New chemical and physical data on keilite from the Zakłodzie enstatite achondrite

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Abstract

Keilite, (Fe,Mn,Mg,Ca,Cr)S, from the Zakłodzie enstatite achondrite is described. Forming xenomorphic grains up to 0.5 mm in diameter, the keilite is associated with troilite (or pyrrhotite), Fe-Ni metal, an (Fe,Zn,Mn)S phase, enstatite (with relict forsterite in cores), plagioclase and accessory schreibersite, oldhamite, graphite, sinoite, and an SiO_2 polymorph. It is brittle and possesses a good cleavage similar to that of galena, parallel to (001), (010), and (100). X-ray diffraction structural data reveal the following: cubic space group *Fm*3*m*, $\alpha = \beta = \gamma = 90°$, a = 5.1717 (18) Å, unit-cell volume V = 138.32 (8) Å^3; D = 3.958 g/cm^3; Z = 4. The chemical formula based on 63 electron microprobe point analyses is: $(Fe_{0.437}, Mn_{0.356}, Mg_{0.160}, Ca_{0.017}, Cr_{0.019}, Zn_{0.001})S_{1.008}$. Compared with previously described keilites from enstatite chondrites, the Zakłodzie keilite is richer in (Mn,Ca,Cr)S and poorer in the Fe- and Mg-end-members and, consequently, it is nearer to alabandite and oldhamite. This is the first detailed description of keilite from a meteorite that is not an enstatite chondrite.

Keywords: Meteorite, keilite, sulfide, XRD data, chemical data, enstatite achondrite, Zakłodzie

Introduction

Keilite, (Fe,Mg)S, first described as niningerite, (Mg,Fe)S, was discovered in enstatite chondrites by Keil and Snetsinger (1967). It was only in 2002 that keilite was characterized as an Fe-dominant analogue of niningerite and named after Dr. Klaus Keil (Shimizu et al. 2002). In that publication, the main structural, physical (including optical), and chemical features of the mineral were described, keilite being approved as a new mineral by the Commission on New Minerals and Mineral Names of the International Mineralogical Association in 2001 (IMA 2001-053). Keilite was reported first from enstatite chondrites: Abee and Adhi-Kot (type EH4), and Saint-Sauveur (EH5) (Shimizu et al. 2002). The latter authors also listed other meteorites in which keilite had been found: LEW 88180 (EH5), RKP A80259 (EH5), LEW 87119 (EL6), LEW 88714 (EL6), Y-791790 (EH4), Y-791811 (EH4), Y-86760 (EH4 melt), and Y-8404 (EH5) (classifications from Grady 2000). Furthermore, similar Fe-Mn-Mg sulfides have been described by Lorenz et al. (2003) from the phlogopite-bearing enstatite achondrite NWA 1235.

The chemical composition of keilite, like that of niningerite, indicates strongly reducing conditions of crystallization during which lithophile elements (e.g., Mg) were incorporated into sulfides instead of silicates (Keil 1968; Lin and Kimura 1998).

We found a mineral suspected to be keilite during chemical, mineralogical, and petrological investigations of the Zakłodzie enstatite meteorite (Przylibski et al. 2003, 2005). In an earlier paper (Karwowski et al. 2001), this phase was described as alabandite. At that time, only the chemical composition typical of keilite could be confirmed, there not being enough material to determine other structural and physical characteristics of this mineral. Further results of detailed investigations, which have shown this mineral to be keilite, are given in this paper.

The classification of the Zakłodzie enstatite meteorite remains controversial. In the recent publication on the mineralogy, petrology, and classification of this meteorite, Przylibski et al. (2003, 2005) discussed various hypotheses concerning its origin, e.g., considering Zakłodzie as an ungrouped enstatite-rich meteorite (Wlotzka et al. 2000—vide Grossman 2000), an EL7 enstatite chondrite (Stępniewski et al. 2000; Manecki and Łodziński 2001), or an enstatite achondrite (Stępniewski et al. 2000; Karwowski et al. 2001). Another widely held view is that of Burbine et al. (2000): the Zakłodzie meteorite is an impact-related melt. The latter interpretation does not allow assignment of Zakłodzie within the meteorite classification system. Przylibski et al. (2003, 2005), based on detailed observation, interpretation, and discussion of textures and mineral compositions, proposed another hypothesis that explained the observed features better, including published results of isotopic age determinations (K-Ar dating and ^{4}He concentration time method) on the parent body (Patzer et al. 2002). Przylibski et al. (2005) classified Zakłodzie as a primitive enstatite achondrite, and proposed that the stone represents a new group of meteorites, similar to the primitive achondrites: lodranites, winonaites, and acapulcoites defined previously. Because the Zakłodzie meteorite is not an E chondrite (Przylibski et al. 2005), this paper is the first report of keilite from a meteorite other than an enstatite chondrite. We are continuing our research efforts to clarify the remaining interpretative controversies.

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Methods of examination

The studied sample of the Zakłodzie meteorite, 250 × 170 mm in size, has a fusion crust, 0.1–2 mm thick, which is strongly weathered and contains Fe oxides and hydroxides. Three irregular but roughly concentric zones are distinguished in the stone (Przylibski et al. 2005). The dark-gray outer zone A (ca. 10–50 mm thick), and the intermediate rusty-gray zone B (10–40 mm thick) contain mostly silicates and ca. 20 vol% of opaque minerals, whereas in the pale-gray inner part C (20 × 40 mm in size) opaques reach 10–15 vol%. We used a collection of seven polished sections indicated as follows: 1, 2, 4, and 5—covering zone A and the fusion crust; 3—reaching to 50 mm deep from the crust; 6—a large thick section covering all three zones (all six were used for microscopic investigation—see e.g., sample = thin section numbers in Table 1, and for microprobe analysis); and 7—a standard covered thin section made of the outer and intermediate zones, for conventional polarizing microscope studies.

The mode of occurrence, appearance, and optical features were determined and the distinction of keilite was made using optical polarizing microscopes (transmitted and reflected light): Nikon Optiphot 2-POL, Axioplan 2 (Zeiss), NU-2, Vertival, and Amplival (Carl Zeiss, Jena). Keilite grains for XRD structural studies were selected under the microscope in reflected light, using a steel needle, in a paraffin oil medium. Attempts at chemical separation with HF and HCl were unsuccessful as keilite appeared to dissolve easily. Hardness was determined using the polishing relief method, and density was estimated based on the X-ray diffraction data.

Chemical microanalyses were made using electron microprobes at the Institute of Geological Sciences, Wrocław University, and at the Electron Microprobe Laboratory, Inter-Institute Analytical Complex for Minerals and Synthetic Substances, Warsaw University. Instrumentation and analytical conditions were: (1) in Wrocław: Cambridge Microscan 9 with two WDS spectrometers, 15 kV accelerating voltage, 50 nA beam current, 20 s counting time, natural and synthetic standards (Fe and S on troilite, Mn on pure metal, Mg on olivine, Cr on Fe-Cr alloy, Ni on niccolite), ZAF correction; (2) in Warsaw: Cameca SX100, three WDS spectrometers, 15 kV accelerating voltage, 20 nA beam current, 20 s counting time, natural and synthetic standards (Fe on hematite, S and Zn on sphalerite, Mn on rhodonite, Mg on diopside, Cr on Cr_2O_3, Ni on NiO), PAP correction.

Structural investigations were carried out at the Institute of Chemistry and Environmental Protection, Jan Długosz University in Częstochowa, using an automatic X-ray Xcalibur 3-circle single-crystal diffractometer with CCD detector, with graphite-monochromized Mo$K\alpha$.

Appearance and physical properties of keilite

A detailed petrographic description of the Zakłodzie meteorite was given by Przylibski et al. (2005). Opaque material in the central part of the meteorite is composed of grainy kamacite with segregations of schreibersite and graphite, and it does not contain keilite (Karwowski et al. 2001; Przylibski et al. 2005). Outside the center, keilite is distributed rather irregularly (Table 1) and it occurs as small grains, from a few to ca. 500 μm in diameter, together with other opaque minerals, enstatite, plagioclase, and rare Na-K feldspars (up to 40% Or). Most often, it is associated with an Fe-sulfide (Figs. 1a and 1b). This sulfide was originally described as troilite (and it is so indicated in Figs. 1 and 2), however, our preliminary investigations, using the Mössbauer technique, did not confirm troilite and identified pyrrhotite only (this study is in progress). The Fe sulfide and keilite commonly form parallel intergrowths with plagioclase (Fig. 1c). Keilite is also found together with kamacite and taenite (Figs. 1a–1c and 2a), schreibersite, and graphite. In places, the keilite contains small inclusions of graphite and sinoite (Fig. 2a) and, rarely, it is in contact with a silica phase (presumably tridymite or cristobalite, but not confirmed due to its small size). An additional accessory component is oldhamite $(Ca_{0,96},Mg_{0,02},Mn_{0,01},Fe_{0,01})S$. Keilite grains near the fusion crust of the Zakłodzie meteorite contain exsolutions of low-Ni Fe metal, as does the troilite/pyrrhotite (Figs. 2c and 2d).

The modal composition of the Zakłodzie meteorite, as determined by point-counting with an optical microscope (10 000 spots in four polished sections), is given in Table 1. Under weathering, keilite tends to transform into Fe-oxyhydroxides. However, all secondary phases were omitted in the micrometric analysis. The keilite abundance given in Table 1 is a minimum.

Keilite is bluish-gray in reflected light and it is isotropic. Its hardness is about 4 on the Mohs' scale, so it is softer than kamacite and slightly harder than troilite/pyrrhotite. The calculated density is 3.958 g/cm^3.

This value is lower than those from the enstatite chondrites studied by Shimizu et al. (2002), which reflects the nearly triple amount of (MnCaCr)S and lower amount of MgS in the former. Keilite is very brittle and displays good cleavage, similar to that of galena, i.e., parallel to (001), (010), and (100). The cleavage is expressed by characteristic triangular patterns of variable elongation, depending on section orientation (Fig. 2b). As a result, during separation, keilite grains tended to split into orthogonal fragments.

We have identified another sulfide in the Zakłodzie meteorite that has the following composition (selected individual point analyses): $(Fe_{0.4619}, Zn_{0.3233}, Mn_{0.1700}, Mg_{0.0449})S$. This rare phase is found in a similar textural position and association as the keilite (Fig. 2a); neither is associated with oldhamite, the latter always occurring with metal. A similar sulfide was reported by Buseck and Holdsworth (1972) in the Yilmia EL6 enstatite chondrite, in association with troilite, alabandite and daubreelite. The composition of the Yilmia sulfide is: $(Fe_{0.538}, Zn_{0.246}, Mn_{0.159}, Mg_{0.004})S$. An Fe-Zn sulfide, with considerable amount of Mn, was reported by Patzer et al. (2004) from the equilibrated enstatite chondrite Grein 002; its composition ranges from $(Fe_{0.45}\,Zn_{0.33}Mn_{0.20})S$ to $(Fe_{0.41}\,Zn_{0.35}\,Mn_{0.22})S$. To date, such a phase has not been formally approved as a new mineral by the IMA.

Chemical composition

Representative microprobe analyses of keilite from the Zakłodzie meteorite are given in Table 2, and shown on the ternary classification diagram (Fig. 3) together with comparative data from Shimizu et al. (2002). In this diagram, as in the original diagram of Shimizu et al. (2002), Ca and Cr are grouped with Mn. Those authors divided the compositional diagram into three fields, based on the 50% rule, and they described keilite as "Fe-dominant niningerite." The Zakłodzie keilite is considerably richer in Mn, and poorer in Fe and Mg, compared with keilites described previously from enstatite chondrites (Shimizu et al.

Table 1. Modal composition of the Zakłodzie meteorite (vol%)

Sample/ number of points	Enstatite/ clinoenstatite	Feldspar (+SiO_2)	Fe-Ni metal	Troilite/ pyrrhotite	Keilite	Sinoite	(FeZnMg)S	Graphite	Schreibersite
1/3000	64.7	17.1	14.1	2.8	0.23	0.03	0.03	0.6	0.3
2/1000	67.9	14.4	13.7	2.8	0.00	0.00	0.00	1.2	0.0
3/5000	65.0	16.5	14.2	2.9	0.18	0.06	0.02	0.6	0.5
4/1000	66.9	15.4	14.1	3.2	0.00	0.10	0.00	0.3	0.0

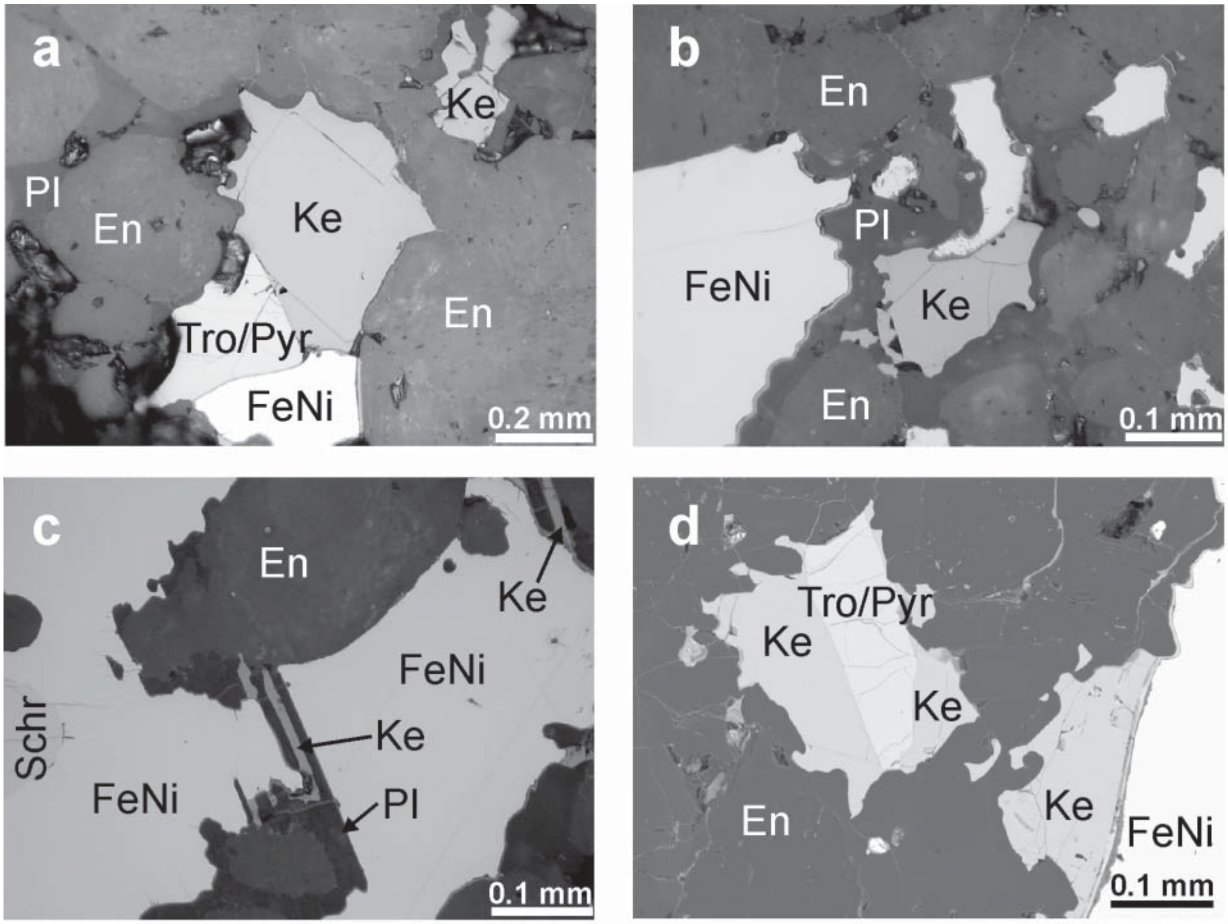

FIGURE 1. Textural settings and mineral associations of keilite in the Zakłodzie meteorite. (**a**) and (**b**) show typical textures of keilite (Ke) in association with troilite/pyrrhotite (Tro/Pyr) and metal (FeNi); gray, mostly oval grains are enstatite (En) and somewhat darker interstices are feldspars (Pl). (**c**) lamellar intergrowth of keilite and plagioclase. (**a**, **b**, **c** = photomicrographs in reflected polarized light). (**d**) back-scattered electron (BSE) image of keilite and other opaques enclosed in enstatite.

TABLE 2. Selected microprobe analyses and cation proportions of keilite from the Zakłodzie meteorite

Analysis	ZAD/0	19/4	1/3	3/3	ZAC/0	3/0	21/4	ZAB/0	20/4	Mean (n)	(n)
S	38.11	39.68	40.52	39.69	37.65	37.37	39.72	37.89	39.18	39.11	63
Fe	33.55	28.32	28.84	29.29	31.88	30.50	29.50	32.17	29.28	29.52	63
Mn	22.75	23.68	22.31	24.38	23.24	23.37	23.03	23.29	23.24	23.68	63
Mg	4.79	4.44	4.57	4.62	5.10	4.74	4.52	5.06	4.53	4.71	63
Ca	0.85	0.85	0.85	0.82	0.89	0.80	0.89	0.85	0.85	0.84	63
Cr	0.94	1.33	1.16	1.17	0.78	1.16	1.27	0.79	1.31	1.17	63
Zn	nd.	0.01	0.19	0.15	nd.	0.09	nd.	nd.	0.14	0.10	42
Total	100.99	98.30	98.45	100.12	99.54	98.03	98.93	100.05	98.51	99.12	
S^{-2}	0.974	1.029	1.044	1.014	0.972	0.981	1.024	0.974	1.017	1.009	63
Fe^{+2}	0.492	0.422	0.427	0.430	0.473	0.460	0.437	0.475	0.436	0.437	63
Mn^{+2}	0.339	0.358	0.336	0.364	0.350	0.358	0.347	0.349	0.352	0.357	63
Mg^{+2}	0.162	0.152	0.155	0.156	0.174	0.164	0.154	0.172	0.155	0.160	63
Ca^{+2}	0.017	0.018	0.017	0.017	0.018	0.017	0.018	0.017	0.018	0.017	63
Cr^{+3}	0.015	0.021	0.018	0.018	0.012	0.019	0.020	0.013	0.021	0.019	63
Zn^{+2}	nd.	0.000	0.002	0.002	nd.	0.001	nd.	nd.	0.002	0.001	42
Total	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	
FeS	0.480	0.434	0.447	0.437	0.460	0.452	0.448	0.463	0.444	0.442	63
MgS	0.158	0.156	0.163	0.158	0.169	0.161	0.158	0.167	0.158	0.162	63
(Mn,Ca,Cr)S	0.362	0.409	0.390	0.405	0.371	0.387	0.395	0.370	0.398	0.396	63

Notes: Analyses with minimum and maximum values for major elements: Fe, Mn, Mg, Ca, and Cr, and the mean composition are shown; nd. = not detected.

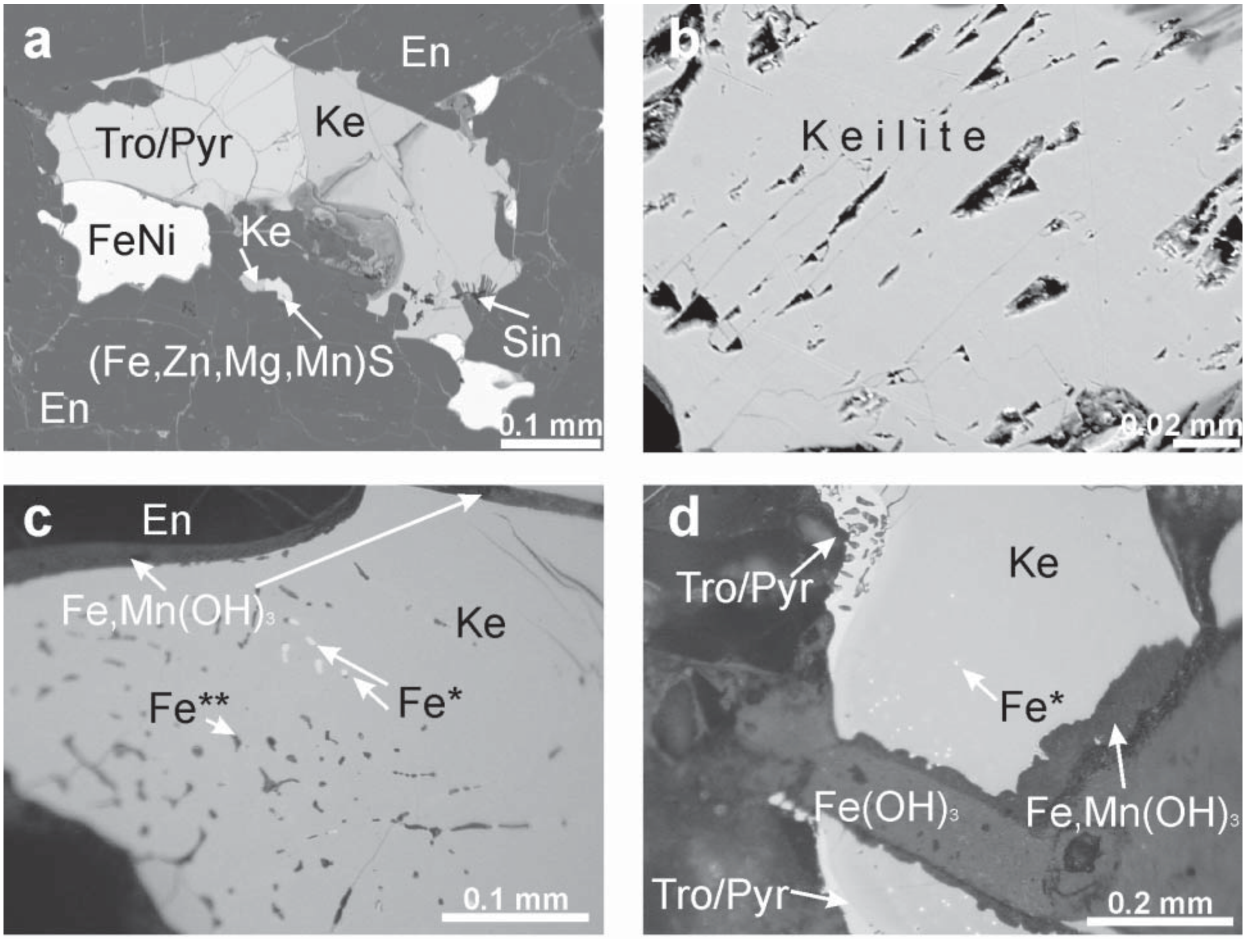

FIGURE 2. Textural settings and mineral associations of keilite in the Zakłodzie meteorite. (**a**) BSE image of keilite (Ke), troilite/pyrrhotite (Tro/Pyr) and kamacite (FeNi); fine intergrowths of sinoite (Sin) in keilite; below the center, a small grain of (Fe,Zn,Mg,Mn)S is intergrown with keilite. (**b**) BSE image of keilite showing triangular patterns reflecting a galena-type cleavage. (**c**) exsolutions of low-Ni iron metal (Fe*) in keilite (Fe** = secondary Fe hydroxides and holes after Fe-metal). (**d**) keilite in the most external zone of Zakłodzie contains low-Ni Fe-metal exsolutions (Fe*); Fe- and Fe,Mn-hydroxides are common near the fusion crust; (c, d = microphotographs in reflected polarized light).

2002). Consequently, it plots in a different part of the keilite field, close to the alabandite sector. Figure 3 also shows that the solid loop [labeled alabandite (EL)], which encloses analyses of minerals originally classified as alabandites (Keil and Snetsinger 1967; Zhang et al. 1995; Brearley and Jones 1998), extends into the keilite field. Thus, some of the minerals described originally as alabandites (e.g., in such meteorites as NWA 1235, Abee, Saint Sauveur, Adhi-Kot, and LEW88180) appear to represent keilite.

The average composition of the Zakłodzie keilite, based on 63 point analyses (Zn on 42 only) of several grains in several thin sections, and recalculated on the basis of 2.0 cations, is:

$$(Fe_{0.437}, Mn_{0.356}, Mg_{0.160}, Ca_{0.017}, Cr_{0.019}, Zn_{0.001})S_{1.008}.$$

Apart from the components included in the formula and given in Table 2, the following elements have been detected in trace amounts in a few cases during the course of microprobe analysis: Co, Ni, Cu, Sr, P, Ti, Si, Al, and Na. However, these may be due to beam overlap on neighboring phases or, simply to analytical errors.

It is worth mentioning that the troilite in the keilite-bearing Zakłodzie meteorite is rich in Cr (mean = 4.39 wt%, n = 14) and Ti (mean = 0.72 wt%, n = 14; data from Przylibski et al. 2003), which apparently results in the absence of such phases as daubreelite and osbornite.

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Structural X-ray results on the keilite studied here are compared with the data of Shimizu et al. (2002) in Table 3. The Zakłodzie keilite belongs to the cubic space group *Fm*3*m*; thus, the angles between the crystallographic axes are $\alpha = \beta = \gamma = 90°$ and all the edges of the unit cell are a = 5.172 (18) Å. The calculated volume of the unit cell is 138.32 (8) Å^3. The R-factor is 0.0182 for 209 reflections collected at a temperature of 293(2) K.

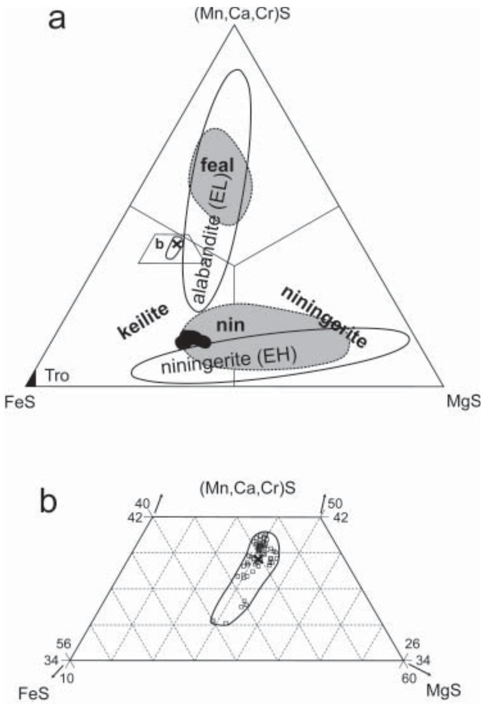

Figure 3. (**a**) FeS-MgS-(Mn,Ca,Cr)S ternary with 50%-lines separating the keilite and niningerite fields; the small trapezoid, enlarged in diagram (**b**), shows the Zakłodzie keilite analyses (small squares and the average X); the approximate compositional range of troilite (Tro) is indicated as the little black triangle near the FeS corner in **a**; the black area in the same diagram represents six analyses of keilite reported by Shimizu et al. (2002); the shaded areas stand for compositional fields of ferroan alabandite (feal) and niningerite (nin) based on data from Keil and Snetsinger (1967)—feal shows analyses from enstatite chondrites type II: Jaih deh Kot Lau, Hvittis, Atlanta, Pillistfer, Ufana, Blithfield, Khairpur, Daniel's Kuil, whereas nin—from enstatite chondrites type I: Abee, Saint Sauveur, Adhi-Kot, Indarch, St. Mark and Kota-Kota; solid loops correspond to the fields of alabandite (EL, comprising EL3 and EL4-6) and niningerite (EH, comprosong EH3 and EH4-6)—based on Keil and Snetsinger (1967), Zhang et al. (1995), Brearley and Jones (1998), Shimizu et al. (2002), and Patzer et al. (2001, 2004).

Discussion and concluding remarks

Our report is the first account of keilite in a meteorite other than the enstatite chondrites. What is the role of that mineral in these enstatite meteorites? The presence of this mineral clearly indicates strongly reducing conditions and (possibly) local concentrations of Mn and Mg, all facilitating the crystallization of keilite besides the more widespread troilite and/or pyrrothite.

We can consider keilite as an accessory phase in the parent rocks of enstatite chondrites and achondrites. The presence of this mineral clearly indicates that the protolith was deficient in Fe, which in the case of the Zakłodzie meteorite is consistent with the composition of silicates, such as enstatite and relict forsterite. Apparently, the melting of the protoliths of the keilite-bearing meteorites, like that of the Zakłodzie meteorite, was a brief event, not allowing for significant differentiation and segregation of sulfides from silicates.

We think that the alabandites (and ninignerites), which are described in earlier works and whose compositions fall into the keilite field (as defined by Shimizu et al. 2002), can be reclassified as keilite, however, this would require re-studying of these sulfides in a range of meteorites. The studies by Shimizu et al. (2002), as well as our new results, indicate that we need a new careful verification of the (Fe,Mn,Mg)S-type sulfides that are present in meteorites, in relation to the classification diagram of Shimizu et al. (2002) and including their structural characteristics.

The keilite composition in Zakłodzie falls between those of alabandite and niningerite, minerals found, separately, in EL chondrites (alabandite) and in EH chondrites (ninigerite) (Patzer et al. 2004). Thus, the presence of keilite in Zakłodzie does not help to discriminate among EL, EH-chondrite, or the intermediate-type compositions of its protolith. Taking into account the keilite occurrence in both EL and EH chondrites (Grady 2000), i.e., in parageneses with alabandite or niningerite, we can see that chemical composition of (Fe,Mn,Mg)S-type sulfides vary considerably between MnS and FeS end-members, and between MgS and FeS end-members. This means that both Mn and Mg can easily be replaced by Fe in these sulfides, whereas the substitution of Mn by Mg appears to be limited. As a result, we can observe niningerite and keilite in EH chondrites and alabandite and keilite in EL chondrites and in their protoliths on the parent bodies. Apparently, the most important chemical difference between sulfides of the (Fe,Mn,Mg)S-type, in the two types of enstatite chondrites and in achondrites, is their Mn/Mg ratio. Finally, it is likely that keilite is more common in various enstatite chondrites and achondrites than supposed previously.

Table 3. X-ray diffraction data of keilite from the Zakłodzie meteorite and earlier published data

h k l	Keilite from Zakłodzie meteorite		Keilite—published data	
	$d_{(meas.)}$	$I_{(meas.)}$	$d_{(meas.)}$*	$d_{(calc.)}$†
1 1 1	2.9859	11		2.985
2 0 0	2.5859	100	2.584	2.585
2 2 0	1.8285	35	1.829	1.828
2 2 2	1.4929	18		1.492
3 1 1	1.5593	2		
3 3 1	1.1865	1		
4 0 0	1.2929	12		1.292
4 2 0	1.1564	8		1.156
4 2 2	1.0557	6		1.055
4 4 0				0.914
4 4 2	0.8619	2		
4 4 4	0.7465	1		
6 0 0	0.8619	2		0.862
6 2 0	0.8177	2		0.817
6 2 2				0.779
6 4 0	0.7172	1		

* From Keil and Snetsinger (1967).
† from Shimizu et al. (2002).

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