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THE TAWALLAH VALLEY METEORITE.

General Description.

By T. HODGE-SMITH, The Australian Museum.

The Microstructure.

By A. B. EDWARDS, Ph.D., D.I.C.,* Research Officer, Mineragraphy Branch, Council for Scientific and Industrial Research.

(Plates i-ii and Figures 1-2.)

General Description.

Little information is available about the finding of this meteorite. Mr. Heathcock, Constable-in-Charge of the Borroloola Police Station, Northern Territory, informed me in April, 1939, that it had been in the Police Station for eighteen months or more. It was found by Mr. Condon, presumably some time in 1937.

The weight of the iron as received was 75.75 kg. (167 lb.). A small piece had been cut off, but its weight probably did not exceed 200 grammes. The main mass weighing 39.35 kg. (863 lb.) is in the collection of the Geological Survey, Department of the Interior, Canberra. A portion weighing 30.16 kg. ($66\frac{1}{2}$ lb.) and five pieces together weighing 1.67 kg. are in the collection of the Australian Museum, and a slice weighing 453 grammes is in the Museum of the Geology Department, the University of Melbourne.

The locality is Tawallah Valley, about forty-eight miles north-west of Borroloola, Northern Territory, Australia, latitude 15° 42' S., longitude 135° 40' E. approximately.

The shape of the iron is quite unusual in that it is more or less flat and there is an almost complete absence of thumb-marks so characteristic of meteoric iron. The iron contains only a few very small inclusions of troilite and no schreibersite has been found. This does support the view that thumb-marks are due to the decomposition of such minerals during the meteorite's flight through the atmosphere. The flat side forms roughly a parallelogram the longer sides of which are very straight and almost parallel. They are 65 cm. in length and approximately 38 cm. apart. The smaller sides of the parallelogram forming the head and tail of the meteorite are not so straight. The tail particularly is curved toward the centre.

The thickness gradually decreases from the head (65 mm.) to the tail (5 mm.). There is a general tapering from one side to the other, though this difference in thickness disappears toward the tail. One wing of the tail is bent at an angle of about 25° to the plane of the iron. It is possible that this was bent when the iron struck the ground, as there is a fairly large oval indentation on the edge just where the bending takes place, and there is no doubt that this indentation was formed ^{to}mechanically.

The reverse side is almost perfectly flat with a number of indentations that have been mechanically formed. The larger ones appear to be the result of the iron striking something hard and sharp on reaching the ground. Their formation and orientation confirm the fact that the thick end of the iron was actually the head during flight. The smaller indentations are not oriented and resemble chisel marks and may have been made by man.

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The obverse side is not so flat. There is a mound rising about 25 mm. above the level surface, situated about half-way between the head and the tail and close to the thicker side. There are also four depressions so arranged as to give the appearance of the foot-mark of some beast. There are chisel-like marks similar to those on the reverse side and, like them, probably the result of human activity.

The outer skin is black and very thin. In a number of places it is worn through, showing the bright iron underneath. There is no sign of rippling of the surface similar to that of the Bugaldi (New South Wales) iron. Around the head on the flat side only is a layer of iron less than 1 mm. in thickness, extending not more than 40 mm. from the periphery and ending in a very irregular edge. This may possibly represent the remnant of a wave of molten metal leaving the forward end.

On the corner of the head and on the thinner side are two large oval indentations mechanically formed, probably at the time of contact with the ground.

The first cutting was kindly undertaken by the Chief Mechanical Engineer's Branch of the New South Wales Railways Department. It was cut by a slitting saw in a milling machine. Later, when it was found that the iron was soft and comparatively free from inclusions, a complete section was cut by hand at the Museum. The Railway Department did the polishing. The iron takes a brilliant polish and does not rust easily.

Chemical Analysis.

\mathbf{Fe}	Ni	Co	\mathbf{Pt}	\mathbf{S}	\mathbf{P}	\mathbf{C}	Total	Sp. Gr.	n
82.29	16.90	1.09	Trace	abs.	abs.	0.03	100.31	8.00	4 ·8

The absence of sulphur would indicate the complete absence of troilite, but this can be true only for the portion analysed, as there are a few scattered inclusions of troilite in a complete section of the iron. The largest of these inclusions does not exceed 2 mm. in diameter. No schreibersite has been detected. The very small amount of carbon is present, in part at least, in a very finely divided state. Of the platinoid metals no iridium was detected and platinum was found to be present to the extent of 0.003 per cent.

The specific gravity is somewhat high. Three separate samples were tested by the ordinary chemical balance method, giving as result 8.002, 8.002, 8.001. Another piece tested by the Chemical Branch of the Department of Mines gave a result of 7.997. The average of these readings is 8.00.

This is the highest value that I have been able to find for any meteorite except for the Botetourt iron (O. Sjostrom, 1898), of which the specific gravity is recorded as 8.186.

The theoretical specific gravity for an iron-nickel of the composition of the Tawallah Valley, using the specific gravities given in the Smithsonian Physical Tables (1920), is for the minimum value 7.903 and for the maximum 8.001.

On etching a polished surface with dilute nitric acid a distinct etch pattern was produced showing in its orientation a resemblance to the usual Widmanstätten figures. The usual bands of kamacite and taenite were replaced by a definite sheen. A similar etch pattern has been recorded for many of the nickel-rich irons. No satisfactory explanation of this structure has been offered. Dr. Edwards very kindly offered to undertake a mineragraphic examination with a view to supplying this explanation. That he has been entirely successful will be realized from his notes published below.

According to the analysis, this iron belongs to the nickel-rich ataxites, Group 3 of Prior's Classification. Spencer records twenty-three nickel-rich ataxites. Since that time the Tschinga iron (Ni 16.71 per cent; n 5) has been recorded, so that this iron makes the twenty-fifth record and the first for Australia. The Arltunga (Central Australia), with a value for n of 8.6, is just on the border-line between the finest octahedrites and the nickel-rich ataxites. It is reported as having a micro-octahedral structure. The Yarroweyah (Victoria) is the only Australian nickel-poor ataxite.

In view of the apparently constant structure to be found in nickel-rich iron and the* fact, as shown by Dr. Edwards for the first time, that this structure is closely related to the octahedral structure, the name ataxite, meaning without order, is no longer applicable. The term ataxite should be confined to those irons in which the nickel content is too low to produce a definite structure of the nickel-iron alloys. It is suggested that the term eotaxite should be applied to those irons rich in nickel in which the early stage of the formation of the Widmanstätten structure is developed.

The Microstructure.

Examination of the polished sections of the Tawallah Valley iron in reflected light reveals that it consists almost wholly of nickel-iron with an occasional bleb of iron sulphide, from 1 to 2 mm. in diameter.

Iron Sulphides.

The blebs of iron sulphide vary somewhat in composition. Some consist of troilite, others of pyrrhotite. The distinction between them is that troilite, which contains more sulphur, effervesces with dilute nitric acid, and gives off hydrogen sulphide, which stains the iron, while the pyrrhotite is practically inert to dilute nitric acid. One bleb of pyrrhotite examined consisted of several allotriomorphic crystals, some of which showed lamellar twinning. The pyrrhotite is creamy-brown in colour, strongly anisotropic, and shows reflection pleochroism. The powder is magnetic. Of the standard etching reagents, potassium hydroxide slowly stains the pyrrhotite brown, while the other reagents are negative. When a portion of the meteorite was dissolved in dilute nitric acid, a complete, but somewhat etched, prismatic crystal of pyrrhotite, about 2 mm. \times 0.5 mm., was set free. The crystal was elongated in the prism direction, and showed the forms (1011), (1010), (0001). Measurements under a microscope gave (0001) \wedge (1011) = 45° ± 30'; (1010) \wedge (1011) = 45° ± 30'.

Nickel-Iron.

When polished sections are etched with a 2 per cent. solution of nitric acid in alcohol for a few seconds (or with bromine water, or 2 per cent. picric acid in alcohol), a microstructure such as is shown in Plate ii, Fig. 1, develops. This structure is composed of two components, and is more or less comparable with the martensite-like structure shown by artificial Fe-Ni alloys with 10 to 25 per cent. Ni that have been annealed from 1300° C. (Marsh, 1938, p. 47). The more readily etchable constituent forms lamellae and more or less diamond-shaped or lens-like bodies in the other constituent, which acts as host. These large bodies are oriented in definite directions, presumably related to the crystallographic directions of the host. Study of Figures 3 and 6 shows that there are three such directions. Two of them are well marked, while the third is only weakly developed, but can be detected in the diamond-like shape of many of the bodies.

In the regions between these large oriented bodies there is developed what appears to be a similar intergrowth, but on a much finer scale. At the margins of the large bodies, however, the host constituent has been drained clear, presumably by diffusion during the growth of the large bodies. The structure is typically that of an ex-solution, and since the materials consist solely of iron and nickel (plus cobalt), it must represent a stage in the transformation of γ -nickel-iron to α -nickel-iron. The ex-solution bodies are presumed to consist of α -nickel-iron, oriented parallel to the octahedral directions of the original γ -nickel-iron crystals.

This oriented microstructure gives rise to a "schiller" on the surface of polished and etched specimens, when the specimen is observed in oblique light (Plate ii, fig. 5). There is a narrow band around the edge of the meteorite (Plate ii, fig. 5), much thicker on one side than on the other, in which the microstructure is lacking. This band consists of γ -nickel-iron, and the absence of ex-solution bodies of α -nickel-iron from it must be attributed to its rapid chilling.

A third constituent of the nickel-iron is also revealed by etching with 2 per cent. nitric acid in alcohol, or other etching reagents. As shown in Plate ii, fig. 2, it consists of small irregular, often elongated or rounded, grains of a substance which is not attacked by the etching reagents, even with prolonged etching. These grains are almost invariably associated with the ex-solution bodies of α -nickel-iron. They were formed prior to the ex-solution, because the bodies of α -nickel-iron are commonly moulded on them, or even enclose them (Plate ii, fig. 4). Apparently the grains of this third constituent served as nuclei about which the ex-solution bodies grew. Careful examination of the unetched polished specimens in reflected light reveals that under high magnification these grains can be distinguished from the main nickel-iron intergrowth by the fact that they show a slight brownish tint. When such polished sections are left exposed, a tarnish film develops on the $(\alpha + \gamma)$ nickel-iron intergrowth, but the grains of the third constituent remain bright (Plate ii, fig. 2).

The chemical composition of the meteorite indicates that this mineral is also an alloy of nickel and iron; and in view of its resistance to acid etching reagents, it is presumed to be one which is rich in nickel. By way of confirmation a piece of the iron weighing about 20 grams was dissolved in 2 per cent. nitric acid, solution being complete at the end of four weeks. A crystal of pyrrhotite, and a small amount of black insoluble powder were left. The powder, when filtered and dried, weighed only 0.005 gram. This was too little for a quantitative analysis with the means available, so qualitative microchemical tests were carried out on it. Strong positive tests were obtained for iron and nickel, and the tests indicated that the iron was the more copious of the two. Tests for cobalt were doubtfully positive. Tests for phosphorus were negative. The mineral is regarded, therefore, as being an iron-nickel alloy in the γ -phase, and containing about 25–30 per cent. of nickel, i.e. *taenite*.

Standard etching reagents affect the iron as follows. Nitric acid (1:1) etches the α -iron, roughening its surface, and slowly attacks the Ni-poor γ -iron surrounding it. The Ni-rich γ -iron grains are not affected. The areas of fine $(\alpha + \gamma)$ intergrowth between the large α -lamellae are blackened instantaneously. This suggests that the blackened areas commonly found associated with taenite (Ni-rich γ -iron) on etching are finely microcrystalline intergrowths of α - and γ -iron, as claimed by Smith and Young (1939), and are not due to the presence of either phosphide or carbon in solid solution in the iron, as has been suggested by other writers (Johnston and Ellsworth, 1921; Vanick, 1925). Hydrochloric acid (1:1) is negative, but the fumes tarnish the surface lightly, and reveal the microstructure of the iron. The iron washes clean. Potassium cyanide and potassium hydroxide are negative; but ferric chloride and mercuric chloride both etch the iron instantaneously. The ferric chloride blackens the α -iron, but leaves all the γ -iron untouched. The orientation of the minute bodies of a-iron in the "groundmass" between the coarser a-iron ex-solution bodies is particularly well brought out by this etching (Fig. 6). Mercuric chloride blackens the surface immediately, and rubs to an etched surface. The α -iron is attacked, but the γ -iron is unaffected. The grains of Ni-rich γ -iron (taenite) stand out sharply in the etched section.

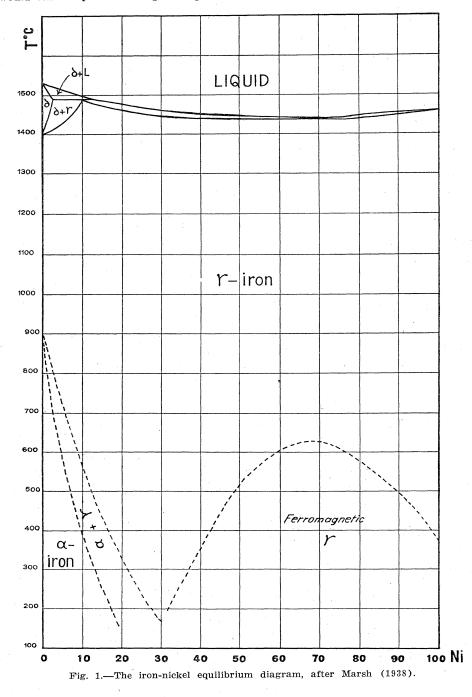
Origin of the Microstructure.

The microstructure of the Tawallah Valley iron is readily explained by reference to the generally accepted iron-nickel equilibrium (as shown by Marsh, 1938, p. 55; Hanson, 1936), which is reproduced diagrammatically in Fig. 1. The exact location of the high temperature points connected with the peritectic region and the boundaries of the ($\alpha + \gamma$) field are uncertain, but the general features of the diagram are firmly established.

Consider the cooling history of an iron-nickel alloy containing about 18 per cent. of nickel, which is the composition of the Tawallah Valley iron, if Co is regarded as Ni. Solidification of the melt would commence at about 1480° C., with the separation of γ -iron containing 11 to 12 per cent. of nickel, leaving the residual liquid enriched in nickel. If cooling was sufficiently slow to permit the establishment of equilibrium, solid diffusion would keep pace with crystallization, and upon complete solidification the iron would contain 18 per cent. Ni, while the final drop of liquid to crystallize would contain about 25 per cent. Ni. If, however, cooling was too rapid for equilibrium

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to be established, the composition of the γ -iron formed would range from 11 per cent. Ni up to 25 per cent. Ni, or even higher, and the Ni-rich crystals, being the last to form, would tend to be small and would form in the interstices and grain boundaries of the earlier-formed Ni-poor γ -iron. As pointed out by March (1938, p. 35), the Ni-atom is very similar to the Fe-atom, so that the probable rate of diffusion of Ni in Fe is very slow. As a result, these differences in Ni-content in different crystals of the alloy would tend to persist during subsequent cooling.



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In an iron-nickel alloy of the composition of the Tawallah Valley meteorite it might well happen that equilibrium would be maintained during the early stages of solidification in view of the more rapid rate of diffusion of Ni in the Ni-poor γ -iron, and fail during the later stages of solidification, when a high Ni concentration had developed in the isolated droplets of residual melt. In this way the mass of the γ -iron would have a relatively uniform composition, about 15 to 17 per cent. of Ni, and only the minute, last-formed, interstitial crystals would consist of Ni-rich γ -iron.

On the further cooling of such an alloy, the γ -iron enters the $(\alpha + \gamma)$ field, and begins to transform to α -iron. This transformation is accompanied by an enrichment in nickel of the remaining γ -phase. As will be seen from Fig. 1, addition of Ni to the γ -iron causes a decrease of the temperature, at which the γ to a transformation begins, to so low a degree that the resistance to transformation finally becomes very great, and transformation more or less ceases as the Ni-content approaches 25 per cent. Meteoric irons with no more than 10 per cent. Ni would readily convert from the γ to the a phase, and only a small quantity of γ -iron with as much as 25 per cent. Ni (taenite) will remain. Moreover, the α -iron so formed will contain not more than 6 per cent. Ni (i.e. will be kamacite). Where, however, the original γ -iron contains about 15 to 18 per cent. Ni, as in the case of the Tawallah Valley iron, it will enter the $(\alpha + \gamma)$ field at so low a temperature that only a partial transformation can take place, and the final product of cooling will be an intergrowth of ex-solution bodies of α -iron in a matrix of γ -iron. Moreover, such crystals of Ni-rich γ -iron as formed the final products of solidification will not undergo any transformation to the a-phase, but will remain as isolated blebs in the $(\alpha + \gamma)$ matrix.

Normally, the addition of Ni to the residual γ -phase iron during the γ to a transformation would yield a residue of γ -iron almost as rich in Ni as these blebs; but in a case such as we are considering, the inability of the transformation to proceed beyond a preliminary stage, owing to the arrest of the process by further cooling, will prevent more than a small amount of Ni being transferred to the residual γ -iron, so that it will fail to approach a composition comparable with the Ni-rich γ -iron blebs. As a result, there will occur γ -iron of two compositions, one considerably richer in Ni than the other; and the difference in Ni-content may be sufficient to produce a slight difference in colour and in etching properties, such as is found in the constituents of the Tawallah Valley iron.

Origin of Widmanstätten Structure.

The ex-solution structure found in the Tawallah Valley iron throws additional light on the origin of Widmanstätten structure. The early formed ex-solution bodies of α-iron segregate in the octahedral crystallographic directions of the γ -iron crystals; and as they grow they develop into laminae elongated in these directions. Thus there developed three sets of parallel lamellae, inclined to one another at 60°. The actual angles seen in sections will, of course, vary with the direction in which the section is cut. As more and more molecules of a-iron are drained from the transforming γ -iron, the residual γ -iron, which is being enriched in Ni, becomes isolated between the widening lamellae of α -iron. Finally, when the Ni-content of this residual γ -iron has been raised to about 25 per cent. Ni, further transformation to α -iron becomes impossible, and the mass of iron becomes stable as an intergrowth of lamellae of α -iron (kamacite) of widths varying according to the Ni-content of the original melt, separated by bands or lamellae of Ni-rich γ -iron. Each α -iron lamella is oriented in one or other of the three octahedral crystallographic directions of the original γ -iron crystals, and the residual taenite lamellae have, perforce, a similar orientation. This combination of oriented, interleaved, lamellae of Ni-poor α -iron (kamacite) and Ni-rich γ -iron (taenite) gives rise on etching to the well-known Widmanstätten structure.

When, as in the Tawallah Valley iron, this process of ex-solution is arrested at an early stage, etching of a polished section gives rise to an octahedral "schiller", showing the same orientation as Widmanstätten structure, and being, in fact, an incipient development of that structure. The Tawallah Valley iron presents, therefore, a confirmation of the explanation of Widmanstätten structure advanced by Derge and Kommell (1937).

The Microstructure of Ni-rich Ataxites.

If the above explanation of the microstructure of the Tawallah Valley iron is correct, then it follows that all iron meteorites with a Ni-content between 15 and 20 per cent. Ni should possess similar microstructures; and from what information is available this would seem to be likely, although few such meteorites have been subjected to mineragraphic investigation.

Thus the Hoba meteorite (Spencer, 1932), which contains about 17 per cent. Ni (plus cobalt), consists of an extremely fine $(\alpha + \gamma)$ intergrowth (described by Spencer as "plessite"), similar to the finer intergrowth structure in the areas between the coarse ex-solution bodies of a-iron in the Tawallah Valley iron. The constituents of this intergrowth show a well-marked orientation, which on etching gives rise to a "sheen" on the etched surface, when it is viewed in oblique lighting. Scattered through this intergrowth are numerous grains of a white mineral not etched by dilute nitric acid. Spencer has suggested that these are grains of (?) schreibersite, or possibly cohenite. They are not likely to be cohenite, because there is only 0.02 per cent. of carbon in the meteorite, and so small an amount would be retained in solid solution in the γ -iron of the intergrowth. Schreibersite, on the other hand, is known to be harder than nickel-iron (Johnston and Ellsworth, 1921), whereas the mineral in the Hoba iron is reported to be softer than the nickel-iron. This, as will be seen from the discussion of the Brinell hardness tests on the Tawallah Valley iron, which follows, agrees with the hardness of taenite; and the probability is that these small grains, which so resemble the taenite grains in the Tawallah Valley iron, will prove to be taenite also.

The so-called "sheen" or "schiller" that is observed on etched surfaces of the Hoba and Tawallah Valley irons, when lit by oblique light, is a macroscopic effect of this particular microstructure; and the fact that other Ni-rich ataxites show such an effect is evidence that they possess a comparable microstructure.

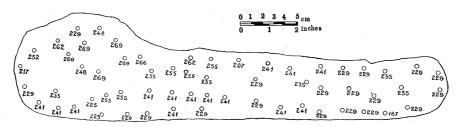


Fig. 2.-Brinell Hardness Survey on section of the Tawallah Valley iron.

Brinell Hardness.

A study of the Brinell hardness (Fig. 2) of the iron was made by Mr. R. A. Holloway, Testing Engineer, Department of the New South Wales Railways, who supplied the following note:

"The hardness survey carried out over a cross-section of the meteorite reveals a remarkable uniformity. Apart from the two low readings of 187 and 207, which are probably due to sponginess at these isolated points, the narrower end of the section shows a hardness range of 229 to 255. If the usual conversion factor of 0.22 is used, this represents a tensile strength range of from 50.4 to 56.1 tons per square inch.

"The thicker end shows a tendency towards higher hardnesses (up to 269) which may be due to some segregation in the centre of the section where it might reasonably be expected to occur.

"The homogeneous nature of the meteorite is also revealed by the shape of the Brinell impressions, which are quite circular and show no sign of irregularity such as is found in heterogeneous bodies." Polished sections were prepared of pieces showing hardnesses of 229, 235 and 241, but they showed no apparent differences in structure or composition when examined under the microscope in reflected light.

In view of the fact that the hardness of annealed iron-nickel alloys ranging in composition between 6 and 25 per cent. Ni shows a marked change with a small change of Ni-content (ranging from 150 at 6 per cent. Ni, to 225 at 9 per cent. Ni, with a maximum of 290 at 17 per cent. Ni, and then falling to 250 at 24 per cent. Ni, and 150 at 26 per cent. Ni) (Marsh, 1938), it seems probable that these slight variations in hardness in the Tawallah Valley iron are due to slight local variations in the Ni-content of the iron, arising from a failure to establish complete equilibrium during solidification. While the hardness behaviour of annealed alloys is not necessarily comparable with those of a meteoric iron, it may be recalled that the annealed artificial alloys of this range of composition develop martensite-like microstructures more or less similar to those shown by the Tawallah Valley iron.

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EXPLANATION OF PLATES.

PLATE I.

Fig. 1.—View looking at the thinner side of the Tawallah Valley iron, showing one tip of the tail bent, and an oval indentation mechanically formed on the edge close to the head. Photograph by G. C. Clutton.

Fig. 2.—The obverse side of the Tawallah Valley iron. Photograph by G. C. Clutton.

Fig. 3.—View looking at the head of the Tawallah Valley iron. Photograph by G. C. Clutton.

PLATE II.

- Fig. 1.—Typical ex-solution structure of the Tawallah Valley iron, showing coarse ex-solution bodies of α -iron growing in the crystallographic directions of the γ -iron. In the areas between these bodies is a host of minute ex-solution bodies of α -iron. The α -iron adjacent to the large bodies has been drained free of these minute bodies. Etched with 2 per cent. nitric acid in alcohol. \times 150. Photograph by A.B.E.
- Fig. 2.—The same as Figure 3, but magnified, showing a large ex-solution body of α -iron, enclosing a grain of nickel-rich iron (taenite), and surrounded by γ -iron of lower nickel content. \times 600. Photograph by A.B.E.
- Fig. 3.—Grains of nickel-rich γ -iron (taenite) set in the $(a + \gamma)$ matrix. Tarnished by exposure, leaving the taenite grains white. $\times 500$. Photograph by A.B.E.
- Fig. 4.—Section of the Tawallah Valley iron etched with FeCl_3 solution showing the orientation of the finer-grained ex-solution bodies of α -iron parallel with the coarser α -iron bodies. \times 150. Photograph by A.B.E.
- Fig. 5.—A plate of the Tawallah Valley iron, which has been polished and etched, so that it shows a "schiller" pattern when photographed in oblique light. Natural size. Photograph by J. S. Mann.



