Metallography of IRON METEORITES

This article describes the microstructures of the various meteorite types, lists the minerals typically found in them, explains color etching techniques, and describes the metallography of the Odessa and Coahuila meteorites.

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Meteorites are grouped into three basic types: stones, stony irons, and irons. Within these groups, the classification of meteorites is a complex subject. For the iron meteorites, classification is based upon chemical composition, macrostructure, and microstructure. Basically, iron meteorites fall into three categories – hexahedrites, octahedrites and ataxites. However, some do not fully fit the requirements of these categories and are classified as anomalous.

Displays of meteorites in museums generally consist of large, solid chunks of iron meteorites and of etched slices. These slices are ground smooth and then etched with a strong acid solution that brings out the growth structure. The octahedrites are commonly exhibited in this manner because they undergo a solid-state phase transformation in which the kamacite (ferrite) nucleates and grows along the octahedral planes of the parent taenite (austenite) phase, producing a beautiful etched pattern.

The kamacite phase in octahedrites grows very slowly. Based upon the known rate of diffusion of nickel in iron, the cooling rate between about 700 and 400ºC, where the phases transform, has been found to vary from about 1 to 250ºC per million years!

It is important to examine the gross macrostructure, defined as the structure revealed by a large ground section, and viewing it with the unaided eye, or at low magnification. However, much more information can be found by microscopic examination. Because iron meteorites are opaque to light, they must be examined with a reflected light microscope, just like man-made metals such as irons and steels.

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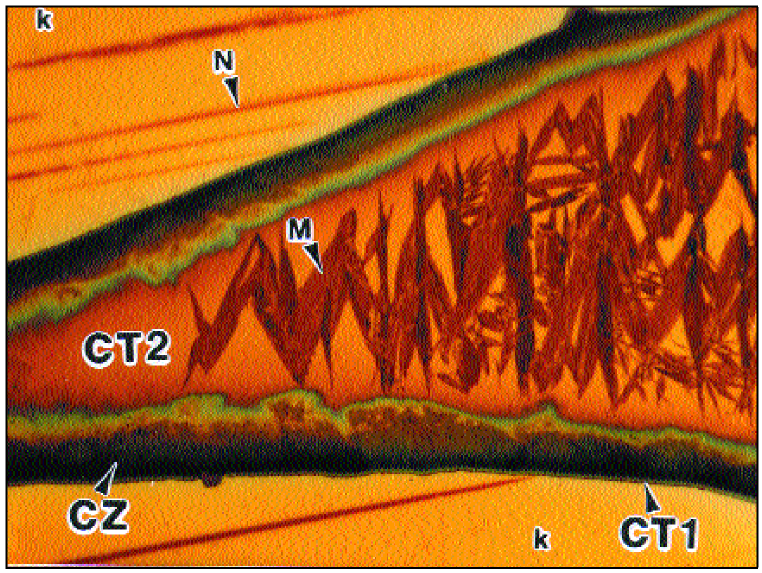

Fig. 1 — Color micrograph (400X) of Odessa, a coarse octahedrite, etched with Beraha's complex CdS tint etch revealing a taenite wedge containing a clear taenite outer rim (CT1), a cloudy zone (CZ), an inner clear taenite zone (CT2,) and martensite (M), surrounded by kamacite (k) containing Neumann bands (N). Beraha's etch.

This article describes the microstructures of the various meteorite types, list the minerals typically found in them, explains color etching techniques, and describes the metallography of the Odessa and Coahuila meteorites.

Microstructures

Iron meteorites are iron-nickel alloys with nickel contents from about 4.3 to 34 weight percent. In general, most have nickel contents from about 5 to 10 wt%. Small amounts of cobalt are also present, generally from about 0.4 to 1.0%. Sulfur, phosphorus, and carbon are also present, but their amounts are quite variable. Trace levels of many other elements can be found.

Iron meteorites are classified into three types, based on the bulk nickel content. These include

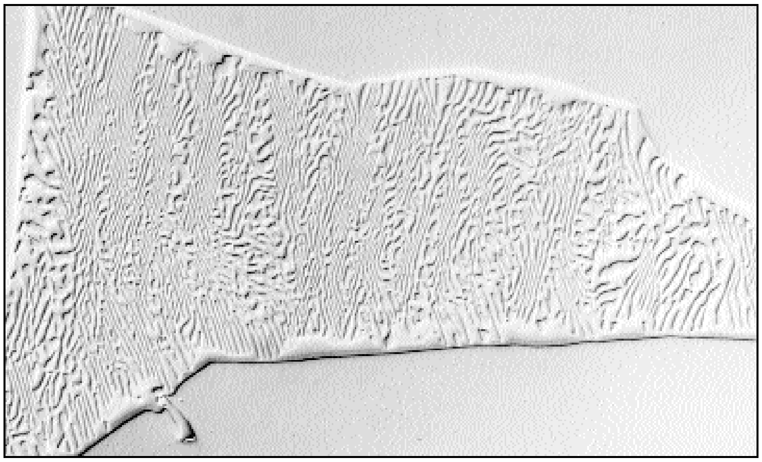

Fig. 2 — A patch of plessite, a mixture of kamacite and taenite, surrounded by kamacite in Odessa, a coarse octahedrite that fell in Texas, viewed with DIC at 400X in the as-polished condition.

Hexahedrites have nickel contents in the range of 5.2 to 5.8 weight percent.

hexahedrites, octahedrites, and ataxites.

• *Hexahedrites* are single crystals of kamacite; that is, no grain boundaries are observed, even in quite large specimens. Typically, hexahedrites have nickel contents in the range of 5.2 to 5.8 weight percent. Other phases, chiefly iron-nickel phosphides, develop in hexahedrites, with the result that not all of the nickel is in solution in the kamacite.

• *Octahedrites* contain about 5 to 10 weight percent nickel, and two main phases — kamacite and taenite. Taenite forms upon solidification. With subsequent cooling, kamacite is nucleated on the octahedral planes of the taenite, and grows in a preferred pattern. The kamacite that forms is lower in nickel content, generally about 5.5 to 7.5%, than the bulk nickel content. Thus, the movement of nickel atoms causes the remaining taenite phase to become enriched in nickel. As the bulk nickel content increases, the bandwidth of the kamacite grains decreases. Octahedrites are classed in five steps from coarsest (lowest nickel) to finest (highest nickel) and then plessitic (distinct bands no longer exist).

Plessite is a two-phase mixture of kamacite and taenite. The morphology of the mixture changes, and the amount increases, with increasing nickel content. A wide variety of rather colorful jargon has been created to describe these different plessitic patterns.

• *Ataxites* contain higher nickel contents than octahedrites, frequently in the 15 to 18% range. Unlike the octahedrites, they do not exhibit gross macrostructural patterns. Relatively few ataxites have been found. The kamacite in ataxites is equiaxed; it is approximately equal in size in all directions. Also, their grain diameter is small, about 30 µm or less. Kamacite grains in octahedrites are quite long and narrow, with lengths of up to several centimeters not unusual. Length-to-width ratios of 10 to 30 are commonly observed.

The kamacite phase in meteorites has a body-centered cubic crystal structure identical to that of α-iron in steels. Taenite has a face-centered cubic crystal structure identical to that of γ-iron in certain types of stainless steels. When kamacite is deformed, as in extraterrestrial impacts between asteroids, twinning occurs if the rate of strain is high (as in a collision) and the temperature is low (as in outer space). These twins are called Neumann bands and they are ubiquitous in kamacite in hexahedrites and octahedrites, unless they have recrystallized due to reheating. Neumann bands are not commonly observed in ataxites, unless the grain size is rather large. Neumann bands are long (up to several centimeters is possible) and narrow (1 to 10 µm).

When kamacite forms and grows in octahedrites, thin films of residual taenite can be observed between portions of some of the adjacent kamacite grains. Also, wedge-shaped patches of taenite can be observed at kamacite grain junctions (Fig. 1). If these patches are relatively large, greater than about 50 µm in diameter, martensite may form in the central region where the nickel content is less than 25%. The nickel content of these taenite patches is highly variable. At the extreme surface, adjacent to the kamacite, nickel contents of about 50% appear in a very thin zone, 1 to 2 µm wide. Below this thin layer, the nickel content gradually drops to about 28 to 30%. This zone is called the "cloudy" zone because of its appearance after etching with nital. Beneath this zone, the nickel content continues to decrease until martensite is observed when less than 25% is present. The structure of these regions is very complex. Transmission electron microscopy is required to study it properly.

Carbides can be observed in certain meteorites, but not all. The most common carbide in meteorites is called cohenite, with the general formula $(Fe,Ni,Co)_3C$. This type of carbide also develops in steels, and is called cementite. It has the general formula M_3C, where M refers to a metal, mainly iron. Small amounts of iron could be replaced by manganese and chromium, but cementite does not contain nickel or cobalt. An alloy carbide, called haxonite, has been observed in a few meteorites. Both carbide types are very hard.

Other meteorite elements

Phosphorus is very common in meteorites, but in much greater concentrations than in steels. When the phosphorus level exceeds about 0.06 weight percent, which is quite common in meteorites, phosphides are formed. They have a tetragonal crystal structure with the general formula $(Fe, Ni)_3P$, but different morphologies are observed.

• *Schreibersite* is the name given to globular shaped phosphides in meteorites. Their size is a function of the phosphorus content. Schreibersite has a variable nickel content, depending upon the temperature at which the globules nucleated, from about 10 to 50% nickel. The higher nickel content particles tend to be smaller in size, and they are more ductile than the larger, lower-nickel particles, which are often cracked.

• *Rhabdites* are phosphides that have plate-like or prismatic shapes. These types of phosphides are most common in hexahedrites. In most instances, the prismatic-shaped particles are crack free, but the plate-like shaped particles exhibit numerous transverse cracks.

Many other mineral phases have been found in meteorites, but they are much less common than those discussed above. Also, within a given meteorite these phases may be rather erratically distributed, rather than uniformly distributed.

Etching methods

Techniques that have been developed for iron and steel specimens are directly applicable to meteorites. After final polishing, the specimens must be etched. Without etching, very little can be observed of the microstructure, unless viewed with differential interference contrast (DIC) illumination. Bright field (BF) enables detection of cracks, terrestrial corrosion, and certain second phases (graphite, sulfides, silicates, etc.). If a small amount of relief is developed during polishing, DIC can reveal the plessite constituent, Fig. 2, and phosphides and carbides very well. This figure shows plessite in Odessa, a coarse octahedrite meteorite that fell in Texas.

Nital and picral are the most common chemical etchants for meteorites, as well as for steel. Nital is a solution consisting of 1 to 4% nitric acid in ethanol. Picral is a solution consisting of 4g picric acid dissolved in ethanol. Both etchants dissolve the kamacite phase preferentially to other phases.

Nital is quite sensitive to the crystallographic orientation of the kamacite, while picral is not. Hence, picral dissolves the kamacite uniformly while nital does not. Nital reveals kamacite grain boundaries, but picral does not. Picral does not reveal the Neumann bands or as-formed martensite. Nital reveals both well. Nital is preferred for the overall study of the microstructure of meteorites, although picral is much better for examining the plessitic structures.

As an example, Fig. 3 shows views of the same patch of plessite in Gibeon, a fine octahedrite that fell in Southwest Africa. (Meteorites are named after the town closest to the fall site.) Picral, Fig. 3a, reveals the very fine taenite particles within the patch. Note the thin band of white taenite around the outer edges of the patch. Just inside the edge is a dark etching zone of "black" plessite, a very fine mixture of kamacite and ferrite. Within the plessite patch, we observe very fine particles of taenite in a kamacite matrix. Etching with nital, Fig. 3b, reveals grain boundaries within the kamacite matrix of the plessite, which obscures the fine taenite particles. Note that some Neumann bands and some subgrain boundaries can now be observed in the kamacite surrounding the plessite.

Figure 4 shows an example of a large patch of taenite between kamacite grains where the nickel content was low enough in the center for martensite to form. At the very edge of the taenite wedge is a thin layer of clear taenite where the nickel content is about 50%. Below this is a dark etching band called the "cloudy zone," which actually contains two phases (only resolvable with the transmission electron microscope). The cloudy zone has a bluish / brownish color when etched with nital. Between the cloudy zone and the martensite central area is a second region of clear taenite.

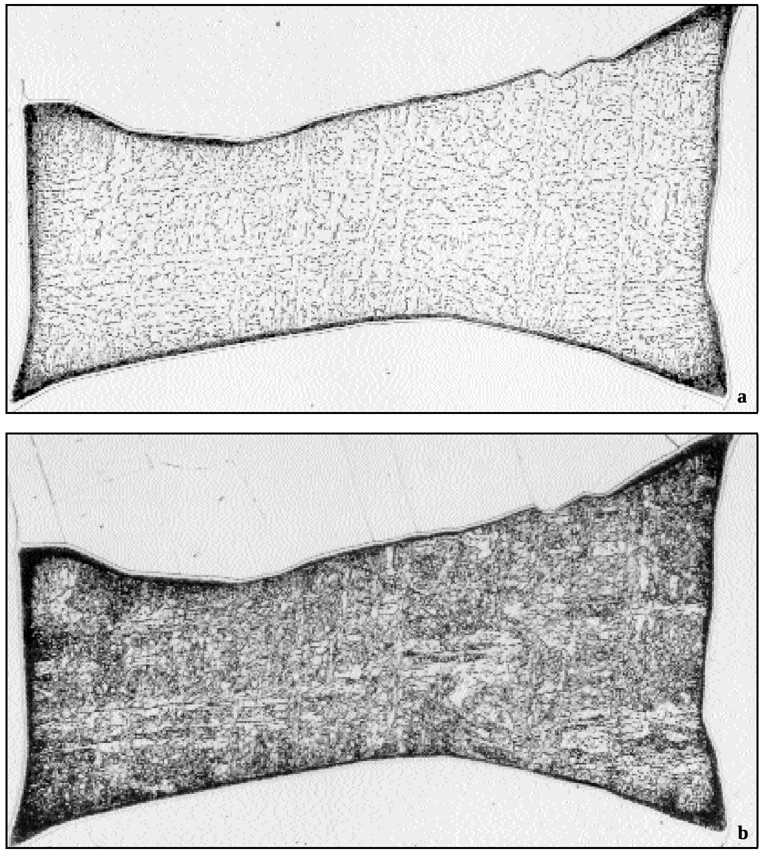

Fig. 3 — An example of finger plessite in Gibeon, a fine octahedrite that fell in Southwest Africa. (a) etched in 4% picral; (b) etched in 2% nital (200X).

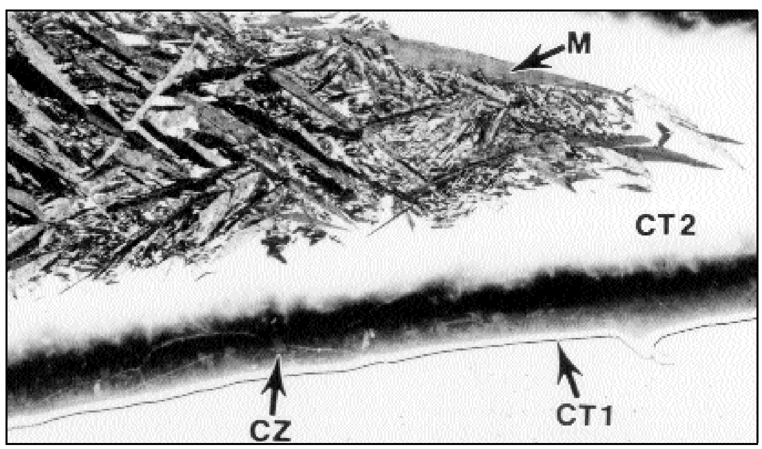

Fig. 4 — A large patch of taenite containing a thin outer layer of clear taenite (CT1), a dark etching cloudy zone (CZ), an inner region of clear taenite (CT2) and martensite (M) in the central region in the Canyon Diablo meteorite, a coarse octahedrite that fell in Arizona (etched with 2% nital, 400X).

Tint etching meteorites

Natural color is not usually seen in meteoritic microstructures. Color can be introduced by DIC, but it has no physical significance or value. Polarized light may produce some color effects with graphite and certain mineral phases, but these are not commonly observed. Crossed polarized light, in some instances, can produce color effects in coarse martensite within taenite wedges, as shown in Fig. 5. This is a high magnification micrograph of coarse martensite in the Odessa meteorite in nearly crossed polarized light and a sensitive tint plate (which has colored the residual taenite magenta).

The best approach to introduce meaningful color is to tint etch ("stain" etch) the specimen, generally after a light etch with picral or nital. Tint etchants are aqueous or alcoholic solutions that are balanced to produce a thin film on the specimen surface, typ-

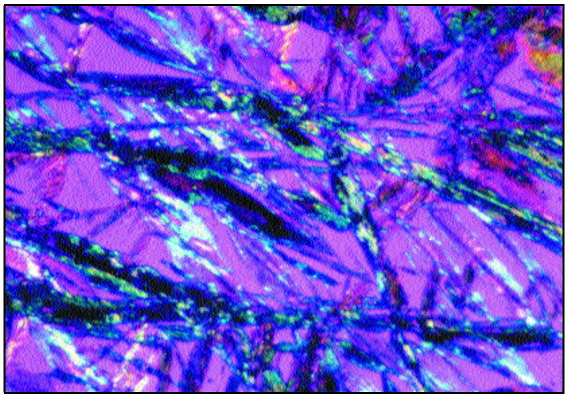

Fig. 5 — Martensite in a patch of taenite (colored magenta) in Odessa observed using nearly crossed polarized light and a sensitive tint plate (etched with 2% nital, 1000X).

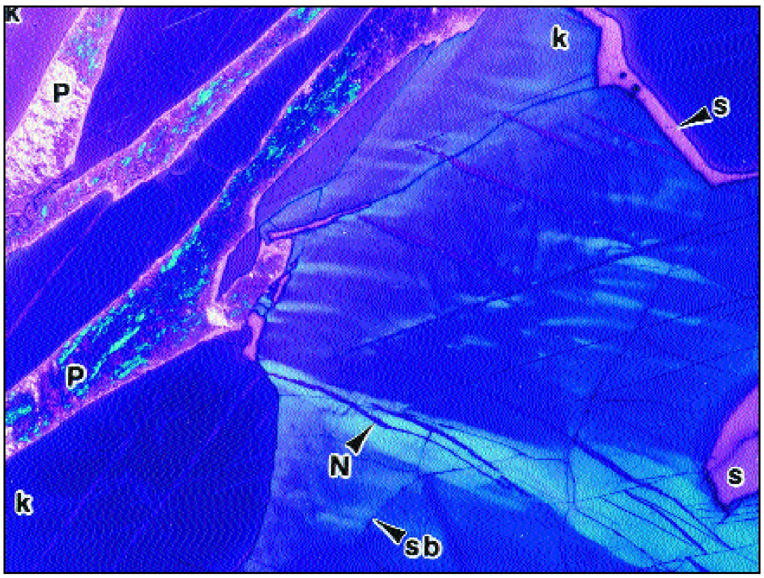

Fig. 6 — Color micrograph of Odessa, a coarse octahedrite, tint etched with sodium metabisulfite and viewed with nearly crossed polarized light and sensitive tint revealing (50X): plessite (P), Neumann bands (N), schreibersite (s), kamacite (k) and subgrain boundaries (sb).

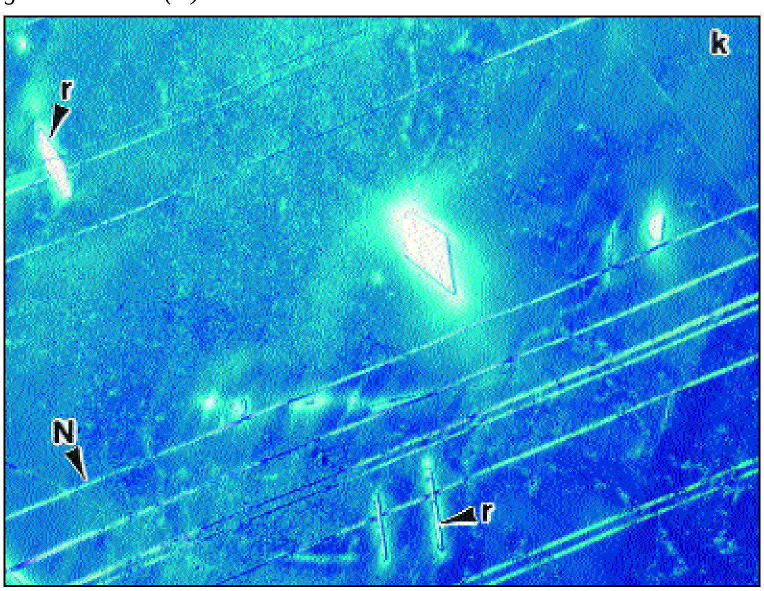

Fig. 7 — Color micrograph of Coahuila, a hexahedrite that fell in Mexico, after tint etching with sodium thiosulfate and potassium metabisulfite revealing (200X): kamacite (k), Neumann bands (N), prismatic-shaped and plate-shaped rhabdites (r).

ically 40 to 500 nm thick, of an oxide, sulfide, complex molybdate, chromate, or elemental selenium. Colors are developed by interference in the same manner as films produced by heat tinting or by vacuum deposition of a dielectric compound with a high index of refraction (the Pepperhoff method). The film thickness controls coloration. As the thickness increases, at a certain thickness interference begins and colors are produced, beginning with yellow, then green, red, violet, blue and silver. Both first order and second order colors can be observed as the film grows.

Experiments have shown that certain tint etchants that work well with carbon and alloy steels do not color meteorites. This is due to the compositional differences between meteorites and ordinary steels. For example, Klemm's I is a standard anodic tint etch that colors ferrite in carbon and low-alloy steels. However, it does not color kamacite (ferrite) in meteorites because of their high nickel contents. Beraha's sodium molybdate cathodic tint etch colors cementite (Fe_3C) in carbon and low- alloy steels, even if some manganese or chromium is dissolved in the cementite (replacing some of the iron). However, it does not color cohenite $(Fe,Ni,Co)_3C$, in meteorites. What works and what does not work, can be determined only by experimentation, although results of past experiences may suggest what to do.

The tint etchants are used by immersion, that is, the polished specimen is placed face up in a container holding about 100 mL of the solution. The surface is never swabbed with a cotton tuft soaked with the solution, because the film does not form under such conditions. Etch time varies between about 45 and 300 seconds, depending on the solution.

Odessa and other meteorites

One of the simplest color etchants is an aqueous solution of 10% sodium metabisulfite ($Na_2S_2O_5$). This solution can tint etch ultra-high strength, ultra-high toughness Ni-Co steels. To get good color effects, it is necessary to view the specimen with nearly crossed polarized light and a sensitive tint plate. Figure 6 shows the microstructure of the Odessa meteorite etched with this solution and viewed in this manner. In the upper left corner are two patches of plessite which, because of the lamellar pattern of the kamacite and taenite, respond brightly. In the upper and lower right areas are pink phosphides (schreibersite) containing cracks. They are pink because of the sensitive tint plate; they would otherwise be white.

The matrix consists of several kamacite grains colored light violet (upper right corner), purple (left side, top to bottom) and blue (variable blue colors in the right half of micrograph). Note the numerous straight or slightly curved red lines in the kamacite. These are Neumann bands from extraterrestrial shock events. In the blue kamacite region, some very faint Neumann bands can be seen. These are probably from much older shock events. Also in the blue kamacite is a series of fine blue lines that are interconnected. These are subgrain boundaries. A few very small phosphides are observed on these lines. Note also the variation in the blue region of kamacite on the right half of the micrograph. This is from either chemistry variations or from residual stress in this area.

• *The Coahuila meteorite* (Fig. 7) is a hexahedrite that fell in Mexico. It was etched with an aqueous solution containing 10% sodium thiosulfate ($Na_2S_2O_3$) and 3% potassium metabisulfite ($K_2S_2O_5$), and is viewed with bright field. Like most hexahedrites, the Coahuila specimen is a single crystal, that is, the kamacite matrix has no internal grain boundaries. Note that all of the kamacite is colored blue. A number of parallel light blue lines run diagonally left to right; these are Neumann bands. The white particles are phosphides, both prismatic and plate shaped (in three dimensions). Note that one prismatically-shaped rhabdite in the upper left corner has cracked due to the intersecting Neumann band. Note also the variation in color around the large prismatically-shaped rhabdite in the center of the image, caused by compositional variations around the phosphide. The kamacite background has a rough appearance because of many very small (<1 μm diameter) phosphides.

Beraha's complex thiosulfate tint etch that forms a cadmium sulfide interference film works very well with meteorites (but not the lead sulfide complex reagent).

• *Arispe* (Fig. 8) is a coarse octahedrite that fell in Sonora, Mexico, tint etched with Beraha's CdS reagent. This shows a high magnification view of plessite in Arispe where both kamacite and taenite are colored. Surrounding the plessite, kamacite is colored brown. In the lower left corner, some noncolored cohenite is present. At the edge of the plessite is a tan rim of taenite. The kamacite within the plessite is light blue, while the taenite particles in the plessite are tan.

• *Henbury* (Fig. 9) is a medium octahedrite that fell in Australia. Thisa specimen was etched with sodium thiosulfate and potassium metabisulfite. The meteorite was subjected to a violent event that ripped it apart, perhaps when it exploded coming into the Earth's atmosphere. The structure is mostly highly deformed kamacite containing many fine mechanical twins (Neumann bands). In the upper right corner a large phosphide is visible.

• *Canyon Diablo* (Fig. 10) was etched with Beraha's selenic acid tint etch. This reagent has no fixed composition, but it consists of hydrochloric acid (up to 20 mL), selenic acid (up to 3 mL), and ethanol (amount added so that the total is 100 mL). One must experiment to find the best composition. Pre-etching with nital is required for best results. The figure shows two patches of cohenite colored orange brown. Note that in the center of the field, greenish patches of schreibersite are attached to the cohenite. The cream and tan patches are taenite. The matrix is highly deformed kamacite containing some faint subgrain boundaries. ■

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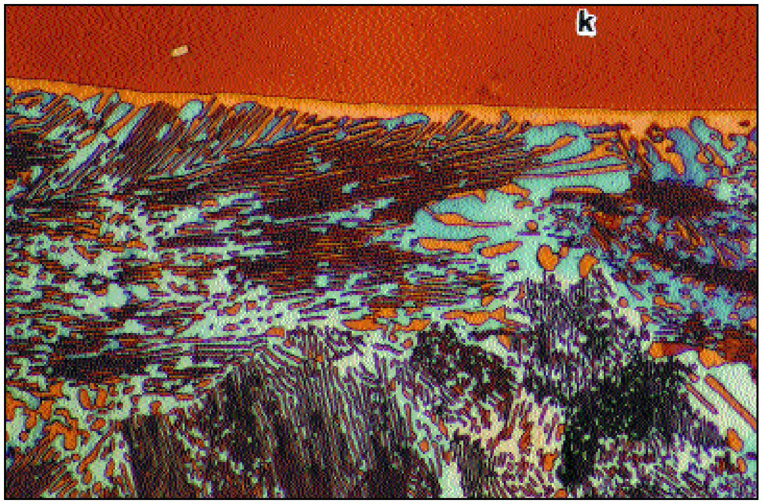

Fig. 8 — Color micrograph of Arispe, a coarse octahedrite that fell in Mexico, tint-etched with Beraha's complex CdS tint etch. It reveals (400X): kamacite (k), plessite where both the kamacite and taenite are colored, and cohenite (C).

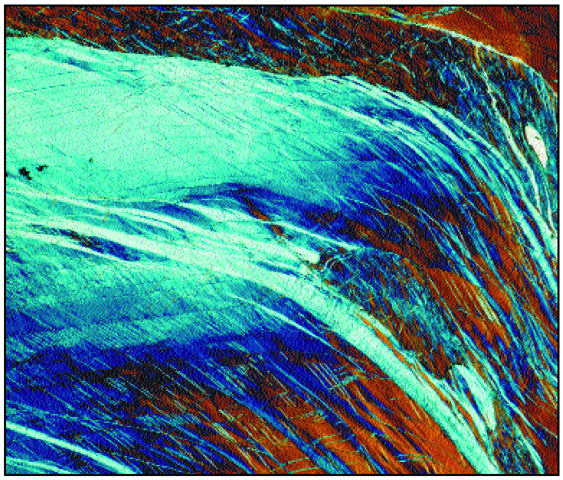

Fig. 9 — The Henbury meteorite after etching with sodium thiosulfate and potassium metabisulfite. Henbury is a medium octahedrite that fell in Australia.

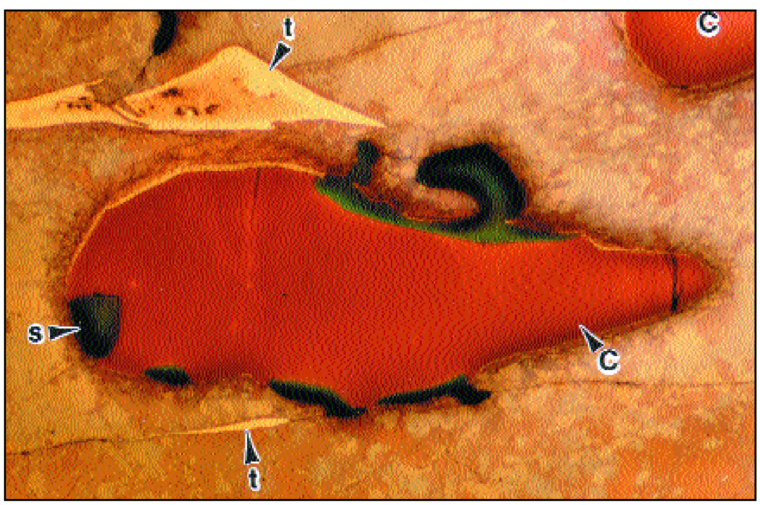

Fig. 10 — The microstructure of the Canyon Diablo meteorite. It was etched with Beraha's selenic acid tint etch.

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Of general interest, **Circle 281**
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