

Partially-Saturated Brines Within Basal Ice or Sediments can Explain the Bright Basal Reflections in the South Polar Layered Deposits

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Submitted to: JGR-Planets

Initially Submitted: May 26, 2022

Key points:

- Brines within ice and icy sediments can explain the bright basal reflection in the south polar layered deposits
- Salt enhancements of 5–16 times the salt-regolith concentration at the Phoenix landing site is needed in brine-sediment mixtures
- Calcium perchlorate brines are stable above their eutectic temperature, which is experimentally measured at 197.3 ± 0.2 K

Abstract

Strong radar reflections have been previously mapped at the base of the Martian South Polar Layered Deposits (SPLD). Here, we analyze laboratory measurements of dry and briny samples to determine the cause of this radar return. We find that liquid vein networks consisting of brines at the grain boundaries of ice crystals can greatly enhance the electrical conductivity, thereby causing strong radar reflection. A liquid brine concentration of 2.7–6.0 vol% in ice is sufficient to match the electrical properties of the basal reflection as observed by MARSIS. When brine is mixed with sediments, we find that the brine-ice mixture in the pores must be 2–5 times more concentrated in salt, increasing the brine concentration to 6.3–29 vol%. Thus, our best fit of the median observed MARSIS value suggests a salt-bulk sample concentration of ~6 wt%, which is ~8 larger than that of the Phoenix landing site. To form brine, the basal reflector must reach a temperature greater than the eutectic temperature of calcium perchlorate of 197.3 ± 0.2 K. Colder metastable brines are possible, but it is unclear if brines can remain metastable for millions of years. Additionally, grey hematite with a concentration of 33.2–59.0 vol% possess electrical properties that could cause the observed radar returns. However, such concentrations are 2–3 times larger than anywhere currently mapped on Mars. We also demonstrate that brines mixed with high-surface-area sediments, or dry red hematite, jarosite, and ilmenite cannot create the observed radar returns at low temperatures.

Plain-Speak Abstract

Previously research has shown that strong radar reflections emanate from the interface between Mars' southern ice cap and their underlying sediments over a region with an area of 20×30 km and 1.5 kms beneath the surface. Radar reflections are caused by changes in electrical properties. Here, we analyze electrical property laboratory measurements of materials under Mars-like conditions. We find that a small amount (~3–6%) of brine in ice samples could create strong radar reflections similar to those that are observed. A greater concentration of salt is needed in sediment ice mixtures, such that a salt enhancement of ~10 would be needed compared to the rest of Mars. Additionally, a calcium perchlorate brine could only form if the temperature was greater than 197.3 K. Colder metastable brines are possible, but it is unclear if brines can remain metastable for millions of years. Additionally, dry grey hematite could also cause the observed radar returns, but the concentration of grey hematite would have to be 2–3 times larger than anywhere else on Mars.

58 We can also rule out any brines mixed with clays, or dry red hematite, jarosite, and ilmenite as
59 they cannot create the observed radar returns at low temperature.
60

1. Introduction

The Mars Advanced Radar for Subsurface and Ionospheric Sounding (MARSIS) instrument on Mars Express has detected strong subsurface radar reflections in the region of Ultimi Scopuli (81°S, 193°E), within the South Polar Layered Deposits (SPLD) (Orosei *et al.*, 2018; Lauro *et al.*, 2021). These subsurface reflections are ~10 dB greater in power than the surrounding reflections and ~3 dB greater than the reflections from the surface (Orosei *et al.*, 2018). The reflecting unit is located at the base of the SPLD, approximately 1.5 km below the topographic surface. Because data acquired by MARSIS do not separate the real (ϵ') and imaginary (ϵ'') parts of the complex permittivity of reflectors, the apparent permittivity (ϵ_a), a single parameter accounting for both ϵ' and ϵ'' (Mattei *et al.*, 2022a; ref. to §2.2) is commonly reported. In their investigation of the SPLD, Orosei *et al.* (2018) obtained reflectivities characterized by a median ϵ_a value of 33 ± 1 at 4 MHz. Using a threshold value of $\epsilon_a = 15$, the largest reflective zone is ~20×30 km (Lauro *et al.*, 2021). Furthermore, Lauro *et al.* (2021) also detected at least three other smaller locations, about 10 km across, within 120 km of the largest anomaly.

The high reflectivity values observed by MARSIS (Orosei *et al.*, 2018) were found to have a 1st and 3rd quartile values ϵ_a of 16 and 91, respectfully. Such high values of ϵ_a significantly limit the number materials that could cause such reflections. Three categories of materials have thus far been proposed to explain the high reflectivity values: clays with adsorbed water (Smith *et al.*, 2021); dry minerals and rocks with high iron content (Bierson *et al.*, 2021; Grima *et al.*, 2022); and saline ice (Bierson *et al.*, 2021).

Smith *et al.* (2021) suggested that the dielectric relaxation of clay with adsorbed water at the base of the SPLD could result in high ϵ_a values at the presumed basal temperature in Ultimi Scopuli. However, Smith *et al.*'s (2021) data were measured at 230 K and are inconsistent with similar types of measurements as reported in the literature (Moore and Maeno, 1993; Stillman *et al.*, 2010; Stillman and Grimm, 2011b; Lorek and Wagner, 2013; Cunje *et al.*, 2018; Kulacz and Orzechowski, 2019; Mattei *et al.*, 2022a).

Bierson *et al.* (2021) suggested that red hematite is a possible source for the bright reflection, because it has an $\epsilon' = 18.1$ (Robinson and Friedman, 2003). In fact, Robinson and Friedman (2003) found that a soil sample of red hematite with 50.5% porosity, likely a reasonable analog for Martian soils, has a bulk $\epsilon' = 4.5$, which is consistent with measurements reported by Stillman and

Olhoeft (2008). The $\varepsilon' = 18.1$ used by *Bierson et al.* (2021) was the estimate of the grain or solid permittivity, which assumes no porosity. Assuming minimal porosity of 10% for a hematite-rich rock, using *Robinson and Friedman's* (2003) mixing models we estimate a bulk ε' value of 10.8, which is lower than the observed 1st quartile value. As an alternative, the same authors also suggested jarosite as a possible material, based on its proposed high value of ε' . This is however inconsistent with dry soil measurements of permittivity for this mineral, as published in the literature (ref. to figure 1 in *Stillman and Olhoeft*, 2008).

Grima et al. (2022) suggested that measurements of bulk $\varepsilon' > 16$ have been reported for ilmenite (FeTiO_4) and basalts (*Parkamento*, 1967; *Shmulevich et al.*, 1971; *Rust et al.*, 1999). We addressed this specific interpretation later on in this paper, where we present and discuss the results of our measurements on dry ilmenite.

Bierson et al. (2021) suggested that the conductivity of saline ice, i.e., ice with a bulk salt mass concentration range of 0.1–3.5 wt%, could return ε_a values observed at Ultimi Scopuli. Liquid brines have high ε' (~80) and are highly conductive; thus, brines in ice or sediments can plausibly create ε_a values within range of those observed by MARSIS (*Mattei et al.*, 2022a).

Here, we report on our experimental investigation of the compositional and thermal range of stability of Martian-consistent brines, which is the principal purpose of this study. We also address the dry material interpretation through our laboratory experiments on selected dry materials. The paper is structured as follows: (i) we briefly provide a background into the MARSIS instrument, electrical properties of materials, formation of brines, and estimated basal temperatures in Ultimi Scopuli (§2); (ii) we then present our measurement methodology (§3); (iii) we follow with a discussion of our results (§4); (iv) we then proceed to interpret our results in the context of published hypotheses on the nature of the reflectors (§5); and (v) our principal findings are summarized in the conclusions (§6).

2. Background

2.1 MARSIS

MARSIS acquires data on four channels, with center frequencies of 1.8, 3, 4, and 5 MHz, each with a bandwidth of 1 MHz (e.g., *Picardi et al.*, 2005). The MARSIS dataset used by *Orosei et al.* (2018) and *Lauro et al.* (2021) was acquired without onboard synthetic-aperture radar processing to ensure the amplitudes were as accurate as possible (*Lauro et al.*, 2021). These MARSIS results

rely on the 4 MHz center frequency data as it is the most complete data set around the Ultimi Scopuli region (Lauro *et al.*, 2021). The radius of the first Fresnel zone is ~3.5-6 km depending on satellite altitude (300-800 km) (Orosei *et al.*, 2018), while the more conservative pulse limited criterion, has a diameter of 7.5 km assuming a satellite altitude of 400 km (Lauro *et al.*, 2021). Thus, the anomalous areas are larger than the pulse limited criterion. Lastly, the vertical resolution is ~55 m, after range compression and Hanning windowing when assuming an $\varepsilon' = 3.1$ of pure ice (Lauro *et al.*, 2021).

2.2 Electrical Properties

The electrical properties of the material control the speed at which radar waves propagate, the amount of energy that is attenuated, and how much energy is reflected at discontinuities. Here and in the following discussion, we ignore the effects of the magnetic permeability, given its small variability (Stillman and Olhoeft, 2008). The real part of the relative dielectric permittivity (ε') primarily controls the speed at which radar waves propagate through the material. In most dry rocks at radar frequencies, ε' can be estimated using the following equation:

$$\varepsilon' = (1.93 \pm 0.17)\rho, \quad (\text{Eq. 1})$$

where ρ is the bulk density of the rock or soil in g cm^{-3} (Olhoeft and Strangway, 1975). The imaginary part of the relative dielectric permittivity (ε'') primarily controls the attenuation of radar waves through the material and is a summation of losses due to dielectric relaxations (ε''_p) and Direct Current (DC) electrical conductivity (σ_{DC}),

$$\varepsilon'' = \varepsilon''_p + \frac{\sigma_{DC}}{2\pi f \varepsilon_0}, \quad (\text{Eq. 2})$$

where f is the frequency of the radar energy and ε_0 is the dielectric permittivity in vacuum ($8.854 \times 10^{-12} \text{ F m}^{-1}$).

Next, we convert ε' and ε'' into ε_a (Mattei *et al.*, 2022a) using

$$\varepsilon_a = \varepsilon_1 \frac{\varepsilon_1 + |\varepsilon_2| + \sqrt{\varepsilon_1^2 + |\varepsilon_2|^2 - 2\varepsilon_1\varepsilon_2'}}{\varepsilon_1 + |\varepsilon_2| - \sqrt{\varepsilon_1^2 + |\varepsilon_2|^2 - 2\varepsilon_1\varepsilon_2'}}, \quad (\text{Eq. 3})$$

where ε_1 is the ε' of the SPLD assumed to be 3.5, ε'' of the SPLD is assumed to be zero, and magnitude of the permittivity of the basal reflector is

$$|\varepsilon_2| = \sqrt{\varepsilon_2'^2 + \varepsilon_2''^2}. \quad (\text{Eq. 4})$$

In **Figure 1**, the combination of the reflector's ε'_2 and ε''_2 that would create ε_a similar to that observed in the MARSIS data at 4 MHz (*Lauro et al.*, 2021) is shown: minimum values of ε'_2 and ε''_2 of 13.5 and 6, respectively, could allow for an $\varepsilon_a \geq 16$, i.e., the observed 1st quartile value. If both the real and imaginary part values of a material are less than these minimum values, then ε_a cannot reach the MARSIS threshold values observed.

Electrical properties are frequency dependent because different polarization mechanisms separate charges at different speeds (e.g., *Stillman and Olhoeft*, 2008; *Stillman et al.*, 2010). To model the material's frequency dependence, we use the Cole-Cole equation (*Cole and Cole*, 1941, *Stillman and Olhoeft*, 2008). Frequency dependent, materials are also temperature dependent, because the speed at which charge separates is temperature dependent: this behavior is modeled with a Boltzmann temperature dependence (e.g., *Stillman and Olhoeft*, 2008, and refs. therein). These models allow us to extrapolate and interpolate electrical properties in frequency and temperature. Thus, ε' and ε'' measured to 1 MHz can be extrapolated to comprise the MARSIS frequency range of 2 – 5 MHz (e.g. *Stillman and Grimm*, 2011a). Additionally, as the frequency dependence shifts to lower frequency at colder temperatures, we can test whether the extrapolation is valid (*Stillman and Grimm*, 2011a).

2.3 Formation of Brines

Salt-H₂O mixtures are characterized by four distinct phases (**Fig 2A**):

- (1) Brine: when the temperature of the mixture is above the melting temperature;
- (2) Brine + ice: when the temperature of a sub-eutectic concentration (<49.8 wt% for Ca(ClO₄)₂) is below the melting temperature, but above the eutectic temperature;
- (3) Brine + hydrate: when the temperature of a super-eutectic concentration is below the melting temperature, but above the eutectic temperature; and
- (4) Ice + hydrate: when the temperature of a mixture is below the eutectic temperature.

Here, we focus on the brine + ice phase for sub-eutectic concentrations. As a sub-eutectic concentration (<49.8 wt% for Ca(ClO₄)₂) sample begins to freeze below the melting point, H₂O is fractionated out of the system as pure ice (or nearly pure ice if Cl⁻, F⁻, or NH₄⁺; for more see *Gross et al.*, 1977; *Petrenko and Whitworth*, 1999; *Stillman et al.*, 2013ab) and the salt-H₂O mass concentration increases in the liquid brine until it reaches a eutectic concentration at the eutectic temperature. The amount of liquid brine present at the eutectic depends on its initial concentration

and salt type (**Fig. 2**). For very low salt concentrations in the mixture, the melt accumulates at the boundaries of the ice grains. Even low salinity brines (e.g., ~3 mM, ~0.03 mass%, or ~0.06 vol%; *Grimm et al.*, 2008) are electrically conductive, they form electrically connected networks across the ice matrix, defined as along Liquid Vein Networks (LVNs). Thus, salt-H₂O mixtures display high electrical conductivity even when most of the ice is frozen (*Grimm et al.*, 2008; *Stillman et al.*, 2010). The electrical conductivity of the mixture drops significantly below the eutectic temperature, when the entire system is frozen.

2.4 Estimated Basal SPLD Temperature

Estimates of the values of SPLD basal temperatures in Ultimi Scopuli have been recently published (*Sori and Bramson*, 2019; *Ojha et al.*, 2019; *Egea-González et al.*, 2022), and are heavily dependent upon the heat flow from the planetary interior, and the thermal conductivity of the SPLD, both of which are not fully constrained. Assuming a temperature-dependent thermal conductivity of pure H₂O ice for the SPLD, a heat flow >84 mW/m² is required to allow liquid Ca-perchlorate brine to form. For a more realistic heat flow range of 14-25 mW m⁻² (*Parro et al.*, 2017), a basal temperature of ~171-176 K is obtained (*Sori and Bramson*, 2019). *Egea-González et al.* (2022) found a basal temperature range of 175-187 K using the maximum surface heat flow (32 mW m⁻²) by assuming a dust proportion of 15%. We speculate that inclusion of CO₂ ice into H₂O ice layers or as pure CO₂ layers within the SPLD could further enhance basal temperatures (*Wieczorek*, 2008), as CO₂ ice has a thermal conductivity that is 5-6 times lower than that of H₂O ice (e.g., *Mellon*, 1996).

3. Methods

This section describes the methodology of electrical property measurements of salt-H₂O mixtures and salt-H₂O mixtures with Martian analogs. Note, new measurements are complemented by data obtained in previous published (*Grimm et al.*, 2008; *Stillman et al.*, 2010; *Stillman and Grimm*, 2011ab) and unpublished work. Low-frequency measurements were conducted at Southwest Research Institute in Boulder Colorado. Solutions of salt-H₂O mixtures were freshly prepared before each measurement. For measurements of salt-H₂O mixtures, the liquid solution was poured into the three-electrode sample holder with a Teflon cup (Solartron 12962A and 12964A). For measurements of salt-H₂O mixtures with Martian analogs, the analogs (e.g., clays,

sands, ilmenite) were dried in a vacuum at ~ 1 mbar and ~ 383 K until their mass changed by less than 0.5% over a ~ 12 -hour period. The granular analog material was then spooned into the Teflon cup sample holder with a spatula, then the liquid solution was poured into the granular material. Lastly, the sample was intimately mixed with a spatula to ensure a homogenous sample was created. The sample was 3.5 cm in diameter and ~ 0.6 cm thick. The sample holder was then placed into the sample chamber.

The sample chamber has been previously described (*Grimm et al.*, 2008; *Stillman et al.*, 2010). For measurements made after Sept 2019, we used a custom vacuum chamber that is placed in the Ultra-Low freezer (So-Low C85-9). Only dry materials were measured in vacuum of ~ 1 mbar, samples with ice were measured at atmospheric pressure. The sample holder sits on a liquid nitrogen cold plate so that temperatures below 180 K can be obtained. Lake Shore Cryotronics Silicon diode (DT-670) temperature sensors are used to measure the sample temperature on the Teflon cup and a second sensor controls two 25 W cartridge heaters (Lake Shore HTR-25-100) via a Lake Shore Cryotronics temperature controller (331). The sample is then connected via BNC cables to a Solartron 1260A impedance analyzer and a Solartron 1296A dielectric interface. The sample temperature was then lowered to reach a temperature that is 2 K below its freezing point for 1 hour to initialize crystallization. Subsequently, the temperature is further decreased to 10 K below the freezing point for an additional 2 hours to ensure crystallization of any metastable water. Reheating of the sample is carried out by raising the temperature back to 2 K below the freezing point for 3 hours. This process guarantees formation of stable LVNs for the entire duration of the experiment, reducing the rate of cracking in the sample, and avoiding the effect of rapid recrystallization. The temperature of the sample is then drastically lowered at a rate that is lower or equal to <1 K min^{-1} to the sample's lowest temperature. Electrical property measurements are conducted non-stop as soon as the sample obtains properties that allow it to be measured easily, typically this occurs when $\sigma_{DC} < 0.1$ S/m. After obtaining the lowest temperature value the sample is slowly heated. To increase temperature accuracy, the target temperature is held to within 0.2 K for at least 20 min. Typically, measurements are made over the range of $1\text{--}10^6$ Hz, which takes ~ 6 minutes to acquire. However, as σ_{DC} increases the lower frequency limit is also increased, which reduces the time required to measure a spectrum. Additionally, once the sample reaches the frequency at which σ_{DC} dominates the electrical properties the uncertainty of ϵ' increases greatly.

The electrical property measurements output complex impedance as a function of frequency, which is converted into complex dielectric permittivity using the electrode geometry. Cole-Cole parameters of each spectrum were modeled by nonlinear curve fitting (Stillman and Olhoeft, 2008; Grimm et al., 2008; Stillman and Grimm, 2011ab). The Cole-Cole parameters were then used to find ϵ_a at 4 MHz as a function of temperature.

4. Results and Interpretations

4.1 Dry Ilmenite

Dry granular ilmenite (purchased from Ward's Scientific) with 35.5% porosity and a bulk density of 3.07 g cm^{-3} possesses a significant σ_{DC} and a polarization mechanism that increases ϵ' and ϵ'' at MARSIS frequencies (Fig. 3). The observed relaxation mechanism is known as an anomalous low-frequency dispersion (e.g., Jonscher 1978; 1999; Shahihi et al., 1975; Stillman et al., 2010). Previously reported data indicate that ilmenite reaches a value of $\epsilon' > 33$ at room temperature (Parkmenko, 1967). In our experiments, at $\sim 273 \text{ K}$, the ϵ_a of ilmenite does not approach the observed median, dropping below the observed 1st quartile value at a temperature of $\sim 252.8 \text{ K}$. Therefore, we conclude that ilmenite is not a plausible candidate as the cause of the strong basal reflections at Ultimi Scopuli.

4.2 Chloride Salt- H_2O Mixtures

Chloride brines in ice were previously shown to obtain high values of σ_{DC} and ϵ'' at temperatures above their eutectic temperatures (Grimm et al., 2008). However, NaCl (Grimm et al., 2008) and MgCl_2 (Primm et al., 2020) have eutectic temperatures of 251 and 240 K, respectively; thus, they are too high to be considered in the context of the SPLD. CaCl_2 has a much lower eutectic temperature of 223 K (Stillman et al., 2010). The ϵ_a data versus temperature and concentration show that a 100 mM (1.1 wt%) of CaCl_2 at the eutectic temperature has an ϵ_a equivalent to the observed 1st quartile value (Fig. 4). The ϵ_a of the 1,000 mM (10 wt%) of CaCl_2 is larger than the observed 3rd quartile value (Fig. 4), indicating a lower concentration is need to match the 3rd quartile value.

4.3 Perchlorate Salt-H₂O Mixtures

Because of their low eutectic temperatures of 198 and 216 K (Toner *et al.*, 2014) respectively, calcium and magnesium perchlorate brines have been proposed as most plausible source for the bright basal reflections in Ultimi Scopuli (Lauro *et al.*, 2021). The ϵ_a versus temperature and concentration show that a concentration of 100 mM (2.2 wt%) of Mg(ClO₄)₂ is just below the observed 1st quartile value (Fig. 5), while 500 mM (10 wt%) of Mg(ClO₄)₂ is greater than the observed 3rd quartile value (Fig. 5).

For the 300 mM (6.9 wt%) of Ca(ClO₄)₂, the selected measurements of ϵ' (Fig. 6A) and ϵ'' (Fig. 6B) display a strong ice relaxation below the eutectic temperature. Above the eutectic temperature, σ_{DC} increases to large values (Fig. 6C) so that ϵ' can no longer be measured accurately due to so much energy being dissipated by ϵ'' . The observed dependence of ϵ_a with temperature demonstrates that concentrations of 300 mM (6.9 wt%) of Ca(ClO₄)₂ are sufficient to obtain the observed 3rd quartile value at the measured eutectic temperature of 197.3±0.2 K (Fig. 7). The brine in the 200 mM (4.7 wt%) of Ca(ClO₄)₂ never frozen into hydrate and ice even though the temperature was dropped to 161.9 K. As will be shown in the next section and Figure 8, ϵ_a has the same value at a given temperature when above the eutectic temperature no matter whether the temperature is warming or cooling. Thus, ϵ_a at the eutectic temperature for the 200 mM Ca(ClO₄)₂ sample shows it is near the median observed value, making this a likely material for the base of the SPLD in Ultimi Scopuli. Meanwhile, the 100 mM (2.4 wt%) Ca(ClO₄)₂ sample shows it is below the 3rd quartile value.

4.4 Metastability of Salts-H₂O Mixtures

The results of the experiments conducted to explore the metastability of perchlorate brines are shown in Figure 8. For the 300 mM (6.7 wt%) Ca(ClO₄)₂, the brine remained in a metastable form down to a temperature of 187.9±0.2 K, i.e., 9.4 K below the eutectic temperature, although the measured value of ϵ_a is below the observed 1st quartile. The decreasing value of ϵ_a with decreasing temperature is attributed to a decreasing volume of the liquid brine combined. Thus we conclude that, to record values of apparent permittivity consistent with the bright MARSIS reflections, metastable brines must form from concentrations above that in this experiment. We were able to hold the brine sample in a metastable form for 6.9 hours before decreasing the temperature to a lower temperature. Metastability of other salts has been previously reported to last at least 72 hours

when held at a temperature a few degrees below the eutectic temperature (Primm *et al.*, 2020). Other $\text{Ca}(\text{ClO}_4)_2$ samples displayed in **Figures 7 & 9** never froze even though they were taken to temperatures below 173 K.

4.5 Salt- H_2O Mixtures in Low-Surface Area Sediments

Previous measurements (Stillman *et al.*, 2010) have shown that brines in low-specific-surface ($<1 \text{ m}^2/\text{g}$) area sediments also show distinct increases of σ_{DC} at the eutectic temperature that indicate that LVNs still dominate conduction in icy sediment mixtures. Our experiments were conducted with fine-grained (mean grain size of $110 \text{ }\mu\text{m}$) sand with a porosity of $\sim 40\%$. We focused on $\text{Ca}(\text{ClO}_4)_2$ salt- H_2O mixture with sand. We found that a 37.8 vol% of 700 mM (15.1 wt%) $\text{Ca}(\text{ClO}_4)_2$ mixed with sand is near the observed ε_a values for the 1st quartile (**Fig. 9**). Similarly, a 40.4 vol% of 1.5 mM (29.1 wt%) $\text{Ca}(\text{ClO}_4)_2$ mixed with sand is near the observed ε_a values for the median observed values (**Fig. 9**), making this a likely material for the base of the SPLD in Ultimi Scopuli.

4.6 Salt- H_2O Mixtures in High-Surface Area Sediments

Clay samples from the Clay Mineral Society were previously measured at Mars-like temperatures (Stillman *et al.*, 2010; Stillman and Grimm, 2011ab). A compilation of multiple samples of STx-1 (Texas Calcium Montmorillonite) measured dry and with slight additions of water at a temperature of $193.2 \pm 0.2 \text{ K}$ are shown in **Figure 10**. STx-1 possesses a large H_2O surface area of $217 \text{ m}^2/\text{g}$ (Jänchen *et al.*, 2009), where 3 and 7 monolayers (ML) represent ~ 14 and ~ 32 wt% water, respectively. (Note a ML of water was assumed to be 0.3 nm thick.) None of the data plotted approach the ε_a threshold for the 1st quartile observations. Furthermore, the full temperature dataset for 7 ML of 100 mM CaCl_2 indicates that, once the temperature drops below 233 K, the electrical properties are not compatible with the 1st quartile MARSIS observations (**Fig. 11**).

For this work, we measured clay specimen SAz-1 (“Cheto” from Arizona with an N_2 surface area of $97.4 \text{ m}^2/\text{g}$) saturated with 54.2 wt% of 500 mM $\text{Ca}(\text{ClO}_4)_2$ brine (**Fig. 12**). While the sample displays σ_{DC} and strong polarization mechanisms, these are not of sufficient magnitude to approach the range of ε_a values observed by MARSIS. Thus, our new experiment corroborates the conclusion that salt- H_2O mixtures in high-surface area sediments do have increased values of σ_{DC}

above the eutectic temperature (*Stillman et al.*, 2010; *Mattei et al.*, 2022ab). While absorbed/bound water does conduct electrical current below and above the eutectic temperature, the ions in adsorbed/bound water are less mobile and follow a much more tortuous path compared to brines in sand or ice (*Stillman et al.*, 2010; 2019).

JSC Mars-1, which consists of hematite and titano-magnetite, has been measured by many researchers (e.g., *Williams and Greeley*, 2004; *Stillman and Olhoeft*, 2008). When measured dry, it does not approach the 1st quartile values. JSC Mars-1 has a large H₂O surface area (106 m²/g; *Pommerol et al.*, 2009) due to a palagonitic surface. However, even with 7 ML (~18 wt%) it still does not obtain large electrical property values approaching the range of ϵ_a values of that observed by MARSIS until temperatures larger than 273 K (**Fig. 13**). In conclusion, salt-H₂O mixtures in high-surface area (>1 m²/g) sediments do not possess a large enough σ_{DC} to provide an ϵ_a value at SPLD-like temperatures that is similar to the bright reflector measured by MARSIS.

5. Discussion

5.1 Dry Minerals

A discussion on red hematite and jarosite is not necessary as previous measurements in the literature can eliminate these minerals, as was discussed in §2.5. While dry ilmenite can have very high ϵ_a values at room temperature (*Parkmenko*, 1967; *Boivin et al.*, under review), temperature dependence significantly lowers ϵ_a values (**Fig. 3**). Thus, ilmenite has an ϵ_a value near ~12 that is lower than the observed 1st quartile value at the approximate temperature of the base of the SPLD.

Intrusive and extrusive igneous rocks were measured by *Shmulevich et al.* (1971) of which 12 of 89 had $\epsilon' > 13.5$ at 500 MHz, which could have an $\epsilon_a > 16$ depending on their ϵ'' , which were not published. There is little data regarding the methodology of the *Shmulevich et al.* (1971) measurements. Leaving many important questions: what temperature were the samples measured at, where the samples were acquired, and were the samples vacuum or thermally dried or measured as collected? Additionally, frequency and temperature dependence are also omitted. Thus, it is impossible to extrapolate these measurements to SPLD temperatures and MARSIS frequencies given the lack of details given in *Shmulevich et al.* (1971).

Many other measurements (*Campbell and Ulrichs*, 1969; *Chung et al.*, 1970; *Gold et al.*, 1970; *Adams et al.*, 1996; *Russell and Stasiuk*, 1997) of volcanic rocks all measured $\epsilon' < 12$ at radar frequencies. While *Rust et al.* (1999) found two of 34 basaltic samples, with an $\epsilon' > 13.5$ at 4 MHz

and at room temperature. Only the MB1 sample has an $\varepsilon_a > 16$ at 4 MHz at room temperature (**Fig. S1**). *Rust et al.* (1999) does comments that this sample hosts thin cracks, thus a layer of absorbed water could be increasing ε' as has been measured by many other (e.g., *Jonscher* 1978; 1999; *Shahidi et al.*, 1975; *Knight and Endres*, 1990; *Stillman et al.*, 2010). Thus, we are skeptical that this single basalt measurement could produce an ε_a value due to adsorbed/bound water that is larger than clay or JSC Mars-1.

Grey hematite possesses a radio-frequency relaxation with a small activation energy of 0.1 eV (*Stillman and Olhoeft*, 2008). A dry sample of 59.0 vol% grey hematite with a porosity of 41.0% can obtain the median MARSIS ε_a value at 4 MHz over possible SPLD-like temperatures (**Fig. 14**). This is a significant amount of grey hematite and about four times greater than the maximum grey hematite value measured at the surface of 15% at Aram Chaos and Meridiani Planum (*Glotch and Christensen*, 2005). Additionally, the grey hematite in Meridiani Planum is a lag deposit and thus concentrated compared to its *in-situ* formation concentration (*Hynek*, 2004). This is likely why no large increases in surface reflectivity have been measured at Aram Chaos or Meridiani Planum even though they have a high surficial concentration of grey hematite.

To further explore the possibilities of grey hematite, we perform dielectric mixing models. Using a Lickenecker power law mixing formula (*Stillman and Olhoeft*, 2008), we estimate the solid grain ε_a of grey hematite to be 137.6 at 4 MHz at 200 K, assuming a grain density δ_G of 5.26 g cm⁻³. We then used *Eq. 1* to estimate an ε_a value of ultramafic and mafic grain assuming a δ_G of 3.8 and 3.0 g cm⁻³, respectively. We assume that any porosity is filled with ice (no brine) with ε_a of 3.15 as ground ice is stable below the SPLD. We then created four mixing models with grey hematite (**Fig. 15; Table 1**) using a power-law mixing model with an exponent of 2.65 (*Shabtaie and Bentley*, 1994; *Stillman et al.*, 2010). First, we mix grey hematite with ice that produces grey hematite volume concentrations of 26.8-45.2-81.0% for the observed 1st quartile-median-3rd quartile values. The replacement of the porosity with ice reduces the volume concentration of the median value from 59.0% (**Fig. 15**) to 45.2%, however this is still three times larger than the largest values detected on the surface of Mars. We then assume an ice-filled porosity of 19% and 10%, while mixing the ultramafic grains with grey hematite. This produces smaller grey hematite concentrations of 12.4-35.6-81.0% and 10.0-33.2-78.6% for ice concentrations of 19% and 10%, respectively, for the observed 1st quartile-median-3rd values. These values of grey hematite are only about two times larger than those detected on the surface, however the bulk density δ_B of

these mixtures are higher than any typical volcanic rocks measured on Earth (*Olhoeft and Johnson*, 1984; *Shmulevich et al.*, 1971; *Kiefer et al.*, 2012; *Rust et al.*, 1999). To reduce δ_B , we then mixed a 10% ice with grey hematite and mafic grains to produce grey hematite concentrations of 18.5-39.2-79.8%. Such a reduction in δ_B leads to an increase in grey hematite concentration, and would likely still produce a measurable gravity anomaly. *Li et al.* (2012) displays no gravity anomaly is present at the Ultimi Scopuli site, albeit greater gravity resolution is needed before this could be totally ruled out. Overall, we find that large amounts of grey hematite can create ε_a values that match the MARSIS observations. However, the concentration of grey hematite (median values from 33.2-59.0 vol% and 3rd quartile value from 78.6-81.0 vol%) and extent (20-30 km ellipse) of the deposits needed rival that of terrestrial banded iron formation, such formations are not expected on Mars.

5.2 Metastability and Premelting

Measurements made while cooling brine mixtures can show significant hysteresis as well as long-term (of at least a few days) stability (**Fig. 8**; *Primm et al.*, 2020). Unlike the measurements of MgCl_2 (*Primm et al.*, 2020), we could not reproduce the metastability of $\text{Ca}(\text{ClO}_4)_2$. One experiment froze as low as 188 K, while many other experiments it did not freeze down to 173 K and in one case down to 163 K. Recall, *Toner et al.* (2014) measured two $\text{Ca}(\text{ClO}_4)_2$ samples, which did not freeze until 153 K. Without freezing no hysteresis was measured. It is far from demonstrated that the longevity of metastable brines shown in laboratory settings extends over geologic timescales. Therefore, below we pursued hypotheses that do not require metastability until future improved temperature models can fully eliminate the possibility of SPLD basal temperatures of 197.3 K.

Figures 5, 6, & 7 show an increase in ε_a and σ_{DC} before the eutectic temperature is reached. This parabolic behavior is known as premelting (e.g., *Rempel et al.*, 2004; *Stillman et al.*, 2010; 2019; *Rempel*, 2012) and is typically seen in terrestrial permafrost samples as they approach their melting temperature near 273 K (*Grimm and Stillman*, 2015). This premelting effect allows very high salt concentrations to reach the observed MARSIS ε_a values a degree or two below the salt's eutectic temperature.

5.4 Brine Mixtures

Partially-saturated brines are capable of increasing ε_a to the observed values, if the temperature is greater than the eutectic temperature of the salt (**Figs 4, 5, & 7**). The significant increase in ε_a is due to the increase in ε'' via σ_{DC} . Our $\text{Ca}(\text{ClO}_4)_2\text{-H}_2\text{O}$ measurements show that 1st and 3rd quartile values are reached at the eutectic temperature with a $\text{Ca}(\text{ClO}_4)_2$ concentration of 140 mM (3.3 wt%) and 310 mM (7.1 wt%), respectively (**Fig. 16**). Note, the lower end of this range overlaps with the upper range of saline ice (0.1–3.5 wt%) range, as suggested by *Bierson et al.* (2021). The bulk brine concentrations of these samples at the 1st and 3rd quartile values are 2.7 and 6.0 vol% brine at a eutectic temperature (**Fig. 2**).

In salt-H₂O-sediment mixtures, the salt-H₂O just occupies the pore space. Thus, a greater concentration of salt is needed when mixed with sediment to match the MARSIS observations compared to the salt-H₂O mixtures without sediment. At the eutectic temperature, we find that for a mixture that is ~60 vol% sand and 40 vol% $\text{Ca}(\text{ClO}_4)_2\text{-H}_2\text{O}$ the observed ε_a values for the 1st and 3rd quartile values would have a $\text{Ca}(\text{ClO}_4)_2$ concentration of 770 mM (16.4 wt%) and 2.5 M (43.4 wt%), respectively (**Fig. 16**). We then modeled the amount of brine in the bulk sample to calculate a brine concentration of 6.3 and 28.7 vol% at the 1st and 3rd quartile values, respectively. Thus, we find that the brine concentration in fine sand must be 2-5 times larger than when the brine is just in ice. This occurs because of increased tortuosity of the LVNs, which leads to a lower σ_{DC} . This drop in σ_{DC} is much larger when the pores and pore throats are even smaller. Therefore, high-specific surface area sediments (i.e., clays and JSC Mars-1) possess LVNs, but with high tortuosity and small σ_{DC} (*Stillman et al.*, 2010; 2019; *Stillman and Grimm*, 2011ab; **Figs. 10-13**) Therefore, these salt-H₂O-sediment mixtures cannot obtain the MARSIS observed ε_a values.

5.5 Salt Enhancement

The salt-regolith concentration discovered at the Phoenix landing site was 0.7 mass% (*Hecht et al.*, 2009). Our 1st quartile, median, and 3rd quartile salt-H₂O-sediment values have a salt-bulk sample concentration of 3.6, 5.7, and 11.3 mass%, respectively. Thus, the sediment at the basal layer of the SPLD would have to be enhanced by a factor of ~5–16 compared to the Phoenix landing site. Below we discuss four salt enhancement mechanisms:

(1) Drainage of an interconnected system of LVNs could drain concentrate brine down through the SPLD. Similar physics occurs within sea ice and has been hypothesized to occur within the

Europa ice shell (e.g., *Hesse et al.*, 2022). LVNs could have drained to the base of the SPLD or have drained into regolith/rock sediments below the SPLD.

(2) The sublimation of CO₂/H₂O snow from a previous SPLD-like construct would have left deposited salt on the remaining sediment or dust upon which the current SPLD now rests. Similar processes occur when snow with small amount of salt blows into the upper dry valleys in Antarctica. This snow then sublimates away, leaving the sediment highly enriched in salt (*Levy et al.*, 2012).

(3) The sublimation of ice due to a change in orbital parameters can concentrate the amount of dust and by proxy the amount of salt mixed into the dust. This process has occurred in the SPLD as images at the edge of the SPLD show layers with varying amount of dust (some likely with more than 10%; *Milkovich and Plaut*, 2008). Note that ~80% of the ice would have to sublimate here to concentrate the ice to our estimated values, as such the resulting layer would be more similar to a salt-H₂O mixture in sediments.

(4) Salt could also be concentrated via the accumulation of water in a paleolake, which then dried up, concentrating salts in the paleolake sediments. Similar processes are thought to have formed the chloride deposits found throughout the southern highlands (*Osterloo et al.*, 2008). Note that these previously identified deposits have a maximum area of 25 km² compared to the strong reflector that has an area of ~600 km².

6. Conclusions

Here, we have compiled new and old measurements of electrical properties of Martian analog materials at SPLD basal temperatures. This paper has shown that of all the materials proposed as the source of the bright reflections at Ultimi Scopuli, only brine-rich ice mixtures are viable options. Ca(ClO₄)₂ brines are the most likely brine as they have the lowest eutectic temperature of any common Martian salt. The concentration of Ca(ClO₄)₂ to H₂O needed is 140 – 310 mM (3.3 – 7.1 mass%) to obtain the observed 1st and 3rd quartile values. Likewise if liquid brines exist in sediment with ~40% porosity, a larger concentration of 0.77 – 2.5 M (16.4 – 43.4 mass%) of Ca(ClO₄)₂ is required to obtain the observed 1st and 3rd quartile values. Thus, the sediments require a factor of ~5-16 concentration in salt enhancement compared to that measured at the Phoenix landing site. However, the large increase by Ca(ClO₄)₂ brines is only stable above its measured eutectic temperature of 197.3±0.2 K, which is too large estimates of simple SPLD thermal models.

We suggest that layers of CO₂ ice or CO₂ mixed in with H₂O could increase the basal temperatures above the eutectic temperature of Ca(ClO₄)₂. Additionally, the volume of brine is estimated to be 2.7-6.0 vol% and 6.3-28.7 vol% in ice mixtures and sand mixed ice, respectively. Thus, partially-saturated ice or ice sediment mixtures (not briny lakes) are required to produce the observed reflection.

If thermal modeling cannot increase temperatures to the eutectic temperature of Ca(ClO₄)₂, then metastable brines are necessary to drop the range at which brines could exist. However, additional research is needed constrain the stability/lifetime of metastable brines, as well as a larger concentration of salt. We also demonstrated that grey hematite is the only known mineral on Mars to possess a large ϵ_a when dry and at SPLD-like temperatures. However, concentrations 2–4 times (33.2–59.0 vol%) than any currently known are needed to match the MARSIS SPLD observations. Such large concentrations of grey hematite would produce a gravity anomaly that could be used to test this hypothesis.

Acknowledgments. We acknowledge the support of the space agencies of Italy (ASI) and the United States (NASA) for the development and science operations of MARSIS. Operations of the Mars Express spacecraft by the European Space Agency (ESA) are gratefully acknowledged. D.S. was supported by NASA grant #80NSSC20K0858. The authors thank Luca Guallini, Francesco Soldovieri, and Robert Grimm for intellectual discussions regarding this subject. D.S. thanks Michael Shoffner for his laboratory assistance. Data for this paper can be found here DOI: 10.5281/zenodo.6600729

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