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Ion microprobe U-Th-Pb dating of phosphates in martian meteorite ALH 84001

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Abstract–Phosphates in martian meteorites are important carriers of trace elements, although, they are volumetrically minor minerals. PO_4 also has potential as a biomarker for life on Mars. Here, we report measurements of the U-Th-Pb systematics of phosphates in the martian meteorite ALH 84001 using the Sensitive High Resolution Ion MicroProbe (SHRIMP) installed at Hiroshima University, Japan. Eleven analyses of whitlockites and 1 analysis of apatite resulted in a total Pb/U isochron age of 4018 ± 81 Ma in the $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$-$^{204}Pb/^{206}Pb$ 3-D space, and a ^{232}Th-^{208}Pb age of 3971 ± 860 Ma. These ages are consistent within a 95% confidence limit. This result is in agreement with the previously published Ar-Ar shock age of 4.0 ± 0.1 Ga from maskelynite and other results of 3.8–4.3 Ga but are significantly different from the Sm-Nd age of 4.50 ± 0.13 Ga based on the whole rock and pyroxene. Taking into account recent studies on textural and chemical evidence of phosphate, our result suggests that the shock metamorphic event defines the phosphate formation age of 4018 ± 81 Ma, and that since then, ALH 84001 has not experienced a long duration thermal metamorphism, which would reset the U-Pb system in phosphates.

INTRODUCTION

ALH 84001, collected from the Far Western Icefield of Allan Hills in 1984, is classified as a martian meteorite based on petrography (Score and Mittlefehldt 1993), mineral chemistry (Berkley and Boynton 1992; Mittlefehldt 1994), and oxygen isotopic composition (Clayton 1993; Farquhar et al. 1998). This meteorite is well-known to be unique among the martian meteorites. First, it is composed of a coarse-grained orthopyroxene-rich component, which is present in the other martian meteorites only as xenoliths in the Elephant Moraine (EET) A79001 basalt (e.g., see McSween and Jarosewich 1983; Mittlefehldt 1994). Second, as explained below, this meteorite has been found to be the oldest (4.0–4.5 Ga) among the martian meteorites, but it has suffered several impact events during its history (Treiman 1995). Third, the most unusual feature of this meteorite is the presence of ellipsoids and patches of carbonate minerals (Mittlefehldt 1994). The origin of these carbonates was controversial (Mittlefehldt 1994; Romanek et al. 1994; Treiman 1995) and has been more so since McKay et al. (1996) discussed possible evidence of past biological activity on Mars, which was preserved within the outermost black rims of the carbonate globules from this meteorite. Formation of the carbonate globules at 0–300°C (Romanek et al. 1994; Valley et al. 1997) may be consistent with biologic activity and biogenic products, while a high-temperature formation at >650°C (Mittlefehldt 1994; Bradley et al. 1996) seems antagonistic to life. Recently, additional ion probe studies on oxygen isotopes have been reported showing a clear systematic correlation between $\delta^{18}O$ values and chemical composition, which is indicative of a systematic change in fluid temperature and/or isotopic composition (Leshin et al. 1998; Saxton et al. 1998). From these results, Leshin et al. (1998) interpreted carbonate formation to occur at no lower than 125°C but at as high as 500°C and, therefore, suggested a non-biological origin. Saxton et al. (1998) also concluded that ALH 84001 carbonates formed in a hydrothermal system, with T <400°C in the early stage and that the later stages of deposition probably occurred at temperatures below 150°C. While based on textural evidence and geochemical systematics, Eiler et al. (2002) suggest 2 populations of carbonates; the first population consists of flow-temperature aqueous precipitates, and the second population was produced by shock melting of the first. Thus, the origin of the carbonate still remains a matter for debate.

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Chronological studies of this unique meteorite, ALH 84001, have been performed by several different analytical approaches. Jagoutz et al. (1994) found that the Sm-Nd isotope systematics for mineral separates fit a 4.56 Ga isochron. Nyquist et al. (1995) also found that both the Rb-Sr and Sm-Nd isotope systems fit a 4.56 Ga isochron. On the other hand, both Ar-Ar and K-Ar ages show younger ages of 4.0 ± 0.1 Ga (Ash et al. 1996), 3.9–4.3 Ga (Bogard and Garrison 1999), 3.8–4.05 Ga (Turner et al. 1997), 3–4 Ga (Miura et al. 1995), and 3.86 Ga (Goswami et al. 1997). These Ar-Ar and Kr-Ar ages are quite similar, suggesting that ALH 84001 underwent an extensive equilibration event ~4 Ga, such as impact metamorphism (Turner et al. 1997; Bogard and Garrison 1999). Recently, Wadhwa and Lugmair (1996) reported that the Rb-Sr internal isochron based on whole rocks and orthopyroxenes gives an age of 3.84 ± 0.05 Ga. Borg et al. (1999) suggested that the age of secondary carbonate mineralization in the martian meteorite ALH 84001 was determined to be 3.90 ± 0.04 Ga by Rb-Sr dating and 4.04 ± 0.10 Ga by Pb-Pb dating, using leachates from a high-graded carbonate (visually estimated as ~5%), whitlockite (trace), and orthopyroxene (~95%). However, the U-Th-Pb systematics of ALH 84001 have not yet been well-documented.

For a better understanding of its thermal history, an in situ U-Th-Pb dating of phosphates in ALH 84001, which are carriers of trace elements and more resistant to secondary process, was performed using the Sensitive High Resolution Ion MicroProbe (SHRIMP II) installed at Hiroshima University, Japan. Our in situ analysis techniques attain high sensitivity (e.g., more than 10 cps/ppm/nA for ^{206}Pb on apatite) at high mass resolution, ~5800, providing the following advantages in comparison with the conventional TIMS analyses: 1) a much smaller amount of sample is required; 2) the mineralogy of the phosphates and textural relationships with other minerals can be investigated; 3) following the U-Pb analysis, other elements can be measured in the same grain; and 4) some information on the closure temperature of the U-Pb system based on the observed grain species and sizes can be obtained. This in situ U/Pb dating method has been applied successfully to some extraterrestrial phosphates in Shergotty (Sano et al. 2000), lunar meteorite EET 96008 (Anand et al. 2003), and some ordinary chondrites (Terada and Sano 2002, 2003). The primary purpose of this work is to directly observe the U-Th-Pb systematics of phosphates in ALH 84001.

SAMPLE AND ANALYICAL METHODS

The ALH 84001 sample used in this study was a polished thin section of #84001.88, provided by NASA-JSC. The polished thin section was carbon coated, and back scattered electron images were obtained, with the major chemical components being analyzed by an Electron Probe Micro Analyser (EPMA), to identify the location and mineralogy of the phosphates. Seven whitlockites and 1 intergrowth of apatite and whitlockite were observed in this polished thin section, the sizes of which range from 40 to 100 μm. All grains were surrounded by orthopyroxenes and/or maskelynite with cracks. Some of the grains had small inclusions or cracks. The interstitial apatite of ~300 μm reported by Wadhwa and Crozaz (1994, 1995) and the large grains of whitlockites up to 800 μm reported by Boctor et al. (1998a, b) were not found in this polished thin section. The major chemical compositions are listed in Table 1.

After EPMA analysis, the thin section was polished slightly using 0.25 μm diamond paste. Then, following cleaning to minimize surface contaminant Pb, it was gold-coated to prevent charging of the sample surface during SHRIMP analyses. The thin section was evacuated in the sample lock overnight to further reduce the already very small $^{x-1}$PbH$^+$ interference on the ^{x}Pb$^+$ peaks. Before the actual analysis, the sample surface was rastered for 3 min to remove possible contaminants. For SHRIMP analysis, only parts of the grains free from such inclusions or cracks were selected.

A ~1 nA O_2^- primary beam at an energy of 10 keV, was focused to sputter an area of ~10 μm in diameter on the phosphates, and positive secondary ions were extracted with an acceleration voltage of 10 kV. The mass resolution was ~5800 at 1% peak height of ^{208}Pb. The magnet was cyclically peak-stepped from mass 159 ($^{40}Ca_2{}^{31}P^{16}O_3^+$) to mass 254 ($^{238}U^{16}O^+$), including the background, all Pb isotopes, and masses 238 and 248 for $^{238}U^+$ and $^{232}Th^{16}O^+$. No significant isobaric interference was detected in this mass range for the phosphates. The ^{238}U/^{206}Pb ratios were obtained from the observed $^{238}U^+/^{206}Pb^+$ ratios using an empirical quadratic relationship between the $^{206}Pb^+/^{238}U^+$ and $^{238}U^{16}O^+/^{238}U^+$ ratios of the standard apatite, which were derived from an alkaline rock of the Prairie Lake circular complex in the Canadian Shield dated at 1156 Ma. Experimental details of the phosphate U-Pb analysis and the calibration of the data against the standard apatite are given elsewhere (Sano et al. 1999, 2000).

RESULTS

The observed U and Th concentrations and ^{238}U/^{206}Pb, ^{207}Pb/^{206}Pb, ^{204}Pb/^{206}Pb, ^{204}Pb/^{208}Pb, and ^{232}Th/^{208}Pb ratios of each analysis are listed in Table 2. The uncertainties for these concentrations are ~30%, estimated by repeated measurement of standard apatites. Average U and Th abundance are 3.9 ppm and 28 ppm, respectively. The average Th/U ratio of 7.2 for all phosphates is comparable to the Th/U ratio of 6.9 in Shergotty phosphate (Sano et al. 2000).

As shown in Table 2, a significant correlation exists between the observed ^{238}U/^{206}Pb ratios and the ^{204}Pb/^{206}Pb ratios and also between the observed ^{207}Pb/^{206}Pb ratios and

Table 1. Major element compositions of phosphates in ALH 84001.[a]

Grain nr	Mineral	CaO (%)	MgO (%)	FeO (%)	Na_2O (%)	P_2O_5 (%)	Total (%)
#1	Whitlockite	46.8	3.4	0.5	2.8	45.9	99.4
#2–1	Whitlockite	46.8	3.4	0.6	2.6	45.2	98.7
#2–2	Apatite	54.3	0.1	0.3	0.3	43.0	98.0
#3	Whitlockite	46.0	3.5	0.9	2.4	45.0	97.8
#4	Whitlockite	46.6	3.3	0.6	2.5	45.0	98.1
#5	Whitlockite	47.5	3.3	0.7	2.7	45.4	99.7
#6	Whitlockite	46.8	3.3	0.6	2.7	45.2	98.6
#7	Whitlockite	47.2	3.5	0.9	2.9	43.0	97.4
#8	Whitlockite	45.7	3.4	0.6	2.7	44.8	97.3

[a]Grain #2 shows the intergrowth structure of whitlockite and apatite.

Table 2. U and Th concentrations and $^{238}U/^{206}Pb$, $^{207}Pb/^{206}Pb$, $^{204}Pb/^{206}Pb$, $^{204}Pb/^{208}Pb$, and $^{232}Th/^{208}Pb$ ratios in phosphates in ALH 84001.

Spot nr	U	Th	$^{238}U/^{206}Pb$	$^{207}Pb/^{206}Pb$	$^{204}Pb/^{206}Pb$	$^{204}Pb/^{208}Pb$	$^{232}Th/^{208}Pb$
84001.02.1	1.9	24.4	0.892 ± 0.133	0.441 ± 0.018	0.0089 ± 0.0013	0.0028 ± 0.0004	4.149 ± 0.526
84001.02.2	1.6	21.0	0.800 ± 0.166	0.572 ± 0.022	0.0241 ± 0.0024	0.0082 ± 0.0008	4.365 ± 0.763
84001.02.3	17.9	24.1	1.355 ± 0.089	0.441 ± 0.009	0.0019 ± 0.0003	0.0042 ± 0.0006	4.662 ± 0.269
84001.03.1	1.4	30.2	1.313 ± 0.255	0.476 ± 0.023	0.0036 ± 0.0006	0.0006 ± 0.0001	5.444 ± 0.894
84001.04.1	2.2	18.2	1.156 ± 0.176	0.386 ± 0.019	0.0018 ± 0.0004	0.0007 ± 0.0002	4.522 ± 0.590
84001.04.2	2.5	22.4	1.262 ± 0.087	0.447 ± 0.008	0.0021 ± 0.0007	0.0056 ± 0.0009	3.509 ± 0.213
84001.05.1	2.4	26.9	1.713 ± 0.235	0.493 ± 0.016	0.0039 ± 0.0007	0.0011 ± 0.0002	6.494 ± 0.753
84001.06.1	0.8	9.7	0.713 ± 0.230	0.475 ± 0.040	0.0133 ± 0.0030	0.0037 ± 0.0008	2.833 ± 0.776
84001.06.2	1.3	12.8	1.423 ± 0.296	0.601 ± 0.034	0.0114 ± 0.0021	0.0033 ± 0.0006	5.000 ± 0.887
84001.07.1	6.7	61.2	0.720 ± 0.163	0.469 ± 0.011	0.0023 ± 0.0003	0.0010 ± 0.0002	3.474 ± 0.332
84001.08.1	5.5	57.6	1.005 ± 0.134	0.524 ± 0.017	0.0102 ± 0.0011	0.0041 ± 0.0004	5.014 ± 0.569
84001.08.2	1.0	19.2	1.017 ± 0.255	0.584 ± 0.031	0.0129 ± 0.0018	0.0029 ± 0.0004	5.100 ± 1.081

the $^{204}Pb/^{206}Pb$ ratios of phosphates in ALH 84001. Based on the least-square fit using the York method (York 1969), the former correlation gives a $^{238}U/^{206}Pb$ isochron age of 3704 ± 440 Ma (MSWD = 2.2; 95% confidence limit), and the latter gives a $^{207}Pb/^{206}Pb$ isochron age of 4022 ± 96 Ma (MSWD = 3.8; 95% confidence limit). The observed $^{207}Pb/^{206}Pb$ and $^{238}U/^{206}Pb$ isochron ages agree well with each other, within the uncertainties, suggesting that the U-Pb systems are concordant.

Next, we calculated a "total Pb/U isochron age" from the linear regression line in the 3-D space ($^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$-$^{204}Pb/^{206}Pb$). Cogenetic samples with an undistributed U-Pb system that share the same common Pb isotopic composition must define a line in $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$-$^{204}Pb/^{206}Pb$ space, the intersection of which with the $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$ plane must fall precisely on the U-Pb Concordia curve, and the intersection of which with $^{207}Pb/^{206}Pb$-$^{204}Pb/^{206}Pb$ is the isotopic composition of the common Pb. The crucial advantages of this calibration method are that it is not necessary to know the isotopic composition of the common Pb, and, in fact, it is part of the regression solution itself (for a detailed discussion, see Wendt 1989), and that all of the relevant isotope ratios are used at the same time, yielding the smaller justifiable age uncertainty for U-Pb systematics (see Ludwig 2001). Figure 1 shows the 3-dimensional linear regression for the total Pb/U isochron projected onto the $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$ plane. The calculation was made using Isoplot/Ex (Ludwig 2001). In Fig. 1, the solid curve is an evolution line of the U-Pb system without initial lead (Concordia line), the dashed line is the projected regression line onto the $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$ plane, and the total Pb/U isochron age is determined as an intersection with the Concordia line on the $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$ plane. Eleven analyses of whitlockites and 1 analysis of apatite indicate a total Pb/U isochron age of 4018 ± 81 Ma in the $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$-$^{204}Pb/^{206}Pb$ 3-D space (95% confidence limit; MSWD = 3.1). Figure 2 also shows a correlation between the $^{204}Pb/^{208}Pb$ ratios and the $^{232}Th/^{208}Pb$ ratios. A least-squares fit using the York method gives a $^{232}Th/^{208}Pb$ isochron age of 3971 ± 860 Ma (95% confidence limit; MSWD = 3.2). This $^{232}Th/^{208}Pb$ isochron age is also consistent with a total Pb/U isochron age, suggesting that the U-Pb systems and the Th-Pb systems are concordant. Taking into account the average of the $^{232}Th/^{208}Pb$ isochron age and a total Pb/U isochron age, the ion microprobe U-Th-Pb dating of ALH 84001 yields a formation age of 4018 ± 81 Ma for the phosphates.

Although the isotopic compositions of common lead of the phosphates ($^{206}Pb/^{204}Pb$ = 12 ± 17, $^{207}Pb/^{204}Pb$ = 12.8 ± 8.9, and $^{208}Pb/^{204}Pb$ = 28 ± 17 at a 95% confidence limit) can also be obtained from the intersection of the regression line with the $^{207}Pb/^{206}Pb$-$^{204}Pb/^{206}Pb$ plane in the 3-D

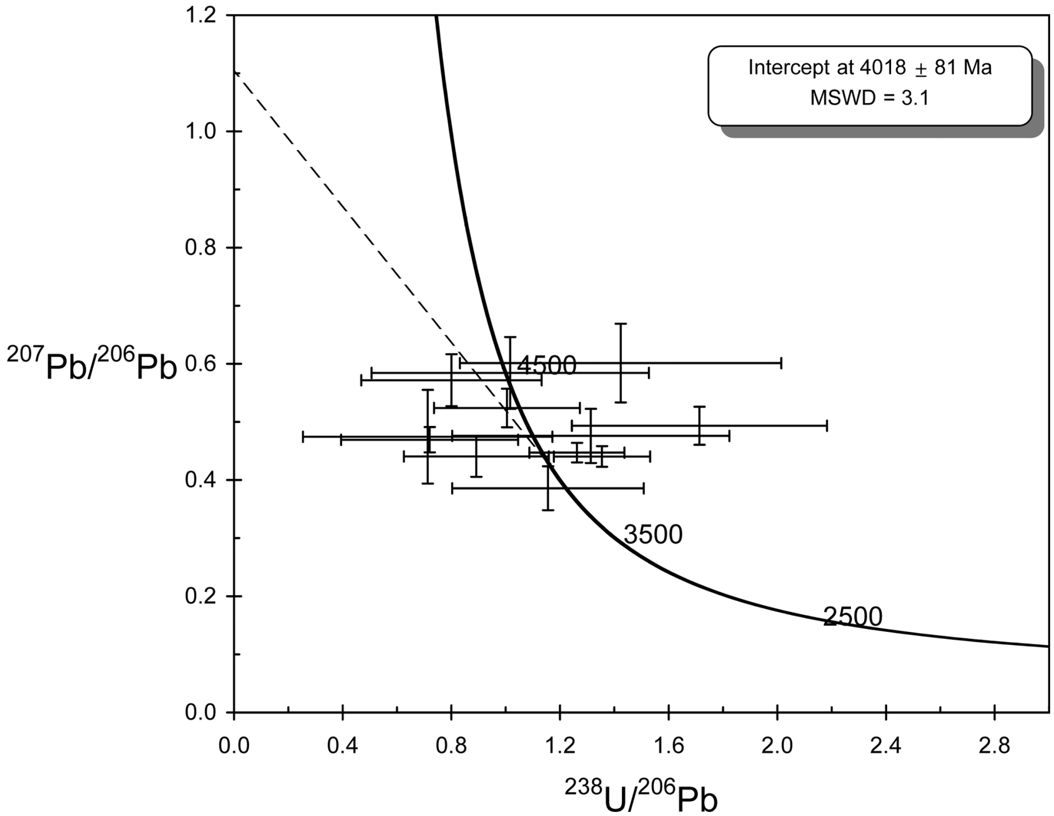

Fig. 1. The result of 3-dimensional linear regressions of ALH 84001 phosphates. Eleven whitlockites and 1 apatite data are projected onto the $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$ plane. Uncerteinties are portrayed at the 1σ level. In this figure, the solid curve is an evolution line of U-Pb systematics (Concordia line), and the dashed line is the projected regression line onto the X-Y plane. Note that the total Pb/U isochron age is determined by the intersection with the Concordia line on the X-Y plane. Three-dimensional linear regression in the $^{238}U/^{206}Pb$-$^{207}Pb/^{206}Pb$-$^{204}Pb/^{206}Pb$ space yields a total Pb/U isochron age of 4018 ± 81 Ma (95% confidence limit; MSWD = 3.1). The linear regressions were calibrated using Isoplot/Ex (Ludwig 2001).

correlation diagram and X-axis in the $^{204}Pb/^{208}Pb$-$^{232}Th/^{208}Pb$ plane, no constraints existed on the Pb-evolution model due to the large uncertainties.

DISCUSSION

So far, the thermal history of ALH 84001 has been examined by several different analytical approaches. The observed U-Th-Pb age of 4018 ± 81 Ma for the phosphates agrees well with the Ar-Ar shock age of 4.0 ± 0.1 Ga from maskelynite (Ash et al. 1996) and other results (3.9–4.3 Ga by Bogard and Garrison [1999] and 4.05–3.8 Ga by Turner et al. [1997]). This age is also consistent with the age of secondary carbonates of 3.90 ± 0.04 Ga via Rb-Sr dating and 4.04 ± 0.10 Ga via Pb-Pb dating, using the leaching techniques (Borg et al. 1999). In contrast, the U-Th-Pb age of phosphates is not consistent with the Sm-Nd age of 4.56 Ga (Jagoutz et al. 1994) and Sm-Nd isochron age of 4.50 ± 0.13 Ga, defined in pyroxene and whole rock samples (Nyquist et al. 1995). It is also slightly older than a Rb-Sr internal isochron age of 3.84 ± 0.05 Ga based on whole rocks and orthopyroxenes (Wadhwa and Lugmair 1996).

Based on petrography, Treiman (1995) suggested that the history of ALH 84001 must include a crystallization from source magma, a first shock (impact) metamorphism, thermal metamorphism, low-temperature chemical alteration, and a second shock (impact) metamorphism to launch it to the Earth. Greenwood and McSween (2001) also suggested that there were 2 shock events, the pre-carbonate shock event and a post-carbonate shock, and that the latter event appears to have been of lower intensity than the pre-carbonate shock event and likely resulted in liberating the sample from Mars. Thus, no disagreement exists on the point that ALH 84001 has experienced at least 2 shock events, although, the relative timing of igneous crystallization, shock events, and carbonate precipitation is highly contested. Kirschvink et al. (1997) argued, on the basis of paleomagnetic evidence, that post-carbonate shock did not heat this meteorite above 150°C for more than a few sec. Weiss et al. (2000) also investigated the images of the magnetic field of ALH 84001 and concluded that ALH 84001 was only lightly shocked during its ejection and that the interior of the rock has not been above 40°C, indicating the last impact event did not cause the disruption of the U-Th-Pb system. On the other hand, Cooney et al. (1999)

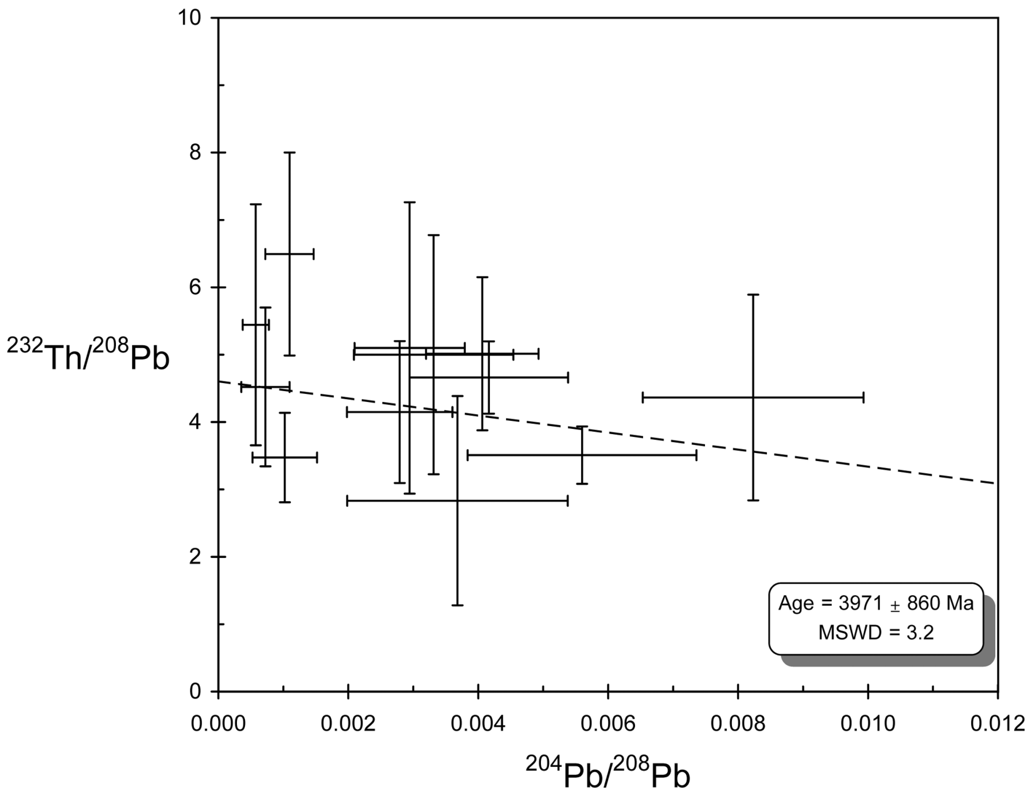

Fig. 2. Correlation between the $^{204}Pb/^{208}Pb$ ratios and the $^{232}Th/^{208}Pb$ ratios of phosphate in ALH 84001. Uncertainties are portrayed at the 1σ level. A least-squares fit using the York method (York 1969) gives a $^{232}Th/^{208}Pb^*$ isochron age of 3971 ± 860 Ma (95% confidence limit; MSWD = 3.2).

argued that phosphates and carbonates in ALH 84001 were shock melted, based on both micro-Raman and IR reflectance spectra. Moreover, recent textual and chemical studies (Greenwood et al. 2003) also revealed that a high temperature event, such as an impact, melted the phosphate and led to growth of augite rims at phosphate-orthpyroxene grain boundaries. Such a high temperature event would have reset the U-Th-Pb system in phosphates. Taking into account these observations and our result, we conclude that the shock metamorphic event defines the phosphate age of 4018 ± 81 Ma and that since then, ALH 84001 has not experienced a long duration thermal metamorphism, the temperature of which would be compatible to the closure temperature of phosphates of ~500°C (Cherniak et al. 1991). This scenario has much in common with the observation that some carbonate grains are cross-cut by 4 Ga glass (Ash et al. 1996), and their ages were reset during impact metamorphism (Rb-Sr age of 3.90 ± 0.04 Ga and Pb-Pb age of 4.04 ± 0.10 Ga; Borg et al. 1999), although, inherent formation of carbonates could be older than the impact event. The resolved difference between the Sm-Nd age of 4.5~4.6 Ga for bulk rock (Jagoutz et al. 1994; Nyquist et al. 1995) and the phosphate ages of 4 Ga in this study could be explained if the shock metamorphic event at 4 Ga did not reset the Sm-Nd system on large scales such as bulk rock, while it caused the redistribution of U-Th-Pb on micro-scales (Borg, personal communication).

In summary, in situ U-Th-Pb dating of ALH 84001 phosphates was successfully carried out by the SHRIMP. Our result suggests that a shock metamorphic event defines the phosphate formation age of 4018 ± 81 Ma and that since then, ALH 84001 has not experienced a long duration thermal event, which would reset the U-Pb system in phosphates. This analytical method allows for rapid analysis of small amounts of sample in thin sections and will be useful for chronological studies of the returned samples from Mars in the near future.

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