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Tetrataenite—ordered FeNi, a new mineral in meteorites

ROY S. CLARKE, JR.

Department of Mineral Sciences
National Museum of Natural History
Smithsonian Institution
Washington, D.C. 20560

AND EDWARD R. D. SCOTT[1]

Department of Terrestrial Magnetism
Carnegie Institution of Washington
5241 Broad Branch Road, N.W.
Washington, D.C. 20015

Abstract

Tetrataenite is a new meteoritic mineral with ideal formula FeNi, which forms by ordering of Fe and Ni atoms in taenite. Albertsen *et al.* (1978a), using Mössbauer spectroscopy and X-ray diffraction, identified ordered FeNi in two iron meteorites. They found it has the $L1_0$ (or CuAu) structure like synthetic FeNi: space group $P4/mmm$ with $a = 2.533 \pm 0.002$, $c = 3.582 \pm 0.002$Å, $Z = 1$. Our electron-probe analyses give (range and mean wt. percent) Ni 48–57, 51; Fe 44–52, 49; Cu 0.11–0.36, 0.20; Co <0.02–2.0, 0.08; P <0.01. Tetrataenite, like taenite, is cream-colored in reflected light, but under crossed polars it can be distinguished by its anisotropy and characteristic polarization colors, which are clearly discernible in well-polished sections.

Tetrataenite has been identified optically in over 50 chondrites and mesosiderites, where it is commonly distributed as 10–50-μm-sized grains in contact with kamacite, troilite, taenite, and silicate. This occurrence was previously described as 'clear taenite' by Taylor and Heymann (1971). It also forms in two other locations identified by Albertsen *et al.*: rims 1–20 μm wide on taenite grains in chondrites, mesosiderites, irons, and pallasites, and grains ≤1 μm in size in adjacent cloudy taenite intergrowths. Massive and rim occurrences of tetrataenite are composed of three sets of irregular oriented crystals, which show intricate lamellar magnetic domains under crossed polars. Tetrataenite forms below 350°C in slow-cooled meteorites that contain taenite, and is most abundant in mesosiderites and some chondrites which have cooled the slowest.

Introduction

Two Fe–Ni minerals are commonly found in meteorites: kamacite, which is body-centered cubic and has 4–7 percent Ni, and taenite, which is face-centered cubic with ~25–45 percent Ni. Albertsen *et al.* (1978a) found another phase with the idealized formula FeNi, which had formed from Ni-rich taenite by ordering of Fe and Ni atoms. We have observed it optically in 60 meteorites and conclude that it forms in all slow-cooled meteorites that contain taenite. In many it is a common accessory mineral and in some it is more abundant than taenite. The mineral and the name tetrataenite,[2] which is derived from its tetragonal structure and taenite, have been approved by the Commission on New Minerals and Mineral Names of the IMA. It is the natural analog of synthetic ordered FeNi, which has been produced by irradiation of Fe–Ni alloys with neutrons or electrons (Paulevé *et al.*, 1962; Gros and Paulevé, 1970; and several other papers referenced therein).

Metallurgical studies of chondrites by Urey and Mayeda (1959) and Knox (1963) revealed what ap-

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[1] Present address: Institute of Meteoritics, University of New Mexico, Albuquerque, New Mexico 87131.

[2] We thank Dr. A. Kato, Chairman of the Commission, for suggesting this name.

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peared to be two varieties of taenite grains: one which develops a cloudy border on etching in nital (a cloudy core if the grains are small or the cooling rate low), and another which remains clear and is now identified as tetrataenite. Using electron-probe analysis, Reed (1964) found that most taenite was strongly zoned in Ni but a few grains were much more homogeneous and contained about 50 percent Ni. Wood (1967) showed that strongly zoned taenite in chondrites formed during slow cooling of metal grains from ∼700°C to ∼400°C, as in iron meteorites. By comparing observed profiles with those calculated using phase diagram and diffusion data for the Fe–Ni system, Wood was able to derive meteorite cooling rates from the compositions of zoned taenite grains. Taylor and Heymann (1971) were the first to discover that 'clear taenite,' as they called the second variety, contained 49–57 percent Ni. They gave a detailed account of its occurrence in nine chondrites and suggested that it formed below 400°C, by nucleating in kamacite.

Ordered FeNi was found by Albertsen (né Petersen) and coworkers in supposedly normal taenite by Mössbauer spectroscopy and X-ray diffraction studies of isolated lamellae from the Cape York and Toluca iron meteorites (Petersen *et al.,* 1977; Albertsen *et al.,* 1978a,b,c). From etching experiments, they concluded that ordered FeNi was concentrated at the edge of the taenite lamellae. Scott and Clarke (1979a) observed that the 'clear taenite' of Taylor and Heymann (1971) was optically anisotropic, and that, in zoned taenite grains from all types of meteorites, the rim which is outside the cloudy border or core and remains clear on etching was also anisotropic. After measuring Ni concentrations of 48–57 percent in the rim in chondrites, mesosiderites, and irons, we deduced that 'clear taenite' was ordered FeNi. In large or fast-cooled grains of zoned taenite that have cloudy borders, there is another region that remains clear on etching. This phase, which is *inside* cloudy taenite and contains 25–30 percent Ni, was called 'clear taenite II' by Lin *et al.* (1979). It is optically isotropic and is, presumably, true taenite.

Electron microscopy of cloudy taenite by Scott (1973) and Lin *et al.* (1979) revealed that it is a submicron intergrowth of two phases with a foam-like structure. Using electron and X-ray diffraction, these authors identified the phases as kamacite and taenite, but Albertsen *et al.* (1978b), using Mössbauer spectroscopy and X-ray diffraction, identified only taenite and ordered FeNi in their samples.

In this paper we review published X-ray and Mössbauer data on ordered FeNi and give a detailed account of the composition, properties, and occurrence of tetrataenite. Ordering in taenite has previously been reported by Ramsden and Cameron (1966a,b) and Stulov (1960). However, the extra X-ray reflections which these authors discovered and indexed on (different) multiple cells have not been confirmed. It is possible that there is a second, ordered metallic phase in meteorites: FeNi$_3$ (awaruite). Clarke *et al.* (1971) found metal of about this composition in the Allende meteorite. It has not yet been shown that Allende metal has an ordered structure (Cu$_3$Au type or L1$_2$).

Results

Chemical composition

We have analyzed tetrataenite in 18 meteorites with the electron probe. Pure Fe, Ni, Co, and Cu metals, Fe–Ni alloys of known compositions, and schreibersite were used as standards, with an accelerating voltage of 15 or 20 kV and specimen currents of 0.04 and 0.1 μA. Samples were lightly etched before analysis to assist in mineral identification. The mean Ni content of 51 wt. percent (Table 1) is close to the theoretical value for FeNi of 51.2 percent. Although cloudy taenite is commonly found in contact with tetrataenite, its Ni content is always distinctly lower, ≤43 percent. Tetrataenite is only slightly zoned in Ni (generally 49–54 percent) compared to taenite (Taylor and Heymann, 1971; Scott and Clarke, 1979a). We have not found any taenite with Ni concentrations inside the tetrataenite range of 48–57 percent, except in the atmospherically heated rims of meteorites.

Analyses of tetrataenite for Ni, Co, and Cu are listed in Table 2. In general, tetrataenite has concentrations of Co and Cu which are proportional to those in coexisting cloudy taenite. Thus in LL chondrites tetrataenite is richest in Cu and Co, just like taenite.

Table 1. Mean composition (wt%) of tetrataenite in meteorites by electron-probe analysis

Element	Range	Mean
Ni	48-57	51
Fe	44-52	49
Cu	0.11-0.36	0.20
Co	< 0.02-2.0	0.08
P	< 0.01	< 0.01
Sum		100.2

Table 2. Electron probe analyses of tetrataenite in 14 meteorites for Ni, Cu, and Co (wt%)

Meteorite	Class	No. grains	Ni		Cu	Co
			Mean	Range		
Kainsaz	CO3	3	53.2	50.0-55.2	0.36	< 0.02
Ngawi	LL3	1	50.5	50.0-56.5	0.19	2.0
Alta'Ameem	LL5	3	52.0	50.5-54.5	0.24	0.22
Olivenza	LL5	2	51.4	49.5-56.2	0.16	0.20
Mezö-Madaras	L3	8	51.2	49.6-56.0	-	0.18
Guibga	L5	2	53.7	52.0-56.0	-	0.10
Fayetteville	H	3	49.8	47.8-51.5	-	0.15
Sena	H4	1	51.0	-	0.28	-
Lost City	H5	3	51.7	49.2-54.8	-	0.04
Guareña	H6	1	50.0	48.0-52.8	-	-
Estherville	MES	9	51.1	48.0-55.5	0.15	0.02
Veramin	MES	6	50.7	49.0-53.8	0.16	< 0.02
Emery	MES	6	50.7	48.0-53.5	0.11	< 0.02
Freda	IIID	2	50.0	49.3-50.7	0.18	0.10

Structure

Evidence for ordering of Fe and Ni in supposedly pure taenite lamellae from the Cape York and Toluca iron meteorites was derived from Mössbauer spectroscopy and X-ray diffraction by Albertsen *et al.* (1978a,b). The six-line Mössbauer spectra are asymmetric due to quadrupole splitting, which shows that the Fe atoms are in non-cubic environments. The narrow width of the lines shows that all the Fe atoms have identical surroundings. After a sample was heated at 460°C for 60 hrs, its spectrum showed broad lines with no quadrupole splitting, indicative of (disordered) taenite. Their etching experiments on a Cape York lamella showed that ordered FeNi is concentrated in the outer few microns.

X-ray diffraction of 'taenite' lamellae from Cape York and Toluca by Albertsen *et al.* (1978a), using CoKα radiation and oscillation and Weissenberg cameras, showed extra, weak reflections. These could be indexed using the taenite unit cell, but should be absent due to face-centering of the lattice. Their first-layer Weissenberg photograph of a Cape York crystal oscillated about [110] showed 32 such weak reflections.

The only structure for ordered FeNi compatible with X-ray and Mössbauer results is the $L1_0$ structure of CuAu (Fig. 1). This tetragonal structure can be derived from the face-centered taenite structure by ordering Fe and Ni atoms on alternate (002) planes. Albertsen *et al.* (1978a) found that the cell constants on taenite axes are both 3.582±0.002Å, although other CuAu-type compounds have detectable tetragonal distortions. On the conventional primitive tetragonal axes (Fig. 1) tetrataenite is $P4/mmm$ with $a = 2.533$, $c = 3.582$Å (±0.002Å), $Z = 1$.

Since the tetrataenite cell on taenite axes is C-centered, there should be some systematic absences in single-crystal diffraction patterns indexed on the taenite unit cell. However 'taenite' lamellae contain regions of tetrataenite oriented in three mutually perpendicular directions, so these absences are not observed.

Extra lines due to ordering of FeNi have recently been detected by Danon *et al.* (1980) on X-ray powder photographs of metal from the chondrite St. Séverin. Danon *et al.* (1979b) also observed asymmetric Mössbauer spectra like those seen by Albertsen *et al.* (1978a) in metal from several chondrites. The Mössbauer and X-ray diffraction data for tetrataenite in irons and chondrites are entirely consistent with those for synthetic ordered FeNi for which an $L1_0$ structure has also been deduced (see Chamberod *et al.*, 1979; Paulevé *et al.*, 1962, and papers referenced therein). Recently Mehta *et al.* (1980) have used transmission electron microscopy to observe superlattice spots in tetrataenite from the Estherville meteorite.

Optical and physical properties

As expected, tetrataenite has similar properties to taenite in reflected light; both are creamy in color when compared to kamacite, which is white, and have similar high reflectivity and polishing hardness. However, under crossed polars, tetrataenite shows anisotropy while taenite does not. Distinct anisotropy is observed in well-polished sections of many meteorites, but with a poor polish the effect is weak or unobservable. Reheating, both cosmic and terrestrial, and shock can also reduce or remove the anisotropy. With polars slightly uncrossed, tetrataenite under oil immersion shows weak anisotropic colors of bluish-green and brownish-orange. Also visible under

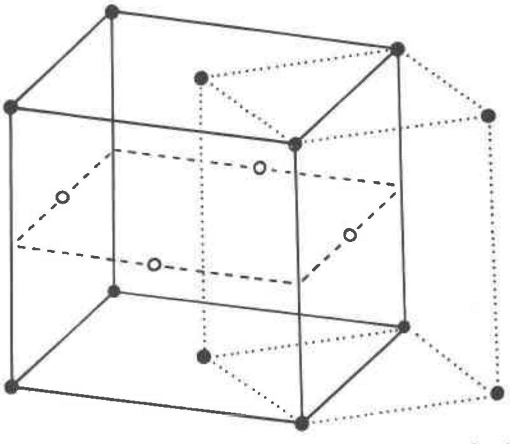


Fig. 1. Unit cells of taenite (solid lines) and tetrataenite (dotted lines). Tetrataenite, which has the $L1_0$ or CuAu structure and space group $P4/mmm$ (Albertsen *et al.*, 1978a), forms when Fe and Ni atoms (●,○) order on alternate (002) planes of taenite as shown.

crossed polars are intricate lamellae (Fig. 2). These are magnetic domains, which like those in cohenite, Fe_3C, are revealed by the Kerr magneto-optical effect and can be moved with a hand magnet. Reflection dichroism and bireflection are not observed, even in oil immersion.

Etching with a 1–2 percent solution of nital (nitric acid in ethanol) is useful in identifying tetrataenite. It is unaffected by light etching whereas cloudy taenite, which is often adjacent, turns brown. Because etching may produce pseudoanisotropy, tetrataenite was characterized only in unetched sections.

The Vickers hardness of tetrataenite grains in Estherville as measured by E. P. Henderson using a 25 g load was 170–200, *cf.* 130–160 in adjacent kamacite. Kamacite hardness varies greatly with the thermal and shock history of the meteorite (Buchwald, 1975, p. 93), and the same is probably true for tetrataenite.

Occurrence

Three major types of tetrataenite occurrence have been observed. Massive tetrataenite, typically 5–50 μm in size but up to 400 μm in Estherville, is widely distributed in H, L, and LL chondrites of all metamorphic types, and in mesosiderites. It is associated with kamacite and is commonly in contact with cloudy taenite, troilite, and silicates. Such occurrences in chondrites were described and analyzed by Taylor and Heymann (1971) under the name of 'clear taenite.' Tetrataenite also occurs as rims 1–20 μm wide on taenite grains in all types of meteorites: chondrites, mesosiderites (Fig. 2), pallasites, and irons. On etching it can be seen that the tetrataenite rim encloses cloudy taenite. In the chondrites and mesosiderites where the rims are widest, there is a complete sequence of occurrences between massive tetrataenite, tetrataenite with small central blobs of cloudy taenite, and normal zoned grains with tetrataenite rims.

Both massive and rim tetrataenite are composed of irregular crystals 1–15 μm in size, which are only visible under crossed (or slightly uncrossed) polars and are oriented in three different directions. Three sets of oriented tetrataenite crystals are observed, because ordering in a single crystal of taenite can occur on one of three equivalent sets of (002) planes. Since lattice distortions on ordering are so minute, boundaries between tetrataenite crystals will be coherent irrespective of their orientation, and are not as a rule crystallographically oriented themselves.

Tables 3 and 4 list some meteorites where massive or rim tetrataenite has been identified by reflected-light microscopy from its characteristic appearance under crossed polars. This list is obviously not exhaustive, but it does illustrate the relative ease of identifying tetrataenite optically in different meteorite classes. In addition to the classes already discussed, CO3 chondrites and diogenites also contain massive and rim tetrataenite. In 18 of the 60 meteorites listed in Tables 3 and 4 (those marked by an asterisk) we have analyzed tetrataenite with the electron probe and found Ni contents in the range 48–57 percent, confirming the optical identification. In irons and pallasites where the tetrataenite rims are narrower (typically 1–3 μm), it is more difficult to observe its characteristic features under crossed polars. But the widespread distribution of clear rims next to cloudy taenite (Scott, 1973) suggests that tetrataenite is common in all irons which contain taenite and have experienced normal slow cooling (*e.g.*, 1–100°C/m.y.).

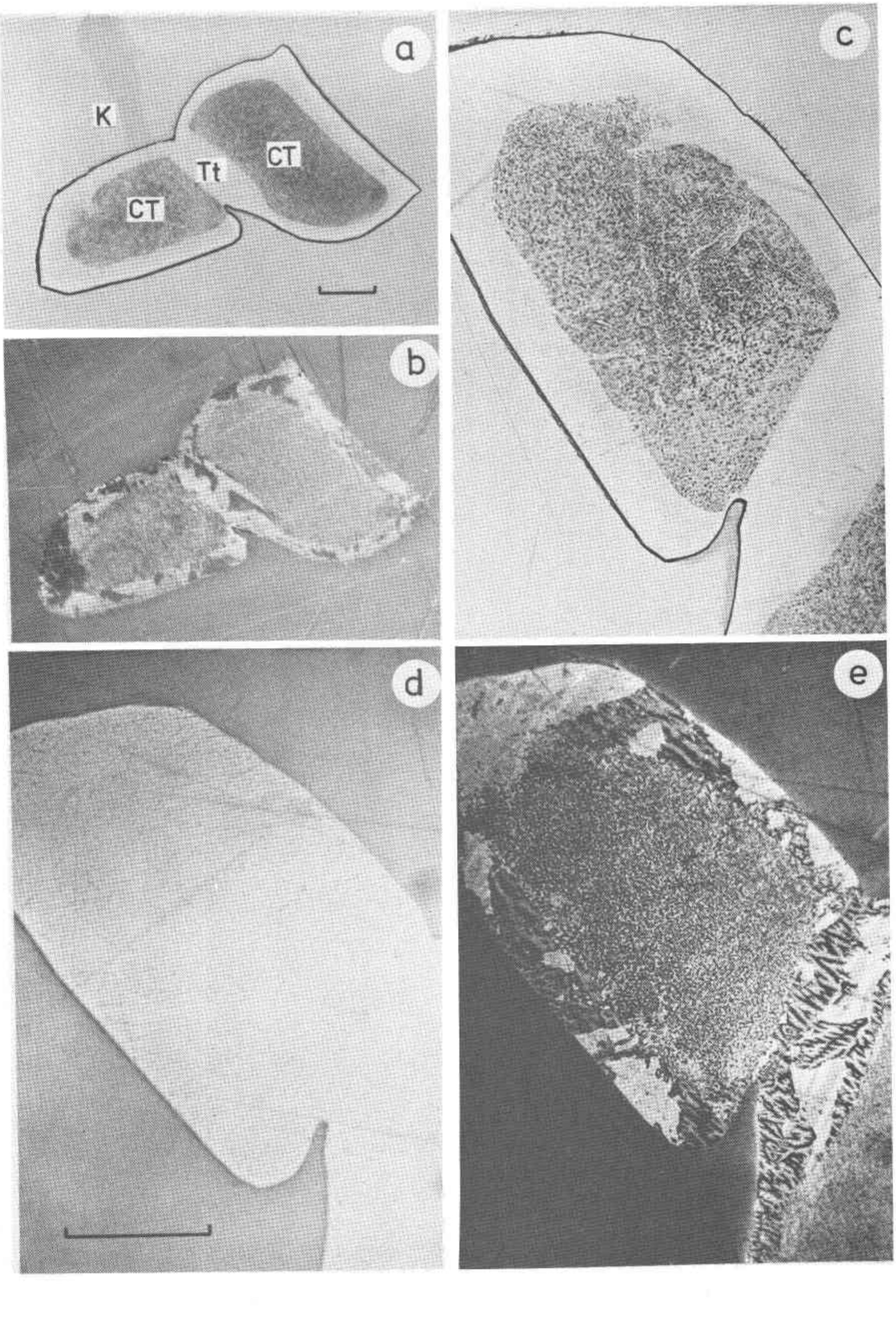
The third type of occurrence of tetrataenite is in cloudy taenite. In cloudy taenite areas of Estherville (Fig. 2), it is possible to see μm-sized crystals of tetrataenite with the same anisotropic colors and orientations as tetrataenite crystals in the rim (Scott and Clarke, 1979b). This confirms the conclusion of Albertsen *et al.* (1978a,b) that ordered FeNi is a component of cloudy taenite. Mesosiderites, like Estherville, which have the slowest cooling rates, contain the coarsest intergrowths in cloudy taenite; normally the crystals are too small for optical identification.

Mössbauer and X-ray studies indicate that tetrataenite is also a major component of the Santa Catharina iron (Danon *et al.*, 1979a), which contains 34 percent Ni (Buchwald, 1975, p. 1068).

Tetrataenite is most readily observed in mesosiderites and slow-cooled chondrites, where it is most abundant. The mesosiderite Estherville has supplied the type material. This meteorite is widely distributed among meteorite collections, including that in the Smithsonian Institution (catalog number 1025).

Discussion

Except for Gooley *et al.* (1975), who gave a brief description of tetrataenite in four meteorites under the name of 'anisotropic taenite,' many authors, including Ramdohr (1973, p. 19) and Scott (1973), have studied tetrataenite with reflected-light microscopy without observing its anisotropy. The explanation for this is not clear, as its anisotropy can be easily observed in a large number of meteorites. Poorly-polished sections may be partly to blame. In-


a
K
Tt
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CT
b
c
d
e

Table 3. Chondrites in which tetrataenite has been optically identified

Meteorite	Type†	Meteorite	Type	Meteorite	Type
H		L		LL	
Dhajala	3	*Mezö-Madaras	3	Bishunpur	3
Farmville	4	Bjurböle	4	Chainpur	3
Menow	4	Saratov	4	*Ngawi	3
*Sena	4	Barwell	5	Soko-Banja	4
*Weston	4	*Guibga	5	*Alta'Ameem	5
Allegan	5	Sao Jose do		*Olivenza	5
Avanhandava	5	Rio Preto	5	Ensisheim	6
Canon City	5	*Wold Cottage	6	St. Séverin	6
*Lost City	5			Uden	7
Nuevo Mercurio	5	CO		Other	
Plainview	5				
Pultusk	5	*Kainsaz	3	Cumberland	
*Guareña	6			Falls (Xenolith)	
Ipiranga	6				
*Fayetteville	–				

*Meteorites in which tetrataenite was analyzed by electron probe.
†Metamorphic types from Wasson (1974).

Table 4. Differentiated meteorites in which tetrataenite has been optically identified

Mesosiderites	Mesosiderites	Irons	Other
Barea	Mincy	IAB	Pallasites
Bondoc	Morristown	Mundrabilla	Brenham
Budulan	Mt. Padbury	Toluca	Glorieta Mtn.
Crab Orchard	Patwar	*Woodbine	
Dalgaranga	*Pinnaroo	IIICD	Diogenite
*Emery	Simondium	Carlton	Roda
*Estherville	Vaca Muerta	Dayton	
Hainholz	*Veramin	*Freda	
Lowicz	ALHA 77219		

*Meteorites in which tetrataenite has been analyzed by electron probe.

terestingly, the discovery of anisotropy in 'clear taenite' by Scott and Clarke (1979a) was purely accidental, despite suggestive clues in the work of Albertsen *et al.* (1978a,b) and Gooley *et al.* (1975). Gooley *at al.* did not realize that they had observed the 'clear taenite' of Taylor and Heymann (1971). They did suspect that their anisotropic phase was ordered FeNi from its composition, optical properties, and loss of anisotropy on heating to 500°C for 24 hours, but were unable to observe extra X-ray diffraction spots to prove it.

Other recent studies that failed to identify tetrataenite include those of Scott (1973), Kimball (1973), and Lin *et al.* (1979), who examined taenite using electron microscopy and diffraction. As the extra diffraction spots are very weak in intensity, tetrataenite could easily have been mistaken for taenite. Lin *et al.* studied two iron meteorites in which we have identified tetrataenite rims optically. Their analysis of these rims, using a scanning transmission electron microscope with a resolution of ~50 nm, gave concentrations of 42 to 48 percent Ni, slightly lower than our values of 48–57 percent Ni measured by the electron microprobe in wider rims of other meteorites. Some, but not all of this discrepancy might be attributed to the larger error of their technique (±2 percent Ni).

Mehta *et al.* (1980) have studied a large tetrataenite grain in the Estherville mesosiderite with electron microscopy. They found the extra superlattice diffraction spots in certain regions 1–3 μm in diameter, but they identified the surrounding area which failed to show these spots as disordered taenite. Now the regions which showed superlattice spots were oriented with [110] parallel to the electron beam. The surrounding area must therefore have contained tetrataenite crystals with [101] and [011] parallel to the beam. Since these orientations do not show superlattice spots, we are confident that Mehta *et al.* also misidentified tetrataenite as taenite.

Danon *et al.* (1979b, 1980) have described a sample of pure ordered FeNi which apparently contained 40 percent Ni. They extracted this sample from the St. Séverin chondrite, using HF and a $CuCl_2/2KCl$ solution. Our results suggest that a sample of this composition must be largely cloudy taenite, and yet they did not detect extra lines from kamacite or taenite in its Mössbauer spectrum.

Our observations of cloudy taenite in Estherville confirm that in some, if not all meteorites, cloudy taenite contains tetrataenite, as Albertsen *et al.* (1978a,b) proposed. The identity of the low-Ni phase which comprises the walls of the foam structure in cloudy taenite is still in question. Scott (1973) and Lin *et al.* (1979) observed kamacite while Albertsen *et al.* found only disordered, paramagnetic taenite containing less than 30 percent Ni. If the two phases in cloudy taenite are kamacite and tetrataenite, the term cloudy taenite becomes a misnomer, as taenite is no longer present.

Fig. 2. Reflected-light photomicrograph of tetrataenite (Tt), 'cloudy taenite' (CT), and kamacite (K) in the Estherville mesosiderite. (a) A lightly etched section showing two cloudy taenite grains surrounded by a white tetrataenite rim. (b) The same area *before* etching under crossed polars, showing optical anisotropy and irregular crystals in the tetrataenite rim. (c,d,e) Magnified views under oil immersion of the left-hand taenite grain. In (c) the etched section shows a sharp boundary between cloudy taenite and tetrataenite and a mottled texture in the cloudy taenite. After a good polish (d) the reflectivity of cloudy taenite and tetrataenite are almost identical, although kamacite and tetrataenite are easily distinguished. The same *unetched* area (e) under crossed polars shows an intricate stripe pattern of magnetic domains in the tetrataenite rim. In cloudy taenite, μm-sized tetrataenite crystals are oriented parallel to rim crystals. Probe analyses show that the tetrataenite rim has 48–55% Ni, cloudy taenite 40.5–43.5% Ni, and kamacite 4–7% Ni. Both scale bars: 50 μm.

Heating experiments by Albertsen *et al.* (1978b) showed that tetrataenite could be disordered by heating at 460°C for 10 hours. For synthetic FeNi an ordering temperature of 320°C has been obtained (Paulevé *et al.,* 1962). Our observations on the distribution of tetrataenite support the conclusion of Albertsen *et al.* that ordered FeNi forms at these low temperatures during slow cooling. We also find that the abundance of tetrataenite is approximately inversely correlated with the meteorite cooling rate. In mesosiderites, for example, which have the slowest cooling rates of 0.1 °C/m.y., metal is composed very largely of tetrataenite and kamacite. Taenite, if Albertsen *et al.* (1978b) are correct, occupies only the walls of the foam-like intergrowth of cloudy "taenite"; if the walls are kamacite, however, there is no taenite in mesosiderites. We suggest that kamacite and tetrataenite are the stable phases for metal of meteoritic Fe–Ni compositions below 300°C.

Tetrataenite has only been synthesized in nuclear reactors and electron accelerators close to 320°C (see Gros and Paulevé, 1970; Chamberod *et al.,* 1979). However, to synthesize ordered FeNi without radiation may not require the excessively slow cooling rates of meteorites. For meteoritic metal, which has bulk Ni compositions of ~5–30 percent, the rate-determining factor in the growth of tetrataenite is probably diffusion of Ni through kamacite and taenite, rather than ordering of Fe and Ni. Mechanisms for the growth of tetrataenite and its implications for cooling rate calculations will be discussed elsewhere.

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