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W. E. Straszheim
Iowa State University

K. A. Younkin
Iowa State University

R. T. Greer
Iowa State University

R. Markuszewski
Iowa State University

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MOUNTING MATERIALS FOR AUTOMATED IMAGE ANALYSIS
OF COALS USING BACKSCATTERED ELECTRON IMAGING

W. E. Straszheim^{*1}, K. A. Younkin¹, R. T. Greer², and R. Markuszewski¹

¹Fossil Energy Program, Ames Laboratory, and
²Department of Engineering Science and Mechanics
Iowa State University, Ames, IA 50011

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Abstract

In order to apply SEM-based automated image analysis (AIA) to the characterization of not only minerals in coal but to the coal itself, sample preparation methods need to be developed beyond common practice. A significant consideration is the degree of contrast achievable between the mount media chosen and the coal. Four low-atomic number materials (epoxy, polyethylene, polystyrene and carnauba wax) were compared for their potential as suitable mounting media for coal samples. Epoxy is satisfactory only for characterization of mineral particles since the contrast between epoxy and coal particles is negligible. Polyethylene or polystyrene have marginal application for use as mounting material for coal characterization due to limited contrast and sample preparation artifacts. Carnauba wax appears satisfactory as a mounting material since it provides good contrast with coal particles with minimal artifacts.

Introduction

Selection of the proper mounting material is critical for performing successful SEM-based automated image analysis (AIA). Sufficient contrast must be maintained between the mounting material and the particles under study. The characterization of mineral particles in coal is of importance in planning and evaluating mechanical and chemical coal cleaning technologies. Mineral particles are easily distinguished from conventional epoxy formulations used for embedding samples when the sample is examined using backscattered electron imaging. However, coal particles have a low mean atomic number, comparable to that of epoxy, and are practically indistinguishable from the epoxy. Therefore, a new mounting material is necessary which provides contrast with both coal and mineral particles in order to characterize coal particles.

Epoxy mixtures have been doped with barium, bromine, and iodine (Moza et al., 1978, Straley, 1983, and Gomez et al., 1984) to raise the mean atomic number of the mounting material and provide contrast. However, the contrast with mineral particles was diminished or lost. Furthermore, the doped epoxies were often difficult to prepare. Therefore, work was undertaken to find an alternative mounting material with a mean atomic number less than that of coal.

Four different mounting materials were studied: conventional epoxy, two polyethylene powders, styrene resin, and carnauba wax. In addition to the contrast they provided with the coal particles, these materials were evaluated for ease of preparation, adhesion to particles, dispersion of particles, and performance in the scanning electron microscope (SEM).

Materials and Methods

Average atomic number

An average atomic number can be calculated for materials that consist of more than one element. This average atomic number, or atomic number factor, can then be used to estimate the contrast between various substances. The atomic number factor (Robinson et al., 1984) is calculated by weighting the average of the atomic numbers of the constituent elements by their mass fraction in the material. For example, consider the polymer polyethylene, $(C_2H_4)_n$. Carbon has an atomic number of 6 and a mass of 12.0 g/mole, and

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*Address for correspondence:

Warren E. Straszheim
B-17 Spedding Hall
Ames Laboratory/Iowa State University
Ames, IA 50011 Phone No. (515) 294-8187

hydrogen has an atomic number of 1 and a mass of 1.0 g/mole. The hydrogen to carbon ratio of 2:1 results in a carbon mass fraction of:

$$\frac{12.0}{12.0 + (2 \times 1.0)} \times 100 = 86\%$$

and a hydrogen mass fraction of 14%. The atomic number factor (ANF) is calculated as:

$$\text{ANF} = \sum_{i=1}^n [(\text{mass fraction}_i) \times (\text{atomic number}_i)] \quad (1)$$

$$= (86\% \times 6) + (14\% \times 1) = 5.28$$

where n is the number of elements comprising the molecule.

The atomic number factors of coal and of a number of prospective polymers have been calculated, and the values are presented in Table 1. The atomic number factors for several minerals are also included for comparison.

The mass fractions for the coal were derived from the ultimate analysis of an Illinois No. 6 coal with the results converted to a dry, mineral matter-free basis. The atomic number factor will vary with the coal composition and especially the sulfur content. For example, the tabulated value of 6.17 is for a sulfur content of 2.0%. Reduction of the sulfur content to 0% led to an atomic number factor of 5.97. In other words, each percent of organic sulfur in the coal will lead to an increase of 0.10 in the atomic number factor.

The atomic number factors of the coal and epoxy (ANF = 5.99) are very similar. Robinson et al. (1984) have stated that contrast as low as 0.1 atomic number units or 3% relative to the ANF of the phase being studied, whichever is greater, can be detected with available backscattered electron detectors. Therefore, it is very difficult to detect the slight difference in backscattered electron intensity between the epoxy and the coal, let alone to consistently differentiate between these two phases with a global threshold as is often employed for image analysis. On the other hand, the difference between the ANF of epoxy and the ANF values of the listed minerals does lead to adequate contrast, and epoxy is quite a satisfactory mounting medium for characterizing the mineral matter.

Polyethylene and polypropylene yield the lowest atomic number factors that can be attained with conventional polymers. They have the highest hydrogen-to-carbon ratio that can be obtained without disrupting the carbon chain. Carnauba and other waxes have ANF values which are practically identical to polyethylene. Polymers such as polystyrene contain less hydrogen and therefore have a higher ANF. Polyesters contain oxygen, and nylons contain oxygen and nitrogen resulting in even higher ANF values.

Sample Preparation

The following sections describe the procedures used to embed the coal particles. Much of the preparation procedure was common to the polymers. Only the production of the solid pellet varied with the polymer.

Samples of several bituminous and sub-bituminous coals have been prepared using the procedures described in this work. Results have largely been independent of the coal used. Samples of an Illinois No. 6 bituminous coal (200 mesh \times 0) were used to produce the pellets described in this work.

Five grams of coal were mixed with an equal weight of polymer and formed into a cylindrical pellet 25 mm in diameter and 15 mm long. The following paragraphs detail the procedures for each polymer.

Epoxy The epoxy was a two component system (LECO Epoxide Resin number 811-164). Portions of resin and hardener were mixed in the recommended 4:1 volume ratio. The liquid was then mixed with the coal particles and cast into a bakelite ring-mold and allowed to harden overnight at room temperature.

Polyethylene Two types of polyethylene powders were used for preparing pellets. The first type was ultra-high molecular weight polyethylene (UHMWPE) from Dow Chemical (designation X0 5317.00). The second was "Allathon D Polythene" resin from DuPont. The Allathon pellets were softer and more translucent than the UHMWPE pellets formed under similar conditions.

The pellets were prepared from both polyethylene powders by mixing five grams of 100 mesh (147 μ m) polyethylene powder with 5 g of coal and transferring the mixture to a preheated (140° C) hydraulic press (LECO model PR10). The mixture was subjected to a pressure of 27.6 MPa (4000 psi) for 7 minutes. The mold was then cooled by forced air convection. Pressure had to be maintained during cooling since both polyethylenes shrank significantly during cooling.

Polystyrene Pellets of polystyrene were prepared from a liquid monomer following a procedure outlined by Shakhashiri (1983). Twenty-five grams of styrene monomer were mixed with aluminum oxide (Baker aluminum oxide neutral, #0540-1, 50-200 μ m nominal particle size) to remove the polymerization inhibitor, stirred a few seconds, and then filtered (Whatman type 50 filter paper). One-half gram of benzoyl peroxide (C_6H_5CO)₂O₂ was dissolved in the styrene to initiate polymerization. The mixture was heated to 97° C to continue polymerization. After 15-20 minutes, the styrene had polymerized to the point where it was viscous and was ready to be mixed with the coal. The mixture of coal and styrene was cast into a bakelite ring-mold and placed in a preheated oven (100° C) and left overnight to finish polymerizing.

Carnauba Wax Approximately 3 grams of carnauba wax (Garrett Wade Cat. No. 51P05.02, M.P.=83° C) were melted on a hot plate at approximately 100° C and mixed with about 2 grams of coal. This mixture was poured into a copper ring mold and allowed to harden for 15 minutes. A greater mass of wax led to shrinkage cracks in the pellet during cooling, apparently related to the increased pellet thickness. No cracking was observed when the total pellet mass was less than 5 grams.

Further Preparation

Hardened pellets of all four materials were prepared for SEM examination using standard petrographic procedures. Pellets were ground to expose a horizontal cross section using 320-grit

Mounting Materials for AIA of Coal

Table 1. Atomic number factors of coal and of selected polymers and minerals.

Material	Representative Chemical Formula	Chemical Composition (mass fraction, %)	Atomic Number Factor
Coal ^a	--	C=81, H=5, O=10, N=2, S=2	6.17
Epoxy ^b	--	C=74, H=7, O=19	5.99
Polyethylene	[C ₂ H ₄] _n	C=86, H=14	5.28
Polypropylene	[CH ₂ CHCH ₃] _n	C=86, H=14	5.28
Polystyrene	[CH ₂ CHC ₆ H ₅] _n	C=92, H=8	5.61
Nylon 6	[CO(CH ₂) ₅ NH] _n	C=72, H=11, O=16, N=14	5.91
Polyester (Dacron)	[CO(C ₆ H ₄)COO(CH ₂) ₂ O] _n	C=63, H=4, O=33	6.45
Carnauba wax ^c	CH ₃ (CH ₂) ₂₉ OH	C=82, H=14, O=4	5.36

Kaolinite	Al ₂ Si ₂ O ₅ (OH) ₄	Al=21, Si=22, O=56, H=2	10.24
Illite ^d	KAl ₄ (Si _{6.5} Al _{1.5} O ₂₀)(OH) ₄	K=5, Al=20, Si=24, O=51, H=0.5	10.99
Calcite	CaCO ₃	Ca=40, C=12, O=48	12.57
Quartz	SiO ₂	Si=47, O=53	10.80
Pyrite	FeS ₂	Fe=46, S=54	20.65

^a Due to the complex and heterogeneous nature of coal, the composition used for coal is typical of high-volatile bituminous coals.

^b Due to the variety of formulas of epoxy, the atomic number factor was calculated for a saturated mixture of 2 moles of the diglycidyl ether of bisphenol A with 1 mole of diethylene triamine. The chemical formula for the ether of bisphenol A is CH₂CHOCH₂O(C₆H₄)C₃H₆(C₆H₄)OCH₂CHOCH₂. The formula for diethylene triamine is NH₂C₂H₄NHC₂H₄NH₂.

^c The formula listed is an approximation. Carnauba wax is a complex mixture of several components (Warth, 1956). The formula given is for myricyl alcohol, a significant component of carnauba wax.

^d A representative formula for illite, after Deer et al. (1966).

silicon carbide paper. The cross section was then polished using 600-grit silicon carbide paper followed by a 5 μm alumina powder suspension on a flocked twill cloth followed by a 0.3 μm alumina powder suspension. The polished sections were coated with about 15 nm of carbon to provide an electrically conductive surface for SEM examination.

Samples were examined in a JEOL-840A SEM with a split annular ring BSE detector and optional high speed amplifier at conditions of 25 kV and a probe current of 1.2 nA. The samples were examined at a working distance of about 35 mm and without any sample tilt.

Results and Discussion

All four materials stood up to examination in the SEM at conditions of 25 kV and 1.2 nA and showed little or no sign of degradation. However, the mounting materials differed in other respects, and especially in the amount of contrast with coal particles (see Figures 1-5 for photos of the epoxy, the two polyethylenes, the polystyrene, and the carnauba wax mounts, respectively). The differences are discussed below.

Epoxy

Epoxy was a relatively easy material to prepare. The epoxy required only mixing the resin and hardener at room temperature. No special cleanliness or precise measurements or critical timing were required to prepare satisfactory pellets. The epoxy produced hard pellets that were easy to polish, and it exhibited good adhesion to the coal particles. The epoxy seemed to be the best material for achieving particle dispersion, but did suffer from some particle settling during polymerization.

The biggest problem with utilizing epoxy for AIA work is its relatively high ANF. As seen in Figure 1, it is practically impossible to distinguish the epoxy from the coal particles in backscattered electron imaging by signal brightness alone. This was expected in view of the similar ANF values for coal and for epoxy.

Polyethylene

The polyethylenes were a little more difficult to work with than the epoxy, but standard hot pressing procedures for thermoplastic materials were adequate. The polyethylene pellets were not as hard as the epoxy pellets and were more difficult to polish. The UHMWPE, especially, did

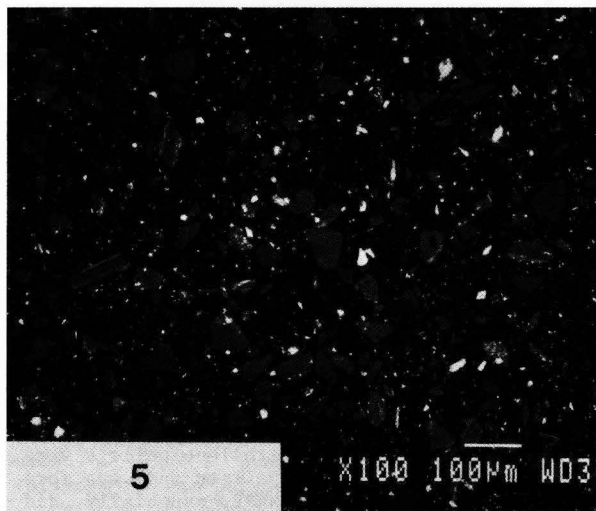
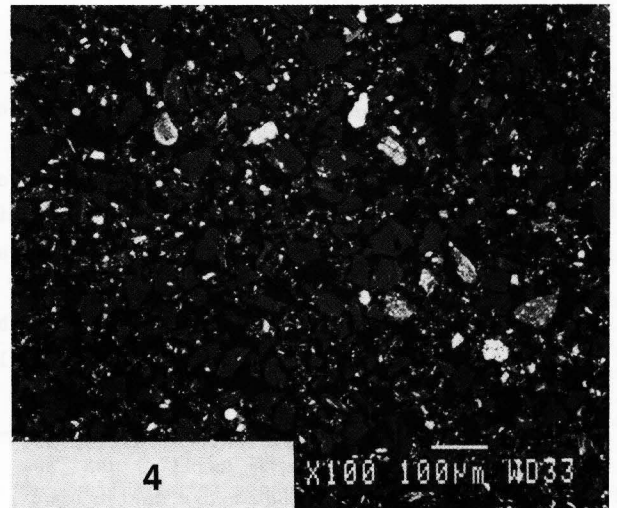
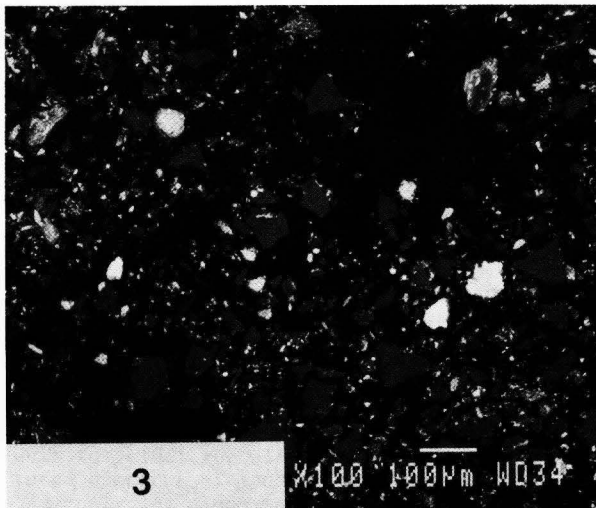
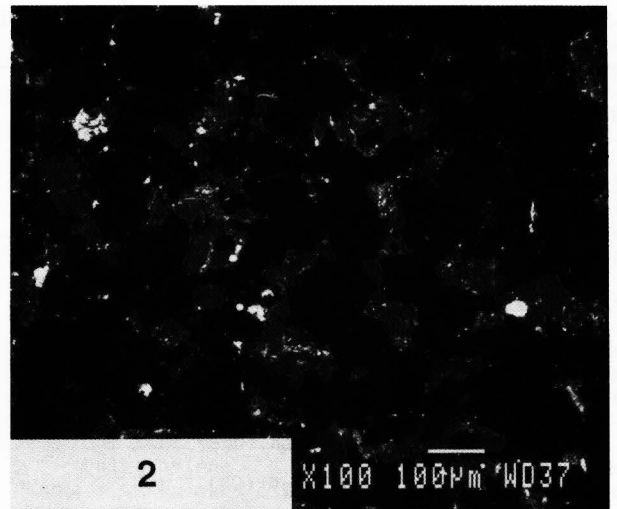
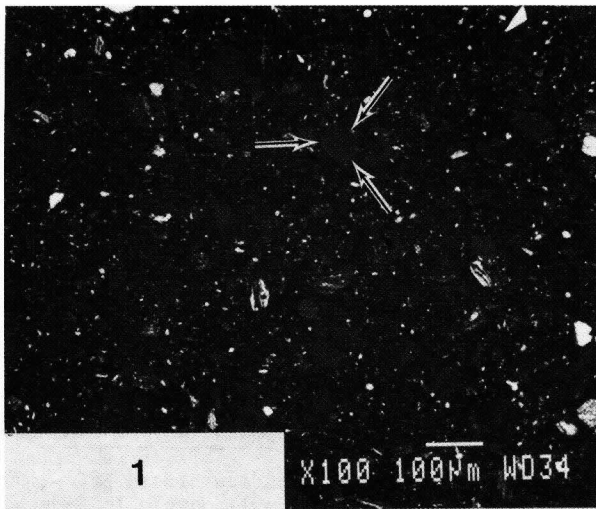


Figure 1. Backscattered electron image of epoxy mount of coal. The coal-epoxy boundary for a typical particle is indicated by the arrows.

Figure 2. Backscattered electron image of ultra-high molecular weight polyethylene mount of coal.

Figure 3. Backscattered electron image of polythene mount of coal.

Figure 4. Backscattered electron image of polystyrene mount of coal.

Figure 5. Backscattered electron image of carnauba wax mount of coal.

Mounting Materials for AIA of Coal

not bond very well to the coal (visible in Figure 2). Greater care was needed to achieve a uniform dispersion of coal particles. Even so, it was practically impossible to mix the coal and polyethylene powders so that coal particles were not in contact. The polyethylene particles commonly forced the coal to be embedded as clusters of particles rather than as separate particles.

The polyethylene did exhibit good contrast with the coal. The ANF difference is $6.17 - 5.28 = 0.89$. However, the problems with surface texture following polishing may restrict the use of the polymer. Intensity variations due to polishing artifacts can be severe enough to exceed the contrast due to the ANF difference.

Polystyrene

The polystyrene pellets were the most difficult to prepare. Polymerization was initiated in a hot water bath. The time at which the coal was mixed with the styrene and the time at which the mixture was cast into pellets were critical. Premature mixing of the coal and styrene monomer resulted in severe settling of the coal and mineral particles, and delayed mixing of the coal and styrene resulted in severe air entrainment. Further curing at elevated temperatures was also necessary for several hours.

The polystyrene did produce hard pellets that were relatively easy to polish, and the polymer exhibited good adhesion to the coal particles. The polystyrene has an ANF of 5.61 and therefore exhibits a contrast of about 0.56 atomic number units with the coal particles. The smoother surface achieved during polishing led to fewer problems with edge effects and noise than were found with the polyethylenes. This allowed easier differentiation of the coal from the polystyrene than from the polyethylenes. However, the relatively small amount of contrast still makes analysis difficult. Small drifts in beam current can cause the coal and/or polystyrene signals to cross over the intensity threshold, giving rise to erroneous results.

Carnauba wax

The carnauba wax was the easiest mounting material to prepare. The wax (M.P.=83° C) melts in about 10 minutes at 100° C, and the melting temperature is not critical. The pellets were relatively easy to polish. Since the coal was

mixed with the molten wax, particles were well dispersed; clumping of coal particles was minimized. Particle settling as the wax solidified was tolerable. The wax does shrink markedly upon cooling, which can lead to cracks in the finished pellet if too much wax is used. However, small cracks do not generally interfere with image analysis. Since the wax is darker than the coal and the cracks are darker still, the cracks appear as part of the background.

The carnauba wax exhibited good contrast with coal particles in BSE imaging and has been used successfully in several studies (Straszheim et al., 1987; Straszheim et al., 1988). The calculated atomic number factor is practically identical to that of polyethylene.

The suitability of the four types of polymers for use as mounting media for image analysis of coal particles is summarized in Table 2.

Conclusions

The carnauba wax appears to be the most satisfactory material for image analysis of coal particles using global thresholding. The only two drawbacks are occasional cracks due to shrinking of the wax during cooling and some settling of particles. The wax does not involve a polymerization where the various chemical functionalities of coal can occasionally inhibit polymerization. Vertical cross sections can be used for image analyses instead of the commonly used horizontal sections, in order to avoid the effects of particle settling. We are also investigating the possibility of mixing sized polyethylene powders with the coal and wax to disperse the coal particles better, and to prevent settling.

Acknowledgments

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Table 2. Comparison of epoxy, polyethylene, polystyrene, and carnauba wax for embedding coal for AIA.

Properties	Material			
	Epoxy	Polyethylene	Polystyrene	Carnauba wax
Ease of preparation	Good	Good	Difficult	Good
Adhesion to coal	Good	Poor	Good	Good
Ease of polishing	Good	Poor	Good	Good
Dispersion of coal	Subject to some settling	Some clumping of particles; no settling	Subject to much settling	Subject to some settling
Contrast with coal:				
Calculated	0.18	0.89	0.56	0.81
Observed	Minimal	Good	Fair	Good

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Discussion with Reviewers

W. Petruk: How good is the adhesion of carnauba wax to mineral grains compared to the adhesion of epoxy resins to mineral grains?

W. Petruk: Is the surface of the carnauba wax hard or semi-plastic? For example, if a mineral particle became dislodged during polishing, would it be re-embedded in the wax surface by the pressure of the polishing wheel, or would it be rolled or smeared beyond the area of the polished section?

Authors: The wax is neither as hard as the epoxy resins, nor does it bond as strongly to the mineral grains. However, we have found the hardness and adhesion to be quite adequate for sample preparation and have observed no problems with particles being "plucked" from the surface of the pellet during polishing. However, we did experience some of the latter problem with the polyethylenes.

F. Huggins: How much variation in atomic number factor might be found across the rank spectrum for U.S. coals? Similarly, how much variation exists among the different maceral types?

Authors: We have calculated the atomic number factor from the ultimate analyses for several coals for which we have data. Those coals ranged in rank from sub-bituminous to low-volatile bituminous. The calculated ANF values ranged from 6.1 to 6.4, but there was no clear relationship with rank.

The major factors affecting the ANF which vary with rank include oxygen, hydrogen, and sulfur contents. Assuming that mature coal can be represented by graphite, the limiting ANF with increasing rank is 6. The effect of additional elements in lower rank coals depends as much on the ratio of abundances of those elements as it does on their individual concentrations. The change in ANF due to the substitution of one percent of each of the above three elements for one percent of ANF=6 material is as follows:

$$\begin{aligned} \text{for hydrogen, } \Delta &= 1\% \times (1.0-6.0) = -0.05, \\ \text{for oxygen, } \Delta &= 1\% \times (8.0-6.0) = +0.02, \\ \text{for sulfur, } \Delta &= 1\% \times (16.0-6.0) = +0.10. \end{aligned}$$

It should be apparent that the hydrogen and oxygen effects will offset each other, and the presence of hydrogen and oxygen in a mass ratio of 2:5 will maintain the ANF of the coal at exactly 6. It should also be obvious that the organic sulfur content has a much larger effect.

Therefore, while the high oxygen-to-hydrogen ratio of lower rank coals will lead to an increase in ANF, so also will the organic sulfur content of the higher rank eastern and midwestern coals. The overall change with rank is hard to predict and depends on the exact composition.

In addition, lower rank coals may contain some moisture that has not been removed during sample preparation. If moisture is present, it will raise the ANF slightly (ANF of water = 7.2). Also, lower rank coals may contain a significant amount of organically bound calcium which serves to significantly raise the ANF, but which is not included in the ultimate analyses. [A calcium content of 1.0% would raise the ANF by 0.14, i.e., $1\% \times (20-6)$]. The final outcome is that the ANF of coal remains near 6, and what substitutions that do occur serve to increase the ANF and provide more contrast with the darker mounting medium.

For similar reasons, we have not seen significant or consistent contrast among maceral types. It has been practically impossible to manually distinguish one maceral from another in BSE imaging or to automatically identify macerals for image analysis.

F. Huggins: It would appear from Table 1 that a mounting medium with an atomic number factor of about 8 would provide two units of contrast between both coal (~6) and mineral matter (>10), and should, therefore, give much better contrast for coal particles than any of the materials considered in the paper, which differ in contrast from coal by less than one unit. Why were mounting media in this range not evaluated?

Authors: As we mentioned in the paper, there has been work done to this end using iodine, barium, and bromine additions to epoxy, and it is true that such a material would provide more contrast with the coal. However, it has also been our impression that it has been difficult to control the ANF of the resultant material, and that it has not been uncommon for the ANF to be so high as to obscure or hide the mineral particles.

Also, our original image analysis hardware would not work with a system where one phase of interest (mineral matter) was lighter than the background and another phase (coal) was darker

than the background. This is no longer a constraint with our present system.

It may well be that materials such as polysulfides or silicone rubbers would provide a consistent ANF between that of coal and mineral matter. However, such materials will also contain elements that are common in coal, and thus they might lead to X-ray signals which would interfere with those from the sample.

D.W. Strickler: In your formula for epoxy you do not mention any chlorine, but it is quite common for these materials to contain several weight percent of chlorine. The presence of chlorine would tend to increase the ANF of the epoxy.

Authors: Indeed, a significant chlorine content would increase the atomic number factor, and it would have an effect on the same order of magnitude that sulfur has on the coal. Thus, it would diminish what very minimal contrast there is between the epoxy and the coal. However, EDS analyses of our epoxy formulation showed chlorine content to be quite low, on the order of 0.2%.

G. Bonifazi: How many samples of each different mounting media have been prepared? Are the reported data related to a significant number of samples, in order to take them to be representative of a phenomenology? Have the results of image analysis applied to the "same family" of mounting media produced different results?

Authors: We have prepared dozens of pellets for each of the polymers described, and have found the results to be very consistent. The variation in contrast that has been observed has been due primarily to variation in the composition of the coals.

W. Petruk: Will the carnauba wax be as stable as epoxy at higher currents, such as 15 nA at 25 kV? **Authors:** Surprisingly, the carnauba wax is at least as stable under the electron beam as the conventional epoxy resin, although there is a strong dependence on the thickness of the carbon coating. We examined one wax sample at 25 kV and 20 nA and at magnifications in excess of 10,000 diameters and detected no degradation. However, with insufficient carbon coating, we have seen degradation at currents as low as 1-2 nA. By comparison, our epoxy resin has been degraded at currents as low as 5 nA.

G. Bonifazi: What kind of image analysis has been applied to the samples (i.e., gray level examination, threshold analysis, texture analysis, etc.) in order to determine the most satisfactory material?

D.W. Strickler: Even though the ANF of the coal is greater than that of the carnauba wax, is this sufficient to allow an automated image analysis of the coals?

Authors: We applied gray level analysis to determine which of the materials was most satisfactory. Ultimately, we were looking for a material that might be distinguished from the coal using global thresholding. By global thresholding we mean that particles are detected on the basis of the gray level signal being in a certain brightness range regardless of the position within the field of view, as opposed to local thresholding where particles are detected only on the basis of

the rapid changes in gray level which occur at particle edges. Therefore, we were looking for a material which would provide a smooth surface of constant gray level. Such characteristics are desired for the background material for any AIA technique.

Figure 6, shown below, is a histogram of the gray levels in a representative BSE image of coal in carnauba wax which is similar to the image shown in Figure 5. The number of pixels having a particular gray level is shown beginning with the darkest gray levels on the left. The two peaks on the left-hand side of the histogram correspond to carnauba wax (left) and to coal (right).

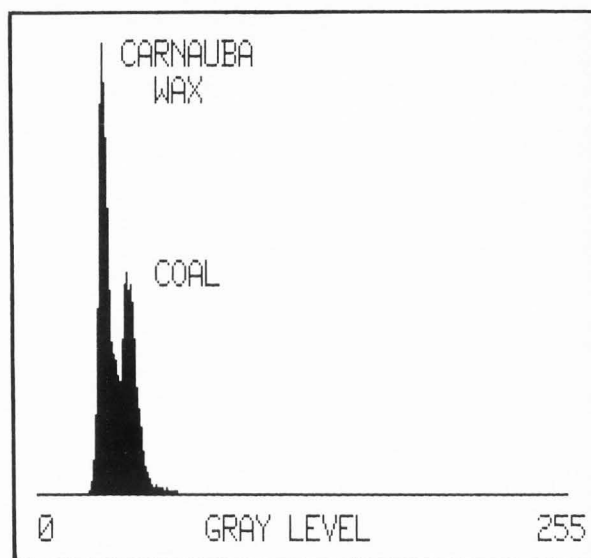


Figure 6. Histogram of brightness levels in an image of coal embedded in carnauba wax.

Ideally, the histogram peaks for the phases would be spikes at a single gray level, but practically, the peaks spread out due to signal noise and polishing artifacts. Even with the spread in the peak width, the peaks are clearly resolvable with a deep trough between the wax and coal peaks. It is in this trough that the global threshold is set to differentiate coal from mounting material.

While there are a few pixels of coal and wax which contribute to the tails of their respective peaks by being much darker or lighter than the average brightness level for that phase, those few pixels can be handled by image processing or analysis software and reassigned to the proper phase. On the other hand, for the polystyrene or epoxy materials described in this paper, the peak overlap is so severe that the peaks are not resolvable.

The contrast between carnauba wax and coal particles has most definitely been proved to be adequate for image analysis. Therefore, we have adopted the carnauba wax as our primary mounting material and have used it for numerous analyses for which we have already reported results (see, for example, Straszheim et al., 1987, 1988).

G. Bonifazi: What are the goals of the subsequent AIA applied to the samples, is it for morphological analysis, pattern recognition and classification, or texture analysis?

Authors: Our specific AIA application is particle recognition, analysis, and classification in determining particle size and phase distributions for both mineral and coal particles in samples of coal. Particles are located using gray level discrimination (i.e., global thresholding). Contiguous pixels within a brightness range are taken as belonging to the same homogeneous particle. Particle shape and size are determined using one of three available algorithms. Those approaches involve either constructing a set of diameters across a particle, constructing a finely spaced grid over a particle, or reassembling particles from contiguous chords passing through the particle. Following size and shape characterization, particles are identified and classified using the relative intensities of the characteristic X-ray emissions. The final results can be expressed as tables or graphs showing the distribution of sample mass as a function of particle size (area-equivalent diameter) and chemical phase. Such results are useful for characterizing coals before and after cleaning by various processes.