Empirical constraints on progressive shock metamorphism of magnetite from the Siljan impact structure, Sweden

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ABSTRACT
Little is known about the microstructural behavior of magnetite during hypervelocity impact events, even though it is a widespread accessory mineral and an important magnetic carrier in terrestrial and extraterrestrial rocks. We report systematic electron backscatter diffraction crystallographic analysis of shock features in magnetite from a transect across the 52-km-diameter ca. 380 Ma Siljan impact structure in Sweden. Magnetite grains in granitoid samples contain brittle fracturing, crystal-plasticity, and lamellar twins. Deformation twins along [111] with shear direction of <112> are consistent with spinel-law twins. Inferred bulk shock pressures for the investigated samples, as constrained by planar deformation features (PDFs) in quartz and shock twins in zircon, range from 0 to 20 GPa; onset of shock-induced twinning in magnetite is observed at ≳5 GPa. These results highlight the utility of magnetite to record shock deformation in rocks that experience shock pressures ≳5 GPa, which may be useful in quartz-poor samples. Despite significant hydrothermal alteration and the variable transformation of host magnetite to hematite, shock effects are preserved, which demonstrates that magnetite is a reliable mineral for preserving shock deformation over geologic time.

INTRODUCTION
Magnetite, with an inverse spinel structure and nominal chemical formula of Fe²⁺Fe³⁺O₄²⁻, is a common accessory mineral in igneous, sedimentary, and metamorphic rocks (e.g., Deer et al., 1992). It forms under a wide range of conditions and is one of the most important magnetic carriers in both terrestrial and extraterrestrial rocks (e.g., Dunlop and Ozdemir, 1997; Louzada et al., 2011). Magnetite, alongside other ferromagnetic minerals, defines magnetic anomalies associated with impact craters on Earth and other bodies that are essential to the discovery and mapping of such features (e.g., Pilkington and Grieve, 1992). Deformation can modify the magnetic signature of rocks on Earth, e.g., in tectonic pseudotachylites (e.g., Ferré et al., 2005), and shock deformation may permanently alter the intrinsic magnetic properties of rocks (e.g., Gilder et al., 2004; Kletetschka et al., 2004; Gattacceca et al., 2007; Reznik et al., 2016). Yet, detailed microstructural characterization of shock deformation in magnetite is limited to one experimental study (Reznik et al., 2016).

We used scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) analysis to conduct a systematic assessment of shock microstructures in magnetite from the Siljan impact structure. We document microstructural shock effects in magnetite from granitoid rocks from the central uplift of a large (>50 km diameter) impact structure. As the samples have well-constrained shock pressure estimates based on systematic studies of quartz (Holm et al., 2011), we are able to correlate progressive deformation of magnetite with increasing shock pressure.

RESULTS
Up to 10–20 magnetite grains were surveyed in each sample using backscattered electron (BSE) imaging, and orientation mapping using EBSD was conducted on two to six

¹Supplemental Material. EBSD data and additional description of methods. Please visit https://doi.org/10.1130/GEOL.S.17072681 to access the supplemental material, and contact editing@geosociety.org with any questions.

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representative magnetite grains per sample. The grains investigated are subhedral to euhedral and 90–800 µm in size. Grains are often intergrown with biotite and/or amphibole, accessory apatite, and zircon. In samples 21, 58, and 5, magnetite is partly altered to hematite, which is consistent with the observed alteration of biotite and amphibole to chlorite (Fig. S1). Orientation analysis of magnetite-hematite intergrowths shows that the transformation is controlled crystallographically, with \{111\}_\text{mag} aligned with basal \{0001\}_\text{hem} and \langle 110 \rangle_\text{mag} aligned with \langle 1010 \rangle_\text{hem} (Figs. S4 and S5). The transformation results in four hematite orientation variants systematically aligned with one another, which are readily discernable in pole figures and consistent with a topotactic martitization transformation (e.g., Barbosa and Lagoeiro, 2010).

**Deformation Microstructures in Magnetite**

Non-planar and planar brittle fractures occur in all investigated magnetite grains from shocked and unshocked samples (Figs. 2 and 3). Fractures are generally not associated with

![Simplified geologic map of the Siljan impact structure in Sweden](https://apps.sgu.se/kartvisare/kartvisare-berg-50-250-tusen.html)

**TABLE 1. SAMPLES INVESTIGATED IN THIS STUDY**

| Sample number | Lithology* | Distance from geographical center (km) | Coordinates† | Pressure range (GPa)‡ | Average pressure (GPa)§ | No. of investigated magnetite grains/ aggregates in sample | Magnetite observations†† |
|---------------|------------|----------------------------------------|--------------|-----------------------|-------------------------|----------------------------------------------------------|--------------------------|
| 69            | Hornblende-biotite granite | 1.7 | N61°03.051′; E014°53.612′ | 15–20 | 16 | 6 | Twins (4) |
| 21            | Hornblende-biotite granite | 8.9 | N60°59.569′; E014°46.780′ | 10–15 | 10.6 | 3 | Twins (3) |
| 58            | Hornblende-biotite granite | 9.2 | N60°57.409′; E014°54.355′ | 5–10 | 7.2 | 2 | Twins (2) |
| 5             | Hornblende-biotite granite | 27.4 | N60°53.270′; E015°18.863′ | No shock** | No shock** | 3 | No twins |

**Note:** *All samples are coarse-grained, porphyritic granitoids that contain alkali feldspar, quartz, plagioclase, hornblende, and biotite with minor magnetite, titanite, zircon, and apatite.

†Coordinates are in Swedish Reference Frame 1999 (SWEREF 99) (WGS 84).

‡Pressure range estimate; see Holm et al. (2011).

§Pressure estimate as an average calculated from pressure values of each individual quartz grain in the sample, based on the planar deformation feature (PDF) population in each grain; see Holm-Alwmark et al. (2018).

**Sample is from outside of zone of PDF occurrences in quartz and shatter cone observations.

††Maximum number of sets of twins observed in grains per sample.

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Figure 2. (A) Backscattered electron (BSE) image of shocked magnetite aggregate (sample 69, grain #1) intergrown with zircon. Ap—apatite; Afs—alkali feldspar; Bt—biotite; Mag—magnetite; Pl—plagioclase; Qz—quartz; Zrn—zircon. (B) Cumulative misorientation map showing up to 14° variation across the host and twin boundaries in red. (C) Inverse pole figure (IPF) z orientation map. (D) High-resolution map of shocked zircon exhibiting one set of shock twin lamellae. Arrows indicate twin orientations in both zircon (zt) and magnetite (t1–t3). (E) Magnetite pole figures plotted as equal area, lower hemisphere projections. (F) Crystallographic relationships between twin and host for twins in magnetite and zircon.
detectable lattice misorientation, although exceptions include areas with high-density sets of straight, parallel and closely spaced fractures, which occur in shocked samples. Orientation mapping shows the straight fractures coincide with twin boundaries. Twin lamellae, the most conspicuous microstructure observed, are only present in the three shocked samples (Table 1; Fig. 2). Twins are polysynthetically and occur as up to four sets of straight lamellae. Twins generally cross each other without apparent offset, except in some instances where planar fractures along twin planes appear to offset other twins (Fig. 2C). Twins have apparent widths of up to 5 μm and crosscut the full length of the host crystal with some showing tapered terminations. The apparent thickness of twins varies among twin sets in the same grain, in the same twin orientation, and between grains, depending on the angle of intersection with the polished section. Individual twins can be separated by as little as 2–3 μm.

Twin lamellae and host crystals share a systematic orientation relationship defined by a misorientation (i.e., minimum misorientation) of 60° about [111]. This orientation relationship results in the coincidence of several [112] crystallographic directions between host and twin that lie normal to the 180° misorientation axis (Fig. 2F). The 180° misorientation axis aligns with the pole to [111], which is consistent with spinel-law twinning, with a [111] compositional/invariant plane (K1) and a <112> shear direction (τ1).

Magnetite crystals exhibit intragrain cumulative misorientation of up to 18° as evidenced by dispersion in pole figures (e.g., Fig. 2E). Internal misorientation is frequently most concentrated near grain boundaries or between twin lamellae. Crystal plasticity has also resulted in the formation of deformation bands with indistinct boundaries in some crystals (Fig. 3). Well-defined deformation bands have systematic crystallographic disorientation about <111> (Fig. 2E; Fig. S6A).

Microstructures in Zircon

In sample 69, some magnetite grains are intergrown with zircon. Two zircon grains (Fig. 2; Fig. S6E) exhibit [112] mechanical twin lamellae, disoriented 65° about <110>. This twin type has only been reported in shocked zircon (e.g., Erickson et al., 2013).

DISCUSSION

Shock-Induced Microstructures in Magnetite

Several types of microstructures, most notably deformation twinning, were observed in magnetite from the Siljan impact structure. Magnetic inclusions with shocked quartz show closely spaced sets of straight, lamellar twins in single or multiple orientations (Fig. 2). Twinning is one of the major deformation modes that enable minerals to change shape in response to shock wave passage through geological materials (e.g., Christian and Mahajan, 1995). Deformation twins have been described in other accessory minerals from shocked rocks including zircon (Erickson et al., 2013), monazite (Erickson et al., 2016), xenotime (Cavosie et al., 2016), and titanite (Timms et al., 2019). Twins described here are consistent with magnetite [111] spinel-law twins and agree with observations of shock-twinning in spinel when the Hugoniot elastic limit (HEL) is exceeded (Schäfer et al., 1983). Twinning in magnetite was not observed outside the zone of shock metamorphism; thus, we conclude that the pervasive lamellar twinning is a shock-induced microstructure.

Under experimental shock-conditions, Reznik et al. (2016) reported fracturing/fragmentation, development of microshear bands (5 GPa), mechanical twins (≥10 GPa), kink-bands (30 GPa), as well as transmission electron microscopy-scale amorphization of magnetite. The microstructural observations were associated with decreasing magnetic susceptibility and increasing coercivity of the investigated crystals. Our observations of twinning in magnetite are broadly consistent with those of that study. In spinel-structured materials, the slip system 1/2 <110> [111] is structurally favored because it comprises the shortest translation vector and the most densely packed lattice plane, operates both in high-temperature and low strain-rate endogenic processes, and is present during shock deformation experiments (Schäfer et al., 1983). In experimentally shocked spinel, Schäfer et al. (1983) reported dislocations that were interpreted as remnants of shock-induced plastic deformation by the slip system 1/2 <110> [111] and [111] mechanical twins. In grains studied here, plastic strain results in systematic misorientation about [111] (Fig. 2F), consistent with the 1/2 <110> [111] slip in spinel-structured minerals.

Few descriptions of natural shock-induced twins in magnetite have been reported. Cloete et al. (1999) reported <1-μm-sized twins in a magnetite inclusion in shocked quartz in granitic gneiss from the Vredefort structure in South Africa, and Timms et al. (2019) reported a grain...
of magnetite with twins in shocked granitoid from the Chicxulub structure in Mexico. Magnetite microstructures formed in shock experiments (Reznik et al., 2016) are on the order of tens of nanometers and thus much smaller in scale than those reported here. The drastic difference in the dimension of twins in our study from the results of Reznik et al. (2016) could result from differences between natural hypervelocity impact events and experimental impact cratering, such as shock pulse duration, geometry of the shock wave, as well as shock impedance, textural features, and the pre-shock temperature of target rocks (e.g., Stöffler, 1972; Huffman and Reimold, 1996; Stöffler et al., 2018; Wittmann et al., 2021). Increasing grain size is known to result in increased volume fraction of microtwins and microbands in metals and alloys (Murr and Esquivel, 2004). However, given the similarity in the size of grains investigated by us and those in Reznik et al. (2016), grain size alone cannot explain the observed differences in shock-induced twinning.

Shock impedance has recently been described as the cause of localized pressure amplification for zircon enclosed in less dense minerals (Wittmann et al., 2021). We did not observe variations in magnetite twins based on different surrounding phases (typically biotite, plagioclase, hornblende, or titanite), but given the high density of magnetite, local pressure amplifications beyond mean pressure estimates may have occurred.

Chronology of Shock Features

Twin formation in the magnetite grains appears unaffected by non-planar fractures, as we observed no abrupt terminations of twins at fractures. Instead, twins are, in some instances, offset by both planar and non-planar fractures, which suggests the fractures post-date twin formation (Figs. 2C and 3; Fig. S6C). Planar fractures may form when twin planes open as tensile cracks during pressure release (e.g., similar to the opening of feather features in quartz; Poelchau and Kenkmann, 2011). Plastic strain is constrained to the host crystal between twin sets, and crosscutting twins and lamellae, indicating that dislocation slip was active during twinning and after.

Magnetite in sample 69 appears relatively unaffected by alteration; however, grains in samples 21 and 58 are highly altered (alteration was also observed in sample 5; Figs. S4 and S5). Hematite alteration appears to utilize twin planes and fractures for fluid ingress and crosscuts some twins. Martitization is interpreted to have occurred in hydrothermal systems within the newly formed crater (e.g., Hode et al., 2003). Impact craters on Earth, and other bodies such as Mars, have been subject to hydrothermal alteration (e.g., Osniski et al., 2013). Despite the extensive alteration of Siljan magnetite, preserved shock features were identified in all samples from the zone of shock metamorphism, indicating that shocked magnetite can be stable for hundreds of millions of years even in hydrothermally altered rocks.

Progressive Shock Metamorphism of Magnetite Recorded by Lamellar Twins

The number of twin sets in Siljan magnetite correlates with distance from the crater center and thus with intensity of shock deformation (Fig. 3). Magnetite grains from sample 69 (15–20 GPa) exhibit up to four sets of twin lamellae, whereas samples subjected to lower shock levels record one to two (rarely three) sets of twin lamellae per grain. Thus, as the magnetite lattice is subjected to higher stress (higher shock pressure), load release happens through twinning in more directions than at lower stress (lower shock pressure). A similar explanation was suggested for the increasing number of twins in shocked monazite grains (Erickson et al., 2016), where the number of twins per grain was suggested to be analogous to PDFs in quartz, which exhibit a greater number of PDF orientations at higher shock pressures (e.g., Holm-Alwmark et al., 2018). The largest magnitude of plastic strain (18° of cumulative misorientation) was observed in grains from sample 69, located closest (1.7 km) to the crater center.

Significance of Shock-Induced Grain-Size Deformation of Magnetite

The volume of rock that experiences low pressure (<5–10 GPa) during impact is two to three times larger than that which is subject to higher pressures (Poelchau and Kenkmann, 2011). Additionally, in eroded or small impact craters, low-pressure deformation may be all that is available for study, especially once ejecta has been removed. Therefore, there is a need to understand shock effects in the pressure range between quasi-static deformation at the HEL and high shock-pressure indicators (e.g., Poelchau and Kenkmann, 2011). While [111] twins in magnetite are not considered diagnostic impact features here, they can be used to characterize the extent of shock deformation in rocks with a known shock provenance, and thus to constrain the effects of shock wave propagation and attenuation. Since magnetite occurs in a wide range of rock types, it can also be used as a shock barometer in rocks that generally lack quartz (e.g., limestone, gabbro, and serpentinite).

We have shown that mechanical twins, plasticity, and fracturing are key features of magnetite in target rocks across a relatively wide range of shock pressures, which first appear at low shock pressures (>5 GPa; average 7.2 GPa). Magnetic domain structures in magnetite are sensitive to lattice defects and modifications caused by plastic deformation (twinning) and other internal stress (e.g., Dunlop and Odzemir, 1997). Twins within experimentally shocked magnetite reported by Reznik et al. (2016) contributed to increasing coercivity in their material. Our results, thus, illuminate processes that can result in impact-modification of magnetic fabrics in crustal rocks on terrestrial planets.

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