JOURNAL OF MATERIALS SCIENCE 9 (1974) 564–568

A structural investigation of the Cape York meteorite by transmission electron microscopy

R. A. JAGO*
Technical University of Denmark, Lundtofte, Denmark

Examination of the kamacite phase of the Cape York meteorite by TEM revealed the following principal microstructural features: subgrains, Neumann bands, slip traces, dislocation networks, dislocation loops and precipitate particles. Quantitative data on many of these features have been collected.

The significance of a pre-terrestrial shock event in explaining the origin of several of these features is discussed and microstructural evidence is presented showing that the maximum shock pressure encountered by the meteorite was in the range 70 to 120 kbar. This result is at variance with that claimed by other workers.

1. Introduction

The examination of iron meteorites by optical microscopy has been carried out systematically over several decades now, and a large volume of literature describing the observed microstructures has accrued. By this means, the origin of certain microstructural features has been inferred, e.g. kamacite (ferrite) lamellae in the extremely low cooling rates of the meteorites, and Neumann bands in some subsequent, but still pre-terrestrial, shock event. These, and other features which are frequently observed are now well documented, and have permitted a classification of meteorites on the basis of their microstructural characteristics.

In spite of the large amount of information gained from optical microscopy, little thought has been given to the systematic application of electron microscopy to the study of meteorites. This technique, with its inherent advantage of high resolution together with the facility of electron diffraction, should make possible a much more detailed study of microstructural features than previously, and permit a better description of the meteorite's pre-terrestrial history to be obtained thereby. The aim of this investigation, therefore, was to document the structural features observed in the electron microscope and to interpret these in the light of the cosmic processes responsible for them.

2. Background details

The Agpalilik meteorite was discovered on the Agpalilik Peninsula[†], Greenland in 1963 [1]. The find is an iron meteorite of 20 ton mass, and belongs to the Cape York group of meteorites.

After transportation to Copenhagen, the mass was sectioned for examination. Fig. 1 is a macrophotograph of part of a polished and etched cross-sectional slice of the meteorite showing the kamacite bands and a troilite (FeS) precipitate. The continuity of the system of kamacite lamellae throughout the slice and elsewhere in the meteorite indicates that the entire mass was, at some stage, a single crystal of taenite (austenite) before transformation occurred.

The meteorite belongs to the classification Om, group IIIA (medium octahedrite), and the major constituents are:

	%		%
Ni	7.85	Co	0.50
P	0.15	C	0.02
S	1.30	Fe	rem.

---

*Present address: Department of Materials Engineering, Monash University, Clayton 3168, Victoria, Australia.
[†] Position: 76° 9′N, 65° 10′W, approximately 100 km S.E. of Thule air base.

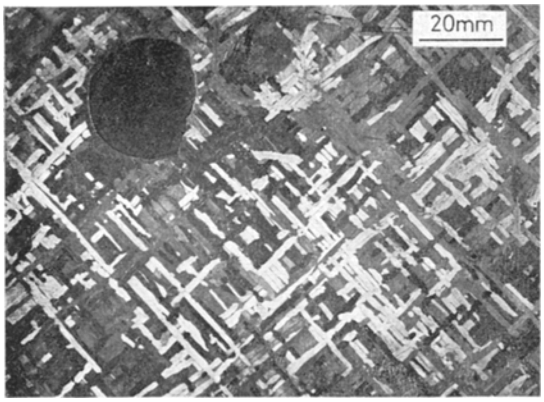

Figure 1 Macrophotograph of polished section of meteorite, showing uniformity of kamacite lamellae. Area at top left is a troilite bleb (× 1).

3. Experimental details

Samples for study were chosen from a region in the meteorite at least 35 cm away from the ablation surface. On microstructural evidence, it was known that the effects of reheating during entry into the earth's atmosphere extended no further than 30 mm below the meteorite's surface, so that the samples chosen are truly representative of the bulk of the meteorite, and contain no artefacts resulting either from atmospheric surface heating or terrestrial corrosion.

Foils for electron microscopy were prepared by thinning blanks in a Struers "Tenupol" jet-polishing machine using an electrolyte consisting of 10% perchloric acid and 90% acetic acid at a temperature of approximately 15°C. These were examined in the Hitachi HU-11A and Jeol JEM-100U electron microscopes at 100 kV, and selected foils were later re-examined in a JEM-1000D at 1 MV. As an initial step, the investigation was restricted to the kamacite phase, as this is by far the most abundant phase present in the meteorite.

4. Experimental results and discussion

4.1. General structural observations

The foils examined showed a high degree of uniformity with regard to the nature and distribution of the microstructural features observed. In general, the kamacite phase contained large subgrains exhibiting Neumann bands of several different orientations surrounded by an extensive and uniform dislocation network. Interspersed among this network were numerous small "black dots", later shown to be dislocation loops. Slip bands could frequently be distinguished in the matrix when suitable contrast conditions were obtained; these reflected the symmetry of the foil orientation. In addition to these features which are characteristic of the kamacite, precipitate particles with various morphologies were present as a secondary feature.

No evidence of recrystallization was found in the as-received specimens, and polygonization was detected only at isolated deformation twin intersections. No shear plates were evident in the microstructure.

4.2. Detailed structural observations

4.2.1. Subgrains

The entire system of kamacite lamellae is divided into numerous subgrains, having diameters ranging from 0.06 to 1.5 mm with a mean value of about 0.6 mm. Where the subgrain boundaries are cut by Neumann bands, very little deflection of the latter is seen to occur, indicating that the boundaries are low-angle and comprise relatively few dislocations. Occasionally, the subgrain boundaries are decorated by small precipitate particles, suggesting their presence before the precipitates formed.

4.2.2. Neumann bands

Neumann bands are distributed quite uniformly throughout the kamacite, and make up about 5% of the kamacite by volume. Their morphology varies markedly from narrow twins with thicknesses of 0.1 to 1 μm having smooth straight sides (Fig. 2), to quite coarse ones (up to 10 μm) with notched surfaces.

Data from single-surface analyses of seventeen twin traces in foils of various orientations showed that these lie close to the traces of {112} planes, generally within the limits of the selected area diffraction analysis technique (±5°). The data are thus not inconsistent with the {112} twinning plane observed for iron.

The presence of deformation twins in meteorites is generally believed to be the result of some shock event. Contrary to popular belief, the observed Neumann bands in meteorites and other evidence of shock loading (when present) do not generally result from the relatively minor impact with Earth [2, 3], but result either from the event that separated the meteorite from its parent body, or from some later collision in orbit. In the few instances where deformation

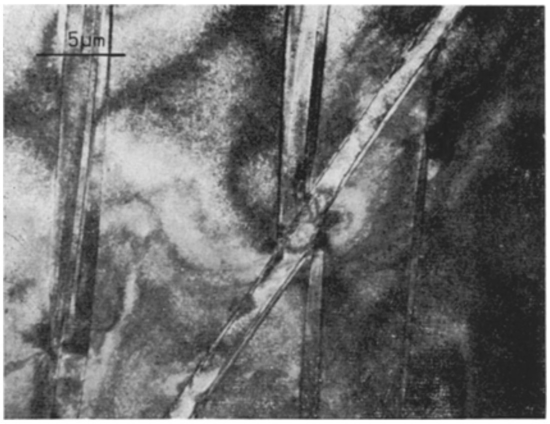

Figure 2 Neumann bands in meteorite showing intersection (1 MV).

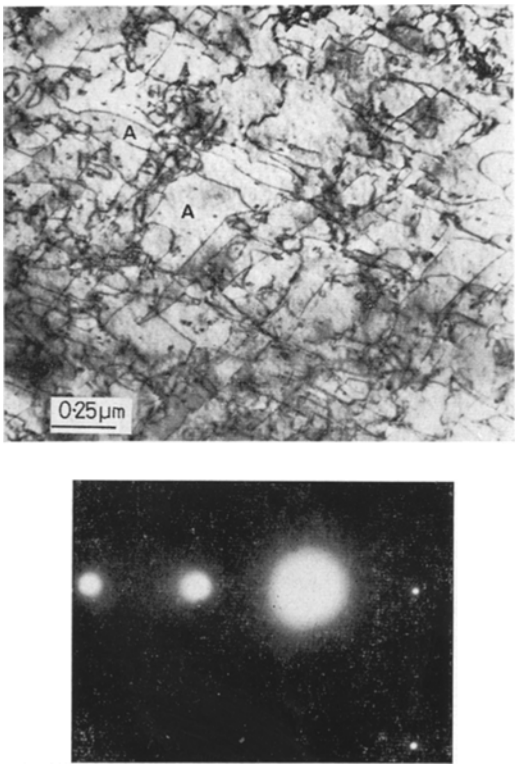

Figure 3 Dislocation configuration in {1 1 0} foil plane and corresponding SAD pattern. Traces are generally parallel to ⟨1 1 1⟩. Note dislocation loops at A.

effects are attributable to the impact with Earth, these effects are also observed in the ablation zone [2]; this was not so in the present case.

4.2.3. Slip traces

Slip traces could be easily seen in foils of any orientation when suitable diffraction conditions were obtained. Analysis of these using the pole-locus method showed that the plane responsible for the traces is {1 1 0}, the slip plane most frequently observed for iron.

4.2.4. Dislocations

The most striking feature of the microstructure of the meteorite is the regular dislocation arrays observed throughout the kamacite phase. Fig. 3 shows a section through the array parallel to the operative slip plane – {1 1 0}. Preferred alignment of the dislocation traces parallel to ⟨1 1 1⟩ is evident, while the bowing of dislocations in several instances indicates that they are strongly pinned, probably by fine precipitate particles.

Consideration of Fig. 3 shows that the dislocations are pure screws. The foil is parallel to the known slip plane and, hence, should contain primarily slip dislocations. Such dislocations are easily distinguished by their relatively greater length, while their screw character is obvious from the fact that they lie parallel to ⟨1 1 1⟩; this is the Burgers vector for slip dislocations in iron.

Determinations of the dislocation density in the kamacite making due allowance for out-of-contrast dislocations, gave a value of 4×10^{10} cm^{-2}. This value is higher than that obtained [4] for the Campo del Cielo meteorite (10^9 cm^{-2}) but is comparable with the dislocation density measured by Hornbogen and Kreye [5] for the Gibeon meteorite ($\sim 10^{10}$ cm^{-2}).

The black dots evident in Figs. 3 and 4 were shown to be dislocation loops by careful tilting experiments. The smallest resolvable loop had a diameter of 60 Å, but it was obvious that there were many smaller than this. Estimates of the loop density gave an order of magnitude value of 10^{15} cm^{-3}, but the number of loops out of contrast under any one set of diffracting-conditions would make this value an under-estimate.

4.2.5. Precipitate particles

Precipitates have several stages of formation. Those that are part of the primary structure formed early in the cooling history of the original mass of metal and are often very large, e.g. the troilite bleb in Fig. 1.

Later precipitates formed by solid state transformation after the entire melt had solidified. Fig. 4 shows a group of Rhabdite – $(\text{FeNi})_3\text{P}$ – particles with the matrix dislocation

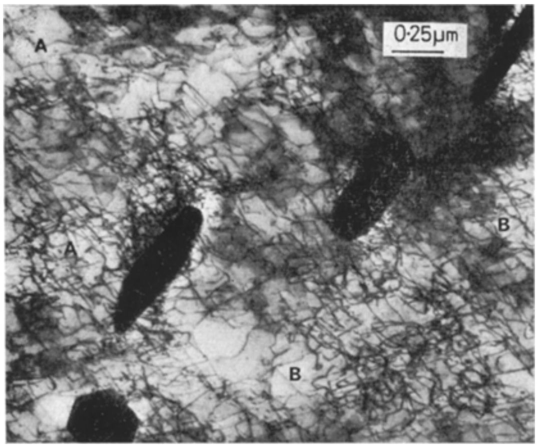

Figure 4 Rhabdite particles showing pile-up and tangling of matrix dislocations at precipitates. Note dislocation loops at A and B.

network in good contrast. It is considered that the particles shown here formed before the shock event that produced the dislocations, as a local disturbance of the regular network is evident adjacent to the particles suggesting pileup of dislocations against existing obstacles and subsequent tangling.

5. General discussion of experimental results

The regular occurrence and similarity of the observed structural features in foils from samples separated by distances of several centimetres in the meteorite demonstrate their general nature. Four of the observed features, namely slip bands, Neumann bands, dislocation networks and loops, are attributable to the shock event that occurred earlier in the meteorite's history, while subgrain and precipitate formation are easily explained in terms of the cooling and subsequent transformation of alloyed austenite containing large quantities of dissolved impurities.

The metallurgical effects of shock waves in iron and iron alloys have been reviewed by Hornbogen [6] and Mahajan [7]. In iron, as in the present case, the effect of shock loading is to cause both slip and twinning, but there is a balance between these, and the proportions of each are governed by the temperature and shock pressure. Together, the two processes account for most of the strengthening effect that accompanies shocking in metals.

Dislocation configurations identical with those observed in the present work have been reported by Hornbogen [8] and Leslie *et al* [9], and the component dislocations identified as screws. The dislocation loops (see for example, regions A in Figs. 3 and 4) are generated by the compression wave, but their role during the passage of the shock wave is uncertain. Hornbogen [8] considers that loops move with the wave, leaving behind a network of screw dislocations, but it is also argued [10] that they remain behind after the wave-front has passed in order to account for the observed strengthening.

The maximum shock pressure encountered by a meteorite in the pre-terrestrial shock event is of importance in theories of the origin of meteorites and in explaining certain of the microstructural features present. Using an X-ray diffraction technique, Jain and Lipschutz [11] concluded that a large proportion of the group III meteorites had undergone shocks of at least 130 kbar at some time in their history, and that Cape York had been shocked in the range 130 to 400 kbar. Their conclusion is not supported by the present work for the Cape York meteorite; the uniform dislocation arrays observed in the kamacite are not consistent with a shock exceeding the transition pressure (120 kbar in this alloy), since a distinct change in microstructure would be observed [9]. Further, optical microscopy does not reveal the characteristic "hatched" microstructure normally observed after the ($\alpha \rightarrow \epsilon$) phase transformation which begins at the transition pressure.

An estimate of the shock pressure experienced may be obtained by resolving the total accumulated plastic strain, γ, into a component γ_T, due to deformation twinning, and a component $\gamma_\perp$, arising from dislocation motion [12]. This gives

$$\gamma = \gamma_T + \gamma_\perp \, .$$

For a large number of twins, the shear strain is given by $\gamma_T = k\alpha$, where k is the twinning shear and α the volume fraction of twinned material. In the present case, $k = 0.707$ and $\alpha = 0.05$, hence $\gamma_T = 0.035$. In the absence of any shear strain component due to dislocation slip, $\gamma = \gamma_T$, and reference to the Hugoniot for Fe-4Ni [13] shows that this strain is associated with a shock pressure of 70 kbar. This is, therefore, the minimum possible shock pressure to which the meteorite could have been subjected, although higher values would be more consistent with the observed structures since slip, has, in fact, occurred.

The upper limit of the range of possible shock pressures is considered to be somewhat less than 120 kbar as applied stresses close to the transition pressure could produce the "hatched" structure on a local scale by the action of stress-raisers such as precipitate particles. Such structures are not, however, observed.

6. Summary and conclusions

1. Examination of the kamacite phase of the Cape York meteorite by transmission electron microscopy revealed the following principal structural features: subgrains, Neumann bands, slip traces, dislocation networks and loops, secondary precipitate particles; the observations are considered general for the whole meteorite.
2. Subgrains have a mean diameter of 0.6 mm, and are only very slightly misoriented relative to one another.
3. Single-surface trace analysis of seventeen Neumann bands using the selected area diffraction technique showed that the bands are not inconsistent with twinning on $\{112\}$.
4. Analysis of slip traces by the pole-locus method identified $\{110\}$ as the operative slip plane.
5. The Burgers vector of dislocations comprising the regular networks was found to be $\langle 111 \rangle$, so that the dislocations have screw character; this agrees with observations on shocked iron by others. The measured dislocation density was 4×10^{10} cm^{-2}.
6. Large numbers of dislocation loops are evident in the microstructure; the loop density is estimated to be at least 10^{15} cm^{-3}.
7. The shock pressure encountered by the meteorite has been shown to lie in the range 70 to 120 kbar.

Acknowledgements

The author expresses his gratitude to V. F. Buchwald for awakening his interest in the field of meteorites generally, for supplying the material for examination, and for numerous interesting discussions. Professors E. W. Langer and R. M. J. Cotterill of the Technical University of Denmark are thanked for their encouragement and constructive criticism. T. Kjer and E. Johnson made many valuable contributions which are gratefully acknowledged. The JEOL-100U microscope was made available by courtesy of the Physics Laboratory of the Oersted Institute, Copenhagen, and the State Technical and Scientific Fund (Denmark) provided a grant enabling use of the 1 MV microscope.

References

1. V. F. BUCHWALD, *Geochim. et Cosmochim. Acta* **28** (1964) 125.
2. H. J. AXON, Pre-Terrestrial Deformation Effects in Iron Meteorites, in "Meteorite Research", edited by P. M. Millman (Reidel, Dordrecht-Holland, 1969) p. 796.
3. V. F. BUCHWALD, Study of Six Iron Meteorites, *Analecta Geologica* No. 2 (1967).
4. K. H. G. ASHBEE and L. F. VASSAMILLET, *Science* **151** (1966) 1526.
5. E. HORNBOGEN and H. KREYE, *Z. Metallk.* **61** (1970) 914.
6. E. HORNBOGEN, Formation of Lattice Defects by Shock Waves, in "High Energy Rate Working of Metals", Proceedings NATO Advanced Study Institute, Sandefjord-Lillehammer (Norway) (1964) Vol. 2, p. 345.
7. S. MAHAJAN, *Phys. Stat. Sol.* [*A*] **2** (1970) 187.
8. E. HORNBOGEN, *Acta Metallurgica* **10** (1962) 978.
9. W. C. LESLIE, E. HORNBOGEN and G. E. DIETER, *JISI* **200** (1962) 622.
10. R. S. DAVIS and K. A. JACKSON, in "Plasticity", Proceedings of the Second Symposium on Naval Structural Mechanics, Brown University, Rhode Island (Pergamon, New York 1960) p. 100.
11. A. V. JAIN and M. E. LIPSCHUTZ, Shock Histories of Hexahedrites and Ga-Ge Group III Octahedrites, in "Metorite Research", edited by P. M. Millman (Reidel, Dordrecht-Holland, 1969) p. 826.
12. J. N. JOHNSON and R. W. ROHDE, *J. Appl. Phys.* **42** (1971) 4171.
13. C. M. FOWLER, F. S. MINSHALL and E. G. ZUKAS, in "Response of Metals to High Velocity Deformation", edited by P. G. Shewman and V. F. Zackay, Metallurgical Society Conferences, Vol. 9 (Interscience, New York, 1961) p. 275.

Received 10 September and accepted 24 October 1973.