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August 1980

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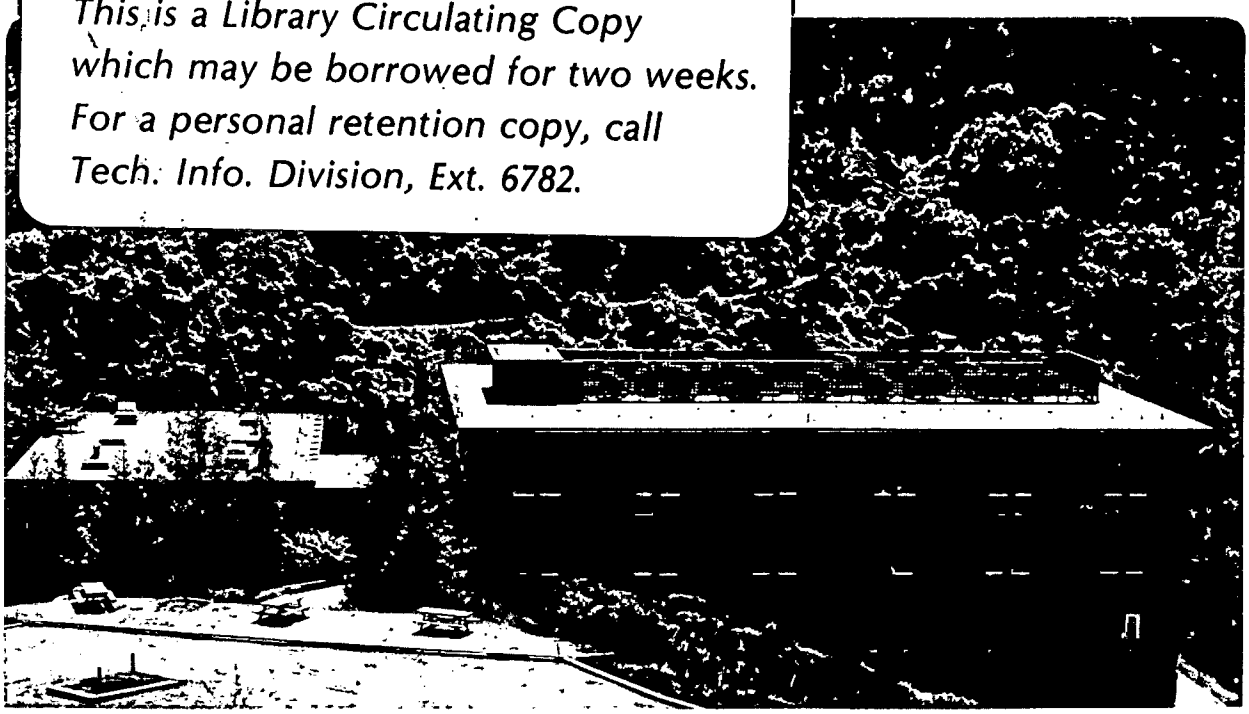

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TRANSMISSION ELECTRON MICROSCOPY STUDIES OF COALS

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ABSTRACT

Various examples of both high and low rank coals from the Eastern and Western United States have been examined using both TEM (transmission electron microscopy) and STEM (scanning transmission electron microscopy) techniques. These techniques allow the characterization of both the coal and mineral matter microstructures as well as the rapid identification of the particular minerals in the coals. Most of the coals were found to contain both quartz-like and alumino-silicate minerals as well as pyrite particles and a variety of other minerals depending on tbe coal studied. The sub-bituminous coals were more difficult to prepare and seemed to show some evidence of partial charring due to interaction with the ion beam used in the ion beam thinning procedure. Such interaction highlights the importance of careful specimen preparation and a method was developed to minimize any artifacts introduced by the preparation method.

Introduction

In recent years a vast amount of research has been concerned with the problems concerning the efficient conversion of various coals and coal products into high grade energy sources via gasification and liquefaction (eg 1-6). Much of the work has focussed on the usual chemical techniques for identification of the various chemical constituents of the coal which are active in conversion processes (eg 7-9), and on gravimetric analysis during conversion experiments to gain data on the reaction rates of different coals in different environments and to measure the activation energies involved (eg 10-12). These studies have been successful but have continued to raise other important questions concerning the more fundamental parameters involved during coal conversion processes, especially at the microscopic level.

Both the structure and the maceral content of the coals are obviously important in subsequent operations. The structure is reflected in the size and distribution of the pores in the coal as well as the grain size and shape of the various constituents. The maceral content will determine the relative amounts of volatile matter and fixed carbon in a coal of a given rank; different macerals may react differently under conversion conditions. A further important factor is that it has recently been realized that the inherent mineral matter in coal has an effect on its behavior during conversion (2-3, 13-14), although neither the precise mode of operation of this effect nor the particular minerals involved are well understood.

These problems suggest microscopic investigation of various coals and, of course, a well established system of coal petrography has existed

for many years (15). However the coal microstructure, the fine scale porosity and the submicron sized minerals do not lend themselves easily to optical microscopic investigation. Consequently, in recent times, both the transmission electron microscope (TEM) and the scanning transmission electron microscope (STEM), fitted with X-ray analytical devices, have made possible the characterization of coal microstructures at very high resolution and the positive identification of minerals incorporated within the coal.

Harris and co-workers (16-19) have investigated both the porosity and mineral matter contained in some Eastern coals, but the very inhomogeneity of coals means that the greatest information can only be gathered from examinations of many different coals and a subsequent comparison and contrasting of the experimental results. Also, coals from the Western states of the U.S. are Cretaceous or Tertiary in age and are therefore derived from plants of quite different anatomy compared with plants in the Carboniferous that gave rise to the coals in the Eastern U.S. (7). This paper will thus report on the characterization of various coals, including several western coals, using transmission electron microscopy.

Experimental

The aim of the study was to examine the microstructures and mineral contents of a wide range of coals and therefore anthracites, bituminous and sub-bituminous coals were studied. Full details of the origins of the coals and the proximate and ultimate analyses are given in Tables 1 and 2.

The inhomogeneity and reactivity of coal means that great care must be taken in preparing representative specimens for the microscope, and to avoid artifacts in the coal microstructure due to specimen preparation. For this reason chemical preparative techniques can be eliminated due to selective attack by any chemical reagents used on different parts of the coal microstructure. Thus an ion-beam thinning technique must be used but again caution is necessary during specimen thinning to ensure representative results. In ion-beam milling a beam of energetic argon ions ($\simeq$ 6kV accelerating potential) is impinged onto the specimen surface which is angled at approximately 10-15° to the beam. During this procedure the sample holder can become quite hot and thus, by inference, the sample also; in certain metallic alloys temperatures as high as 200°C have been found to occur (20). Also the argon ions thin the sample by sputtering away the surface atoms, layer by layer, until a small hole develops, the edges of which are then thin enough for electron transmission. This process can give rise to surface structures resulting from the ion bombardment which are not truly representative of the specimen. To overcome both of these drawbacks a procedure can be adopted which also allows the investigation of the coal macerals and their relationship to the microstructures observed in the electron microscope. The procedure is elementary in that it merely involves very careful hand grinding and polishing on successive diamond pastes down to a 0·25 μm paste, until the specimens become optically transparent. At this stage specimens are approximately 5-10 μm thick and as ion-thinning machines thin at approximately 2 μm/hour then a mere 2-3 hours are required for final thinning. A liquid nitrogen cooled cold stage on the ion-thinner is also advantageous to avoid heating effects and the use of a helium sputtering atmosphere is being investigated.

Another advantage to the thinning procedure is that during optical transmission the various maceral groups in the coal are easy to distinguish due to each one transmitting a different color ranging from a light yellow through red to brown and black. Thus the final procedure is as follows:

1. Specimens are hand ground to optical transparency and a montage of photographs made to indicate the distribution of the macerals.
2. The specimen is mounted between a folding copper grid and a second photograph is taken showing the relative position of the sample and the grid.
3. A low magnification (x60) electron microscope image is taken to correlate both optical and electron optical images.
4. Finally the various areas can be viewed in the electron microscope at high magnification and the microstructure or mineral matter appropriate to a given area of the coal can be examined.

This procedure allows the correlation of microstructures to macerals but for merely viewing the mineral morphology, simple electron microscope observation is sufficient and some examples of this will also be reported here. Most observations reported were made on a Phillips 400 TEM/STEM electron microscope with an X-ray analyser, although use was also made of a Phillips 301 microscope, and a Hitachi 650 kV microscope to observe thicker areas.

Results and Discussion

Anthracites

High-ash. The reflected light optical micrograph (Fig. 1) shows

this high-ash anthracite to be a mixture of grey and black areas with a microstructure in which it is very difficult to identify individual macerals. However, with increasing rank the individual macerals become less and less different in reflectance so that it becomes more difficult to differentiate between them under the microscope (15). The very high mineral matter content of this coal will also tend to obscure identification of the macerals. The electron micrographs reveal the extent to which this mineral matter is intimately connected to the coal structure. This coal was found to contain many particles which contained only titanium as seen in the X-ray display (Fig. 2)at A. (The spectrometer will only successfully detect elements with atomic weights greater than approximately that of magnesium.) By using a finely focussed electron beam with a probe diameter of approximately 100 Å, micro-micro diffraction patterns can be obtained from a single particle and thus here the titanium bearing particle could be identified as anatase, a mineralogical form of TiO_2. Identification of titanium bearing minerals in coal can be important because in the electrolyte production of aluminum, even a few ppm of titanium in the carbon electrodes are harmful, because they cause brittleness in the finished metal (15). Also, titanium has a detrimental effect on the Co-Mo catalyst used in liquefaction processes. The other minerals most abundant in this coal, as in most other coals, were different forms of the clay group minerals. Fig. 2(Band C) shows a large grain of clay and the intimate association of this clay with the nearby coal grain. The extensive streaking of the diffraction spots in the diffraction pattern indicate that the grain is composed of very many thin clay platelets in identical orientation as may be expected from the

layer like structures of the clay minerals. Unfortunately these minerals underwent rapid degradation under the electron beam forming amorphous areas (C) and occasionally displaying even more severe damage in the STEM unit (Fig. 3) and so a series of diffraction patterns was difficult to obtain. (This possibility of specimen/beam interaction has always to be considered when examining volatile materials like coal.) However, by measuring the streaked diffraction pattern shown, and with evidence from the X-ray specimen, this material can be tentatively labelled as sericite, a form of muscovite($K\ Al_2Si_3Al\ O_{10}(OH)_2$). This would also explain the instability in the electron beam as the material could lose water of crystallization and/or hydroxyl groups. This is known to occur in kaolinite, a similar hydrous layer lattice silicate, which dehydrates to form an intimate mixture of silica and alumina (21). The presence of kaolinite like materials is important in that a high kaolite content of a coal leads to a substantial rise in the ash fusion point, and this is an important factor in the evaluation of boiler coal. However, Mukherjee et al. (22) found a continual rise in conversion via hydrogenation, with percentage kaolin in the coal fraction, an effect which Gray (13) attributes to the kaolin acting as a dispersant. Thus knowledge of the kaolin content of a coal can be extremely important.

Low-ash. The low-ash anthracite was almost featureless in the optical microscope (Fig. 1) with none of the structure evident in the high-ash sample. This would confirm the influence of the mineral matter on the microstructure of the high-ash sample. In the electron microscope the low-ash anthracite showed occasional small particles (Fig. 4) which in this case could be identified by microdiffraction as aragonite ($CaCo_3$).

The microstructure of the coal material itself showed similar characteristics in both anthracites (Fig. 5) and seemed to indicate a fairly dense structure with little evidence of porosity. The absolute morphology of the coal grains may, in part, reflect the ion-beam thinning procedure as both anthracites were ion-thinned from approximately 50 μm. However, as will be seen with the bituminous coals, comparison of morphologies between such ion-thinned coals and those ion-thinned from merely 5-10 μm shows quite good agreement and thus these morphologies can be thought to give some indication of the true coal morphology. The X-ray spectrum shows there to be sulphur present in the coal grains but at a relatively low level as can be estimated by comparing the sulphur peak height with the general background X-radiation (bremsstrahlung) and this is confirmed in the analysis.

Bituminous Coals

High volatile Class A (Eastern). Figure 6 shows a reflected light optical micrograph in which can be seen various macerals of different groups associated together. The dominant microlithotype observed in this coal is vitrite with bands of other microlithotypes and macerals ranging in width from 5-15 mm. Fig. 6 shows several vitrite bands with areas of fusinite and thin layers of cutinite. The electron microscopic examination of such an inhomogeneous sample obviously presents difficulties in recognizing the exact area from which the observation is made and so the method of examination outlined in the experimental section was used. While this procedure is being used with all of the bituminous and sub-bituminous coals the full discussion will only be given in this section to avoid unnecessary repetition. Figure 7 is a montage of micrographs

which shows the sequence of events from identification of the macerals to examination in the electron microscope. Figure 7a is an optical micrograph in reflected light showing vitrinite at C and a fusinite area at A. At this stage the sample would also be examined in transmitted light. In the next three micrographs the same sample can be seen mounted between a folding copper grid before (7b) and after (7c, 7d) various stages of ion-milling. Thus the original areas can be easily correlated with those areas having holes after the ion-thinning. The final two micrographs show how a low power electron image can be used to locate a hole seen in the optical microscope and finally the magnification increased to examine the micromineral matter and the structure of the coal. Figure 8 shows micrographs of the two different areas 'A' and 'C' shown in Fig. 7. Figure 8a shows an example of the large particles observed in the region 'A' of the specimen. These particles of average size between 0.25 μm and 2 μm were tentatively identified as kaolinite from the X-ray and diffraction data. The micrograph of Figure 8b shows an area of the region 'C' in the vitrinite region of the specimen. In contrast only a very few small particles were found in this region which seemed to contain aluminum and silicon. Figure 8c shows an electron micrograph from the Telocollinite region with very small regularly shaped particles of average size approximately 0.2 μm. X-ray analysis showed these particles to contain mainly aluminum with a small amount of calcium. It is important to note that the morphology of the coal grains showing various flake like structures is very similar in both those samples ion-thinned from 5-10 μm and those ion-thinned from 50 μm, thus indicating that ion thinning is not producing massive damage in the specimen.

High volatile Class A (Western). Both reflected and transmitted light micrographs of this western coal show it to be very similar to the Eastern coal. Again vitrinite is the most common maceral although the proportions of maceral, sub-macerals and microlithotypes vary between the coals. A detailed study of this difference has not yet been made. Both the bituminous and sub-bituminous coals studied showed similar types of mineral particle and therefore all descriptions of these particles will be given in this section to facilitate the explanations. By far the most widely found particles in these coals and generally in most coals are those particles containing only silicon, i.e. quartz-like minerals, and those containing both aluminum and silicon and sometimes potassium and/or sodium, i.e. the alumino-silicate class of minerals. Fig. 9 shows examples of both of these minerals. The particle at A was found to contain only silicon and, by using a finely focussed electron beam to obtain various micro-micro diffraction patterns, was identified as melanphlogite, a form of SiO_2. The alumino-silicate particle at B was not absolutely identified due to the tendency for it to degrade under the electron beam, presumably because most of these minerals are associated with hydroxyl groups and water of crystallization which can be lost during prolonged exposure to the beam. The X-ray spectrum of this area also shows a comparison with the nearby coal grain at C which shows merely the presence of organic sulphur with no diffusion of aluminum or silicon into the coal. The various particles range in size from 100 Å to a few microns in diameter and appear homogeneously distributed in the coal. An important mineral found in the western bituminous coal studied here was pyrite as seen in Fig. 10. The distribution of pyrite in coal is important because an excessive sulphur content in a coking coal leads to a corresponding

sulphur content in the coke, which is undesirable. Also a high pyrite content of coal means a correspondingly high emission of SO_2 on burning and so more expensive gas treatment processes. However, it is possible that the interconversion of pyrite and pyrrhotite may be an important step in catalysis by mineral matter. Thus identification of these sub-micron sized particles and the subsequent investigation of their role during coal gasification will be very important.

Sub-bituminous coals

The main coal studied in this category appeared different from the bituminous coals in reflected light optical micrographs (Fig. 11). As can be seen, the microstructure is very complicated and a full analysis of all of the constituents has not yet been undertaken. This coal also shows various minerals of the quartz-like and alumino-silicate groups but also a higher percentage of calcium and titanium bearing minerals. Also the higher moisture content of this coal together with its high volatile content may make it even more susceptible to partial charring in the ion-beam thinner especially after long exposures. This may be evident in Fig. 12 where the microstructure appears indicative of some chemical reaction having occurred due to the more rounded edges of the sample and in such a region it is thus possible that the coal examined is in fact a semi-char. Unfortunately attempts to hand thin this coal to an optically transparent 5-10μm have so far proved unsuccessful due its extreme brittleness. The catalytic effect of certain minerals within coals is often ascribed to their ability to 'wet' the surface of the coal and provide additional reaction sites (10). As a semi-char this coal will already have 'gasified' to some extent and therefore it is instructive to compare the elemental distribution on the coal. This can

be seen in Fig. 13. Elemental mapping of the silicon and titanium exposes the silica and titania particles which appear as essentially discrete particles against a background of random "noise" from the X-ray detector. The sulphur scan shows a general level of sulphur throughout the sample as would be expected from the organic sulphur in the coal grains, but the calcium scan shows no localization of calcium bearing particles but instead a high level of calcium spread evenly over the coal grains of this particular area. Thus it would appear that calcium does have a good ability to 'wet' the surface of the coal. However, this may only be a reflection of the fact that calcium can be organically bound to carboxyl groups in low rank coals.

Conclusions

Coal is a very heterogeneous substance at both macro and micro levels and the more characterization that we have of it using as many techniques as possible the greater will our understanding be. In this respect the scanning transmission electron microscope is well suited to the examination of basic coal microstructures giving both localized diffraction information and some ideas of the general morphology of coal grains. With added X-ray analysis capabilities it can easily identify minerals down to sub-micron sizes and thus enable full characterization of this mineral matter both before and after any gasification or liquefaction reactions. In this way it will be possible to focus on the catalytic ability of any one mineral and determine its effects during the course of a reaction. These minerals have been shown to be intimately associated with the coal grains which may enhance their catalytic effect. The low-ash anthracite was shown to have only a few small particles of $CaCO_3$ and SiO_2 whereas, as expected, the high ash anthracite contained much silaceous material including much clay-like mineral matter. The class A bituminous coals

contained mostly quartz-like or alumino-silicate minerals although the western coal particularly showed many pyrite particles. Certain differences between the microstructures of the coal macerals themselves could also be seen in the bituminous coals. The sub-bituminous coal proved very difficult to prepare and the time spent in the ion-thinning machine may indeed have produced a semi-char. However the subsequent examination of the sample showed calcium to have wetted the coal and spread evenly over the coal grains and this may be evidence of the ability of calcium to effectively wet the coal and produce additional reaction sites to enhance gasification rates. This shows that careful preparation of specimens is very important and in this respect the use of ion-beam thinners must be made with some care. If possible modern machines capable of high thinning rates and with liquid nitrogen cooled cold stages should be used to avoid artifacts introduced into the final microstructure.

Acknowledgement

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TABLE 1

PROXIMATE ANALYSES

Coal Type	Source	% c daf	Fixed c (a)	Ash	Volatile Matter	Moisture	BTU (a)
High-ash Anthracite	Pennsylvania	88.93	52.14	40.57	6.36	1.94	7840
Low-ash Anthracite	Wilkes-Barre, Pensylvania	89.74	94.31	1.67	4.02	2.93	13701
High Volatile Class A (Eastern)	West Virginia	60.78	58.76	3.28	37.36	1.46	14453
High Volatile Class A (Western)	Geneva Mine East Carbon, Utah	56.28	51.52	8.22	38.96	2.67	13384
Sub-bituminous (Western)	Dave Johnston Mine Clenrock, Wyoming	48.53	44.33	6.26	34.02	27.65	11376

(a) Dry basis

TABLE 2

ULTIMATE ANALYSES (as received)

Coal Type	Source	Moisture	Carbon	Hydrogen	Nitrogen	Chlorine	Sulphur	Ash	Oxygen (difference)
High-ash anthracite	Pennsylvania	1.94	51.05	1.17	0.9	0.01	0.9	40.57	3.46
Low-ash Anthracite	Wilkes-Barre, Pennsylvania	2.93	85.65	2.05	0.63	0.00	0.73	1.62	6.39
High Volatile Class A (Eastern)	West Virginia	1.46	80.73	5.3	1.22	0.02	1.01	3.28	6.98
High Volatile Class A (Western)	Geneva Mine, East Carbon, Utah	2.67	73.16	4.98	1.5	0.02	0.86	8.22	8.59
Sub-bituminous (Western)	Dave Johnston Mine, Glenrock, Wyoming	27.65	48.17	3.56	0.6	0.00	0.54	6.26	13.22

Figure Captions

FIGURE 1 Reflected light micrographs of high ash (a) and low ash (b) anthracites.

FIGURE 2 Transmission electron micrograph of high-ash anthracite showing a titanium bearing particle at A and a partially degraded clay particle (B and C).

FIGURE 3 Transmission electron micrograph showing the damage caused to a clay particle by the finely focussed beam of the STEM unit.

FIGURE 4 Transmission electron micrograph of aragonite ($CaCo_3$) particle in low-ash anthracite.

FIGURE 5 Transmission electron micrograph of the typical coal microstructure seen in the anthracites (sample thinned from $\simeq$ 50μm).

FIGURE 6 Reflected light micrograph of the high volatile class A bituminous (Eastern) coal.

FIGURE 7 Montage of optical and electron micrographs showing the preparation method used to accurately characterize the coals and correlate both optical and electron-optical micrographs. a) Optical micrograph showing vitrinite (c) and fusinite (A) bands. b) The same areas as a) mounted in a folding grid. c) The same area as a) and b) viewed in transmitted light. d) The area after ion beam thinning. e) Low magnification electron micrograph of the sample. f) Higher magnification electron micrograph showing more image detail.

FIGURE 8 Electron micrographs showing microstructures and particles associated with the different macerals in the coal. a) Particles of Kaolinite in the fusinite band. b) Microstructure of the vitrinite band. c) Microstructure and particles from a tellocollinite region.

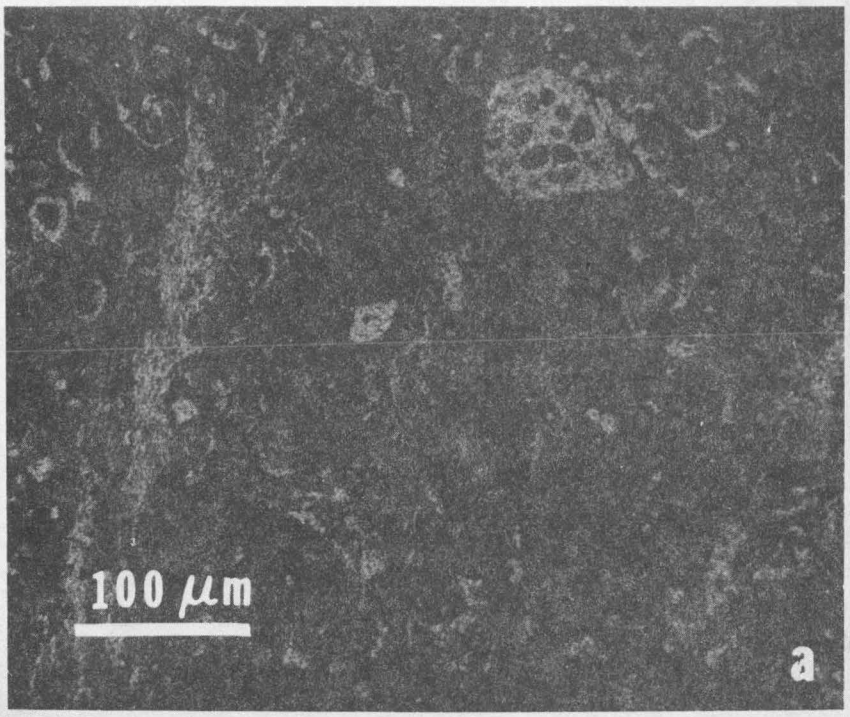
FIGURE 9 Electron micrographs from the high volatile class A (Western) coal showing silica particles (A) and alumino-silicate particles (B).

FIGURE 10 Electron micrograph of pyrite in coal.

FIGURE 11 Optical micrograph of western sub-bituminous coal.

FIGURE 12 Electron micrograph of the sub-bituminous coal after ion-thinning showing a more rounded morphology possibly indicating a semi-char. (Specimen ion-thinned from $\simeq$ 50μm.)

FIGURE 13 Elemental distribution in the sub-bituminous coal specimen. a) STEM micrograph of area. b) X-ray analysis of the whole area. c) Silicon map. d) Titanium map. e) Sulphur map. f) Calcium map.



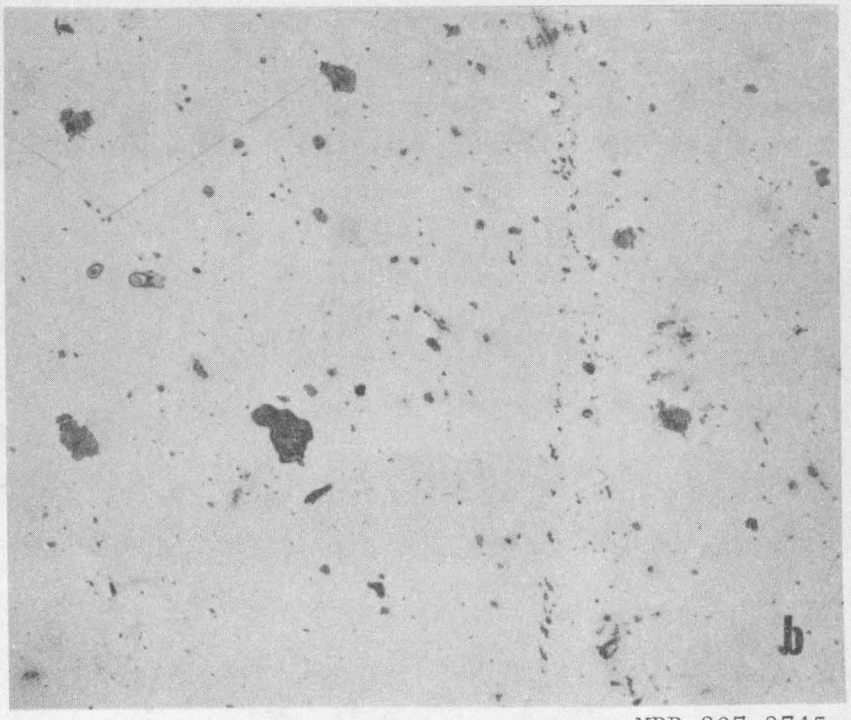


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Figure 1 Reflected light micrographs of high ash (a) and low ash (b) anthracites.

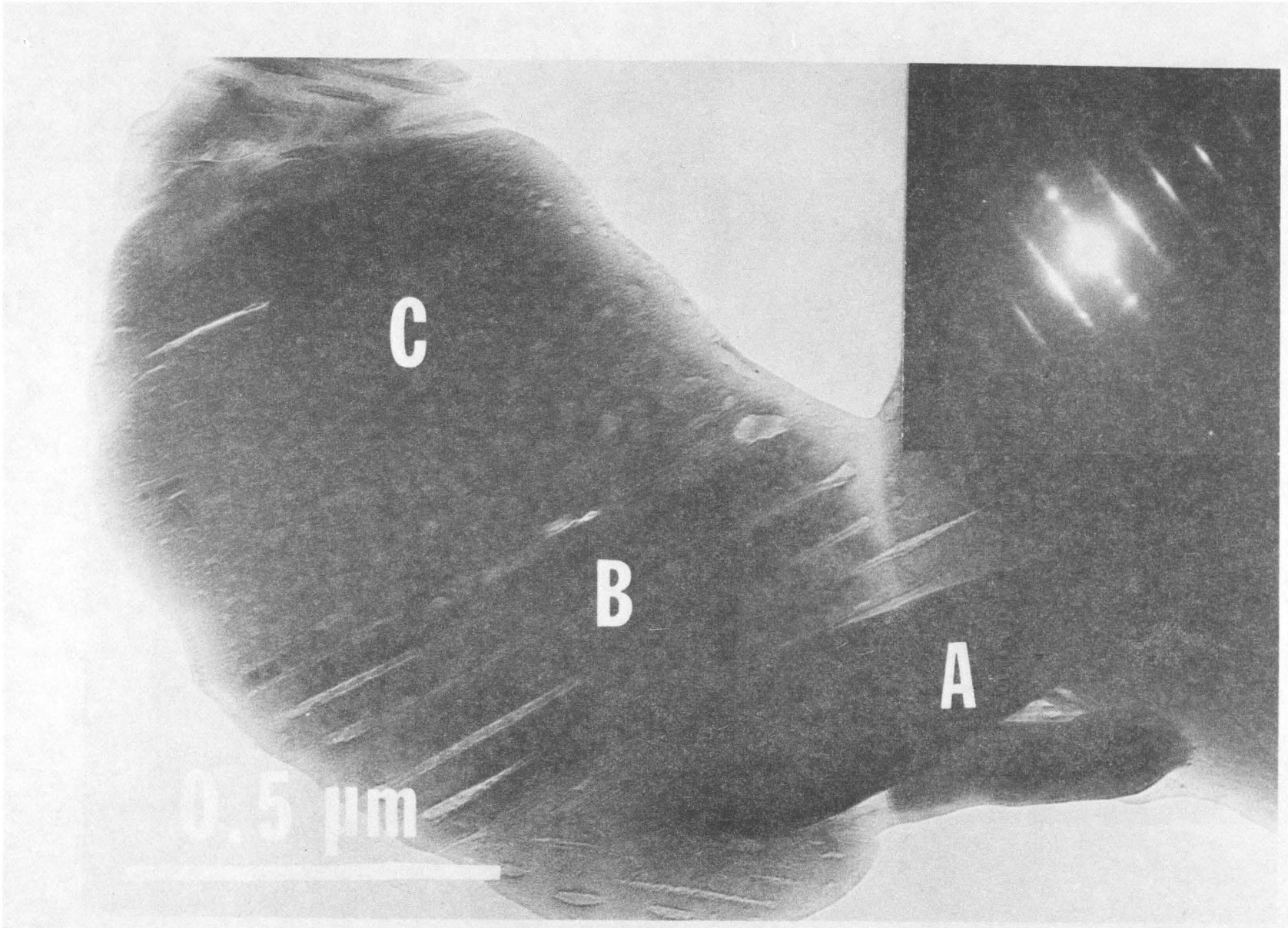

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Figure 2 Transmission electron micrograph of high-ash anthracite showing a titanium bearing particle at A and a partially degraded clay particle (B and C).

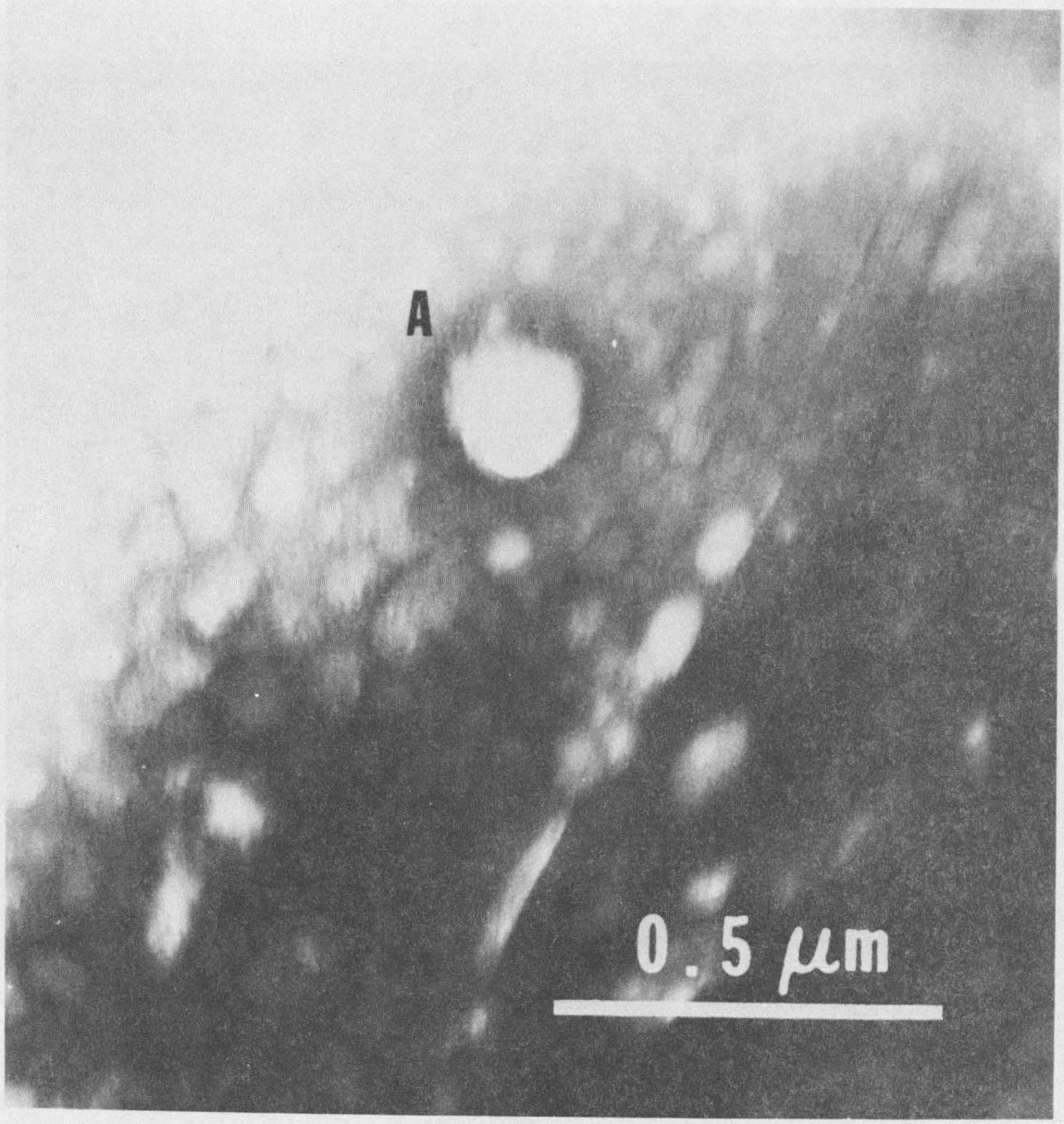

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Figure 3 Transmission electron micrograph showing the damage caused to a clay particle by the finely focussed beam of the STEM unit.

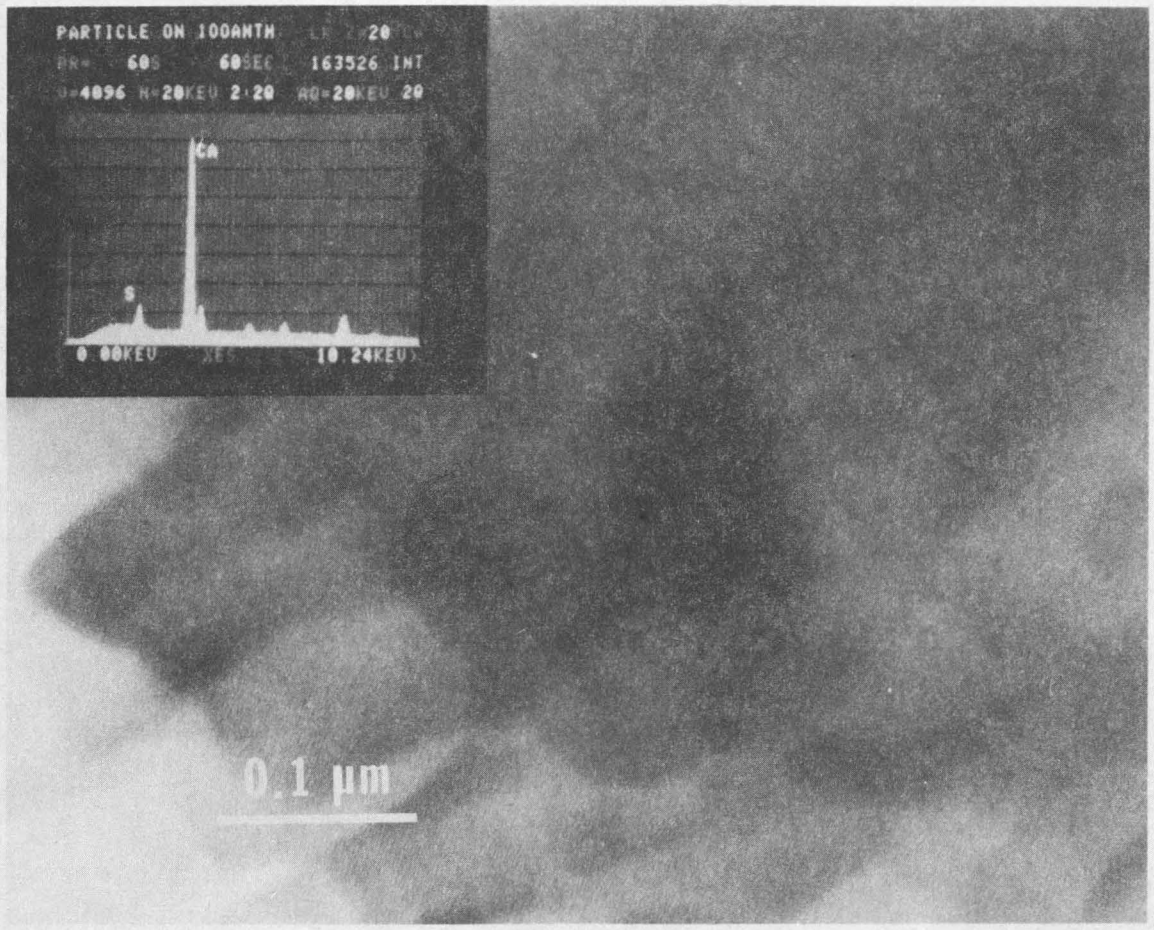

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Figure 4 Transmission electron micrograph of aragonite ($CaCO_3$) particle in low-ash anthracite.

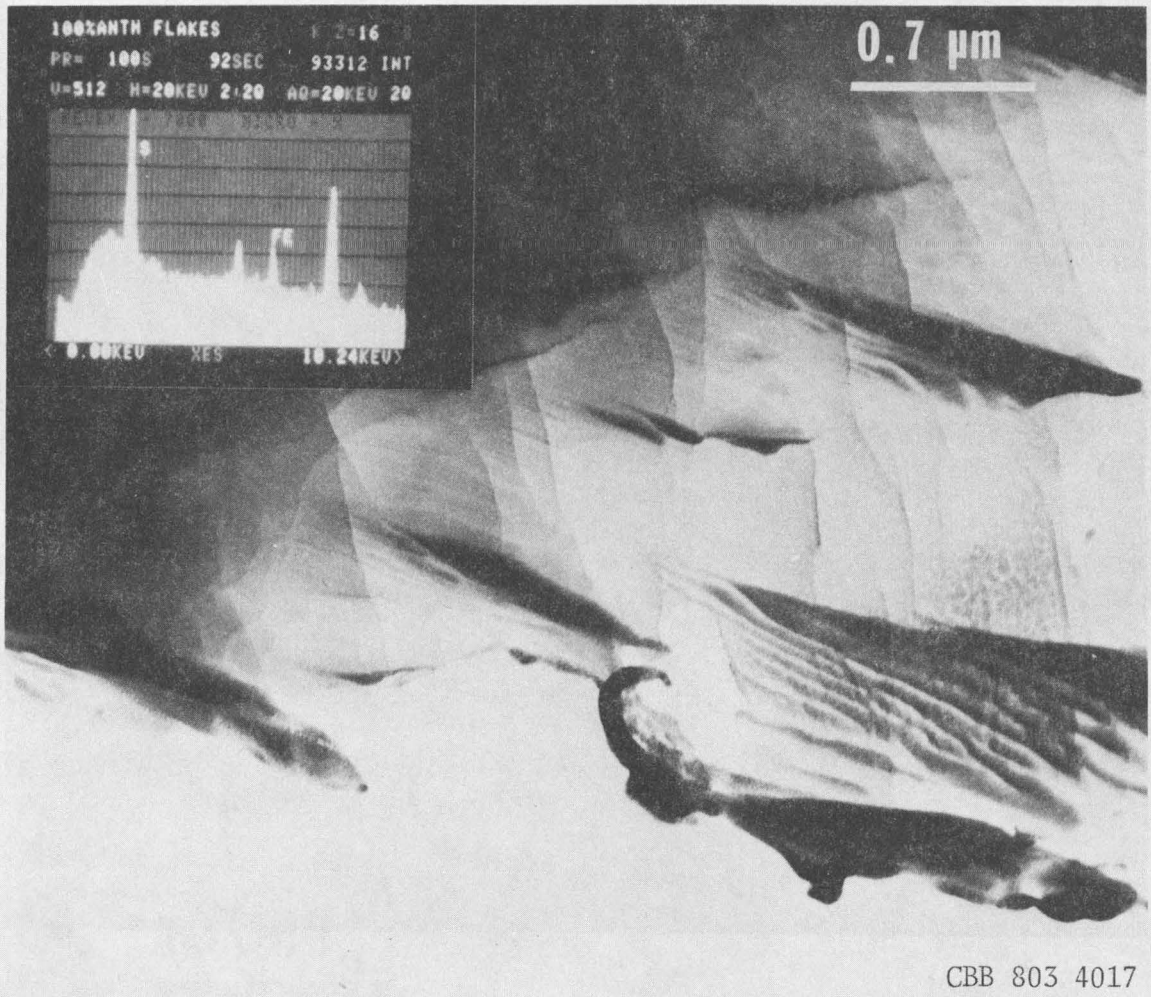

Figure 5 Transmission electron micrograph of the typical coal microstructure seen in the anthracites (sample thinned from ≃ 50μm).

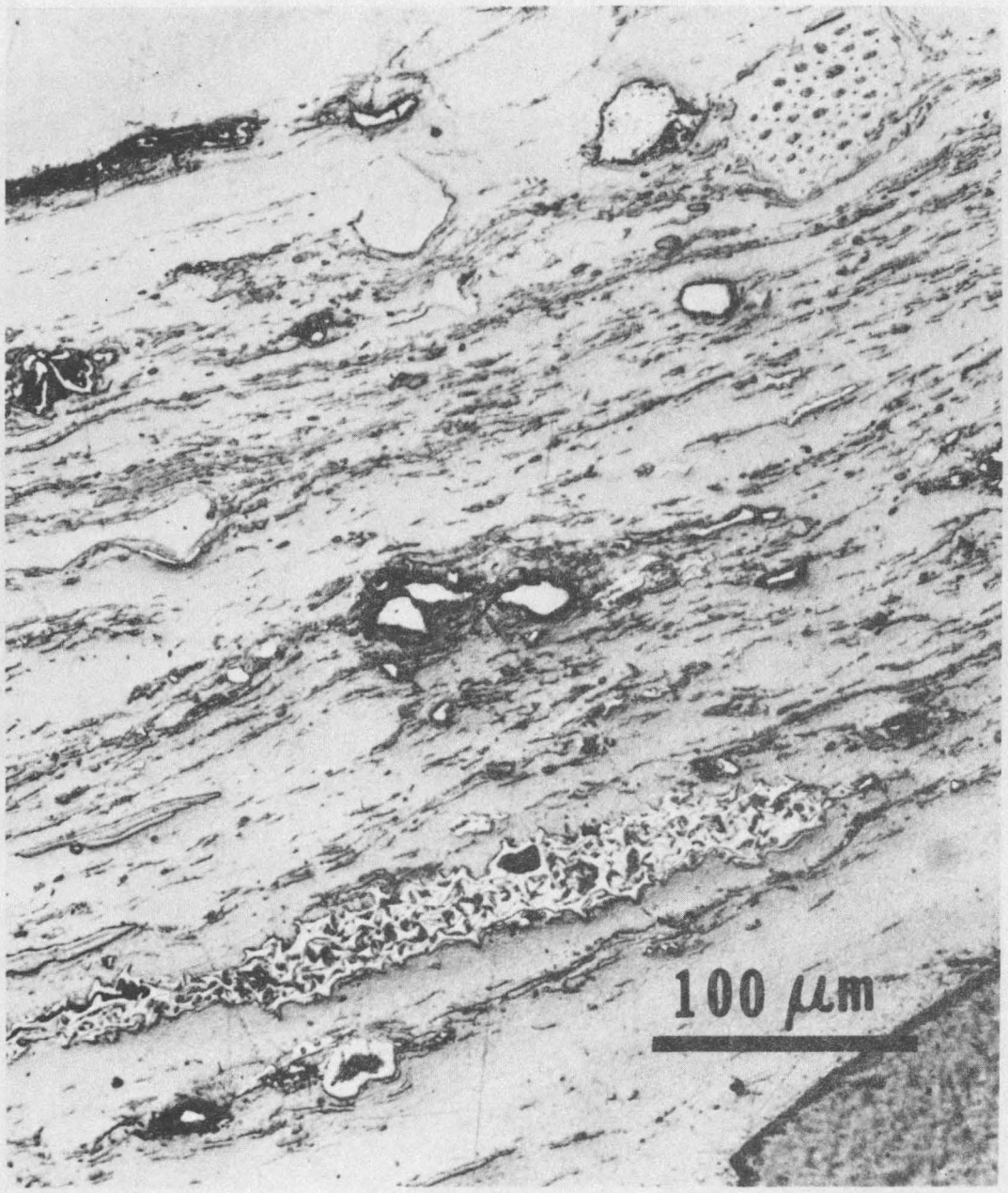

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Figure 6 Reflected light micrograph of the high volatile class A bituminous (Eastern) coal.

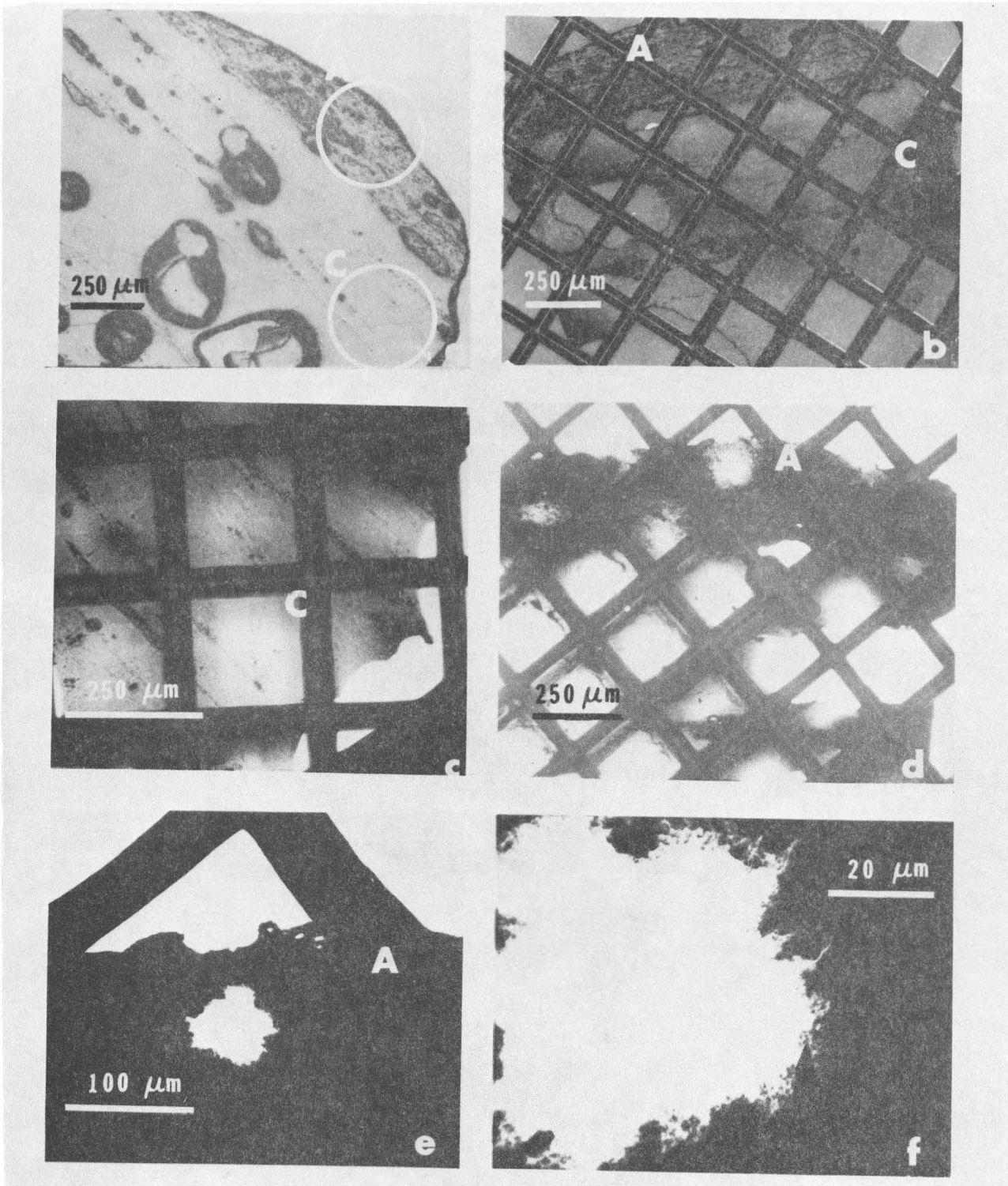

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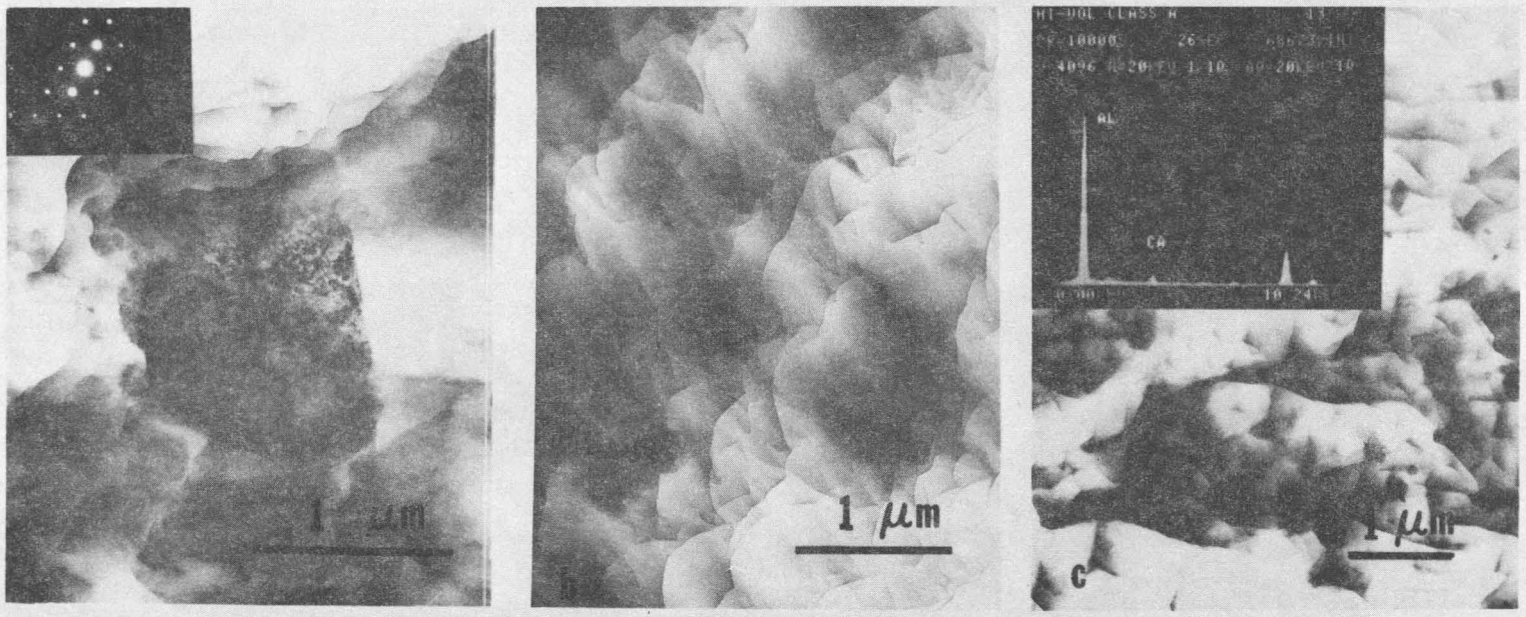
Figure 7 Montage of optical and electron micrographs showing the preparation method used to accurately characterize the coals and correlate both optical and electron-optical micrographs. a) Optical micrograph showing vitrinite (c) and fusinite (A) bands. b) The same areas as a) mounted in a folding grid. c) The same area as a) and b) viewed in transmitted light. d) The area after ion beam thinning. e) Low magnification electron micrograph of the sample. f) Higher magnification electron micrograph showing more image detail.



XBB 807 8895

Figure 8 Electron micrographs showing microstructures and particles associated with the different macerals in the coal. a) Particles of Kaolinite in the fusinite band. b) Microstructure of the vitrinite band. c) Microstructure and particles from a tellocollinite region.

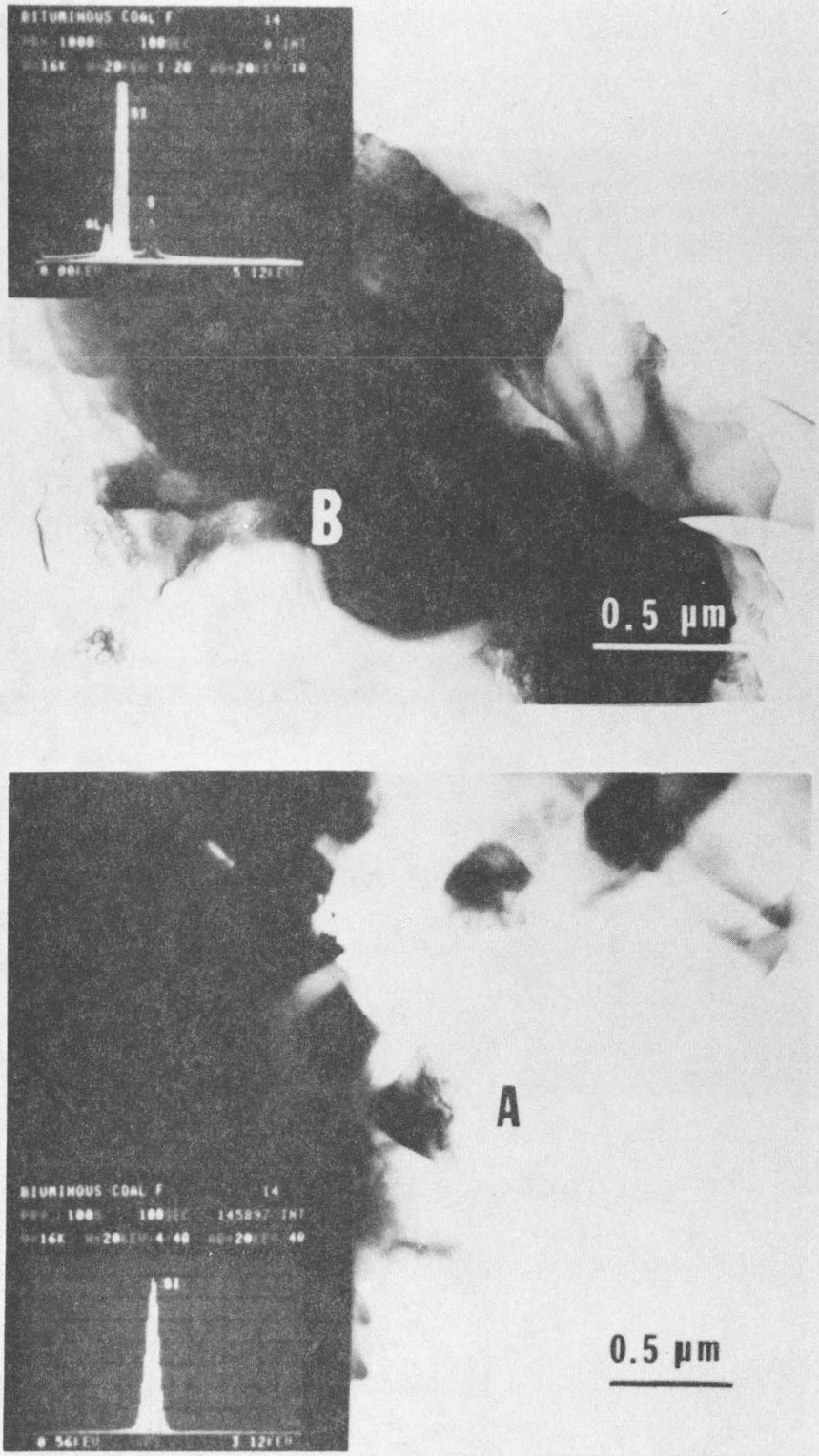


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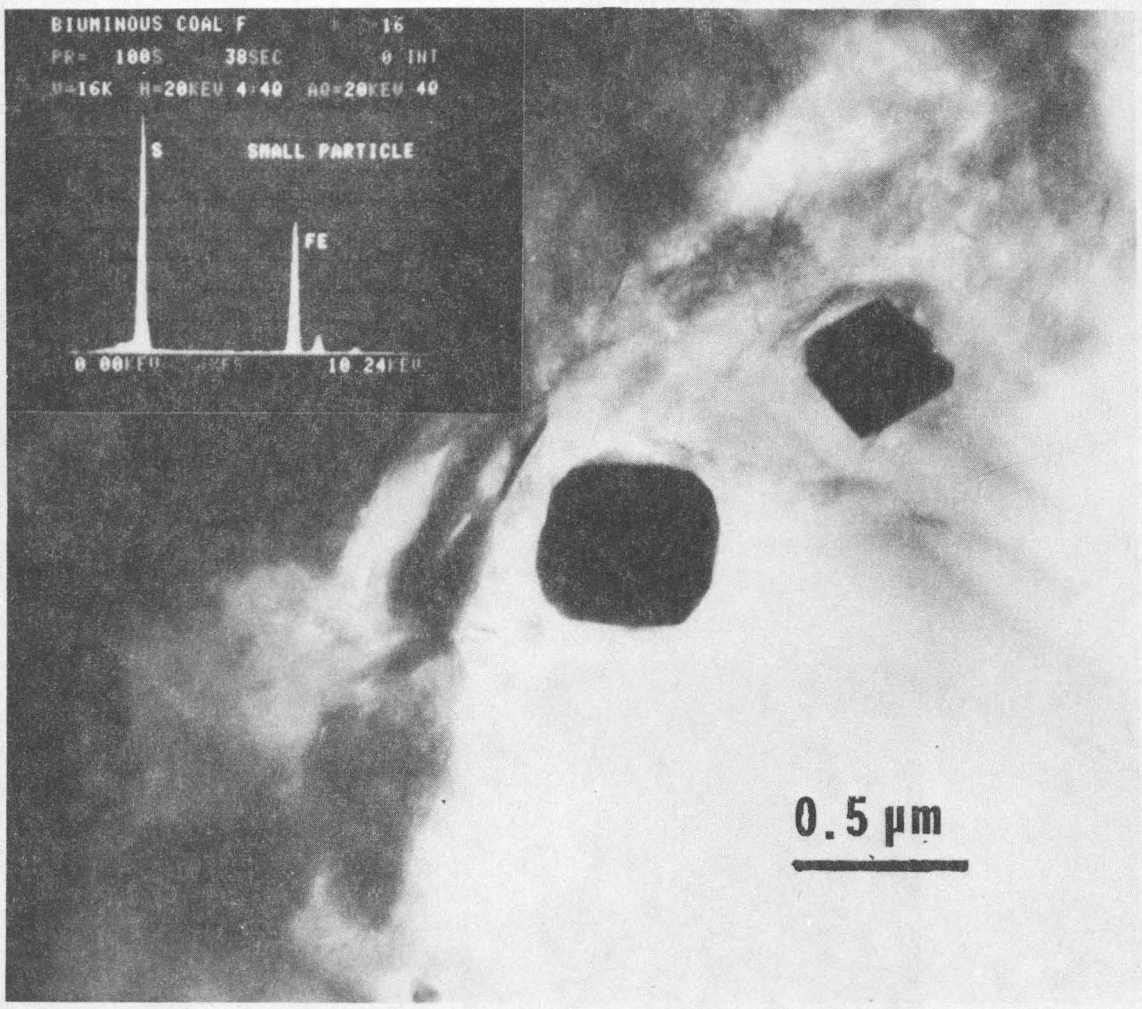
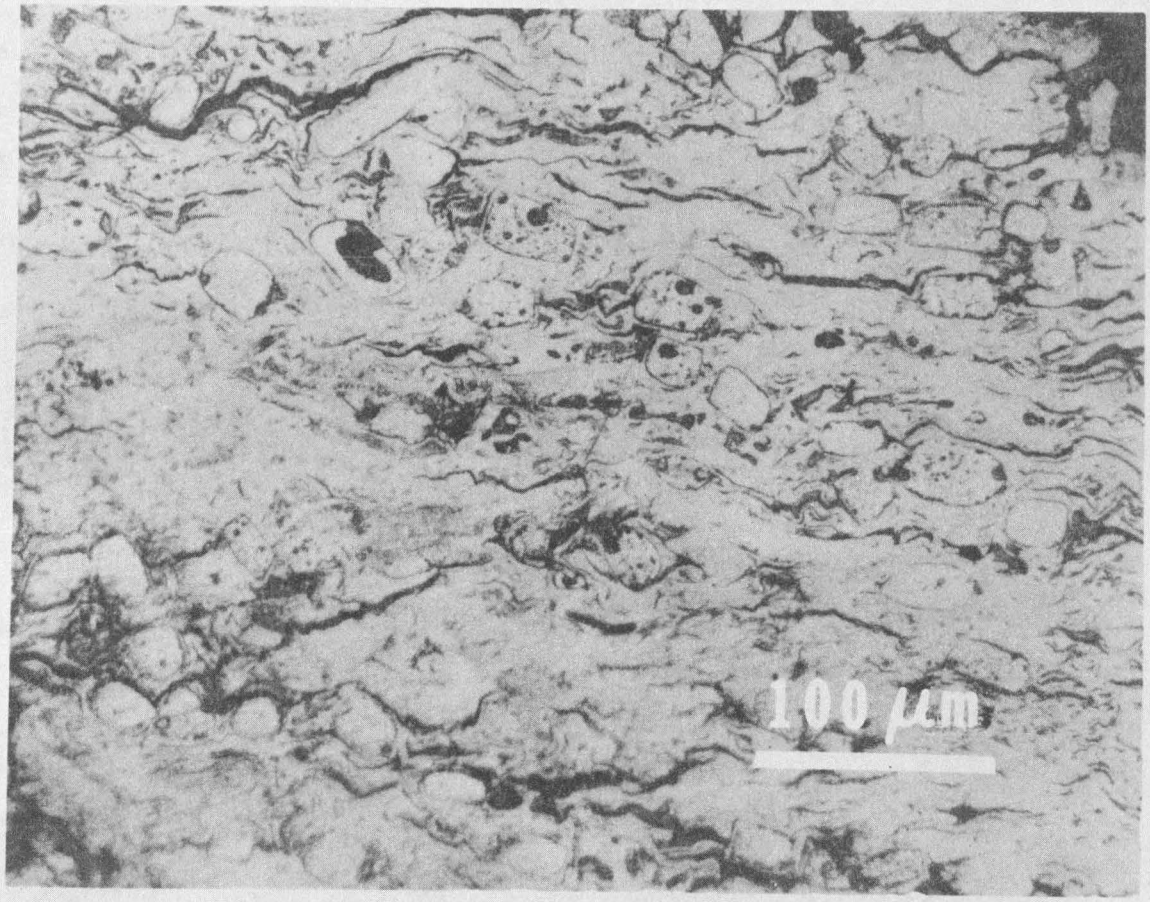
Figure 9 Electron micrographs from the high volatile class A (Western) coal showing silica particles (A) and alumino-silicate particles (B)



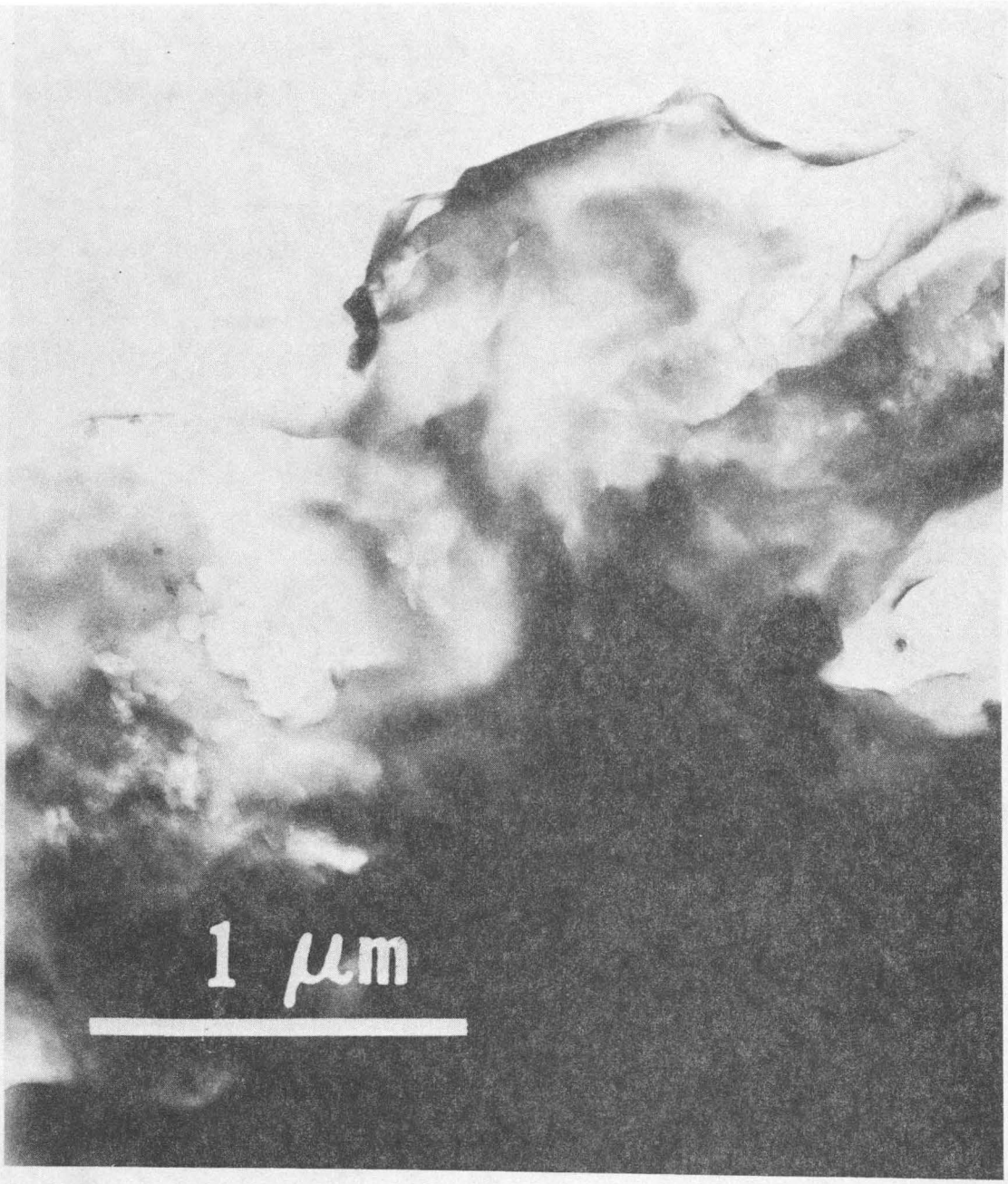
CBB 803 4013

Figure 10 Electron micrograph of pyrite in coal.



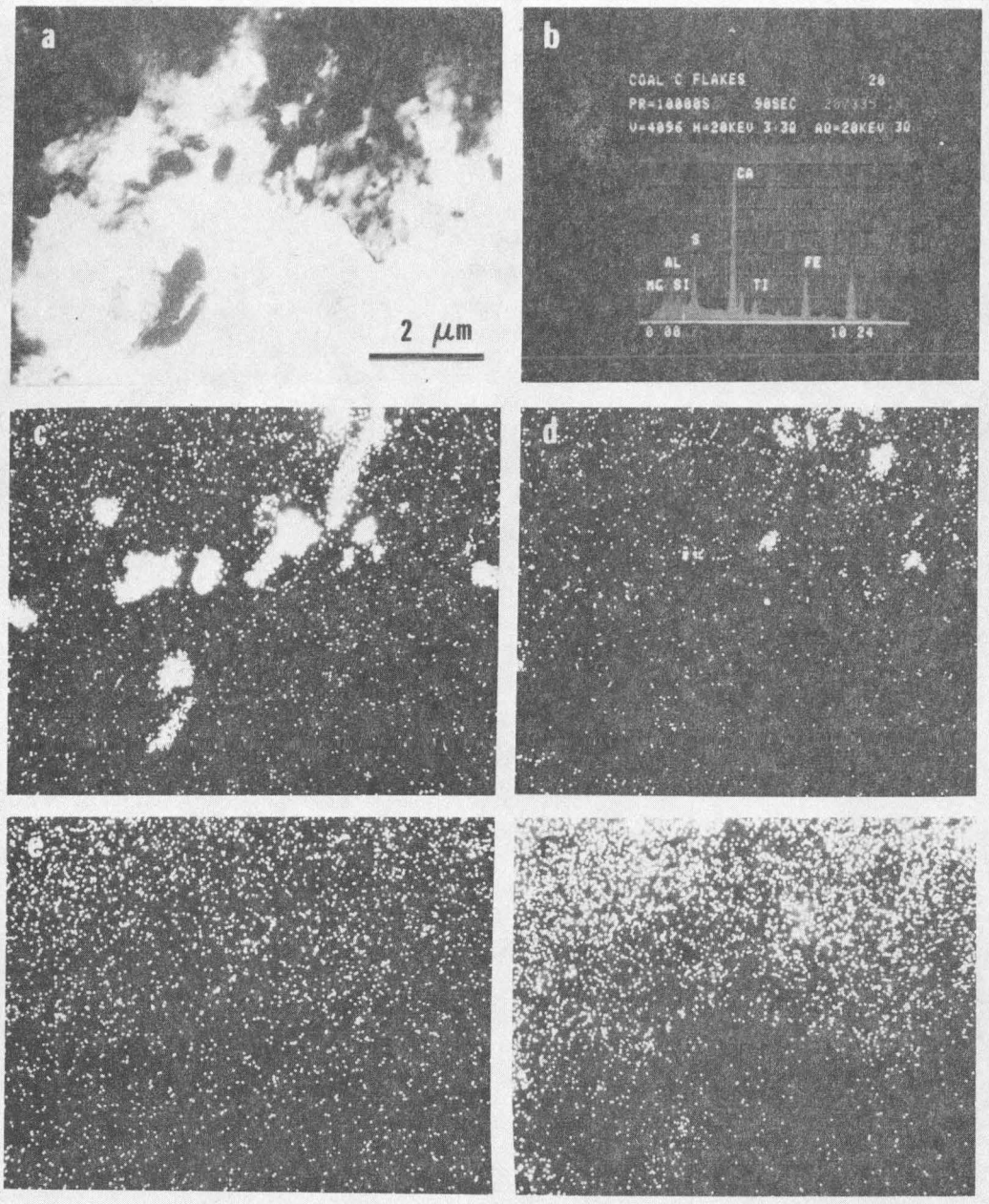
XBB 807 8744

Figure 11 Optical micrograph of western sub-bituminous coal.



XBB 807 8743

Figure 12 Electron micrograph of the sub-bituminous coal after ion-thinning showing a more rounded morphology possibly indicating a semi-char. (Specimen ion-thinned from ≃ 50μm.)



XBB 807 8747

Figure 13 Elemental distribution in the sub-bituminous coal specimen. a) STEM micrograph of area. b) X-ray analysis of the whole area. c) Silicon map. d) Titanium map. e) Sulphur map. f) Calcium map.

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