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ELECTRON MICROPROBE ANALYSIS OF SOME RARE MINERALS IN THE NORTON COUNTY ACHONDRITE

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Electron microprobe analysis of some rare minerals in the Norton County achondrite

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Abstract—The composition of the minerals constituting the Norton County achondrite has been ascertained by measuring a large number of individual grains of each mineral, using electron microprobe techniques. The following minerals were found: enstatite, forsterite, metallic nickel-iron, metallic copper, daubreelite, troilite, and two new species, titanoan troilite and ferromagnesian alabandite. The unusual association of minerals indicates that at the time when this meteorite was formed the environment was strongly reducing, and any hypothesis on the origin of meteorites based on comparison with terrestrial rock-forming mechanism must take this into account.

INTRODUCTION

THE Norton County achondrite shower fell shortly before 4.56 p.m., 18 February 1948, near the boundary between Norton County, Kansas, and Furnas County, Nebraska, after a brilliant detonating fireball was seen. More than 100 stones fell, among them the largest known stone meteorite and largest observed fall, weighing more than a ton. In 1951 BECK and LA PAZ published a description of this meteorite, a microscopical investigation of its constituents, and a bulk chemical analysis of it (another bulk chemical analysis was performed by WIIK in 1956). BECK and LA PAZ described the meteorite as consisting of essentially enstatite crystals in a ground mass of enstatite and olivine, with small amounts of nickel-iron inclusions. Table 1 lists the minerals present in this meteorite according to their study.

Table 1. Minerals present in the Norton County achondrite as identified microscopically by BECK and LA PAZ (1951)

Mineral	Remarks
Metallic nickel-iron (kamacite)	Inclusions up to 50 × 35 × 20 mm
Schreibersite	Together with the nickel-iron inclusions
Troilite	Small amounts together with the nickel-iron inclusions, none found in the polished sections
Graphite	Small flakes
Enstatite	Two types: 1. Light gray with cleavage 2. Clear, glassy, without cleavage
Clinoenstatite	Present as narrow bands in some of the cleavage enstatite crystals
Diallage	Small amounts
Olivine	

The chemical analysis of the meteorite's metallic portion, as given by those authors, shows a certain peculiarity; namely, an unusually high copper content of

1·20 weight per cent. Such an extremely high value might well be due to the presence of either metallic copper or minerals with high copper content. This consideration led in part to the present study. Although metallic copper has been mentioned several times as existing in meteorites (see Table 2), these studies generally have been based on macroscopical or microscopical identification without much analytical proof.

Table 2. Metallic copper in meteorites, according to the authors listed. Classification of the meteorites as in PRIOR-HEY (1953) and in KEIL (1960)

Author	Year	Name of the meteorite	Classification of the meteorite
T. T. QUIRKE	1919	Richardton	Veined spherical bronzite-chondrite
J. D. BUDDHUE	1937	Toluca	Medium octahedrite
J. A. DUNN	1939	Rangala	Veined white hypersthene-chondrite
P. K. GHOSH	1940	Bherai	Veined white hypersthene-chondrite
H. H. NININGER	1941	Garnett	Chondrite
		Toluca	Medium octahedrite
I. A. YUDIN	1952	Vengerovo	Gray crystalline enstatite-olivine-chondrite
	1958	Saratov	Gray spherical hypersthene-chondrite
		Kainsaz	Black enstatite-olivine-chondrite
	1960	Nikolskoje	Bronzite-olivine-chondrite
P. RAMDOHR and G. KULLERUD	1961 and 1962	Several, but not specified*	
B. MASON	1962b	Miller (Arkansas)	Gray enstatite-chondrite
		Ochansk	Polymict brecciated spherical bronzite-chondrite
		Morito	Medium (or coarsest) octahedrite

* Note added in proof: Among the 140 chondrites investigated, more than 90 were found to contain small amounts of metallic copper (P. RAMDOHR, personal communication).

When microscopic examination of a number of polished sections of the Norton County meteorite was begun, some red grains, presumably copper, with a diameter up to almost 1 mm, were found. These grains, as well as those of a number of other rare minerals (including two new ones) found in close contact with them, were analysed quantitatively by use of electron microprobe techniques. The results of these analyses, together with those of certain minerals already described by BECK and LA PAZ, (1951) are given below.

METHOD

The chemical composition of selected mineral grains was measured with a modified ARL electron microprobe X-ray analyser.† For these measurements the polished sections, 1·5 to 2·5 cm in diameter, were coated with a layer of carbon a few hundred angstoms thick. At least three measurements, 3–5 μ/mu apart, were usually made on each grain. The homogeneity of the troilite, the metallic copper,

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† ARL refers to Applied Research Laboratories, Glendale, California.

the daubreelite, the ferromagnesian alabandite, and the titanoan troilite was ascertained by moving the sample in steps of 4 μ under the fixed beam, thus covering the grain with rows of analyses. Table 3 shows the averages of all the measurements carried out for each of the minerals. Generally, the chemical composition of each mineral was constant, usually varying less than ±0·4 weight per cent for a specific element (exception is troilite with varying titanium content from grain to grain.)

Table 3. Results of the electron microprobe analysis of the minerals found in the Norton County achondrite in weight per cent

No.	Mineral	Fe	Ni	Mg	Ca	Mn	Cu	S	Ti	Cr	Total	Number of grains analysed
1	Metallic nickel-iron (kamacite)	96·5	3·6								100·1	3
2	Troilite	55·9–63·1						36·6	4·1–0·4			51
3	Titanoan troilite	56·0					0·8	38·0	6·4		101·2	1
4	Enstatite, light gray, with cleavage	$<0·1$		23·8	0·45							30
5	Enstatite, clear, glassy, without cleavage	$<0·1$		23·9	0·32							30
6	Olivine	$<0·1$		34·3	~0·05							20
7	Metallic copper	4·2					96·5				100·7	8
8	Ferromagnesian alabandite	15·2		10·0		32·4		41·4			99·0	4
9	Troilite exsolution lamellae in No. 8	56·8	2·8				4·0	38·4			102·0	10
10	Daubreelite	17·8				1·0		44·5		35·5	98·8	12

For the quantitative analyses of nickel-iron and of copper, pure iron, nickel, and copper were used as standards. Titanium in troilite and titanoan troilite, and chromium in daubreelite were measured against metallic titanium and chromium. The content of iron and sulfur in the sulfides was ascertained by using a chemically analysed pyrite (Fe = 46·21; S = 53·60 weight per cent) as a standard. In every case the proper corrections for background and mass absorption were made. The iron and magnesium content of pyroxene and olivine was determined against chemically analysed pyroxenes and olivines. The chemical analyses and the degree of homogeneity of these standards were controlled with the microprobe by analysing them against pure metallic iron and magnesium standards. After corrections were made for background, dead time, mass absorption, fluorescence (according to the suggestions made by WITTRY, 1962), and atomic number, agreement with the chemical analyses was found to be excellent; namely, within ±0·3 per cent for Fe. With use of these values, calibration curves were drawn for both pyroxenes and olivines; and further analyses were evaluated graphically (FREDRIKSSON and KEIL, 1963; detailed description, KEIL and FREDRIKSSON, in preparation). The relative standard error of the results for both pyroxenes and olivines was found to be ±0·1 per cent for Fe and ±0·2 per cent for Mg; the standard error for the elements measured in the other minerals listed in Table 2 is usually the same order and does not exceed ±0·4 per cent.

An important application of the electron microprobe is the use of a scanning beam. This technique enables the electron beam to be moved over a microscopically selected area of the sample. The extent of the area covered by this beam can be varied in four steps. For the Figs. 5 through 8, with the accelerating potential of 25 kV that corresponded to an area of 430 × 430μ to 55 × 55μ, this amounts to enlargement of the image on the oscilloscope screen 175–1,400 times. The resulting X-ray emission from a single element in the surface of the sample can be selected by using a crystal spectrometer and is then detected with the aid of a proportional counter. The signal from the counter is used to modulate the brightness of a beam scanning a cathode ray tube in synchronism with the probe scanning the sample. The image produced on the screen corresponds with the area scanned on the specimen. From this image, photographic pictures are taken by use of a polaroid camera. The photographs obtained of the X-ray image give semi-quantitatively the distribution of the chosen element in the scanned area. Using the backscattered electrons to produce the image reveals topographical details of the surface of the scanned area, and in addition it shows differences in the average atomic numbers of the minerals involved (as the brightness of the image increases with increasing atomic number; compare Figs. 5 through 8).

RESULTS

The Norton County meteorite is rather inhomogeneous with regard to the rare minerals listed in Table 3. Only one of the sections examined contained nickel-iron, daubreelite, native copper, and ferromagnesian alabandite. These minerals occur in close contact, but the troilite is distributed uniformly throughout the sections studied.

1. *Rhombic pyroxene*. The pyroxene is the predominant mineral in this meteorite. BECK and LA PAZ, (1951) differentiated two types: (1) light gray, with cleavage; (2) clear, glassy, without cleavage. However, a large number of analyses of single grains in the present study showed their chemical identity. The iron content of both types is less than 0·1 weight per cent (Table 3); therefore, the pyroxene is enstatite ($MgSiO_3$), (possibly with some clinoenstatite of the same composition).

2. *Olivine*. This mineral is fairly abundant in occasional large crystals. According to the analyses, it is pure forsterite (Mg_2SiO_4) with less than 0·1 weight per cent iron (see Table 3).

3. *Metallic nickel-iron*. As was stated by BECK and LA PAZ, (1951) this meteorite contains nickel-iron in inclusions as large as 50 × 35 × 20 mm. They are scattered irregularly throughout the meteorite. In the polished sections used in the present study, small nickel-iron grains ($<$0·1 mm) were found closely related to the metallic

Fig. 1. Reflected light, magnification 155 times. Association of metallic copper (center, high reflecting), metallic nickel-iron (left, high reflecting), and titanoan troilite (upper right-hand corner, gray). Dark: silicates; deep black: holes in the section. The titanoan troilite is surrounded by copper (high reflecting, but mostly broken out during the process of polishing).

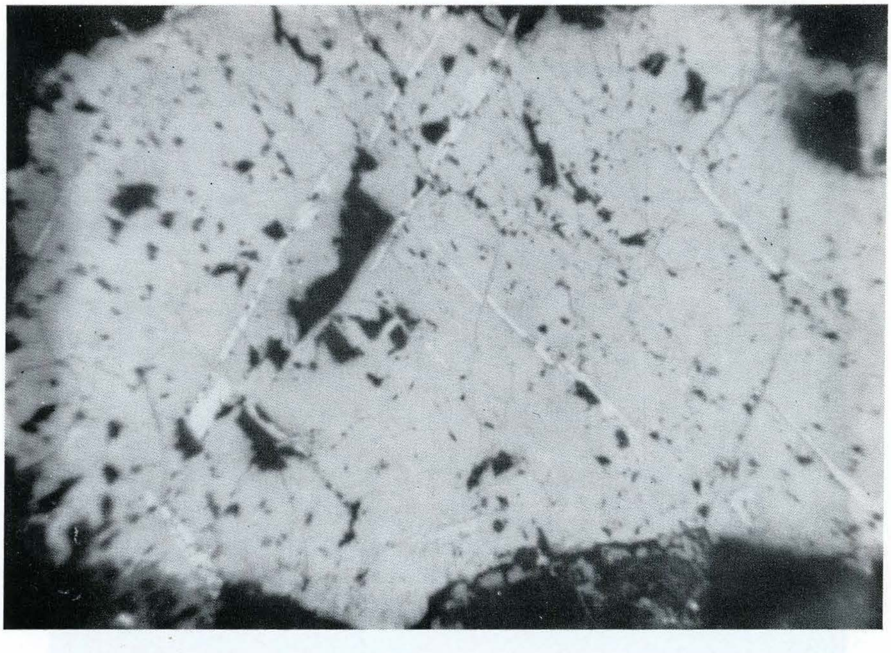


Fig. 2. Reflected light, magnification 240 times. Large grain of ferromagnesian alabandite (gray main mass), with orientated exsolution lamellae of troilite (white). Dark: silicates; deep black: holes in the section. Notice the offsets in the troilite lamellae in places where they were cut by the cleavage.

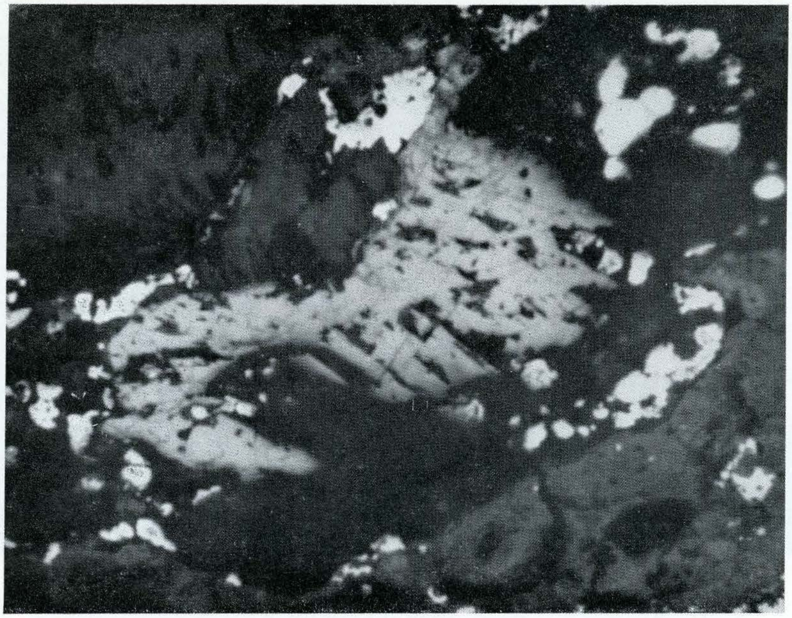


Fig. 3. Reflected light, magnification 320 times. Titanoan troilite (center, gray), surrounded by metallic copper (white, high reflecting, but mostly broken out during the process of polishing, therefore appears deep black). Dark: silicates; deep black: holes in the section.

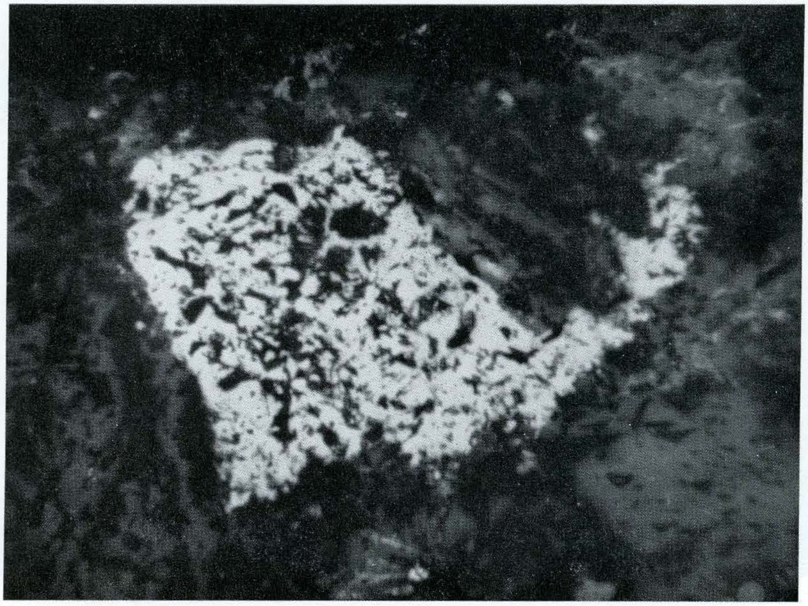


Fig. 4. Reflected light, magnification 240 times. Daubreelite (center). Dark: silicates; deep black: holes in the section.

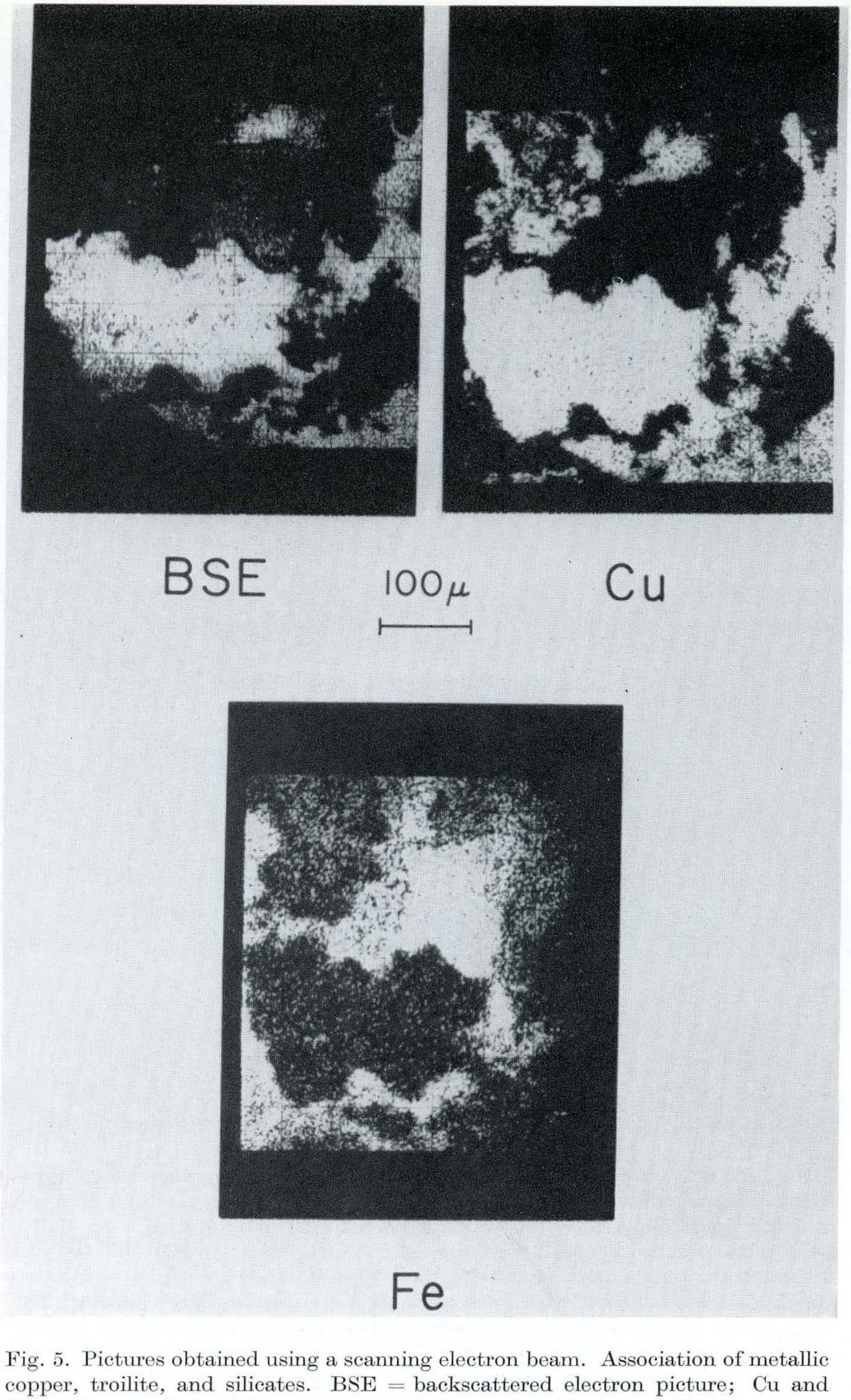


Fig. 5. Pictures obtained using a scanning electron beam. Association of metallic copper, troilite, and silicates. BSE = backscattered electron picture; Cu and Fe = pictures of the $\mathrm{Cu}_{K\alpha}$ and $\mathrm{Fe}_{K\alpha}$ radiation respectively. In BSE the mineral with the highest average atomic number (Cu) gives the brightest image; less bright is FeS; deep black are the silicates or holes in the minerals. The $\mathrm{Cu}_{K\alpha}$ picture shows the metallic copper grains; in some places there is a $\mathrm{Cu}_{K\alpha}$, but no BSE image (upper left-hand corner). This is due to holes in the copper, which give an image in $\mathrm{Cu}_{K\alpha}$ radiation, but none with backscattered electrons. The $\mathrm{Fe}_{K\alpha}$ shows the high Fe in the FeS, and the uniform Fe distribution in the metallic copper.

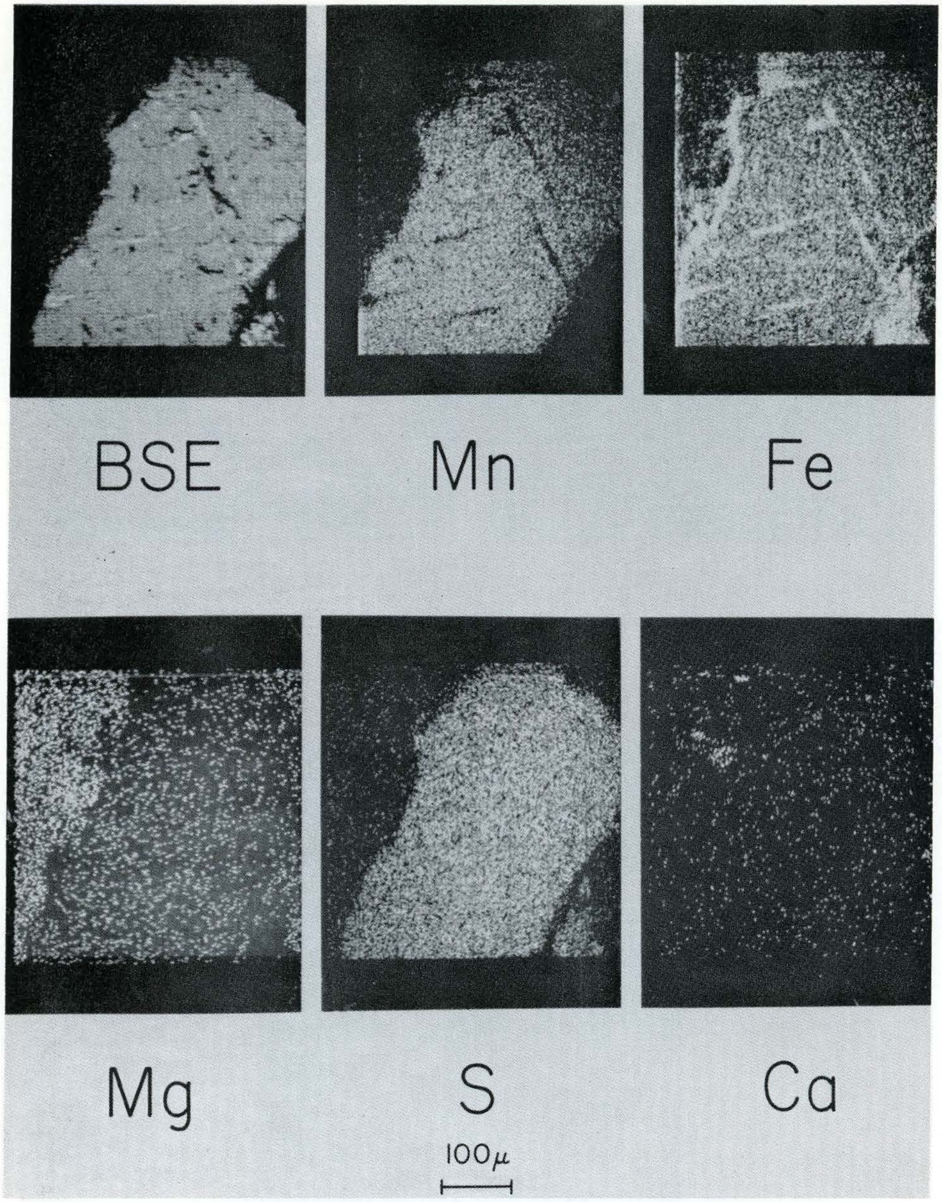


Fig. 6. Pictures obtained using a scanning electron beam. Association of ferromagnesian alabandite with FeS exsolution lamellae and surrounded by a thin rim of FeS, in silicate matrix. BSE = backscattered electron image; Mn, Fe, Mg, S, Ca = pictures taken in the K_α radiation of these elements. In the BSE the FeS with highest average atomic number gives brightest image (orientated exsolution lamellae); main mass is ferromagnesian alabandite; deep black are silicates. The $Mn_{K\alpha}$ image shows no Mn in the exsolution lamellae and in the silicate matrix, but uniform content in the large host grain. The $Fe_{K\alpha}$ image shows high amounts of Fe in the ferromagnesian alabandite, but even higher amounts in the exsolution lamellae and in the thin FeS rim around the ferromagnesian alabandite. The $Mg_{K\alpha}$ image shows homogeneous Mg distribution in the ferromagnesian alabandite, but much higher amounts in the silicate matrix (pure $MgSiO_3$). The sulfur, as given in the $S_{K\alpha}$ image is present in equal amounts in both ferromagnesian alabandite and the FeS exsolution lamellae. There is no Ca present in FeS and ferromagnesian alabandite.

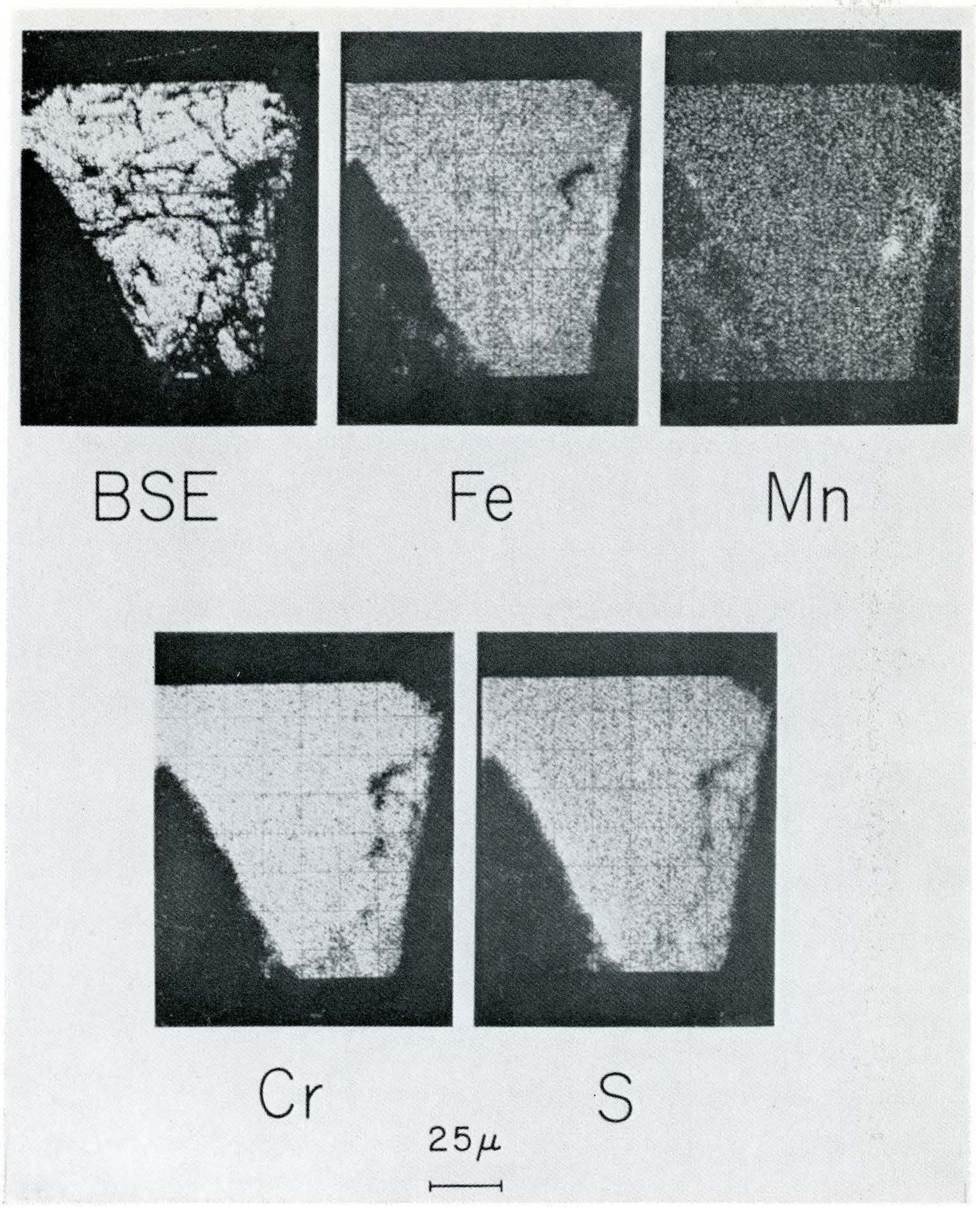


Fig. 7. Pictures obtained using a scanning electron beam. Association of daubreelite in silicate matrix. BSE = backscattered electron image; Fe, Mn, Cr, S = pictures taken in the K_α radiation of those elements. In the BSE picture the bright image is due to the daubreelite (highest average atomic number); deep black are silicates. The K_α pictures of Fe, Mn, Cr, and S show the presence of those elements in the daubreelite in a uniform distribution.

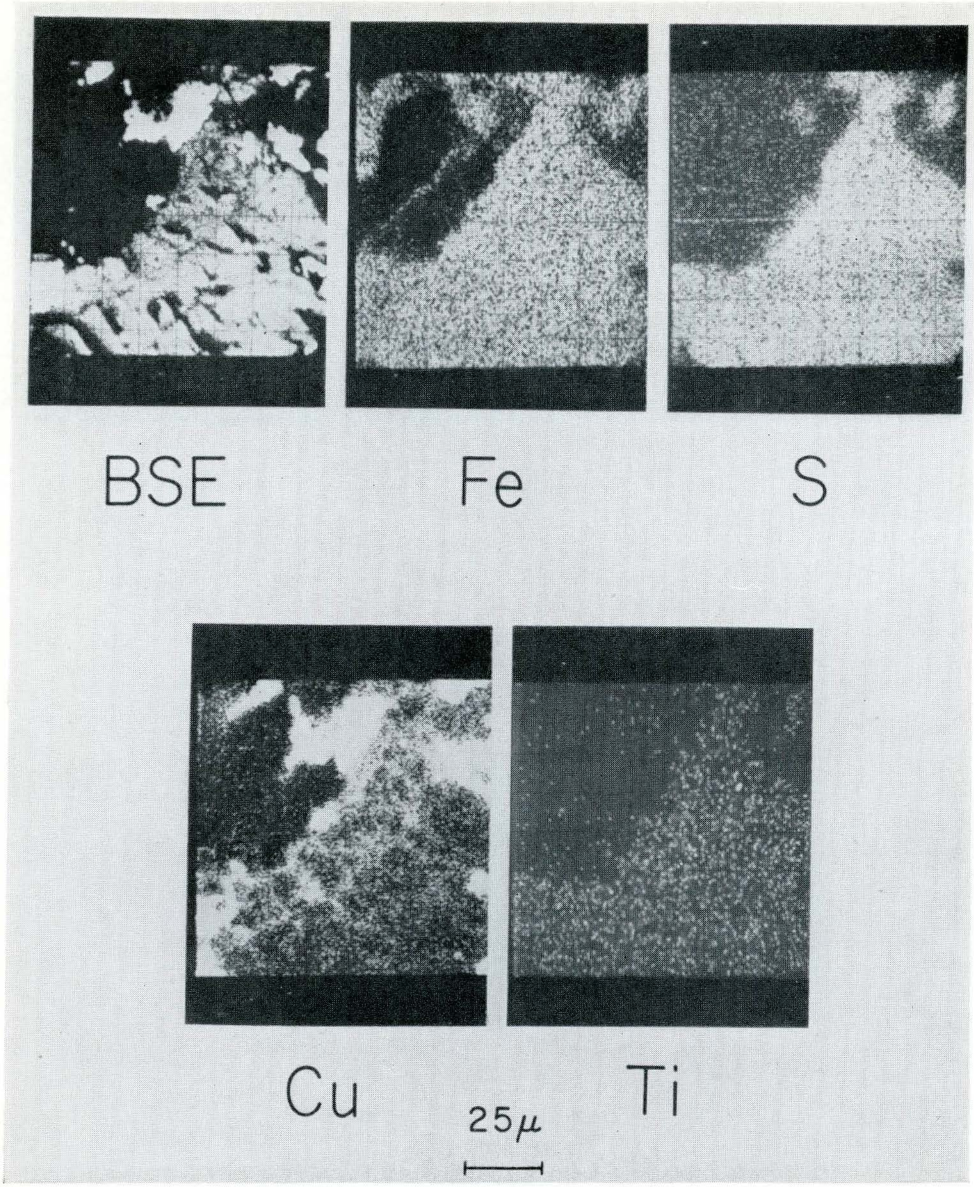


Fig. 8. Pictures obtained using a scanning electron beam. Association of titanoan troilite, metallic copper, silicate matrix. BSE = backscattered electron image. Fe, S, Cu, Ti = pictures taken in the K_α radiation of these elements. In the BSE picture the brightest image is due to metallic copper (highest average atomic number), followed by the less bright image of the titanoan troilite; deep black are silicates. The K_α pictures of Fe, S, and Ti show the uniform distribution of these elements in the titanoan troilite. The CuK_α image shows several grains of metallic copper as well as a small amount of Cu irregularly distributed in the titanoan troilite.

copper and the titanoan troilite (see Fig. 1). The average nickel value was found (in 9 analyses) to be 3·6 weight per cent (see Table 2). This indicates that the nickel-iron is kamacite. No taenite was discovered in these sections.

4. *Metallic copper*. Native copper has been described several times in other stone meteorites as well as in iron meteorites (see Table 2), but thus far without chemical evidence. Metallic copper was found in one of the sections of the Norton County achondrite (see Figs. 1, 3). Several grains of this mineral were found, the largest having a diameter of almost 1 mm. The chemical analyses of several grains reveal a considerable amount of iron in the copper. The average of 11 analyses showed 4·2 weight per cent (see Table 3). Fig. 5 illustrates this semi-quantitatively in the scanning pictures for the back-scattered electrons (BSE), the $\mathrm{Cu}_{K\alpha}$ and the $\mathrm{Fe}_{K\alpha}$ radiation of an area consisting mainly of native copper with some troilite and silicate grains. Traces of silver in the order of 0·1 weight per cent seem to be present in the copper.

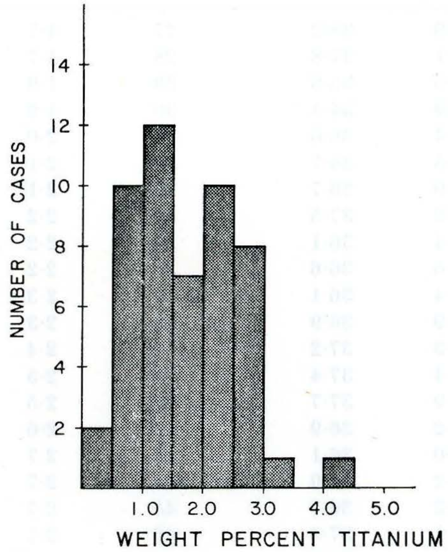


Fig. 9

5. *Ferromagnesian alabandite*. This new species was found in one large grain and several small ones in close association with the metallic copper, titanoan troilite, and nickel-iron. (See Fig. 2.)

Manganese sulfide (alabandite) from a meteorite was first reported by Dawson *et al.*, (1960) as occurring in a single chondrule of the Abee stone meteorite. The mineral was identified in an X-ray diffraction pattern; no chemical analysis had been made. Yet the slight difference in the cell edge, in comparison to standard MnS may well be due to the presence of some iron. Magnesium sulfide (MgS) was described by Du Fresne and Anders, (1961) to exist in the Pesyanoe achondrite. Manganese sulfide with a considerable iron content (no analysis being given) was described as a new mineral, "Eisenalabandin", from the metallic iron-bearing basalt from Bühl, near Kassel, Germany (Ramdohr, 1952), and later found also in a pyrrhotite inclusion of the phonolite from Fohberg, near Oberschaffhausen, Germany (Ramdhor, 1957). The "alabandite" discovered in some (not specified) meteorites was mentioned as being an iron manganese sulfide (Ramdohr and Kullerud, 1961, 1962.) In recognition of Ramdohr's "Eisenalabandin", the new mineral found in the Norton County achondrite may appropriately be named ferromagnesian alabandite. Fig. 2 shows the largest grain found in this meteorite; Fig. 6 illustrates semi-quantitatively its distribution of the elements present; Table 3 shows its chemical composition as ascertained by electron microprobe techniques. Manganese is the predominant cation in this mineral (32·4%), followed by iron (15·2%) and magnesium (10·0%). No Ca (<0·1%) was found. Characteristic are systems of exsolution lamallae of FeS (troilite), which contain small amounts of nickel and copper.

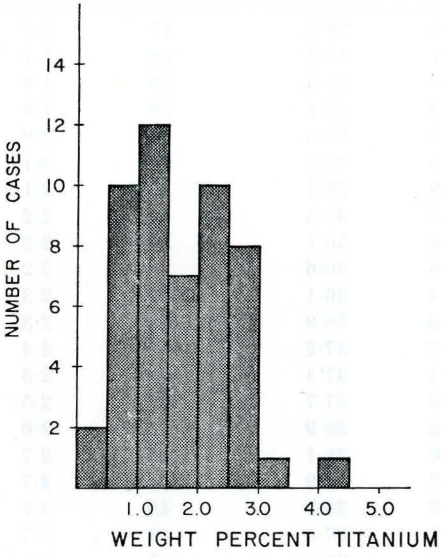
Table 4. Composition of 51 individual troilite grains, in weight per cent

No.	Ti	Fe	S	No.	Ti	Fe	S
1	0·4	55·9	38·2	27	1·7	59·1	35·4
2	0·4	63·1	37·8	28	1·7	60·0	35·2
3	0·7	58·5	35·8	29	1·9	59·9	36·3
4	0·8	58·9	34·4	30	1·9	58·6	34·9
5	0·8	62·4	36·6	31	2·0	57·7	35·3
6	0·8	61·5	36·7	32	2·1	57·5	36·1
7	0·8	61·9	36·7	33	2·1	60·0	36·9
8	0·9	62·2	37·5	34	2·2	61·1	38·3
9	0·9	59·4	36·1	35	2·2	61·0	37·6
10	0·9	60·6	36·6	36	2·2	60·7	37·1
11	1·0	60·4	36·1	37	2·3	57·1	35·8
12	1·0	61·9	36·9	38	2·3	57·5	37·3
13	1·1	61·3	37·2	39	2·4	57·8	36·3
14	1·1	62·4	37·4	40	2·5	58·0	36·1
15	1·1	62·9	37·7	41	2·5	59·4	38·3
16	1·1	61·2	36·9	42	2·6	59·4	36·1
17	1·2	60·0	36·1	43	2·7	59·4	36·9
18	1·2	61·2	37·9	44	2·7	56·0	34·4
19	1·2	61·3	36·7	45	2·7	56·0	35·8
20	1·2	62·1	37·5	46	2·7	60·4	37·9
21	1·4	59·4	35·9	47	2·8	59·0	37·1
22	1·4	60·4	36·6	48	2·9	57·8	36·0
23	1·5	60·3	36·2	49	2·9	58·5	36·7
24	1·5	59·7	36·9	50	3·2	58·7	37·5
25	1·6	60·3	36·5	51	4·1	56·1	36·1
26	1·6	59·3	36·0	Average	1·7	59·7	36·6

6. *Troilite and titanoan troilite.* Contrary to the observations by Beck and La Paz (1951), many small flakes and grains of troilite were found uniformly distributed throughout the sections examined. The composition of each of 51 grains in three polished sections was measured, and a surprising amount of titanium was observed in the troilite. Table 4 lists the results of the analyses in the order of increasing titanium. Fig. 9 gives the frequency distribution of the titanium values for these

copper and the titanoan troilite (see Fig. 1). The average nickel value was found (in 9 analyses) to be 3·6 weight per cent (see Table 2). This indicates that the nickel-iron is kamacite. No taenite was discovered in these sections.

4. *Metallic copper*. Native copper has been described several times in other stone meteorites as well as in iron meteorites (see Table 2), but thus far without chemical evidence. Metallic copper was found in one of the sections of the Norton County achondrite (see Figs. 1, 3). Several grains of this mineral were found, the largest having a diameter of almost 1 mm. The chemical analyses of several grains reveal a considerable amount of iron in the copper. The average of 11 analyses showed 4·2 weight per cent (see Table 3). Fig. 5 illustrates this semi-quantitatively in the scanning pictures for the back-scattered electrons (BSE), the $Cu_{K\alpha}$ and the $Fe_{K\alpha}$ radiation of an area consisting mainly of native copper with some troilite and silicate grains. Traces of silver in the order of 0·1 weight per cent seem to be present in the copper.



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5. *Ferromagnesian alabandite*. This new species was found in one large grain and several small ones in close association with the metallic copper, titanoan troilite, and nickel-iron. (See Fig. 2.)

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In close relation to the copper, nickel-iron, and ferromagnesian alabandite, there was found an iron sulfide having a considerably higher titanium content (6·4 weight per cent). Fig. 3 shows the grain surrounded by copper. The scanning-beam pictures in Fig. 8 illustrate semi-quantitatively the distribution of elements in that grain. Since this appears to be the first case in which titanium was found to be chalcophile and enriched in the troilite, this mineral can appropriately be named titanoan troilite.

7. *Daubreelite.* In addition to the other sulfide minerals, considerable daubreelite ($FeCr_2S_4$) was found in this meteorite. Table 3 shows its chemical composition. The mineral contains a small amount of manganese. Fig. 4 shows a daubreelite grain in a photomicrograph. Fig. 7 illustrates semi-quantitatively in several scanning-beam pictures, the distribution of the elements which it contains.

DISCUSSION

The rather uncommon composition of the major minerals, also the presence and composition of certain minor minerals, leads to the conclusion that this meteorite was formed in a specific environment; namely, under extreme reducing conditions. The facts in support of these conclusions may be summarized thus:

1. Both olivine and pyroxene are free of iron. The small amount of iron in the meteorite is reduced to clusters of metallic nickel-iron, with a nickel content of only 3·6 per cent, although there is a considerable amount of iron in the sulfide phases.

2. The copper is reduced to metallic copper, however, some copper seems to be present in solid solution in the troilite exsolution lamellae occurring in the ferromagnesian-alabandite.

3. The generally lithophilic chromium does not appear in chromite ($FeCr_2O_4$), as usually in meteorites, but forms daubreelite ($FeCr_2S_4$), thus indicating a chalcophilic tendency caused by the reducing conditions.

4. The manganese is not incorporated in the silicates, but forms ferromagnesian alabandite and appears to some extent in the daubreelite, thus indicating a chalcophilic character caused by the reducing conditions.

5. The predominently lithophilic titanium does not form ilmenite ($FeTiO_3$), as usually in meteorites, but appears chalcophilic and is enriched in the troilite, owing to the extraordinary reducing conditions. Titanium has not been noted before to have chalcophilic tendency, although both the monosulfide and disulfide of titanium are well-known chemical compounds (PEARSON, 1958).

It seems likely that the described paragenesis is in chemical equilibrium, and that the meteorite formed by a rather slow cooling from a melt. The general texture of the rock, as observed in thin sections, is consistent with this view. It was suggested by MASON (1962a) that a differentiation from material similar to the enstatite chondrites could have been the source for Norton County type achondrites. Although the enstatite chondrites are highly reduced, they still contain small amounts of oxidized iron in the silicates (FREDRIKSSON, unpublished microprobe analysis of St. Marks, Indarch, and Hvittis). The metal phase in these meteorites is kamacite with approximately 6 per cent Ni. Assuming a separation of the metal from the silicate by melting and gravitational separation, the small amount of oxidized iron would be reduced to metal. The remaining traces of metallic nickel-iron left in the silicate portion should, therefore, be low in nickel. This is in agreement with the findings in the Norton County achondrite. The nickel content in the metallic nickel-iron of that meteorite amounts to only 3.6 per cent, thus being consistent with Mason's suggestion. However, this process took place in a strongly reducing environment; one quite different from the conditions under which comparable terrestrial rocks have been formed. This difference must be considered in any hypothesis on the origin of meteorites based on a comparison to terrestrial rocks and rock forming processes.

Acknowledgements—We wish to express our gratitude to Professors Hans E. Suess and Gustaf Arrhenius for stimulating discussions and constant interest in this work. Samples from the Norton County achondrite were made available through the generous cooperation of Dr. Carleton B. Moore. Professor T. G. Sahama, Dr. C. Ross, and Drs. A. E. and C. G. Engel kindly provided us with their analysed standard olivines and pyroxenes. Thanks are due to Miss D. Bryan and Miss B. Koontz for preparing the samples, and to Mr. C. Andersen for assistance in the microprobe work. The National Aeronautics and Space Administration provided support for the study (Contract Nos. NsG-317-63 and NsG-322), which is gratefully acknowledged.

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