Inversion twinning in troilite

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ABSTRACT

The crystal structure of troilite from chondrites Etter and Georgetown and a troilite analog synthesized by sulfurization of an iron wire was refined using single-crystal X-ray data. Troilite is known to be hexagonal, with space group $P\bar{6}2c$, which is non-centrosymmetric, allowing two non-identical inversely related spatial arrangements of atoms within the unit cell. All three samples represent the so-called inversion twins. They contain both inversely related atomic orientations instead of a single atomic arrangement. The inversion twinning may have developed as a result of a phase transition from the ideal centrosymmetric NiAs-type structure to troilite-type structure during cooling. In addition, all samples were found to be cation-deficient. The departure from ideal stoichiometry—up to almost 3.5 rel% of metal atoms are missing—is also possibly related to atomic ordering when the crystals cooled.

Keywords: Troilite, crystal structure, absolute structure, inversion twin, meteorite, chondrite, Etter, Georgetown

INTRODUCTION

Troilite is an ubiquitous constituent in many meteorite types, lunar rocks, and interplanetary dust particles (Buchwald 1975; Brearley and Jones 1998; Mittlefehldt et al. 1998; Papike et al. 1998; Rietmeijer 1998 and references therein). Rarely, it has been found also in terrestrial rocks (e.g., Anthony et al. 1990)

Troilite crystallizes in the space group $P\bar{6}2c$ (No. 190). This space group is non-centrosymmetric and achiral [type NA of Flack (2003)] which results in two possible different spatial arrangements of constituting atoms within the unit cell. In this particular case, the two structurally *non-identical* arrangements are related by the inversion symmetry operation (see Fig. 1 displaying the coordination of iron atom in troilite). The correct orientation of the crystal structure is called the *absolute structure*. The theory and practical aspects of absolute structure determination from single-crystal structure data are presented by Flack (1983, 2003) and Flack and Bernardinelli (1999, 2000). The application of the X-ray single-crystal diffraction approach to the determination of the absolute structure for meteoritic schreibersites was shown in Skála and Císařová (2005).

None of the previous structure refinements of troilite or its synthetic analogs (e.g., Evans 1970; Keller-Besrest and Collin 1990a, 1990b; King and Prewitt 1982) included the test of the absolute structure. Here, we report the results of the absolute structure determination for two chondritic troilites and a synthetic analog, with their possible genetic implications.

EXPERIMENTAL METHODS

Single-crystal data were collected using a 4-circle single-crystal diffractometer Nonius KappaCCD (at the Center of Molecular Structures at the Faculty of Science, Charles University, Prague) equipped with a position-sensitive area CCD detector. The program COLLECT (Nonius 1997–2000) was used for data collection and software HKL SCALEPACK (Otwinowski and Minor 1997) was employed for the unit-cell refinement. The data reduction was carried out with program HKL DENZO (Otwinowski and Minor 1997). After correcting the data for absorption (Coppens 1970; Blessing 1997), the crystal structures were solved by applying the SIR92 program (Altomare et al. 1994), and the subsequent structure refinement was performed with the SHELXL97 software (Sheldrick 1997). The structure refinement and reporting programs were operated through the WinGX graphical interface (Farrugia 1999). The graphical presentations of the structure were prepared with DIAMOND (Crystal Impact 2005) program. Characteristics of the polyhedra were calculated from refined data employing the program PLATON (Spek 1998).

The chemical composition of the troilite samples studied was measured on carbon-coated polished (thin) sections using an electron microprobe JEOL JXA-8200 (at Bayerisches Geoinstitut, University Bayreuth, Bayreuth, Germany). Accelerating voltage was 20 kV, sample current 20 nA. The elements analyzed included Fe, Co, Ni, Mn, Ti, Cr, S, and P. The standards used were pyrite (for Fe and S), synthetic $MnTiO_3$ (for Mn) and GaP (for P), and pure metals for the remaining elements. All the elements were analyzed on their $K\alpha$-lines.

MATERIALS

Several grains of troilite from meteorites Cape York (IIIAB/Om), Etter (L5/S5), Flagg (L5), Forest Vale (H4/S2), and Georgetown (H6) were tested for suitability for single-crystal study. Most gave polycrystalline diffraction patterns or the diffraction spots in the frames recorded were too diffuse or streaky, and thus, not suitable for the collection of single-crystal data of a reasonable quality. In the end, single crystals from ordinary chondrites Etter and Georgetown were used for the data acquisition.

For comparison, also a synthetic troilite analog was studied. Single crystals were prepared by heating an iron wire (Koch-Light Lab, Ltd., England, diameter 0.7 mm, 99.998% purity) with a stoichiometric amount of high purity sulfur at 800 °C for 50 h in an evacuated silica-glass tube. The preparation of synthetic troilite simulates the sulfurization process suggested for the formation of some troilites in lunar rocks (Papike et al. 1998).

RESULTS

The crystal structure refinements of all three samples show that the overall crystal structure motif is consistent with previ-
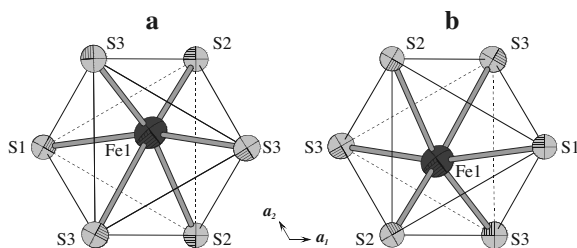
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FIGURE 1. Coordination octahedron around the iron atom for two inversely related structure models of troilite showing the atom-numbering scheme as viewed down [001]. The atomic setting in (**a**) corresponds to the atomic arrangement published in earlier reports whereas (**b**) represents the inverted one. Displacement ellipsoids are at the 90% probability.

ously published data for this mineral (Evans 1970; Keller-Besrest and Collin 1990a, 1990b; King and Prewitt 1982). To test which of the two non-identical inversely related crystal structure arrangement prevails in the crystals studied, we carried out a complete structure refinement for these structure settings and then compared the calculated Flack's x parameters as well as agreement factors for all three pairs of refinement (Table 1). Both Flack's parameter calculated during the structure refinement and the simple comparison of agreement factors indicates that in all three cases the single crystals studied correspond to the so-called *inversion twins* (Flack and Bernardinelli 1999)—they contain, in significant amount, simultaneously both of the two possible atomic configurations related by the inversion symmetry operation.

To allow easy comparisons, the final structure refinement was performed in the structure setting reported previously in literature though actually in all three crystals the inverse atomic setting dominates. Crystal data and structure refinement details are summarized in Table 2. The volume of inversely related domain, also reported in Table 2, was refined with SHELXL97, applying TWIN/BASF commands as suggested by Flack and Bernardinelli (2000). Such an approach allows a full-matrix least-squares refinement of the Flack's x parameter, which is otherwise calculated by the hole-in-one algorithm (see Flack and Bernardinelli 2000 for details) in the SHELXL97 program. The refined volumes of inverted twin domains in our inversion twins are 56(6)% for the crystal from Etter, 67(6)% for that from Georgetown, and 55(8)% for the synthetic material. These results confirm the preliminary tests summarized in Table 1 and indicate that in all three crystals the domain with inverted crystal structure dominates over the structure setting reported traditionally in the literature.

Both structure refinement and electron microprobe data show that the two natural troilites, as well as the synthetic one, are relatively homogeneous with a slight deficit in iron and other metallic elements. The departure from ideal stoichiometry based on microprobe data is 3.2 ± 0.5 for Etter, 2.5 ± 0.3 for Georgetown, and 2.8 ± 0.3 rel% for synthetic troilite (see empirical formulae in Table 2). The deficiency of metals derived from the structure refinements is slightly smaller: 2.1 ± 0.6 for Etter, 1.7 ± 0.6 for Georgetown, and 2 ± 1 rel% for synthetic troilite.

Atomic coordinates corresponding to spatial arrangement

TABLE 1. Evaluation of absolute structure for the troilite crystals studied

Sample	Etter		Georgetown		Synthetic	
Structure model	normal	inverted	normal	inverted	normal	inverted
R_{all}	0.0304	0.0301	0.0306	0.0278	0.0364	0.0356
wR_{all}	0.0709	0.0703	0.0759	0.0647	0.0893	0.0858
GooF	1.161	1.146	1.177	1.144	1.148	1.146
Flack's x parameter	0.55(7)	0.43(6)	0.67(7)	0.33(6)	0.54(9)	0.44(9)
Interpretation	inversion twin		inversion twin		inversion twin	

TABLE 2. Crystal data and structure refinement details for the troilite crystals studied

Sample	Etter	Georgetown	Synthetic
Empirical formula*	$Fe_{0.968}S$	$Fe_{0.975}S$	$Fe_{0.972}S$
Formula weight	86.09	86.47	86.35
Temperature (K)		293(2)	
Wavelength (Å)		0.71073	
Crystal system, space group		Hexagonal, $P\bar{6}2c$ (no. 190)	
Unit-cell dimensions, a (Å)	5.9650(2)	5.9650(2)	5.9660(4)
Unit-cell dimensions, c (Å)	11.7570(5)	11.7590(5)	11.7650(8)
Unit-cell dimensions, V (Å³)	362.28(2)	362.34(2)	362.65(4)
Z		12	
Calculated density (g.cm⁻³)	4.735	4.755	4.744
Absorption coefficient (mm⁻¹)	12.946	13.021	12.985
F (000)	494	496	495
Absorption correction routine	Coppens (1970)	Coppens (1970)	Blessing (1997)
T_{min} (mm)	0.303	0.182	0.268
T_{max} (mm)	0.530	0.484	0.504
Crystal size (mm)	0.90×0.89×0.54	1.9×0.7×0.7	0.86×0.74×0.45
θ range for data collection (°)	3.94 to 29.99	3.94 to 29.99	3.94 to 27.41
Limiting indices	−8≤ h ≤8	−8 ≤ h ≤8	−7 ≤ h ≤ 7
	−8 ≤ k ≤ 8	−8 ≤ k ≤8	−6 ≤ k ≤ 6
	−16 ≤ ℓ ≤ 16	−16 ≤ ℓ ≤ 16	−15 ≤ ℓ ≤ 15
Reflections collected/unique	9077/380	9104/380	1040/296
R_{int} †	0.0582	0.0603	0.013
Completeness to θ$_{max}$	98.80%	98.80%	98.40%
Refinement method		Full-matrix least-squares on F^2	
Data/restraints/parameters	380/0/23	380/0/23	296/0/23
GooF on F^2 ‡	1.183	1.159	1.12
Weight function parameters, a ‡	0.0321	0.0295	0.0546
Weight function parameters, b ‡	0.7495	1.0735	0.6629
Final R indices [$I > 2\sigma_I$], R §	0.0251	0.0239	0.0303
Final R indices, wR #	0.0615	0.06	0.0771
R indices (all data), R	0.0285	0.0263	0.0342
R indices, wR	0.0636	0.062	0.0821
Extinction coefficient	0.0041(10)	0.0051(11)	0.0066(17)
Largest diff. peak (e/Å³)	0.902	0.859	1.05
Largest diff. hole (e/Å³)	−0.627	−0.638	−0.7
Twin volume (inverted orientation)	0.56(6)	0.67(6)	0.55(8)

* Determined using electron microprobe.
† $R_{int} = \Sigma|F_o^2 - F_{o\,mean}^2|/\Sigma [F_o^2]$.
‡ *GooF*, Goodness-of-fit, $GooF = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n-p)\}^{1/2}$ where n is the number of reflections and p is the total number of parameters refined, $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where P is $[2F_c^2 + Max(F_o^2,0)]/3$ and a, b refinable parameters.
§ $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$.
# $wR = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$.

consistent with that given previously in the literature and equivalent isotropic displacement parameters yielded by SHELXL97 are listed in Table 3. In addition, Table 3 summarizes the parameters of individual polyhedra within the structure. Table 4 lists the anisotropic displacement parameters, and Table 5 gives selected interatomic distances and angles.

Troilite crystal structure represents a superstructure of the niccolite NiAs-type structure with unit-cell dimensions $a_T = \sqrt{3}a_N$ and $c_T = 2c_N$, where subscript T corresponds to troilite unit cell and N indicates the cell edges of the NiAs-type structure. The ideal NiAs structure possesses the $P6_3/mmc$ symmetry and consists of a primitive hexagonal cation sublattice, with anions

TABLE 3. Atomic coordinates, equivalent isotropic displacement parameters (in Å²), and polyhedral characteristics for the troilite crystals studied

Sample	Etter	Georgetown	Synthetic
Fe1, 12i: x, y, z			
x	0.3791(1)	0.3788(1)	0.3786(2)
y	0.0549(1)	0.0548(1)	0.0546(2)
z	0.12303(5)	0.12311(5)	0.12302(7)
U_{eq}	0.0152(2)	0.0145(2)	0.0180(3)
Coordination number	6	6	6
Polyhedral volume (Å³)	20.13	20.13	20.13
Quadratic elongation	1.019	1.019	1.019
Angle variance (°²)	58.4	58.2	57.6
S1, 2a: 0, 0, 0			
U_{eq}	0.0136(4)	0.0125(4)	0.0172(6)
Coordination number	6	6	6
Polyhedral volume (Å³)	19.22	19.20	19.19
Quadratic elongation	1.110	1.110	1.110
Angle variance (°²)	342.8	342.8	343.3
S2, 4f: $^1/_3$, $^2/_3$, z			
z	0.0198(1)	0.0200(1)	0.0198(2)
U_{eq}	0.0125(3)	0.0112(3)	0.0149(5)
Coordination number	6	6	6
Polyhedral volume (Å³)	15.28	15.29	15.30
Quadratic elongation	1.176	1.176	1.176
Angle variance (°²)	480.8	481.3	481.1
S3, 6h: $x, y, ¼$			
x	0.6653(3)	0.6652(2)	0.6656(3)
y	−0.0035(3)	−0.0033(3)	−0.0029(4)
U_{eq}	0.0130(3)	0.0118(2)	0.0150(4)
Coordination number	6	6	6
Polyhedral volume (Å³)	14.98	14.97	14.99
Quadratic elongation	1.251	1.251	1.251
Angle variance (°²)	696.1	695.3	694.6

TABLE 4. Anisotropic displacement parameters (in Å²) in the troilite crystals studied

Sample	Etter	Georgetown	Synthetic
Fe1			
U_{11}	0.0183(3)	0.0177(3)	0.0215(5)
U_{22}	0.0155(3)	0.0153(3)	0.0182(5)
U_{33}	0.0130(3)	0.0119(3)	0.0154(5)
U_{23}	−0.0009(2)	−0.0009(2)	−0.0013(3)
U_{13}	−0.0003(2)	−0.0004(2)	−0.0006(3)
U_{12}	0.0093(2)	0.0093(2)	0.0108(3)
S1			
$U_{11}, U_{22}, 2U_{12}$	0.0115(6)	0.0101(6)	0.0161(9)
U_{33}	0.0178(9)	0.0175(8)	0.0194(11)
S2			
$U_{11}, U_{22}, 2U_{12}$	0.0114(5)	0.0099(4)	0.0146(7)
U_{33}	0.0147(7)	0.0138(7)	0.0157(9)
S3			
U_{11}	0.0129(6)	0.0120(6)	0.0141(9)
U_{22}	0.0110(5)	0.0110(5)	0.0144(8)
U_{33}	0.0149(6)	0.0127(5)	0.0172(8)
U_{12}	0.0059(5)	0.0059(5)	0.0075(8)

TABLE 5. Selected interatomic distances and angles

Sample	Etter	Georgetown	Synthetic
	interatomic distances (Å)		
Fe1–S1	2.5636(6)	2.5633(6)	2.5628(8)
Fe1–S2[ii]	2.381(1)	2.385(1)	2.384(1)
Fe1–S2[iii]	2.5059(9)	2.5052(9)	2.506(1)
Fe1–S3[i]	2.360(1)	2.360(1)	2.359(2)
Fe1–S3	2.420(1)	2.420(1)	2.422(1)
Fe1–S3[iv]	2.719(1)	2.719(1)	2.720(2)
Fe1–Fe1[v]	2.925(1)	2.927(1)	2.929(1)
Fe1–Fe1[vi]	2.948(1)	2.950(1)	2.949(2)
	interatomic angles (°)		
S1–Fe1–S3[iv]	84.32(3)	84.36(3)	84.36(4)
S2[iii]–Fe1–S1	85.82(3)	85.84(2)	85.86(4)
S2[ii]–Fe1–S1	88.46(3)	88.40(3)	88.44(3)
S2[ii]–Fe1–S2[iii]	90.60(2)	90.57(2)	90.57(3)
S2[iii]–Fe1–S3[iv]	79.64(4)	79.62(4)	79.70(5)
S2[ii]–Fe1–S3	102.78(4)	102.76(4)	102.68(5)
S2[ii]–Fe1–S3[iv]	168.23(4)	168.18(4)	168.26(5)
S3[i]–Fe1–S1	92.09(3)	92.15(3)	92.19(4)
S3–Fe1–S1	165.95(4)	166.02(4)	166.10(6)
S3–Fe1–S2[iii]	85.67(4)	85.66(4)	85.72(5)
S3[i]–Fe1–S2[ii]	104.62(4)	104.60(4)	104.61(5)
S3[i]–Fe1–S2[iii]	164.59(5)	164.65(5)	164.65(7)
S3–Fe1–S3[iv]	83.18(6)	83.21(5)	83.27(7)
S3[i]–Fe1–S3[iv]	84.96(6)	85.04(5)	84.95(6)
S3[i]–Fe1–S3	93.15(5)	93.11(5)	93.01(7)
Fe1[v]–Fe1–Fe1[i]	60.0	60.0	60.0
Fe1[v]–Fe1–Fe1[vi]	95.25(1)	95.230(9)	95.22(1)
Fe1[i]–Fe1–Fe1[vi]	101.09(2)	101.05(2)	101.03(3)

Notes: Symmetry transformations used to generate equivalent atoms:
(i) $−x + y + 1$, $−x + 1$, z; (ii) $x − y + 1$, $−y + 1$, $−z$; (iii) x, $y − 1$, z; (iv) $−y$, $x − y − 1$, z; (v) $−y + 1$, $x − y$, z; (vi) $x − y$, $−y$, $−z$.

are highly deformed and can no longer be described as trigonal prisms. Instead, their topology approaches strongly deformed tetragonal scalenohedra. Triangles at the bases of trigonal prisms around S3 atoms are parallel to (001) yet they are not equilateral as in ideal NiAs-type structure; angles defining the trianglar bases are ~65, ~46, and ~69°.

CONCLUDING REMARKS

The crystal structure of troilite from two ordinary chondrites (Etter and Georgetown) and a synthetic analog prepared by sulfurization of an iron wire is in general consistent with what has been reported for troilite or its synthetic analogs (Evans 1970; Keller-Besrest and Collin 1990a, 1990b; King and Prewitt 1982). However, a detailed structure study shows that it is formed by two inversely related atomic arrangements—the crystals represent the so-called inversion twins. The Flack's absolute structure parameter x (Flack and Bernardinelli 1999), which determines the fraction of a domain with inverted crystal structure, is 0.56(6) for Etter, 0.67(6) for Georgetown, and 0.55(8) for the synthetic analog, indicating that, contrary to previous literature data, the inverse atomic arrangement dominates.

In older structure studies, the absolute structure was not reported because the quality of the data did not allow a determination of the volume of the twin domains. It was the usual practice that only a part of the reciprocal space was recorded, which permitted the structure determination and refinement, but not enough of the Bijvoet pairs were recorded to find the correct of the two possible atomic arrangements. In our study, we acquired almost a complete reciprocal space (see Table 2) for a given range of diffraction indices. This data redundancy then allowed us to determine the absolute structure for the studied materials.

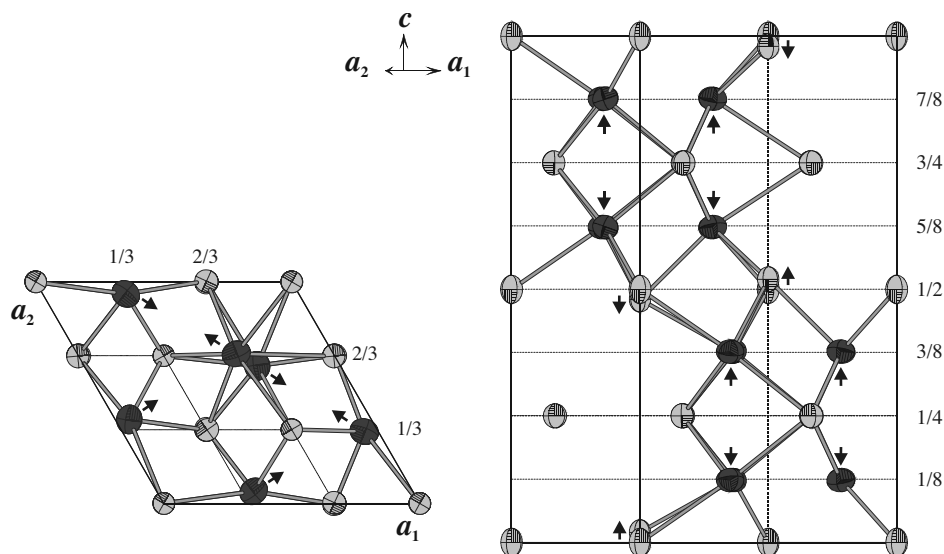In contrast to the absolute structure determination of sch-

lying in a hexagonal close packed sublattice. Cations in this structure are octahedrally coordinated, and anions are located at the centers of trigonal prisms. The octahedra share their faces parallel to the (001) plane and form infinite edge-sharing layers perpendicular to the hexagonal **c** axis. Figure 2 shows the unit cell of troilite in two principal projections and indicates how the atoms are displaced from ideal NiAs-type structure. This shift of atoms results in a deformation of all polyhedra within the troilite crystal structure. Octahedra around iron atoms are the least deformed (see also quadratic elongation and angle variance in Table 3). Nevertheless, the face defined by S1 and S2 atoms, which is parallel to the (001) plane in NiAs-type structures, is inclined by about 8° in troilite. Coordination environment of sulfur atoms is shown in Figure 3. Polyhedra around S1 and S2

FIGURE 2. Crystal structure of troilite (from Georgetown chondrite) viewed down [001] (left) and [120] (right). Arrows indicate the displacement of atoms from the ideal position in NiAs-type sublattice.
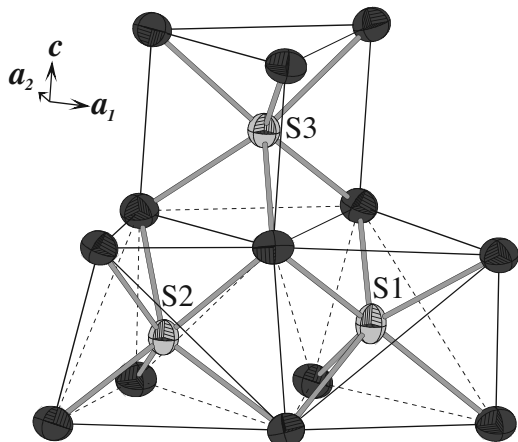

FIGURE 3. All sulfur atoms are in sixfold coordination. Whereas the coordination of the S1 and S2 atoms is irregular and approaches a strongly deformed tetragonal scalenohedron, the coordination polyhedron around S3 corresponds to a slightly deformed trigonal prism inherited from the original ideal NiAs-type structure.

reibersite (Skála and Císařová 2005), troilite displays inversion twinning in all three crystal structure refinements . This difference, together with an almost equal proportion of both atomic orientations, is most probably due to a polymorphic transition of FeS during cooling. At only slightly elevated temperatures, the stable form of FeS possesses the NiAs-type structure, which is centrosymmetric. When cooled, FeS undergoes a phase transition (~140 °C; Selivanov et al. 2003) to troilite-type structure that is non-centrosymmetric. As a consequence of a phase transition from higher to lower symmetry, inversion twins form consisting of two mutually intergrown domains with inverted atomic arrangements. In the case of schreibersite, the forma-

tion temperature is significantly higher and direct nucleation possibly provides a driving force strong enough to "switch" the absolute structure.

Iron occupancy refined from X-ray diffraction data as well as the content of Fe and other metals measured with the microprobe indicates that the studied troilites are cation-deficient—up to almost 3.5 rel%. Such departures from ideal stoichiometry may be attributed to a local disorder. This, at least in some instances (e.g., Pósfai and Buseck 1997), results in the formation of other FeS polytypes or interstitial defects locally present within troilite (a regular 2H polytype) crystals. However, these locally developed misfits in stacking sequence are random and consequently do not introduce sharp satellite diffraction spots; instead, they may deteriorate the diffraction patterns by producing diffuse or streaky diffraction maxima.

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