatility of lead under lunar conditions, and diffusion of Ar, respectively.

In the case of Rb-Sr dating, many rock types became accessible for dating due to an extreme refinement of the experimental techniques by the California Institute of Technology group (ref. 1); but this still does not apply to anorthosites (an important lunar rock type) or to very small rock fragments extracted in great variety from lunar soils ("coarse fines").

Fortunately, the pioneering work of G. Turner made it possible to account for Ardiffusion losses in K-Ar dating by introducing an improved version of the K-Ar method, the so-called <sup>39</sup>Ar-<sup>40</sup>Ar method (refs. 2 and 3).

At present, Rb-Sr and <sup>39</sup>Ar-<sup>40</sup>Ar dating are the two principal tools for dating lunar rocks. Each of the two methods has its strengths and weaknesses, but together they complement each other rather favorably.

In this paper some general features and methodical problems of <sup>39</sup>Ar-<sup>40</sup>Ar dating will be described first. They will be illustrated by means of results obtained in dating rocks from the Apollo 17 site. Then the problems involved in the geological interpretation of measured radiometric ages will be discussed. This leads to discussion of the selenochronological significance of the results obtained for Apollo 17 samples in particular and to a short glimpse at the timetable of the evolution of the lunar crust in general. Finally, similar studies concerning the Malvern achondrite will be reported, and relations which might exist between the early evolution of the Moon and of meteorite parent bodies will be discussed.

## <sup>39</sup>Ar-<sup>40</sup>Ar DATING

#### Principles

A basic feature of the <sup>39</sup>Ar-<sup>40</sup>Ar method is the extraction of the radiogenic <sup>40</sup>Ar contained in a sample in a stepwise fashion at progressively higher temperatures in order to resolve the <sup>40</sup>Ar released from various phases or lattice sites of different activation energies. In addition, one is aiming at a simultaneous determination of the K concentration in exactly the same sites that release their <sup>40</sup>Ar at a given temperature. This is achieved by converting a fixed proportion of <sup>39</sup>K into 269 y-lived <sup>39</sup>Ar by a fast neutron irradiation via the reaction  ${}^{39}$ K (n,p)  ${}^{39}$ Ar. Provided that the release of <sup>39</sup>Ar produced in a given type of lattice sites follows the release of radiogenic <sup>40</sup>Ar from the same sites, one can in principle determine the K-Ar age of each phase by a massspectrometric determination of <sup>40</sup>Ar/<sup>39</sup>Ar isotopic ratios. If the less retentive phases had suffered <sup>40</sup>Ar losses during geological times, the apparent ages obtained at lower degassing temperatures would be lower than ages obtained at temperatures sufficiently high to degas those phases that did not undergo natural diffusion losses. If a sample contains at least some highly retentive phases that quantitatively retained their radiogenic <sup>40</sup>Ar since the initial cooling of the rock, then the ages obtained above a certain release temperature must approach a constant (maximal) value that defines a "plateau" in a plot of age versus release temperature or <sup>40</sup>Ar/<sup>39</sup>Ar-ratio versus cumulative fraction of <sup>39</sup>Ar released. It is this plateau age not affected by natural <sup>40</sup>Ar losses

that might imply geochronological significance.

## Practical Conditions

Elaborate descriptions of experimental details may be found in the literature (e.g., refs. 3, 4, and 5). Here we will repeat some facts of more general interest.

The threshold of the <sup>39</sup>K(n,p)<sup>39</sup>Ar reaction is 0.218 MeV. Assuming a typical reator spectrum,  $10^{19}$  neutrons/cm<sup>2</sup> with energies above 0.1 MeV produce ~  $5 \cdot 10^{-4}$  cm<sup>3</sup>STP <sup>39</sup>Ar per gram of potassium. A total neutron dose largely in excess of  $\sim 5 \cdot 10^{19}$  fast neutrons/  $cm^2$  (> 0.1 MeV) should be avoided since the radiation damage caused by such a high fluence could lead to a complete breakdown of the crystal structure (metamictization). If we consider a lunar rock with an extremely low K content of, say, 10 ppm, we obtain ~ 5·10<sup>-10</sup> cm<sup>3</sup>STP <sup>39</sup>Ar in a 100-mg sample, or ~  $5 \cdot 10^{-11} \text{cm}^3 \text{STP}^{-39} \text{Ar}$  in a single temperature step in which 10 percent of the total <sup>39</sup>Ar is released. With an advanced rare gas mass spectrometer one can easily handle such Ar-quantities. This consideration illustrates once more the nearly universal applicability of the K-Ar method.

In an actual experiment, <sup>36</sup>Ar, <sup>37</sup>Ar, and <sup>38</sup>Ar are recorded, together with <sup>39</sup>Ar and <sup>40</sup>Ar, in order to gain additional information about cosmic-ray-produced Ar-isotopes and to infer appropriate corrections for side reactions leading to <sup>39</sup>Ar and <sup>40</sup>Ar, as well as for a possible <sup>40</sup>Ar-contribution entrapped in the sample at the lunar surface.

The principal target element for cosmicray-produced <sup>38</sup>Ar in lunar samples is calcium. Therefore, 35.1-day-lived <sup>37</sup>Ar produced in the reactor by <sup>40</sup>Ca  $(n,\alpha)^{37}$ Ar allows one to infer cosmic ray exposure ages from <sup>38</sup>Ar/<sup>37</sup>Ar ratios in a fashion very similar to the determination of <sup>39</sup>Ar-<sup>40</sup>Ar ages. In addition, the <sup>37</sup>Ar/<sup>39</sup>Ar ratios obtained in the stepwise release pattern yield the apparent Ca/K ratios of the mineral phases responsible for the gas release at a given temperature. Since the Ca/K ratio is indicative of the mineral composition, this information can be very helpful in the interpretation of complex  ${}^{39}\text{Ar}/{}^{40}\text{Ar}$  release patterns (ref. 4).

The most serious side reaction is the production of <sup>39</sup>Ar from n-reactions with <sup>42</sup>Ca and <sup>43</sup>Ca.

Typical Ca/K ratios for common lunar rock types range between ~ 100 and ~ 1000. This corresponds to Ca-derived <sup>39</sup>Ar contributions of about 5 to 35 percent of the total <sup>39</sup>Ar. A correction can be made based on the <sup>37</sup>Ar/<sup>39</sup>Ar ratio produced from Ca. Since this ratio depends on the energy spectrum of the neutrons under the actual irradiation conditions, K-free Ca targets like CaF<sub>2</sub> must be irradiated together with the samples.

Other side reactions of neutrons with various K and Ca isotopes are less severe, but must nevertheless be taken into account (ref. 6). Further complications caused by thermal neutron capture reactions can be prevented by appropriate Cd shielding. Atmospheric Ar adsorbed at the samples is usually released by predegassing at ~  $300^{\circ}$ C, but it may occasionally account for high  $^{40}$ Ar/ $^{36}$ Ar ratios in the very first temperature fraction.

Surface exposed samples rich in implanted <sup>36</sup>Ar, <sup>38</sup>Ar, and <sup>40</sup>Ar from the solar wind or the lunar atmosphere should be avoided whenever possible. Small residues of trapped <sup>40</sup>Ar can be recognized from their <sup>36</sup>Ar and <sup>38</sup>Ar complements. A regular isochron plot of <sup>40</sup>Ar/<sup>36</sup>Ar versus <sup>39</sup>Ar/<sup>36</sup>Ar for the various temperature fractions may help to elucidate the proper <sup>40</sup>Ar/<sup>36</sup>Ar ratio of the trapped component (actual values range from ~ 0.5 to ~ 10; see fig. 1).

To convert the measured Ar isotope ratios into ages, a monitor is required for which age, K content, and Ca content are accurately known. Precambrian hornblende standards have proved to be most suitable for this purpose. Flux gradients along the ampoule can be recognized from the <sup>58</sup>Co activity induced by fast neutrons in various segments of an included Ni wire.

<sup>40</sup>Ar/<sup>39</sup>Ar ratios are transformed into ages according to

 $t_s = 4.34 \, {}^{10} \log \left( 1 + R_s \cdot J \right)$  where

$$J = [\exp(t_m/\tau) - 1]/R_m$$
  
= mean life of <sup>40</sup>K · 1 885 ×

 $\tau = \text{mean life of } {}^{40}\text{K}; 1,885 \times 10^9 \text{yr}$ 

$$R = {}^{40}\mathrm{Ar}_{\mathrm{radiogenic}}/{}^{39}\mathrm{Ar}_{\mathrm{corrected}}$$

Subscripts s and m refer to sample and monitor, respectively. The <sup>38</sup>Ar-Ca exposure age is given by

$$T = \left(rac{{}^{38}\mathrm{Ar}_{\scriptscriptstyle C}}{{}^{37}\mathrm{Ar}_{\scriptscriptstyle B}}
ight) rac{\Sigma \;\;{}^{37}\mathrm{Ar}_{\scriptscriptstyle m}}{\mathrm{Ca}_{\scriptscriptstyle m}} oldsymbol{\cdot} rac{1}{P} \,\mathrm{where}$$

 $\gtrsim$  <sup>37</sup>Ar<sub>m</sub> is the total <sup>37</sup>Ar released from the monitor at all temperature fractions, Ca<sub>m</sub> is the Ca content of the monitor in g/g sample, and P is the <sup>38</sup>Ar production rate per gram Ca and unit time.

 ${}^{38}\mathrm{Ar}_o$  is cosmogenic  ${}^{38}\mathrm{Ar}$  as deduced from a split of the total  ${}^{38}\mathrm{Ar}$  and  ${}^{36}\mathrm{Ar}$  contents and the isotopic compositions of cosmogenic and trapped Ar (see ref. 5).  ${}^{37}\mathrm{Ar}$  must be corrected for decay between irradiation and measurement.

The total K and Ca contents of a sample are obtained by

$$egin{array}{rcl} {
m K}_s & = {
m K}_m \ (\Sigma \ {}^{39}{
m Ar}_s/\Sigma \ {}^{39}{
m Ar}_m) \, {
m and} \ {
m Ca}_s & = {
m Ca}_m \ (\Sigma \ {}^{37}{
m Ar}_s/\Sigma \ {}^{37}{
m Ar}_m) \end{array}$$



Figure 1.—Ar release patterns of anorthositic breccia 77017,32,A and glassy vein 77017,32,B. Apparent K/Ca ratios are also shown. Small numerals indicate degassing temperature in 100°C. The insert illustrates the determination of the <sup>40</sup>Ar/<sup>36</sup>Ar ratio of nonradiogenic Ar included in the glass.

In the further discussion, the Apollo 17 highland rock 77017 will be used as an example. This rock is a white anorthositic breccia (subsample "A") veined by a black vuggy glass (subsample "B"). After separation of the two components, samples of 100 mg and 32.6 mg, respectively, were cleaned in ethanol, wrapped in Al foil, stacked alternatingly in quartz ampoules, and vacuum sealed together with three samples of a  $2.67 \pm 0.02$ -b.y.-old hornblende monitor, two  $CaF_2$  crystals, and an Ni wire. The Cdshielded ampoule was irradiated for 78 hours in the core of the BR-2 reactor at Mol, Belgium. The total neutron dose above 0.1-MeV energy was  $\sim 1.5 \times 10^{19}/\text{cm}^2$ . The flux gradient along the whole length of the ampoule was 3.6 percent. For the sample positions, J values were 0.0628 and 0.0631.

After degassing for 1 hour each at 600, 700, 800, 900, 1000, 1100, 1300, and 1500°C, the extracted Ar was analyzed in a high sensitivity mass spectrometer. The results are summarized in table 1.

### Ar Release Patterns, Exemplified by Apollo 17 Samples

Figure 1 shows a plot of the apparent ages at the various temperature fractions, together with the apparent K/Ca ratios of the corresponding release fractions. The age pattern for the anorthositic breccia 77017,32,A exhibits three typical features of general interest:

- 1. A relatively high apparent age in the very first temperature step. This can be due either to an atmospheric contamination or to Ar that was lost from less retentive sites, but partially preserved through uptake at grain boundaries.
- 2. A monotonous increase of ages with increasing temperature, reflecting gas loss in the less retentive mineral sites.
- 3. A rather constant plateau age at higher temperatures, indicating that the rock does in fact contain retentive domains that behaved as isotopically closed

microcells, since the rock was formed at the time indicated by the plateau age— $4.05 \pm 0.04$  b.y. ago in our example. Note that conventional K-Ar dating would have yielded an age of  $3.23 \pm 0.07$  b.y. only.

Another feature frequently observed in highland rocks but not displayed in our example is a decrease of apparent ages from the plateau value at very high temperatures. Turner et al. (ref. 7) noticed that pyroxenes are responsible for this effect. The physical reason is not clear at present. Subsolidus crystallization of pyroxenes from glass long after rock formation could be responsible (early Ar losses from low-retentive glass, high activation energy of pyroxene today) (ref. 5).

A further frequent deviation of age patterns from the ideal case is a sudden increase of age above the plateau value at very high temperatures close to the liquidus (refs. 8 and 9). There are two possible explanations:

- 1. Relicts of ancient rock clasts with sufficient retentivity to resist the thermal metamorphism that had reset the radiometric clocks for the other constituents are included within a breccia.
- <sup>39</sup>Ar deficit occurred due to the recoil displacement of <sup>39</sup>Ar by a few hundred Å in statu nascendi. Following intense preheating in the earlier degassings, a more than proportional share of <sup>39</sup>Ar may have been lost.

More generally, physico-chemical reactions during gas extraction may lead to mixing of phases that were previously earmarked by different <sup>39</sup>Ar/<sup>40</sup>Ar ratios. Also, perceptible quantities of <sup>39</sup>Ar may be directly lost during production in the irradiation from very fine-grained minerals ( $\leq 1 \mu$ m diameter). Apart from these experimental reasons, structured release patterns may be due either to contributions from various mineral phases with different ages and/or different solidstate properties or to different types of imperfections in the same type of mineral, either of primary nature or caused by such things as shock effects or radiation damage. The latter case is most likely when the K/Ca variation through the whole temperature range is as insignificant as for our example 77017,32,A (fig. 1). If, on the other hand, K-rich interstitials or feldspars exist in a breccia, then the K/Ca ratio may vary by orders of magnitude.

In our opinion, one should be rather reluctant in assigning geological significance to singularities in release patterns like "humps" and "dips" in various temperature ranges. Even if such a deviation is resolved into some closely spaced temperature fractions, one should keep in mind that an ideal crystal with just one type of gas-bearing lattice sites would release its gas over a range of temperatures rather than at one temperature. Obviously, methodical studies are required before too much emphasis is given to the interpretation of details. At present one should be satisfied if it is possible to infer a plateau age. This is by no means always the case. Quite often release patterns show a continuous increase with temperature up to completely unreasonable ages, and no plateau is defined at all. These samples contain either excess  ${}^{40}$ Ar (neither produced in situ nor accompanied by trapped  ${}^{36}$ Ar), or their retention characteristics are completely disturbed by some unidentified process that must be rather radical but still not sufficient to completely reset the radiometric clock for all constituents of the sample.

The glassy vein 77017,32,B must be the result of a melt injection into the breccia, possibly at the time of the final brecciation. Shock melting in situ can be excluded from the fact that the glass contains large quantities of <sup>36</sup>Ar that must be derived from the solar-wind-loaded regolith as source material of the glass. The breccia itself is essentially free of trapped Ar (table 1). Trapped Ar contains variable proportions of <sup>40</sup>Ar (refs. 10 and 11). For samples like our glass, this proportion must be known in order to distinguish the in-situ-produced radiogenic <sup>40</sup>Ar. It may be obtained from a plot of

 Table 1.—Analytical Results for Highland Breccia 77017,32

77017,32,A K 410 $\pm$ 30 ppm; Ca 9.6 $\pm$ 0.8%; Exposure age 80 $\pm$ 10 m.y. Total Ar age 3.23 $\pm$ 0.07 b.y.; Plateau age 4.05 $\pm$ 0.04 b.y.							77017,32,B (glassy vein) K 475 $\pm$ 30 ppm; Ca 10 $\pm$ 0.8%; Exposure age 90 $\pm$ 40 m.y.; Total Ar age 1.67 $\pm$ 0.3 b.y.; Plateau age 1.5 $\pm$ 0.2 b.y.				
${\stackrel{\rm Temperature}{\stackrel{{}^\circ}{\rm C}}}$	$^{40}\mathrm{Ar}_{R}$	$^{39}\mathrm{Ar}_{K}$	${}^{38}\mathrm{Ar}_{C}$	<sup>37</sup> Ar <sub>corr</sub> .	$^{36}\mathrm{Ar}_{T}$	$^{40}\mathrm{Ar}_{R}$	<sup>39</sup> Ar <sub>K</sub>	$^{38}\mathrm{Ar}_{C}$	<sup>37</sup> Ar <sub>corr</sub> .	$^{36}\mathrm{Ar}_{T}$	
600 700 800 900 1000 1100 1300 1500	$\begin{array}{r} 68.1 \\ 62.8 \\ 166 \\ 153 \\ 200 \\ 335 \\ 229 \\ 169 \end{array}$	$1.80 \\ 2.91 \\ 4.99 \\ 1.72 \\ 1.67 \\ 2.78 \\ 1.89 \\ 1.38$	0.65 1.39 2.81 1.03 1.06 1.85 2.17 1.11	$     193 \\     314 \\     545 \\     193 \\     194 \\     338 \\     375 \\     194 $	$\begin{array}{c} 0.20\\ 0.16\\ 0.30\\ 0.12\\ 0.14\\ 0.17\\ 0.23\\ 0.11\\ \end{array}$	$\begin{array}{r} 83.2 \\ 27.9 \\ 24.4 \\ 21.7 \\ 25.7 \\ 20.5 \\ 94.7 \\ 204 \end{array}$	$\begin{array}{c} 0.312\\ 0.586\\ 1.284\\ 0.914\\ 1.154\\ 1.350\\ 5.77\\ 10.8 \end{array}$	$\begin{array}{c} 0.08\\ 0.23\\ 0.65\\ 0.49\\ 0.57\\ 0.80\\ 4.76\\ 8.04 \end{array}$	15     44     112     70     84     120     653     1352	$1.33 \\ 1.50 \\ 3.57 \\ 5.15 \\ 6.14 \\ 8.85 \\ 142 \\ 177$	
Total	1385	19.2	12.1	2345	1.43	500	22.2	15.6	2450	346	

NOTES: Explanation of subscripts: R-radiogenic

K—potassium-derived

C—cosmogenic

corr.-corrected for decay

T—trapped

Due to rounding, figures may not add to totals.

 $^{40}\mathrm{Ar}/^{36}\mathrm{Ar}_{\mathrm{s.w.}}$  versus  $^{39}\mathrm{Ar}/^{36}\mathrm{Ar}_{\mathrm{s.w.}}$  (insert of fig. 1). In spite of large errors introduced by this correction, we can infer a plateau age of  $1.5\pm0.2$  b.y. for the glass. Obviously, the glass was injected into the breccia  $\sim2.5\,$  b.y. after the crystallization of the anorthositic breccia clasts. The extremely high "age" in the 600°C release of the glass sample could be caused by surficial inclusion of extraneous  $^{40}\mathrm{Ar}$  or atmospheric argon.

Apart from samples 77017 discussed above, we have dated 10 more Apollo 17 samples in an analogous manner. For our discussion, the data given in table 2 are sufficient. With reference to the foregoing discussion, a short characterization of the release type of each sample is also included. Further details are given in Kirsten et al. (ref. 12) and Kirsten and Horn (ref. 9).

## INTERPRETATION OF PLATEAU AGES

It is very encouraging to note that the agreement between <sup>39</sup>Ar-<sup>40</sup>Ar plateau ages and Rb-Sr-ages is very good in nearly every case for which comparisons have been made (see ref. 13 and Kirsten and Horn, unpublished). This applies also to samples for which the age plateau covers only an intermediate temperature range. Three conclusions can be drawn from the observed consistency.

- 1. In general, <sup>39</sup>Ar-<sup>40</sup>Ar-plateau ages are reliable also in cases for which Rb-Sr ages cannot be obtained (e.g., coarse fines).
- 2. The "geological" half life of <sup>87</sup>Rb is once more confirmed.
- 3. Rock-forming processes or metamorphism on the Moon reset K-Ar and Rb-Sr clocks at essentially the same time ( $\pm 20$  m.y.).

The latter point leads to the discussion of the geological interpretation of plateau ages. <sup>39</sup>Ar-<sup>40</sup>Ar ages are gas-retention ages. The time at which the temperature dropped below a critical level and the rock became isotopically closed is determined. Implied is the assumption that preexisting <sup>40</sup>Ar was expelled at that time and that the K-Ar clock was in fact set to zero.

On the Moon the critical thermal event could be

- 1. Crystallization of a cooling magma or of volcanic pyroclastics; metamorphism might occur at the contacts
- 2. Local impact melting or volcanic fire fountaining
- 3. Displacement of rocks passing different P-T levels (e.g., uplift)
- 4. Large-scale basin-forming impacts with all grades of shock metamorphism

The first case most likely applies to the mare basalts, but younger lava dikes or sills penetrating older ones may affect their ages. The second case is responsible for ages of particular glass samples or glass splashes and veins such as our sample 77017,32,B.

Most highland rocks are rather complex multistage breccias. They are believed to be ejecta of the large basin-forming events that occurred during the childhood of the Moon. Further brecciation was caused by more local impacts. The question then is whether the ages of highland rocks can tell us the sequence of the major basin-forming events and of the respective lunar formations. Here are two crucial problems:

- 1. It is by no means clear to what extent the large-scale events have reset the radiometric clocks.
- 2. It is quite ambiguous to associate a given lunar breccia with a certain formation and/or event.

These two points will now be discussed in some detail.

Some authors have concluded, mainly from the predominance of a certain age for a suite of samples tentatively assigned to the same formation, that the basin-forming event has equilibrated the radioisotopes by (1) extensive thermal metamorphism associated with the event (refs. 14, 15, and 16) and (2) uplift from depths where the temperature prevents retention of Ar (refs. 15 and 17).

Sample Number	Rock Type	Sampling Site and Probable Origin	Exposure Age (4) (m.y.)	Total Ar-Age (b.y.)	Plateau Age (b.y.)	Type of Release Pattern <sup>(6)</sup>
70215,21	Mare basalt fine grained	Near Lunar Module Camelot Crater <sup>(1)</sup>	$100 \pm 12$	$3.81 \pm 0.10$	$3.84\pm0.04$	a,b,B
79155,24	Mare basalt coarse grained, shocked	Rim of Van Serg Crater, Cochise, Shakespeare, Hanny Craters?	$575 \pm 60$	$3.73\pm0.07$	$3.80\pm0.04$	a,b,c,C
75055,11,2	Mare basalt	Camelot Crater	$85 \pm 10$	$3.62\pm0.07$	$3.82\pm0.05$	a,b,d,C
78503,13,B	Mare basalt medium grained	Base of Sculptured Hills; Camelot Crater <sup>(1)</sup>	$105 \pm 15$	$3.68\pm0.08$	$3.83\pm0.04$	a,b,C
74243,4,A	Mare basalt coarse grained	Shorty Crater (2)	$315~\pm~40$	$3.76\pm0.08$	$3.78\pm0.04$	a,b,d,C
74243,4,B	Mare basalt very fine grained	Shorty Crater (2)	$57 \pm 10$	$4.04\pm0.10$	No plateau	Steady increase to 5.9 b.y. (excess Ar): C
74243,4,C	Poikilitic mare basalt, medium grained	Shorty Crater (2)	58 ± 8	$3.82\pm0.08$	(3.93) (5)	b,c,d,C
77017,32,A	Anorthositic breccia, poikilitic clasts	Base of North Massif	$80 \pm 10$	$3.23 \pm 0.07$	$4.05 \pm 0.04$	a,b,c,A; see figure 1
77017,32,B	Black glass vein penetrat- ing A	Base of North Massif	$90 \pm 40$	$1.67\pm0.3$	$1.5 \pm 0.2$	See discussion and figure
76055,4,1	Polymict breccia, noritic matrix	Base of North Massif	$120 \pm 15$	$4.05\pm0.08$	$4.05\pm0.07$	a,A
70053,8	Anorthositic breccia, monomict	Base of North Massif ®	$160 \pm 20$	$4.06\pm0.08$	(4.3?)	
78503,13 A	Poikilitic gabbro	Base of Sculptured Hills	$290\pm35$	$4.26 \pm 0.08$	$4.28 \pm 0.04$	Steady increase from 3.5 to 4.3 b.y.;A

Table 2.—Summary of <sup>39</sup>Ar-<sup>40</sup>Ar Results for Apollo 17 Rocks

Notes: (1) Inferred from exposure age.

(2) Probably preirradiated since exposure age of Shorty Crater is  $\sim 30$  m.y.

- (3) Picking pot; eventually broken from 76335.
- (4) Based on  $({}^{38}\text{Ar}/{}^{38}\text{Ar})_T = 0.187$ ,  $({}^{38}\text{Ar}/{}^{36}\text{Ar})_C = 1.7$ ; production rate  $1.7 \pm 0.17 \times 10^{-8}$  ccSTP  ${}^{38}\text{Ar/g}$  Ca, m.y. Fe and Ti neglected.
- (5) Maximum value, only in one temperature fraction.

(6) Key: a—well-defined plateau; b—initial increase (gas loss); c—decrease at high temperatures; d—raised value at 1500°C; e—high value at 600°C; A—K/Ca ratio constant within factor of ~ 3; B—K/Ca ratio decreases monotonously by factor of ~ 20; C—K/Ca ratio decreases monotonously by factor of ~ 200.

However, it becomes increasingly clear that, in fact, a range of ages occurs also for rocks from within *one* formation or even among clasts of the same breccia (refs. 8 and 17). This unavoidably asks for the inheritance of some of the preexisting radiogenic species that were not equilibrated in the basin-forming event. Also, from the variety of metamorphic effects recorded in Apollo 14 breccias (ref. 18) it was concluded that these rocks cannot have been produced and/or metamorphosed by a single event, but that they also retain imprints of previous igneous, metamorphic, and collisional phenomena.

We have concluded that radiometric ages do not necessarily date the events responsible for the deposition of rocks, but that, nevertheless, the ages measured on rocks from a given formation can be used to infer an upper age limit for the formation itself since a selenologic formation cannot be older than the youngest rock found within this formation. The youngest rock age may date the respective event, possibly because the rocks were melted or heated at that time, but probably because they were excavated from a depth in which temperatures were high and isotopic systems were open (ref. 17).

All these discussions depend on the proper assignment of rocks to formations and of formations to basins. Such assignments are usually made on the basis of

(1) proximity of the basin closest to the sampling site; (2) stratigraphic relations (ejecta rays, etc.); and (3) petrographical similarities among various rocks.

An additional approach is a distinction based on meteoritic trace element admixtures related to the different basin-forming projectiles (ref. 19).

As the observational evidence increases, it seems that most authors (including those of this paper) may previously have been too optimistic in accepting proposed relations between rocks and formations, especially since mixing of ejecta from various formations, as well as locally derived components, is more important than had been thought before (see, for example, refs. 20 and 21). Schaeffer and

Husain (ref. 15) propose to use statistical predictions of the abundance of ejecta from different basins at a given site (ref. 22) for the respective assignments, but the used ejecta blanket thicknesses are inferred from a rather simplistic model. Without supporting evidence, it is not justified to ascribe different ages found within one sampling site to rocks from different formations. For example, the chemical classification of Morgan et al. (ref. 19) does not lead to a correlation between formations and the *face* values of radiometric ages. In conclusion, only a firm assignment of samples, together with the use of minimum ages of related rock suites, can lead to the establishment of a proper chronological sequence of the major lunar structures as tentatively attempted in the next sections.

## DISCUSSION OF APOLLO 17 RESULTS

The Apollo 17 module has landed in the Taurus-Littrow highlands in a topographical low filled by high Ti basalts and bordered by three highland units: North Massif, Sculptured Hills, and South Massif. We have dated seven mare basalts (three larger rocks: 70215, 79155, 75055; four independent small fragments: 78503,13B, 74243,4,A,B, and C) and four highland rocks (breccias 77017, 76055, small fragments 70053,8 and 78503, 13A) (table 2).

#### Mare Basalts

For five out of seven dated mare basalts, we obtain plateau ages within the very narrow range of  $(3.78 \pm 0.04) - (3.84 \pm 0.04)$  b.y. (table 2). This age is interpreted as the crystallization age of the upwelling magma and very probably dates the end of valley filling at Taurus-Littrow almost contemporaneously with the end of the filling of Mare Tranquillitatis (Apollo 11). Since the five dated basalts represent at least two different types, subsequent lava flows must have occurred within only 60 m.y. or so.

As can be seen from the total argon ages,

gas losses have been rather moderate. In the 1500°C-fraction, some of the samples display an increase in apparent age. This could be due to inherited ancient crustal material incorporated into the upwelling basaltic magma at this locality, analogous to comparable contaminations in many continental terrestrial basalts. For a more detailed discussion, see Kirsten and Horn (ref. 9).

### Highland Rocks

Breccias 77017,32,A and 76055 represent material from the North Massif. They contain rock clasts within a crushed and annealed matrix. Both breccias give well-defined plateau ages of 4.05 b.y., which is within the age range of other Apollo 17 highland samples recently reported by various authors at the Fifth Lunar Science Conference (3.9 to 4.3 b.y.).

Contrary to 76055, breccia 77017,32,A has suffered appreciable gas loss. This and the observed shock effects are probably related to the earlier mentioned glass injection caused by a local impact about 1.5 b.y. ago.

The age of 4.05 b.y. could eventually date the time of metamorphism of these rocks. However, before we continue in the interpretation let us first discuss a topic that was rather controversial until the Fifth Lunar Science Conference. It concerns the observation of <sup>39</sup>Ar-<sup>40</sup>Ar ages in excess of 4.1 b.y. in small rock fragments extracted from coarse fines (ref. 23). Skepticism was expressed because of the dominant opinion that basinforming impacts equilibrate isotopic systems and because no larger rocks of comparable age had been found. A cataclysm had been proposed to account for the observation that highland rocks from the various basins were isotopically equilibrated within a rather narrow time interval of  $3.95 \pm 0.1$  b.y. (refs. 24 and 25). Accordingly, all the major circular lunar basins should have been produced during this violent era by an episodic period of giant impacts. Consequently, older rocks should have had little chance to survive that catastrophic period without isotopic reequilibration. In the meantime, ages between 4.1 and 4.3 b.y. have been repeatedly found by various authors in Apollo 16 and 17 coarse fines (refs. 16, 17, 26, and 27) and also in larger rocks or clasts (refs. 8, 16, 27, and 28).

If the current view of the relation between formations and impact basins is adopted, all the accumulated age data would indeed suggest that the major lunar basins were formed within a time span of  $\sim 200$  m.y. centered at  $\sim 3.95$  b.y. ago (see next section). However, such a distribution can also be interpreted in terms of an exponential decrease of craterforming projectiles (above 200 m in diameter) with time. The rocks with ages in excess of 4.1 b.y. could have been stored in that violent early phase at a depth of a few kilometers, thereby escaping destruction through the high meteorite flux rate at early times. Their ages, as well as those of the old breccia clasts included in some highland breccias, are proof that isotopic equilibration is not an unavoidable consequence of the metamorphism occurring  $\sim 4$  b.y. ago, whatever its cause might have been.

Returning to the data given in table 2, coarse fine fragment 70053,8 is possibly, fragment 78503,13A certainly, another example for very old highland fragments. Anorthositic breccia 70053,8 shows a continuous increase in apparent age. It is doubtful whether the higher temperature fractions can be interpreted in terms of a plateau age near 4.3 b.y. On the other hand, poikilitic gabbro 78503,13A displays a very well-defined plateau age of 4.28  $\pm 0.04$  b.y. The plateau is paralleled by a very constant K/Ca ratio, indicating that essentially only one single mineral phase has contributed to the <sup>39</sup>Arrelease. The gas loss of this sample is as low as 2.7 percent.

Altogether, the evidence supports the idea that rocks have crystallized within the lunar crust as early as 4.3 b.y. ago and that some of them have survived ever since (ref. 26).

Before we draw some conclusions from the results of the investigated highland samples with respect to the origin of Taurus-Littrow, let us briefly discuss the measured cosmic ray exposure ages.

#### Cosmic Ray Exposure Ages

Due to the limited range of the cosmic radiation, exposure ages date the integral time period in which a particular rock was deposited in the upper meter or so of the lunar regolith. Frequent turnover may give rise to a complex exposure history, and only in favorable cases does it become possible to associate certain exposure ages with particular local craters. At a smaller scale, the problem of assignment is quite analogous to the case of gas retention ages, and only from the repeated occurrence of a certain exposure age within the range of a particular local crater can the age of the latter be concluded with some confidence.

The exposure ages of our Apollo 17 rocks range from 57 to 575 m.y. Samples 70215, 75055, and 78503,13B were collected near Camelot Crater and yield within the limits of error a consistent exposure age of  $95 \pm 15$ m.y. (table 2). The exposure ages of the other mare basalts cannot be related to specific cratering events. The exposure age of 575 m.y. for 79155 is among the highest so far measured for larger rocks. For small soil particles only, exposure ages occur up to at least 1.5 b.y. (ref. 5). The exposure ages of 74243, A,B, and C may reflect the typical complex exposure history of small regolith constituents. The similar exposure ages of  $\sim 58$  m.y. for subsamples B and C seems to be coincidental, since these fragments are taken from a mixed lunar soil and are not a priori interrelated. They do not date Shorty Crater (near their sampling site), since the latter is known to be  $\sim 30$  m.y. old (ref. 30). The exposure ages of highland rocks (table 2) have to be interpreted in terms of mass wasting at North Massif. No specific events can yet be recognized.

## Chronology of the Lunar Highlands

Provided that the association of the Apollo 17 highland breccias to the Serenitatis event is correct, we may conclude from the age of 77017 and 76055 that this event occured  $\leq 4.05$  b.y. ago. Crater counts and strati-

graphical observations indicate that Serenitatis is the oldest of all circular basins (ref. 31). In particular, it is older than the Imbrium basin with an age of  $\sim 3.9$  b.y. (ref. 17). Consequently, the Serenitatis basin was excavated between 3.9 and 4.05 b.y. ago, consistent with the observation that the stratigraphically younger basaltic lava flows at Taurus-Littrow solidified between 3.78 and 3.84 b.y. ago (ref. 12).

The youngest lunar basin dated is Orientale (3.82 to 3.85 b.y.) (ref. 17). Lunar areas inaccessible for sampling can be included in the established time sequence on the basis of crater counts and stratigraphical relations. Accordingly, at least 28 lunar basins must have been formed between 3.82 and 4.05 b.y. ago. The sequence of the most prominent of those basins is Orientale, Imbrium, Nectaris, Crisium, Serenitatis (ref. 31).

## LUNAR TIMETABLE

Since a review of the lunar time scale is given elsewhere in this conference (ref. 32), only a brief outline on this subject will be given in our context. It is summarized in figure 2.

At least a part of the lunar material must have condensed within 100 m.y. after chondrite formation  $\sim 4.6$  b.y. ago. This can be concluded from the observation that certain lunar breccias contain the recycled decay products of the now extinct radioisotopes  $J^{129}$ and Pu<sup>244</sup>, which have half lifes of 17 and 82 m.y., respectively (ref. 33). However, it remains possible that this decay occurred in solidified protolunar material that only later became incorporated into the Moon, provided that cold accretion occurred. Based on Rb-Sr and U-Pb studies, it has been found that the primary chemical separation of the primitive lunar crust must have occurred between 4.6 and 4.3 b.y. ago (refs. 32 and 34). This requires in the beginning a molten state at least for the outer 100 km or so.

As time went on between 4.3 and 4 b.y., the heat proceeded inward, and a solid lithosphere developed on top of the molten layers. It was impinged or penetrated by asteroid-



Figure 2.—Lunar Timetable based on radiometric age determinations. A = A pollo mission; L = LunaMission.

sized bodies that formed basins like Nubium or Fecunditatis. Due to the high impact rate at that time, rocks already solidified had little chance to survive. The oldest surviving rocks with ages between 4.3 and 4.1 b.y. were probably partially protected at some depth. The oldest interrelated rock suites have ages of  $\sim 4$  b.y. and are believed to be associated with the Serenitatis basin. Many basins were formed between 3.85 and 4.05 b.y. ago. Such ages are found for many rocks sampled from ejecta blankets of large basins, the most prominent one being the Imbrium basin that was excavated between 3.85 and 3.9 b.y. ago.<sup>1</sup> The youngest large circular basin is Orientale with an age between 3.82 and 3.85 b.y. Beginning about 3.85 b.y. ago, the face of the Moon was changed by the onset of a new type

of igneous activity, the extrusion of mare basalts, which were upwelling from depths of about 150 to 250 km. These basalts are the differentiation product of a Fe- and Mg-rich source magma at greater depths. The fluid basaltic liquids filled the various basins in subsequent floodings, where for each mare the uppermost lava flow is the latest one. The Tranquillitatis basin was flooded between  $\sim 3.8$  and  $\sim 3.5$  b.y. ago, Mare Imbrium between 3.35 and 3.15 b.y. ago, and the Oceanus Procellarum between  $\sim 3.3$  and  $\sim 3.1$  b.y. ago. Thus, the period of mare filling extended from  $\sim 3.8$  to 3.1 b.y.

No lunar igneous rock of younger age has been found. It seems clear that the largescale features of the Moon are today the same as 3.1 b.y. ago. Since that time, only occasional impacts like that producing the Crater Copernicus have altered the face of the Moon. However, on a smaller scale, exposure ages tell us that most rocks that are now found at the top of the surface of the Moon were excavated by smaller impacts during the last 1 b.y., and that regolith formation and mixing is an ongoing process, although at progressively lower intensity.

A proper time scale provides random conditions that limit the choice of possible models for the evolution of planetary objects that include condensation, accretion, differentiation, heat transfer, and metamorphism. For instance, the occurrence of lava flows as late as 1.5 b.y. after the formation of the Moon is incompatible with an initially cold Moon (ref. 36). Considerations of this type must be seen in the context of the planetary system as a whole. For this reason, meteorite chronology is presently experiencing a renaissance, as will be discussed in the following section.

### THE MALVERN ACHONDRITE

We became curious about Malvern because of its striking textural similarity with annealed polymict lunar highland breccias, e.g., those collected at the Fra Mauro site (ref. 37). The meteorite was found in South Africa in 1933. Based on chemical evidence, Malvern has been classified as a howardite,

<sup>&</sup>lt;sup>1</sup>This is not the place to discuss the entirely controversial view of Schonfeld and Meyer (ref. 35) who give arguments not easily disproved that Imbrium is in fact 4.3 to 4.4 b.y. old and that dated rocks result from more local events. However, this illustrates the difficulty in the geological assignments.



Figure 3.—Sections of the achondrite Malvern in plain transmitted light. Left: dark grey and black areas are glass and recrystallized glass, respectively; large fractured angular fragments are orthopyroxenes; plagioclase is clear white. Widths of picture equals 2 mm. The textural resemblance of this breccia is that of moderately annealed Apollo 14 breccias. Right: as above, with an intact rock clast.

but texturally it seems rather unique (Ramdohr, personal communication). It is highly brecciated (fig. 3) and contains various clasts. The dominant clast type (denoted A) is glassy to devitrified with small, but abundant, disseminated opaques and various amounts of trapped angular mineral fragments (fig. 3). Clast C is a coarse-grained aggregate of oithopyroxene, plagioclase, and clinopyroxene. As a whole, the meteorite resembles a moderately annealed Apollo 14 breccia. The glass occurs in the form of spherules and angular fragments up to 1 mm in diameter. The mineral fragments are shocked orthopyroxenes (hypersthene-bronzite), clinopyroxene, small olivine fragments, and larger plagioclases. The plagioclases appear to be recrystallized since they are clear with no signs of shock. We have separated the major constituents and performed <sup>39</sup>Ar-<sup>40</sup>Ar dating as well as rare gas analyses. The results are compiled in table 3; the age release patterns are shown in figure 4.

The apparent age of the glassy clast A1 shows an initial rise to 3.8 b.y. at 900°C. At higher temperatures it assumes values between 3.6 and 3.8 b.y. The ages of clast C vary in a similar fashion between 3.4 and

37 b.y. (the larger errors for sample C are mainly due to the low K content of 63 ppm). The fluctuations are not understood in detail. but it may be safe to state that Malvern was affected by a strong outgassing event between 3.4 and 3.8 b.y. ago. (In an earlier investigation, Podosek and Huneke (ref. 41) found nearly the same age range for the Stannern achondrite.) Most likely the minimum age of 3.6 b.y. for clast A1 corresponds to the age of the glass, whereas small admixtures of older material cause the scatter. This is substantiated by microscopical observation and the behavior of the K/Ca ratios for clasts A1 and C. The drastic decrease of this ratio above 1300°C in clast C suggests that the high ages for the 1300°C and 1400°C fractions are due to Ar release from distinct



Figure 4.—<sup>39</sup> $Ar^{49}Ar$  age release pattern for the Malvern achondrite. Sample A1 is enriched in glass splashes and spherules, sample C is a pyroxenerich clast. Apparent K/Ca ratios are also shown. Degassing temperatures are indicated. The age patterns reveal a strong outgassing event between 3.4 and 3.8 b.y. ago, very probably associated with the formation of the glass. Only the most retentive sites of the lithic clast escaped complete outgassing.

residuals of material inherited from the parent.

#### Rare Gas Analysis

The rare gas contents of glassy clasts and glass-free matrix chips of Malvern are given in table 3. Malvern is not a "gas-rich" meteorite. but the matrix does contain moderate amounts of heavily fractionated trapped gasses (table 3). This indicates heavy outgassing during intensive metamorphism. Trapped Ne and Ar included in the glassy

Table 3.—Analytical Results of the Malvern Achondrite

Notes: (1) <sup>39</sup>Ar-<sup>40</sup>Ar-analysis.

(2) Unit for rare-gas isotopes is  $10^{-8}$ ccSTP/g.

(3) Sample weight 10 mg only.

- (4) Corrected for reactor-produced isotopes.
- (5) Subscripts C and T refer to "cosmogenic" and "trapped," respectively. A three-isotope plot of all Ne data gave  $({}^{20}Ne/{}^{22}Ne)_T = 10.5 \pm 1$ ;  $({}^{22}Ne/{}^{21}Ne) \sim 32$ ; assumption:  ${}^{20}\text{Ne}_{C} = {}^{21}\text{Ne}_{C}.$

(6)  $\sim$ 1 percent of the pyroxenes of this particular sample contain heavily annealed solar flare tracks (Storzer and Pellas, private communication).

- (7) Based on  $({}^{36}\text{Ar}/{}^{38}\text{Ar})_{c} = 0.59$ ;  $({}^{36}\text{Ar}/{}^{38}\text{Ar})_{T} = 5.35$ .
- (8) Allowance is made for a possible contribution of  ${}^{3}\text{He}_{T}$ .

(9) <sup>4</sup>He<sub>radiogen</sub> not known, possibly 100 percent of total <sup>4</sup>He.

(10) T denotes cosmic ray exposure age.

Production rates are calculated from the chemical composition (ref. 37) and relations given by Herzog and Anders (ref. 38), Yaniv et al. (ref. 39), and Bogard et al. (ref. 40). We arrive at  $(2.7 \pm 0.3) \times 10^{-8}$ cc <sup>3</sup>He/g, m.y.;  $(0.465 \pm 0.050) \ 10^{-8}$ cc <sup>21</sup>Ne/g, m.y.;  $(0.12 \pm 0.012) \times 10^{-8} \text{cc} {}^{38}\text{Ar/g}$  meteorite, m.y.

(11) <sup>37</sup>Ar-<sup>38</sup>Ar exposure ages. Production rate derived from relations mentioned above is  $(2.4 \pm 0.24) \times 10^{-8} cc^{-38} Ar/g$  Ca, m.y. To account for Fe and Ti, it was multiplied by a factor 1.2. samples are probably due to the adherent matrix material.

Due to diffusional loss of spallogenic gases, the <sup>3</sup>He and <sup>21</sup>Ne exposure ages are much lower than the <sup>38</sup>Ar exposure ages (table 3). The <sup>38</sup>Ar/Ca exposure age of clast C is 14 m.y. for fractions released up to 1200°C and rises to  $\sim 20$  m.y. at the same high temperatures at which the <sup>39</sup>Ar-<sup>40</sup>Ar age rises above 4 b.y. This fact is suggestive of a preirradiation of the inherited parent material in clast C, but it could also be due to an exceptionally high Fe content of the mineral phase responsible for the gas release at very high temperatures.

#### Implications

There are two indications that Malvern must have contained solar-type rare gases sometime in its early history:

- 1. The high trapped  ${}^{36}\text{Ar}/{}^{84}\text{Kr}$  ratio of  $\sim 1000$  makes it unlikely that all the trapped  ${}^{36}\text{Ar}$  is dissolved gas of the planetary type.
- 2. About 1 percent of the pyroxenes from sample B2 contain heavily annealed solar flare-induced tracks (Storzer and Pellas, private communication).

Where then is the trapped He and Ne complementary to trapped <sup>36</sup>Ar? The relative abundance of Ne is depleted by more than a factor of 200 if compared to solar abundance ratios and it is still an order of magnitude lower than in lunar soil. The only explanation is that Malvern has once experienced extreme thermal stress and was heavily outgassed and annealed.

It needs little imagination to suppose that it was the same event that was responsible for the formation of the glass and for the resetting of the K-Ar clock in all but the most retentive constituents at some time between 3.4 and 3.8 b.y., as indicated by the  $^{39}$ Ar- $^{40}$ Ar data.

The occurrence of light trapped gases (ref. 42) and solar flare tracks (ref. 43) in meteorites indicates that the respective meteorites were derived from a parent body that was covered by a regolith (ref. 44), a rather common feature on top of planetary bodies (ref. 45).

The Moon as well as meteorite parent bodies were both bombarded with interplanetary debris, and giant impacts like those on the Moon must also have occurred on meteorite parent bodies. As on the Moon, such impacts had the potential to reset radiometric clocks to some extent. The age patterns of Malvern suggest that similar impact events took place on the Moon and meteoritic parent bodies and led to striking similarities in texture and retention characteristics of rare gases from various sources. For example, concentrations and fractionation patterns of trapped gases in lunar breccia 14303 (ref. 5) are nearly identical to those observed in Malvern (table 3). Evidence that collisional reheating of meteorite parent bodies is relatively frequent, not restricted to a certain time period and not restricted to a particular meteorite class, was first inferred from the distribution of total U-He and K-Ar ages (ref. 46). More recently, substantial details have been added to our knowledge about giant collisions in the solar system. Ages substantially below 4.6 b.y. have been found for an iron meteorite and two achondrites by Rb-Sr dating (refs. 47 and 48), four Carich achondrites (ref. 41), and four chondrites (ref. 49). The plateau ages reported by authors of the latter reference are all below 0.6 b.y. However, resistant phases with higher ages coexist quite frequently with the annealed matrix (e.g., Malvern C, and Pasamonte) (ref. 41). Consistent with the near but not total obliteration of early irradiation records, this indicates intensive, but not annihilating, metamorphism.

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