CHARACTERIZATION OF CARBON MATERIALS USING QUANTITATIVE OPTICAL MICROSCOPY

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INTRODUCTION

Research which was directed towards the characterization of ultra-fine coal has been utilized in the analysis of vitrinite and high-temperature carbon materials. Equipment and procedures have been developed which permit full reflectance characterization on particles as small as 1.5 micrometers diameter using rotational polarization techniques. Microstructural characterization using optical methods in conjunction with density gradient centrifugation (DGC) have been employed to assess carbon density within its spacial context. Using reflectance mapping techniques with apparent density data obtained from DGC processing, reflectance "images" recorded on the sample can be correlated to reflectance data obtained from DGC fractions. By comparing reflectance values associated with specific density fractions to reflectance values obtained from specific areas within the mapped reflectance image the spacial distribution of reflectance and density can be determined for a complex heterogeneous material.

PROCEDURES

Samples are processed for both petrographic examination and DGC processing. Petrographic samples are processed using standard metalurigical mounting and polishing techniques used for vertical white-light observation. Reflectance data is obtained using a Leitz MPVIII compact microscope modified for rotational polarization reflectance. The modified optical configuration uses the same components that are employed in conventional reflectance. The only variance with convention is the adaptation of the polarizer to permit rotation through 360 degrees, instead of the standard fixed 45 degree orientation, and the use of a fixed stage position during analysis. All polarizer rotation, data acquisition and data processing functions are computer controlled using a 80386/20 mHz computer. Microscope interfacing is made via a gear coupled polarizer and stepping stage, stepping stage controller and IEEE bus. A separate A/D converter controls data acquisition. Optical correction is made on all measurements to remove the effect of residual polarization within the vertical illuminator and transmission optics. Without this correction a pronounced anisotropic signal, induced by rotating one polarizer against a "stationary polarizer" within the light path, would be recorded. This "stationary polarizer" should not be confused with an actual optical component but represents the optical effect caused by residual polarization within the vertical illuminator. Sequential measurements are made across samples with both the maximum reflectance and the spacial orientation of the point stored within a data file.

DGC processing requires samples to be crushed to a particle size $>15\mu m$. The sample is then dispersed in a brig 35 solution to obtain a uniform suspension. The suspension is applied to the top of a vessel filled with an aqueous sodium polytungstate gradient ranging from 1.0 to 2.2 g/mL. The vessel is centrifuged forcing particles to their appropriate density level. After centrifugation the vessel is fractionated by pumping, filtering and weighing the resulting density layers from the density gradient. The resulting density profile accurately represents the density composition of the samples constituents.

DISCUSSION

Figures 1 and 2 illustrate the results obtained from sequential reflectance measurements. The area boxed out with squares represent the point locations where individual reflectance measurements were made. Figure 1 illustrates the results obtained from a vitrinite maceral in coal while figure 2 illustrates reflectance mapping of a C-C composite sample. Figures 3 and 4 represent DGC profiles of the coal sample and C-C composite sample respectively. The indicated points on the DGC profile indicate measured fractions with the average fraction density and reflectance values indicated. Figures 5 and 6 illustrate the spacial density relationships calculated for the "mapped" telocollinite and C-C composite samples respectively.

Figures 1 and 5 illustrate the level of heterogeneity present in vitrinite macerals in both reflectance and density properties. Figures 2 and 6 show the effects of "stress" graphization in graphitized phenolic pitch. In both cases heterogeneity of the carbon can be quantitatively evaluated using combined DGC/Ro mapping techniques.

CONCLUSIONS

Both reflectance mapping and DGC analysis can be used to quantitatively assess carbon heterogeneity. By combining these analysis the spacial distribution of reflectance and density can be determined enabling assessment of physical properties within a morphological context.

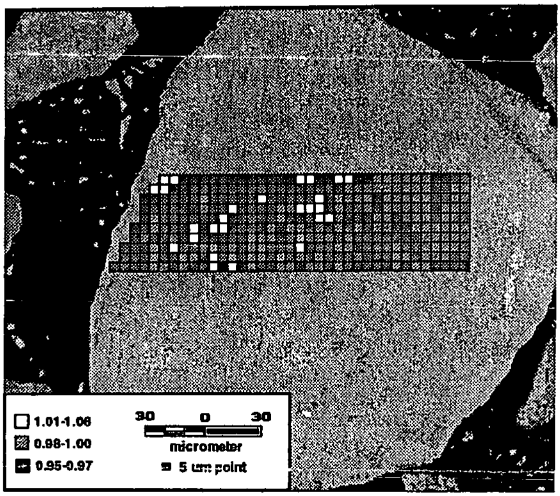

Figure 1: Reflectance distribution as measured on a apparently homogeneous vitrinite maceral.

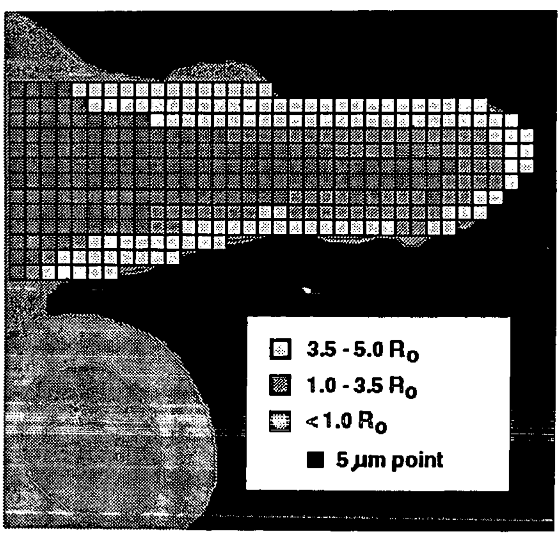

Figure 2: Reflectance distributions measured on a C-C composite sample.

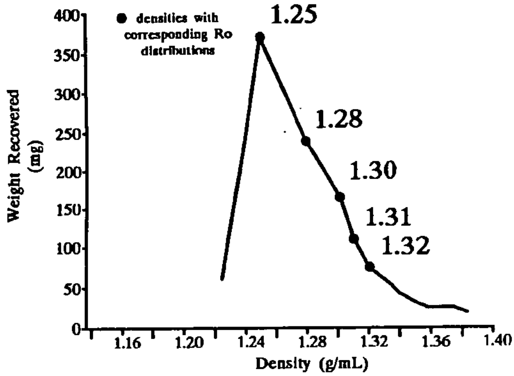

Figure 3: DGC profile of coal sample from which a single vitrinite maceral was mapped (figure 1).

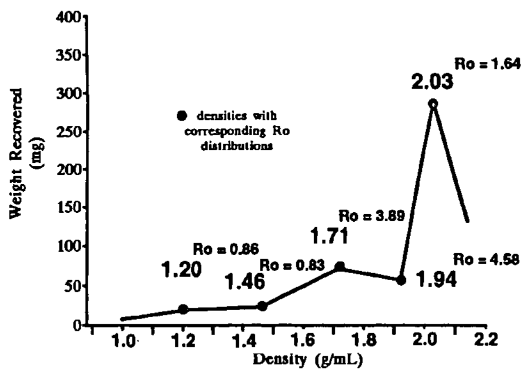

Figure 4: DGC profile of a C-C composite sample from which a single area was mapped (figure 2).

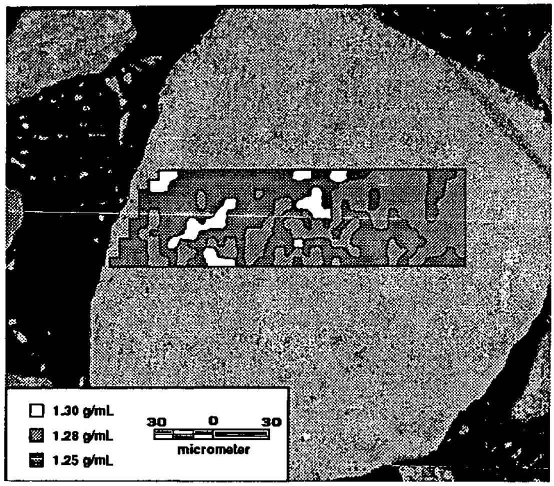

Figure 5: Density correlations made on the mapped vitrinite maceral using combined reflectance mapping/DGC techniques.

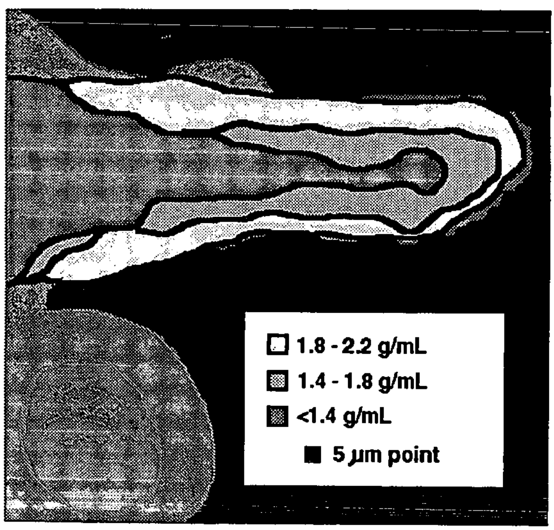

Figure 6: Density correlations made on the mapped C-C composite area using combined reflectance mapping/DGC techniques.