# THE STRUCTURES OF METEORIC IRONS. GERHARD DERGE AND ARTHUR R. KOMMEL.1

### ABSTRACT.

The perfection of crystalline structures in Cañon Diablo and Amalia Farms meteorites was determined by X-rays and by measurement of the trace directions of the kamacite plates. The metallographic features of the Cañon Diablo sample were observed. A Widmanstätten pattern, comparable with those found in meteorites, was developed in an alloy of iron with 27 per cent nickel. These findings are discussed in relation to currently accepted metallurgical knowledge and the conclusion is drawn that the structures found in metallic meteorites were formed during slow cooling from high temperatures.

The origin and history of meteoric bodies are of both scientific and philosophical interest. Many older explanations of the structures found in metallic iron-nickel meteorites are no longer tenable. New studies of the crystallographic relationships in Widmanstätten patterns, when correlated with similar studies on synthetic alloys and a modern knowledge of physical metallurgy, have contributed considerably to an understanding of these questions.

The crystallographic relationships resulting from the transformation of the face-centered cubic lattice of gamma iron to the body-centered cubic lattice of the alpha phase have been the object of numerous researches on iron and its alloys with nickel and carbon. A detailed discussion of this problem has just been published<sup>2</sup> and only a brief review of the important features need be given here.

In a careful X-ray study of quenched 1.4 per cent carbon steels Kurdjumow and Sachs<sup>3</sup> found that the alpha orientations could be derived from those of the parent gamma by the following relationship:

$$(111)$$
  $\gamma$   $11$   $(110)$   $\alpha$   $[1T0]$   $\gamma$   $11$   $[1T1]$   $\alpha$ .

Other work indicated that all Widmanstätten structures involving these lattices could be described in the same way. However,

<sup>3</sup> Kurdjumow, G., and Sachs, G.: Ueber den Mechanismus der Stahlhärtung, Zeitschrift für Physik 64, 325-343, 1930.

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Mehl, R. F., and Derge, G.: A. I. M. E., Iron and Steel Division Preprint, T. P. 797 (1937). The press releases on this paper were not approved by the authors and do not express their ideas on the subject.

Nishiyama<sup>4</sup> found that when alloys of iron with 30 weight per cent of nickel were transformed in liquid air, the following relationship existed:

$$(111)$$
  $\gamma$  11  $(011)$   $a$   $[\overline{2}11]$   $\gamma$  11  $[01\overline{1}]$   $a$ .

Wassermann<sup>5</sup> confirmed both relations but did not explain why two different crystallographic mechanisms should operate in these alloys. In studying this latter problem Mehl and Derge found that both mechanisms could operate in the same alloy, that of Kurdjumow and Sachs being effective above room temperature while that of Nishiyama was effective at —195° C. In the course of this study some interesting information on meteoric structures was accumulated which is described in this

The crystallographic studies of the Widmanstätten pattern found in meteorites have already been reviewed,6 the most important being the X-ray work of Young.7 His results did not indicate clearly which of the above mechanisms operated in meteorites, for reasons which will become apparent. Bøggild<sup>8</sup> also studied kamacite orientations with a goniometer, using the reflections from the oriented rhabdites (iron-nickel phosphides) within the kamacite plates. This work was of a lower order of accuracy than Young's, but nevertheless is in good agreement with it.

## STRUCTURES IN METEORITES.

The gross patterns found in meteorites offer a unique opportunity for studying orientation relationships which cannot be equaled in alloys synthesized in the laboratory, and it is for this reason that these materials were re-studied to determine which of the two proposed transformation mechanisms operated in

Reports Tôhoku Imp. Univ. Series I, 23, 637-664, 1934.

<sup>6</sup> Wassermann, G.: Ueber den Mechanismus der Alpha-Gamma Umwandlung des Eisens, Mitt. Kaiser-Wilhelm-Inst. für Metallforsch. 17, 149-155,

Young, J.: The Crystal Structure of Meteoric Iron as Determined by

X-rays, Pro. Roy. Soc. 112, 630, 1926.

\* Βφggild, O. B.: The Meteoric Iron from Savik, Near Cape York, North Greenland. Saertryk of Meddeldser om Grφnland, 74, 1-30, 1927.

<sup>&</sup>lt;sup>4</sup> Nishiyama, Z.: X-ray Investigation of the Mechanism of the Transformation from Face-Centered Cubic Lattice to Body-Centered Cubic, Science

<sup>&</sup>lt;sup>6</sup> Mehl, R. F., and Barrett, C. S.: Studies upon the Widmanstätten Structure, I. Introduction. The Aluminum-Silver and Copper-Silicon Systems, Trans. A.I.M.E. 93, Institute of Metals Division 78-122, 1931. Also, see

low Ni alloys. The orientation of the original taenite (f.c.c.) can be determined stereographically from measurement of the trace directions of the kamacite (b.c.c.) plates of the specimen, since all investigations agree that these plates form parallel to the {111} planes of the taenite. The orientations of the individual kamacite lamellae can then be determined by back reflection Laue photograms and related to the taenite orienta-



Fig. 1. The Widmanstätten Pattern of the Amalia Farms Meteorite. x 1.

tion. Since the kamacite orientations which would result from the operation of the Nishiyama mechanism would differ from those resulting from the Kurdjumow and Sachs mechanism by only 5° 16′, the specimen must have a high degree of crystalline perfection if a differentiation between the two relationships is to be made.

A section of the Amalia Farms<sup>11</sup> fall was selected because of the exceptional regularity of its pattern, Fig. 1. The cross-

<sup>&</sup>lt;sup>o</sup> See reference 6.
<sup>10</sup> Greninger, A. B.: Determination of Orientations of Metallic Crystals by Means of Back Reflection Laue Photographs, Trans. A.I.M.E., Institute of Metals Division 117, 61-74; describes a recent simplification of this, 1935.
<sup>11</sup> This was loaned through the courtesy of Doctor Avinoff of the Carnegie Museum, Pittsburgh, Pennsylvania.

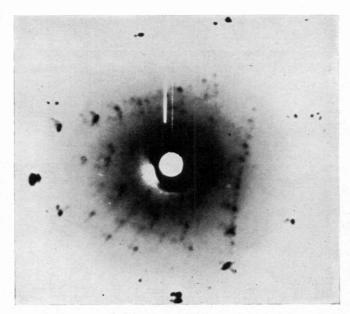


Fig. 2. Back-Reflection Laue Photogram of a Kamacite Plate in Amalia Farms Meteorite.

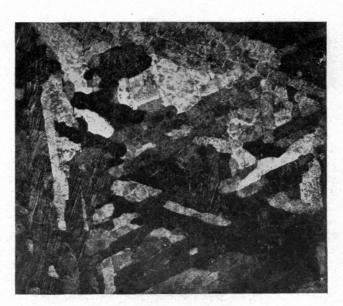


Fig. 3. The Widmanstätten Pattern of the Cañon Diablo Meteorite. x 1.5. Streaks which appear to be scratches are Neumann Bands.

section of this sample was about ten inches square. Within this area the three well defined trace directions showed a maximum deviation from the average of 0.3°, 0.8°, and 1.2° respectively. However, back-reflection Laue photograms of the individual plates showed that the lattice was highly imperfect, Fig. 2, and therefore no attempt was made to solve the orientation relationships in this sample.

Samples of the Cañon Diablo fall were also available. This, when compared with that from Amalia Farms, has a very coarse and much less regular Widmanstätten pattern, Fig. 3.

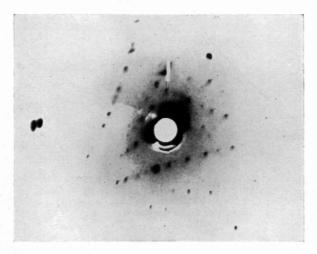


Fig. 4. Back Reflection Laue Photogram of a Kamacite Plate in Cañon Diablo Meteorite.

In an area ten inches square, the best defined trace direction showed a deviation of  $1.8^{\circ}$  from the average. However, backreflection Laue photograms showed that the individual kamacite plates had a very perfect lattice so that their orientations could be determined with an accuracy of  $\pm$  1°, Fig. 4. It was estimated that by confining the measurements to a two by six inch area, the taenite orientation could be determined within  $\pm$  2°. In spite of this macro-imperfection, the orientations of all kamacite plates could be plotted with regard to the same reference position so that a pole figure derived in this manner might be expected to indicate which orientation relationship existed. The {110} pole figure for fourteen kamacite plates was made and all of the data rotated into a unit stereographic triangle. This is shown in Fig. 5, which also gives the posi-

tions of {110} a poles predicted by the mechanisms in question. Evidently the scatter of orientations is so great that it precludes any choice between mechanisms. For this reason

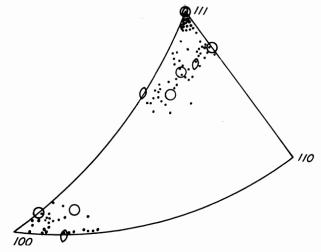


Fig. 5. {110} α Pole Figure for 2" x 6" Area of Cañon Diablo Meteorite. Unit stereographic triangle showing:

(a) The {110} α poles predicted by Kurdjumow and Sachs, 0.
(b) The {110} α poles predicted by Nishiyama, 0.
(c) The {110} α poles determined experimentally, 0.
(d) The gamma poles as designated.

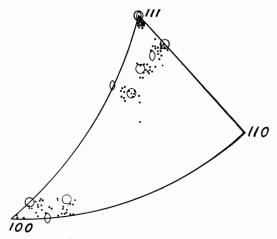


Fig. 6. {110}  $\alpha$  Pole Figure for  $1\frac{1}{4}$ " x  $1\frac{1}{4}$ " Area of Cañon Diablo Meteorite. Notation the same as for Fig. 5.

the data of Young, which are based upon a few isolated taenite and kamacite orientations, cannot be considered as conclusive proof for the existence of either mechanism in the meteorite studied.

In view of the crystalline perfection of the individual plates and the accuracy with which their orientations could be determined, it was reasonable to assume that a choice of mechanisms could still be made by confining the measurements to a sufficiently small area. Orientations were obtained for eighteen kamacite lamellae passing through an area  $1\frac{1}{4}$  inches square and the resultant  $\{110\}$  pole figure is plotted in Fig. 6. In spite of a great deal of scatter, the experimental points fall into three distinctly separate groups about the (111)  $\gamma$  pole. Since this meets the requirements of the Kurdjumow and Sachs relationship while that of Nishiyama calls for only two positions in this region of the projection, it is concluded that although the gross structure of this meteorite is imperfect, the  $\gamma$ —a transformation followed the mechanism of Kurdjumow and Sachs.

The contrast in types of perfection shown by these two meteorites is striking. The Widmanstätten pattern of the Cañon Diablo sample is coarse and highly imperfect, while the crystal lattice of any individual lamella is very perfect. On the other hand, the Widmanstätten pattern of the Amalia Farms specimen is comparatively regular, but the lattices of the kamacite lamellae are badly distorted. It appears that in this meteorite the conditions during transformation were such that the kamacite plates were forced to follow the pattern dictated by the octahedral planes of the well formed taenite matrix with consequent distortion of the kamacite lattice, because of stresses resulting from the volume changes accompanying the transformation. Comparatively rapid cooling probably occurred. On the other hand the larger plates of the Canon Diablo sample indicate that it cooled more slowly so that stress relief by annealing occurred during formation of the kamacite, consequently its lattice is more perfect. This is submitted only as a reasonable explanation of the experimental facts.

A micrograph made of the Cañon Diablo sample is of interest, Fig. 7. The large central area, horizontal on the page, is an individual kamacite plate. Apparently this is composed of many small grains, but all of the back-reflection Laue photograms, taken at random positions on over thirty different lamellae, showed that each plate had a single orientation and

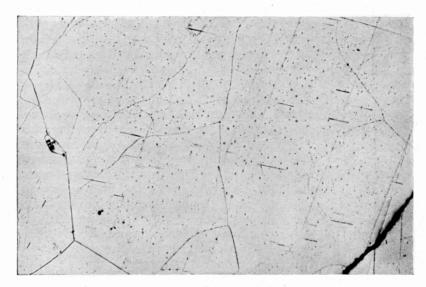


Fig. 7. Photomicrograph of Cañon Diablo Meteorite. x 60. Nital Etch.

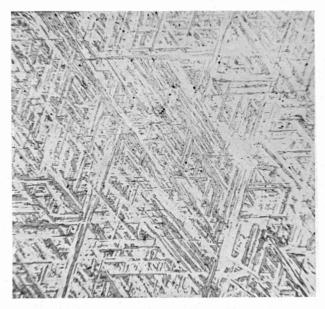


Fig. 8. The Widmanstätten Pattern of a Slowly Cooled Alloy of Iron with 27% Nickel.  $\times$  50.

a quite perfect lattice. Closer examination of the micrograph confirms this. The long faint streaks, making an angle of about 15° with the vertical, are Neuman bands which are evidently independent of the fine structure. The short plates, making an angle of about 5° with the horizontal, are rhabdites as judged from their angular appearance at high magnification and by characteristic phosphide etching tests. These also have the same orientation throughout the lamella. The actual kamacite grain boundaries are sharper and more angular than the pseudoboundaries, this being shown especially well in lower left section of the micrograph. Recent studies of veining<sup>12</sup> in metals attribute it to separation of oxides from solid solution during slow cooling of the metal. Thus, these markings within the lamellae can best be explained as a form of ferrite veining which is much more pronounced than usual, as might be expected in this otherwise exceptionally coarse structure.

Numerous attempts have been made to produce a Widmanstätten pattern, comparable with those found in meteorites, in alloys melted in the laboratory.<sup>13</sup> These have not met with unqualified success and the pattern has appeared on a microscopic scale only. In the course of our experiments with alloys of higher nickel content a sample containing 27 per cent nickel was allowed to cool from 1400° C. to room temperature in about 12 hours. When polished and etched the exceptionally well developed structure shown in Fig. 8 was found, this is quite visible with the naked eye. Although containing more nickel than the average meteorite, there is little reason to doubt that this is a laboratory demonstration of the production of the Widmanstätten pattern which was first observed in meteorites.

# DISCUSSION.

The uncommon interest attached to meteorites has led to numerous scientific investigations of their structure. These have in turn led to a wide variety of theories concerning the origin and history of meteorites. Metallurgists have used many devices to explain the formation of the Widmanstätten patterns commonly observed. Most of these have involved a eutectoid decomposition in the iron-nickel system, <sup>14</sup> but X-ray studies have shown that this eutectoid does not exist. The

<sup>&</sup>lt;sup>12</sup> Northcott, L.: Veining and Sub-Boundary Structures in Metals, J. Inst. Met. 59, Ad. Copy, 1936.

See reference 6.
 Benedicks, C.: Chimie et Industrie, Vol. 16, Part 2, 406-410, 1926.

formation from the melt of kamacite plates as the  $\delta$ -iron phase, stabilized by the presence of phosphorous, so that they are retained on rapid cooling, has been suggested more recently. <sup>15</sup> The simple manner in which the orientations of all the kamacite

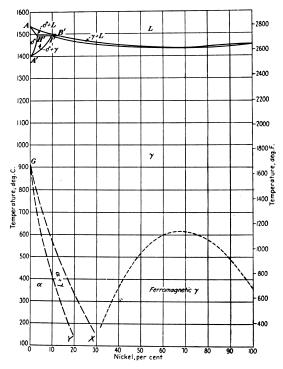


Fig. 9. The Iron-Nickel Constitution Diagram.

plates can be related to that of a single taenite orientation makes this seem extremely unlikely. The generally accepted iron-nickel constitution diagram, <sup>16</sup> Fig. 9, in conjunction with the established properties of this system, will explain these patterns quite directly. The exact locations of the high temperature points connected with the peritectic reaction  $\delta$  + melt  $\rightarrow$   $\gamma$  and the boundaries of the  $\alpha$  +  $\gamma$  field are uncertain.

<sup>16</sup> Marsh, J. S.: The alloys of Iron and Nickel, Alloys of Iron Research Series. Advance Copy, Chapter II. Not yet published.

<sup>&</sup>lt;sup>15</sup> Vogel, R.: Eine umfassenden Deutung der Gefüge-erscheinungen des Meteoreisens durch das Zustandsdiagramm der ternären Systems Fe-Ni-P. Abhandl. d. Gellsechaft d. Wissenschaften zu Göttingen-Math.-Phys. Klasse III Folge, Heft 6 1-31, 1932.

However, this is unimportant for the present purpose as the general features of the diagram are well established. sider the history of an alloy of meteoric composition, in the range between five and 15 per cent nickel. The complications introduced by the  $\delta$  phase need not be discussed. Below approximately 1400° C., the entire mass will be in the f.c.c. γ form and these alloys have a pronounced tendency to form large grains at these temperatures, thus accounting for the huge taenite crystals required for the formation of the Widmanstätten pattern. On further cooling, as the alloy reaches the line GX it will begin to transform to the b.c.c. a phase, forming the kamacite plates. During this process the remaining  $\gamma$ will become richer in nickel until it contains about 25 per cent. material containing more nickel than this does not transform readily at ordinary temperatures and will be the component known as taenite. Some of this taenite will have a composition in the two phase  $\alpha + \gamma$  field and will therefore transform slowly on further cooling, either partially or completely, depending upon its composition and rate of cooling, giving rise to the various forms of plessite. It is believed that all structures found in metallic meteorites can be explained on this general theory. Additional work on the phases present in the different varieties of plessite and the orientation relationships existing in these fields would be of great interest and value. Varying amounts of impurities and different rates of cooling undoubtedly affect these forms markedly.

Some investigators have been led to the conclusion that the metallic portions of meteorites were formed by reduction of metallic chlorides with hydrogen below about  $400^{\circ}$  C.<sup>17</sup> There are metallurgical reasons for questioning such a possibility. As just described, the most probable means of formation of large taenite grains and subsequently the coarse Widmanstätten patterns is by slow cooling of the metal from the molten condition, or at least from relatively high temperatures. Also, the research of Mehl and Derge<sup>18</sup> demonstrates that the orientation relationships and microstructure in meteorites are such that the  $\gamma \longrightarrow \alpha$  transformation must have occurred at relatively high temperatures. Likewise the veining found in kamacite plates can be interpreted best as precipitation of metallic oxides during slow cooling, and the same process accounts for the oriented rhabdites within the kamacite lamellae.

 <sup>&</sup>lt;sup>17</sup> Merrill, G. P.: Proc. U. S. Nat. Museum 73, 1-7, 1928.
 <sup>18</sup> See reference 2.

Since this report only includes results incidental to an investigation of another problem, it is not amiss to point out that a more complete study of the crystalline perfection and orientation relationships of a large number of different meteorites would undoubtedly yield a great deal of information pertaining to their past history. The use of X-rays has increased the certainty of our knowledge of transformations in alloy systems to such an extent that the highly speculative character of previous meteoric studies could be practically eliminated.

### SUMMARY.

- 1. The Amalia Farms meteorite has a highly perfect macrostructure but a very imperfect kamacite crystal lattice, while the Cañon Diablo meteorite has an imperfect macro-structure and a relatively perfect kamacite lattice.
- 2. The kamacite orientations of the Cañon Diablo meteorite can be related to the parent taenite orientation by the conditions of Kurdjumow and Sachs.
- 3. A very good Widmanstätten pattern was produced by slowly cooling an alloy of iron with 27 per cent nickel.
- 4. Metallurgical evidence indicates that the Widmanstätten patterns formed in meteorites are due to slow cooling from high temperatures.

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