

Practical limitations of selected area electron diffraction techniques for identifying mineral particles

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Manuscript received in final form November 25, 1976

Selected area electron diffraction techniques are being suggested as potentially useful ones for the identification and characterization of mineral particulates in studies of industrial and environmental health hazards. In many of these studies it has proven to be of great value. However, there are serious limitations in the method which make the interpretation of diffraction patterns ambiguous or drastically increase the amount of operator time required to confirm the identification of each individual particle in a complex sample.

The problems most frequently encountered arise because of variations in the thickness of individual crystallites, their degree of crystal perfection and the presence of polycrystalline aggregates. Well-developed cleavages may also eliminate diagnostic reflections. The precise measurement of interplanar distances requires internal standardization and even then the results from different minerals are so similar that they cannot be differentiated. Electron diffraction patterns are difficult to index and interpret without some prior knowledge of the identity of the particles.

The results obtained from a variety of fibrous minerals indicate that the technique is not well suited for mineral identification in polymineralic samples. It cannot be recommended for quantitative identification techniques but may be useful in a qualitative sense to tell whether a particle was crystalline, fibrous, twinned or a complex aggregate. Electron diffraction must be supplemented with other information in order to obtain an unambiguous identification.

INTRODUCTION

Electron microscopy is an extremely valuable analytical technique which has been employed for many years to study the morphology of finely divided particulate materials. The electron microscope can also be used to obtain a selected area electron diffraction (SAED) pattern which contains information about the crystal structure of the material in the electron beam and in some cases can be used to identify the crystallites. The two techniques are especially useful because the observations are frequently unique enough to provide an adequate identification procedure.

In the field of mineralogy the combined observations are useful in many cases where individual small crystals must be identified. The

technique has been used to determine crystal structures, identify traces of minerals in rocks and characterize the phases formed during thermal decomposition or nucleation of micron-sized particles. There is an increased interest in the mineralogical applications of electron diffraction results as explained by Ross and Christ (1958) because the method potentially can be used to identify small particulates in air and water samples. For example, Clark and Rudd (1974) presented electron micrographs, SAED patterns, and X-ray diffraction data for asbestos standards supplied by the IUCC (International Union Against Cancer) and have suggested ways that the results might be useful in studies of industrial and environmental health hazards.

The SAED technique is extremely difficult to use with some minerals and its application to others should not be attempted without an understanding of the potential sources of error. Among the most serious limitations are the

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complications arising from the variations in sample thickness, the presence of polycrystalline aggregates and the amount of departure of a given (*hkl*) plane from perpendicularity to the incident electron beam. Güven (1974) has discussed some of these problems in detail with respect to micaceous minerals and has even demonstrated that the symmetry of the electron diffraction pattern can be altered by minor changes in the objective lens current. The vacuum and thermal instability of some minerals makes them difficult to examine. Internal standards must be employed to precisely measure interplanar spacings.

The following is a brief review of the principles of electron diffraction and a discussion of the method's practical limitations for identifying particulates in air and water samples. In most cases, diffraction data collected from several specimens of cummingtonite, an amphibole asbestos mineral, will be used as examples of the problems encountered and results obtained. A comparison will be made among the electron diffraction data from minerals with a composition and/or habit similar to the above. The method is subject to more ambiguities than conventional X-ray diffraction techniques but some generalizations and partial identifications can be made. Electron diffraction methods are more suited for monitoring changes in monomineralic samples than identifying the phases present in polymineralic ones. This discussion is similar to ones by Alderson and Halliday (1965) and McConnell (1967).

PRINCIPLES OF ELECTRON DIFFRACTION

A simple device for electron diffraction is illustrated in Fig. 1. High energy electrons are focused by an electromagnetic lens and formed into a coaxial beam. As it passes through the crystal, the electrons are scattered elastically and diffracted beams are produced in directions which may be predicted by the Bragg equation ($n\lambda = 2d \sin \theta$). The diffracted beam may be viewed directly by projection on a fluorescent screen as in the diagram or the deviation may be increased by lenses between the specimen and the viewing screen. In the simplest case, the radial displacement (*R*) of the diffracted beam from the undeviated one is a function of

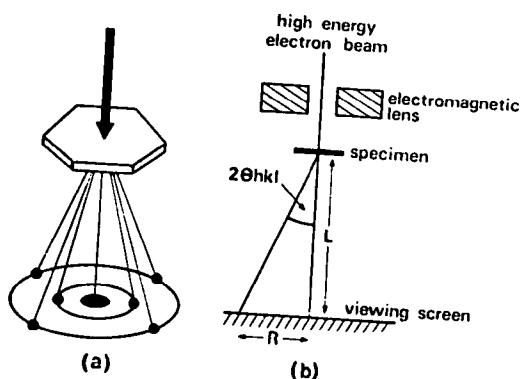


Fig. 1. Basic relationships in electron diffraction. (a) Diagrammatic illustration of diffraction rings or spots produced by the interaction of the specimen and the electron beam. (b) Geometry of diffraction in a simple device. *R* is the radial displacement of the diffraction spot or ring, *L* is the diffraction camera constant and 2θ is the Bragg diffraction angle.

the specimen to screen distance (*L*) and the diffraction angle (θ) such that:

$$\tan 2\theta = R/L, \text{ or } 2 \sin \theta \cos \theta / \cos 2\theta = R/L$$

The energy of the electron beams used in these devices is sufficiently great that only very small values of 2θ are observed and:

$$\cos \theta / \cos 2\theta \approx 1$$

Thus for all practical purposes, the Bragg diffraction condition is:

$$\sin \theta = R/L$$

Substituting the above in the Bragg equation an expression is derived which can be used to determine the direct lattice spacing (*d*) from the diffraction pattern by measuring *R*, when *L* and λ are known.

Thus:

$$d = L\lambda/R$$

In practice, $L\lambda$, the diffraction camera constant, is difficult to measure. It is evaluated by obtaining the diffraction pattern from a standard with known *d*-values. Unknown *d*-spacings can then be obtained by a simple ratio:

$$d_{\text{standard}}/d_{\text{unknown}} = R_{\text{unknown}}/R_{\text{standard}}$$

Variations in $L\lambda$ can be quite pronounced because of the possible fluctuation in a number of the operating conditions. If precise measurements are to be made on electron diffraction

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Fig. 2. Typical electron diffraction patterns showing irregular arrays of diffraction spots caused by the rows of close packed atoms. The pattern

patterns, an internal standard is a necessity. In all results to be discussed below, a thin gold film was used for calibration. The gold diffraction effects are the continuous rings in the electron diffraction patterns (Fig. 2).

Electron diffraction phenomena are fairly

easy to interpret because the pattern produced on the fluorescent screen is a direct image of a section of the reciprocal lattice and the reciprocal lattice is directly related to the mineral's atomic structure. For example, the arrangement of atoms in cummingtonite can be reduced to a

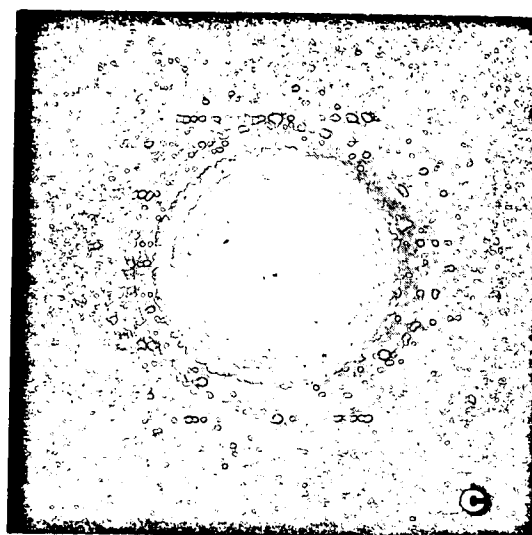
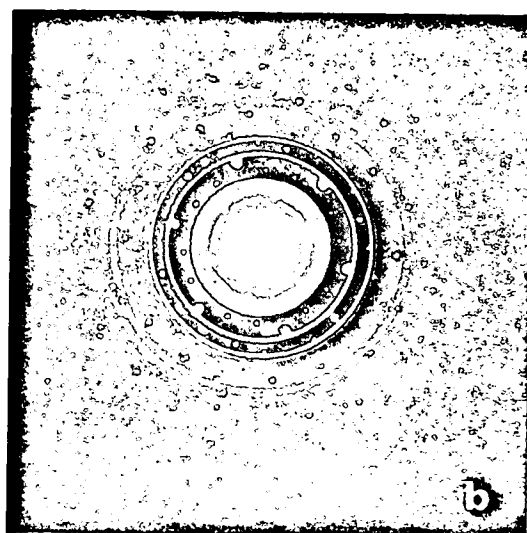


Fig. 2. Typical electron micrograph and electron diffraction patterns produced by fibrous minerals in air or water samples. The micrograph (a) illustrates the straight sides produced by cleavage in crystals of cummingtonite and the tendency for the particles to agglomerate. The irregular background is caused by the thin discontinuous layer of gold applied to the sample for internal standardization of the diffraction effects. Some crystals produce clear pseudo-hexagonal arrays of diffraction spots (b) while others are more complicated (c). The continuous rings are caused by the gold standard. A typical pattern of cummingtonite is illustrated by the latter. The rows of closely spaced diffraction dots are perpendicular to c^* and the rows are separated by d_{001} . The pattern is complicated by a second row of dots from another crystal in the electron beam which lies at an angle to the major crystal. $\times 9,000$.

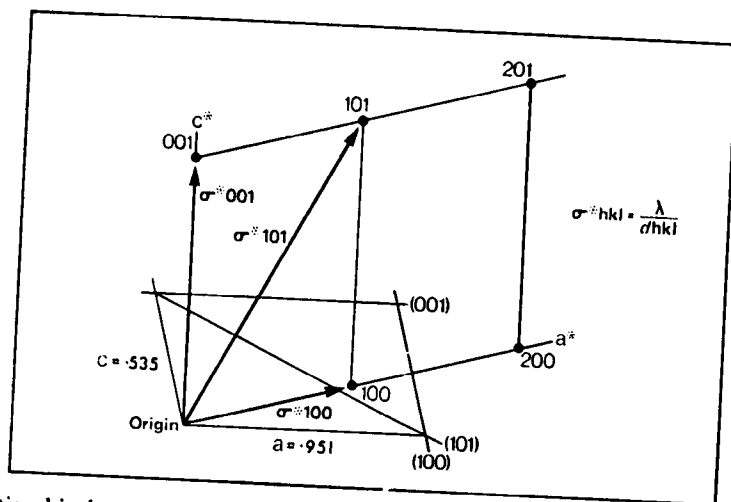


Fig. 3. Relationship between the ac projection of the cummingtonite space lattice and the reciprocal lattice nodes generated from it. Indices in parentheses are actual planes and σ_{hkl}^* is the reciprocal lattice vector.

primitive set of equivalent points which form a lattice with monoclinic symmetry. The vectors defining the actual unit cell are $a = 0.951\text{nm}$, $b = 1.819\text{nm}$, $c = 0.533\text{nm}$ and $B = 101.90^\circ$.

The relationship between an actual lattice plane and the corresponding reciprocal lattice network is shown in Fig. 3. This is a representation of the $(h0l)$ plane of cummingtonite and it can be used to predict the arrangement of spots when the electron beam is parallel to the zone axis (010) . Several planes in the real lattice are shown. The indices of these are enclosed in regular parentheses. The reciprocal lattice points are along lines drawn through the origin and perpendicular to a given reflecting plane. A reciprocal lattice point is, in effect, a point which represents a real plane in the crystal structure. The lengths of the reciprocal lattice vectors which locate the points are inversely related to the distance between the lattice planes and are arbitrarily scaled in the illustration.

The lattice point 100 is on the line representing the reciprocal lattice direction a^* which is perpendicular to (100) and the point 001 is in the direction of c^* which is perpendicular to (001) . The points representing these planes are located at the ends of the reciprocal lattice vectors σ_{100}^* and σ_{001}^* , respectively. The points associated with the planes (101) and (200) are also shown. If the d -spacing of one plane is one-half that of another, the reciprocal lattice point will be twice as far from the origin, i.e. 100 and 200. The resulting pattern would contain all

points in the a^* and c^* plane such as 100, $\bar{1}00$, 001, $00\bar{1}$, 101, $\bar{1}01$, etc.

A complete planar array of reciprocal lattice points could be constructed as in the above fashion for other planes in the crystal such as $h1l$ or $h2l$ and a three-dimensional network would result. This is the reciprocal lattice, but all of the points in it would not appear in the diffraction pattern. Only those points which intersect the Ewald sphere are observed. This sphere which limits diffraction is drawn through the origin of the reciprocal lattice; its centre is in the electron beam and its radius is $1/\lambda$.

In electron diffraction, a number of reciprocal lattice points are usually observed because the reciprocal lattice vectors are very small and the Ewald sphere is large. A section through the reciprocal lattice and the limiting sphere helps explain this situation (Fig. 4). As shown, the reciprocal lattice points are elongate spikes because the crystals yielding good diffraction are usually thin and, therefore, are not perfectly triperiodic. The resulting diffraction pattern would contain the points 000, 001, $00\bar{1}$, 002, 013 and $01\bar{3}$.

Single crystal electron diffraction patterns can be interpreted in a fairly straightforward manner because with a little knowledge of crystallography one can predict the kind of image to be formed by certain minerals. Close agreement between observed and theoretical patterns identifies the particles. However, in

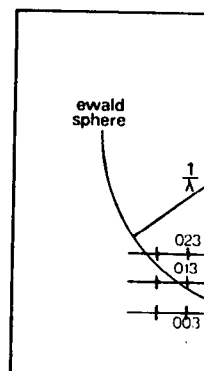


Fig. 4. A hypothetical sphere, which limits the reciprocal lattice of cummingtonite, intersecting the Ewald sphere.

practice, electron diffraction patterns are rather ambiguous and the problems are often solved in more detail later.

1. Crystals may be distinguished by measurement of internal standard reflections.
2. Well-developed diagnostic reflections of planar spacings are because the zone may not be coincident.
3. Polycrystalline diffraction may produce spectra which may be more difficult to interpret.

MATERIAL

After a careful study of electron diffraction phenomena from a standpoint, several Philips EM-100 transmission electron microscope and actual diffraction patterns obtained. The mineral properties are listed in Table 1. The samples were selected because they were commonly occurring and their ratios greater than 100. The test the applicability of the identification of the samples were the following manner.

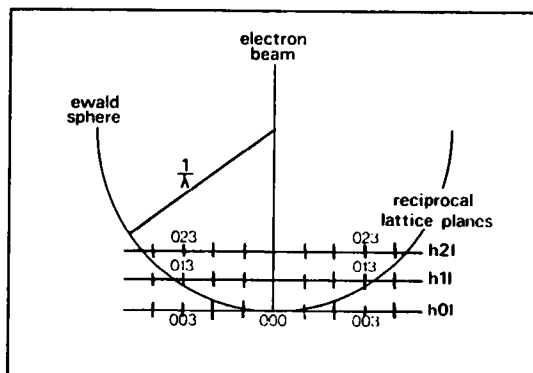


Fig. 4. A hypothetical section through the Ewald sphere, which limits diffraction, and the reciprocal lattice of cummingtonite. The elongate lattice nodes intersecting the Ewald sphere are the points which appear in the electron diffraction pattern.

practice, electron diffraction patterns can be rather ambiguous or difficult to interpret. Some of the problems are outlined below and discussed in more detail later.

1. Crystals may be imperfect. 2. The precise measurement of interplanar spacings requires internal standardization. 3. The d -spacings of many minerals are so similar that they cannot be distinguished by electron diffraction methods. 4. Well-developed cleavages may eliminate diagnostic reflections. 5. The measured interplanar spacings are usually longer than expected because the zone axis of the diffracting plane may not be coincident with the electron beam. 6. Polycrystalline or fibrous mineral aggregates may produce special diffraction effects which may be more difficult to analyse.

MATERIALS AND METHODS

After a careful consideration of electron diffraction phenomena from a theoretical standpoint, several minerals were examined in a Philips EM-100 transmission electron microscope and actual diffraction patterns were obtained. The minerals studied and some of their properties are listed in Table 1. These minerals were selected because they are representative of commonly occurring ones with length to width ratios greater than 3:1, and are good ones to test the applicability of the SAED method for the identification of micron-sized particles.

The samples were prepared and examined in the following manner: 1. A thin, discontinuous

gold film was applied to a collodion-coated 200 mesh electron microscope grid. The gold film forms the grainy background of Fig. 2. 2. The mineral particles were dispersed in water and a droplet of each suspension was applied to separate grids. 3. Electron diffraction was produced in the electron microscope operated at 80kV. 4. The patterns for the minerals with the internal standard were recorded on photographic film. 5. A working copy of the diffraction pattern was prepared by placing it in an enlarger and projecting the spots on a piece of plain white typing paper. The positions of the dots were marked with a pencil. Alternately one could directly measure from the projected image, but we found a copy to be more desirable for reproducible measurements. 6. Uniform enlargement of the diffraction patterns was obtained by adjusting the projector until the gold diffraction ring with a spacing of 0.0695nm was exactly coincident with a previously drawn standard circle having a radius of 2.56in. 7. The patterns were indexed, if possible, and R was measured with a scale having fifty divisions to the inch. Repeated measurements indicate this technique has an error of ± 0.001 nm. The camera constant, including the enlargement step, was 890.12. This value was used to simplify the data handling and reduction. It is, of course, a unique value for each laboratory. 8. The interplanar spacings were measured and recorded as the number of units on the scale, then divided by 890.12, the camera constant in this procedure, to convert to nm units.

DISCUSSION OF RESULTS

The electron diffraction patterns of cummingtonite are characterized by two rows of dots (Fig. 2). In one direction, the spacing between the dots is much smaller than the other and the pattern has a layered appearance. The dots form a regular planar array that is almost rectangular instead of the 102° parallelogram as illustrated previously. Because of this difference, one must assume that the crystals are in some orientation other than the one described and it is necessary to try and determine the orientation before proceeding.

Cummingtonite has a prominent cleavage $\{110\}$, which creates elongate crystals and enhances the possibility of preferred orientation. The crystals most likely will come to rest on $\{110\}$ or some other face in the form. The c -axis

Table 1. General properties of minerals which may have a fibrous habit and therefore may be difficult to distinguish from cummingtonite. The unit cell dimensions commonly overlap and the presence of well developed prismatic cleavages increase the likelihood that an identification by electron diffractions techniques will not be unambiguous

Mineral	Crystal system	Cleavage	Approximate unit cell dimensions (nm)			Composition	Remarks
			a	b	c		
Actinolite	Monoclinic	{110} good	0.99	1.81	0.53	$\text{Ca}_2\text{Mg}_6[\text{Si}_8\text{O}_{22}](\text{OH})_2$	Long, needle-like crystals
Amosite	Monoclinic	{110} perfect	0.96	1.83	0.53	$(\text{Fe}, \text{Mg})_7[\text{Si}_6\text{O}_{22}](\text{OH})_2$	Iron rich fibrous variety of cummingtonite
Anthophyllite	Orthorhombic	{210} perfect	1.85	1.77	0.527	$(\text{Mg}, \text{Fe})_7[\text{Si}_8\text{O}_{22}](\text{OH}, \text{F})_2$	Long, needle-like crystals or laths
Crocidolite	Monoclinic	{010}{100} imperfect {100} good	0.97	1.77	0.53	$\text{Na}_2\text{-Mg}_3\text{Al}_2[\text{Si}_8\text{O}_{22}](\text{OH})_2$	Fibrous, blue coloured variety glaucophane
Diopside	Monoclinic	{110} good	0.975	0.891	0.525	$\text{Ca Mg}[\text{Si}_2\text{O}_6]$	Usually blocky, irregular crystals
Grunerite	Monoclinic	{110} good	0.96	1.83	0.53	$(\text{Fe}, \text{Mg})_7[\text{Si}_8\text{O}_{22}](\text{OH})_2$	Long, needle-like crystals common
Hedenbergite	Monoclinic	{110} good	0.985	0.902	0.526	$\text{Ca Fe}[\text{Si}_2\text{O}_6]$	C* usually present, patterns generally incomplete
Hornblende	Monoclinic	{110} good	0.99	1.80	0.53	variable amphibole with $(\text{Na}, \text{K})\text{Ca}_2(\text{Mg}, \text{Fe}, \text{Al})_5 \dots$	
Tremolite	Monoclinic	{110} good	0.99	1.81	0.53	$\text{Ca}_2(\text{Mg}, \text{Fe})_6[\text{Si}_8\text{O}_{22}](\text{OH}, \text{F})_2$	Long, needle-like crystals
Albite	Triclinic	{001} perfect {010} good {110} poor	0.814	1.278	0.716	$\text{NaAl Si}_3\text{O}_8$	Usually tabular crystals
Enstatite	Orthorhombic	{210} good	1.823	0.881	0.519	Mg SiO_3	Stubby irregular crystals
Hypersthene	Orthorhombic	{210} good	1.82	0.88	0.52	$(\text{Mg}, \text{Fe})\text{SiO}_3$	Usually not fibrous
Wollastonite	Triclinic	{110} perfect {001}{102} good	0.794	0.732	0.707	Ca SiO_3	Diffraction points from a nearly square array
Chrysotile	Monoclinic	fibrous parallel to X	1.466	0.924	0.533	$\text{Mg}_3[\text{Si}_2\text{O}_5](\text{OH})_4$	Occurs as flexible fibre formed by rolling up plates
Cummingtonite	Monoclinic	{110} good	0.96	1.83	0.53	$(\text{Mg}, \text{Fe})_7[\text{Si}_8\text{O}_{22}](\text{OH})_2$	Long acicular crystals

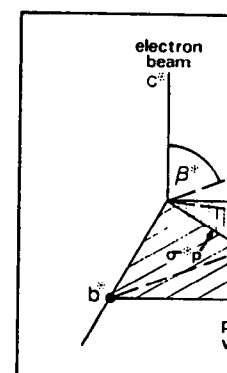


Fig. 5. Preferential developed cleavages and diffraction patterns difficult to interpret from perpendicular plane to the electron beam. The reciprocal lattice vectors are shown.

The above analysis of the observed patterns. The patterns represent the distance between reciprocal lattice points correspond to multiple points are in reciprocal space. The distances on the pattern correspond to the d -spacings in the crystal. The prominent interpretation of the patterns reduces the amount of information that can be obtained. The patterns in the spacings to be compared with the lattice vectors are of these limitations with any reliability.

The results of the ten different specimens shown in Table 2. The spacing of 0.5465 nm as compared with the methods. This is in agreement that one of the two methods, but the

therefore will be parallel to the specimen support grid and the repeat distance along it should therefore appear in the diffraction pattern. The other orientation represents some plane parallel to c and intersecting the a and b axis, but its precise orientation cannot be determined. The maximum spacing to be expected should not exceed about 2nm. (Fig. 5).

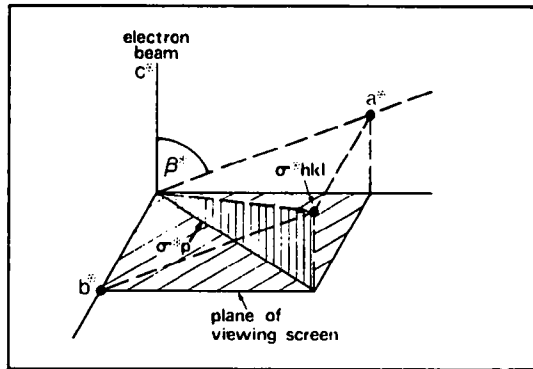


Fig. 5. Preferential orientation of crystals on well-developed cleavages may make some electron diffraction patterns difficult to interpret. The departure from perpendicularity of a given reciprocal lattice plane to the electron beam causes the projected reciprocal lattice vector σ^*p to be different from σ_{AH}^* .

The above analysis is consistent with the observed patterns. The closely spaced points represent the distances along the unidentified reciprocal lattice vector. The other points correspond to multiples of 001. Because the points are in reciprocal space the shortest distances on the pattern correspond to the largest d -spacings in the crystal.

The prominent {110} cleavage helps in the interpretation of the pattern, but drastically reduces the amount of useful information that can be obtained. There is considerable variation in the spacings to be observed and the reciprocal lattice vectors are difficult to identify. Because of these limitations only d 001 can be measured with any reliability.

The results of the measurement of d 001 for ten different specimens of cummingtonite are shown in Table 2. The average spacing is 0.5465nm as compared to 0.5215nm by X-ray methods. This is approximately the amount of agreement that one would expect between the two methods, but the deviation about the mean

Table 2. Results of cummingtonite d -spacing measurements by electron diffraction

Average value for d -001	0.5465nm
Standard variation in 10 measurements	0.027nm
Range of observed values	0.5267–0.6268nm
Average d -001 determined by X-ray diffraction	0.5215nm
Range of measured values in unidentified crystallographic direction	1.0049–1.9074nm

is very large for electron diffraction results. For example, the measured values ranged from 0.5267–0.6268nm. This is because electron diffraction is a single crystal measurement for the most part, and slight variations in the orientation of the crystal or mistakes in indexing the pattern will have pronounced effects.

In the unidentified reciprocal lattice direction the measured d values vary from 1.0049nm–1.9074nm. In some cases, it is difficult to decide what to measure. As suggested earlier, these values have no practical significance for mineral identification because you would have to have prior knowledge of the minerals present in order to make the determination. In other words, the orientation of cummingtonite on its well developed cleavage restricts the number of actual d -spacing measurements that can be made without assuming *a priori* identification and orientation.

The indexing of the electron diffraction patterns can be difficult if systematic absences reduce the number of dots or if twinning, crystal defects, or polycrystalline particles increase the number of dots. In Fig. 2c, the array of reciprocal lattice points has a pseudo-hexagonal appearance because reflections with $l = 1$ are absent. Twinning and rotation of the $h^*k^*l^*$ reflecting plane about the electron beam produce a pattern which is typical of polycrystalline materials. The cummingtonite pattern has begun to develop diffraction rings. Unfortunately these rings are no more useful than the spots because you cannot identify the spacings they represent. The number of rings produced by a polycrystalline aggregate may also be too great and closely spaced for effective measurement.

Electron diffraction methods are limited in the identification of cummingtonite. In good, single crystal patterns, the appearance of the

Occurs as flexible fibre formed by rolling up plates

Long acicular crystals

$\text{Mg}_3[\text{Si}_2\text{O}_5](\text{OH})_4$ 0.533 0.533 0.924 1.466
fibrous parallel to X {001} {102} good

Monoclinic

Chrysotile

$(\text{Mg}, \text{Fe})_7[\text{Si}_8\text{O}_{22}](\text{OH})_2$ 0.53 0.53 1.83 0.96
{110} good

Monoclinic

Cummingtonite

reciprocal lattice points is characteristic in a qualitative sense because the closely spaced rows of dots are readily observed. Because of the good {110} cleavage, quantitative measurements are restricted to $d\ 001$ and even this may vary considerably.

Many of the other minerals in this study set possess a good or perfect cleavage parallel to the c -crystallographic axis. The symmetry may be slightly different but the cleavage forms are usually {110} or {210}. Therefore the same general prismatic forms and orientations would be expected and only c -axis spacings of the same magnitude as that of cummingtonite should be observed. Good, complete single crystal electron diffraction patterns of these should be identical within the limits of the technique.

Amosite, anthophyllite, hornblende, crocidolite, hedenbergite, actinolite, diopside, tremolite and grunerite have the requisite cleavage. The c -axis dimensions also fall within the narrow limits 0.5251nm to 0.5348nm. This range is narrower than the one observed for the cummingtonite samples. Enstatite and hypersthene are also likely candidates for the above category, but their patterns should exhibit a truly orthogonal array.

As predicted, most of the SAED patterns from the above minerals were like those of cummingtonite. When good single crystal patterns were obtained, they could be overlaid and the distances between the points along c^* were similar (identical within experimental error) and the spacings in the other directions were variable and difficult to relate to a specific crystallographic direction.

Four hornblende diffraction patterns provide good examples to illustrate the extreme variability in d -spacing measurements caused by the preferred orientation of crystallites. Circles were drawn through the points on incomplete diffraction patterns in order to determine how many discrete reflections were present. The number of d -spacings observed varied from seven to eighteen on the different specimens. These patterns were then placed on a light table and the coincident circles marked on each. The maximum agreement between circles and therefore d -spacings was four. In most cases the circles corresponding to a multiple of $d\ 001$ were the ones which overlapped perfectly. This should make it very clear that the only consistent parameter to be determined for these minerals by the electron diffraction technique

is the repeat distance between reciprocal lattice points along c^* and this dimension is not unique enough to identify the minerals of the amphibole family or closely related mineral species.

Wollastonite, albite, and chrysotile specimens produced electron diffraction patterns which were different from cummingtonite. The wollastonite reciprocal net was almost a square with edges of 0.7418nm and 0.7120nm. This is very close to what would be expected from a crystal of wollastonite and the spacings represent b^* and c^* , respectively. One of the albite patterns formed a partial parallelogram and spacings of 0.8092nm and 0.7418nm were measured and identified tentatively as a^* and c^* . The results were not this good for all the specimens and therefore most of the incomplete patterns would not have been identified as wollastonite or albite, or even distinguished from cummingtonite. Therefore, even in cases where the minerals being examined have very different crystal structures, the inability to obtain a complete diffraction pattern from all particles makes specific identification of each small crystal impossible. Thickness variations and crystal defects are thought to be the major causes of this problem.

One of the most unique diffraction patterns observed was that of chrysotile. The characteristic rolled tube crystal morphology of this mineral creates a situation where all possible reciprocal lattice planes parallel to the fibre axis are in a position to satisfy the condition for diffraction. As a result the pattern is a series of streaks perpendicular to the fibre axis. Occasionally, spots may be superimposed on the streaks or small segments of arcs from fibres parallel to the electron beam are seen. One does not find a great deal of difference between the chrysotile and cummingtonite spacings because the axis about which the rolling takes place is the a -axis and its length is 0.534nm. The streaked diffraction lines are essentially equivalent to the pattern produced in a rotation X-ray camera and are quite distinctive for identification of chrysotile. However, all chrysotile particles did not yield the same results and there are other minerals which may exist as rolled tubes, i.e. halloysite.

SUMMARY

The results obtained during the study support the contentions of others (Brindley and de Kimpe, 1961, as well as Ross and Christ, 1958)

that electron diffraction is a useful technique for X-ray diffraction of polymineralic samples to identify single crystals and avoid the problems encountered with X-ray diffraction patterns.

Electron diffraction patterns can be obtained and only the best quality crystal patterns for identification. Different orientations can be obtained and interpreted quantitatively. Quantitative measurements of cleavages in many minerals can be obtained from a single crystal and restrict selection. In the amphibole family, only repeat distances $d\ 001$, could be measured. There is more variation in $d\ 001$ for samples of the same mineral than for separate species.

Other minerals such as chrysotile may be identified by their electron diffraction patterns in some cases. The shape and the quality of the diffraction pattern are important in identifying characteristics of the patterns are available for identification of circles from polymineralic samples good for identification.

In conclusion, electron diffraction patterns are difficult to interpret and are not recommended for the identification of mineral particles. It would be very difficult to obtain results to determine whether a sample is amorphous, an amphibole, or a chrysotile. In a qualitative sense, electron diffraction can determine whether a fibre was a mineral. The difficulties involved in electron diffraction are especially important in identifying the so-called "halloysite" which may exist as rolled tubes, i.e. halloysite.

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Brindley and de
and Christ, 1958)

that electron diffraction is not as amenable as X-ray diffraction to mineral identification in polyminerallic samples. It is virtually impossible to identify single crystal particles because of the problems encountered in interpreting SAED patterns.

Electron diffraction patterns are difficult to obtain and only those relatively complete single crystal patterns from specimens in special orientations can be unambiguously indexed and interpreted quantitatively. Well-developed cleavages in many monoclinic minerals prevent the crystals from assuming this special orientation and restrict severely the methods utility. In the amphiboles and pyroxenes examined only repeat distances of 0.53nm, equivalent to d_{001} , could be measured and unfortunately there is more variation in the measurement of samples of the same species than there is among separate species.

Other minerals such as wollastonite, albite and chrysotile may be distinguished from amphiboles in some cases. The cell dimensions, its shape and the qualitative appearance of the diffraction pattern may be used as identifying characteristics when relatively complete patterns are available. Rings or diffraction circles from polycrystalline particles are not good for identification purposes.

In conclusion, electron diffraction analyses are difficult to interpret and the technique cannot be recommended for quantitative identification of mineral particles in composite samples. It would be very difficult to use electron diffraction results to determine whether a fibre was amorphous, an amphibole or a non-amphibole. In a qualitative sense, it may be possible to tell whether a fibre was chrysotile or another mineral. The difficulties inherent in the method are especially important to consider when attempting to identify the source of particulate pollutants

because electron diffraction techniques have to be supplemented with other information such as micro-probe determinations in order to obtain unambiguous information. It should not be used as the sole means for establishing the identity of mineral particles in complex samples.

In this paper, we have attempted to call attention to some of the negative aspects or difficulties associated with the SAED technique because there are many electron microscopists entering this field who might not be aware of its limitations. There are ways to overcome many of the problems which we have discussed and we cannot deny that electron diffraction is a very powerful analytical technique. However, the difficulties do exist and are very prevalent when dealing with natural samples composed of many mineral varieties. Practical considerations such as time and effort do restrict the utility of the SAED technique.

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