



Meteoritic evidence for a previously unrecognized hydrogen reservoir on Mars



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ABSTRACT

Fluvial landforms on Mars suggest that it was once warm enough to maintain persistent liquid water on its surface. The transition to the present cold and dry Mars is closely linked to the history of surface water, yet the evolution of surficial water is poorly constrained. Based on *in situ* hydrogen isotope (D/H) analyses of quenched and impact glasses in Martian meteorites, we provide evidence for the existence of a distinct but ubiquitous water/ice reservoir ($D/H \approx 2\text{--}3$ times Earth's ocean water) that lasted from at least the time when the meteorites crystallized (173–472 million years ago) to the time they were ejected by impacts (0.7–3.3 million years ago), but possibly much longer. The origin of this reservoir appears to predate the current Martian atmospheric water ($D/H \approx 5\text{--}6$ times Earth's ocean water) and is unlikely to be a simple mixture of atmospheric and primordial water retained in the Martian mantle ($D/H \approx$ Earth's ocean water). This reservoir could represent hydrated crust and/or ground ice interbedded within sediments. Our results corroborate the hypothesis that a buried cryosphere accounts for a large part of the initial water budget of Mars.

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1. Introduction

Recent Mars exploration missions have provided compelling evidence for the presence of liquid water and warmer climatic conditions during the earliest geologic era (Noachian: ~ 3.9 to 4.5 Ga) of Mars. Observations of dense valley networks and evaporites (e.g., gypsum) and hydrous minerals (e.g., clays) that are commonly formed by aqueous processes imply that some time in the past Mars had an Earth-like hydrological cycle with large lakes or oceans (Carr, 2006). In contrast to this ancient warm environment, today the surface of Mars is cold and dry. The recent desert-like surface conditions, however, do not necessarily indicate a lack of surface or near-surface water/ice. Massive deposits of subsurface ice have been detected using subsurface radar sounder observa-

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tions by the European Mars Express mission (Mouginot et al., 2012). Furthermore, landforms that appear to be glacial commonly occur even in the equatorial regions; this has led to the hypothesis that, like on Earth, Mars had recent ice ages in which water–ice would have globally covered the surface (Head et al., 2003, 2005). The amount and physical state (ice, liquid, and vapor) of surficial water are closely linked to climate and the potential for life on Mars. Hence, it is important to constrain the global inventory of surficial water/ice reservoirs on Mars and how they have changed with time.

The evolution of surface water/ice and its interaction with the atmosphere can be traced by measurements of hydrogen isotope ratios (D/H: deuterium/hydrogen). Hydrogen is a major component of water (H_2O) and its isotopes fractionate significantly during hydrological cycling between the atmosphere, surface waters, ground ice, and the polar ice caps. Telescopic studies have reported that the hemispheric mean D/H ratio of the Martian atmosphere is ~ 6 times the terrestrial values ($\delta D \approx 5000\text{‰}$; Owen et al., 1988); $\delta D = [(D/H)_{\text{sample}} / (D/H)_{\text{reference}} - 1] \times 1000$, where the reference is

Standard Mean Ocean Water (SMOW). The high atmospheric D/H ratio has been interpreted to result from the preferential loss of hydrogen relative to the heavier deuterium from the Martian atmosphere throughout the planet's history (Bibring et al., 2006; Jakosky and Phillips, 2001; Kurokawa et al., 2014). Recent telescopic measurements have revealed a strong latitudinal variation in the hydrogen isotopic composition of atmospheric water, ranging from low δD ($\sim 3000\text{‰}$) near the polar regions to high δD ($\sim 6000\text{‰}$) near the equatorial regions (Novak et al., 2011). This latitudinal D/H variation is explained by the interplay between multi-latitudinal reservoirs with different average D/H ratios or different latitudinal water/ice abundances (Fisher et al., 2008; Fisher, 2007). While telescopic measurements help constrain the hydrogen isotopic compositions of present-day surficial water reservoirs, subsurface water–ice and the historical variations in the isotopic composition(s) of surface or near-surface water/ice reservoirs are potentially contained in Martian meteorites that formed at different times over the past 0.15 to 4.1 Ga (Lapen et al., 2010; McSween, 2007; Nyquist et al., 2009).

Martian meteorites are all igneous rocks that were either extruded onto the surface as lava flows or solidified as magma bodies in the shallow crust (McSween, 2007), except for the recently found Martian breccia NWA 7034/7533 (Agee et al., 2013; Humayun et al., 2013). Hydrogen isotope analyses of these Martian meteorites provide a significantly greater δD range (-111‰ to $+6034\text{‰}$, Boctor et al., 2003; Greenwood et al., 2008; Hallis et al., 2012a, 2012b; Hu et al., 2014; Leshin, 2000; Usui et al., 2012; Watson et al., 1994) than the δD range for most terrestrial water (-350‰ to $+50\text{‰}$; Hoefs, 2009). Such variation has been interpreted to result from the mixing of two distinct components: a D-enriched component derived from crustal fluids that had interacted with the Martian atmosphere ($\delta D = \sim 5000\text{‰}$), and a D-poor component that is from the Martian mantle and/or terrestrial contamination. The D/H ratio of the D-poor, primordial mantle component has not been well constrained, ranging from -111‰ (Hallis et al., 2012a) to $\sim +900\text{‰}$ (Leshin, 2000). Such uncertainty has arisen both from the scarcity of primitive Martian meteorites and as a result of secondary processes (including contamination by terrestrial water) that obscure the signature of the Martian mantle. The most promising estimate for the Martian primordial water ($\delta D \leq 275\text{‰}$) is obtained from olivine-hosted melt inclusions in the most magnesian olivine-phyric shergottite Yamato 980459 (Usui et al., 2012).

Martian meteorites contain hydrogen-bearing magmatic phases such as apatite (e.g., Boctor et al., 2003; Filiberto and Treiman, 2009; Hallis et al., 2012a; McCubbin et al., 2012) and water-bearing melt inclusions trapped in nominally anhydrous minerals (e.g., olivine) (e.g., Boctor et al., 2003; Usui et al., 2012). Analyses of these magmatic phases provide the hydrogen isotopic compositions of water in their source magmas that originated from the deep crust and mantle (Boctor et al., 2003; Greenwood et al., 2008; Hallis et al., 2012a; Usui et al., 2012). On the other hand, some Martian meteorites contain post-magmatic glass formed either during volcanic eruption or by the impact events that ejected their host meteorites from Mars and delivered them to Earth. These impact melts are known to have trapped surficial and atmospheric components (Bogard and Johnson, 1983; Rao et al., 2011; Treiman et al., 2000). Thus, comprehensive hydrogen isotope analyses of residual volcanic glass and impact melt-glass can potentially reveal the history of Martian surface water over the last ~ 4 Ga.

Hydrogen isotope measurements have been mostly obtained from apatite because it contains high water abundances (~ 5000 ppm) and is commonly found in Martian meteorites (Boctor et al., 2003; Greenwood et al., 2008; Hallis et al., 2012a; Leshin, 2000); melt inclusion measurements are less common, due in part to their scarcity (Boctor et al., 2003; Usui et al., 2012). Despite the fact that several Martian meteorites contain ample glass

in their matrix, only a few measurements have been reported (e.g., Boctor et al., 2003). The absence of D/H impact melt data mainly reflects the analytical difficulties posed by their low water contents and potential terrestrial contamination.

This study reports hydrogen isotope compositions (δD) and volatile abundances (H_2O , CO_2 , F, Cl, S) of matrix glass phases in Martian meteorites. A recently developed sample preparation method (Usui et al., 2012) enables low-volatile contamination, *in situ* ion microprobe analyses. Three Martian basaltic meteorites (olivine-phyric shergottites): Yamato 980459 (Y98), Elephant Moraine 79001 (EETA79), and Larkman Nunatak 06319 (LAR06) were studied. These shergottites are geochemically distinct in terms of their radiogenic isotopes (e.g., Sr, Nd and Hf) and relative trace element abundances (e.g., rare earth elements), suggesting that they originated from different magmatic sources (see Section 3). They also have different crystallization and ejection ages, indicating different magmatic events and launching sites from Mars, respectively. These lines of evidence suggest that δD values of matrix/impact melt glasses from the three meteorites potentially provide geochemical information of surficial water–ice reservoirs from different localities and different times on Mars. Remarkably, *in situ* ion microprobe analyses of these geochemically different shergottites (this study) demonstrate that Mars may have a ubiquitous hydrogen reservoir with an intermediate D/H ratio ($\delta D = 1000\text{--}2000\text{‰}$) that has previously gone unrecognized.

2. Analytical methods

2.1. Low-contamination sample preparation

Hydrogen contamination of thin section samples during preparation, storage, and during the ion microprobe analysis can lead to analytical artifacts as shown by Boctor et al. (2003), Greenwood et al. (2008). Sources of these contaminants include air left in the vacuum system, oils (and/or water) used as lubricants during polishing, and epoxy (or acrylic) resin used as a mounting agent. Among them, resins are the most unavoidable blank sources for Martian meteorites that were highly shocked (typically to >20 GPa, Nyquist et al., 2001) and as a result have many micro-fractures. Resins penetrate into these fractures and cannot be removed.

We have employed a sample preparation method using indium metal instead of resin. Rock chips of three meteorite samples (Y98: Y-980459, 67; EETA79: EETA79001, 654; LAR06: LAR 06319, 50) were first embedded into molten indium metal set in aluminum holders (1-inch dia.) on a hot plate ($\sim 160^\circ\text{C}$ for ~ 5 min), then cooled under vacuum until the indium metal solidified. The samples embedded in the indium metal were sliced and polished with diamond grit for electron and ion microprobe analyses (Fig. S1). A final polish with corundum powder was performed to remove carbon contamination from the diamond polish. We made four Y98, two EETA79, and two LAR06 polished sections for this study. This method greatly reduced contamination of volatile elements. The potential for contamination was further reduced by using detailed petrographic observations and checking of secondary ion images (Section 2.3), and possible levels of contamination were assessed using mixing calculations (Section 5.1).

2.2. Electron microprobe analysis

The polished sections were carbon coated and analyzed using an electron microprobe (Cameca SX-100) at the NASA Johnson Space Center (JSC). They were examined by reflected optical microscopy and by scanning electron microscope (JEOL JMS 5910LV) at JSC before the microprobe analyses. Mineral and glass compositions were determined by wavelength dispersive spectrometry

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