

1 Empirical Constraints on Progressive Shock Metamorphism of  
2 Magnetite from the Siljan Impact Structure, Sweden

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12

13 **ABSTRACT**

14 Little is known about the microstructural behavior of magnetite during hypervelocity  
15 impact events, even though it is a widespread accessory mineral and important magnetic carrier  
16 in terrestrial and extraterrestrial rocks. We report systematic electron backscatter diffraction  
17 crystallographic analysis of shock features in magnetite, from a transect across the 52-km-  
18 diameter ~380 Ma Siljan impact structure in Sweden. Magnetite grains in granitoid samples  
19 contain brittle fracturing, crystal-plasticity, and lamellar twins. Deformation twins along {111}  
20 with shear direction of <112> are consistent with spinel-law twins. Inferred bulk shock pressures  
21 for investigated samples, as constrained by planar deformation features (PDFs) in quartz and  
22 shock twins in zircon, range from 0–20 GPa; onset of shock-induced twinning in magnetite is

23 observed at >5 GPa. These results highlight the utility of magnetite to record shock deformation  
24 in rocks that experience shock pressures >5 GPa, which may be useful in quartz-poor samples.  
25 Despite significant hydrothermal alteration, and variable transformation of host magnetite to  
26 hematite, shock effects are preserved, demonstrating that magnetite is a reliable mineral for  
27 preserving shock deformation over geologic time.

28

29

## 30 **INTRODUCTION**

31 Magnetite, with an inverse spinel structure and nominal chemical formula of  $\text{Fe}_2^{3+}\text{Fe}^{2+}\text{O}_4^{2-}$ , is a  
32 common accessory mineral in igneous, sedimentary, and metamorphic rocks (e.g., Deer et al.,  
33 1992). It forms under a wide range of conditions and is one of the most important magnetic  
34 carriers in both terrestrial and extraterrestrial rocks (e.g., Dunlop and Özdemir, 1997; Louzada et  
35 al., 2011). Magnetite, alongside other ferromagnetic minerals, defines magnetic anomalies  
36 associated with impact craters on Earth and other bodies that are essential to the discovery and  
37 mapping of such features (e.g., Pilkington and Grieve, 1992). Deformation can modify the  
38 magnetic signature of rocks on Earth, e.g., in tectonic pseudotachylites (e.g., Ferré et al., 2005),  
39 and shock deformation may permanently alter intrinsic magnetic properties of rocks (e.g., Gilder  
40 et al., 2004; Kletetschka et al., 2004; Gattacceca et al., 2007; Reznik et al., 2016). Yet, detailed  
41 microstructural characterization of shock deformation in magnetite is limited to one experimental  
42 study (Reznik et al., 2016).

43 Here we use scanning electron microscopy (SEM)-based electron backscatter diffraction  
44 (EBSD) analysis to conduct a systematic assessment of shock microstructures in magnetite from  
45 the Siljan impact structure. We document microstructural shock effects in magnetite from

46 granitoid rocks from the central uplift of a large (>50 km diameter) impact structure. As the  
47 samples have well-constrained shock pressure estimates based on systematic studies of quartz  
48 (Holm et al., 2011), we are able to correlate progressive deformation of magnetite with  
49 increasing shock pressure.

50

## 51 **GEOLOGICAL SETTING, SAMPLES AND METHODS**

52 Siljan is a ~52 km-diameter impact structure located in Sweden (Fig. 1) which formed  $380.9 \pm$   
53 4.6 Myr ago (Reimold et al., 2005; Jourdan, 2012; Holm-Alwmark, 2021, and references  
54 therein). Target rocks are crystalline basement rocks that were overlain by a ~3 km thick  
55 sequence of sedimentary rocks (Holm-Alwmark et al., 2017). Siljan consists of a 32-km wide  
56 central plateau surrounded by an annular depression partly filled by lakes. The area has not  
57 undergone any large-scale post-impact tectonic deformation. Four granitoid samples from  
58 outcrop in the central plateau of Siljan were investigated (Fig. 1; Table 1; DR Item 1). Samples  
59 69, 21, and 58 were previously assigned to a shock pressure range of 5 to 20 GPa based on PDFs  
60 in quartz (Holm et al., 2011; Holm-Alwmark et al., 2018). Sample 5 is from outside the annular  
61 depression, beyond the domain of shock metamorphism.

62 Mineral phase information, crystallographic orientation, and microstructural data by  
63 EBSD were collected with a Tescan Mira3 field emission SEM equipped with an Oxford  
64 Instruments EBSD detector. Details of operating conditions are specified in DR Items 2, 7.

65

## 66 **RESULTS**

67 Up to 10-20 magnetite grains were surveyed in each sample by backscattered electron (BSE)  
68 imaging, and orientation mapping using EBSD was conducted on 2-6 representative magnetite

69 grains per sample. Investigated grains are subhedral to euhedral and 90 to 800  $\mu\text{m}$  in size. Grains  
70 are often intergrown with biotite and/or amphibole, accessory apatite and zircon. In samples 21,  
71 58, and 5, magnetite is partly altered to hematite, consistent with observed alteration of biotite  
72 and amphibole to chlorite (DR Item 1). Orientation analysis of magnetite-hematite intergrowths  
73 shows that the transformation is controlled crystallographically, with  $\{111\}_{\text{mag}}$  aligned with basal  
74  $\{0001\}_{\text{hem}}$ , and  $\langle 110 \rangle_{\text{mag}}$  aligned with  $\langle 10\bar{1}0 \rangle_{\text{hem}}$  (DR Item 4, 5). The transformation results in  
75 four hematite orientation variants systematically aligned with one another, which are readily  
76 discernable in pole figures, and consistent with a topotactic martitization transformation (e.g.,  
77 Barbosa & Lagoeiro, 2010).

78

#### 79 **Deformation microstructures in magnetite**

80 Non-planar and planar brittle fractures occur in all investigated magnetite grains from shocked  
81 and unshocked samples (Figs. 2, 3). Fractures are generally not associated with detectable lattice  
82 misorientation, although exceptions include areas with a high density of sets of straight, parallel,  
83 closely spaced fractures, which occur in shocked samples. Orientation mapping shows the  
84 straight fractures coincide with twin boundaries. Twin lamellae are the most conspicuous  
85 microstructure observed, and are only present in the three shocked samples (Table 1; Fig. 2).  
86 Twins are polysynthetic, occurring as up to four sets of straight lamellae. Twins generally cross  
87 each other without apparent offset, except in some instances where planar fractures along twin  
88 planes appear to offset other twins (Fig. 2C). Twins have apparent widths up to 5  $\mu\text{m}$ , crosscut  
89 the full length of the host crystal, and some show tapered terminations. The apparent thickness of  
90 twins varies between twin sets in the same grain, in the same twin orientation, and between

91 grains, depending on angle of intersection with the polished section. Individual twins can be  
92 separated by as little as 2-3  $\mu\text{m}$ .

93 Twin lamellae and host crystals share a systematic orientation relationship defined by a  
94 disorientation (i.e., minimum misorientation) of  $60^\circ$  about  $\langle 111 \rangle$ . This orientation relationship  
95 results in coincidence of several  $\langle 112 \rangle$  crystallographic directions between host and twin that lie  
96 normal to the  $180^\circ$  misorientation axis (Fig. 2f). The  $180^\circ$  misorientation axis aligns with the  
97 pole to  $\{111\}$ , consistent with spinel-law twinning, with a  $\{111\}$  compositional/invariant plane  
98 (K1) and a  $\langle 112 \rangle$  shear direction ( $\eta_1$ ).

99 Magnetite crystals exhibit intragrain cumulative misorientation, up to  $18^\circ$  as evidenced  
100 by dispersion in pole figures (e.g., Fig. 2e). Internal misorientation is frequently most  
101 concentrated near grain boundaries or between twin lamellae. Crystal plasticity has also resulted  
102 in formation of deformation bands with indistinct boundaries in some crystals (Fig. 3). Well-  
103 defined deformation bands have systematic crystallographic disorientation about  $\langle 111 \rangle$  (Fig. 2e,  
104 DR item 6a).

105

## 106 **Microstructures in zircon**

107 In sample 69, some magnetite grains are intergrown with zircon. Two zircon grains (Fig. 2 and  
108 DR Item 6E) exhibit  $\{112\}$  mechanical twin lamellae, disoriented  $65^\circ$  about  $\langle 110 \rangle$ . This twin  
109 type has only been reported in shocked zircon (e.g., Erickson et al., 2013).

110

## 111 **DISCUSSION**

### 112 **Shock-induced microstructures in magnetite**

113 Several types of microstructures were observed in magnetite from the Siljan impact structure,  
114 most notably deformation twinning. Magnetite grains in samples with shocked quartz show  
115 closely spaced sets of straight, lamellar twins in single or multiple orientations (Fig. 2).  
116 Twinning is one of the major deformation modes that enable minerals to change shape in  
117 response to shock wave passage through geologic materials (e.g., Christian and Mahajan, 1995).  
118 Deformation twins have been described in other accessory minerals from shocked rocks  
119 including zircon (Erickson et al., 2013), monazite (Erickson et al., 2016), xenotime (Cavosie et  
120 al., 2016), and titanite (Timms et al., 2019). Twins described here are consistent with magnetite  
121  $\{111\}$  spinel-law twins, and agree with observations of shock-twinning in spinel when the  
122 Hugoniot elastic limit (HEL) is exceeded (Schäfer et al., 1983). Twinning in magnetite was not  
123 observed outside the zone of shock metamorphism, thus we conclude the pervasive lamellar  
124 twinning is a shock-induced microstructure.

125 Under experimental shock-conditions, Reznik et al. (2016) reported  
126 fracturing/fragmentation, development of microshear bands (5 GPa), mechanical twins ( $\geq 10$   
127 GPa), kink-bands (30 GPa), as well as transmission electron microscopy-scale amorphization of  
128 magnetite. The microstructural observations were associated with decreasing magnetic  
129 susceptibility and increasing coercivity of the investigated crystals. Our observations of twinning  
130 in magnetite are broadly consistent with that study. In spinel-structured materials, the slip  
131 system  $1/2 \langle 110 \rangle \{111\}$  is structurally favored because it comprises the shortest translation  
132 vector and the most densely packed lattice plane, and operates both in high-temperature and low  
133 strain-rate endogenic processes, and during shock deformation experiments (Schäfer et al.,  
134 1983). In experimentally shocked spinel, Schäfer et al. (1983) reported dislocations interpreted  
135 as remnants of shock-induced plastic deformation by the slip system  $1/2 \langle 110 \rangle \{111\}$  and  $\{111\}$

136 mechanical twins. In grains studied here, plastic strain results in systematic misorientation about  
137 {111} (Fig. 2f), consistent with  $1/2\langle 110 \rangle\{111\}$  slip in spinel-structured minerals.

138         Few descriptions of natural shock-induced twins in magnetite have been reported. Cloete  
139 et al. (1999) reported  $<1\ \mu\text{m}$  sized twins in magnetite inclusions in shocked quartz in granitic  
140 gneiss from the Vredefort structure (South Africa), and Timms et al. (2019) reported a grain of  
141 magnetite with twins in shocked granitoid from the Chicxulub structure (Mexico). Magnetite  
142 microstructures formed in shock experiments (Reznik et al., 2016) are on the order of 10s of  
143 nanometers, thus much smaller in scale than those reported here. The drastic difference in  
144 dimension of twins in our study from the results of Reznik et al. (2016) could result from  
145 differences between natural hypervelocity impact events and experimental impact cratering, such  
146 as shock pulse duration, geometry of the shock wave, as well as shock impedance, textural  
147 features, and pre-shock temperature of target rocks (e.g., Stöffler, 1972; Huffman and Reimold,  
148 1996; Stöffler et al., 2018; Wittman et al., 2021). Increasing grain size is known to result in  
149 increased volume fraction of microtwins and microbands in metals and alloys (Murr and  
150 Esquivel, 2004). However, given the similarity in size of grains investigated by us and those in  
151 Reznik et al. (2016), grain size alone cannot explain observed differences in shock-induced  
152 twinning.

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

158

159 **Chronology of shock features**

160 Twin formation in investigated magnetite grains appears unaffected by non-planar fractures, as  
161 we have observed no abrupt terminations of twins at fractures. Instead, twins are in some  
162 instances offset by both planar and non-planar fractures, suggesting the fractures post-date twin  
163 formation (Fig. 2C, 3; DR Item 6C). Planar fractures may form as twin planes open as tensile  
164 cracks during pressure release (e.g., similar to opening of feather features in quartz; Poelchau  
165 and Kenkmann, 2011). Plastic strain is constrained to the host crystal between twin sets, and  
166 crosscutting twins and lamellae, indicating dislocation slip was active during twinning and after.

167 Magnetite in sample 69 appear relatively unaffected by alteration, however, grains in  
168 samples 21 and 58 are highly altered (alteration was also observed in sample 5; DR item 4, 5).  
169 Hematite alteration appears to utilize twin planes and fractures for fluid ingress, as well as  
170 crosscutting some twins. Martitization is interpreted to have occurred in hydrothermal systems  
171 within the newly formed crater (e.g., Hode et al., 2003). Impact craters on Earth, and other  
172 bodies such as Mars, have been subject to hydrothermal alteration (e.g., Osinski et al., 2013).  
173 Despite extensive alteration of Siljan magnetite, preserved shock features were identified in all  
174 samples from the zone of shock metamorphism, indicating that shocked magnetite can be stable  
175 for hundreds of millions of years, even in hydrothermally altered rocks.

176

177 **Progressive shock metamorphism of magnetite recorded by lamellar twins**

178 The number of twin sets in Siljan magnetite correlates with distance from the crater center, and  
179 thus with intensity of shock deformation (Fig. 3). Magnetite grains from sample 69 (15-20 GPa)  
180 exhibit up to four sets of twin lamellae, whereas samples subjected to lower shock levels record  
181 one to two (rarely three) sets of twin lamellae per grain. Thus, as the magnetite lattice is



182 subjected to higher stress (higher shock pressure), load release happens through twinning in more  
183 directions than at lower stress (lower shock pressure). A similar explanation was suggested for  
184 increasing number of twins in shocked monazite grains (Erickson et al., 2016), where the number  
185 of twins per grain was suggested to be analogous to PDFs in quartz, which exhibit a greater  
186 number of PDF orientations at higher shock pressures (e.g., Holm-Alwmark et al., 2018). The  
187 largest magnitude of plastic strain ( $18^\circ$  of cumulative misorientation) was observed in grains  
188 from sample 69, 1.7 km from the crater center.

189

### 190 **Significance of shock-induced grain-scale deformation of magnetite**

191 The volume of rock that experiences low pressure ( $\sim 5$ -10 GPa) during impact is 2-3 times larger  
192 than that which is subject to higher pressures (Poelchau and Kenkman, 2011). Additionally, in  
193 eroded or small impact craters, low-pressure deformation may be all that is available for study,  
194 especially once ejecta has been removed. There is therefore a need to understand shock effects in  
195 the pressure range between quasi-static deformation at the HEL and high shock-pressure  
196 indicators (e.g., Poelchau and Kenkmann, 2011). While  $\{111\}$  twins in magnetite are not here  
197 considered diagnostic impact features, they can be used to characterize the extent of shock  
198 deformation in rocks with a known shock provenance, and thus constrain effects of shock wave  
199 propagation and attenuation. Since magnetite occurs in a wide range of rock types, it can also be  
200 used as a shock barometer in rocks generally lacking quartz (e.g., limestone, gabbro, and  
201 serpentinite).

202 We have shown that mechanical twins, plasticity, and fracturing are important features of  
203 magnetite from target rocks across a relatively wide range of shock pressures, first appearing at  
204 low shock pressures ( $>5$  GPa; average 7.2 GPa). Magnetic domain structures in magnetite are

205 sensitive to lattice defects and modifications, caused by plastic deformation (twinning), and other  
206 internal stress (e.g., Dunlop and Özdemir, 1997). Twins within experimentally shocked  
207 magnetite reported by Reznik et al. (2016) contributed to increasing coercivity in their material.  
208 Our results, thus, illuminate processes that can result in impact-modification of magnetic fabrics  
209 in crustal rocks on terrestrial planets.

210

## 211 **ACKNOWLEDGMENTS**

212 SHA was supported by the Swedish Research Council (grant #2017-06388) and the Royal  
213 Physiographical Society in Lund. Thanks to C. Alwmark for help with resolving SEM-related  
214 issues. Professional editorial handling by M. Norman, and thorough reviews by A. Wittman and  
215 two anonymous reviewers are gratefully acknowledged.

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336

### 337 **FIGURE CAPTIONS**

338 Figure 1. Simplified geologic map of the Siljan impact structure (modified after Swedish  
339 Geological Survey (SGU) bedrock map viewer (2021); Holm et al., 2011; Holm-Alwmark,

340 2021). Sample localities are indicated by black dots and corresponding numbers. Ages from  
341 SGU bedrock map viewer (2021) and Stephens (2020).

342

343 Figure 2. A: BSE image of shocked magnetite aggregate (sample 69, grain #1) intergrown with  
344 zircon. B: Cumulative misorientation map showing up to 14° variation across the host and twin  
345 boundaries in red. C: Inverse pole figure (IPF) z orientation map. D: High-resolution map of  
346 shocked zircon exhibiting one set of shock twin lamellae. Arrows indicate twin orientations in  
347 both zircon (zt) and magnetite (t1-3). E: Pole figures plotted as equal area, lower hemisphere  
348 projections. F: Crystallographic relationships between twin and host for twins in magnetite and  
349 zircon. Pl = plagioclase; Ap = apatite; Afs = alkali feldspar; Mag = magnetite; Bt = biotite; Qz =  
350 quartz; Zrn = zircon.

351

352 Figure. 3. Progressive shock metamorphism of magnetite with distance from the crater center.  
353 Cumulative misorientation maps are shown for representative magnetite in each sample. Special  
354 boundaries (yellow) are consistent with topotactic martitization transformation of magnetite to  
355 hematite. Twin boundaries in samples 21, 58 and 69 are marked in red. A planar deformation  
356 band (PDB) is labeled in sample 58.

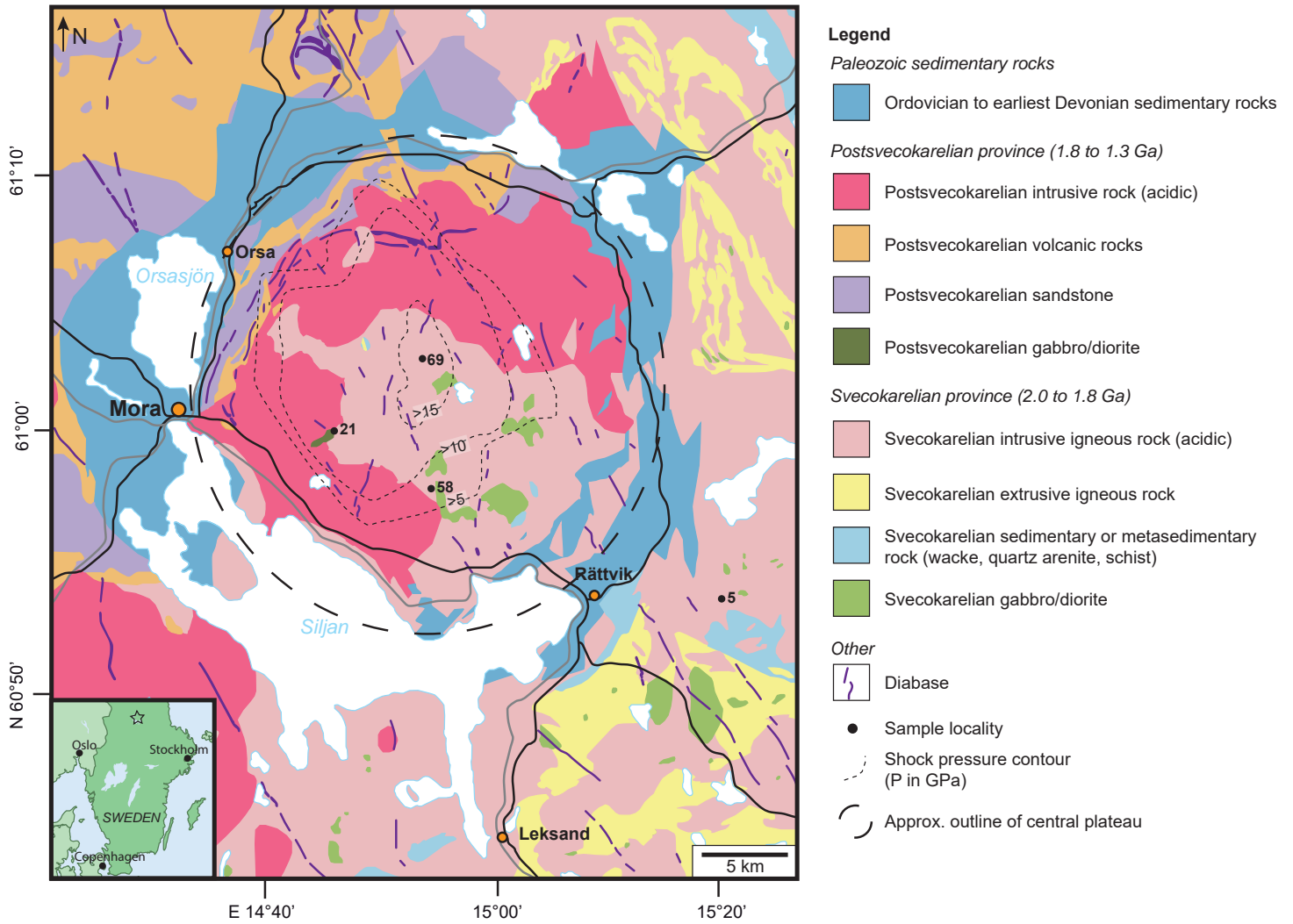
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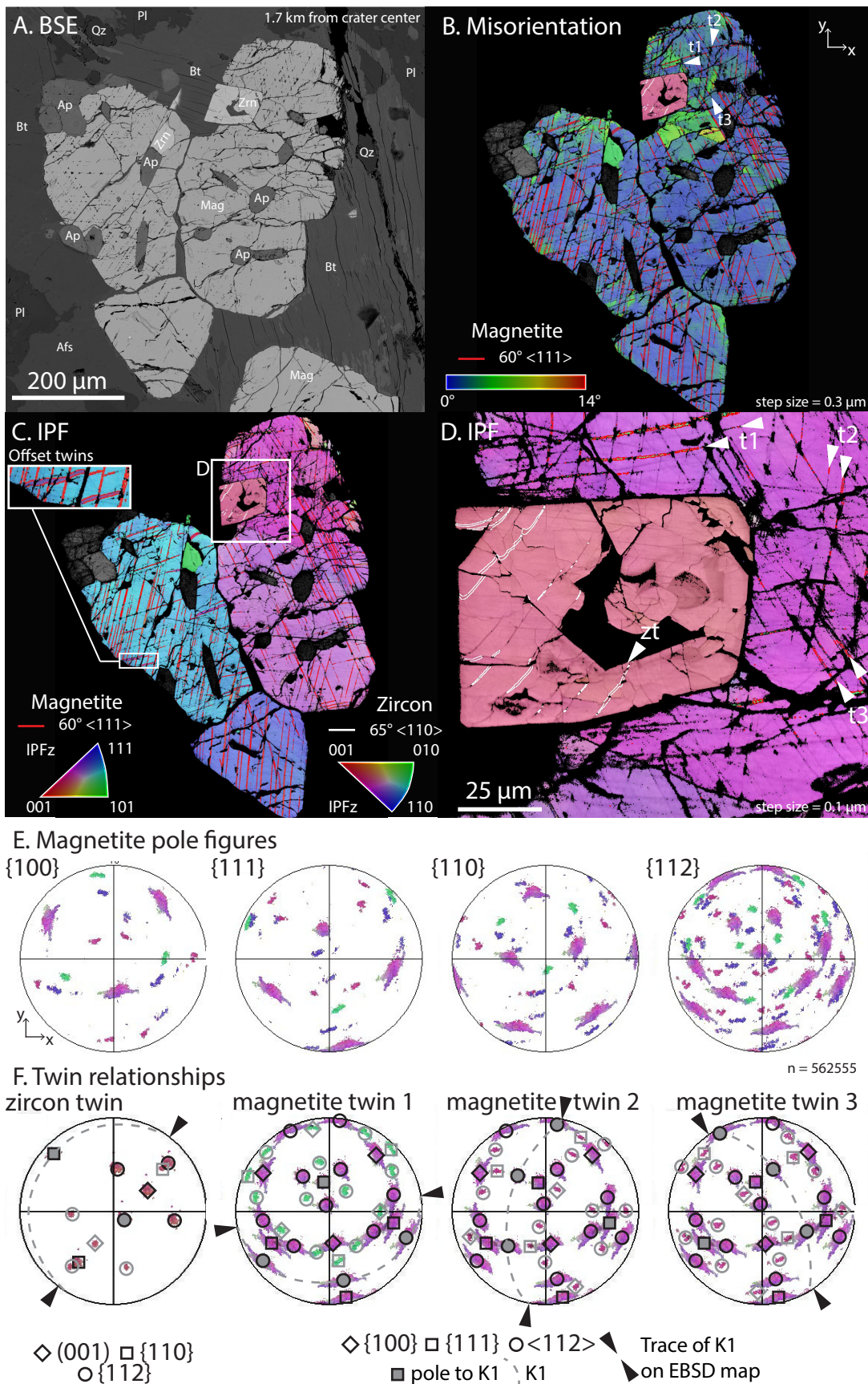
359 <sup>1</sup>GSA Data Repository item 20XXxxx, [EBSD data and additional description of methods], is  
360 available online at [www.geosociety.org/pubs/ft20XX.htm](http://www.geosociety.org/pubs/ft20XX.htm), or on request from  
361 [editing@geosociety.org](mailto:editing@geosociety.org).



# Figure 1



# Figure 2



# Figure 3

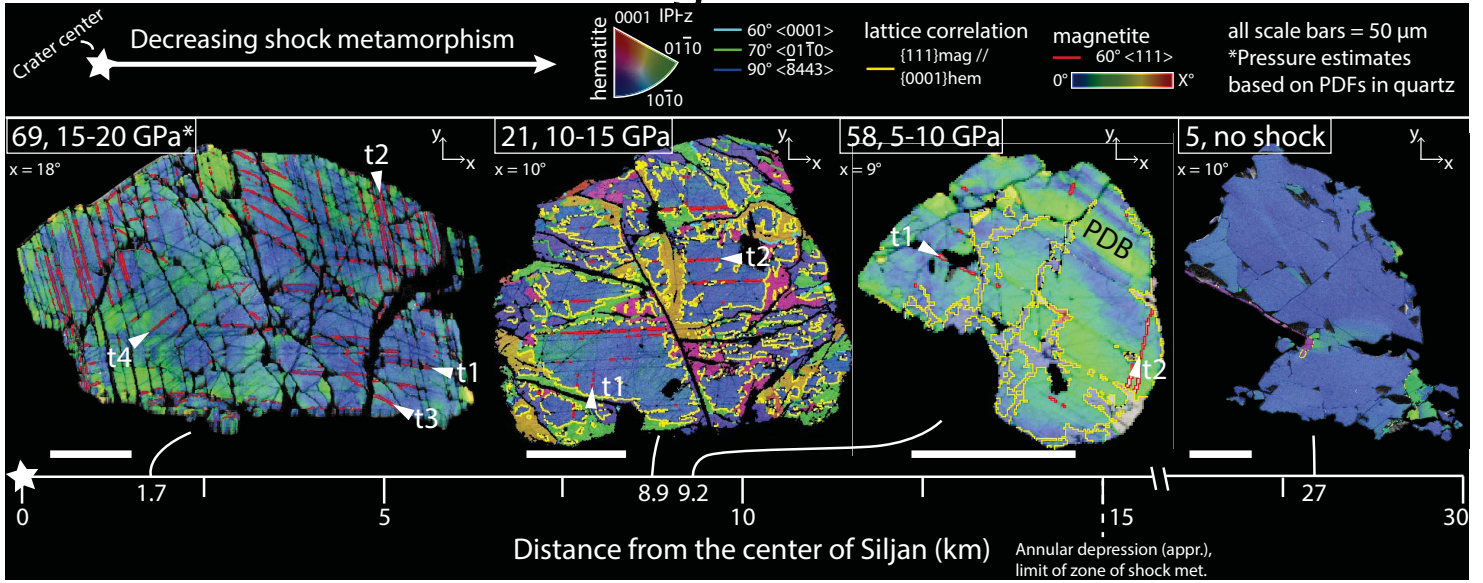


TABLE 1. SAMPLES INVESTIGATED IN THIS STUDY

Sample number	Lithology*	Distance from geographical center (km)	Coordinates <sup>†</sup>	Pressure range (GPa) <sup>§</sup>	Avg. pressure (GPa) <sup>#</sup>	No. of investigated magnetite grains/aggregates in sample	Magnetite observations (Maximum number of sets of twins observed in grains per sample)
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°46.760'	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

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

<sup>†</sup>Coordinates are in SWEREF 99 (WGS 84).

<sup>§</sup>Pressure range estimate, see Holm et al. (2011).

<sup>#</sup>Pressure estimate as an average calculated from pressure values of each individual quartz grain in the sample, based on the 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.