Report Title:

COMPREHENSIVE INTESTIGATION OF THE LIBERATION CHARACTERISTICS OF PYRITE AND OTHER MINERAL MATTER FROM COAL

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DISCLAIMER

ABSTRACT

A technique has been developed for generating scanning electron microscope images of sections of polished coal and coal particles that are suitable for the measurement of the linear intercept distribution functions and the distribution of three-phase linear grades. The three phases used are; pyrite, ash forming minerals, and coal. The phase-to-phase transition probabilities for the unbroken ore can also be estimated using conventional image analysis techniques. The distribution of linear intercept lengths through the pyrite, ash and coal phases were found to be described by sums of 2, 3 and 4 exponentials respectively. This reflects the presence of distinct textural regions in the coal. The linear grade distributions were determined in 710 - 1000 micron coal particles that had previously been carefully fractionated using dense liquids. The three phase linear grade distributions reflect the variation in particle composition that results from the fractionation and they provide a detailed picture of the three-phase composition of the various fractions. It is not yet possible to stereologically correct these three-phase distributions. However, the measured uncorrected distributions are very encouraging and a viable stereological correction procedure will be developed during the next phase of work on this project.

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EXECUTIVE SUMMARY

The objectives of the project are:

1. Develop laboratory and theoretical techniques for the quantitative measurement of the liberation spectra in samples of coal particles in the size range 50μm - 1 mm. Three dimensional spectra will be required to account for the mineral matter and pyrite separately.

2. Establish, experimentally, the Andrews-Mika diagram for a number of typical U.S. coals and to develop an appropriate parameterized description for the Andrews-Mika diagram so that it may be determined easily and quickly for coals of different origin and different type.

3. Establish an effective and reliable simulation technique so that liberation of both pyritic sulfur and ash during comminution operations can be modeled and the operation of coal preparation facilities simulated. These models will be incorporated into a computer simulation system for coal cleaning plants.

The main conclusion from the work done during this reporting period is that the essential information required to predict and measure the liberation of ash and pyrite from coal during grinding can be measured using image analysis techniques. Linear intercept distributions and linear grade distributions have been successfully measured in "unbroken" coal and particles in the 1000 - 710 μm size range.

RESULTS AND DISCUSSION

1. Experimental

The experimental work undertaken during the reporting period has been directed primarily towards the generation of SEM images of coal samples. To achieve the objectives of this project these images must be of sufficient quality to allow the three phases of interest, namely pyrite, ash-forming minerals and the remaining "coal" to be identified correctly. Furthermore the images must be obtained at sufficient resolution to ensure that all of the essential geometrical information that describes the texture of the material can be established. Imaging of coal is difficult. Our technique is based on back-scattered electron images. Two major difficulties had to be overcome in order to produce images of acceptable quality: sufficient contrast must be obtained between each component in the sample and the mounting medium and sufficient contrast and definition must be obtained between each phase in each particle cross section. The former problem was solved by mounting particles in epoxy (Buehler Epo-thin) saturated with iodoform. The heavy iodine atoms in the matrix provide sufficient BSE intensity to ensure that the background mounting material can be

clearly distinguished from the other phases. Contrast and definition of the phases was achieved by the development of a careful polishing program. Details of the mounting and polishing technique are summarized below.

- After size and density fractionations, the coal particle batches were split in the Pulverit sample splitter to produce samples of about 3 grams.
- The individual samples were rinsed in a bath of ethyl alcohol to remove any residual fines.
- The samples were dried and mounted in Epo-thin epoxy saturated with iodoform and cured at room temperature for about 24 hours.
- Two stage grinding was performed using 320 and 600 grit Carbimet silicon carbide papers for about 5 minutes at each stage with a low viscosity oil as the lubricant (Buehler Polishing Oil).
- Between each stage the samples were thoroughly cleaned in an ultrasonic bath of the same polishing oil.
- Polishing was performed using 9,3, and then 1 μm oil based diamond slurries on Texmet for 4 to 7 minutes each, followed by an ultrasonic bath at each stage.
- Some samples required final polish of 0.25 μm oil based diamond slurry for a few seconds.
- Each of the grinding and polishing stages were carried out by hand with a wheel speed of about 120 rpm (automatic polishing will probably be used for future samples).

Basic linear intercept data has been generated for a sample of Pittsburgh #8 coal. The coal was available as lumps approximately 3 cm in size. Eight lumps were hand picked choosing lumps to cover the range of obvious visible differences in the sample. The lumps were sectioned, mounted, and polished using the procedure outlined above. Images of excellent quality were produced by using high-resolution very low speed scanning of the beam in the scanning electron microscope. These scans generated images 1970 x 2046 pixels at a resolution of 0.06 μm^2 per pixel. Scan speed was about 10 rows per second. A typical image of the “unbroken” coal is shown in Figure 1. The contrast level is set so that the grey levels associated with the ash-forming minerals cover a sufficiently wide range for subsequent segmentation of the phases. This setting leads to grey level saturation of the pyrite phase and to unsaturation of the coal phase while the ash grey levels are well defined and broad. This helps in the subsequent image processing because phase separation between the ash, pyrite and coal phases is optimized.

SEM Image Acquisition Procedures for Unbroken Coal

- Coal pieces were sectioned across the stratification using a slow speed diamond cutting saw.
- 8 pieces 20 to 30 mm in size were mounted in a 52 mm mold using Epo-thin epoxy saturated with iodoform
- Polishing procedures are described above.
- Samples were coated with carbon and the sides painted with silver paint
- SEM Settings:
    - 200X magnification
    - 30 kV accelerating voltage
    - Working distance was 40 mm
    - Brightness was set at a minimum
    - Contrast was set so that the pyrite was saturated at 255, the coal was saturated at 0, and the ash was in between

- Images were the true averages of 2 images (1970 x 2046)
- The scan speed was 0.0005 frames per second (the lowest possible)
- Between 2 and 8 images were taken in each coal piece, depending on the size of the piece.
- The sample was then rotated counter clockwise 90 degrees on the SEM stage and images were taken again (between 2 and 8 piece)

Particles of coal were prepared by crushing using the procedure outlined in Figure 2. This procedure was adopted to maximize the yield of particles in the size range1000 - 710 μm. These particles will be used as parent particles in the proposed experiments for the measurement of the Andrews-Mika diagram for this material.

A sample of 1000 - 710 μm particles was carefully fractionated using heavy liquids as described in our previous report. Representative samples of particles from these fractions were mounted and polished using the procedure described below. Figures 3, 4 and 5 show images collected from the fractionated coal particles and thus containing different relative amounts of ash and pyrite. Several images have been generated for each particle mount. These images give some idea of the success that we have had in the generation of suitable images of coal particles.

SEM Image Acquisition Procedures for Particulate Coal (-1000 + 710 μm)

- Samples were coated with a thin film of carbon, and silver paint was used on the sides to provide electrical contact with the SEM mount.
- SEM images were photo images (1898 x 1480)
- Backscattered electron images
- thirty to thirty-five images per sample
- 25X magnification
- Working distance was 67 mm

- 30 kV accelerating voltage
- The brightness was set at the minimum allowable
- Contrast was set so that the coal was completely saturated at zero and the background registered a peak just to the right of zero.

The images of the “unbroken” coal were ultimately converted to ternary images by segmentation of each of the three phases. The segmentation procedure used is complex, and relies in background correction, edge detection followed by delineation, “finger print” identification of the ash phase and a series of Euclidean Distance Map segmentations to eliminate artifacts in all phases. The linear intercept length distributions were measured using the usual line scanning technique. The measured linear intercept length distributions for each of the phases is shown in Figure 6. These distributions have the characteristics of exponential sums. The distributions of linear intercepts for the coal phase is a sum of four exponentials

$$1 - F(\ell) = 0.454e^{-0.083\ell} + 0.312e^{-0.022\ell} + 0.143e^{-0.0042\ell} + 0.139e^{-0.00078\ell} \quad (1)$$

The linear intercept distribution is the sum of three exponentials

$$1 - F(\ell) = 0.598\,e^{-0.229\ell} + 0.291\,e^{-0.051\ell} + 0.081\,e^{-0.013\ell} \quad (2)$$

and that for the pyrite phase in a sum of two exponentials

$$1 - F(\ell) = 0.858\,e^{-0.100\ell} + 0.136\,e^{-0.017\ell} \quad (3)$$

These functional forms are not unusual and have been observed before in other mineral systems (King 1994). However, the need for 4 exponentials to describe the coal phase is unusual and this reflects the geometrical complexity of the texture of this material. Each exponential in the sum accounts for a single textural region from small scale (approximately 12 pixels) to large scale (approximately 1200 pixels).

The linear intercept distributions are the primary data required to generate a liberation model for the three components in the coal together with the phase transition probabilities. These were measured at the same time as the linear intercept distributions and are recorded in Table 1.

Table 1. **Phase Transition Probabilities for the Three Phases In A Sample of Pittsburgh #8 Coal**

	Coal	Ash	Pyrite
Coal	–	0.462	0.016
Ash	0.464	–	0.021
Pyrite	0.015	0.022	–

These transition probabilities represent the relative frequency of observing the indicated phase transition as the material is traversed by a linear probe. Reference 1 should be consulted for a detailed discussion of the role played by the linear intercept distributions and the transition probabilities in defining the liberation model.

The particle images were processed in a similar way and the distribution of 3-phase linear grades was determined in each of these dense liquid fractions. The results are shown in Figures 7 to 12. They represent the linear grade distributions uncorrected for stereological bias.

Personnel

Personnel who have contributed to this project during the reporting period are:

Professor R. P. King	Principle Investigator
Dr. C. L. Schneider	Postdoctoral Associate
Mr. Naiyang Ma	PhD Student
Mr. K. Oliphant	Undergraduate Student
Mr. D. FitzGerald	Technician
Mrs. C. Gundersen	Program Administrator

CONCLUSION

1. A procedure has been developed to measure the linear intercept distribution of the three phases, coal, ash, and pyrite in samples of “unbroken” coal and in particles after comminution.

2. The linear intercept distribution for each phase is described well by a sum of exponentials with the coal phase exhibiting the greatest geometrical complexity then the ash and finally the pyrite, which appears to consist of no more than two distinct grain populations.

3. The measured linear grade distributions, when plotted as three-phase histograms, are consistent with the expected volumetric distributions for each dense-liquid fraction that has been studied.

REFERENCES

King, RP. *Linear Statistic Models for Mineral Liberation*. Powder Technology 81 (1994) 217-234.

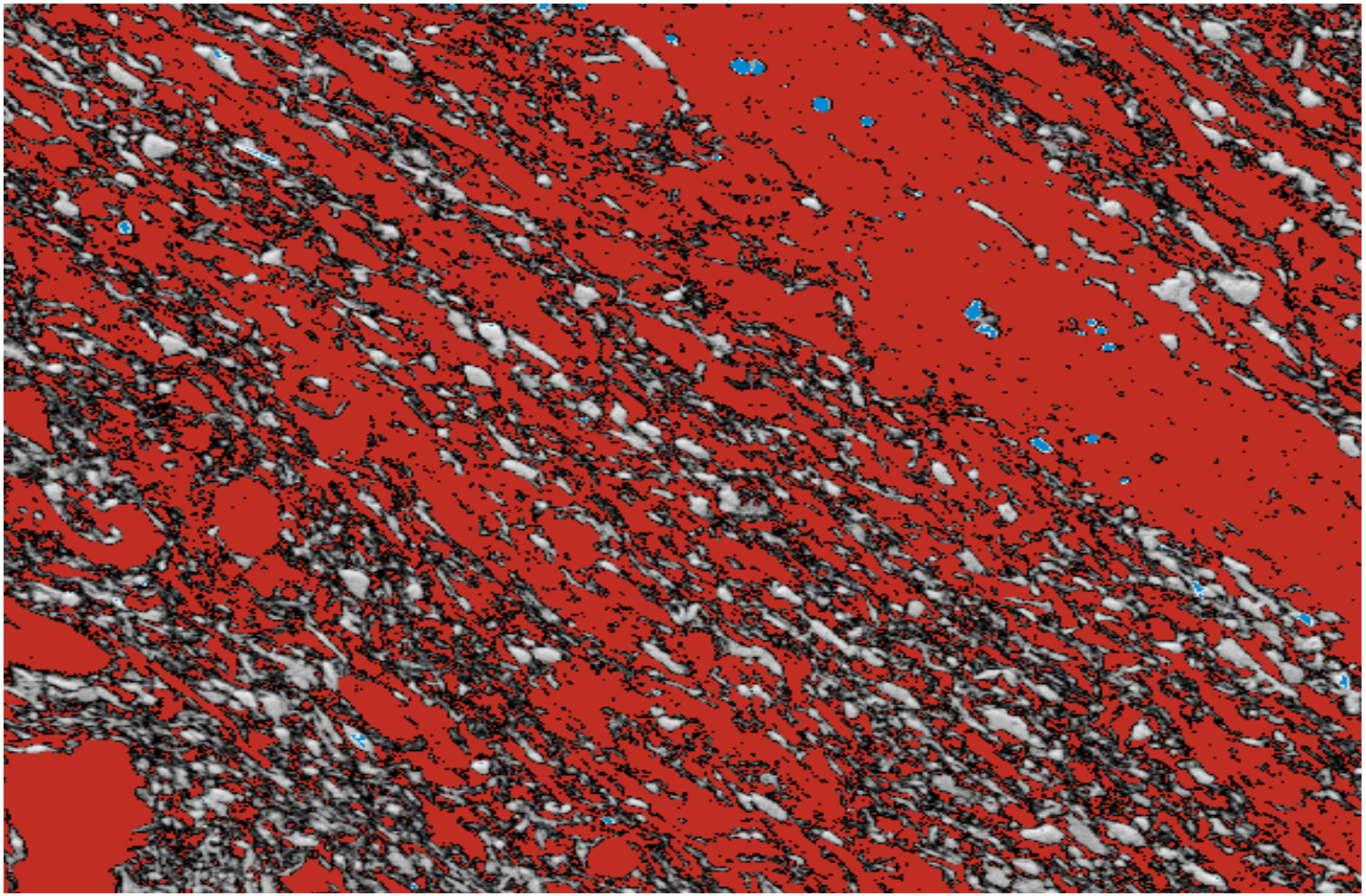

Figure 1: High resolution, high contrast BSE image from a Pittsburgh #8 coal lump. Red and black pixels represent "coal", grey is ash forming minerals and white and blue pixels are pyrite.

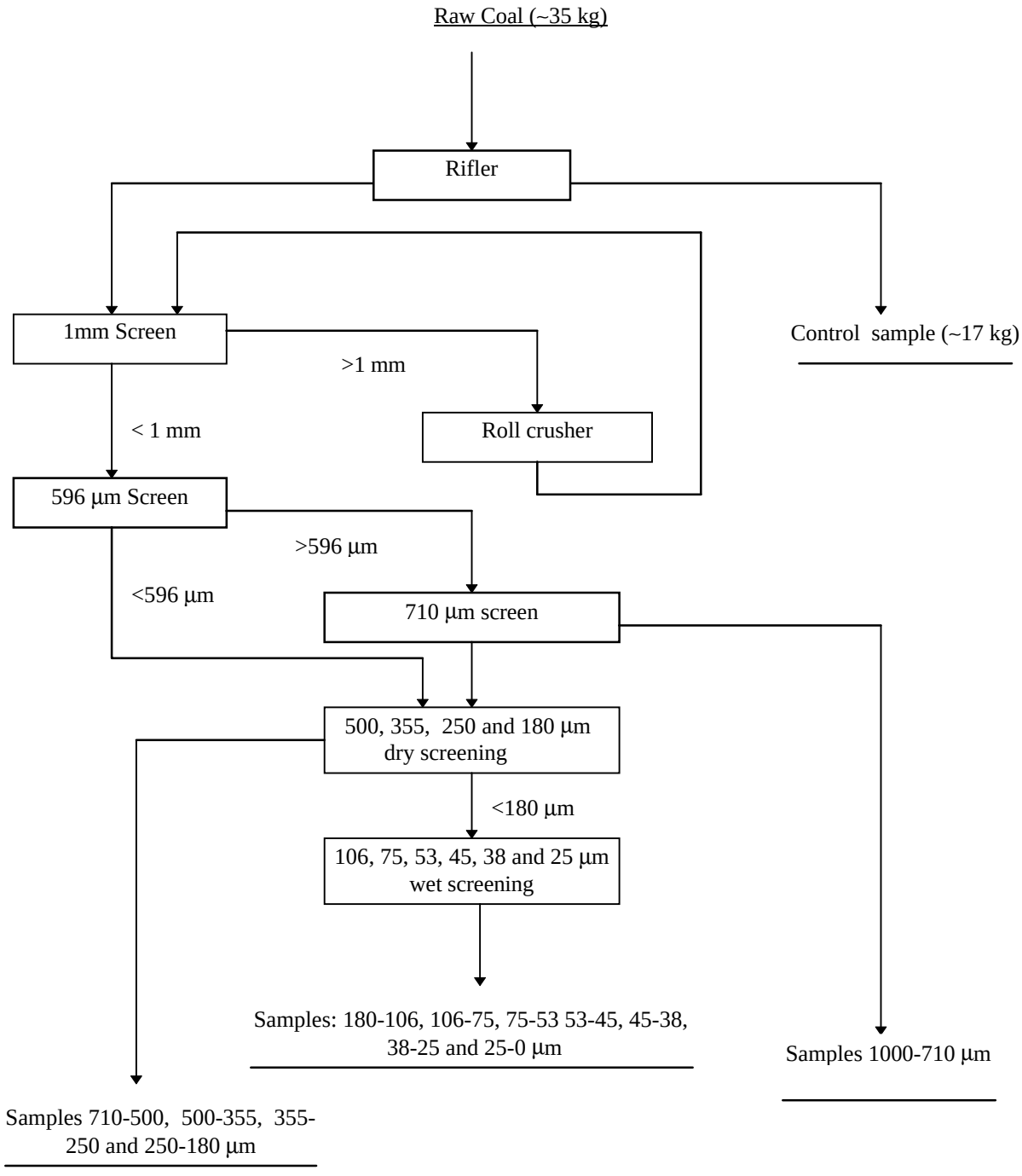

Figure 2: Coal particles crushing procedure

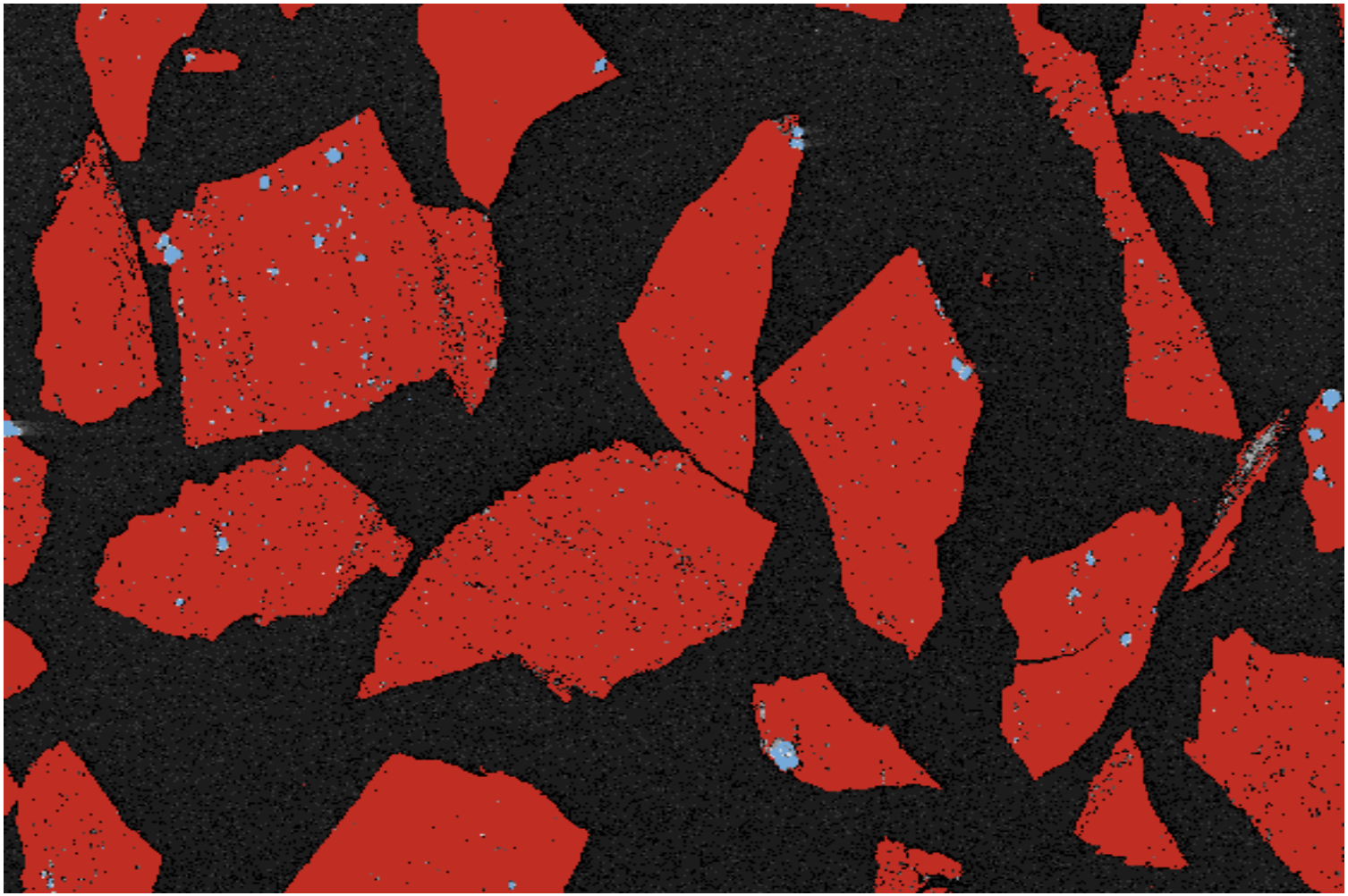

Figure 3: BSE image from Pittsburgh #8 fractionated coal particles, -1000 +710 microns, -1.33 +1.31 g/cc. Red and black pixels represent "coal", dark grey is background, light grey is ash forming minerals and white and blue pixels are pyrite.

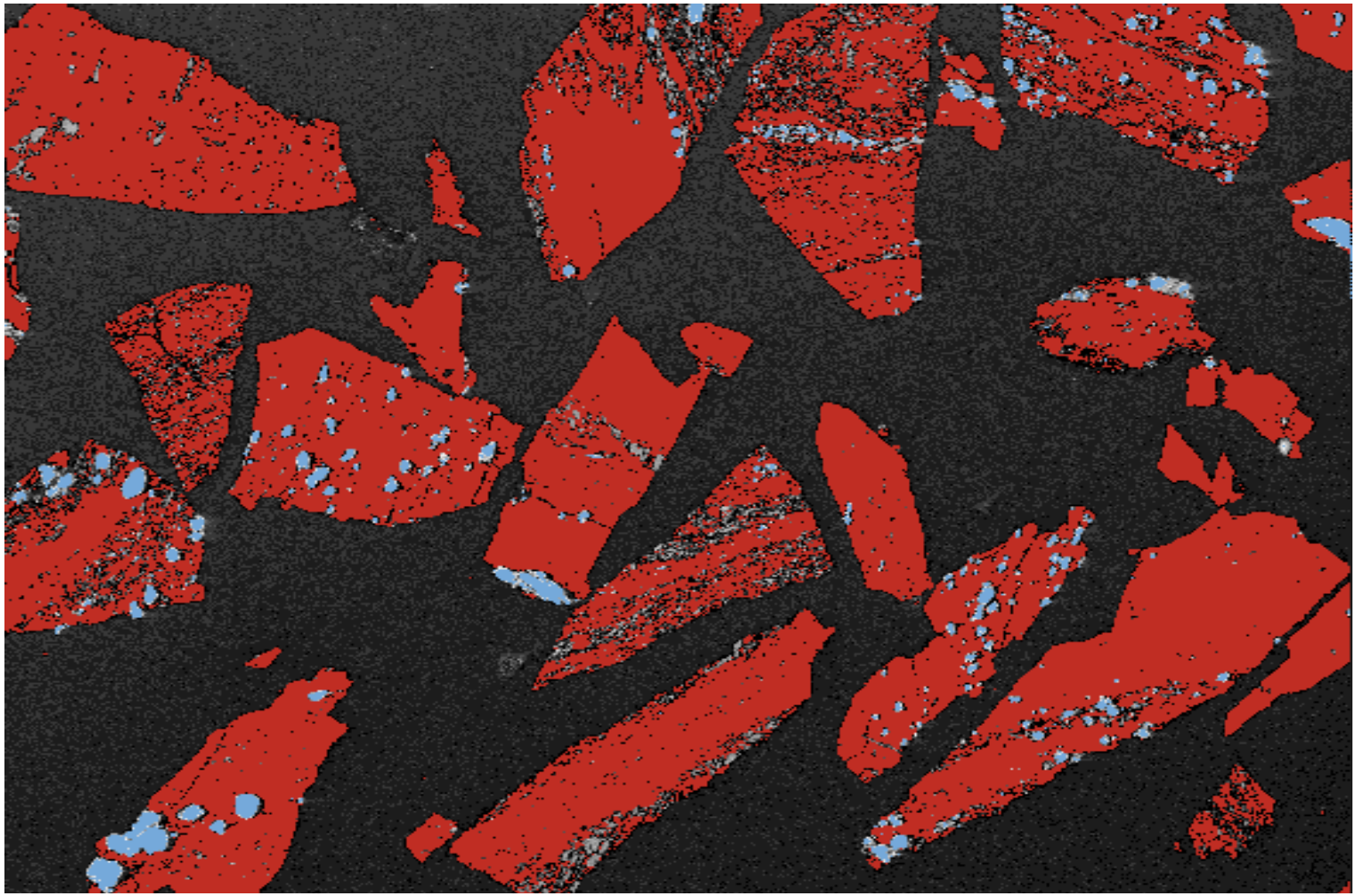

Figure 4: BSE image from Pittsburgh #8 fractionated coal particles, -1000 +710 microns, -1.50 +1.39 g/cc. Red and black pixels represent "coal", dark grey is background, light grey is ash forming minerals and white and blue pixels are pyrite.

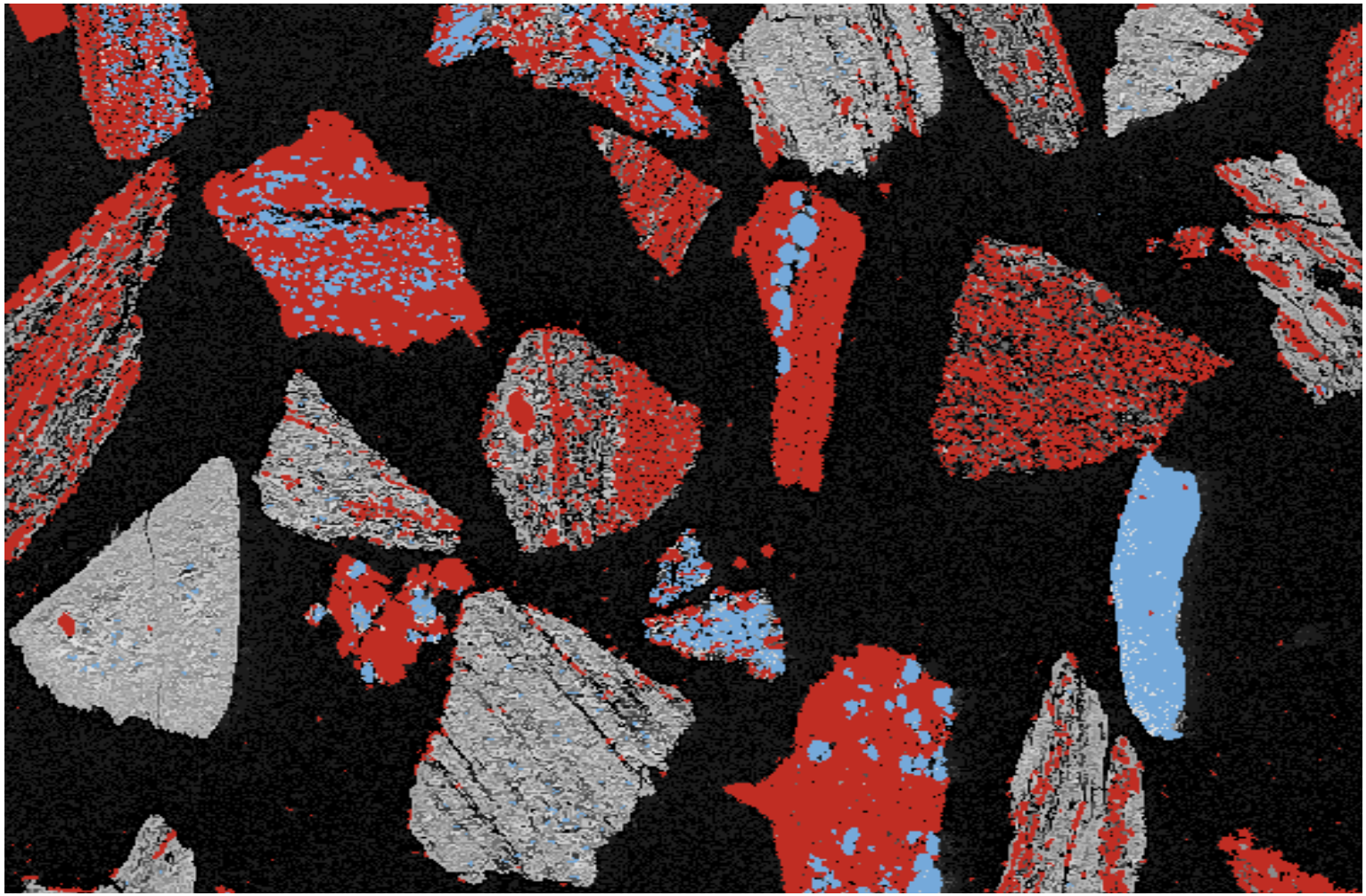

Figure 5: BSE image from Pittsburgh #8 fractionated coal particles, -1000 +710 microns, +1.60 g/cc. Red and black pixels represent "coal", dark grey is background, light grey is ash forming minerals and white and blue pixels are pyrite.

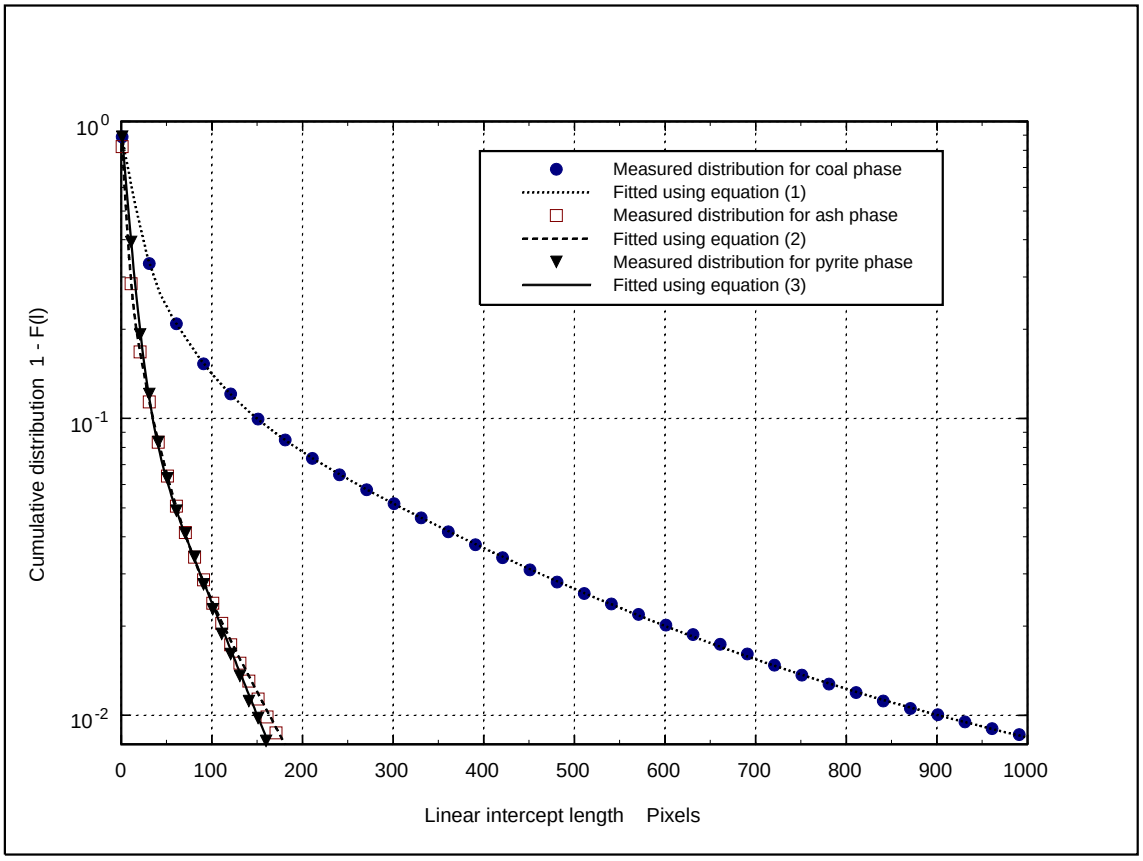

Figure 6: Measured intercept distributions on each phase of unbroken Pittsburgh #8 lumps and the best fits using the exponential models in equations 1, 2 and 3

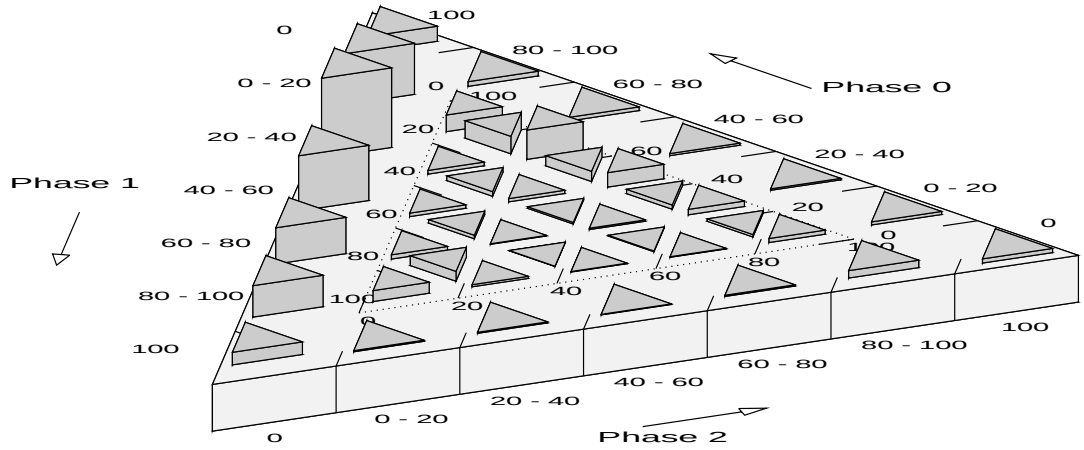

Figure 7: Measured linear grade distibution on fractionated Pittsburgh #8 particles, -1000+710 microns, +1.60 g/cc

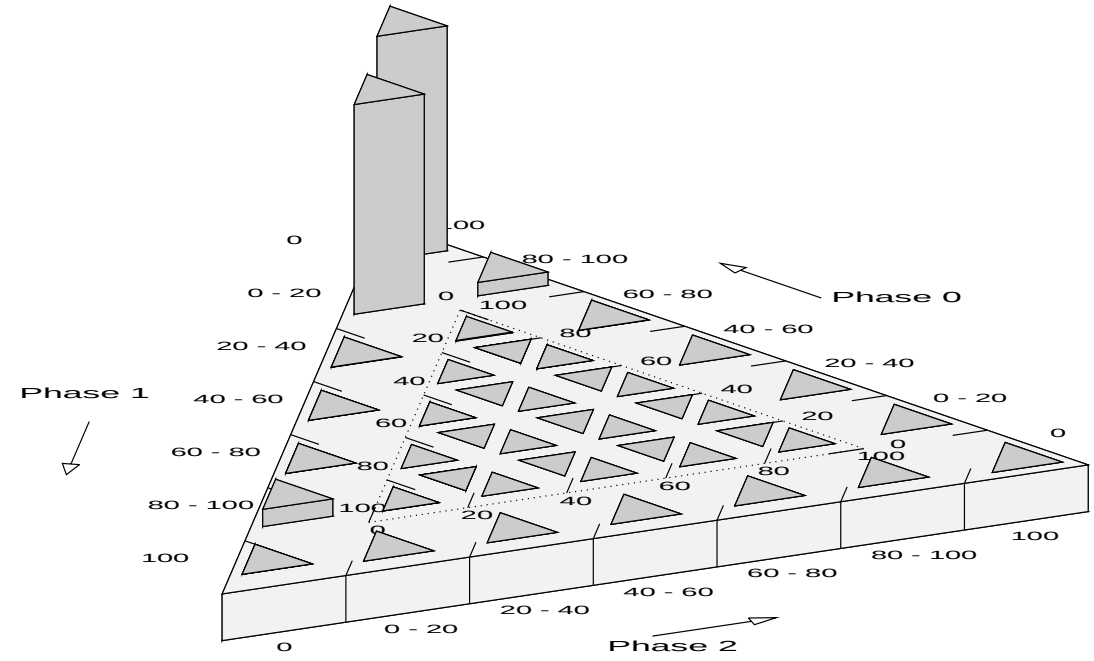

Figure 8: Measured linear grade distribution on fractionated Pittsburgh #8 particles, -1000+710 microns, -1.27+1.25 g/cc

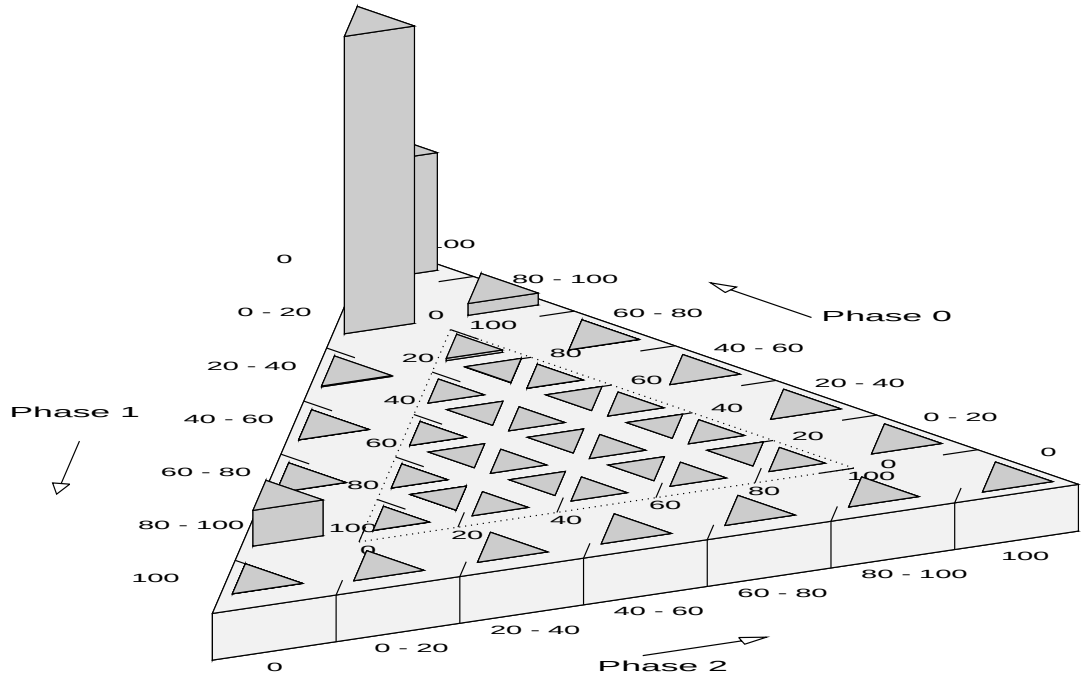

Figure 9: Measured linear grade distribution on fractionated Pittsburgh #8 particles, -1000+710 microns, -1.29 +1.27 g/cc

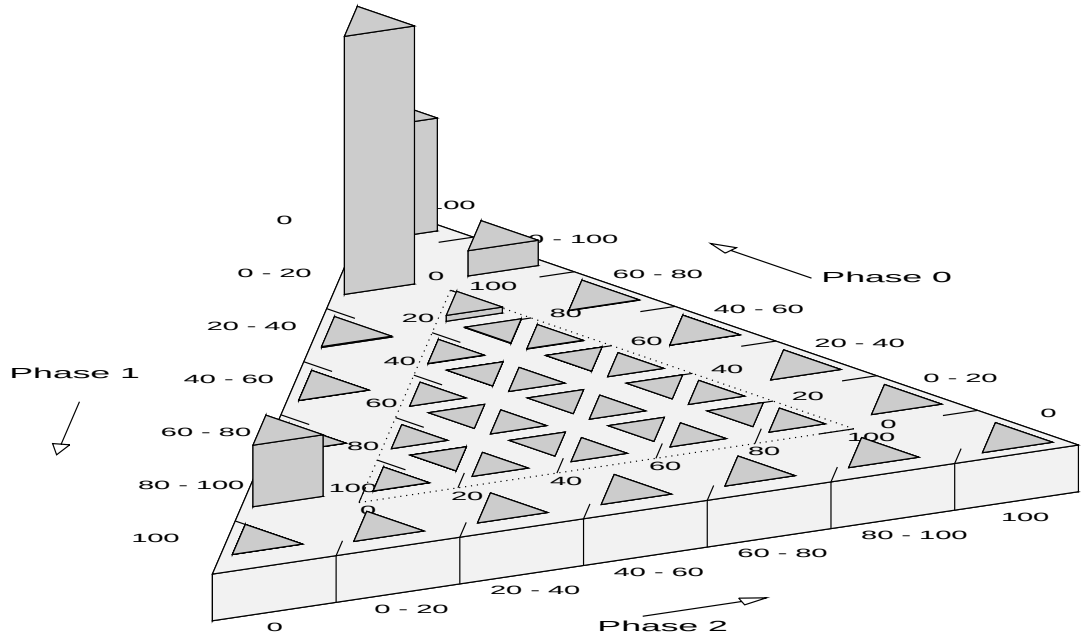

Figure 10:Measured linear grade distribution on fractionated Pttsburgh #8 particles,-1000+710 microns, -1.31 +1.29 g/cc

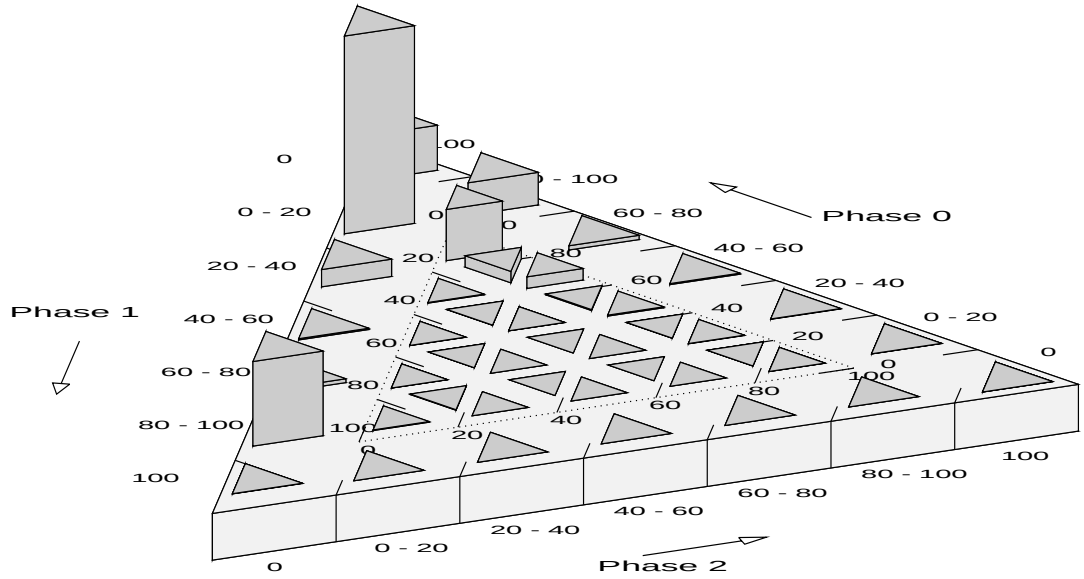

Figure 11: Measured linear grade distribution on fractionated Pittsburgh #8 particles, -1000+710 microns, -1.50 +1.39 g/cc

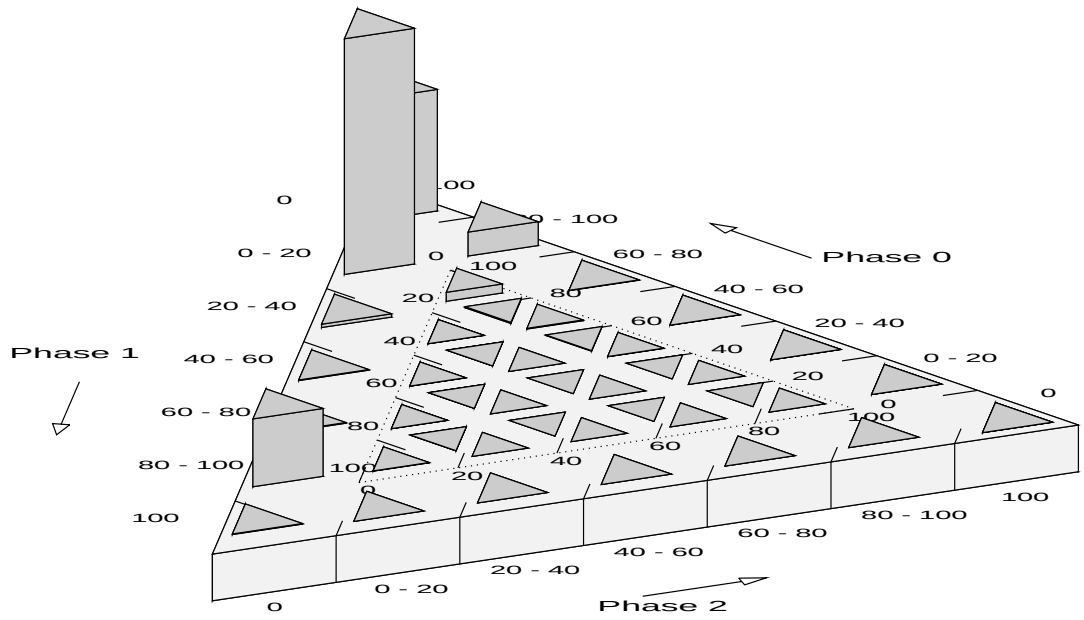

Figure 12: Measured linear grade distribution on composite Pittsburgh #8 particles, -1000 +710 microns