STRUCTURAL PARAMETERS OF COALS FROM 002 LATTICE FRINGE IMAGES USING HRTEM

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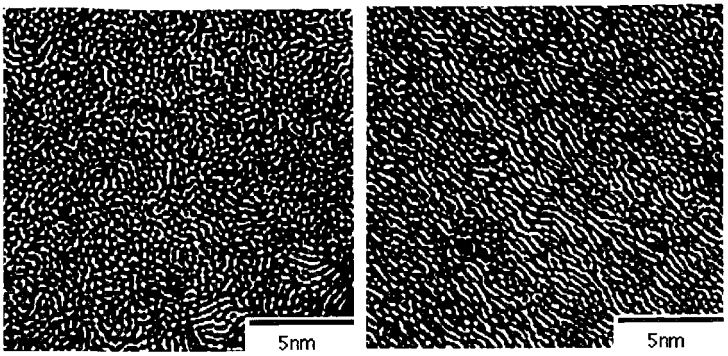
INTRODUCTION

Over the years several studies have been done using different techniques to get the insight into the coal structure. All these studies converge on one understanding that coal is heterogeneous in nature and made up of very small aromatic layers more or less randomly orientated. The layer size and stacking number increases with the rank of coal. Direct evidence about the arrangement of these aromatic layers, obtained by the electron microscope lattice imaging, is obviously an attractive complementary technique. Not much literature have been available on TEM observation of coals as such and most of the studies report about the heat treated coals. McCartney and Ergun [1] used electron microscopy to determine crystalline dimensions of meta-anthracite coal. Another attempt was made by Evans et al. [2] who observed anthracite coal and heat-treated coals. They found the coal samples to be completely amorphous and no evidence of any ordered layers. Millward [3] also reported that when coals were observed by electron microscope, no fringes which could be evidence of layered molecules was found. He highlighted the problems associated with instrument and irradiation damages which make it difficult to observe raw coals. Oberlin [4] developed dark field image technique to observe the coal structure. The electron microscope operation and theoretical consideration of image formation are now fully understood. With recent advances in electron microscope instrument like anti-contamination device which successfully preserve the specimen from irradiation damages during high-resolution observation, it is possible to image even the highly electron beam-sensitive materials like coals.

In this article we are presenting the structure of three different coals as observed by HRTEM. We also attempted to obtain stack distribution using image analysis technique developed by us and described elsewhere [5].

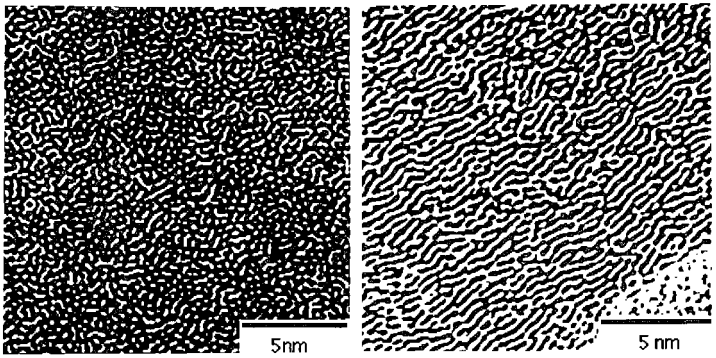
EXPERIMENTAL

Argonne Premium Coal Samples, Pocahontas no. 3 (POC) , Illinois no. 6 (IL) and Beulah-Zap (BZ), were selected for the investigation. The coal samples, ground in ethanol and sprayed on a copper microgrid, were observed under a TEM (JEOL-2010) operating at 200 kV for high resolution images. The transmission electron microscope was equipped with computerized imaging system and EDS for elemental analysis. To get a general view of the coal samples, nearly 7 to 8



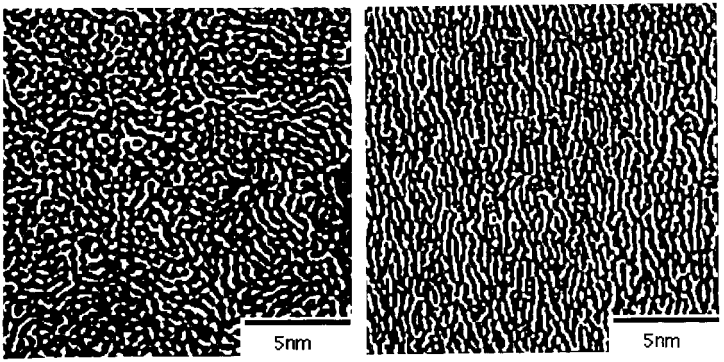
(a) (b)

Figure 1 HRTEM images of Beulah-Zap coal: (a) amorphous structure and (b) ordered structure.



(a) (b)

Figure 2 HRTEM images of Illinois no.6 coal: (a) amorphous structure and (b) ordered structure.



(a) (b)

Figure 3 HRTEM images of Pocahontas no.3 coal: (a) amorphous structure and (b) ordered structure.

different spots were observed. A quantitative analysis was done to evaluate stack distribution using our newly developed image analysis technique [5].

OBSERVATION OF COALS USING HRTEM

It is necessary first to ascertain that the TEM micrographs presented here are those of coals. In all cases, first a sub-micron size particle was selected for observation at a very low magnification and its diffraction pattern was taken and elemental analysis was carried out. The elemental analysis showed signatures of all the elements present in the coals as mineral matter which was also confirmed by the sharp spots observed in the SAD patterns. After confirming that this sub-micron size particle is coal, very thin edges of this particle were observed at a high magnification(x 500k). To prevent the sample from irradiation damages, the current density on the screen was kept at less than 1 pa/cm^2. At this illumination almost nothing can be observed on the fluorescent screen. However, the structure can be observed on the TV screen by increasing the aperture and sensitivity of the camera. By this approach we considerably reduced the number of electrons hitting the sample.

RESULTS AND DISCUSSION

In Figures 1 to 3, the HRTEM images from two different spots for BZ, IL and POC coals are shown. All three coals show the presence of both amorphous and ordered structure. Even in ordered structures, layers are not plane and show lack of mutual orientation. It is known that coals contain small condensed aromatic units. These units show a parallel stacking but lack orientation and planarity because of the presence of hetero-atoms or hydroaromatic portions. These fringes or layers observed from HRTEM images are in accordance with the above understanding, and hence these layers are not graphite-like layers. Figure 4 shows a typical example of extracted images from HRTEM images using the filtration technique [5] for quantitative analysis. The extracted images from five different spots were subjected to image analysis algorithm [5] to evaluate stack distribution and the results are shown in Figure 5. The distributions show that the fraction of single layers or amorphous part is higher in BZ coal than in POC coal and the fraction of highly stacked group is relatively large with POC. However, the number of photographs taken in this study are too less to discuss further on the difference among the three coals. Figures 6a-c show the presence of mineral matter in the three coals. BZ and IL show very clear lattice fringes from the mineral matter. To make the fringes more clear, we separeted these from the organic part by drawing a white line. In Figure 6a, these are enclosed by the white line while in Figure 6b, these are towards left of the white line. In POC coal, mineral matter is present as nearly circular dark spots. Figure 6d shows an onion-like structure similar to fullerene as suggested by Fang et al [6]. This was observed only in POC coal and only in one spot.

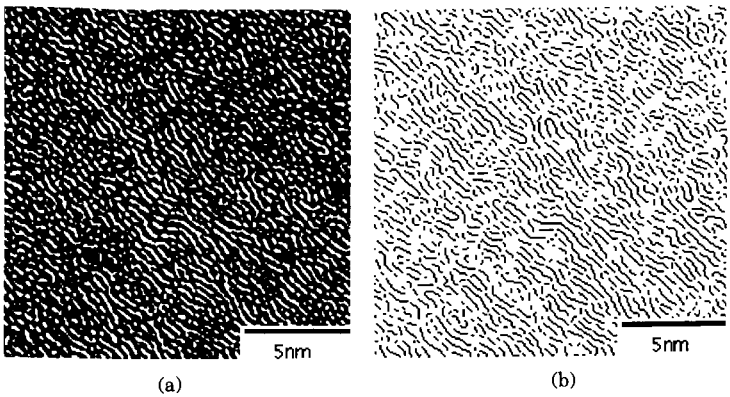


Figure 4: (a) HRTEM image and (b) corresponding extracted image.

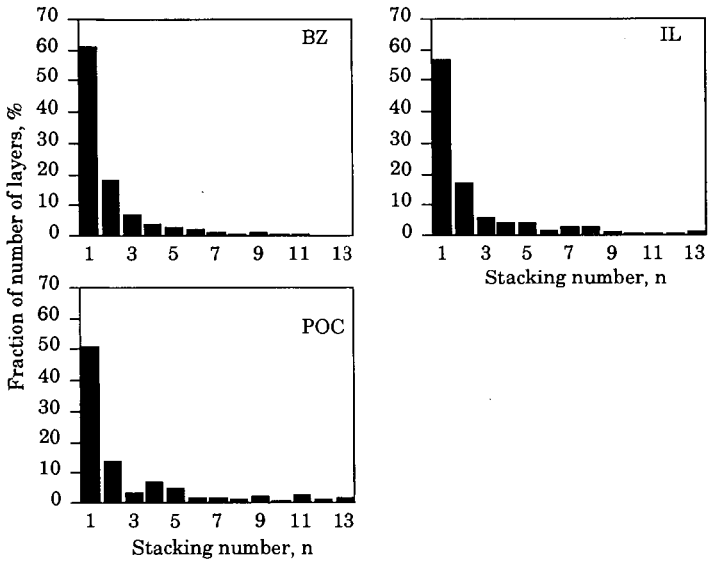


Figure 5 Fraction of number of layers occuring in *n* layers.

CONCLUSIONS

The HRTEM technique is powerful tool for extracting microstructure information of coals. Both amorphous and ordered structures were observed. Direct evidence of lattice fringes which can be ascribed to layers was found.

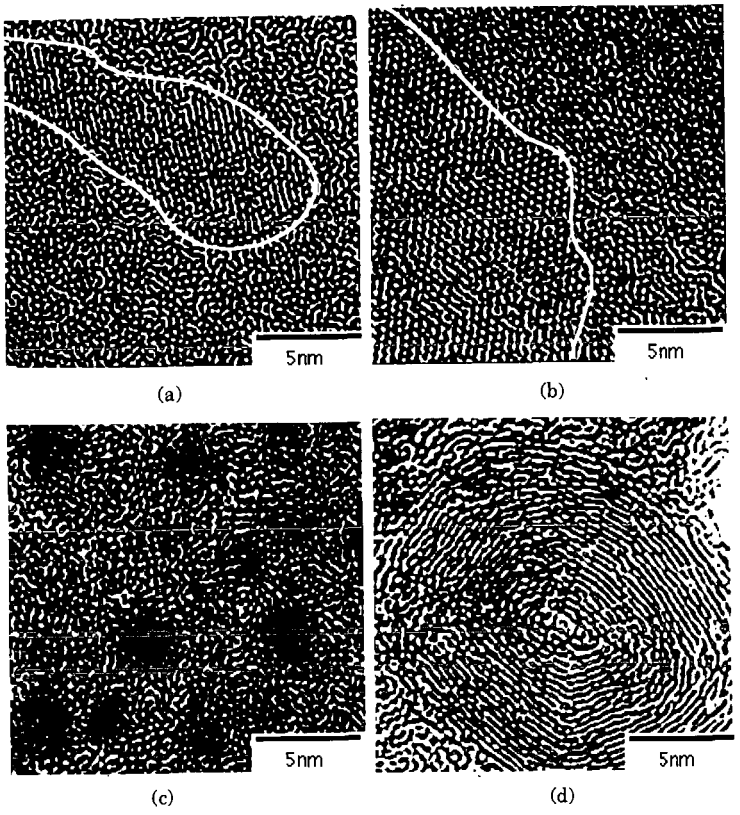


Figure 6 HRTEM images of: (a) mineral matter in Beulah-Zap coal, (b) mineral matter in Illinois no.6 coal, (c) mineral matter in Pocahontas no. 3 coal, and (d) onion-like structure in Pocahontas no. 3.

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