

Image Analysis of Portland Cement Concrete and Asphalt Concrete Pavements Using Scanning Electron Microscope Images

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The major objective of this study 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 demonstrated that the low vacuum scanning electron microscope is better suited to doing rapid analyses. The conventional high vacuum SEM can be used for AC and PCC analyses, but some distortion within the sample matrix will occur. Images with improved resolution can be obtained from SEM 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.

Determining air content and air void parameters in hardened PCC using the linear traverse or point count methods is tedious and time-consuming. Using digital image analysis to measure air voids is of much interest but the need to obtain sufficient contrast between voids and the surrounding matrix presents a serious obstacle. Although image analyzers typically are capable of differentiating at least 256 levels of gray, serious problems can result from uneven illumination, brightness fall-off of the camera lens system, or differential shadowing within voids viewed via a light microscope-based system. This paper describes two techniques developed by the authors to enhance the contrast of images obtained from the scanning electron microscope (SEM). A brief description of the instrumentation and techniques used is included.

IMAGE ANALYSIS

A thorough explanation of image analysis is beyond the scope or intent of this paper. Those interested in further information may find it elsewhere (1-3).

However, for those unfamiliar with image analysis, a definition of what it is, how it works, and what results can be expected may be useful. 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. Images analyzed in this study were obtained from an SEM.

Computers work with numbers, not images; thus the images obtained from the microscope must first be converted into numbers, that is, digitized. If an image is considered 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; one number defines brightness, and two other numbers define location, i.e., x and y coordinates. Typical image analysis systems use a numerical scale for levels of gray that ranges from 0 (pure black) to 255 (pure white).

Because the major concern is to distinguish air voids from surrounding matrix, an image may be simplified so that all pixels above a threshold value are presented as pure white and all pixels below the threshold are presented as pure black. Once such a binary image is obtained, a series of mathematical operations can be applied to the stored image to calculate, for instance, the percentage of air void area, by simply summing the number of pure white (or pure black) pixels. More complex operations can be used to determine void sizes, perimeters, edge-to-edge mean free path, for example, on the basis of gray level and spatial location. The key is to ensure sufficient contrast between air voids and matrix, that is, the aggregate and paste.

BSE IMAGING

A particularly useful method of imaging in the SEM uses backscattered electrons (BSE). In the BSE mode, signal intensity is dependent upon the mean atomic number of the specimen at the point of excitation. The strong dependency (Figure 1) has been used successfully to differentiate hydration states of cement pastes (4). However, BSE imaging is not capable of differentiating air voids, because the resulting gray level values are not unique.

HEAVY GOLD SPUTTERING

To accentuate differences between the aggregate/paste matrix and air voids, the authors hypothesized that an extremely thick coating of gold would provide a high-intensity signal from the matrix, whereas signals from voids would undergo more scattering and deflection with a resulting loss in signal intensity. Those familiar with electron microscopy may recognize that a sputtered gold coating 20 to 30 nm thick, is often used on specimens to provide a con-

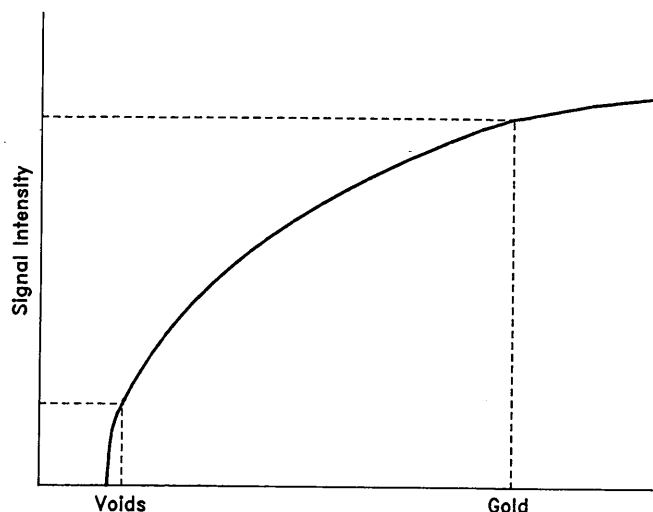


FIGURE 1 Relationship between atomic number and signal intensity.

ductive pathway and to eliminate "charging." To enhance signal contrast, a coating thickness in the range of 200–300 nm is necessary (Figure 2).

DISADVANTAGES OF SEM AND SPUTTERING TECHNIQUE

The purpose of this work was to develop a method to perform rapid analysis over the entire surface of 10-cm diameter portland cement concrete (PCC) cores. 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, because they set the stage for the development of a second technique.

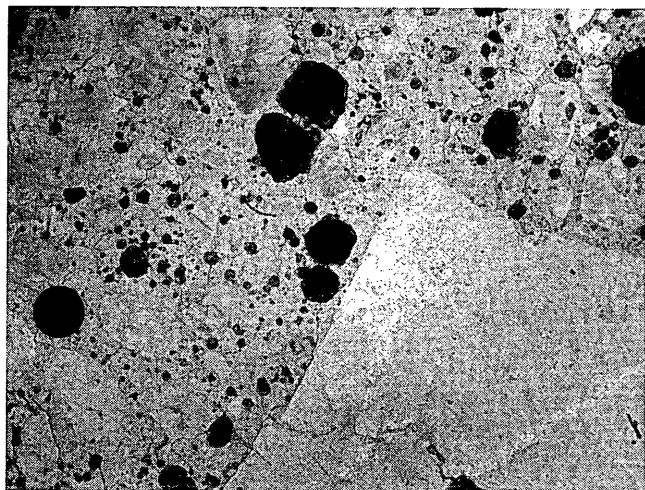


FIGURE 2 BSE image of heavy gold sputter coated PCC core.

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

Second, microcracking occurred on the sample surface because of the excessive vacuum applied to the samples. Although microcracks can be eliminated easily by image processing techniques, it is diagnostically useful to know whether microcracking has occurred before a sample is subjected to a vacuum.

In addition, several other limitations are imposed by the conventional SEM. Standard high vacuum SEMs 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 × 100-mm × 5-mm sample is beyond the capability of most conventional systems, and novel approaches to repositioning a sample while it is under high vacuum must be devised.

Finally, the minimum magnification of conventional SEMs is typically 10×, yet it is desirable to image at magnifications of 1× or 2× to cover a larger field of view, especially for characterizing large, entrapped air voids. With some instruments, it is possible to achieve 2× 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. Note that the extent to which a sample can be lowered beyond the normal position is also contingent on the amount of residual free play in the particular instrument's coarse focus control knob.

LOW VACUUM SEM

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

BARIUM SULFATE WITH RESIN TREATMENT

Barium sulfate paste (BaSO_4 suspended in water) is used routinely 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 with the SEM. The paste exhibits excessive drying shrinkage, especially in large or deep voids. That 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 g BaSO_4 to 5 g LR White. After thorough mixing, one drop of accelerator is added to the paste which is then stirred quickly until completely mixed. The paste is spread onto the core and worked in with a flat-edged blade held at a 45-degree angle. Excess paste can be removed easily with glassine paper.

The paste is allowed to cure for 15 min. 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 BaSO₄ resin treated core prepared by the procedure described is shown in Figure 3. Note that to obtain the level of contrast exhibited in the micrograph, minimal effort is required in optimization of SEM controls. A low- to mid-range setting of contrast control, moderate beam current, and very little time and effort are required to obtain satisfactory results.

EXPERIMENTAL ANALYSIS

A JEOL 840A high vacuum SEM operated at 25 kev in the BSE mode was used for this study. A magnification of 20 \times yielded images covering an area of 0.187 cm². Ten images were acquired randomly for each sample, providing a total analysis area of 1.87 cm². Although the sampling scheme required only about 15 min for image digitization and subsequent analysis of all 10 frames, the actual number of voids analyzed far exceeded the number obtained using the linear traverse system. The latter operated over a 241-cm (95-in.) traverse (Figure 4) and required about 7 hr.

Further work is necessary to evaluate optimum magnification and the number and location of areas to be analyzed. It appears likely

that a combination of analyses would be needed to accurately characterize any given core. For example, a 1 \times 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 \times –400 \times), as are required to elucidate entrained voids, are not useful for characterizing the entrapped air void system.

In addition, at issue is whether it is necessary to include large aggregate in an analysis scheme. Efficiency and accuracy in characterizing an air void system are increased if large aggregate is excluded from the area analyzed. Some coarse aggregate contains large air voids that inadvertently may be included by some operators. Using low magnification, may be possible to analyze the large aggregate fraction in a similar way to, or in conjunction with, the entrapped air void system.

A possibility may be to use compositional imaging. For example, characteristic X-rays detected by an energy dispersive spectrometer could be used to map elementally a sample with elemental concentration scaled to intensity of a unique color (5,6). The resulting images could then be analyzed for feature sizes by image analysis. Success of this method depends on the development of a vastly improved X-ray collection device, because high quality compositional images currently require up to 20 hr of collection time per image.

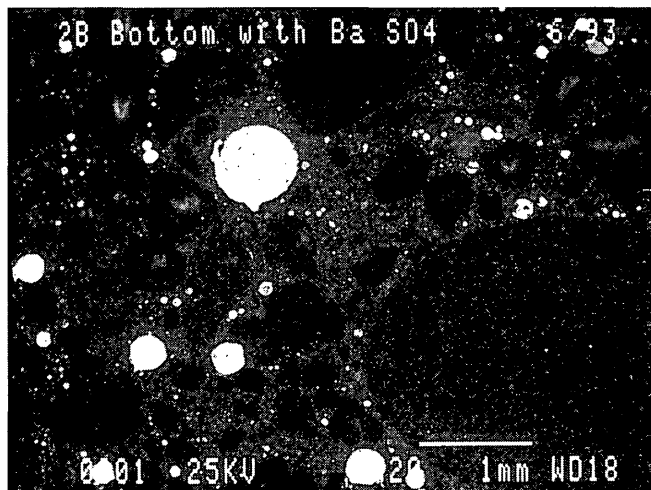


FIGURE 3 BaSO₄ with resin-treated PCC core, 14.8 \times magnification.

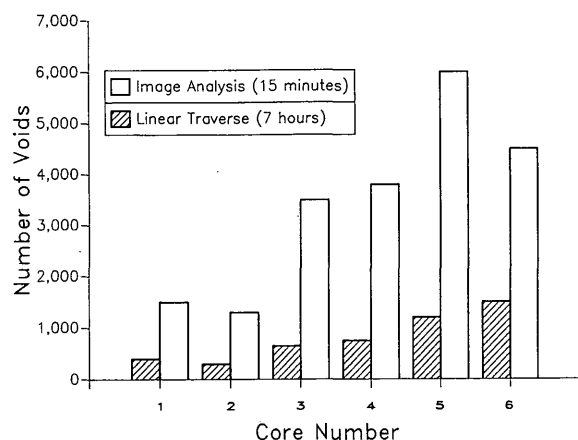


FIGURE 4 Number of voids detected by each method.

COMPARISON OF PCC VOID DETERMINATIONS

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

The American Concrete Institute *Guide to Durable Concrete* recommends 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)

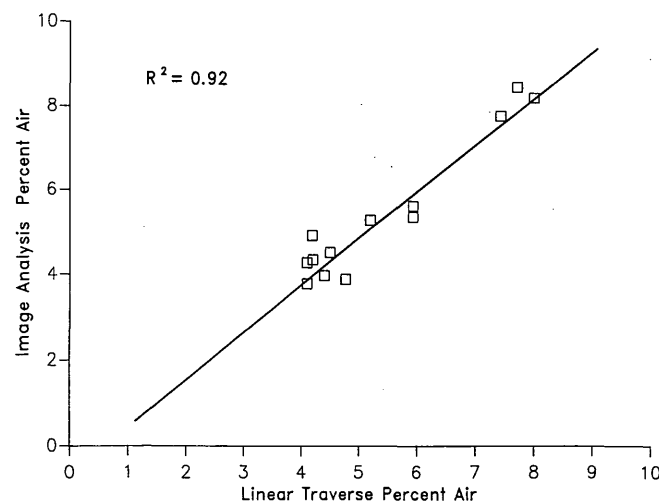


FIGURE 5 Percent air; linear traverse versus image analysis.

TABLE 1 Core Air Content Data

Core No.	Air Content		Specific Surface				Spacing Factor			
	Linear Traverse	Image Analysis	Linear Traverse		Image Analysis		Linear Traverse		Image Analysis	
			mm ² /mm ³	in ² /in ³	mm ² /mm ³	in ² /in ³	mm	in	mm	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	--	----	--	----	----	----	----	----

of greater than 24 mm²/mm³ (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 as compared with 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

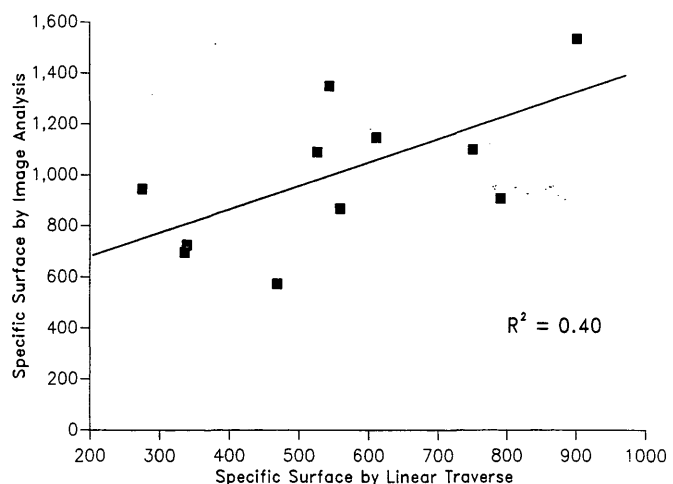


FIGURE 6 Specific surface; linear traverse versus image analysis.

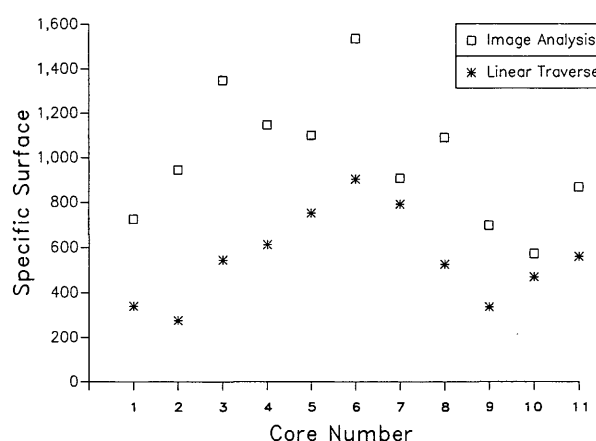


FIGURE 7 Specific surface values.

the linear traverse and that obtained by image analysis would clarify the reason for the two methods' differences in specific surface. As shown in Figure 4, image analysis detected more voids than linear traverse. Perhaps image analysis detected substantially more very small voids. Unfortunately, the linear traverse systems employed in this study were not configured to provide information on bubble size.

One rudimentary way to evaluate the image analysis bubble-size distribution is 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). 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

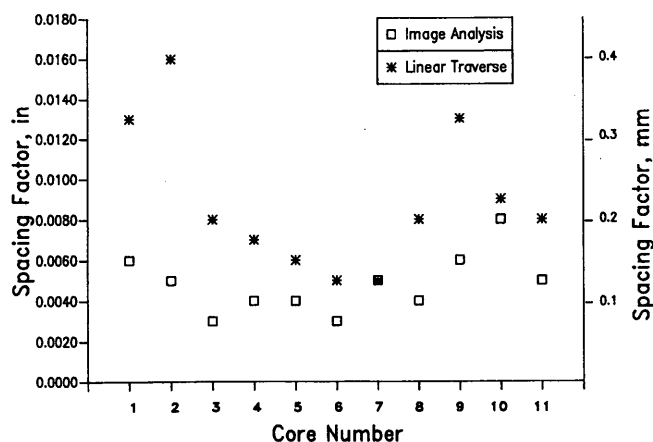


FIGURE 8 Spacing factor.

literature is less than that obtained by image analysis and, in fact, also is less than that determined by calculating average chord length using the published bubble-size distribution. Powers (8) notes, "The number of bubbles calculated from the specific surface diameter is considerably smaller than that calculated from size distribution. . . ." Ideally, it would be helpful to analyze hundreds of cores by each method; however, obtaining quality linear traverse results has proven difficult.

ADVANTAGES OF SEM IMAGE ANALYSIS

Analysis performed on SEM images offers much potential for understanding air-void distribution in concrete. Unquestionably, the speed at which a computer can classify and size objects is superior to manual methods. Many thousands of voids can be analyzed more accurately, providing more 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 the difficulty of achieving adequate contrast to allow successful automated differentiation of one object from another. To an image analyzer, all voids must be within a given range of gray values, and these gray values must be unique to voids. An image analyzer

cannot reason that a bright area within a void is part of the void. Image analysis systems lack the human brain's superb ability to interpret many such details. Light microscope-based analysis systems have inherent difficulties that make it difficult 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 on the basis of varying intensities 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 using a high atomic number material such as the BaSO_4 resin. The heightened difference in the signal results in significantly better images. BSE imaging yields far better resolution of voids' boundaries.

The newer LVSEM, with its ability to operate in a low-vacuum environment, is obviously the instrument of choice; although the conventional high-vacuum SEM will work, it may require pre-pumping of samples. Several benefits are derived from using the LVSEM, and they warrant further discussion. Adequate characterization of samples requires analysis of a number of cores, therefore minimizing specimen preparation and analysis time per sample is important. The LVSEM not only virtually eliminates pumpdown time, it significantly shortens specimen preparation, because BaSO_4 resin treatment takes only minutes.

Second, modifying an LVSEM to achieve lower magnification is much simpler. The specimen stage can be removed easily, as it is attached to the roll-out door (Hitachi instruments). The entire door and stage assembly can be removed and replaced with a fabricated plate. Further, because a high vacuum is not necessary, the replacement door is easy and inexpensive to construct. In this manner, the column is unobstructed, and a sample can be lowered farther 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-dimensional approximation of void content, whereas image analysis is a two-dimensional estimate. By observing the image, one can determine whether the void distribution is uniform.

ANALYSIS OF ASPHALT CONCRETE

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

Analysis of a 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

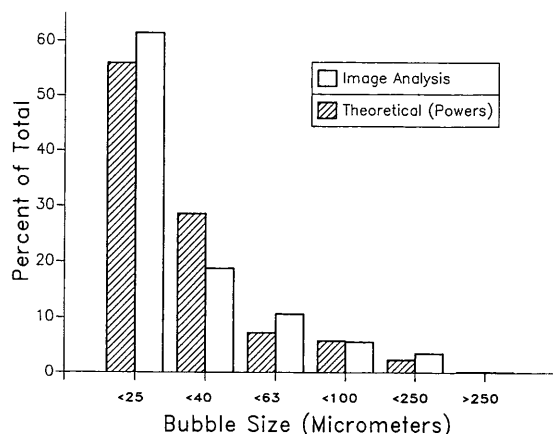


FIGURE 9 Void size distribution; theoretical versus measured.

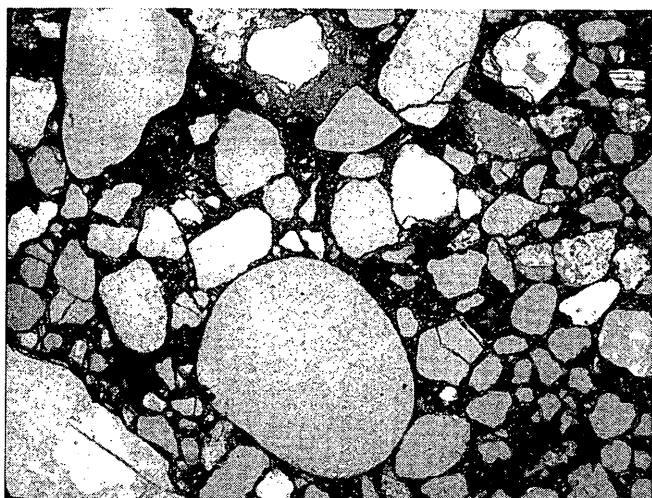


FIGURE 10 BSE image of uncoated AC core.

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, (a) analyze aggregate versus void and asphalt, (b) apply gold sputtering and analyze to delineate voids from aggregate and asphalt.

LIMITATIONS OF BaSO₄ RESIN TREATMENT

Implementation of the BaSO₄ treatment appears to be a more logical approach, because atomic number differences between AC, aggregate, and BaSO₄-filled voids would be significant and would permit a three 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 AC matrix. Water miscible resins have been used successfully, but further research is needed. The AC will appear nearly black in the SEM image and allow determination of the AC 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 1×10^{-6} torr, although no damage is apparent at approximate vacuums of 1×10^{-3} torr. The latter figure, 1×10^{-3} torr, was observed in samples viewed with a conventional SEM operating in a "poor" vacuum state. The instrument of choice for AC analysis is obviously the LVSEM.

CONCLUSIONS

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

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

- Gold sputter coating yields excellent contrast of voids in a BSE image for PCC or AC samples.
- Barium sulfate resin-filled voids yield an excellent BSE contrast of voids; voids were distinguishable from asphalt cement and aggregate in an AC sample as they were generally in a PCC sample.
- Image analysis of SEM images detects significantly more voids than does linear traverse.
- There is a good correlation between percent of air by image analysis and linear traverse.
- There is a poor correlation between specific surface by image analysis and linear traverse.
- Image analysis using SEM images consistently yields a greater specific surface and a lower spacing factor than linear traverse because it detects more voids.

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DISCUSSION

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One of the things the workers on concrete microstructure need is a guide to the use of image analysis. The authors have provided good material for that purpose. I trust their research gets studied by the ASTM Committee on Petrography of Concrete as material for revision of ASTM standard C 457 on the Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete.

The reason some people who look at the surface of a random plane through concrete see more and smaller sections of air voids has to do with the quality of preparation of such surfaces for examination (1; discussion). If preparation of the surface leaves roughness that cannot be distinguished from small air-void sections, those sections will not be detected. Yet the authors do not discuss how their image analysis procedure detects such small sections when linear traverse fails to do so, even though no different degree of surface preparation was involved.

Remember that there are no very small air bubbles. There are, however, very small air-void sections on surfaces when the plane of

the surface intersects a void near the point of tangency of the surface and the void. Such small sections are very shallow when the void is in the removed concrete and, in comparison, very deep when the void is mostly below the surface. The former are very hard to detect; the latter are often artificially enlarged in surface preparation—more so the weaker the paste and the larger the void. Procedures for calculating air-void parameters from air-void section data were given elsewhere (1–4).

The authors do not compare any of their results from tests of hardened concrete with actual values of air content of unhardened concrete. There has been much discussion of why values for air content of hardened concrete do not agree with those of unhardened concrete. Presumably logical reasons were adduced for why the air content of hardened concrete should be lower than that of fresh concrete or why that of hardened should be higher than that of fresh concrete. My view is that if one has a properly adjusted apparatus and uses it in accordance with ASTM C 231, allows the concrete sample so tested to harden in the bowl of the pressure meter, and then takes appropriate samples of the concrete for test by ASTM C 457, either point count or linear traverse, all three will agree. They will agree, that is, so long as things that look like air-void sections on the surface, but are not, are not counted as air voids. The pseudo air-void sections that I am most aware of are cenospheres from fly ash and fallen out spherical sand grains cut below their equatorial plane when the surface to be examined was “facing up” as the concrete hardened. In that case, there is a lack of bond at the underside of a sand grain from bleeding. If one does not fill the holes on the surface and looks at it carefully he or she can tell a bleed-water lined void as under a sand grain or one lined by glass from one lined by the “soap” film surrounding an entrained-air void. In the case of cenospheres, giving the prepared surface a light acid etch will cause a collar of glass to be revealed at the edges of cenospheres. Part of the variation between image-analysis results and linear-traverse results found by the authors may be attributable to discriminating observations made by an operator, which the image analyzer cannot make because the holes (air voids and others) are filled before examination.

Advice given by Walker (5) regarding air voids in hardened concrete merits careful study. In her discussion of image analysis she comments

... systems that require filling the voids (thus hiding their interior surface) cannot be used to make certain distinctions possible by a human operator, can often mentally reconstruct what the surface if this or that flaw had not occurred. The human operator can judge if a void observed is an air void, a fly ash cenosphere, or the hole left where a small round grain of sand has fallen out.

In her discussion of fly ash (6), she wrote

The walls of the cenospheres are frequently thin enough and the cenospheres large enough to be mistaken for entrained air voids. To detect and distinguish from air voids, etch, rinse, blot, and examine with the stereomicroscope. The glass walls can be seen with low-angle illumination projecting above the paste.

I invited my colleague, G. Sam Wong, to comment on the foregoing discussion. He added the following (personal communication):

I agree with your comments and to take it even further concerning the differentiating of material such as cenospheres, there is also the differentiation of paste from aggregates. This is easily done by the petrographer using a microscope; this is not done so easily using image

analysis. Chemistry is often not useful since most elements that are common in portland cement are also common in aggregates. Color often is not useful since the color of aggregate may have similar qualities as that of portland cement paste. They did not show how they distinguish paste from aggregate. It is easy with asphalt if you do not use a black aggregate. The voids filled with some sort of enhancing substance has been used successfully and it suffers the same sort of programs using light microscopy for image analysis as that in the SEM.

On page 8 they mention the examination of 10 random images of 0.187 cm^2 . I calculate this to make an area which would be 4.3 mm on a side. Sand begins at 4.75 mm, which would mean that if they took a representative sample and they have 75 percent aggregate in the concrete then 75 percent of the sections examined would have nothing but aggregate in them and that would include the fine aggregate. It would be impossible to see a single coarse aggregate particle. I believe that it is only by good fortune that the figure 5 information is like it is. Total area with paste and voids that would be available for examination would be 25 percent of 1.87 cm^2 or 0.4675 cm^2 .

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AUTHORS' CLOSURE

Image analysis performed on SEM images detects more small air voids than does linear traverse performed via light microscopy. Unfortunately, the linear traverse systems used in this study did not provide individual measurements on air-void sizes, so direct comparison of size distributions was not possible. However, we do know that the average chord lengths provided by linear traverse were larger than that obtained from image analysis of SEM images.

Powers (8) offers one clue for that difference in a discussion of the linear traverse, in which he states, “. . . accuracy is limited by the resolving power of the microscope.” A typical light microscope has a resolving power (the ability to differentiate two objects as being separate entities) of about 2,500 angstroms. A modern SEM typically has a resolution better than 35 angstroms, which represents a very significant improvement above the light microscope's ability to resolve small objects.

Further, the SEM is noted for its increased depth of focus, which makes it much easier for an operator to view the magnified specimen clearly. Light microscopes require constant focusing because of the poor depth of focus; that, combined with their much poorer resolution, serves to render even the most dedicated operator eye weary in a short period of time. Analyzing a linear traverse via the light microscope is indeed a time-consuming and monotonous task, subjecting an operator to continual eye strain, and boredom can have very serious consequences.

Two points Walker makes regarding the identification of plucked sand grains, fly ash cenospheres, and surface flaws bear discussion.

First, I agree that filling of the voids can obscure fly ash cenospheres and plucked sand grains. In fact, before filling voids with BaSO_4 , we always make a cursory examination of cores with the SEM. A few minutes' examination gives one a clear picture (much more than viewing them with a light microscope) and can be extremely useful for resolving poor sample preparation or the distribution of small and large aggregate and air voids, generally. In fact, during one particular analysis, we were able to determine quickly the presence of numerous cenospheres that, upon subsequent examination using the light microscope, were seen only with much difficulty. The difference in the two imaging systems' ability to identify minute structures is dramatic. Again, the resolving power and depth of focus of the SEM is unquestionably far superior to the light microscope; it serves to greatly aid an investigator's efforts and provides an improved method for differentiating minute structures.

Second, I am less concerned about a few random sand grains plucked from a sample than I am about the total number of voids measured. A case in point is illustrated in Figure 4. A 7-hr linear traverse detected and measured approximately 1,200 air voids; whereas, with the image analyzer, a 7-hr analysis would have measured approximately 168,000 voids on the same sample. The dramatic speed provided by the image analyzer means that one can analyze many more cores instead of relying on the questionable practice of analyzing only a few.

Assuming one does not intend to fill the voids, the gold-coating technique is the method of choice. Figure 2 indicates that, even with a heavy gold-coating, one can still differentiate fine and large aggregate and, thus, presumably cenospheres. If in fact there is a question regarding cenospheres, the energy dispersive spectrometer (attendant on most SEM'S) can provide the conclusive chemical analysis within seconds. The interactive software on image analysis systems can be used to quickly subtract the voids from analysis—as well as from dislodged sand particles. The chief drawback to interactive image processing, however, is that it requires an operator's interpretation, which dramatically increases analysis time.

It has nearly been 50 years since the linear traverse came into use; it needs to be replaced with a more efficient method. Although many questions remain regarding image analysis, it has tremendous potential. To adequately characterize the air content in a road, one has to measure a very large number of voids. Linear traverse does not allow one to do that economically; it is that simple.

Just as simply, we need to be able to measure voids long before the road has been finished, because once the road has been built void content is somewhat of a moot point. I question whether we should relegate highly trained petrographers to task of sitting before a light microscope hour after hour to perform simple duties.

With respect to using color for analysis, electron microscopes "see" shades of gray only, so color is not an issue. In truth, color is not a very useful characteristic for distinguishing voids; the use of BSE offers much more potential. As the number of BSE is related to atomic number, tremendous signal differences can be achieved between the hydrocarbon asphalt cement and aggregate, regardless of color. BSE imaging (See Figure 10) is straightforward and produces outstanding images. BSE analysis can be performed with very little effort to determine in-place gradation of aggregate. In

terms of concrete, published research demonstrates the ability to quantitatively determine W/C ratios and hydration states of paste using the electron microscope in the BSE mode. A challenge for earlier investigators was to successfully differentiate air voids; this paper demonstrates two methods for doing that.

In addition, there are BSE systems that reportedly are capable of measuring .1 differences in atomic number. Although we have not used a sensitive BSE detector, it would be interesting to view concrete and asphalt with such a system.

I too was initially surprised with the high correlation between linear traverse and image analysis in terms of percent of air. I had expected correlation to be very poor considering the extremely low number of voids measure by the linear traverse in combination with human error. However, percent air is dominated by the larger bubbles that the linear traverse can resolve, and percent air is a fairly crude measure. The last reason is justification for calculating either specific surface or the spacing factor; unfortunately, even these measures fall short because they rely on average chord length and really do not consider large discrepancies—beyond which basic equation to use. One must calculate the average chord length as accurately as possible; if one cannot resolve smaller voids, one cannot measure them.

With respect to area analysis, I am not sure whether Mather means to discredit its use or only challenge our not collecting a sufficient number of frames. If we are willing to accept lineal analysis (or point count), we must accept area analysis because the stereological development of the two methods relates to the assumption that $V_v = AA = LL$.

Regarding the number of frames collected for our comparisons, I agree that more area is desirable; even 50 or 100 frames (total analysis times of approximately 1 or 2 hr respectively) may be warranted if one wants to thoroughly characterize a core. A good statistician, however, would recommend reducing the area scanned on one core and greatly increasing the number of cores analyzed. In future, one might wish to forget about measuring the large aggregate and collecting information from the paste fraction only. If interested in air content one might not want to waste time analyzing large aggregate. Also, it may be better to obtain only five frames of data on 10 cores, instead of 50 frames of data from only one core. However, from just five frames, one can get a reasonable estimate of what is happening on any particular core. If results indicate a possible problem, then more extended analysis is warranted; otherwise it is better to move on to another sampling location. Statistically, the analysis of one core, whether by image analysis or linear traverse, is of little use. I believe that what we will see in the future is a totally new way of measuring things, accompanied by a different set of measurements. One could argue the virtues of linear traverse or image analysis until blue in the face. What we really need to concern ourselves with is how to assess the quality of the roads as we build them, not after we build them. We cannot do that with linear traverse.

The contents of this report reflect the views of the author and do not necessarily reflect the official views of ISU and the Iowa Department of Transportation. This report does not constitute any standard, specification, or regulation.

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