

RESEARCH LETTER

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Key Points:

- Five clay minerals known to exist on Mars were experimentally shocked from 10 to 40 GPa and products were analyzed with infrared spectroscopy
- At 10 GPa, chlorite exhibits impact-induced oxidation of Fe, and at 20–30 GPa, kaolinite exhibits signs of structural disorder
- Laboratory results are applied to infrared remote sensing of clays on Mars

Supporting Information:

- Supporting Information S1

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Shock metamorphism of clay minerals on Mars by meteor impact

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Abstract A large fraction of clay minerals detected on Mars by infrared remote sensing represent materials exhumed from the subsurface by meteor impact, begging the question of whether the infrared features used to detect the clays are affected by shock associated with the impacts. We used X-ray diffraction and infrared and Mössbauer spectroscopy to evaluate the mineralogy of five clay minerals after experimentally shocking them to six shock pressures from ~10 to 40 GPa. The shocked clays exhibit three main relevant shock effects: (1) an overall decrease in infrared spectral contrast in the impact-fragmented materials, (2) oxidation of Fe in ferrous clays, and (3) loss of some spectral structure in relatively well-ordered clays such as kaolinite. Other than the widespread oxidation of ferrous clays, shock metamorphism likely has little effect on the accurate interpretation of clay mineralogy on Mars from remote sensing data. However, we are able to identify rare cases of extreme shock in some Martian clay deposits.

Plain Language Summary One of the most significant achievements in planetary sciences in the last 20 years has been the identification and mapping of Martian surface mineralogy by infrared remote sensing. Of major interest is the characterization of clay minerals that formed in ancient habitable environments on Mars >3.5 Ga ago. Because most of these deposits are extremely ancient, they exist within crustal materials that have experienced numerous meteor impacts and therefore could have been affected by heat and pressure associated with those events. The paper contains three main points: (1) the main features that are used to interpret clay mineralogy from infrared data remain intact despite shock metamorphic effects up to pressures of ~40 GPa, (2) shocked clays display some key features that can be used to identify effects of shock remotely, and (3) we describe some of the global-scale spectral biases in interpretation of clays on Mars that are likely to arise from shock effects and we show some concrete examples of shocked Martian clays in certain cases. We believe that this would be the first publication of clear evidence for shocked minerals detected by remote sensing on any planet.

1. Introduction

Over a decade of near-infrared remote sensing exploration of Mars has revealed thousands of deposits of clay minerals, a large fraction of which occur in materials exhumed from the subsurface by impact craters [Ehlmann *et al.*, 2011]. Clay minerals detected by near-infrared spectroscopy ($\lambda \approx 1\text{--}3 \mu\text{m}$) occur within igneous rocks, sedimentary strata, and hydrothermal deposits in the central peaks [Sun and Milliken, 2015], uplifted rims, and ejecta of impact craters [Carter *et al.*, 2013] (Figure 1). Given this context, it stands to reason that clay minerals exhumed from subsurface environments could be affected by shock metamorphism associated with meteor impacts [Boslough *et al.*, 1986; Johnson *et al.*, 2002]. While lithostatic pressures in the shallow Martian crust (<50 km) only are <1 GPa, meteor impacts result in large parts of impacted crust experiencing shock pressures of 10–50 GPa, with heterogeneous, local peak pressures reaching an order of magnitude higher [French, 1998]. The question is whether shock-induced pressure [Kraus *et al.*, 2013] or temperature effects [Gavin and Chevrier, 2010; Che *et al.*, 2011] would confound attempts to interpret clay mineralogy [Weldon *et al.*, 1982; Boslough *et al.*, 1986] of Mars from remote sensing data.

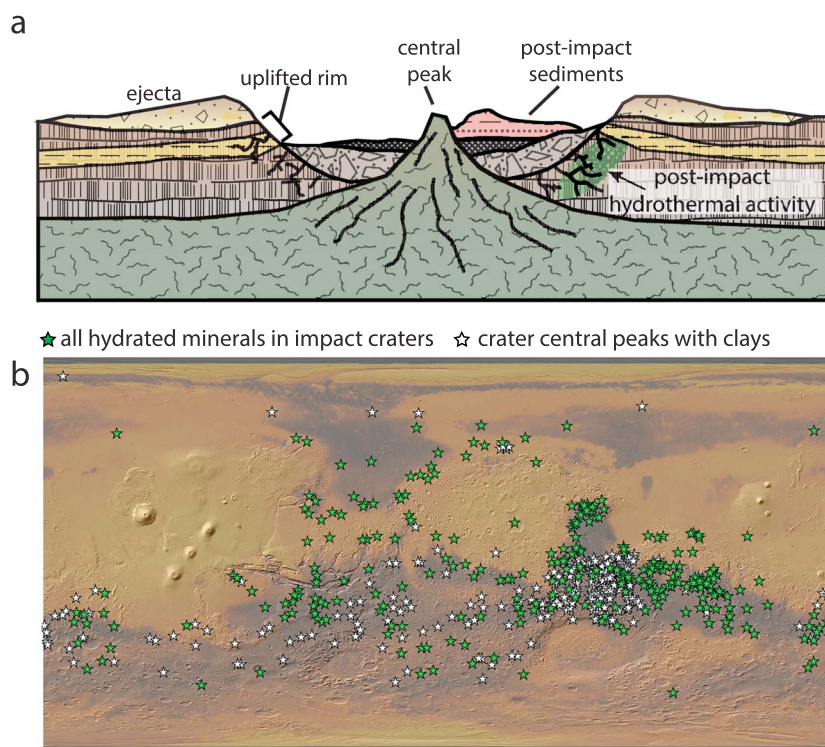


Figure 1. Clay minerals are found in several different contexts associated with impact craters on Mars. (a) Some contexts represent preimpact clays that have been exhumed by the impact event (e.g., central peaks, ejecta, and uplifted rims) and might contain shocked materials. Other contexts include clays deposited postimpact into crater basins. A global map shows alteration minerals associated with impact craters on Mars. (b) The green stars show all hydrated minerals occurring in impact craters [Carter *et al.*, 2013], and the white stars show only clay minerals associated with crater central peaks [Sun and Milliken, 2015]. Both maps include many examples of exhumed clay minerals and some examples of postimpact clays.

Not all clay minerals associated with craters were necessarily uplifted/exposed by the impact, and clay minerals could be formed by impacts themselves [Tornabene *et al.*, 2013]. Undoubtedly, impact-induced clay formation must have happened on Mars [Marzo *et al.*, 2010]. However, most clay minerals associated with impact craters occur within units that appear to be uplifted from the subsurface in fractured but intact clay-bearing terranes [e.g., Michalski and Niles, 2010], rather than postimpact hydrothermally altered rocks (Figure 1). Impact craters are also the most common type of sedimentary basin on Mars. Therefore, it is important to differentiate between rocks exhumed by the impact and rocks that formed later in the basin, many of which could contain clays and other hydrated phases detectable by infrared remote sensing (Figure 1).

Understanding the mineralogy of clay minerals exhumed by meteor impact on Mars is important for several reasons. The detailed mineralogy and crystal chemistry of the clays trace the chemical conditions of ancient aqueous processes within the crust [Ehlmann *et al.*, 2013]. These include diagenetic and metamorphic processes relevant to understanding the nature of the planet's crust and overall water budget. Also, life could have formed and been sustained within deep crustal hydrothermal systems [Michalski *et al.*, 2013]. But to truly understand the 3-D composition of the crust and subsurface alteration, as indicated by clay minerals exhumed by impact, it is important to determine how shock affects those interpretations of clay mineralogy. Some recent studies have investigated the effects of shock on clay mineralogy at low shock pressures (<20 GPa) [Gavin *et al.*, 2013] and described some detailed relationships between shock and clay spectroscopy at higher pressures [Friedlander *et al.*, 2015, 2016]. Here we summarize the results of a series of shock experiments carried out on clay minerals in the laboratory and relate those results to the spectroscopically observed mineralogy of Mars.

2. Experimental Setup and Analytical Methods

The <2 μm size fraction of each of five well-characterized phyllosilicate samples was separated, dried, and gently ground with mortar and pestle into loose powder. In an effort to decrease porosity, which can

result in increased shock melting during experimental impacts, ~150 mg samples of each powder were pressed at ~70 MPa into disks ~2 mm thick using a hydraulic hand press. Each pellet was loaded into a sample container precisely milled to fit the pellet, minimizing void space. These samples were then shocked experimentally using the flat plate accelerator (FPA) at NASA's Johnson Space Center.

At the FPA, stainless steel or fansteel flyer plate projectiles were launched horizontally at the secured sample containers. Lasers mounted in the flight path were used to determine the projectile velocity and mounted cameras were positioned to characterize projectile tilt (experiments with tilt $>3^\circ$ were rerun). Velocities of 0.872–1.349 km/s were converted to pressure using one-dimensional shock-stress relationships [Gault and Heitowitz, 1963].

The experimental setup was designed to produce six runs resulting in six peak shock pressures of approximately 10, 20, 25, 30, 35, and 40 GPa for each of five clay samples; achieved shock pressures differed by up to a few percent. Recovery was nearly 100% for most runs, although the highest-pressure experiments resulted in some sample loss because the sample holder was highly deformed. Samples chosen for shock experiments include five important phyllosilicates known to exist on Mars from infrared remote sensing: two dioctahedral clay minerals (kaolinite and nontronite) and three trioctahedral clay minerals (serpentine, chlorite, and saponite).

Of primary interest in this work were the near-infrared reflectance (NIR) ($\lambda = 1\text{--}3\ \mu\text{m}$) spectra because these data are directly relevant to measurements of clay minerals made on Mars by orbital remote sensing instruments. In addition to NIR analyses, the unshocked and shocked samples were analyzed by Mössbauer spectroscopy, X-ray diffraction (XRD), and transmission electron microscopy (TEM). We provide a brief summary of analytical methods here. Additional details are available in Friedlander *et al.* [2016].

Bidirectional NIR data were collected under a nitrogen-purged environment from $\lambda = 0.35\text{--}2.5\ \mu\text{m}$ with a sampling interval of 1.4–2 nm at the Center for Planetary Exploration at Stony Brook University. Mössbauer spectra were acquired using a source of ~60 mCi ^{57}Co in Rh on a WEB Research Co. (now SEE Co.) model WT302 spectrometer (Mount Holyoke College) at 295 K. Spectra were calibrated against an Fe metal foil and fitted using standard methods [Cuadros *et al.*, 2013]. XRD analyses were carried out using a Bruker D8 diffractometer at Indiana University, with Cu K α radiation and a SolX solid-state point detector. All samples were analyzed before shock experiments using random-powder cavity mounts, and run products were mounted on “zero-background” quartz plates. The clay particles were examined by TEM using a Philips CM200FEG S/TEM instrument in the LeRoy-Eyring Center for Solid State Science at Arizona State University. Bright field images were used to characterize particle size and morphology, and selected area electron diffraction (SAED) patterns were collected to determine crystallinity and disorder.

3. Effects of Shock on Near-Infrared Spectroscopy of Clay Minerals

The most important result of this work is the demonstration that shock effects generally do not obfuscate identification or interpretation of clay minerals from NIR spectra even if they are shocked to pressures up to ~40 GPa. Major absorptions used to identify and characterize clay minerals with NIR remote sensing remain intact despite shock effects [Gavin *et al.*, 2013], although interesting and important modifications to some of those spectral features occur. The conservation of most NIR spectral features upon exposure to high shock pressures stands in contrast to the midinfrared spectral characteristics, which, depending on the species, typically begin to resemble amorphous silicates between 20 and 40 GPa [Friedlander *et al.*, 2016].

Essentially all clay minerals exhibit some similar spectral features related to basic phyllosilicate mineralogy [Farmer, 1968] (Figure 2). Absorptions located near 1.4 μm result from OH stretching overtones. H $_2$ O adsorbed onto surfaces and between layers within the phyllosilicate structure result in infrared absorption at ~1.9 μm . Metal-OH deformations within the octahedral sheets produce diagnostic absorptions located from 2.2 to 2.35 μm , where the position of this feature is an important indication of octahedral chemistry. Absorption at 2.2 μm is indicative of AlOH in kaolinite (the nontronite has a kaolinite contaminant in it). FeOH in nontronite absorbs at ~2.28–2.29 μm . MgOH in saponite, serpentine, and chlorite absorbs at approximately 2.32, 2.33, and 2.34 μm , respectively. Absorptions at 2.35–2.5 μm are less diagnostic, resulting from complex combination absorptions in the octahedral and tetrahedral sheets. Lastly, a broad absorption located between 1 and 2 μm results from electronic crystal field effects in Fe within Fe $^{2+}$ -bearing samples. This is most pronounced in the chlorite sample, which contains significant amounts of Fe $^{2+}$.

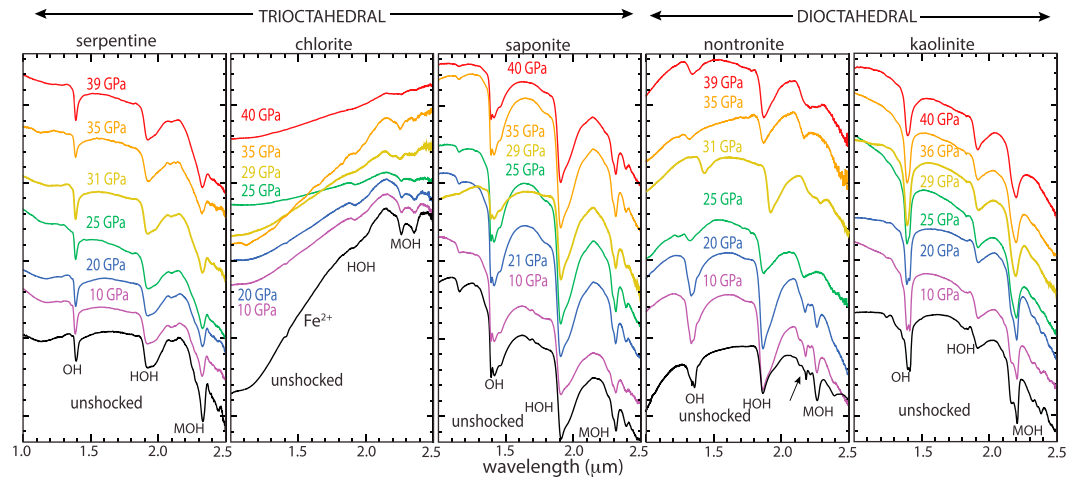


Figure 2. NIR spectra are presented for shocked clay minerals, as well as for the corresponding unshocked samples. “MOH” corresponds to metal-OH features for each mineral located from 2.2 to 2.35 μm. “HOH” corresponds to vibrational overtones in adsorbed H₂O. “Fe²⁺” corresponds to a spectral slope located from 1 to 2 μm related to electronic transitions in Fe. “OH” corresponds to hydroxyl deformation overtones located near 1.4 μm. “GPa” corresponds to the shock pressure calculated for each experimentally altered mineral.

NIR spectra of shocked clays are affected by both physical and mineralogical changes. Physical changes result from intense fragmentation of the sample during shock decompression. Such changes are evident in the XRD analyses, which show peak broadening of the 001, 02 ℓ, and 06 ℓ peaks in all shocked samples, consistent with a decrease in crystallite size in the shocked samples relative to the unshocked samples.

Changes in crystallite sizes and physical particle sizes are known to affect NIR spectral shape and contrast. Here we quantify the effects of impact-induced changes on spectral contrast for the most diagnostic of

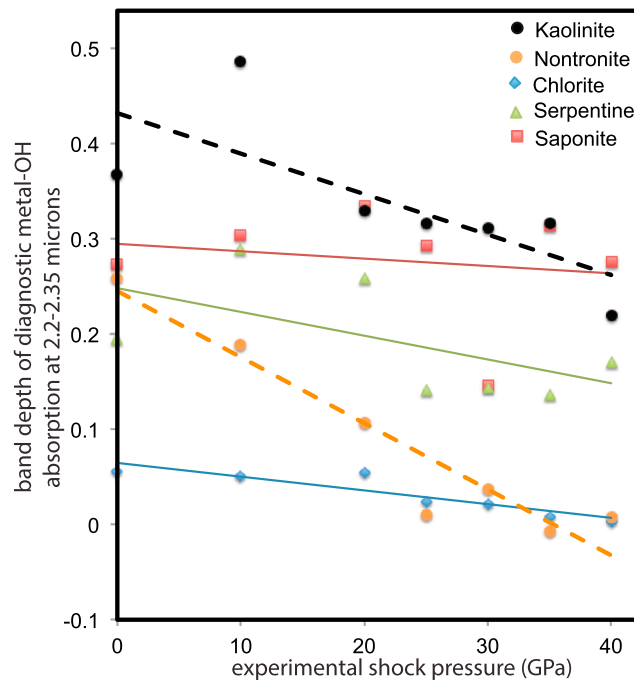


Figure 3. Shock metamorphism and fragmentation result in decreased spectral contrast in the diagnostic metal-OH features used to identify clay minerals on Mars with NIR data. The effect is more pronounced for dioctahedral clays (e.g., kaolinite and nontronite) (dashed lines) than for trioctahedral clays (e.g., chlorite, serpentine, and saponite).

the NIR absorptions for clays, the metal-OH feature located from (λ) 2.17 to 2.35 μm (see supporting information for details). For each mineral, we created a simple spectral index defined as

$$BD = 1 - (R_{\lambda 1} \times 2) / (R_{\lambda 2} + R_{\lambda 3}),$$

where BD is the band depth, $R_{\lambda 1}$ is the reflectance at the metal-OH absorption center, and $R_{\lambda 2}$ and $R_{\lambda 3}$ are the continuum reflectance values on either side of the absorption. Using kaolinite as an example, $R_{\lambda 1}$ corresponds to 2.210 μm, $R_{\lambda 2}$ corresponds to 2.127 μm, and $R_{\lambda 3}$ corresponds to 2.231 μm. The result of this analysis shows that the band depth of the metal-OH feature decreased by an average of ~40% due to shock effects for all samples (Figure 3). However, the effects are more pronounced for dioctahedral clays (average $R^2 = 0.74$) than for trioctahedral clays (average $R^2 = 0.40$). The band depth for saponite (trioctahedral) is essentially unaffected.

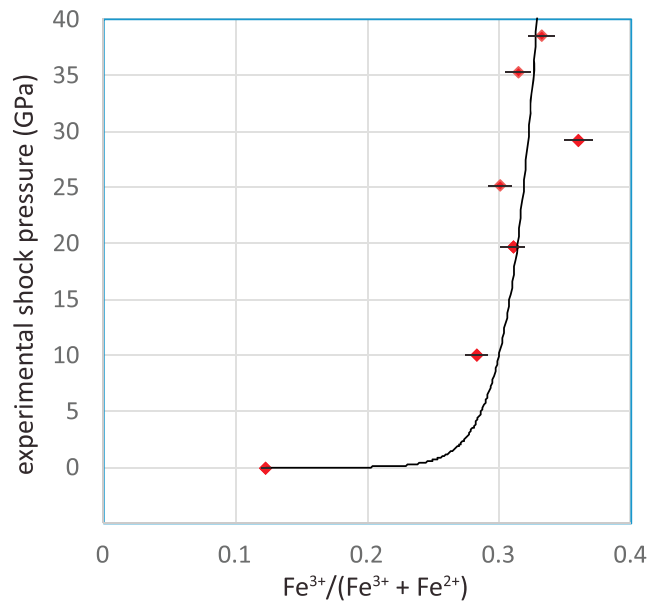


Figure 4. The amount of oxidized iron (Fe^{3+}) versus total Fe is plotted against experimental shock pressure for chlorite. Even minor amounts of shock (10 GPa) dramatically affect oxidation of Fe. The effect is nonlinear at high shock pressures (>20 GPa). Estimated error bars of $\pm 3\%$ are shown for Fe^{3+} data derived from Mössbauer spectroscopy.

Changes in the spectral slope of chlorite between $\lambda = 1$ and $2 \mu\text{m}$ suggest that Fe-oxidation is related to shock, as has been observed in igneous minerals [McCanta and Dyar, 2017]. Mössbauer spectroscopy results relating the amount of oxidized Fe to total Fe show that even relatively low amounts of shock pressure (10–20 GPa) have a profound effect on Fe oxidation for chlorite (Figure 4). The trend is nonlinear and incremental increases in shock pressure above 20 GPa do not produce a proportional increase in Fe oxidation (Figure 4). We interpret this to result from heterogeneous distribution of peak heating within the samples likely due to collapse of residual pore space.

In fact, the TEM data of kaolinite samples show strong evidence for heterogeneous distribution of shocked

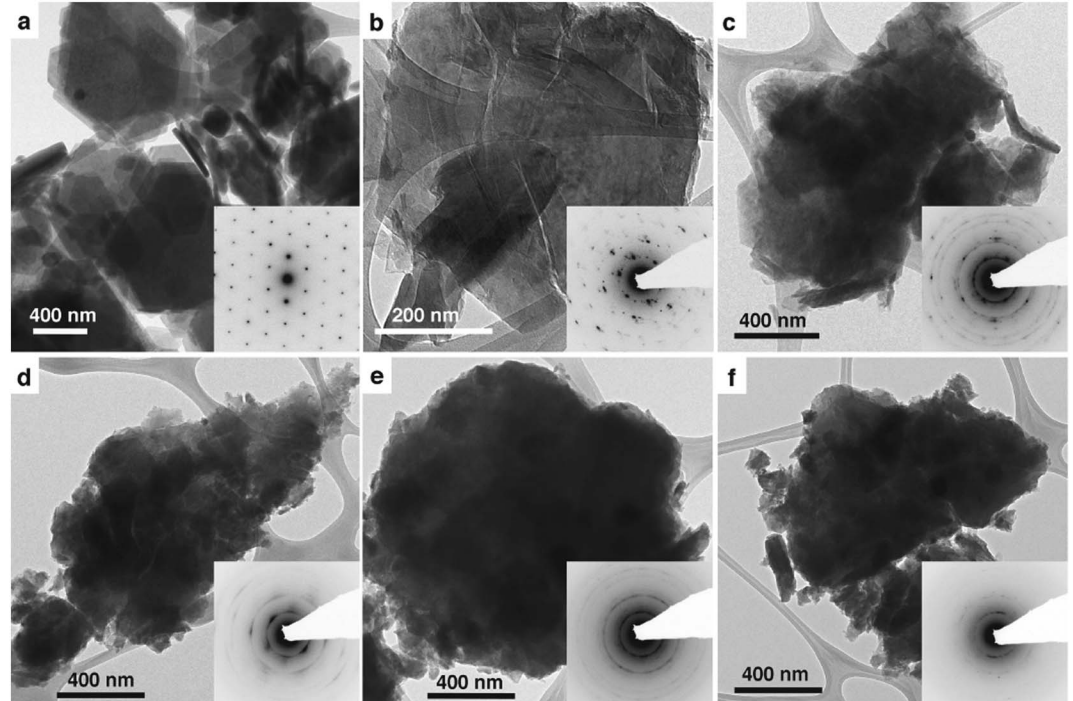


Figure 5. Bright-field TEM images and inset selected area electron diffraction (SAED) patterns of (a) unshocked kaolinite and kaolinite shocked to (b) 10, (c) 20, (d) 29, (e) 36, and (f) 40 GPa. These are examples of the most deformed materials that show a progression from ordered pseudo hexagonal plates (Figure 5a) through nearly amorphous material at 40 GPa. At 10 GPa (Figure 5b), the crystals show minor deformation and rotational disorder in the SAED pattern. At 20 GPa (Figure 5c), the plate-like morphology is reduced and the SAED shows a prominent and sharp ring pattern indicative of rotational disorder. At 29, 36, and 40 GPa, the fragments have lost their plate-like morphology and the SAED patterns show progressively more diffuse and weakening diffraction intensity.

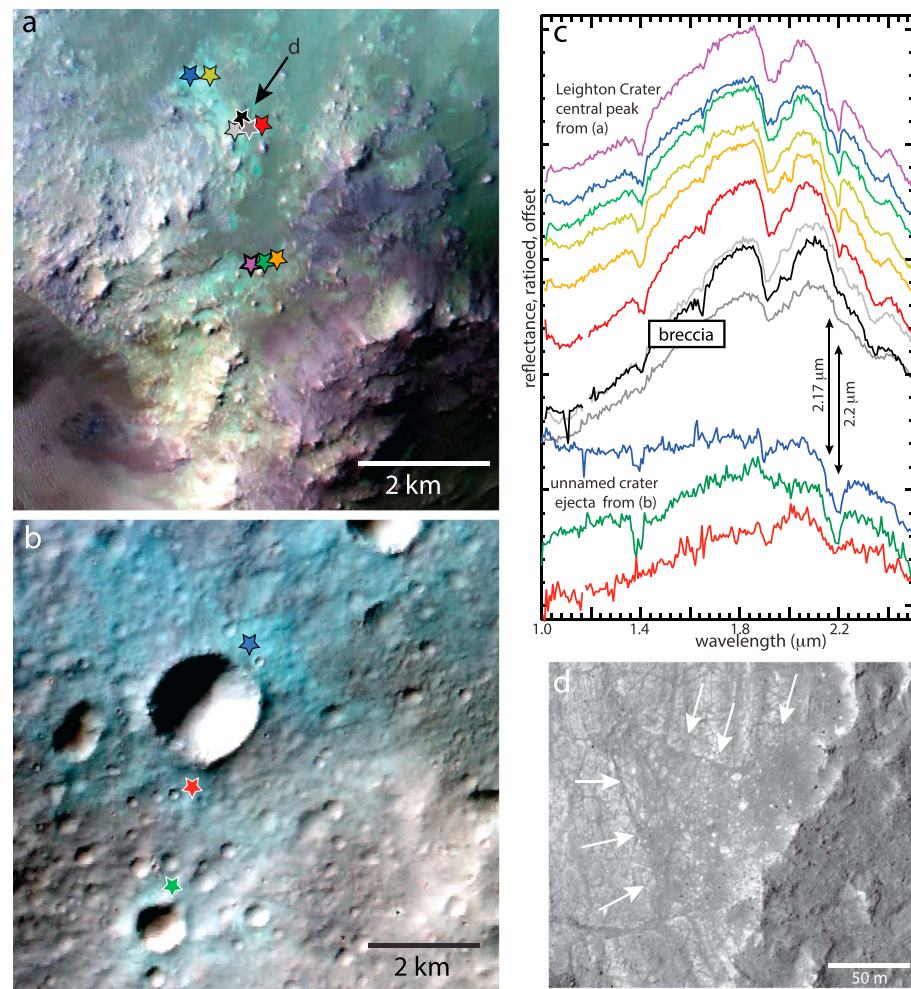


Figure 6. CRISM spectra show evidence for shocked kaolinite on Mars. (a and b) The blue-green colors in the CRISM images correspond to kaolinite-bearing surfaces. (c) The central peak of Leighton crater (Figure 6a) and ejecta of an unnamed crater located at 158.9°E, 25.83°S (Figure 6b) show systematic changes in their spectral features that could be related to shock. Specifically, variations in the spectral structure of AlOH overtones at 2.17 and 2.21 μm are consistent with the observed trends in experimentally shocked kaolinite. Some spectra within the kaolinite unit in Leighton crater show extreme spectral changes (gray-black lines in Figure 6c). (d) The surface bearing these spectra appears brecciated and possibly melted in HiRISE data, and therefore, the features observed with CRISM in these surfaces could correspond to melt or altered melt rocks. CRISM false color images display I/F at 1.08 μm as blue, 1.51 μm as green, and 2.53 μm as red.

material. The unshocked kaolinite crystals are relatively coarsely crystalline, with hexagonal plates $\sim 1\text{--}2\ \mu\text{m}$ in diameter and tens of nanometer thickness (Figure 5). Like the unshocked sample, the 10 GPa shock material was generally composed of large crystals and displays SAED patterns indicative of well-ordered material. Significant changes are observed at 20 GPa where the material contains both amorphous domains and relatively undeformed domains. This trend continues up to 25, 30, and 36 GPa, where the proportion of highly deformed and disordered material steadily increases. The 40 GPa material that is nearly completely amorphous, but even in this highly shocked sample, domains of crystalline kaolinite exist (Figure 5).

These progressive shock effects in kaolinite result in discernible changes to NIR spectral structure of the samples (Figure 2). The NIR spectra of unshocked and low-shock (10 GPa) samples show spectral structure in the 2.17–2.21 μm region diagnostic of the AlAlOH vibrations in relatively well-ordered kaolinite-group minerals [Crowley and Vergo, 1988]. With increasing shock, these overlapping features are broadened and merged into a single spectral absorption centered at $\sim 2.2\text{--}2.21\ \mu\text{m}$. Similar impact-induced disorder is observed in the loss of the doublet spectral structure of the OH overtone absorptions located at 1.41 μm in shock pressures above 20 GPa.

Nontronite and chlorite exhibit slightly more complex behavior in the 2.2–2.35 μm spectral region. Just as in the pure kaolinite sample, the admixed kaolinite contaminant in the nontronite sample loses its spectral structure at ~ 20 GPa [Friedlander *et al.*, 2015], but a strong 2.2 μm AIOH feature persists to high pressures. Over the same pressure range, the FeOH absorption at 2.28 μm becomes weaker and broader to the point that it is unrecognizable against the dominant, stronger AIOH absorption profile. Similarly, chlorite contains two metal-OH absorptions relating to AlMgOH at lower wavelength (~ 2.26 μm) and FeMgOH at higher wavelength (2.35 μm). With increasing shock, the 2.35 μm feature is lost, potentially due in part to oxidation of Fe, which destabilizes MgFeOH bonds in the octahedral sheet. The AlMgOH bonds are less affected, and therefore, the 2.26 μm absorption persists to high shock pressures.

4. Detection of Shocked Clays on Mars

Impact-driven fragmentation of the Martian crust would have resulted in the decrease in lithic particle size and clay crystallite size in ancient, phyllosilicate-bearing materials. Some of the record of ancient Martian clays is likely, as a result, either spectrally undetectable or spectroscopically similar to amorphous materials [Friedlander *et al.*, 2016]. However, some particular features of shocked clays allow for remote infrared detection of shock effects in some cases on Mars.

Spectra from the Compact Reconnaissance Imaging Spectrometer for Mars, of the ejecta of an unnamed crater located at 158.9°E, 25.83°S, and the uplifted central peak of Leighton crater (57.76°E, 3.16°N) both show spectral trends consistent with shock metamorphism of kaolinite (Figure 6) (see supporting information for data processing details). NIR spectra of kaolinite in crater ejecta (Figure 6c) show a decrease in spectral structure and increasing breadth. Spectra of kaolinite in the central peak of Leighton display systematic changes to the AIOH spectral structure, as is observed in lab data of shocked kaolinite (Figure 6). Breccia observed within the kaolinite unit in high-resolution visible images exhibits interesting spectral features that might represent melted bedrock or altered melted bedrock. While neither of these examples demonstrates the detection of shocked clays beyond a shadow of a doubt, both show trends consistent with shock processes in appropriate geologic contexts. Shock effects might be more widespread on Mars, but the effects are more easily detectable as they pertain to kaolinite, which is why these two examples were chosen.

5. Conclusions

Most of the Martian surface has been modified by impact cratering. Clay minerals that formed on early Mars at the surface and in the subsurface have been affected by impact processes, potentially including shock metamorphism. Given the importance of Martian clay mineralogy for understanding ancient climate and early habitability of the planet [Bibring *et al.*, 2006], the questions of if and how shock processes influence our interpretation of the mineralogy of clay minerals on Mars are both interesting and necessary. Importantly, our results show that most of the essential aspects of clay mineral identification by infrared remote sensing of Mars are not obfuscated by shock effects. This result is important not only for remote sensing of Mars but also for other impacted bodies with clay minerals such as asteroids and Ceres [Ammannito *et al.*, 2016].

Effects of shock up to 40 GPa do not dominate the NIR spectral features of clay minerals, but some effects are useful, interesting, and important for Mars. On a global scale, impact fragmentation has reduced the spectral contrast of impacted clays, and therefore, it is important to recognize that the current assessment of clay distribution and abundance on Mars is an underestimate. Second, impact-induced shock could affect the detection of Fe²⁺-rich clays and our analysis of the oxidation state under which early clays formed on Mars. Therefore, it is likely that our interpretation clay mineralogy in the ancient, heavily impacted Martian crust is biased against ferrous clays, which might have been abundant on early Mars.

Acknowledgments

HiRISE and CRISM spectral data used in this work are available through the Planetary Data System (<https://pds.nasa.gov>). Near-infrared spectra of shocked clays will be made available through the following URL in the event of publication: <http://www.clays.space>.

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