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Dating recent aqueous activity on Mars

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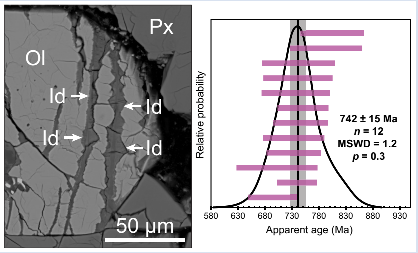
Abstract



Amazonian-age Martian meteorites contain products of indigenous aqueous alteration; yet, establishing when this alteration occurred, and therefore when liquid water was available in the planet's crust, has proven challenging. New $^{40}Ar/^{39}Ar$ dates for iddingsite within the Martian meteorite Lafayette show these minerals precipitated from liquid water at 742 ± 15 Ma (2σ). This age is the most precise constraint to date on water–rock interaction on Mars, and postdates formation of the host igneous rock by ~580 Myr. We infer that magmatic activity most likely induced melting of local permafrost and led to alteration of the nakhlites, suggesting that activation of localised hydrological cycles on Amazonian Mars by magmatism was infrequent and transient, but not unusual.

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Introduction

A key objective of ongoing and future missions to Mars is determining when the planet's hydrological cycle was active in the geologic past. For much of the Amazonian period (2.9 Ga to present), Mars's surface was cold and arid with a thin atmosphere, making liquid water unstable at the surface (*e.g.*, Carr and Head, 2010). However, minerals that formed by aqueous alteration of Amazonian-aged rocks, which have travelled to Earth as meteorites, show that liquid water was available at some points during this time period (Gooding *et al.*, 1991; Treiman *et al.*, 1993).

The nakhlite meteorites are a group of igneous rocks that crystallised between 1416 ± 4 and 1322 ± 5 Ma, and were subsequently ejected by an impact event at 10.7 ± 0.4 Ma (Cohen *et al.*, 2017). Several nakhlites contain aqueous alteration products (*e.g.*, Treiman, 2005) that (1) are crosscut by fusion crusts that formed upon atmospheric entry to Earth (*e.g.*, Gooding *et al.*, 1991; Treiman *et al.*, 1993) and (2) have D/H ratios indicative of fluid equilibration with the Martian atmosphere (Leshin *et al.*, 1996), requiring that these secondary phases formed *via* interaction of the igneous rocks with liquid water on Mars (*e.g.*, Leshin *et al.*, 1996).

Lafayette is one such member of the nakhlites. It is a 0.8 kg olivine-rich pyroxenite find with negligible evidence for terrestrial weathering (Graham *et al.*, 1985; Treiman *et al.*, 1993; Lee *et al.*, 2015). Olivine grains and the mesostasis of Lafayette host aqueous alteration products that include K-bearing hydrous silicates (*e.g.*, Piercy *et al.*, 2022, and references therein; see Supplementary Information). We refer to this alteration mineral assemblage as "iddingsite". The focus of the present study is to determine the age of iddingsite in the olivine-hosted veins (Fig. 1), which comprise 2.3–2.7 volume percent of Lafayette (Lee *et al.*, 2015).

The question of when Lafayette and the other nakhlites were exposed to liquid water on Mars remains unresolved. The most widely cited constraint derives from the Rb-Sr systematics of acid-leachates from the meteorites Lafayette and Yamato (Y) 000593 (Shih *et al.*, 1998; Misawa *et al.*, 2005). These experiments were designed to measure the isotopic systematics of the primary igneous minerals. The acid leach component was meant to remove the iddingsite and any terrestrial weathering products, but was not designed to extract chronological information. Nonetheless, these measurements have also been used to extract apparent, two-point isochron ages of 674 ± 68 Ma and 653 ± 80 Ma (2σ, recalculated; see Supplementary Information for Rb-Sr systematics and $^{40}Ar/^{39}Ar$ methods) for Lafayette (Shih *et al.*, 1998) and Y000593 (Misawa *et al.*, 2005), respectively. With an isochron defined by two points, there is no way to test for contamination or isotopic disturbance, the latter being particularly important as the leaching procedure may produce Rb/Sr fractionation (Clauer *et al.*, 1993). And while these two datasets have been aggregated to estimate a single 'date' for nakhlite alteration (Borg and Drake, 2005), they are not consistent with a single isochron (see Supplementary Information), which raises questions about the overall chronological significance of these data.

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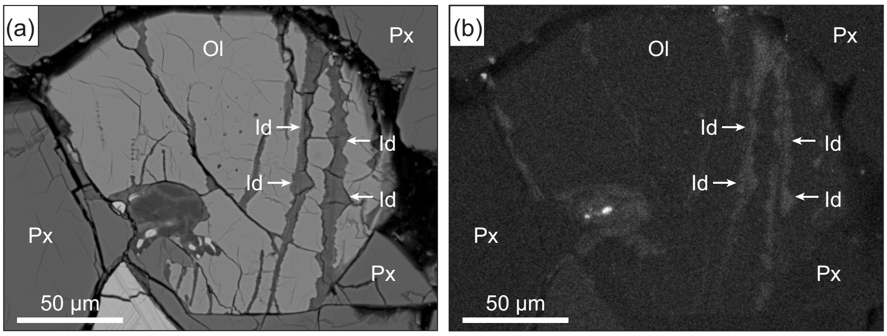

Figure 1 Petrographic context of Lafayette iddingsite. **(a)** Backscattered electron image showing an olivine grain (Ol) surrounded by augite crystals (Px) and cut by iddingsite-filled veins (Id). **(b)** Energy Dispersive Spectroscopy (EDS) X-ray map showing potassium enrichment (brighter grey) in iddingsite. Data were collected using a Carl Zeiss Sigma scanning electron microscope (SEM) at the University of Glasgow, operated in high-vacuum mode at 20 kV and ~2 nA.

Previous work applying the ^{40}K-^{40}Ar chronometer to Lafayette is also ambiguous, suggesting that the nakhlites interacted with aqueous fluids sometime between 670 and 0 Ma (Swindle *et al.*, 2000). These dates were likely impacted by inhomogeneous K distribution between the distinct aliquots of material measured for their ^{40}K and ^{40}Ar content.

Methods

We revisited the age of iddingsite in Lafayette using the ^{40}Ar/^{39}Ar technique, a variant of the ^{40}K-^{40}Ar dating method used by Swindle *et al.* (2000). We obtained 0.216 g of Lafayette from the Smithsonian Institution, sourced from an interior core >20 cm from the fusion crust. Iddingsite was physically separated from the host olivine (see Supplementary Information). During the neutron irradiation required for ^{40}Ar/^{39}Ar dating, the ^{39}Ar atoms produced recoil with an average distance of 0.08 µm in silicate minerals (Turner and Cadogan, 1974). Thus, fine-grained materials with high surface area-to-volume ratios, like the iddingsite studied here, may lose ^{39}Ar during the irradiation process, resulting in spuriously old ages (Onstott *et al.*, 1995). The dating of fine-grained materials using the ^{40}Ar/^{39}Ar technique thus requires a non-conventional approach, which consists of micro-encapsulation to prevent loss of recoiled isotopes (Dong *et al.*, 1997). To achieve this, we encapsulated twelve ~1 µg aliquots of the hand-picked iddingsite in evacuated, flame-sealed quartz glass capsules prior to neutron irradiation. Following irradiation, we measured the Ar isotopic composition of the iddingsite in two stages. First, the glass capsule was cracked under vacuum to measure any recoiled ^{37}Ar and ^{39}Ar. Second, the samples recovered from the cracked glass tubes were then fused with a diode laser. The ^{37}Ar and ^{39}Ar measurements from the cracked tubes were added to the total fusion isotope measurements (^{36}Ar, ^{37}Ar, ^{38}Ar, ^{39}Ar, and ^{40}Ar). Data were corrected for backgrounds, mass discrimination, radioactive decay since irradiation, cosmogenic Ar, and trapped Martian atmospheric Ar. Full analytical procedure details are provided in the Supplementary Information.

Results

Upon cracking the quartz capsules, we observed that significant ^{39}Ar and ^{37}Ar recoil occurred during neutron irradiation into the quartz capsule of 16 ± 2 % and 13 ± 2 %, respectively. When the Ar isotopic data from the two-stage analytical procedure are combined, the data define a normal age distribution with a mean ^{40}Ar/^{39}Ar age of 741.8 ± 15.0/15.2 Ma (analytical precision/full external precision at 2σ). There is no statistically significant difference between the individual aliquot ages, defining a mean square weighted deviation (MSWD) of 1.2 and a *p*-value of 0.3 (Fig. 2). This age for the iddingsite in Lafayette agrees within uncertainty with the previously estimated age from Rb-Sr acid-leachates (Shih *et al.*, 1998) but is significantly more precise. It is slightly older than the wide age range previously estimated from K-Ar measurements (Swindle *et al.*, 2000).

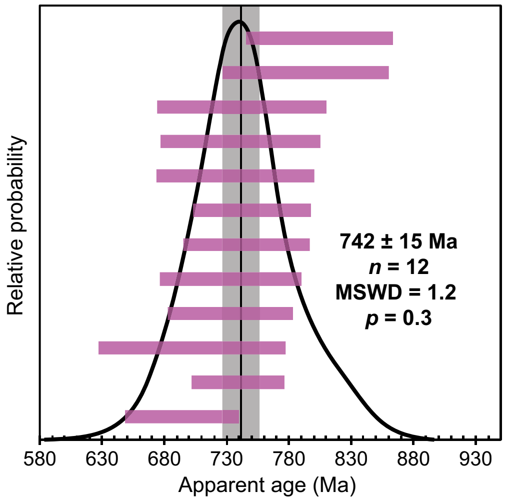

Figure 2 Rank age (purple bars, 2σ) and relative probability (black curve) of ^{40}Ar/^{39}Ar dates for 12 aliquots of Lafayette iddingsite. The data define a Gaussian distribution and indicate no scatter beyond what is expected from measurement precision.

Discussion

Potential for diffusive loss of Ar. Because we conducted total fusion ^{40}Ar/^{39}Ar measurements on the iddingsite aliquots, we did not recover information about the spatial distribution of radiogenic ^{40}Ar in the constituent phases. Therefore, we must evaluate the possibility that the aqueous alteration in Lafayette occurred earlier than 742 Ma, and that heating events at 742 Ma, or later, caused partial loss of radiogenic ^{40}Ar. The maximum possible ^{40}Ar loss is 44 %, based on the difference between the measured iddingsite age and Lafayette's igneous crystallisation age (~1322 Ma; Cohen *et al.*, 2017).

The bulk ^{40}Ar/^{39}Ar systematics of the nakhlites reported by Cohen *et al.* (2017) preclude protracted heating of Lafayette during its residence in Mars's crust. Therefore, we consider three aspects of Lafayette's history that could have induced ^{40}Ar loss: (1) heating during an impact event, such as the impact that ejected Lafayette from Mars *ca*. 10.7 Ma; (2) heating during Lafayette's transit in space before falling to Earth; and (3) heating during entry into Earth's atmosphere. To assess the influence of heating during these events on the ^{40}Ar/^{39}Ar age, we modelled the diffusive loss of Ar from iddingsite. Following others who have applied ^{40}Ar/^{39}Ar dating to authigenic clays (*e.g.*, Clauer *et al.*, 2003), we assume the diffusion kinetics of Ar in muscovite are comparable to those in the K-bearing phases comprising iddingsite and use the kinetic parameters for Ar diffusion in muscovite reported by Harrison *et al.* (2009). The grain size of the iddingsite in Lafayette is not well constrained, but it cannot be greater than 10–20 µm based on the width of the olivine-hosted veins. We therefore explored models with diffusion radii between 0.01 and 10 µm, assuming the grain size defines the diffusion length scale.

In the case of the 10.7 Ma impact event and/or atmospheric entry, we use approximations for fractional loss during a square pulse heating event (Fig. 3a; see Supplementary Information). The duration of heating for both events is geologically brief: up to several hours after ejection (*e.g.*, Fritz *et al.*, 2005) or hundreds of seconds during atmospheric entry (*e.g.*, Parnell *et al.*, 2008). For these scenarios, we therefore assume radiogenic ^{40}Ar production during heating is negligible. However, in the case of Lafayette's 10.7 Myr transit in space, the production of radiogenic ^{40}Ar is nontrivial. We therefore use a solution for radiogenic ^{40}Ar production and diffusive loss during an isothermal heating event, which incorporates the initial age prior to heating. Although Lafayette's temperature would have varied as it transited the inner Solar System, we use the isothermal heating solution as an end member to estimate the minimum temperatures that would yield diffusive loss (Fig. 3b; see Supplementary Information).

For an impact and atmospheric entry, temperatures >~250 °C would need to be sustained for several days, or longer, for Ar loss to exceed 1 % (Fig. 3a). Lower temperatures are required for Ar loss during space transit due to its longer duration (Fig. 3b). For example, for a grain radius of 0.01 µm, temperatures would need to exceed ~160 °C for >1 % diffusive loss of Ar to take place. Higher temperatures during space transit are permitted without diffusive loss over durations <10.7 Myr.

When paired with other observations, these first-order thermal constraints suggest that an age for the Lafayette iddingsite significantly older than 742 Ma age is highly unlikely. For heating during impact ejection, independent estimates based on the shock petrography of Lafayette indicate that shock and post-shock temperatures were at most several tens of degrees Celsius above ambient Martian surface temperatures (*e.g.*, Fritz *et al.*, 2005; Daly *et al.*, 2019). Following ejection, the maximum temperatures that Lafayette could have experienced during space transit are 200–250 °C, which would occur if its orbit approached Mercury (Butler, 1966). However, orbital dynamics calculations demonstrate that a prolonged duration near Mercury is extremely unlikely for a Martian meteorite that falls to Earth (*e.g.*, Gladman, 1997). Therefore, Lafayette likely spent most, or all, of its 10.7 Myr space transit at temperatures well below those required to cause Ar loss (150–200 °C; Fig. 3b). Finally, we expect heating during atmospheric entry to be insignificant, considering that our samples were sourced ~20 cm from Lafayette's fusion crust. The Stone 5 experiments, which placed rocks on the nose of a spacecraft to simulate atmospheric entry, only documented re-entry heating >100 °C at <2 cm depth below the rocks' fusion crust (Parnell *et al.*, 2008). Collectively, these observations and our calculations indicate that while partial Ar loss from Lafayette iddingsite is possible under certain conditions, it is very unlikely given other constraints about Lafayette's history.

Significance of the ^{40}Ar/^{39}Ar age. Given that (1) the ^{40}Ar/^{39}Ar data show no significant scatter beyond what is expected from the measurement precision, (2) radiogenic ^{40}Ar loss from the Lafayette iddingsite is highly unlikely, and (3) the new ^{40}Ar/^{39}Ar date is broadly consistent with previous but significantly less precise age estimates, we interpret the ^{40}Ar/^{39}Ar age of 742 ± 15 Ma as recording the time when the iddingsite in Lafayette formed as a result of water–rock interaction close to the surface of Mars.

Our data show that the impact event recorded by Nakhla at ~913 Ma (Cassata *et al.*, 2010) was not coeval with the aqueous activity that led to iddingsite formation in Lafayette. The impact

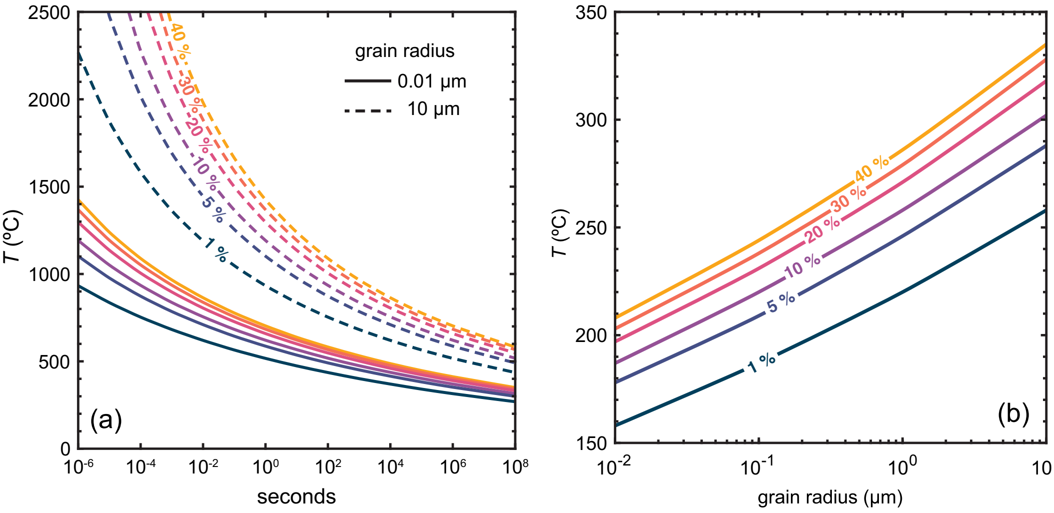

Figure 3 Thermal limits on Ar diffusive loss from Lafayette iddingsite. **(a)** Argon fractional loss (0–40 %) as a function of duration and temperature of a square pulse heating event, such as during an impact or atmospheric entry, for two different end member iddingsite grain sizes. **(b)** Argon fractional loss as a function of grain size and temperature for an isothermal heating event lasting 10.7 Myr, the duration of Lafayette's transit in space (Cohen *et al.*, 2017).

event at 913 Ma is the only evidence that the nakhlites have experienced impact-induced shock following cooling of the parent lavas and prior to their ejection at 10.7 Ma. The impact event at 913 Ma was likely responsible for producing the shock features in Lafayette documented by Daly *et al.* (2019), and for increasing the porosity and permeability for later aqueous solutions to infiltrate grain interiors within Lafayette at 742 Ma (Lee *et al.*, 2015), but did not induce the aqueous alteration of Lafayette or the nakhlites directly. The data do not preclude the occurrence of a third impact event at 742 Ma. However, such an event would have to be low enough energy to not disturb the Ar isotope systematics of primary phases in the nakhlites, but high enough energy to induce subsurface melting of localised permafrost, providing the heat source for water–rock interaction that led to alteration of the nakhlites (*e.g.*, Changela and Bridges, 2010). Moreover, it is unlikely that three large impact events occurred at the same place on the Martian surface during the last 1 Ga (*ca*. 913 Ma, 742 Ma, 10.7 Ma).

An alternate and simpler explanation is that magmatism acted as a localised heat source for melting of subsurface ice and water–rock interaction during the Amazonian on Mars. On Earth, iddingsite is commonly observed in olivine grains of igneous rocks that experience post-emplacement hydrothermal activity not linked to impact cratering (*e.g.*, Carlson and Rodgers, 1975), so it is reasonable to infer a similar process happening on Mars. The spatial variability of alteration within the nakhlites (Lee *et al.*, 2015) is consistent with a model of heat from magmatic eruptions or intrusions at ∼742 Ma, inducing localised, short-lived melting of permafrost. Such spatial variability is diagnostic of a small-scale hydrologic system, whereas higher water to rock ratios, and more pervasive alteration, are more commonly associated with an allochemical, impact-driven hydrothermal cell (Bridges and Schwenzer, 2012). A model for a short-lived heat source of magmatic origin is thus congruent with the low temperature models for nakhlite alteration proposed previously (*e.g.*, Treiman *et al.*, 1993). Fluid–rock interaction at ∼742 Ma selectively widened and extended the fractures within olivine through carbonation (*e.g.*, Tomkinson *et al.*, 2013). The dissolution of the host olivine provides many of the cations required to precipitate the secondary minerals observed in the nakhlites (Lee *et al.*, 2015)—another key requirement of a highly localised, isochemical system. Similarly, the potassium found within the iddingsite (Fig. 1) was likely sourced locally from dissolution of feldspar and glass in the mesostasis (Bridges and Schwenzer, 2012).

The timing of iddingsite formation in Lafayette coincides with a volcanically active period of Mars's history, albeit eruption frequency was decreasing. Amazonian-age volcanism is restricted to the Tharsis and Elysium regions of Mars and their peripheries (Carr and Head, 2010); the nakhlites are most likely sourced from these regions (Herd *et al.*, 2024). Crater ages of tens of millions of years for volcanic surfaces in Tharsis and Elysium, including the crater that formed when the nakhlites were ejected, suggest that Mars is still episodically volcanically active.

Conclusions

The new $^{40}Ar/^{39}Ar$ geochronology presented here demonstrates that the timing for aqueous alteration of Martian volcanic rocks by water was unrelated to their emplacement or an impact event, but most likely related to ongoing magmatic activity on Mars *ca.* 742 Ma. Our proposed model is consistent with previous models for the alteration environment of the nakhlites. Considering the low eruption rate for Amazonian volcanoes and the prevalence of permafrost across Mars, our data support interpretations that activation of localised hydrological cycles on Amazonian Mars by magmatic activity was infrequent but not unusual.

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Additional Information

Supplementary Information accompanies this letter at https://www.geochemicalperspectivesletters.org/article2443.

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