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IMAGE ANALYSIS OF PCC AND AC PAVEMENTS USING SEM IMAGES

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Iowa Department of Transportation

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Iowa Highway Research Board
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TABLE OF CONTENTS

DISCLAIMER

ABSTRACT

The major objective of this work was to evaluate the potential of image analysis for characterizing air voids in Portland Cement Concrete (PCC), voids and constituents of Asphalt Concrete (AC) and aggregate gradation in AC. Images for analysis were obtained from a scanning electron microscope (SEM).

Sample preparation techniques are presented that enhance signal differences so that backscattered electron (BSE) imaging, which is sensitive to atomic number changes, can be effectively employed.

Work with PCC and AC pavement core samples has shown that the low vacuum scanning electron microscope (LVSEM) is better suited towards rapid analyses. The conventional high vacuum SEM can also be used for AC and PCC analyses but some distortion within the sample matrix will occur.

Images with improved resolution can be obtained from scanning electron microscope (SEM) backscatter electron (BSE) micrographs. In a BSE image, voids filled with barium sulfate/resin yield excellent contrast in both PCC and AC. There is a good correlation between percent of air by image analysis and linear traverse.

INTRODUCTION

The determination of air content and air void parameters in hardened PCC using the linear traverse or point count methods is both tedious and time-consuming. Much interest has been expressed in using digital image analysis to measure air voids but the need to obtain sufficient contrast between voids and the surrounding matrix presents a serious obstacle. Although image analyzers are typically capable of differentiating at least 256 levels of gray, serious problems can be encountered due to sources such as uneven illumination, brightness fall-off of the camera lens system, and differential shadowing within voids viewed via a light microscope based system. This paper describes two techniques developed by the authors for enhancing contrast of images obtained from the SEM. A brief description of the instrumentation and techniques employed are included.

IMAGE ANALYSIS

A thorough explanation of image analysis is beyond the scope or intent of this paper. For those interested in further information, several excellent publications are included in the references of this article (1,2,3).

However, for those unfamiliar with image analysis, it may be useful to define what it is, how it works, and what results can be expected. For the purposes of this paper, an image analyzer is a computer based system capable of measuring the size, shape,

and spatial relationship of voids. The images analyzed in this study were obtained from a SEM.

As an introduction, it is important to realize that computers work with numbers, not images, thus the images obtained from the microscope must first be converted to numbers (digitized). If an image is considered as being simply a large array of dots (pixels) with each pixel having a value assigned to it representing its brightness or gray level, then each pixel point can be thought of as having three attributes, namely, one number defining brightness, and two other numbers defining location (x and y coordinates). Typical image analysis systems employ a numerical scale for gray level that ranges from 0 (pure black) to 255 (pure white).

As the major concern is only whether any given pixel is air void or surrounding matrix, the image may be further simplified by thresholding the image so that a binary image is formed that defines all pixels above the threshold value to pure white versus all pixels below the threshold (represented as pure black). Once a binary image has been obtained, a series of mathematical operations can be applied to the stored image to calculate, for instance, the percent void area by a simple summation of the number of pure white (or pure black) pixels. More complex

operations can be employed to determine void sizes, perimeters, edge-to-edge mean free path, etc., based upon gray level and spatial locations.

The key point to remember, however, is the need to obtain sufficient contrast between the air voids and the matrix which constitutes the aggregate and paste.

BACKSCATTERED ELECTRON MODE (BSE) IMAGING

A particularly useful method of imaging in the SEM is provided by backscattered electrons. In the BSE mode, signal intensity is dependant upon the mean atomic number of the specimen at the point of excitation. This strong dependency (Figure 1) has been successfully used to differentiate hydration states of cement pastes (4). However, BSE imaging was not capable of differentiating air voids as their resultant gray level values were not unique.

HEAVY GOLD SPUTTERING

To accentuate differences between the aggregate/paste matrix and the air voids, we hypothesized that an extremely thick coating of gold would provide a very high intensity signal from the matrix while the signals from voids would undergo more scattering and deflection with a resultant loss in signal intensity. Those readers familiar with electron microscopy will recognize that a sputtered gold coating, 20 to 30 nanometers thick, is often used

on specimens to provide a conductive pathway to eliminate "charging". For enhancing signal contrast, a coating thickness in the range of 200-300 nanometers is necessary (Figure 2).

DISADVANTAGES OF SEM AND SPUTTERING TECHNIQUE

The overall intent of this work was to develop a method to perform rapid analysis over the entire surface of 10 cm diameter PCC cores, and although signal differences provided by the gold sputtering technique proved satisfactory, several problems became apparent during subsequent analyses. A short discussion of these difficulties follows as they set the stage for the development of a second technique.

Two potential problems arose that were related to the high vacuum requirement of the conventional SEM. First, pre-pumping of large samples (10 cm diameter cores, 5 mm thick) was necessary to prevent a shutdown of the SEM due to excessive outgassing. The required amount of pre-pumping varied from several hours to overnight, depending on the condition of the PCC which, of course, detracts from the utility of the method from the standpoint of speed.

Secondly, microcracking occurred on the sample surface, due to the excessive vacuum applied to the samples. Although the microcracks can easily be eliminated by image processing

techniques, it is diagnostically useful to know if microcracking occurred before the sample was subjected to a vacuum.

Additionally, several other limitations are imposed by the conventional SEM. Standard high vacuum SEM's typically have a fairly restricted range of stage movement in the X and Y direction of a sample surface. To adequately sample across the specimen plane of a 100 mm x 100 mm x 5 mm sample is beyond the capability of most conventional systems and novel approaches to re-positioning a sample while under high vacuum must be devised.

Lastly, the minimum magnification of conventional SEM's is typically 10x, yet it is desirable to image at magnifications of 1x or 2x to cover a larger field of view, especially for characterizing large entrapped air voids. With some instruments, it is possible to achieve 2x magnification by fully tilting the stage so that it is out of the viewing area (as much as possible) and placing the specimen well below the normal viewing plane. This method, however, eliminates the possibility of moving a sample and reduces signal intensity. It should be noted that the extent to which a sample can be lowered beyond the normal position is also contingent upon the amount of residual free play in the particular instrument's coarse focus control knob.

LOW VACUUM SEM

Because of the limitations imposed by the high vacuum SEM, subsequent efforts have been directed at the potential of a newer low vacuum SEM (LVSEM). This system is capable of operating at near atmospheric pressure. The technique of gold sputter coating then becomes the limiting time factor as several hours of pre-pumping is required to obtain sufficient vacuum for sputtering. For this reason, a second technique was devised for use with the LVSEM.

BARIUM SULFATE ($BaSO_4$) WITH RESIN TREATMENT

Barium sulfate paste ($BaSO_4$ suspended in water) is routinely used in medicine as a contrast agent for x-ray examinations. Similarly, $BaSO_4$ can be used to fill the voids in a polished concrete specimen to yield high contrast BSE images in the SEM. The paste exhibits excessive drying shrinkage, especially in large or deep voids. This problem can be eliminated by mixing $BaSO_4$ powder with an acrylic resin such as LR White (a low viscosity embedding resin used for electron microscopy) in a ratio of approximately 20 grams $BaSO_4$ to 5 grams LR White. After thorough mixing, 1 drop of accelerator is added to the paste and quickly stirred until completely mixed. The paste is then spread onto the core and worked in with a flat-edged blade held at a 45 degree angle. Excess paste can be easily removed with glassine paper.

The paste is then allowed to cure for fifteen minutes. A few final strokes across a 1200 grit paper (dry) followed by wiping with a soft, lint-free cloth are all that is necessary to prepare the surface for the LVSEM.

A micrograph of a $BaSO_4$/resin treated core prepared by the described procedure is shown in Figure 3. It should be noted that to obtain such contrast as exhibited in the micrograph, very minimal effort is needed concerning optimization of controls on the SEM. A low to mid-range setting of contrast control, moderate beam current, and very little time and effort is required to obtain very satisfactory results.

EXPERIMENTAL ANALYSIS

A JEOL 840A high vacuum SEM operated at 25 kev in the BSE mode was utilized for this study. A magnification of 20x was used to yield images covering an area of 0.187 cm^2. Ten images were randomly acquired for each sample providing a total analysis area of 1.87 cm^2. Although such a sampling scheme requires only about 15 minutes for image digitization and subsequent analysis of the entire 10 frames, the actual number of voids thus analyzed far exceeded that obtained by the linear traverse system operated over a 241 cm (95 in.) traverse (Figure 4) and requiring about 7 hours.

Further work is necessary to evaluate optimum magnification, number of areas to be analyzed, and location of areas for analysis. It appears likely that a combination of analyses are required to accurately characterize any given core. For example, a 1x magnification may serve to characterize the entire entrapped air void system, yet such a low magnification cannot detect small entrained voids. Conversely, higher magnifications (100-400X), as required to elucidate entrained voids, are not useful for characterizing the entrapped air void system.

Additionally, at issue is the necessity of including large aggregate into the analysis scheme. More efficiency, as well as accuracy, in characterizing the void system can be gained by excluding large aggregate from the area analyzed. Some coarse aggregate contains large air voids that may be inadvertently included by some operators. It seems plausible, using low magnification, to analyze the large aggregate fraction in a fashion similar, or perhaps, in conjunction with the analysis of the entrapped air void system.

One future possibility may be offered by compositional imaging. For example, characteristic x-rays detected by an energy dispersive spectrometer could be used to elementally map a sample with elemental concentration scaled to intensity of a unique color (5-6). The resultant images could then be analyzed for feature sizes by image analysis. Success of this method is

dependent upon the development of a vastly improved x-ray collection device as high quality compositional images currently require up to 20 hours of collection time per image.

COMPARISON OF PCC VOID DETERMINATIONS

The majority of work to date was conducted using the conventional SEM. Comparative results of percent air (on the same surface) are very encouraging and indicate there is a strong correlation ($R^2 = 0.92$) between linear traverse and SEM based image analysis (Figure 5). The cores for Figure 5 are from Iowa DOT samples and the linear traverse results were from the Iowa DOT and an outside testing laboratory. The linear traverse data was obtained as described in ASTM C457 and was based on a 95" traverse.

The American Concrete Institute "Guide to Durable Concrete" gives recommended parameters for freeze-thaw resistant concrete. With current linear traverse air content data, the air content should be 6.0 ± 1.5 percent, a specific surface (surface area of the air voids) of greater than 24 mm^2/mm^3 (600 $in.^2/in.^3$) and a spacing factor (average maximum distance from any point in cement paste to the edge of the nearest void) of 0.2 mm (0.008 in.) or less.

The specific surface was determined by both linear traverse and image analysis (Table 1). There was a poor correlation ($R^2 = 0.40$) between linear traverse specific surface and image analysis specific surface (Figure 6). Image analysis resulted in

higher specific surface values when compared to linear traverse (Figure 7). The spacing factor by image analysis was consistently lower than linear traverse (Figure 8). A comparison of the bubble size distribution obtained by the linear traverse and by image analysis would clarify the reason for the differences in specific surface by the two methods. As shown in Figure 4, image analysis detected more voids than linear traverse. Speculation is that image analysis detected substantially more very small voids. Unfortunately, the linear traverse systems employed in this study were not configured to provide bubble size information.

One means of evaluating the image analysis bubble size distribution, in a rather rudimentary fashion, was to compare the data from the bubble size distribution of published data (7,8) for a concrete of similar water-cement ratio and air content (Figure 9). The results of such a comparison indicate that image analysis yields similar results in terms of bubble size distribution. Again, however, the specific surface area reported in the literature is lower than that obtained by image analysis and in fact, is also lower than that determined by calculating average chord length using the published bubble size distribution. Powers (6) notes that "the number of bubbles calculated from the specific surface diameter is considerably smaller than that calculated from size distribution...".

Optimally, it would be helpful to analyze hundreds of cores by each method, however, obtaining good quality linear traverse results has proven difficult.

ADVANTAGES OF SEM/IMAGE ANALYSIS

Image analysis performed on images obtained via the scanning electron microscope offers much potential towards understanding air void distribution in concrete. Unquestionably, the speed at which a computer can classify and size objects is far superior to manual methods. Many thousands of voids can be more accurately analyzed giving a greater amount of information to the investigator in less time. Data can be acquired regarding other measurements such as shape and chemistry.

The chief drawback to image analysis, however, continues to be achieving adequate contrast to allow successful automated differentiation of one object from another. To an image analyzer, all voids must totally be within a given range of gray values and these gray values must be unique only to voids. An image analyzer cannot reason that a bright area within a void is part of the void. Image analysis systems lack the superb ability of the human brain to interpret many such details. Light microscope based analysis systems have inherent difficulties that make it more problematic to properly manage light intensities to the extent required by an image analyzer. By using BSE imaging, the pitfalls associated with attempting to establish distinct

gray level differences based upon varying intensity of reflected light are avoided. Contrast, or gray level distinction, is achieved by evaluating differences in atomic number. An atomic number difference of 1 can result in significant contrast differences for sensitive backscattered systems. Consider then, the signal difference between a calcium rich concrete environment (calcium has an atomic number of 20) and a barium rich material (barium has an atomic number of 56). Tremendous differences in contrast can be achieved by utilizing a high atomic number material such as $BaSO_4$/resin. Such heightened difference in signal results in significant gains in obtaining satisfactory images. BSE imaging yields far better resolution of the boundaries of voids.

The newer LVSEM with its ability to operate in a low vacuum environment obviously makes it the instrument of choice, although the conventional high vacuum SEM will work but may require prepumping of samples. Several benefits derived by using the LVSEM warrant further discussion. Adequate characterization requires analysis of a number of cores, thus minimization of specimen preparation and analysis time per sample is important. The LVSEM not only virtually eliminates pumpdown time considerations but further, significantly shortens specimen preparation as the $BaSO_4$ with resin treatment takes only minutes.

Secondly, modification of the LVSEM to achieve lower magnification is greatly simplified. The specimen stage can be easily removed as it is attached to the roll-out door (on Hitachi's instruments). The entire door/stage assembly can be removed and replaced with a fabricated plate. Further, because a high vacuum is unnecessary, the replacement door is easy and inexpensive to construct. In this manner, the column is unobstructed and a sample can be lowered further down the column without difficulty.

Image analysis presents another way of observing air voids because area fractions of the specimen can be analyzed versus lineal fractions as provided by the linear traverse. The linear traverse is a one dimension approximation of void content while image analysis is a two dimensional estimate. A visual observation of the image allows one to determine if void distribution is uniform.

ANALYSIS OF ASPHALT CONCRETE (AC)

Adaptation of the described instrumentation to the characterization of AC appears realistic. Using BSE imaging at 2x magnification, AC cores exhibit significant differences in contrast between the large aggregate and the asphalt matrix in uncoated cores (Figure 10). Another advantage obtained by utilizing BSE imaging is the ability to easily detect even dark colored aggregate as atomic number, not color, provides signal intensity.

The analysis of the void system can be achieved by either heavy gold sputtering or filling the voids with $BaSO_4$ in resin. Each of these techniques however, has limitations for AC.

LIMITATIONS OF HEAVY GOLD SPUTTERING ON AC

The application of a heavy gold layer on the core surface provides the necessary contrast between voids and the surrounding matrix, but eliminates differentiation between the asphalt matrix and aggregate as both are heavily coated with gold. Thus, to provide a measure of void area, aggregate, and asphalt, it is necessary to perform the analysis in two steps, namely, analysis of aggregate versus void and asphalt, followed by gold sputtering and analysis to elucidate void versus aggregate and asphalt.

LIMITATIONS OF $BASO_4$ RESIN TREATMENT

Implementation of the $BaSO_4$ treatment appears to be a more logical approach as atomic number differences between asphalt cement, aggregate, and $BaSO_4$ filled voids would be significant and would permit a 3 gray level analysis scheme. Unfortunately, the action of solvents used in the LR White resin are too disruptive to the asphalt matrix. Barium clearly becomes part of the asphalt cement matrix. Recent success has been experienced with water miscible resins but further work is needed. The asphalt cement will appear nearly black in the SEM image and allow determination of the asphalt cement content without extraction using toxic solvents.

INSTRUMENTAL LIMITATIONS FOR AC ANALYSIS

The high vacuum requirement of the conventional SEM can be detrimental to AC surfaces. Mild distortion of the matrix is apparent at vacuums of 1 x 10 -6 torr, although no damage is apparent at approximate vacuums of 1 x 10 -3 torr. The latter figure of 1 x 10 -3 torr was taken from observations of samples in a conventional SEM operating in a "poor" vacuum state. Obviously, the instrument of choice for AC analysis is the LVSEM.

CONCLUSIONS

The following conclusions are supported by the SEM image analysis research:

1. Improved image resolution can be obtained from SEM BSE images.

2. Gold sputter coating yields excellent contrast of voids in a BSE image of a PCC or AC sample.

3. Barium sulfate/resin filled voids yield an excellent BSE contrast of voids, asphalt cement and aggregate in an AC sample and voids in a PCC sample.

4. Image analysis of SEM images detects significantly more voids than linear traverse.

5. There is a good correlation between percent of air by image analysis and linear traverse.

6. There is a poor correlation between specific surface by image analysis and linear traverse.

7. Image analysis using SEM images consistently yields a higher specific surface and a lower spacing factor than linear traverse due to detection of a greater number of voids.

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TABLE TITLE

1. Core Air Content Data

Table 1 Core Air Content Data

Core No.	Air Content Linear Traverse	Air Content Image Analysis	Specific Surface Linear Traverse mm²/mm³	Specific Surface Linear Traverse in²/in³	Specific Surface Image Analysis mm²/mm³	Specific Surface Image Analysis in²/in³	Spacing Factor Linear Traverse mm	Spacing Factor Linear Traverse in	Spacing Factor Image Analysis mm	Spacing Factor Image Analysis in
1	4.77	3.88	13	339.	29	725.	0.33	0.013	0.15	0.006
2	4.19	4.91	11	275.	37	945.	0.41	0.016	0.13	0.005
3	5.93	5.35	21	544.	53	1348.	0.20	0.008	0.08	0.003
4	5.93	5.61	24	611.	45	1146.	0.18	0.007	0.10	0.004
5	7.72	8.45	30	751.	43	1099.	0.15	0.006	0.10	0.004
6	7.43	7.76	36	902.	60	1534.	0.13	0.005	0.08	0.003
7	4.50	4.51	31	791.	36	907.	0.13	0.005	0.13	0.005
8	4.10	3.78	21	526.	43	1098.	0.20	0.008	0.10	0.004
9	4.10	4.27	13	336.	27	697.	0.33	0.013	0.15	0.006
10	5.20	5.27	18	469.	23	573.	0.23	0.009	0.20	0.008
11	4.40	3.97	22	559.	34	867.	0.20	0.008	0.13	0.005
12	4.20	4.34	--	----	--	----	----	----	----	----
13	8.00	8.19	--	----	--	----	----	----	----	----

FIGURE CAPTIONS

1. Relationship Between Atomic Number and Signal Intensity
2. BSE Image of Heavy Gold Sputter Coated PCC Core, 10X Magnification
3. $BaSO_4$ With Resin Treated PCC Core, 20X Magnification
4. Number of Voids Detected by Each Method
5. Percent Air - Linear Traverse vs. Image Analysis
6. Specific Surface - Linear Traverse vs. Image Analysis
7. Specific Surface Values
8. Spacing Factor
9. Void Size Distribution - Theoretical vs. Measured
10. BSE Image of Uncoated AC Core, 10X Magnification

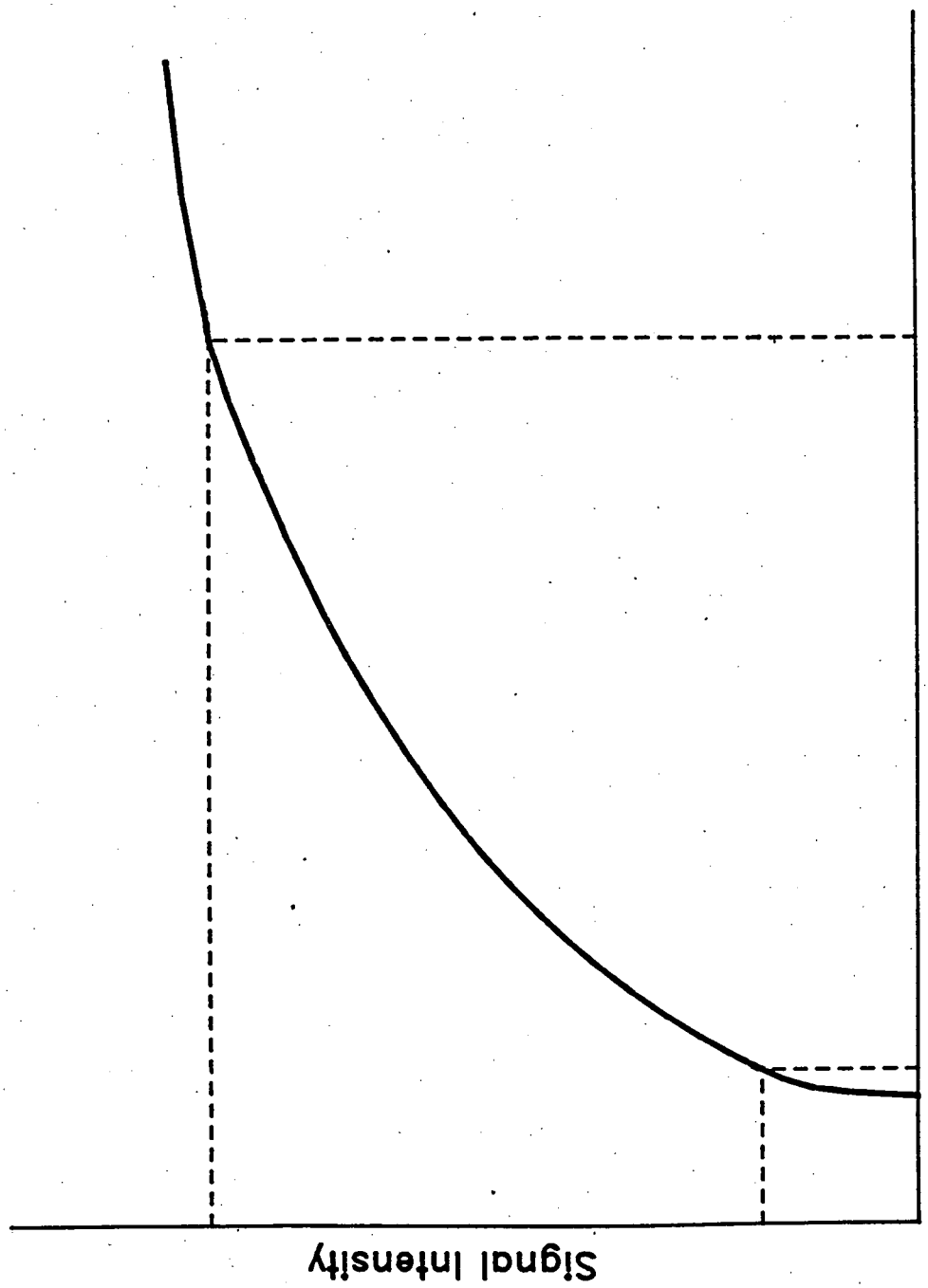


Figure 1— Relationship Between Atomic Number and Signal Intensity

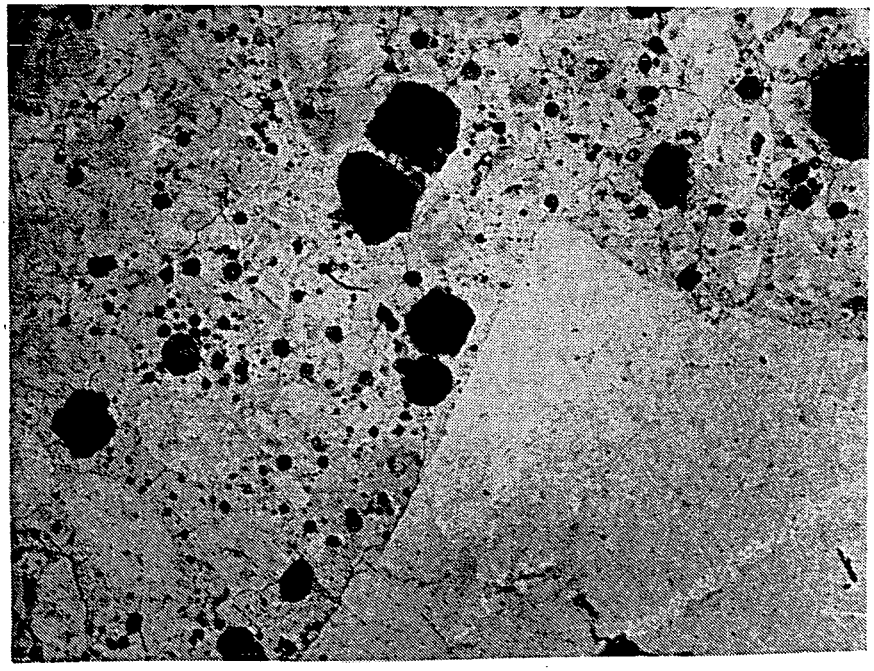


Figure 2 - BSE Image of Heavy Gold Sputter Coated PCC Core

10x Magnification

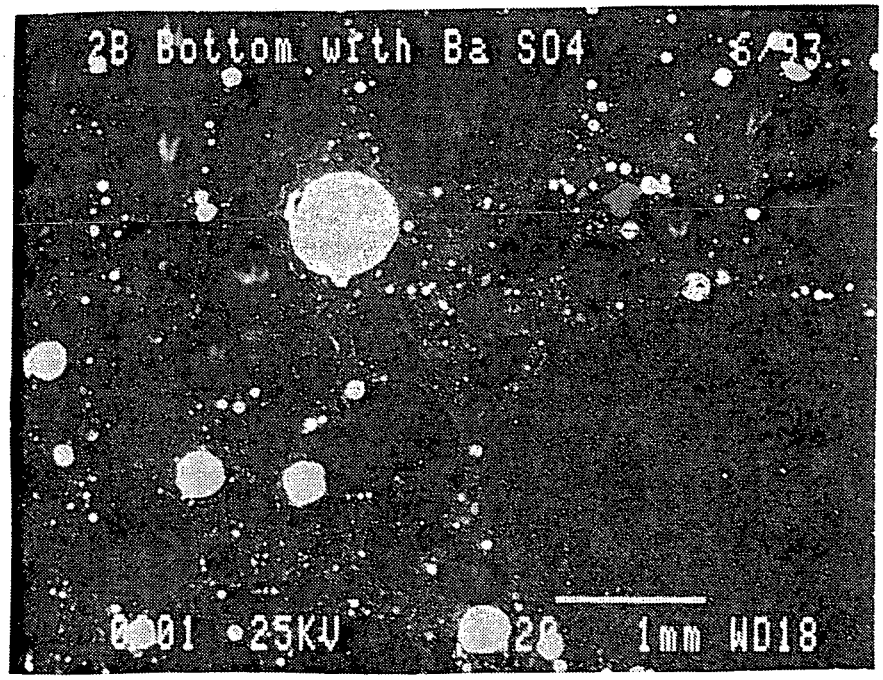


Figure 3 - $BaSO_4$ With Resin Treated PCC Core

20x Magnification

Figure 4 - Number of voids detected by each method

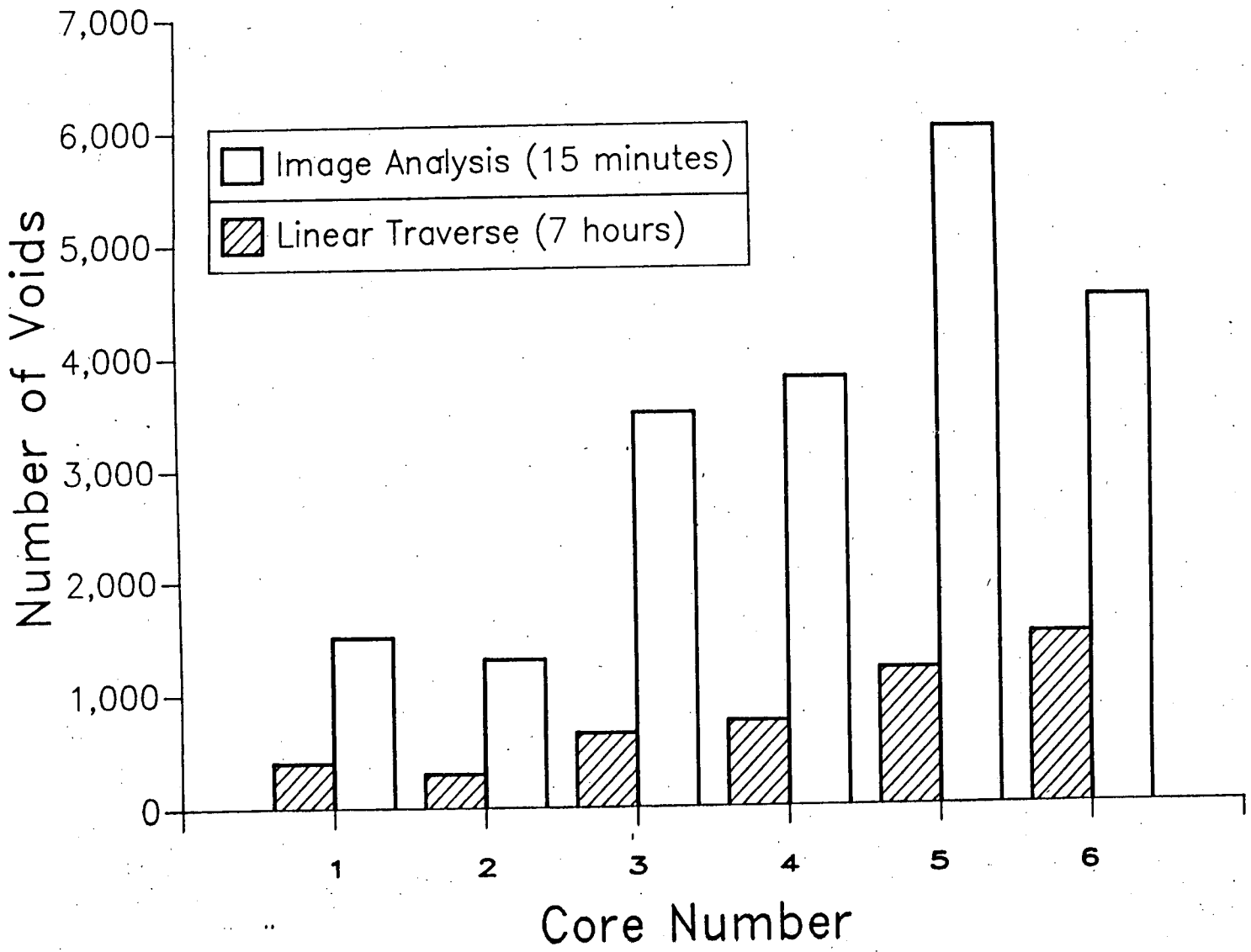


Figure 5 - Percent Air
Linear Traverse Vs. Image Analysis

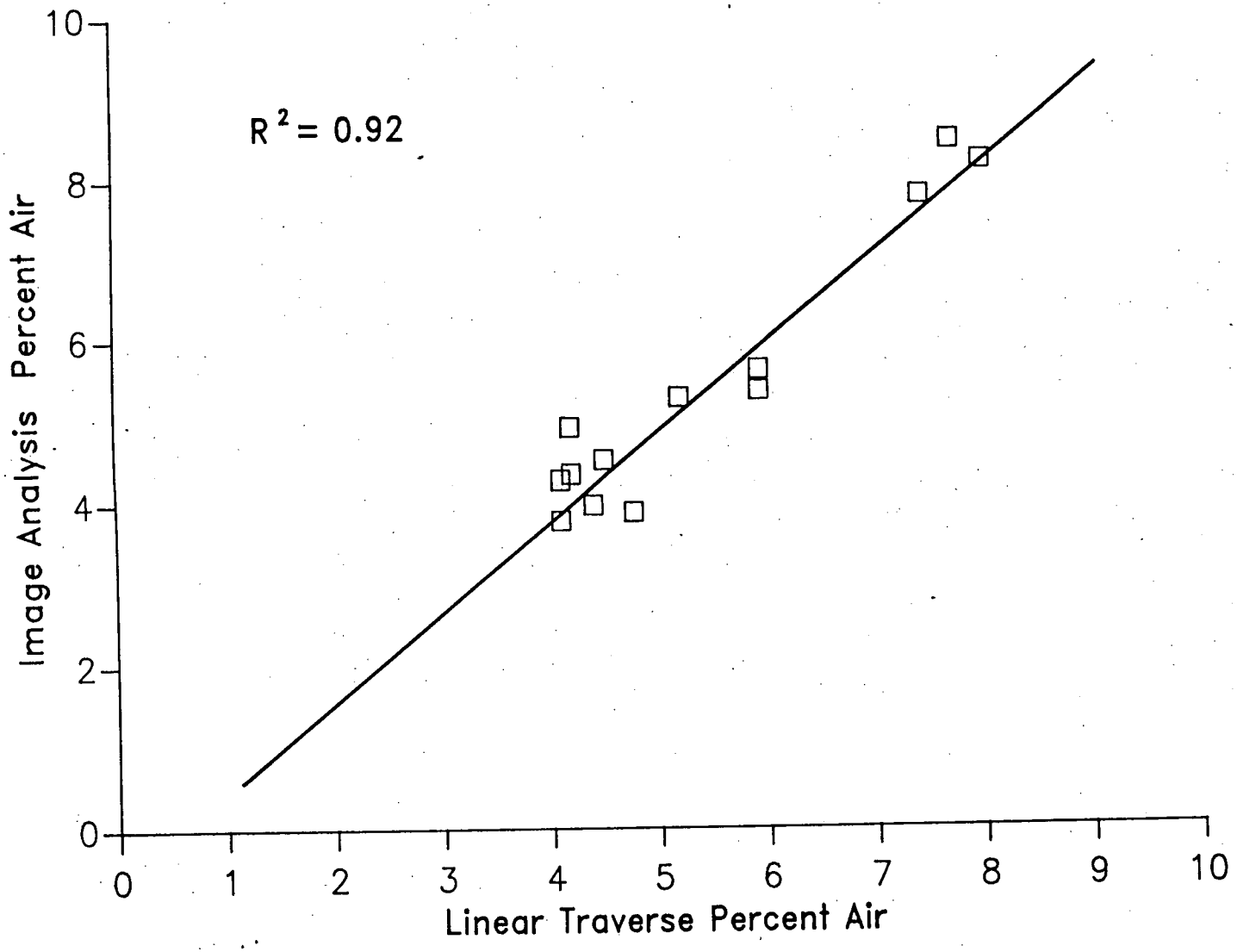


Figure 6 - Specific Surface
Linear Traverse Vs. Image Analysis

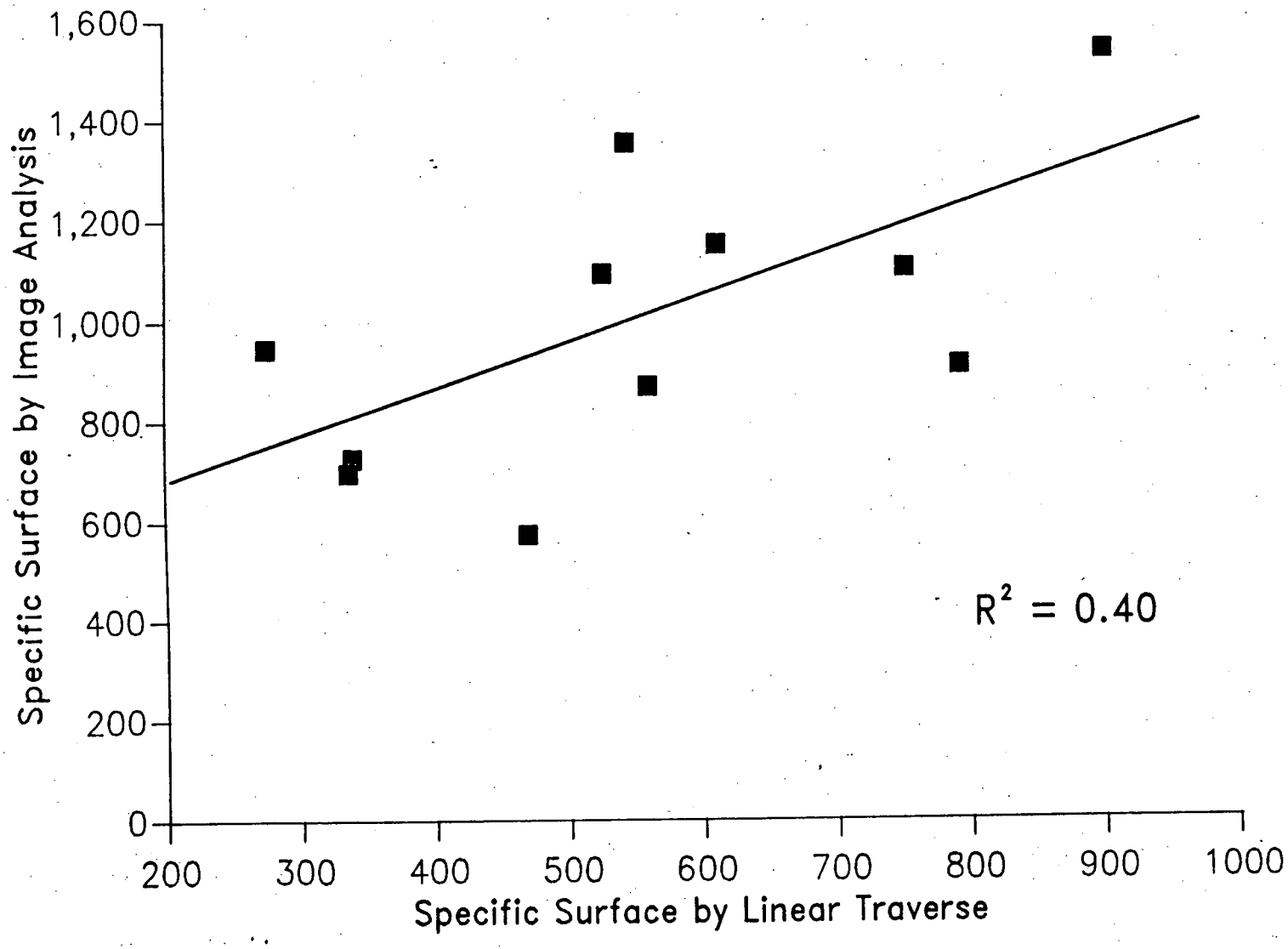


Figure 7 - Specific Surface Values

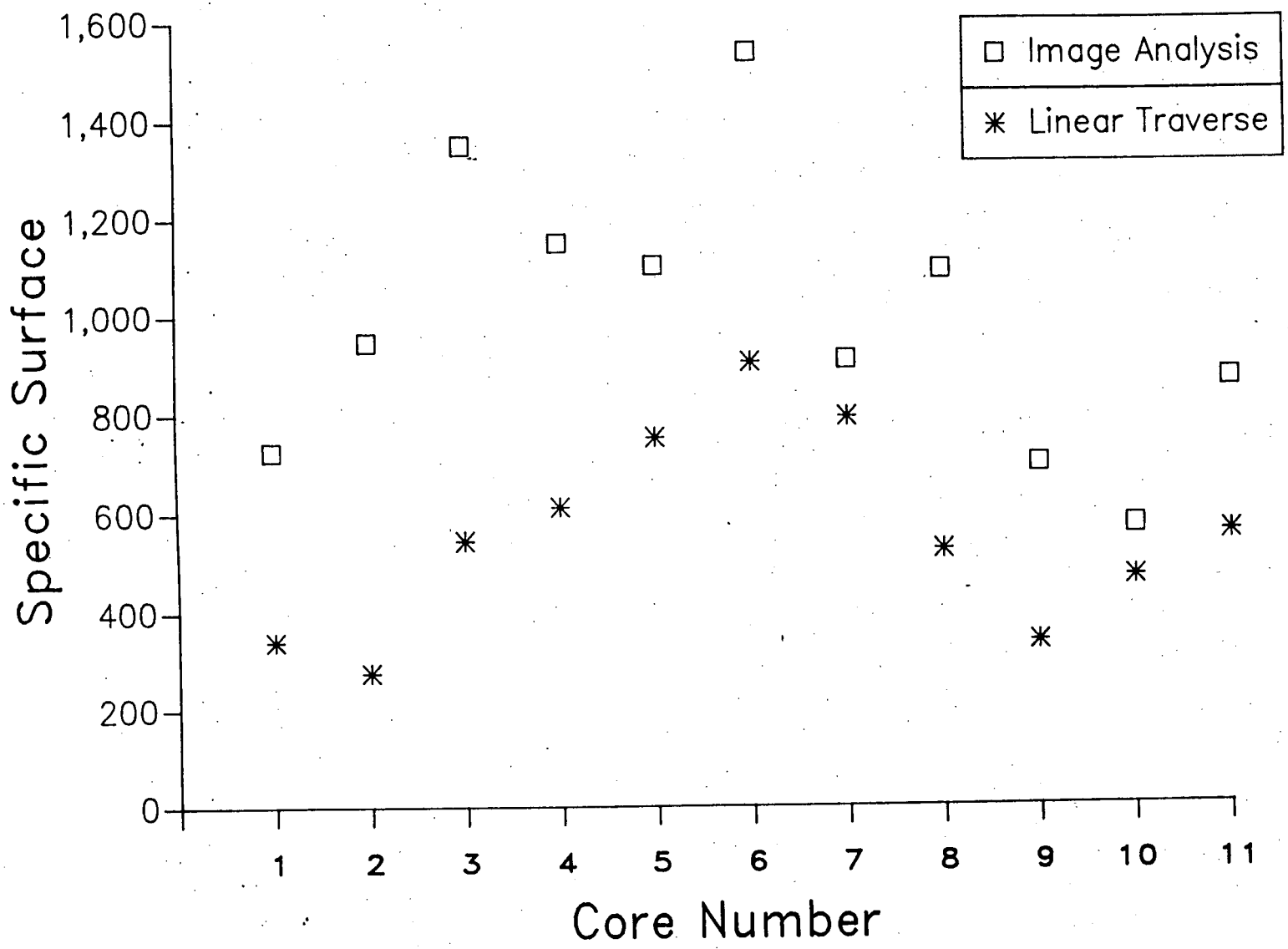


Figure 8 - Spacing Factor

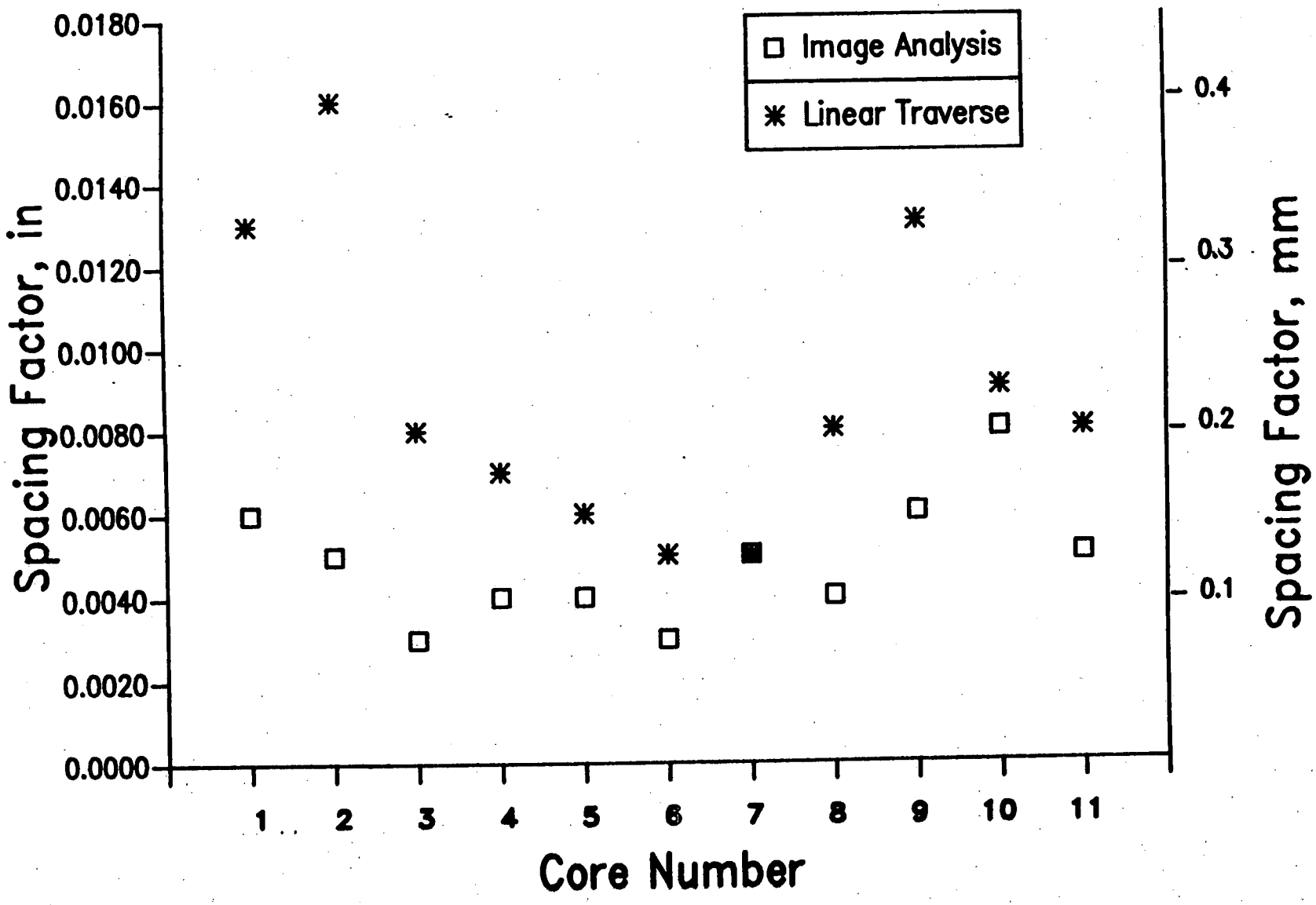
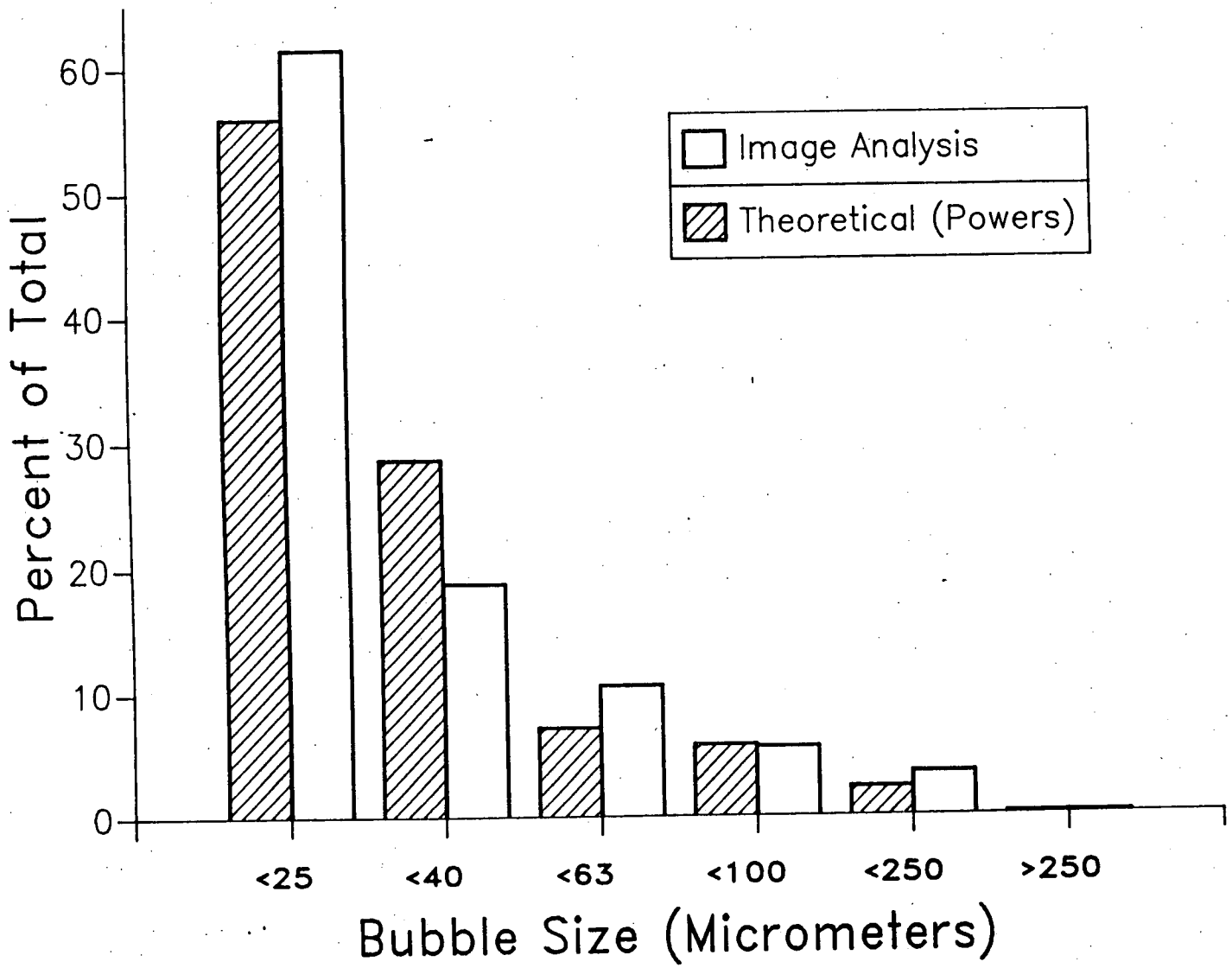


Figure 9 - Void Size Distribution
Theoretical Vs. Measured



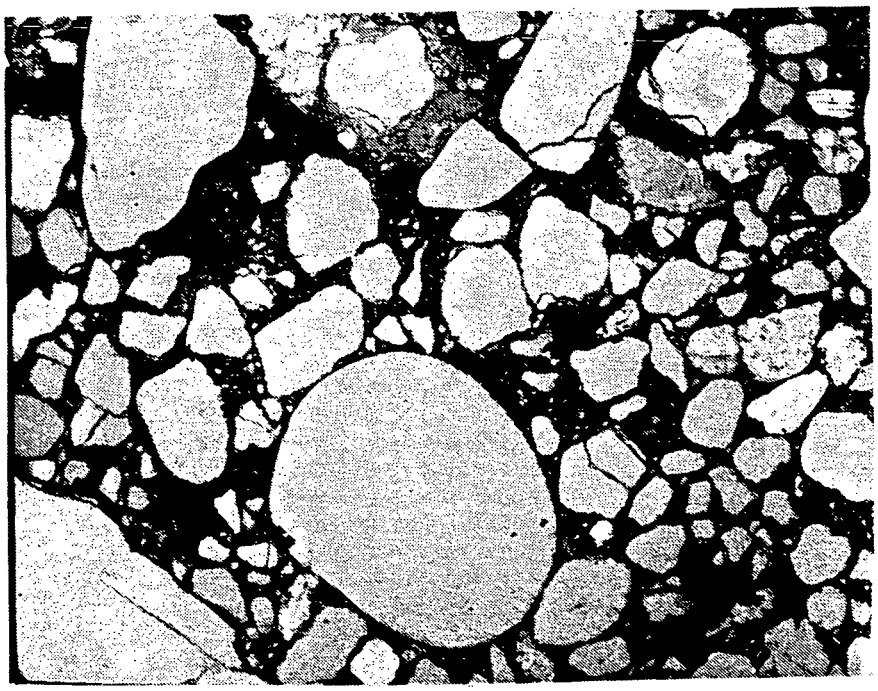


Figure 10 - BSE Image of Uncoated AC Core

10x Magnification