

MARS GEOLOGY

Hematite is a mineralogical marker of ancient climate change on Mars

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The ancient climate of Mars changed from warm to cold surface conditions. This climate transition is demonstrated by geomorphological evidence but lacks suitable mineralogical indicators. We investigated the crystallographic properties of hematite (iron oxide) in Gale crater measured by the Curiosity rover and compared them with laboratory experiments. Hematite crystallite sizes are about 5 to 65 nm in the oldest sedimentary rocks investigated by the rover (the Murray formation) and less than 10 nm in the younger overlying strata (the Mirador and Carolyn Shoemaker formations). We attribute the larger crystallites in the Murray formation to postdepositional coarsening by groundwater in warm and wet conditions that persisted for several million years. Hematite with small crystallites co-occurs with goethite (iron oxyhydroxide) in the overlying layers, consistent with colder and water-limited conditions.

The surface of Mars is cold and dry, but it once had abundant surface water and groundwater. Ancient lake, river, and delta deposits record an active hydrological cycle ~3 to 4 billion years (Ga) ago (1). Secondary minerals formed by water-rock interactions in these sedimentary deposits reveal the past environmental conditions. However, there is little direct mineralogical evidence of the climate transition from warm to cold. Hematite (α -Fe₂O₃) is a secondary mineral formed by reactions between water and iron-rich minerals. On Mars, iron is released from the silicate minerals in basalt rocks and from sedimentary rocks derived from basalts, which occur planetwide (2). The crystallographic characteristics of hematite, such as the size of crystallites (single-crystal domains within a grain) and shapes (morphology), are sensitive to formation conditions (3, 4). We investigated how these crystallographic parameters, and the iron-bearing minerals found with hematite, changed on ancient Mars.

The Mars Science Laboratory Curiosity rover is exploring Gale crater (fig. S1). Before rover landing, hematite was identified in this region using orbital visible and near-infrared spectroscopy (5). Orbital observations

showed that hematite is concentrated in the Vera Rubin ridge area of lower Aeolis Mons (informally called Mount Sharp), an ~5-km-tall mound of layered sedimentary rock within Gale crater (5, 6). In situ measurements by the rover have since shown that hematite is not limited to the Vera Rubin ridge region, but rather is widespread in Gale crater sedimentary rocks (7).

To characterize mineral assemblages in Gale crater, Curiosity carries the Chemistry and Mineralogy (CheMin) instrument, which performs x-ray diffraction (XRD) measurements of rock powder samples collected using the rover's drill (8). XRD peaks identify specific minerals, and their width and shape can quantify the crystallite size and morphology. We investigated the hematite crystallite sizes and shapes in the sedimentary rocks in the stratigraphic sequence of the Murray, Carolyn Shoemaker, and Mirador formations (Fig. 1) and compared the results with experimental laboratory data to determine the processes by which hematite formed and grew (9).

Stratigraphic context

Previous studies using CheMin have detected 1 to 16 wt % hematite (10) in lacustrine (formed in a lake) mudstones and fluvial (river) and eolian (wind-blown) sandstones in the Mount Sharp group (Fig. 1), which were deposited ~3.6 to 3.2 Ga ago (11). We restricted our analysis to samples containing ≥ 1.5 wt % hematite, for which the XRD peaks are sufficiently strong to determine the crystallite sizes and shapes. By sol 3627 (Mars days since landing, equivalent to October 2022), the rover had investigated three formations in the Mount Sharp group and found that (i) the Murray formation, the lowest formation, consists of lacustrine mudstones and intervals of fluvial sandstones (12); (ii) the Carolyn Shoemaker formation conformably (deposited without interruption) overlies the Murray formation and includes mudstone, siltstone, and sandstone deposited in lake margin or fluvial environments (13); and (iii) the Mirador formation unconformably overlies the Carolyn Shoemaker formation and is dominated by eolian sandstone (14).

The Murray formation contains abundant smectite, a phyllosilicate mineral (fig. S2), indicating that the ancient fluvial-lacustrine freshwater environments in which it formed had near-neutral pH (15–17). Some drill samples also contain jarosite [$(\text{H}_3\text{O}^+, \text{K}^+, \text{Na}^+)\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6$] and akaganeite (β -FeOOH) (Fig. 1), which are secondary minerals produced by interaction with acidic and saline fluids. The jarosite formed 2.12 ± 0.36 Ga ago, indicating that those acidic and saline fluids probably arose during a later postdepositional alteration phase (18).

The Carolyn Shoemaker formation contains abundant smectite and minor siderite (FeCO₃), an Fe(II) carbonate, indicating that the formation was deposited in near-neutral and fresh waters and was not subsequently altered by acidic fluids (17).

The eolian deposits in the Mirador formation indicate a shift to much drier depositional environments. Smectite is absent in this formation, but some locations have hydrated magnesium sulfate and siderite, indicating saline and alkaline water-limited environments produced by the evaporation of near-surface groundwater (19, 20).

None of these formations contains minerals indicative of widespread burial diagenesis (postdepositional changes). Thermal models show that these sedimentary rocks did not experience temperatures $>125^\circ\text{C}$, which is consistent with the preservation of smectite (21).

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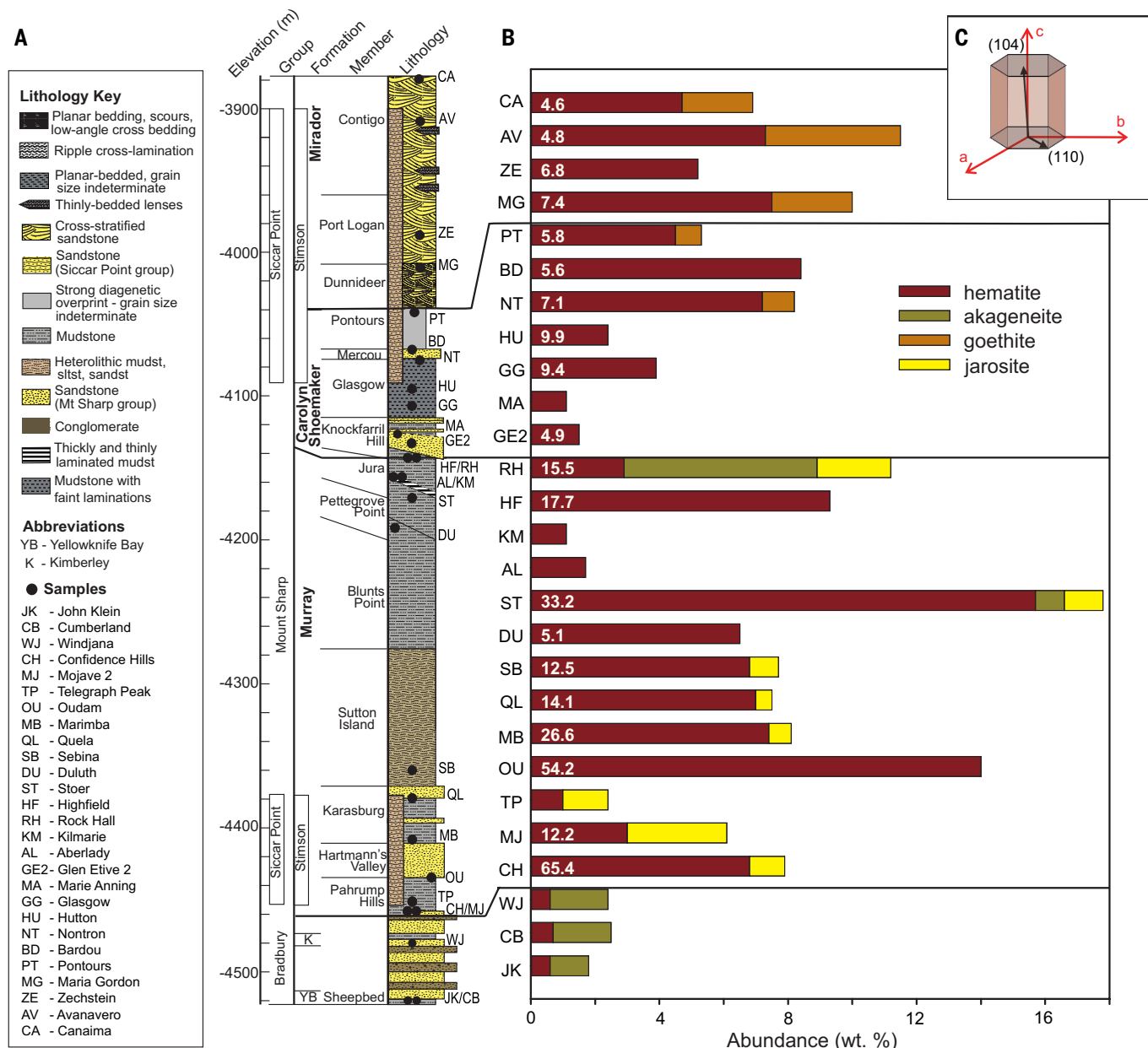


Fig. 1. Variations of hematite crystallite size with stratigraphy. (A) Stratigraphic column of geologic units investigated by Curiosity from landing until sol 3627. Colors and hatching styles indicate the lithology (see legend). The elevation is relative to the Mars geoid. Sample names and abbreviations are listed in the legend. (B) Colored bars indicate the abundances of hematite, goethite, akageneite, and jarosite (see legend). White numbers are the hematite crystallite sizes in nm measured from the (104) XRD peak; samples with no numbers were excluded from our analysis due to their low hematite abundance. (C) Crystal structure of hematite. The (104) XRD peak primarily constrains the size in the c direction.

Hematite crystallite sizes

Hematite forms hexagonal crystals and crystallites (Fig. 1C). We used its most intense XRD peaks, from the (104) and (110) crystallographic planes (figs. S3 to S24), to determine the crystallite sizes in two directions (9). The (110) peak width provides crystallite size in the a-b plane, whereas the (104) peak width reflects size in the c direction (Fig. 1C). We used the whole-powder pattern-modeling method with independent fitting of the mean sizes for each hematite peak (9). The results (Fig. 1B) showed that the (104) crystallite sizes vary substantially with the stratigraphy.

The Carolyn Shoemaker and Mirador formations contain hematite crystallites <10 nm, whereas the Murray formation has crystallites

ranging from ~5 to ~65 nm. The largest crystallites occur in the Oudam and Confidence Hills drill targets in the stratigraphically lowest section of this formation. The unit cell parameters do not vary with crystallite size, indicating negligible ionic substitution (such as Al^{3+}), which would otherwise affect crystallite dimensions and shape (fig. S25 and table S1).

The sizes derived from the (110) and (104) peaks (Fig. 2A and table S1) showed that the crystallites are of approximately equant thickness in every crystallographic direction, implying uniform growth. This size evolution differs from observations of hematite on Earth, which adopts both platy and equant shape morphologies in a wide range of formation and diagenetic conditions. Figure 2B shows equivalent size measurements

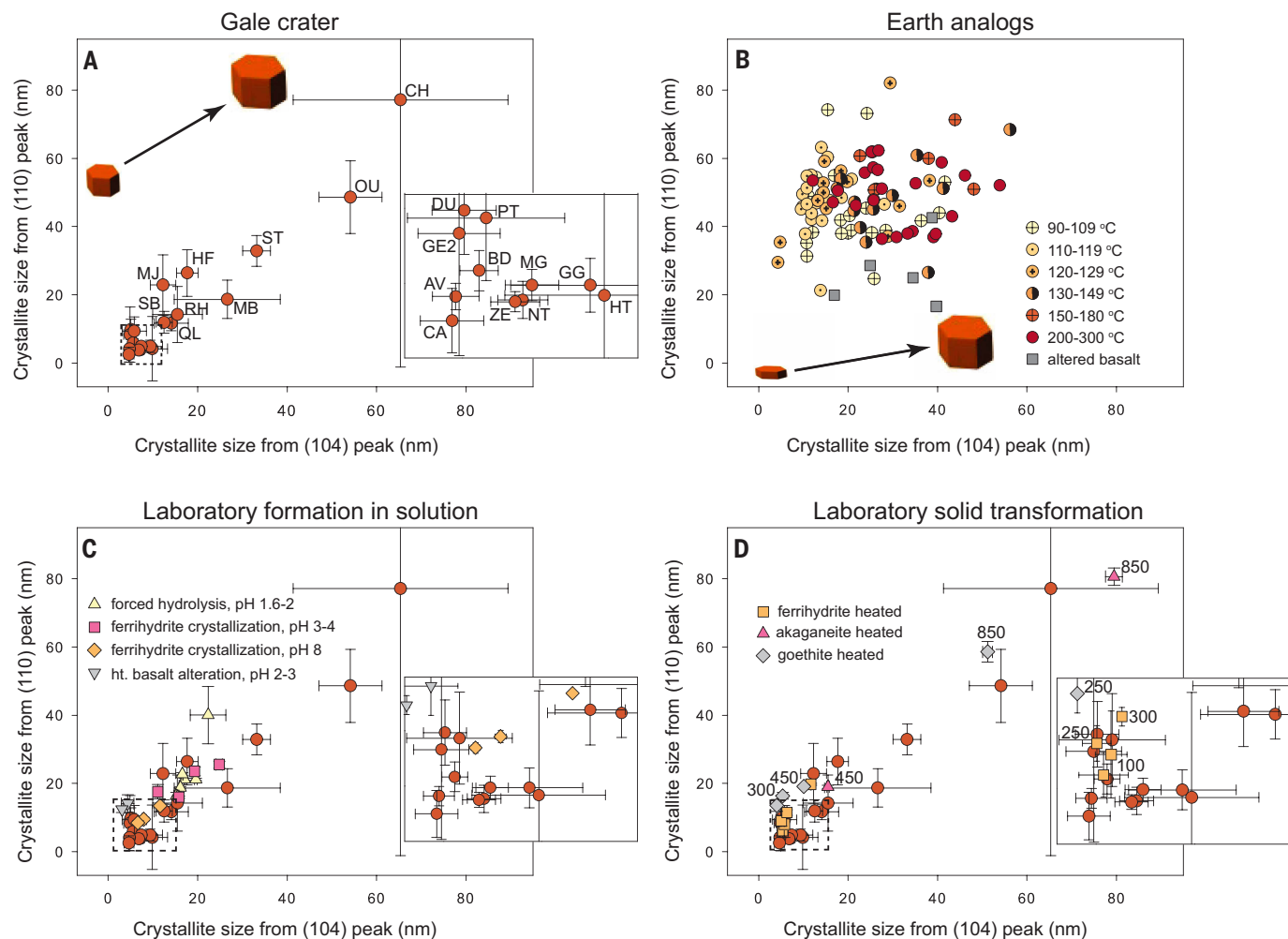


Fig. 2. Two-dimensional mean sizes of hematite crystallites. Sizes were derived from whole-powder pattern modeling with independent fitting of the (110) and (104) diffraction peaks of hematite (9), which constrain the sizes in two crystallographic directions (Fig. 1C). **(A)** Samples from Gale crater (individual XRD spectra are shown in Figs. S5 to S24). Data points are mean values with 1σ error bars. The inset is a magnification of samples with crystallites <10 nm. **(B)** The same parameters measured from Earth analog samples of natural hematite from the Ediacaran redbeds, paleosols, hydrothermally altered basalt, and tuffs of the East European Craton, non-Ediacaran sedimentary rocks (circles, colors indicate the maximum paleotemperatures), and altered basalt in Hawaii (gray squares) (9) (tables S3 and S4). **(C)** Same as panel (A) but compared with laboratory experiments (Figs. S26 to S31) that formed hematite by acidic hydrolysis (yellow triangles), ferrihydrite crystallization at pH 3 to 4 (purple squares) and pH 8 (orange diamonds), and hydrothermal (ht.) basalt alteration (gray downward triangles). **(D)** Same as panel (A) but compared with laboratory experiments (Figs. S32 to S34) that produced hematite by thermal alteration of ferrihydrite (orange squares), akaganeite (pink triangles), and goethite (gray diamonds). Labeled numbers are the temperature in degrees Celsius. For ferrihydrite, the two lowest points correspond to 100°C for 2 weeks and 4 months.

for Earth samples: Ediacaran redbeds, paleosols, hydrothermally altered basalt and tuffs of the East European Craton, non-Ediacaran sedimentary rocks, and hydrothermally altered Hawaiian basalt (tables S3 and S4). Hematite crystallites in Gale crater are also smaller than in gray volcanic hematite on Earth (table S4).

Hematite formation in Gale crater

We considered how the equant-shaped hematite formed and grew in Gale crater and how this relates to the ancient climate of Mars. For comparison with the rover data, we considered laboratory experiments that synthesized hematite by four different processes: forced hydrolysis of Fe(III) solutions (22); aqueous alteration of ferrihydrite [poorly crystalline hydrous Fe(III) oxyhydroxide (22–24)]; oxidation of Fe(II) during hydrothermal alteration of (Mars analog) basaltic glass (25); and thermal decomposition of Fe(III) oxyhydroxides (9). We applied these results to test the plausibility of previous proposals that hematite

in Gale crater formed by direct precipitation from solution and alteration of Fe(III) oxyhydroxides (15, 26). Although the experimental data do not cover all conditions that could influence hematite formation in complex natural settings such as Gale crater, they do include the presence of Mars-relevant ions (ClO_4^- , Cl^- , SO_4^{2-} , Al^{3+} , Si^{4+} , and Mg^{2+}) and a solid substrate (Mars analog basaltic glass) (9).

We found that the hematite in Gale crater could have formed from aqueous alteration of ferrihydrite at neutral to slightly alkaline pH. Crystallites of synthetic hematite formed from ferrihydrite at pH 8 were 7 to 12 nm in size and of equant shape (Fig. 2C), matching the samples from the Murray formation (Duluth, Mojave 2, and Sebina) and slightly exceeding sizes in the Carolyn Shoemaker and Mirador formations. Goethite, an iron(III) oxyhydroxide ($\alpha\text{-FeOOH}$), usually forms together with hematite produced from ferrihydrite under a broad pH range (3). Goethite occurs with hematite in some samples from the Carolyn Shoemaker and Mirador formations (Fig. 1). The abundances of hematite

(*H*) as a fraction of the total hematite and goethite (*G*) are $H/(H + G) = 0.88 \pm 0.14$ for Nontron, 0.85 ± 0.27 for Pontours, 0.75 ± 0.13 for Maria Gordon, 0.63 ± 0.23 for Avanavero, and 0.68 ± 0.18 for Canaima. These ratios are consistent with ferrihydrite crystallization to hematite and goethite at pH 6 to 8 (27). Goethite is absent in the Murray formation, which we attribute to the loss of goethite during later diagenesis (discussed below).

Small hematite crystallites might alternatively form by dehydration of ferrihydrite at elevated temperatures, such as would be experienced by burial diagenesis in Gale crater. We performed thermal alteration experiments on three Fe(III) oxyhydroxides that have been identified in Gale crater: goethite, akaganeite, and ferrihydrite (16, 26, 28). The resulting crystallites were either of platy morphology (formed from goethite; Fig. 2D), unlike the equant shapes found in Gale crater, or required higher temperatures (from akaganeite at 450°C; Fig. 2D) than the estimated maximum during burial diagenesis (125°C) for rocks in Gale crater (21). Only ferrihydrite heated to 100°C produced equant <10 nm hematite, consistent with the small <10 nm crystallites in Gale crater (Fig. 2D). We consider this temperature an upper limit on anhydrous ferrihydrite alteration; however, small hematite crystallites with equant shape can also form from ferrihydrite at lower temperatures [such as ~17°C (29); table S1].

We also investigated the formation of hematite under acidic conditions (Fig. 2C) (9). Crystallites of equant shape formed under extreme acidic pH <2, which is unlikely for Gale crater. At higher pH (pH 3 to 4), Fe(III) sulfate minerals would have formed instead of hematite. We therefore excluded this possibility.

The combination of equant shapes and small crystallite sizes indicates that ferrihydrite is a plausible hematite precursor in Gale crater. We found that <10 nm hematite crystallites of equant shape could form by ferrihydrite alteration through low-temperature solid-state reactions or in solutions with neutral or slightly alkaline pH. However, an additional mechanism is required to explain the larger crystallites in the Murray formation.

Ostwald ripening of Murray formation hematite

Hematite crystallites in the Murray formation have log-normal size distributions (Fig. 3A). The samples with the largest hematite crystallites (Confidence Hills and Oudam) from the lowest stratigraphic levels

lack <10 nm crystallites, whereas all other samples do include such small crystallites (Fig. 3A) (9). We infer that hematite grows by Ostwald ripening, whereby the dissolution of smaller crystallites feeds the growth of larger ones (30). The measured evolution of the log-normal distributions is consistent with theoretical predictions for Ostwald ripening in a closed system (30) (Fig. 3B).

Interpretation of hematite in Gale crater

Our analyses indicated that hematite in the sedimentary rocks in Gale crater could have formed from a single precursor mineral, ferrihydrite, but crystallite growth differed between the Murray formation and the overlying Carolyn Shoemaker and Mirador formations. Figure 4 shows our conceptual model for how this might have occurred.

We propose that ferrihydrite in the Murray and Carolyn Shoemaker formations formed in lacustrine settings, but in the Mirador formation, ferrihydrite formed through interaction with oxidizing groundwater as conditions became drier and water limited. The sedimentary rocks then experienced two phases of diagenesis. During early diagenesis, neutral or slightly alkaline groundwater partially or completely transformed ferrihydrite into hematite and goethite. Small hematite crystallites continued forming from ferrihydrite after sediment drying and lithification under low-temperature [<125°C, (21)] diagenesis (Fig. 4). Goethite likely did not form at this point because low moisture favors hematite crystallization from ferrihydrite (31). Previously formed goethite must have been unaffected because we did not observe the platy hematite that this would produce (Fig. 2D).

We attribute the lack of goethite in the Murray formation to its removal during late diagenesis. Goethite transforms to hematite in warm ($\geq 50^\circ\text{C}$), neutral to alkaline (pH ~7 to 12) solutions (9, 32, 33). Similarly warm, late diagenetic fluids have been proposed to explain hematite coarsening in the Murray formation (26). We hypothesize that these warm fluids removed goethite (or lowered its abundance below the CheMin detection limit), followed by hematite precipitation (Fig. 4). This late diagenesis favored hematite coarsening by Ostwald ripening in a closed system by facilitating the dissolution of small crystallites and the uptake of Fe(III) into larger crystallites. The measured variable degree of coarsening in the Murray formation (Fig. 1B) could reflect differences in water activity

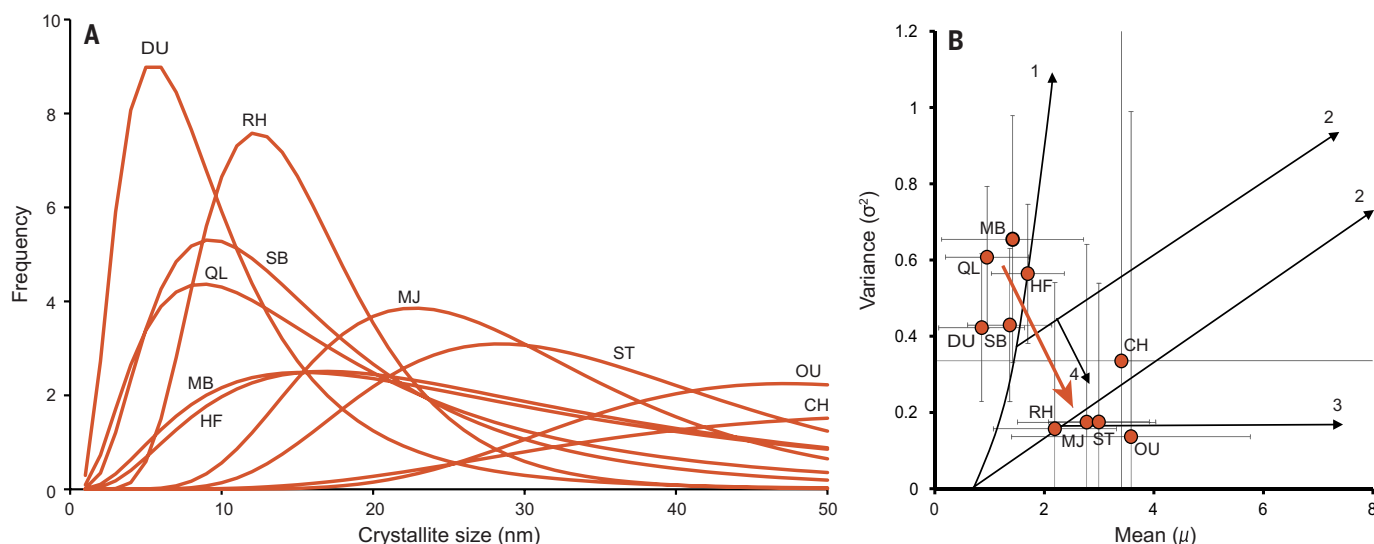


Fig. 3. Size distributions of hematite crystallites. (A) Volume-weighted size distributions of hematite crystallites derived from the XRD model fitting (9). Each line represents a different sample location (labeled as in Fig. 1). (B) The mean (μ) and variance (σ^2) of number-weighted log-normal models fitted to each distribution. Error bars are 1σ (table S2). Numbered arrows indicate the expected trends for different growth mechanisms: continuous nucleation and growth (1) and surface-controlled growth (2), both in an open system, and random ripening (3) and Ostwald ripening (4), both in a closed system (30). The directional trend (orange arrow) from smaller to larger crystallites is consistent with Ostwald ripening (arrow 4).

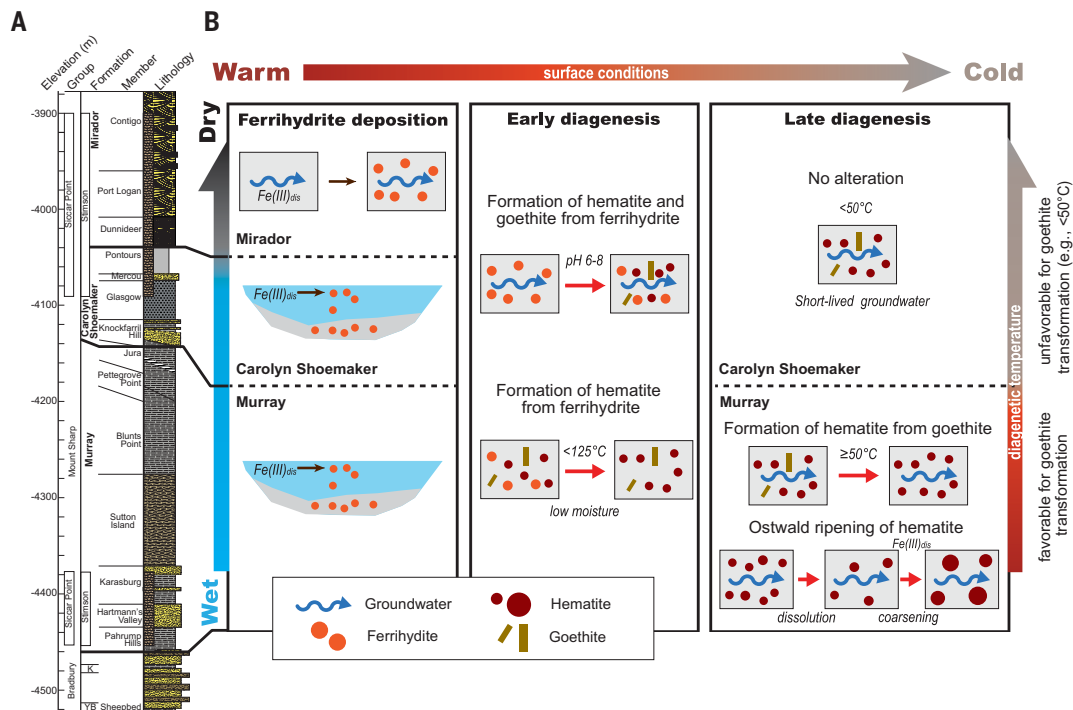


Fig. 4. Proposed conceptual model of hematite formation and evolution in Gale crater. (A) Stratigraphic column from Fig. 1A showing boundaries between the Murray, Carolyn Shoemaker, and Mirador formations (black lines). (B) Left, ferrihydrite formation from dissolved $\text{Fe(III)}_{\text{ds}}$ and deposition. The vertical arrow indicates a transition from wet (blue) to dry (dark gray) surface conditions, reflecting a shift from lacustrine deposition in the Murray and Carolyn Shoemaker formations to groundwater-dominated settings in the Mirador formation. Middle, early diagenesis produced small hematite crystallites and goethite from ferrihydrite. Right, late diagenesis. The vertical arrow shows decreasing diagenetic temperatures from warmer conditions (red), favoring goethite-to-hematite transformations and hematite coarsening in the Murray formation, to colder conditions (gray) that inhibit further alteration. The horizontal arrow (top) indicates cooling surface temperatures from $\geq 0^\circ\text{C}$ to present-day conditions. Dotted horizontal lines are boundaries between formations. Red arrows indicate warm conditions. Shapes indicate hematite, ferrihydrite, goethite, and groundwater (see legend).

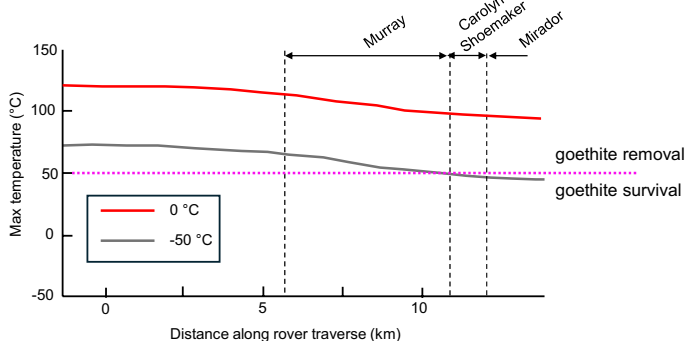


Fig. 5. Maximum burial temperatures experienced in Gale crater. Colored lines are results from previous thermal modeling of sedimentary rocks along the Curiosity traverse (21). The red line represents warm early Mars and the gray line represents present-day cold conditions (surface temperatures of 0° and -50°C , respectively) (9). The pink dotted line at 50°C marks the lowest temperature at which goethite transforms to hematite in laboratory experiments (9). Vertical dotted lines are the boundaries between formations, labeled above. Under warm surface conditions, goethite would transform into hematite in all three formations, contrary to its detection in the Carolyn Shoemaker and Mirador formations. Under cold conditions, goethite remains stable above the Murray formation, in agreement with the rover data.

frequency, duration, or solution pH. We estimate that the time necessary for crystallites to grow from the size of Duluth crystallites ($\sim 5\text{ nm}$) to the size of Confidence Hills crystallites ($\sim 65\text{ nm}$) was 1.1 to 4.7 million years (Ma) at pH 7 and ~ 4 days to 27 years at pH 2 and 50°C , dependent on the solution composition (9, 34). The calculated time reflects the total duration of water presence, whether continuous or episodic. A minimum pH of ~ 7 is necessary for concurrent goethite alteration and hematite coarsening. Under this scenario, warm groundwater might have persisted for up to $\sim 4.7\text{ Ma}$, at least in the deepest layers of Gale crater. Such long-lived aquifers could have been potentially habitable, although other criteria are also required for habitability.

Goethite is still present in the Carolyn Shoemaker and Mirador formations, indicating that lower temperatures ($< 50^\circ\text{C}$) prevented its transformation into hematite (9). Adopting a thermal model of the sedimentary rocks along the Curiosity traverse (20), we suggest that this difference in rock temperatures could reflect the shift of Mars' climate from warmer (surface temperatures $\geq 0^\circ\text{C}$) to the colder conditions that persist today (Fig. 5) (9). We attribute the presence of $< 10\text{ nm}$ hematite crystallites in these formations to a combination of the shorter duration of water activity and the climate transition to drier and colder conditions compared with the formation and evolution of hematite in the Murray formation (Fig. 4).

The proposed model is an extension of our hypothesis that in the Murray formation, goethite formed with hematite and was then removed by diagenesis. Alternatively, hematite alone could have formed

from ferrihydrite in saline solutions (35), as previously inferred in Gale crater (19). We cannot distinguish between these possibilities.

Hematite as a climate change marker

The hematite crystallographic characteristics, and its co-occurrence with goethite, reflect diagenetic and climate conditions on ancient Mars. In the Carolyn Shoemaker and Mirador formations, small hematite crystallites coexist with goethite, indicating lower burial temperatures and shorter duration of water exposure than in the Murray formation. The latter experienced warmer and wetter conditions, resulting in goethite loss and hematite coarsening. We conclude that hematite growth persisted for up to 4.7 Ma total exposure to groundwater at near-neutral pH, either intermittently or continuously.

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SUPPLEMENTARY MATERIALS

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Materials and Methods; Supplementary Text; Figs. S1 to S36; Tables S1 to S8; References (36–66)
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Hematite is a mineralogical marker of ancient climate change on Mars

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Editor's summary

Ancient river channels on Mars demonstrate that it once had a wetter and warmer climate. The Curiosity rover has been investigating the sedimentary rocks in Gale crater, and a mineralogical marker could relate that stratigraphy to the ancient climate. Szczerba *et al.* analyzed microscopic iron oxide crystals in rocks examined by the rover and compared the crystallographic properties with laboratory experiments. The shape of the crystals indicates the formation mechanism, and the presence or absence of a related mineral depends on the subsequent subsurface temperature. The authors argue that a measured transition in the crystal properties coincides with the ancient climate change. — Keith T. Smith

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