

THE STUDY OF COAL BY A SCANNING ELECTRON MICROSCOPE AND ELECTRON PROBE

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The object of this study of selected coal samples using a Scanning Electron Microscope (SEM) and Electron Probe (EP) was to ascertain whether coal macerals, normally observed by reflected light in an optical microscope, could be identified in the emission images of backscattered secondary electrons. As it was difficult to characterize finely disseminated mineral matter in coal macerals using an optical microscope, it was also important to explore the possibility of evaluating the distribution and chemical character of the mineral matter in the maceral types using the x-ray electron probe capability of the Philips SEM.

To compare the optical and SEM microscopes it was essential to select coal samples containing a wide variety of maceral types and to polish the surface to be examined as flat as possible. This was essential to prevent surface relief from contributing artifacts to the secondary electron emission. Everhart (1) has shown that changing the surface inclination to the beam by only a few degrees produces an appreciable change in the number of secondary electrons produced.

Kimoto and Russ (2) point out that the resolution of an image with a SEM is limited to the size of the area emitting photons or electrons at any moment. When the electron probe hits the specimen, scattering causes the probe to spread so that the final volume of electron capture is roughly teardrop-shaped as shown in Figure 1. Secondary electrons, with energies up to about 50 eV, are produced throughout this volume; however they are reabsorbed after travelling only about 100\AA , so it is only the volume within 100\AA or less of the surface that emits secondary electrons that can be detected. This volume is only a few tens of angstroms larger than the diameter of the incident probe which has not had much chance to spread. Hence the secondary electron image provides the highest resolution.

Backscattered electrons come from a greater depth, and hence from a point where the probe has spread further, so that the resolution of the back-scattered image is poorer than the secondary electron image. Elements with high atomic numbers backscatter a greater fraction of incident electrons than ones with low atomic numbers.

The photons of x-rays or visible light come from essentially the entire teardrop volume and hence give the poorest resolution.

EXPERIMENTAL

A sample of Moss #3 coal was polished flat and examined with a Leitz Pan Pol Phot Microscope at a magnification of 450 using an air objective. Figure 2 shows a location selected for examination which contained vitrinite, semifusinite, and a large extremely dark wedge-like structure of what appears to be spore material, which could be readily identified by its wedge shape in the various other modes of examination in the scanning electron microscope. A comparison of Figure 2 with Figure 3, which was taken of the same location in the secondary emission mode, showed that dark bands of exinite (E) at the top of Figure 2 may be associated with similar dark bands at the top of Figure 3. The broad band of semifusinite (SF) in Figure 2 corresponds with a lighter region in Figure 3. The dark wedge-like structure of what appears to be spore material in Figure 2 seems to have an outer rim of high electron emission, as shown in Figure 3, with a characteristic dark thread-like structure midway between the two walls of the bright zone. Possibly this structure has a very high electrical resistance due to the high hydrogen content and thus builds up a negative charge that might reflect the electrons from the probe. The significance of this thread-like structure is not clear at present. The dark apparent voids in the semifusinite in Figure 2 do not correspond in shape with sufficient accuracy to be positively matched with the bright areas in the semifusinite in Figure 3.

Figure 4 shows the same location in the backscattered mode. The white areas in Figure 4 in the semifusinite correspond with the white areas in the secondary electron mode in Figure 3. The converse of this statement is not true. The definition is sufficiently sharp to permit the shapes of the white areas in Figures 4 to be accurately matched with those in Figure 3. The areas of high electron emission in the backscattered electron mode are thought to be due to mineral matter. The high electron emission of these particular areas is attributed to the much higher atomic number of the mineral matter as compared with that of the coal substance. It is noted that bright areas in Figure 3 do not necessarily correspond to bright areas in Figure 4. A striking example of this is the particle marked X.

This preliminary assessment was done using a Cambridge Scanning Electron Microscope which at the moment has no facilities for microprobe analysis. In

view of the importance of characterizing the mineral matter in coal, this preliminary investigation was extended using a Philips Model 4500 Electron Probe Analyzer with beam scanner.

A sample of coal from the Tantalus Butte Mine, N.W.T., Canada, was selected for examination for its relatively high concentration of semifusinite-containing mineral matter. This sample was polished flat and a location was selected that was approximately half vitrinite, and half semifusinite. This was done using a Leitz Microscope at a magnification of 300 with a water-immersion lens, Figure 5.

This same location was then examined in the backscattered electron mode and the resulting image, shown in Figure 6, reveals numerous light areas corresponding to the presence of the mineral matter in the semifusinite. On examining this same area with the microprobe analyzer using the first-order Si $K\alpha$ line, the bright areas as shown in Figure 7 indicate the location of the silica. These areas of high silicon concentration are located in the region occupied by the vitrinite. With the Fe $K\alpha$ first-order line, the bright areas correspond to areas of high iron concentration as shown in Figure 8. The area, in which the iron occurs, appears to be largely concentrated in the semifusinite regions of the field. Using the Ca $K\alpha$ line from a mica crystal the distribution of calcium is shown to be concentrated in the semifusinite as may be seen from the location of the bright areas in Figure 9. Similarly, the carbon content was shown to be higher in the semifusinite region than in the vitrinite portion of the field, as may be seen in Figure 10. In this case, the $CK\alpha$ first-order line from a lead stearate crystal was used.

CONCLUSION

1. The macerals (vitrinite, exinite, fusinite, and semifusinite) are visible in the secondary electron image. The indications are that the optical interpretation can be considerably extended by taking into account the differences observed between the secondary electron image and that obtained from back-scattered electrons.
2. The backscattered emission image generally indicates the presence of mineral matter in and between the maceral types.
3. The X-ray electron probe analyzer shows the iron and calcium to be concentrated in the semifusinite and fusinite portion of the field while the silica is concentrated in the vitrinite in the particular coal being studied. This tends to confirm the data recently obtained from washability studies that the high ash content is associated with higher concentrations of fusinite and semifusinite.(3)
4. The carbon content appears to be higher in the fusinite and semifusinite portion of the

portion of the field than in the vitrinite. This would be expected from the existing information on the chemical character of these macerals.

ACKNOWLEDGEMENT

The authors extended their special thanks to the following persons: Dr. E. Smith, Research Scientist and Dr. K.M. Pickwick, Research Scientist, Metal Physics Section of Physical Metallurgical Laboratories, Mines Branch, for taking photographs of Cambridge Scanning Electron Microscopy and for constructive suggestions and discussions; Mr. S.E. Nixon, technician, Fuels Research Centre, for his help in preparing sample.

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BNN:DSM:KMB:gdb

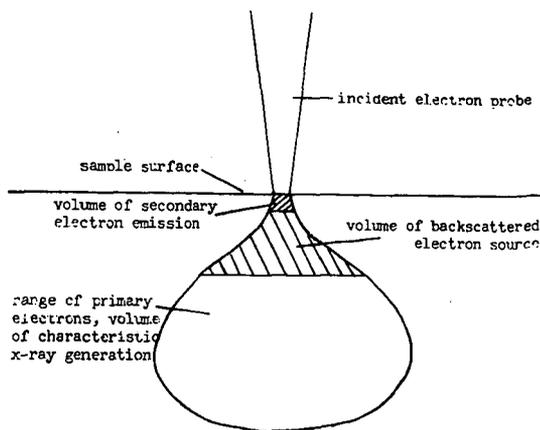


FIG. 1 Penetration of incident electron probe into sample.



FIG. 2 Optical micrographs of the macerals air objective reflected light X450.

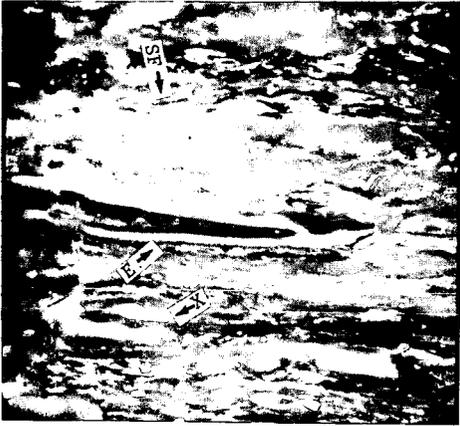


FIG. 3 Scanning micrograph of the macerals of the same location as in Fig. 2 X400 approximately.



FIG. 4 Back-scattered electron micrograph of the same location as in Fig. 2 approximately X400.



FIG. 5 Optical micrograph of the macerals of coal from Tantalus Butte Mine; water immersion reflected light X300.

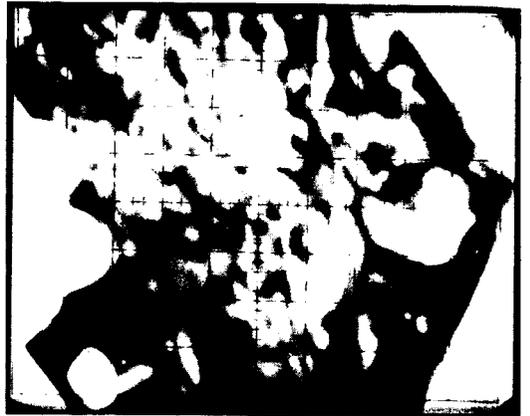


FIG. 6 Back-scattered electron micrograph of the same location as in Fig. 5, approximately X500.

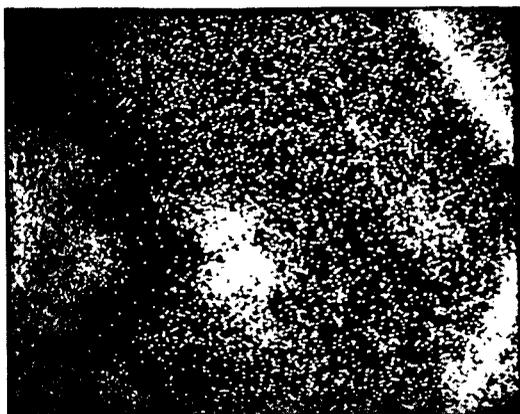


FIG. 7 Microprobe photograph of the silica
X500



FIG. 8 Microprobe photograph of the iron
X500

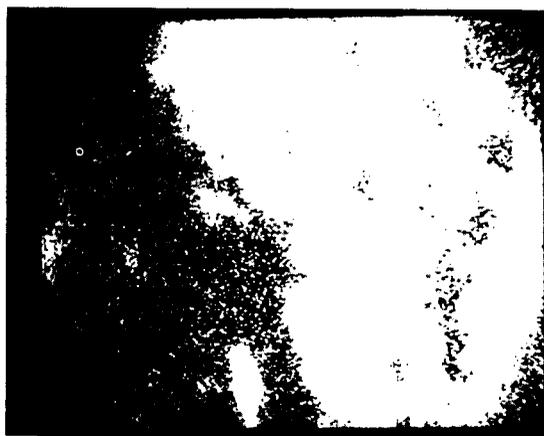


FIG. 9 Microprobe photograph of the calcium
X500



FIG. 10 Microprobe photograph of the
carbon X500