Synthesis and Magnetic Characterization of Siderite Particles with Varying Morphologies: Implication for Rock Magnetic Properties

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Introduction

Siderite is an iron(II) carbonate ($FeCO_3$) mineral commonly found in a range of natural environments, including anaerobic aqueous ecosystems, systems containing biomineralization and extraterrestrial formations.[1-5]

Siderite can be identified by a characteristic magnetic transition at low temperatures below 40 K.[1,2] It is important to be able to detect siderite through low temperature rock magnetic analysis due to its ability to undergo phase transformation to magnetite under certain conditions.[2] It can also complicate carbon and oxygen isotopic analyses in natural rock samples.[6] Additionally, it may greatly assist in the identification of biomineralization processes and paleoclimates.[4]

The goal of this project was to synthesize and characterize siderite particles of varying morphologies and grain sizes. Using X-ray diffraction (XRD), scanning electron microscopy (SEM), and Magnetic Properties Measurement System (MPMS) analyses, we aim to determine trends between particle morphology, grain size, and rock magnetic properties.

Experimental

The following synthetic procedure[4] was completed at varying initial Fe(II) concentrations: 0.033 M, 0.067 M, and 0.10 M.

- One solution of 0.2 M sodium carbonate (Na_2CO_3) was combined with a solution containing 0.2 M ascorbic acid and varying concentrations of ferrous sulfate ($FeSO_4$) in an autoclave with a total volume of 60 mL.
- Particle suspension was aged for 4 hours at 130 °C. Once removed from oven, suspension was cooled overnight to room temperature.
- The suspension was then purified using dialysis until particles settled from solution.

Discussion

At different initial Fe(II) concentrations, there was a difference in particle morphology, particle size, and magnetic data obtained.

- SEM confirms radial growth texture consistent with naturally occurring siderite.
- XRD confirms pure siderite samples when the suspension was aged at 130 °C in the autoclave.
- MPMS data is inconsistent between various concentration trials and current literature. There is a shift in Néel temperature (from <40 K to >50 K) from previous literature.[2,3]
- Higher Fe(II) concentrations result in increased initial growth of particle seeds. This contributed to a greater variety in morphologies, but particles are smaller and have less growth due to resources being used more quickly.
- Lower Fe(II) concentrations result in fewer particle seeds. This contributed to less variety of morphologies but larger particles, due to more growth since resources are used slowly.

Future Work

- Perform characterization on a siderite standard for comparison
- Determine if drying effects are present
- Conduct a time series to determine size dependency of the Néel temperature
- Determine single domain size
- Apply Mössbauer spectroscopy to determine any presence of Fe(III)
- Perform synthesis with different reducing agents

Results

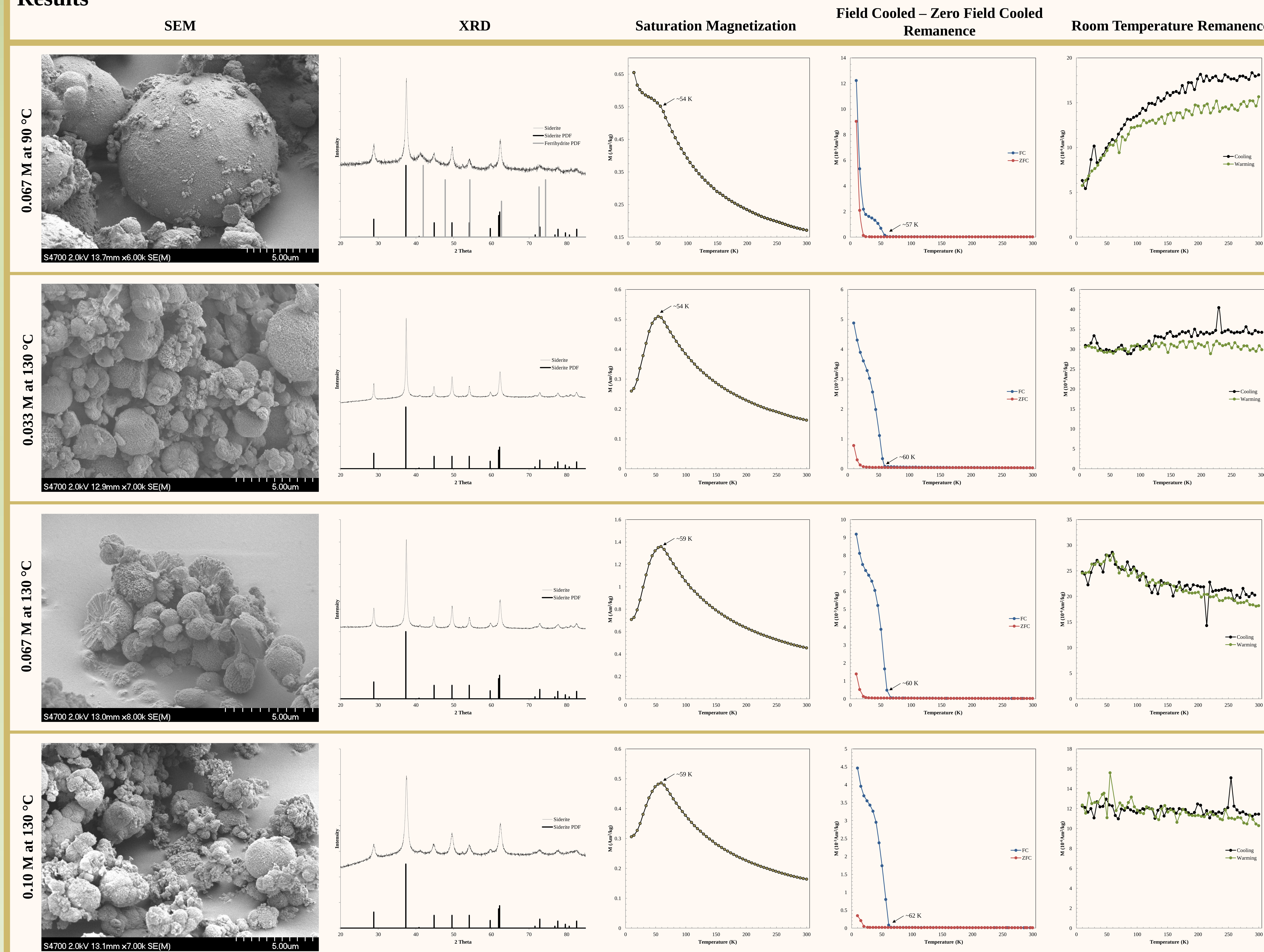

Figure 1: Analysis of siderite particles at various temperatures and initial Fe(II) concentrations. (left to right) Results of SEM, XRD, saturation magnetization, field cooled and zero-field cooled remanence and room temperature remanence. (top to bottom) Concentration and temperature series including 0.067 M Fe(II) at 90 °C, 0.033 M Fe(II) at 130 °C, 0.067 M Fe(II) at 130 °C and 0.10 M Fe(II) at 130 °C.

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