

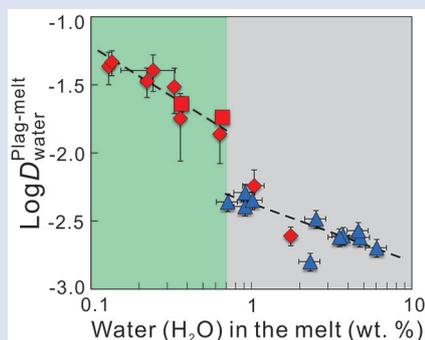
A lunar hygrometer based on plagioclase-melt partitioning of water

Y.H. Lin^{1*}, H. Hui^{2,3*}, Y. Li⁴, Y. Xu², W. van Westrenen^{1*}



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Abstract



>99 % hydrogen degassing occurred during the evolution of the LMO.

The Moon was initially covered by a magma ocean. Hydrogen detected in plagioclase of ferroan anorthosites, the only available samples directly crystallised from the lunar magma ocean (LMO), can be used to quantify LMO hydrogen content. We performed experiments to determine plagioclase-melt partition coefficients of water under LMO conditions with water contents of co-existing plagioclase and melt quantified using Fourier-Transform Infrared Spectroscopy. Results indicate lunar plagioclase can incorporate approximately one order of magnitude more water than previously assumed. Using measured water contents of lunar plagioclase, this suggests that $\sim 100 \mu\text{g/g H}_2\text{O}$ equivalent was present in the residual magma when 95 % of the initial LMO had crystallised. Our results constrain initial LMO water contents to $\sim 5 \mu\text{g/g H}_2\text{O}$ equivalent if water was conserved throughout LMO evolution. If on the other hand the initial LMO contained $>1000 \mu\text{g/g}$ water as suggested by experiments on LMO crystallisation,

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Introduction

The canonical view of a dry lunar interior has been challenged by detections of hydrogen (H or OH, reported here as equivalent amounts of H_2O in $\mu\text{g/g}$) in picritic glass beads (Saal *et al.*, 2008), apatites (*e.g.*, McCubbin *et al.*, 2010; Lin and van Westrenen, 2019), olivine-hosted melt inclusions (*e.g.*, Hauri *et al.*, 2011) and plagioclases (Hui *et al.*, 2013). Sample-based inferences about water in the Moon have been complemented by experimental and modelling studies of lunar magma ocean (LMO) crystallisation (Elkins-Tanton and Grove, 2011; Lin *et al.*, 2017a,b; Charlier *et al.*, 2018; Rapp and Draper, 2018). Quantification of the evolution of the lunar interior volatile budget would provide further insight into the thermal and magmatic evolution of the Moon. However, converting hydrogen abundance data measured in lunar samples or estimated from laboratory experiments to models of the temporal and spatial evolution of water in the Moon, is far from straightforward (McCubbin *et al.*, 2015a).

This study focuses on improving constraints on the water content in the Moon specifically during the LMO stage. Plagioclase is thought to have crystallised and floated to the surface during the late stages of LMO crystallisation, forming the lunar primary feldspathic crust (Warren, 1985).

This indicates that plagioclase in lunar ferroan anorthosite could be our best candidate for estimating the water content of the LMO (Hui *et al.*, 2013, 2017). In addition, plagioclase could have formed continuously from $\sim 70\%$ all the way up to $>99\%$ of LMO crystallisation (Lin *et al.*, 2017a; Charlier *et al.*, 2018; Rapp and Draper, 2018). Therefore, the water content of plagioclase formed at different stages during LMO crystallisation could in principle be used to track and quantify the LMO water content through time.

While nominally anhydrous, terrestrial plagioclase can incorporate trace amounts of H as structural OH and/or molecular H_2O . In magmatic feldspars, concentrations from less than a few to more than $1000 \mu\text{g/g H}_2\text{O}$ have been reported (Johnson and Rossman, 2003, 2004; Johnson, 2006; Mosenfelder *et al.*, 2015). Only very few studies have measured water contents of lunar feldspars from a primary crystallisation product of the LMO so far (Hui *et al.*, 2013; 2017).

To link the water content in plagioclase to that in the melt from which the mineral crystallised, plagioclase-melt partition coefficients D of water are required, with $D_{\text{water}}^{\text{plag-melt}} = C_{\text{water}}^{\text{plag}} / C_{\text{water}}^{\text{melt}}$. Literature values for $D_{\text{water}}^{\text{plag-melt}}$ range between 0.002 ± 0.0004 and 0.006 ± 0.0009 (recalculated using the plagioclase absorption coefficient determined by Mosenfelder *et al.* (2015) based on measurements carried out

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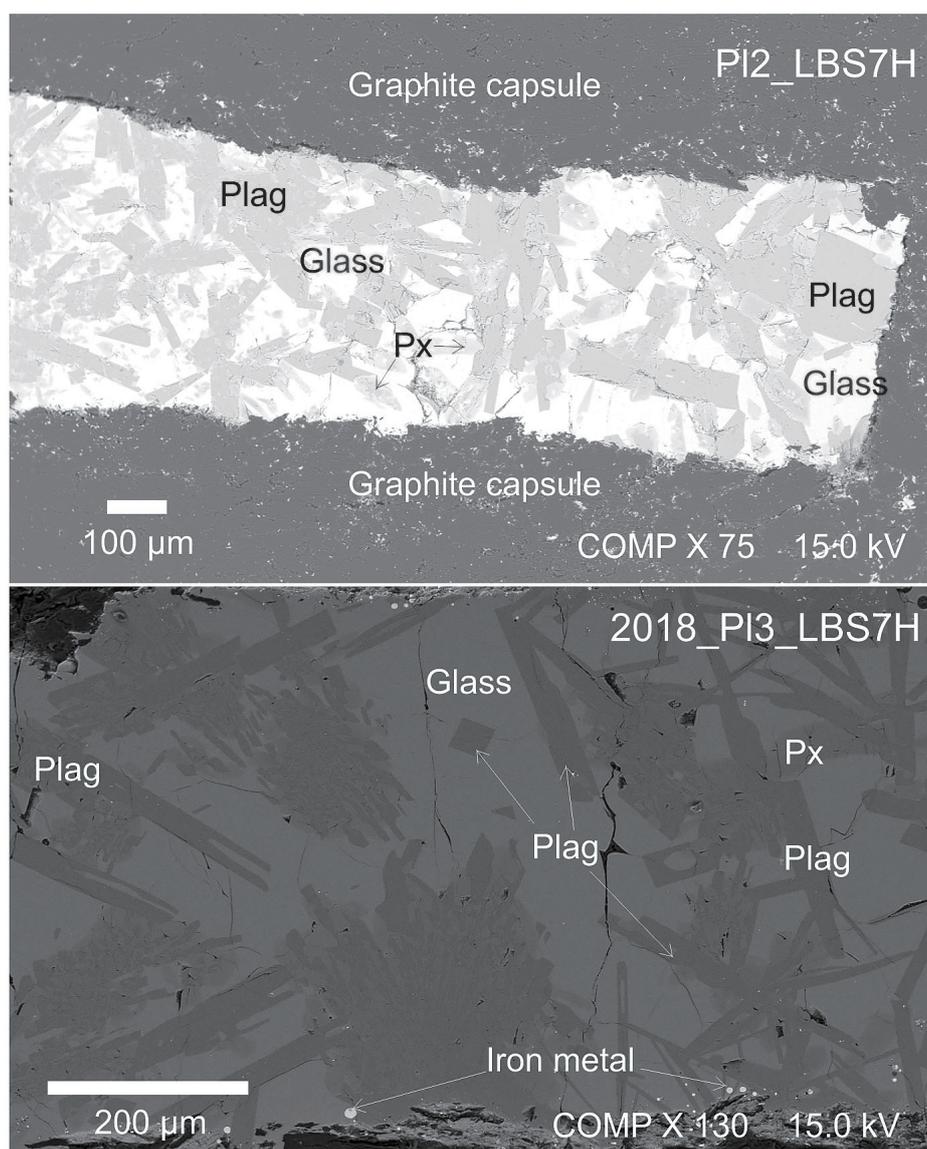


Figure 1 Backscattered electron (BSE) images of representative experimental run products (PI2_LBS7H, 0.4 GPa – 1200 °C; and 2018_PI3_LBS7H, 0.3 GPa – 1160 °C). Px = pyroxene; Plag = plagioclase.

in both natural and experimental systems, which have focused solely on magmatism on Earth (*e.g.*, Hamada *et al.*, 2013). To date, no plagioclase-melt partition coefficient of water under lunar conditions has been published. This is problematic, for example in terms of oxygen fugacity, as it has previously been suggested that fO_2 can affect hydrogen solubility in plagioclase (Yang, 2012). The available $D_{\text{water}}^{\text{plag-melt}}$ data, which are applied to terrestrial systems, were obtained at relatively oxidising conditions. The fO_2 in the Moon is thought to be significantly lower, at $\sim IW$ to $\sim IW-2$ (IW: iron-wüstite) (Sato *et al.*, 1973; Rutherford and Papale, 2009) based on sample analyses. In addition, although Yang (2012) suggests plagioclase composition, temperature and pressure have insignificant effects on $D_{\text{water}}^{\text{plag-melt}}$, this suggestion was based on experiments conducted in a limited temperature-pressure range.

In this study, $D_{\text{water}}^{\text{plag-melt}}$ was determined at pressure-temperature-composition conditions occurring in the lunar magma ocean using high pressure and high temperature experiments and Fourier-Transform Infrared Spectroscopy (FTIR). The pressures (0.4–0.6 GPa) and temperatures (1130–1220 °C) were chosen to be consistent with plagioclase formation during crystallisation of a water-bearing lunar magma ocean (Lin *et al.*, 2017a). The main purposes of this paper are: (1) to

quantify the effects of composition and fO_2 on $D_{\text{water}}^{\text{plag-melt}}$, and (2) to offer further constraints on the water content of the LMO at the time of plagioclase crystallisation.

Water Partition Coefficients

Details of high pressure, high temperature, plagioclase-melt partitioning experiments are given in the Supplementary Information. Table 1 provides a summary of experimental P - T - fO_2 conditions. Starting compositions, EMPA analyses of the major element concentrations in plagioclase and melt phases in the experimental run products, and $\log(fO_2)$ calculations are shown in Tables S-1 and S-2 of the Supplementary Information. All experimental charges contain plagioclase, pyroxene, and quenched glass (Fig. 1). One experimental charge contains Fe metal in addition, indicating an oxygen fugacity at or below that of the iron-wüstite buffer. Representative unpolarised FTIR spectra of plagioclase are shown in Figure 2. All plagioclases show absorption bands (~ 3000 – 3600 cm^{-1}) in the mid-infrared region typical of O–H bonds (Johnson and Rossman, 2004). No H_2 bands have been observed in our plagioclase spectra. Further descriptions are shown in the Supplementary Information.

Table 1 gives FTIR-derived H₂O concentrations in plagioclase and glass from our work, Hamada *et al.* (2013) and Caseres *et al.* (2017). The H₂O equivalent concentrations in our samples range from 42 ± 6 to 99 ± 36 µg/g in plagioclase and 0.13 ± 0.01 to 1.74 ± 0.01 wt. % in silicate glass. The corresponding partition coefficients range between 0.0020 ± 0.0004 and 0.0460 ± 0.0096.

The H₂O concentrations in our plagioclase crystals are significantly below water solubility at our experimental conditions (Yang, 2012). Sample 2018_PI17_LBS8H, with glass containing the highest water concentration (1.74 wt. % H₂O), has the lowest water concentration in plagioclase (42 µg/g H₂O) and hence the lowest partition coefficient ($D_{\text{water}}^{\text{plag-melt}} = 0.0020 \pm 0.0004$). This lowest value is at the lower end of the range of previously published partition coefficients ($D_{\text{water}}^{\text{plag-melt}} = 0.002\text{--}0.006$) by Hamada *et al.* (2013). Our highest partition coefficient is ~7–20 times higher than values from the Hamada *et al.* (2013) data set. The water partition coefficients reported by Caseres *et al.* (2017; $D_{\text{water}}^{\text{plag-melt}} = 0.018\text{--}0.023$ at the IW buffer) overlap with our results.

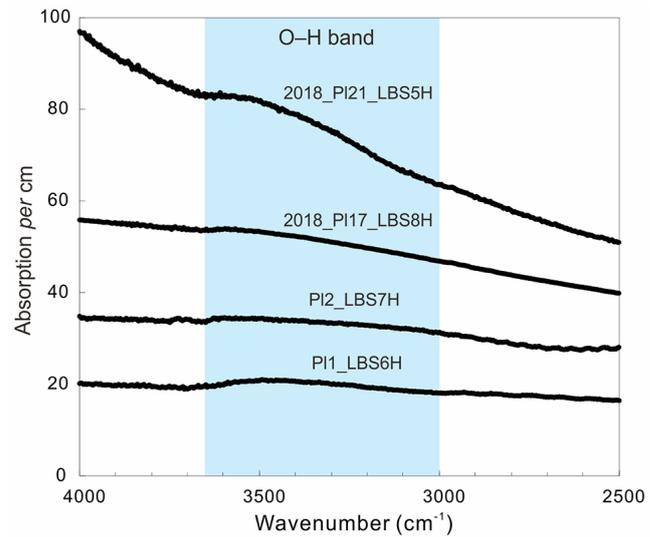


Figure 2 Representative unpolarised infrared spectra of plagioclase, normalised to 1 cm thickness. Spectra are shifted vertically to facilitate comparison.

Table 1 Summary of experimental conditions and water contents of run products in our experiments and literature data.

Sample	Conditions			Plagioclase			Glass			Water partition coefficient		Oxygen fugacity				
	P (GPa)	T °C	Duration (h)	OH (µg/g H ₂ O)	n	1 s.d.	OH (wt.% H ₂ O)	n	1 s.d.	$D_{\text{water}}^{\text{plag-melt}}$	1 s.d.	Oxygen buffer	Log (<i>f</i> O ₂)			
This study																
PI1_LBS6H	0.4	1160	14	58.2	11	17.9	1.03	9	0.16	0.006	0.002	Graphite-COH (C-COH)	-10.4			
PI2_LBS7H		1200	16	63.2	9	32.8	0.35	10	0.02	0.018	0.009		-10.0			
PI3_LBS8H_1		1160	22	96.0	4	28.8	0.24	12	0.09	0.040	0.012		-10.4			
PI4_LBS8H_2		1180	14	85.5	5	33.8	0.63	8	0.04	0.014	0.005		-10.2			
PI5_LBS8H_3		1180	18	99.1	6	36.4	0.32	10	0.01	0.030	0.011		-10.2			
2018_PI2_LBS8H		1170	24	73.4	7	17.0	0.22	8	0.02	0.034	0.008		-10.3			
2018_PI17_LBS8H		1180	24	42.2	8	6.64	1.74	9	0.01	0.002	0.000		-10.2			
2018_PI21_LBS5H	1190	24	54.2	6	14.2	0.13	8	0.01	0.043	0.012	-10.1					
2018_PI3_LBS7H	0.3	1160	24	61.1	7	12.602	0.13	11	0.004	0.046	0.010	Iron-Wustite (IW)	-12.4			
Caseres <i>et al.</i> (2017)																
1#	0.8	1150		82		11	0.36		0.003	0.023	0.003	Iron-Wustite (IW)	-12.3			
2#				118		6	0.65		0.008	0.018	0.001		-12.3			
Hamada <i>et al.</i> (2013)																
MTL04	0.35	1130	24	89.8		13.5	3.70		0.56	0.002	0.000	> Ni-NiO (NNO)	-4.7			
MTL05		1170	24	80.8		12.1	2.50		0.38	0.003	0.000					
MTL17		1220	24	35.9		5.4	0.90		0.14	0.004	0.001					
MTL22		1130	24	36.0		5.4	2.30		0.35	0.002	0.000					
MTL26		1160	24	30.1		4.5	0.70		0.11	0.004	0.001					
MTL29		1170	24	45.4		6.8	0.90		0.14	0.005	0.001					
MTL33		1230	24	44.4		6.7	1.00		0.15	0.004	0.001					
MTL37		1070	24	111		16.6	4.70		0.71	0.002	0.000					
MTL39		1100	24	82.9		12.4	3.50		0.53	0.002	0.000					
MTL40		1100	24	121		18.2	4.60		0.69	0.003	0.000					
MTL41		1050	24	119		17.8	6.00		0.90	0.002	0.000					
Melt Inclusion																
PI19-MI					15.3		2.3	0.32		0.05	0.005			0.001	Fe ₂ SiO ₄ -Fe ₃ O ₄ - SiO ₂ (FMQ)	
PI21-MI				10.6		1.6	0.24		0.04	0.004	0.001					
PI22-MI				16.4		2.5	0.26		0.04	0.006	0.001					

Note: n, number of analysed plagioclases; s.d., 1 sigma standard deviation; Log *f*O₂(buffer) corrected in the Supplementary Information; Melt inclusion data not used in this study because we do not know exact T, P, and whether there was any water loss from the inclusions after formation; The latest infrared absorption coefficient (Mosenfelder *et al.*, 2015) was used for calibrating water contents of all plagioclases.



The Effects of Oxygen Fugacity and Water Content in Melt

It has been shown that a number of parameters can affect water partitioning between nominally anhydrous minerals and silicate melts, including the presence and abundance of chemical impurities and vacancies, the possibility of substitutions with charge-balancing species, temperature, pressure, and oxygen fugacity (e.g., Yang, 2012 and references therein). Previous work on water solubility in feldspar has shown that there is no obvious compositional dependence on the incorporation of H in the feldspar group except for a possible link with potassium content or sodium-hydrogen diffusion during heating (Yang, 2012; Johnson and Rossman, 2013). Potassium, however, has very low concentrations in ferroan anorthositic plagioclase, <0.03 wt. % (Dixon and Papike, 1975) and is absent in our experiments. Pressure and temperature effects cannot be assessed on the basis of our experiments, and those of Hamada *et al.* (2013) and Caseres *et al.* (2017), due to the overall limited pressure (0.3–0.8 GPa) and temperature (1000–1230 °C) range.

The H solubility in Fe-poor plagioclase near the IW buffer was demonstrated to be more than twice that determined at more oxidising conditions (Yang, 2012), leading to the hypothesis that oxygen fugacity could cause the difference between data obtained at lunar conditions, including our data and the data of Caseres *et al.* (2017), and those obtained at terrestrial conditions, *i.e.* the data of Hamada *et al.* (2013). However, Figure 3a shows that there is no correlation between $D_{\text{water}}^{\text{plag-melt}}$ and fO_2 in the overall data set. The terrestrial data set at relatively oxidising conditions yields lower $D_{\text{water}}^{\text{plag-melt}}$ than the lunar data sets obtained at lower fO_2 , similar to the trend between oxygen fugacity and hydrogen solubility in plagioclase (Yang, 2012). One possibility is that the number of vacancies available for hydrogen incorporation is increased at low fO_2 , for example due to the enhanced incorporation of divalent iron in Al sites (Mosenfelder *et al.*, 2019). However, although high values are found at low fO_2 , some low- fO_2 experiments

show low $D_{\text{water}}^{\text{plag-melt}}$ (Fig. 3a). Oxygen fugacity is therefore not the main factor affecting $D_{\text{water}}^{\text{plag-melt}}$. Instead, the experiments suggest that the water content of the silicate melt plays a key role in determining the partition coefficient of water between plagioclase and melt (Fig. 3b). There is an inverse relationship between the measured $D_{\text{water}}^{\text{plag-melt}}$ and the water concentration in silicate melt. In the absence of a theoretical framework to guide the functional form used to describe these inverse relations, we provide the following best-fit equations:

$$D_{\text{water}}^{\text{plag-melt}} = -6 \cdot 10^{-2} \cdot x + 0.05 \quad (x \leq 0.7; R^2 = 0.85), \quad \text{Eq. 1a}$$

$$D_{\text{water}}^{\text{plag-melt}} = -5 \cdot 10^{-4} \cdot x + 0.005 \quad (x > 0.7; R^2 = 0.60), \quad \text{Eq. 1b}$$

where x is wt. % H_2O in the silicate melt. Equations 1a and 1b provide quantitative estimates of $D_{\text{water}}^{\text{plag-melt}}$ given ranges of the magma oxygen fugacity and water content in silicate melt.

The exact mechanism controlling the observed variations of $D_{\text{water}}^{\text{plag-melt}}$ with fO_2 and water content cannot be derived from our experiments, and it is not feasible to construct a thermodynamics-based predictive model of $D_{\text{water}}^{\text{plag-melt}}$ with the currently available data sets. Clearly water partitioning shows strong non-Henrian behaviour in our experiments, pointing to non-ideal behaviour of the relevant hydrogen-bearing species in mineral and/or melt. Previous work has suggested that changes in the OH site in plagioclases occur as a function of plagioclase OH content (Hamada *et al.*, 2013), but we cannot identify variations in the shape of the FTIR spectra that would be consistent with such a change in our experiments. It therefore seems more likely that the non-Henrian behaviour is caused by water activity coefficient changes in the melt. The dominant hydrogen-bearing species in hydrous melts at the hydrogen levels in our experiments could be OH (Stolper, 1982; Newcombe *et al.*, 2017), but non-linear increases in the H_2O/OH ratio with increasing silicate melt hydrogen content have previously been observed (Stolper, 1982). These speciation changes affect the OH activity in the silicate melts, consistent with the observed trend in $D_{\text{water}}^{\text{plag-melt}}$ values, though future work is needed to quantify this correlation.

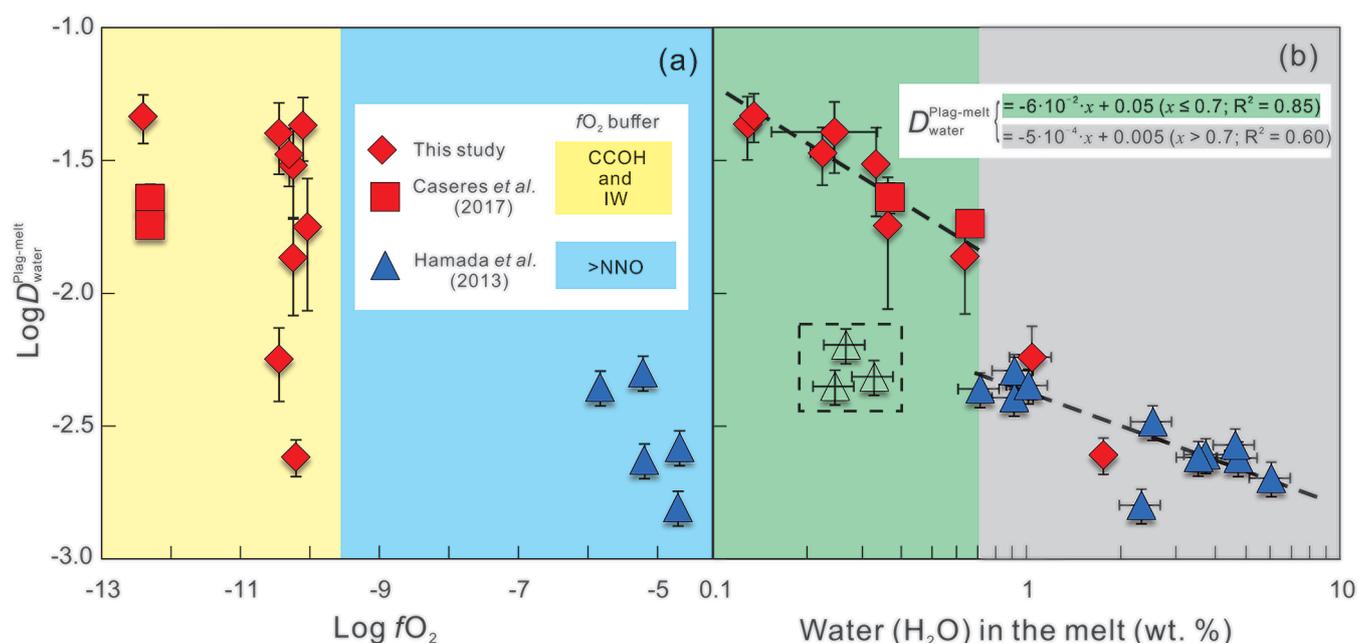


Figure 3 Partition coefficients of water between plagioclase and melt from this study and literature data (Hamada *et al.*, 2013; Caseres *et al.*, 2017), plotted versus (a) oxygen fugacity, and (b) water concentration in silicate melt. Melt inclusion data (Hamada *et al.*, 2013) in the dotted box are not used in this study because the formation temperature of these inclusions and the degree of water loss from inclusions after formation are uncertain.

Water Content of the Lunar Magma Ocean

Equation 1a can be used to calculate the water content of the LMO at lunar oxygen fugacity conditions. This calculation requires estimates of (a) the abundance of water in lunar plagioclase, and (b) the degree of crystallisation of the LMO at the time of plagioclase formation.

The latest study published to date on the water content of lunar plagioclase from a primary crystallisation product of the LMO reported ~5 µg/g water (H₂O) in ferroan anorthosite samples including Apollo sample 60015 (Hui *et al.*, 2017). We constrained the degree of crystallisation of the LMO when this plagioclase was formed by comparing the Mg# (molar (MgO/MgO + FeO) × 100) of plagioclase from sample 60015 (Mg# of 17–47) (Dixon and Papike, 1975) to the Mg# of plagioclase formed at different stages from our recent experimental study of LMO crystallisation (Lin *et al.*, 2017a,b). The first plagioclase formed during LMO crystallisation has a Mg# of ~60. This Mg# decreases with progressive crystallisation. Plagioclase with Mg# as low as 17–47 forms after ~95 % crystallisation of the LMO.

Based on the above constraints, the amount of H₂O equivalent in the residual LMO after ~95 % solidification of the initial magma ocean is calculated to be ~100 µg/g by solving the equation $D_{\text{water}}^{\text{plag-melt}} = 5/C_{\text{water}}^{\text{melt}} = -6 \cdot 10^{-2} \cdot (C_{\text{water}}^{\text{melt}}/10000) + 0.05$ (here, $C_{\text{water}}^{\text{melt}}$ is in µg/g). If the LMO hydrogen budget remained constant throughout LMO solidification, this implies a very low initial LMO water content of just 5 µg/g H₂O equivalent, consistent with inferences from petrology and magma ocean modelling (Elkins-Tanton and Grove, 2011), lunar sample measurements (McCubbin *et al.*, 2015b), and the experimental LMO solidification studies of Rapp and Draper (2018) and Charlier *et al.* (2018). In contrast, if the initial LMO contained >500–1800 µg/g water as suggested by the experimental LMO solidification study of Lin *et al.* (2017a) the minimum amount of water in the residual LMO at the time of lunar plagioclase formation would have exceeded 1 wt. %, far exceeding the ~100 µg/g estimated using the plagioclase hygrometer in this study. In this case, the early Moon experienced extensive degassing, with >99 % of the initial LMO water budget lost during LMO crystallisation. Such a high degree of degassing is consistent with observations based on the isotopic compositions of hydrogen of lunar plagioclase (Hui *et al.*, 2017) and of chlorine in lunar apatites (Sharp *et al.*, 2010) if degassing of Cl occurred primarily through metal chlorides (*e.g.*, Schaefer and Fegley, 2004; Sarafian *et al.*, 2017), and would point to a highly dynamic volatile cycle during early lunar evolution.

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Author Contributions

YHL, HH, and WvW. designed this project. YHL. performed the experiments. YJX, YHL, and HH performed the FTIR analyses. YHL wrote the paper with input from all co-authors.

Additional Information

Supplementary Information accompanies this letter at <http://www.geochemicalperspectivesletters.org/article1908>.



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