

DIGITAL IMAGE ANALYSIS OF OPTICAL TEXTURE AND POROSITY OF PETROLEUM COKES

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INTRODUCTION

The microstructure of coke particles can be considered to consist of their optical texture and porosity¹. Optical texture refers to the appearance of the surface under a polarized-light microscope. The size and shape of isochromatic areas observed on polished surfaces can be used to identify different types of texture². Characterization of the optical texture is very difficult, since the optical domains in petroleum cokes are often connected and tortuous. Several image analysis techniques have been used to determine the size, shape, and orientation of the optical domains in calcined petroleum cokes^{3,6}. For texture analysis of calcined cokes, the pores are either ignored⁴, or masked out by image processing⁵. In the present study, we propose a digital analysis method to simultaneously characterize the texture and porosity of the calcined cokes. An image analysis method was developed to extract the boundaries of optical domains and pores in petroleum cokes. For semi-cokes, only the boundaries of optical domains were extracted to characterize the optical texture. For calcined cokes, the boundaries of the optical domains and the pores were used to characterize the microstructure of the samples. Feature indices were defined and calculated based on the shape and orientation of the extracted boundaries for the optical domains and pores. Before image analysis, thermal expansion measurements were carried out on the same calcined coke particles to see if there is any relationship between the feature indices and the thermal expansion coefficients (CTE) of calcined coke samples. The CTE is one of the critically important properties of the calcined needle cokes used for manufacturing graphite electrodes for electric-arc furnaces⁷.

IMAGE PROCESSING TECHNIQUES

It is well known that there are no distinct gray levels which separate two different isochromatic regions in a polarized-light image of an anisotropic carbon³. Therefore, one cannot readily identify the boundaries of these isochromatic regions. It is, however, possible to use a local derivative operator to extract the boundaries of different objects in a given image. For example, the Robert's cross operator⁸ acts on a 3x3 pixel region of an image (the z 's are gray levels), using the two arrays shown below to obtain a new gray level at point z_5 as $(z_1+z_3)-(z_7+z_9)+|(z_3+z_9)-(z_1+z_7)|$.

z_1	z_2	z_3
z_4	z_5	z_6
z_7	z_8	z_9

1	0	1
0	0	0
-1	0	-1

-1	0	1
0	0	0
-1	0	1

These operators are adopted to detect the edges and to draw the boundaries of the objects using horizontal and vertical gradients. Figure 1 shows different images of a domain structure in a semi-coke sample, including a gray level image (a), boundary image (b), and a binary boundary image (c), created by image processing techniques. As seen Figure 1(a), interpreting optical domains as objects would give complex object shapes. In many cases, an optical domain is not an enclosed object. Therefore, instead of analyzing the complex shapes of optical domains directly, one can analyze the boundaries of these optical domains. The boundary image shown in Figure 1(b) was created by the Robert's cross operator. Although the boundary image does not exactly reflect the shape and size of the optical domains it preserve many features of the optical domains from which it was derived.

After a boundary image is created, it is necessary to expand the gray levels of boundary image to the whole gray scale (0=black, 245=white) by using the stretching technique⁸, since the gray levels of the boundary image occur only at the dark end of the gray scale. The stretching technique is used to increase the contrast of the image. Figure 2 shows the linear operation of contrast transformations. After stretching the gray levels of the boundary image shown in Figure 1(b), a threshold of gray level 67 was chosen to obtain the binary image shown in Figure 1(c). Only binary images can be used for feature analysis.

EXPERIMENTAL

Both commercial calcined needle coke samples and semi-coke samples produced in the laboratory were used for image analysis. Thirteen semi-coke samples were prepared by carbonizing commercial coker feedstocks in closed tubing reactors at 500°C for 3h. Before carbonization, 4 g of each sample was weighed in an aluminum foil tube and then placed in the stainless steel reactor. After carbonization, the resultant lump coke was mounted in an epoxy resin

pellet longitudinally. Polished pellets of semi-coke samples were placed on the microscope stage such that the long axis of the coke samples was parallel to the x-axis of the stage.

Before the calcined coke particles were examined under the microscope, thermal expansion measurements were performed in an ORTON 1600D dilatometer. Nine particles were picked from four calcined coke samples and cut into cylinders of approximately 2.50 cm in length and 0.75 - 1.00 cm in diameter. The particles were cut such that their apparent long dimension was parallel to the long axis of the cylinders. For CTE measurements, samples were heated at a rate of 3°C/min in an argon atmosphere and linear changes of samples were recorded from room temperature to 800°C. For comparison with the published literature^{3,5}, the CTE values were calculated for the same temperature range (300-700°C) for all the particles. When three replicate measurements were performed on each sample, the results showed a standard deviation of 1-2%. After CTE measurement, the samples were placed longitudinally in epoxy resin blocks to prepare polished pellets for microscopic examination.

Image acquisition from a polarized-light microscope (Nikon, Microphot-FXA) was carried out via a high resolution video camera and an image analysis system (PGT, IMAGIST)⁹. After the binary image was created from the boundary image as described in the previous section, it was used for feature analysis¹⁰. Usually, thirty images were analyzed for each pellet. Only the features in the size range of 50 μm^2 and 1500 μm^2 were analyzed to exclude large cracks and polishing artifacts from the measurements.

A manual point counting technique was also used to characterize the semi-coke samples to compare the results with those obtained by digital image analysis. An automated microscope stage was used to scan the sample by moving a mask of 1mmx1mm on each pellet⁸.

RESULTS AND DISCUSSION

For feature analysis of the binary boundary image, we used three parameters, longest dimension (LD), breadth(B), and horizontal chord (HC). Using these three parameters, a feature index, called FRI, was defined to reflect the shape and orientation of the boundaries of optical domains. The following equation was used to calculate the feature indices for each pellet:

$$FRI = (1/n) \sum_{j=1}^n \left(\frac{LD_j}{B_j} \times HC_j \right) / m \quad (1)$$

where: m is the number of features in an image; n is the number of images (normally, n=30). Cokes with more anisotropic and well oriented structures should have higher values of feature index. Using a semi-automated point counting data and the assigned factors for four texture classifications, another optical texture index (OTI) was calculated as described before⁸.

Figure 3 shows a plot of feature index (FRI) against optical texture index (OTI) for semi-coke samples. It appears that the boundary analysis of optical domains can represent the characteristics of the optical texture of semi-cokes. Unlike point counting, the boundary analysis method does not require texture identification for each image. Instead of analyzing the complex shape of the optical domains, the method relies on the boundary properties of the texture domains.

Figure 4 shows a gray level image (left) and a binary boundary image (right) of a calcined coke sample with a highly anisotropic needle coke texture. The comparison of the two images shows that the extracted boundaries represent both the texture domains and the pores seen in the gray level image. It can also be seen that there is a close correspondence between the boundaries of the texture domains and the pores. For feature analysis of the boundary image, an area exclusion formula was used to remove the large cracks (>1500 μm^2) and small pores (< 50 μm^2). From the feature analysis, a feature index (FRI) of 106 was calculated by Equation 1, which reflects the highly anisotropic microstructure of the needle coke image.

In comparison, Figure 5 shows images of a less anisotropic calcined coke sample, with much smaller sizes of optical texture elements and more isometric boundaries of the pores. The feature analysis of the binary boundary image gave a much smaller FRI (25) compared to that obtained for the highly anisotropic needle coke structure shown in Figure 4 (106). These examples show that FRI calculated from the boundary images of the texture domains and pores in the calcined cokes provides a measure of the degree of microstructural anisotropy.

Figure 6 shows a plot of the coefficient of thermal expansion (CTE) against the calculated feature index of the nine calcined coke particles. The plot shows that there is a good correlation between the CTE and FRI within a narrow range of low CTE values desired for the needle cokes used for manufacturing graphite electrodes. This sensitive relationship between the CTE and FRI suggests that the characteristics of both the optical texture and the pores which have the similar size range of optical texture can affect the CTE of calcined cokes¹¹. The apparent close correspondence between the optical texture and the pore structure can be explained by considering that the pores analyzed in this study were produced most likely by shrinkage during calcination⁷.

CONCLUSIONS

Boundary imaging and analysis can be used to characterize the microstructure of semi-cokes and calcined cokes in terms of a feature index. A feature index was defined to represent the size, shape, and orientation of the optical domains obtained by polarized-light microscopy and digital image processing. The principal advantage of this technique is that there is no need to identify the individual texture elements in a given image. A good correlation was observed between the thermal expansion coefficients of the calcined cokes and feature indices derived by digital image

analysis of the optical texture and pore structure. For feature analysis, there is no need to exclude the pores which have a comparable size range to that of the texture elements.

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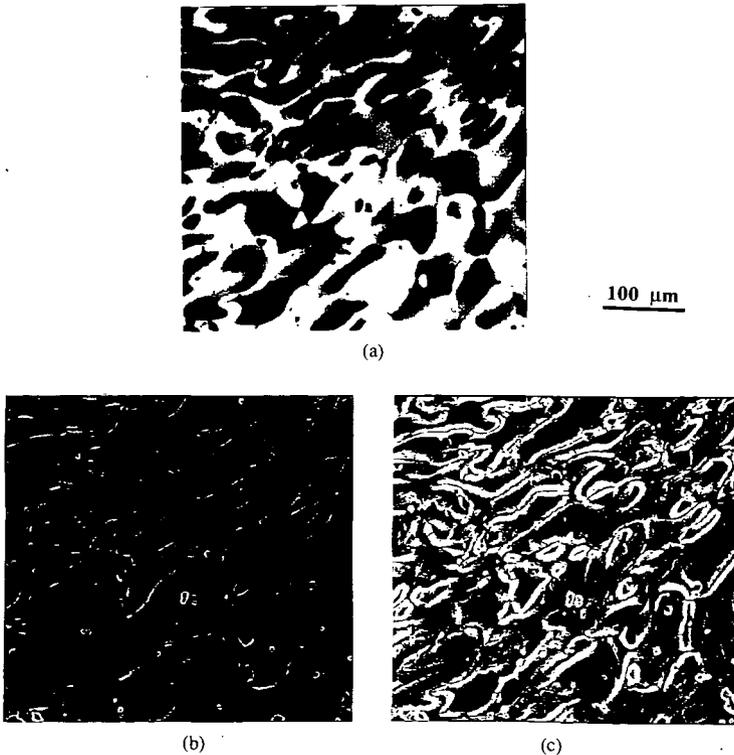


Figure 1. Typical images of domain structure in semi-cokes: (a) gray level image; (b) boundary image; (c) binary boundary image.

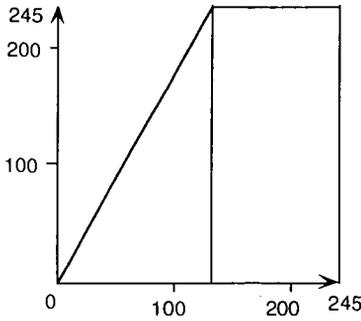


Figure 2. The linear form of transformation function used in contrast stretching.

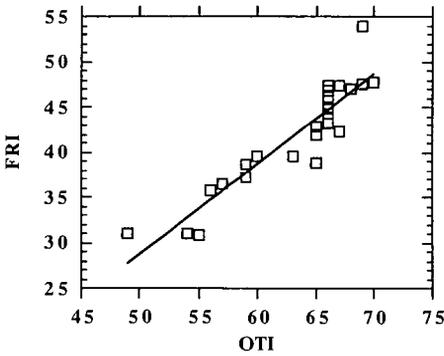


Figure 3. Comparison of feature index (FRI) with optical texture index (OTI).

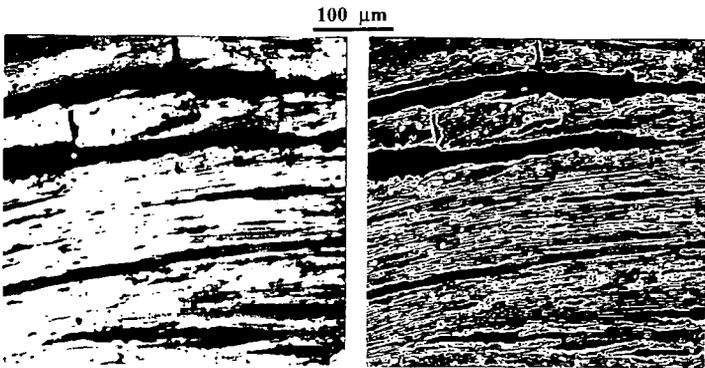


Figure 4. The images of calcined cokes with needle structure; gray level image (left), binary image (right), FRI = 106.

100 μm

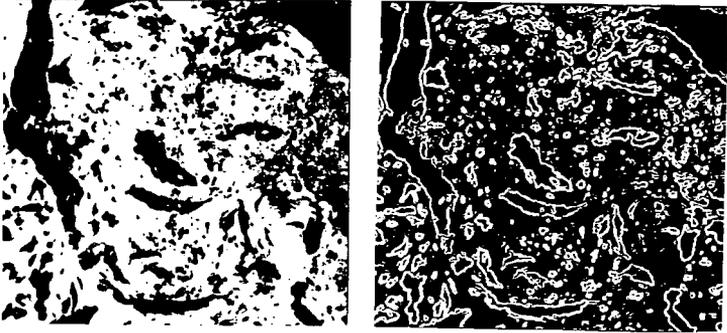


Figure 5. The images of calcined coke with small domain structure; gray level image (left), binary image (right), FRI = 25.

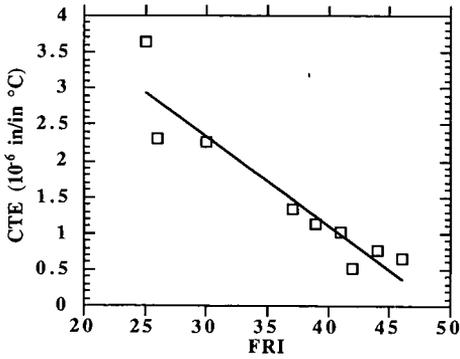


Figure 6. The relationship between coefficient of thermal expansion (CTE) and feature index (FRI) of calcined coke.