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Considerations in image analysis as applied to investigations of the ITZ in concrete

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Abstract

A number of considerations are discussed with regard to the study of the Interfacial transition zone (ITZ) in concrete by image analysis of backscatter SEM images. These include selection and sampling of the concrete, selection of individual aggregates for the ITZ measurements, and design and positioning of individual sampling units around the chosen aggregates. Emphasis is placed on the importance of analyzing the lateral variation in properties around the aggregate as well as variation in mean composition with distance from the aggregate. Statistical considerations that can be applied to the interpretations of the results are illustrated.

Keywords: Interfacial transition zone (ITZ); Image analysis; Microstructure; Backscatter images; Scanning electron microscopy (SEM); Statistics

1. Introduction

Investigation of the internal microstructures of concretes by backscatter-mode scanning electron microscopy has become a relatively common technique in the last few years. In addition to qualitative investigations, various image analysis programs have been used to develop quantitative descriptions and comparisons.

In such microstructural studies of concrete, methods of field image analysis are commonly applied to determine the proportions of the various distinguishable components of the hardened cement paste (hcp). Examples of such field image analyses studies for cement pastes include those of Scrivener et al. [1], Wang and Diamond [2], and Diamond and Wang [3]. The components of the microstructure that are specifically analyzed usually include pores, calcium hydroxide (CH), and residual unhydrated cement. C–S–H, the major hcp component, is usually not susceptible to direct quantification, but its content can be estimated by difference between the sum of the areas of the other components and the total paste area. Ettringite is not usually determined, except in special studies relating to delayed ettringite formation or sulfate attack.

The field image analysis procedures commonly used are straightforward. Digital images are obtained by backscatter SEM on representative areas of polished surface specimens. Pixels corresponding to each of the components are separated by binary segmentation (i.e., thresholding) based on gray level. The percentage area of each component is determined by dividing the number of pixels of each component by the total number of pixels in the field. This percentage area is assumed to provide an appropriate estimate of the volume percent of the component. In studies of concrete, it is common to disregard the pixels corresponding to the aggregates, and express the area percentages of the different hcp components on the basis of the area of the paste, rather than the area of the concrete.

The binary segmentation process within the hcp is less than perfect. It is generally easy to accurately separate residual unhydrated cement at the bright end of the gray scale from the other components. Pores, usually filled with epoxy resin in specimen preparation, can be fairly accurately segmented at the dark end of the gray scale. The segmentation of CH from the only slightly darker C–S–H is slightly more uncertain.

One unavoidable difficulty is that pixels that are at a borderline between two components exhibit a gray level intermediate between them, and are usually misallocated.

There is also a special difficulty with respect to evaluating the percentage area of pores. The area percent

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that is tallied cannot include pores smaller than a single pixel; thus, very fine capillary and 'gel' pores cannot be separated by the gray level thresholding, and are necessarily included in the area of undifferentiated C–S–H.

In the present paper, the writer discusses the practical aspects of a specialized form of such field image analysis applied to the study of the interfacial transition zone (ITZ) in concrete. The object is to establish the degree to which the proportions of components within the ITZ of a given concrete differ from that of the 'bulk' hcp of the same concrete.

Following the lead of Scrivener et al. [4], it is customary to analyze the paste in successive narrow strips or sampling units arranged in stacks starting at the paste-aggregate interface and proceeding through the ITZ and beyond it into the bulk paste. An illustration showing a backscatter image of an area in a 3-day old quartz aggregate concrete is shown in Fig. 1(a). Portions of two adjacent sand grains are visible. To the right of Fig. 1(a) are grouped four versions of a single strip, 0–10 μm wide, representing the innermost portion of the ITZ adjacent to the sand grain on the left portion of Fig. 1(a). For ease of comparison, the images are oriented parallel to the sand-grain perimeter in Fig. 1(a). The first separated image, Fig. 1(b), is the gray level image; the remaining images, Figs. 1(c)–(e), indicate the locations of the pixels identified as pore space, calcium hydroxide, and residual unhydrated cement, respectively, as determined by the segmentation process.

In the analysis, the percentage area corresponding to each of these components is compiled for the strip. Similar information is obtained from a second parallel strip 10–20 μm from the sand grain, a third parallel strip, and so on. In contrast to the usual field analysis which aims at determination of overall composition, in ITZ analyses, it is the variation of composition with distance from the sand grain that is of primary importance.

An example of the results obtained in such investigations is provided in Fig. 2, containing data taken from the results of Huang [5] at Purdue University. In this figure mean values of percentage area found versus distance from the interface are given for detectable pores, unhydrated cement, and CH. The concrete in question is a 100-day old $w : c$ 0.5 concrete batched with dolomite sand and coarse aggregate. The points are plotted at the distance from the centers of the sampling units to the aggregate surface as measured on the plane of the specimen.

The data plotted in Fig. 2 show that in this case, even after 100 days of hydration, the ITZ clearly exhibits the effects of exclusion of cement grains commonly considered to be the basis of the ITZ effect. However, percentage area of pore space found in the ITZ is less than 10%, a modest value compared to the range of 30% or thereabouts indicated by other workers. The content of CH is relatively high, and is higher in the innermost unit. Not shown in the figure is the content of "C–S–H" estimated by difference between the sum of the components shown and 100%. This tally (which includes minor amounts of intermixed ettringite, perhaps other minor hydrated phases, and very fine pores) covers about two-thirds of the paste, and is much the same within the ITZ

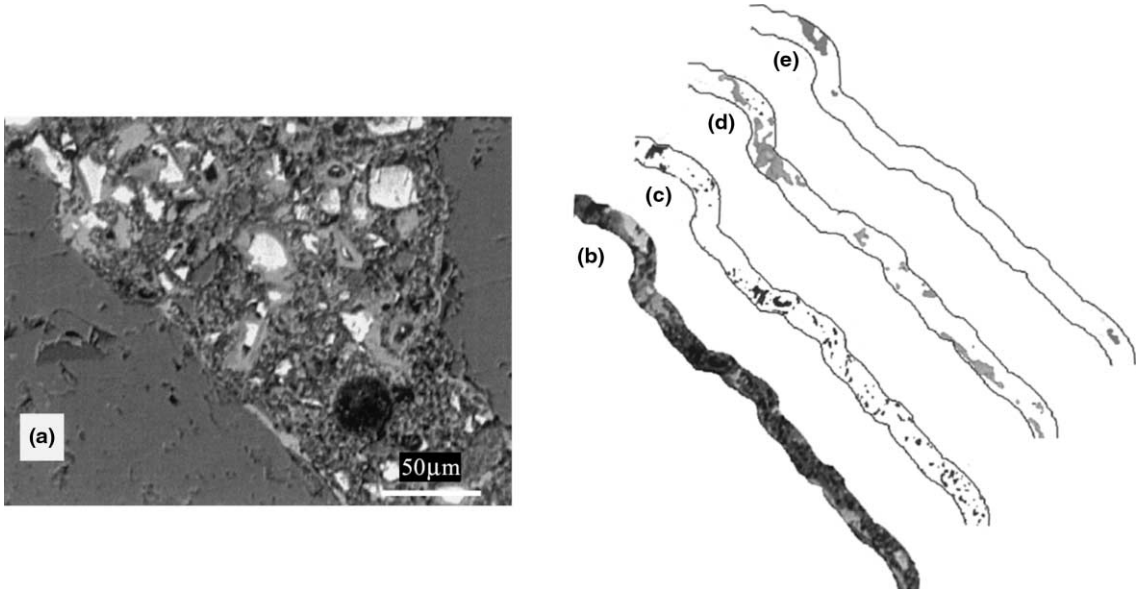

Fig. 1. Illustration of the analysis scheme: (a) shows the area around a sand grain to be analyzed; (b) shows the first sampling unit adjacent to this portion of the sand grain; (c), (d), and (e) show the locations of the segmented pixels within the unit corresponding to pores, calcium hydroxide, and unhydrated cement, respectively.

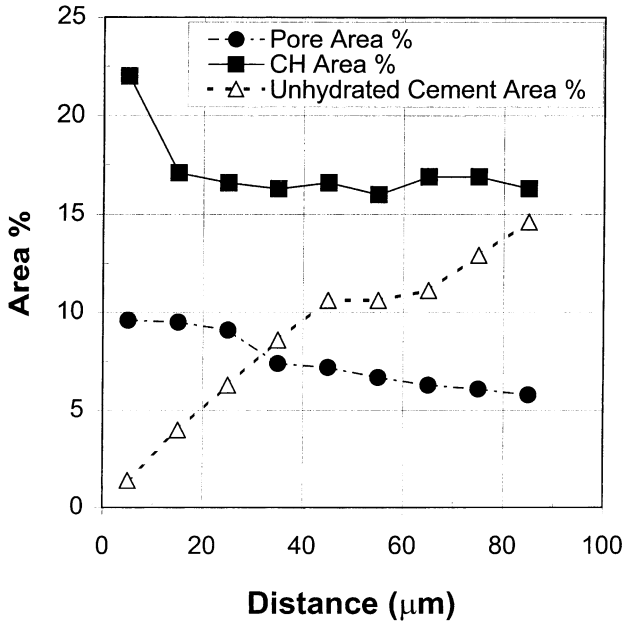

Fig. 2. Mean values of pore percentage area, CH percentage area, and unhydrated cement percentage area with distance from the aggregate for a particular concrete.

as it is in the bulk hcp. The significance of results such as these is explored in a companion paper in this Symposium [2].

The relations found in Fig. 2 are not atypical for well-mixed plain portland cement concretes, but results may be expected to vary depending on cement content, $w:c$ ratio, sand and aggregate compositions and size distributions, age, presence of supplementary cementing components, use of superplasticizers, and many other factors. Thus the details of the ITZ vary from concrete to concrete, and may require specific investigation in a given case. It is the purpose of this paper to review some of the considerations that arise in designing and carrying out such ITZ investigations and in interpreting the results obtained.

2. Choice of concrete for investigation

The concrete to be examined may be sampled from field structures, from industrial production, or from concrete or mortar prepared in the laboratory, depending on the purpose of the investigation. It should be noted that not all concretes lend themselves equally well to ITZ investigations.

One consideration is the mineralogy of the aggregate used, and specifically the range of gray levels produced by the minerals present. Major differences in gray level often occur between different aggregates and in some cases within different parts of individual aggregate grains, due to compositional variations; these often overlap the gray levels of the cement paste components to be analyzed. An illustration is provided in Fig. 3 taken from an industrial concrete examined by the writer in the course of his consulting activities. This difficulty may be circumvented, since many programs permit the investigator to accurately delineate the

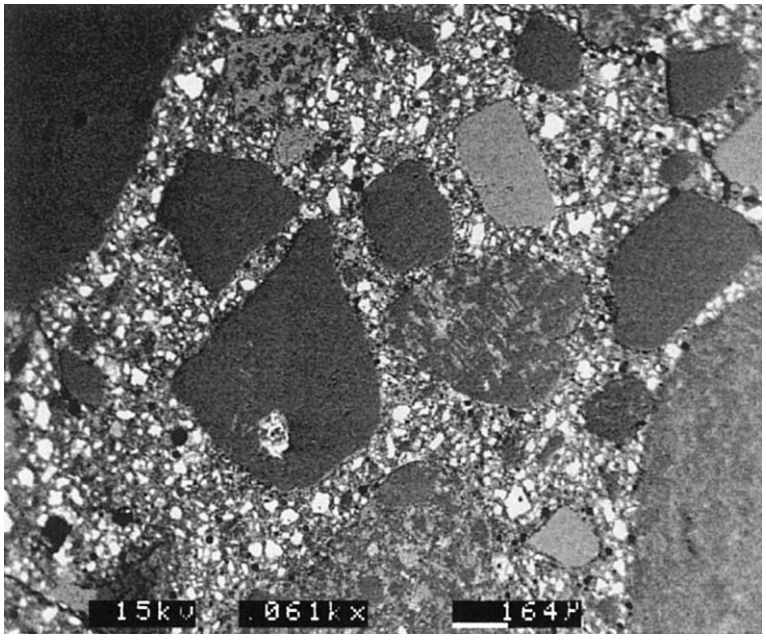

Fig. 3. Illustration of a concrete in which the gray levels vary between and within aggregate grains, making automatic delineation of the edges of aggregate difficult to accomplish.

boundary of the aggregate and remove all of the aggregate pixels from the image. Unfortunately, given the variation in gray level of aggregates in the general case, this is not a procedure that can be easily automated.

Some concretes incorporate supplementary cementitious components that can give rise to a variety of difficulties. Fly ash particles, for example, vary extensively from each other in chemical composition, and most are internally heterogeneous as well. When incorporated within the hcp in concrete they commonly produce gray levels overlapping, and indeed transcending, the normal gray level range. Carbon residues in some fly ashes give rise to dark gray levels that are difficult to segment from those of the epoxy-filled pores. Concretes with silica fume also present special features. As is now well understood, many commercially available silica fumes contain undispersed agglomerated particles ranging up to several hundred μm in size. Their gray levels are typically in the darker part of the C–S–H range, making them difficult to segment from other hcp components. Illustrations showing this feature have been provided by Scrivener et al. [6] and many others.

A further complication common to nearly all concretes (except those mixed under vacuum) is the presence of air voids. Air voids are important components of field concretes and are often themselves the objects of image analysis. In normal specimen preparation for backscatter SEM analysis they are filled with epoxy and consequently exhibit the same gray level as "capillary pores", i.e., smaller pores within the hcp. As will be indicated later, in ITZ studies their presence is a particular cause of complication, and a definite procedure must be established for dealing with them.

The previous considerations apply both to field and to laboratory-mixed concretes. One special concern that arises with laboratory-mixed concretes (or mortars) is that fairly arbitrary choices are often made by the investigator with respect to mix design and mixing procedures. If the object of the investigation is to study the properties of the ITZ as it occurs in 'normal' concrete, the mix design used should bear at least some resemblance to a normal concrete mix design. Similarly, laboratory mixing procedures that to some extent resemble those used in practical concrete production should be employed. Unsophisticated use of the so-called 'model systems' employing little mixing (or no mixing at all) in various ITZ studies in the past has led to misinterpretations of the nature of the ITZ in concrete by the present writer and others.

3. Sampling and specimen preparation

Image analysis is carried out on small specimens obtained by sampling much larger concrete masses. Various considerations concerning the way the concrete mass is sampled and the specimens prepared and produced are of concern.

One concern is that for most purposes the portion of the concrete to be sampled should be selected from areas not influenced by special characteristics. For example, areas near the concrete surface are likely to be carbonated; specimens taken from such areas likely will yield atypical results for CH, and perhaps for pore content and unhydrated cement content as well. Similarly, areas in which extensive cracking has occurred, and areas influenced by sulfate attack, alkali silica reaction, or other forms of attack which affect concrete microstructure would be poor choices, unless the object is to specifically study the effects produced by these external influences on the ITZ. Also, portions of concretes that may have been subject to bleeding effects should ordinarily also be avoided.

Having selected the portion of the concrete to be sampled, the customary procedure is to cut a thin, often wafer-shaped portion from the area to be studied, dry it in a way that does not introduce excessive cracking, impregnate it with epoxy resin, harden the resin, produce the surface to be examined using a diamond saw, and finally lap, polish, and coat the surface in the appropriate manner. The details of such procedures have been provided by various authors, including for example Stutzman and Clifton [7]. Ultra-low-viscosity epoxy resins marketed by several manufacturers can be used. In our procedures and those of others, "dry potting" is usually employed; sawn specimens are gently dried at low temperature, immersed in the ultra low viscosity epoxy, and a vacuum is applied and then released to force the epoxy into the pores. Where the object of the investigation involves delineation of existing cracks, the epoxy can be introduced without drying by replacing the pore solution with ethanol and subsequently replacing the ethanol with the epoxy resin. In either case, low-temperature curing is usually necessary to harden the resin. A fresh surface is then normally generated by slow cutting using a low-speed diamond-blade saw, lubricated with propylene glycol or other non-aqueous fluid. The cutting of the new, impregnated surface is then followed by the grinding and diamond-polishing steps needed to create a smooth, plane surface for SEM imaging. The process of correct specimen preparation is an absolute requirement for obtaining appropriate image analysis results.

4. Selection of areas of the specimen to be analyzed

A critical feature in ITZ investigations is the selection by some sampling process of the aggregate grains to be studied, since each ITZ area to be studied necessarily is

associated with and surrounds some particular sand or coarse aggregate grain. A single SEM specimen surface may contain hundreds of sand grains of various sizes as well as one or more coarse aggregate grains. Some unbiased sampling procedure is necessary to select the relatively small proportion of the grains that are to be the centers of specific ITZ areas to be examined.

One reasonable procedure, used in the writer's laboratory, is to subdivide the specimen surface into a set of numbered areas of equal size. A numbered area from which the first aggregate grain is to be selected is chosen from a random number table. A transparent two-dimensional grid with numbered intersections is placed over a low-magnification micrograph of the area, and a pair of random numbers is drawn to select a candidate grid intersection. If the candidate grid intersection falls within aggregate grain, then that grain is chosen for the ITZ study. Once a grain is selected from a given area of the specimen, a new area is again chosen randomly, and the aggregate-selection process is repeated within this new area. The process is continued until the desired number of aggregate grains has been selected.

If the hcp around the entire periphery of the grain is to be analyzed, no further random selection procedure is required. If only a single stack of sampling units covering a small part of the periphery is to be analyzed, prudence would suggest that some definite randomization procedure be adopted in selecting the location of the part of the aggregate periphery that is to be studied. As will be indicated subsequently, there is significant variation from place to place around the same sand or coarse aggregate grain. The possibility of conscious or unconscious selectivity on the part of the operator cannot be overlooked.

5. Sampling units

As previously mentioned in ITZ image analysis studies, the analysis is carried out on narrow sampling units arranged successively in stacks in the direction normal to the perimeter of the aggregate. These sampling units are usually 5 or 10 μm wide, although sometimes even smaller widths have been used [8]. One factor that influences the selection is pixel size, which depends on the digital resolution and magnification to be used. It is common to use a magnification of 500× to provide an appropriate compromise between area covered and the details required, and to use a digital resolution of 512×512 pixels in the overall image, nominally 10 cm in width. These choices fix the pixel size at just slightly smaller than 0.5 μm in each direction. Thus 5-μm wide sampling units are 10 pixels in width, and 10-μm wide sampling units are 20 pixels in width.

The length of the sampling unit in the direction parallel to the aggregate interface is also influenced by the magnification used. At 500×, the length of a typical unit to be examined is usually about 250 μm. As seen in Fig. 1, the actual outline is irregular, and follows that of the perimeter of the sand grain. Such a unit contains either about 5000 pixels or about 10 000 pixels, depending on whether the 5 or 10 μm width was chosen. It should be emphasized again that the sampling units are not rectangular, but follow the local curvature and configuration of the aggregate perimeter.

There is a statistical concern that impinges on the minimum size of a sampling unit, i.e., the minimum number of pixels it should contain. If variation between individual sampling units is to be studied, it is desirable to have the sampling unit large enough so that the tally of pixels registered for each component within it is reasonably accurate, so that the results for each unit are not greatly affected by random statistical errors stemming from counting small numbers of pixels. This is important if statistical variations between units of the same nominal position are to be evaluated.

A formula derived from counting statistics is available to estimate the number of pixels required for a given degree of accuracy. The relative standard error of the count is given by $1/N^{1/2}$, N being the number of pixels counted. As an illustration of how this works out in practice, consider a hypothetical 10 000 pixel unit yielding a pore space tally of 8 area percent. This means that the number of individual pore space pixels in the unit is 800. The purely statistical uncertainty of this allocation is thus $1/800^{1/2}$, i.e., 3.5% of the 8 area percent estimate. For a 5000 pixel unit of the same area percent tally, the statistical uncertainty is somewhat greater, i.e., $1/400^{1/2}$, or 5%.

Of course, the allocation provided by the binary segmenting operation is never perfect, and the statistical accuracy is only one component of the overall accuracy. A major difficulty inherent in the thresholding process is that boundary pixels forming the border between two different components exhibit a gray level intermediate between them, and are thus often misallocated.

It appears to have been common practice to analyze only a single stack of ca. 250-μm long units for any one sand grain that is studied. For a given effort, this allows one to sample the ITZ around a large number of different sand grains. The percentage area values for a given component in the first-tier (say 0–10 μm) sampling units are averaged over all of the sand grains. The same is done for the second, third, and all subsequent tiers. A profile of *average* percentage area versus distance for each component can thus be generated. Interpretations can then be made from the profiles as to how and to what extent the hcp within the ITZ differs from the

'bulk' hcp, and to what extent gradients of composition with distance from the aggregate can be established.

Such a procedure is extremely useful. However, it does not allow evaluation of the local differences that may be characteristically encountered as one moves around the aggregate grain at a given distance from it.

An alternative procedure can be followed, in which one can choose to analyze the ITZ around fewer sand grains, but to examine the total ITZ around each of them. This procedure has been used in the writer's laboratory. In this procedure, stacks of ca. 250-μm long sampling units are placed adjacent to each other so as to completely surround each grain. We find that between 8 and perhaps 20 side-by-side stacks are required to cover the periphery of most sand grains. Very large sand grains and, of course, coarse aggregate grains, require many more.

This procedure has the advantage that the collection of data from adjacent sampling units at the same distance from the aggregate permits evaluation of local variations in the ITZ around the grains studied. It turns out that the variations within a given tier around the periphery of the grain are often as great as, or greater than, the mean ITZ effects as customarily measured [9].

The two approaches could certainly be combined. Coverage of a portion of the ITZ around each grain using a smaller number of side-by-side stacks that go part way around the perimeter would permit some evaluation of lateral variation, while permitting the study of a ITZ around a larger number of individual grains.

6. Special considerations in carrying out ITZ analyses

Air voids have been mentioned previously as a significant feature of concretes, whether the concrete is deliberately air entrained or not. Air voids are typically much larger than the paste pores within hcp, ranging up to several hundred μm in size. Thus a given air void may lap over a large number of sampling units within a stack or over adjacent stacks, and its effect may completely dominate local areas of what is nominally the ITZ. Thus studies of the ITZ require some procedure for handling air-void effects when encountered in a zone selected for analysis. Fortunately, air voids are easily recognizable by their circular shape on the plane of the exposed specimen surface. It is usually practical to either reject a specific aggregate from the evaluation if its surrounding paste contains air voids, or alternatively, to reject individual sampling units from the tally if any portion of an air void appears within its boundary.

Another important concern that occasionally arises is the proper placement of the boundary of the first-tier strip with respect to the actual surface of the aggregate. Occasionally thin gaps or 'bond failures' are found separating a portion of the periphery of an aggregate from the surrounding paste. This is not a bleeding effect, but one inherent in some concretes; it may be augmented by specimen preparation. It is generally agreed that such areas should not properly be counted as part of the 'pore space' of the innermost sampling unit of the ITZ. Accordingly, in such cases care is taken to avoid this by positioning the innermost surface of the first-tier strip at the boundary of the paste rather than at the surface of the sand grain.

Finally, a specific protocol needs to be obeyed with respect to limiting the number of successive strips to be analyzed in a given stack. The reasonable requirement is that successive strips should not be analyzed beyond the half-way point to the next aggregate grain in the direction concerned. It is apparent from Fig. 1 that sand grains are not infinitely distant from one another. It is obvious that if one continues beyond the half-way point, the influence of the next grain becomes increasingly felt, and the purpose of the ITZ analysis is confounded. This constraint causes variation in the number of units that should be analyzed in different directions around a given grain. Indeed, even in normal concretes it is not uncommon to find occasional sand grains positioned so close to each other that only a few sampling units can be fitted into the narrow gap between them, and the typical ITZ gradient for the material cannot develop.

7. Assessment of lateral variation within the ITZ: statistical approach

When analyses are available for adjacent stacks surrounding each aggregate examined, it is desirable to provide some sort of statistical evaluation of the degree of lateral variation for adjacent sampling units at a fixed distance from the aggregate. Such variation for successive elements of nominally the same character is commonly expressed in terms of standard deviation and coefficient of variation.

An example is provided in Table 1. The data, also taken from results of Huang [5] are a set of pore percentage area values for a full set of 19 "first-tier" sampling units (i.e., those adjacent to the aggregate interface), that entirely surrounds a particular sand grain. The concrete in question is a well-mixed, 3-day old $w : c$ 0.5 concrete made with a Type I cement using a dolomite coarse aggregate and dolomite sand. The mean value for the percentage area of detectable pores in this first-tier set is 10.03%. This mean value would ordinarily be pooled with the corresponding mean values of first-tier sampling units around the other aggregate grains studied in the same sample. The "grand mean" of the first-tier sampling units would be the value ascribed to

Table 1
Pore percentage area values found in adjacent first-tier ITZ sampling units surrounding a sand grain

Sampling unit no.[a]	Pore percentage area	Sampling unit no.	Pore percentage area
1	16.6	11	8.2
2	16.2	12	10.4
3	16.2	13	13.0
4	7.2	14	11.7
5	4.8	15	8.3
6	7.4	16	7.1
7	13.3	17	4.4
8	6.4	18	7.6
9	12.5	19	13.8
10	5.4		

[a] Units are numbered in sequence proceeding consecutively around the sand grain.

the first point in a plot such as that of Fig. 2; the other points would be corresponding grand means of second, third, and subsequent tiers of sampling units.

It is seen that the area percent of pores for these first-tier individual sampling units around the grain studied in the data of Table 1 varies significantly from each other as one proceeds around the grain. The range of values exhibited among the different sampling units is between 4.4 and 16.6 area percent pores. The sample standard deviation for the set is close to 4.0 area percent, and the coefficient of variation is almost exactly 40%.

It is obvious that a coefficient of variation of 40% represents a large degree of non-uniformity in the pore content of these nominally similar sampling units taken at the same distance from the same aggregate grain. With so much variation between individual local units, it is apparent that a mean value by itself, however statistically precise the mean value may be, does not adequately describe the situation. Measurements specifically reflecting the local variation encountered around the grain are also required for a proper picture of the ITZ to emerge.

In point of fact, the calculated standard deviation and coefficient of variation do not perfectly represent the situation either. These statistics assume that each sampling unit is independent of its neighbor. Examination of successive values in Table 1 indicates that there is clustering: that is, there are groups of successive values that are similar to each other. For example, the first three pore area percent values, adjacent to each other around the periphery, are all about 16%. The next three are between 4% and 8% pores. This local clustering of values is characteristic of the ''patchy'' microstructures that have been found to be characteristic of both ITZ and bulk hcp in concrete [9].

Variations and clustering similar to those illustrated in Table 1 were found in the corresponding pore percentage area values in the second and all subsequent tiers of sampling units, including those within the bulk hcp. Thus the commonly studied ITZ effect – the change in *mean* value with distance from the aggregate, as illustrated in Fig. 2 – is seen to be superimposed on a complex pattern of patchy variations within the ITZ, and indeed within all of the hcp.

8. Discussion and conclusions

The theme of this special issue of the present journal was stated by the Guest Editor as being ''automatic image analysis'' applied in various ways to concrete. As illustrated in the previous comments, concrete microstructure in and around the ITZ is complicated and variable. Many special features of the ITZ in concrete that influence the problems of its image analysis would seem to make fully automated determinations quite difficult.

In any event, these special features of concrete need to be considered in setting out the details of any image analysis scheme if misleading results are to be avoided.

Finally in the writer's view it is essential at the end of the day for the investigator to compare the numerical results and interpretations derived from the image analysis with the microstructure that he sees in the micrographs of a representative set of the images analyzed. Only by 'closing the loop' in this manner can possible weaknesses in the image analysis procedures be uncovered. Only by comparison of the results with the substance of the images from which they were derived can the full implications of the results be brought to light.

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References

[1] Scrivener KL, Patel HH, Pratt PL, Parrott LP. Analysis of phases in cement paste using backscattered electron images, methanol adsorption, and thermogravimetric analysis. Materials Res Soc Symp Proc 1987;85:67–76.
[2] Wang Y, Diamond S. An approach to quantitative image analysis for cement pastes. Materials Res Soc Proc 1995;370:23–32.
[3] Diamond S, Wang Y. A quantitative image analysis study of the influence of superplasticizers on cement paste microstructure. Proc 18th Intl Conf Cement Microscopy 1996. p. 465–479.

[4] Scrivener KL, Crumbie AK, Pratt PL. A study of the interfacial region between cement paste and aggregate in concrete. Materials Res Soc Proc 1988;114:87–8.
[5] Huang J. Microstructural study of the interfacial transition zone in concrete using backscatter mode scanning electron microscopy with image analysis, Ph. D. thesis, Purdue University, 1998.
[6] Scrivener KL, Bentur A, Pratt PL. Quantitative characterization of the transition zone in high strength concretes. Adv Cement Res 1988;1(4):230–7.
[7] Stutzman PE, Clifton JR. Specimen preparation for scanning electron microscopy. Proc 21st Intl Conf Cement Microscopy, 1999. p. 10–22.
[8] Scrivener KL, Pratt PL. Characterization of interfacial microstructure. RILEM Report 11, Interfacial Transition Zone in Concrete, E and FN Spon, London 1996. p. 1–16.
[9] Diamond S, Huang J. The interfacial transition zone: reality or myth? Proc RILEM 2nd Intl Conf on the Interfacial Transition Zone in Cementitious Composites, Haifa, 1998. p. 1–40.