Revisiting Critical Flaws in Cement-Based Composites

Eric N. Landis, M.ASCE1; Chula Gangsa2; Lauren S. Flanders, A.M.ASCE3

Abstract: In this work, X-ray microtomographic images were analyzed to quantify the influence of void spaces in small mortar specimens, with a particular focus on the porosity of the interfacial transition zone (ITZ). Specimens were nominally 5-mm-diameter, 4-mm-long cylinders with 0.5-mm-diameter glass bead aggregates. Specimens were scanned via synchrotron-based X-ray microtomography while they were positioned in an in situ loading frame in a split cylinder configuration. Scans of undamaged specimens were evaluated for porosity both in the bulk paste and in the ITZ. Specifically, voids in the paste and porosity in the ITZ were superimposed onto a map of the principal tensile stress in the specimen in an attempt to identify critical flaws and to measure their role in split cylinder strength. Results indicate that a stress intensity factor-type approach can be used to identify critical flaws in cement paste specimens. Similarly, a critical ITZ region can be identified based on local principal stress and local ITZ porosity. In mortar specimens, this critical ITZ region could account for most of the splitting failures that were not accounted for by a critical flaw. However, some specimens exhibited neither a critical flaw nor a critical ITZ region, suggesting prepeak microcracking or some other nonlinear fracture phenomena. DOI: 10.1061/(ASCE)NM.2153-5477.0000111. © 2016 American Society of Civil Engineers.

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Introduction

That flaws govern the strength and toughness of brittle materials is axiomatic. Characterizing flaws and quantifying their effects on multi-scale heterogeneous materials is problematic. In portland cement-based composites, flaws have many forms, including but not limited to air voids, capillary pores, and high porosity regions associated with the interfacial transition zone (ITZ) around aggregate particles. Characterization of these flaws is confounded by the nonuniform geometry and spatial distribution of aggregates at a mesoscale, and the irregular, fractal-like geometry of capillary pore spaces at a microscopic scale. (Indeed, one could further attribute complex geometry to heterogeneities in cement gel at a nanoscale.)

Because microstructural features are difficult to characterize, they are difficult to represent in a computational model. Recent developments in 3D discrete modeling approaches, including Man and van Mier (2008, 2009, 2011), Cusatis et al. (2011a, b), and Asahina et al. (2011), for example, include aggregate particles at a mesoscale, and do not explicitly represent defects. Multiscale approaches (e.g., Pichler and Hellmich 2011) have been used to include flaws and voids at the level of capillary pores. Homogenization schemes are then used to scale the problems to practical levels.

The challenge is both the difficulty and computational expense of expressly representing flaws, and the quantitative relationships between the flaws and the fracture properties of the material. More recent work by Graham-Brady et al. (2015) expressly included slit-like flaws and air bubbles in a micromechanics-based approach to evaluate dynamic failure of cement-based materials under compression. A linear elastic fracture mechanics framework was employed to evaluate a statistically generated microstructure, validated by X-ray computed tomography (X-ray CT) (Mayercsik et al. 2015).

For the work described in this paper, the results of micromechanical tests using X-ray microtomography were used to revisit the questions of flaws and their effect on fracture strength. The flaws considered in this work are at a scale of several micrometers and larger, and have no limitation with respect to shape within the resolution of the imaging. The ultimate experimental question was whether or not a critical flaw could be identified in small cement paste and mortar specimens loaded in a split cylinder configuration. Additionally, if a critical flaw cannot be identified, are there other measurable microstructure features on which one might base a microstructure-based computational model? This work is part of a larger program to incorporate detailed micromechanical data into physically based material models (Landis and Bolander 2009). From that work came several experimentally verified predictions, including one that larger aggregate volume fractions lead to lower split cylinder strength, and another that, within certain bounds, split cylinder strength was relatively insensitive to interfacial bond strength (Asahina et al. 2011).

In addition to the search for a critical flaw, the work described herein addresses two hypothetical questions. First, is the observed decrease in split cylinder strength with higher aggregate fractions due simply to the larger number of ITZ zones, or does the higher volume fraction tend to cause larger flaws in the cement matrix? Second, what is the basis for the aforementioned indifference to interface strength? Is it because the variation in interface strength is more significant than the mean value of interface strength? That is, even though smooth surface aggregates have, in general, weaker interfaces than rough surface ones, is there such variation in the rough surface interface strength that the “weak links” are comparable to the smooth surface cases? In the examination of both these hypotheses, ITZ porosity is assumed to be a proxy of ITZ strength. We consider this assumption in our acceptance or rejection of the hypotheses.

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Materials and Methods

Details of the micromechanical experiments can be found in de Wolski et al. (2014), but they are briefly summarized here for completeness.

Central to the experimental program was the use of synchrotron-based X-ray microtomography (Landis and Keane 2010), a high-resolution 3D imaging technique. The extremely bright X-rays emanating from the synchrotron lead to high-contrast images that allow one to isolate and identify phases with subtle differences in X-ray absorption. Tomographic scans were made by taking 1,500 projection images of the specimen rotated over 180°. Tomographic reconstruction produced 3D images of nominally 1,000 × 1,000 × 800 voxels, with each voxel being a 6 μm cube. Spatial resolution was not quite that of the 6 μm voxels, but was close because of the high-contrast images. Each scan took between 2 and 3 h to complete.

Specimens consisted of 5-mm-nominal-diameter cylinders labeled here as cement paste and mortar. The small specimen size allowed the authors to scan the specimens using high-resolution tomography. All specimens had a water-cement ratio of 0.5, and all specimens included very fine sand particles (passing a #80 sieve). Mortar specimens included 0.5-mm-diameter glass beads as aggregates. The glass beads were used for their well-defined geometry, and because the surface can be easily modified to change interface properties. In this work, two different surfaces were considered: smooth (untreated) and etched using an ammonium bifluoride solution. Two different aggregate volume fractions were used, roughly 10 and 50%, identified here as sparse and dense, respectively. Including the paste specimens there were five different specimen types. (Heretofore these are labeled as P, U10, U50, E10, and E50, for paste, untreated-10%, untreated-50%, etched-10%, and etched-50%, respectively.)

Specimens were loaded in a split-cylinder configuration in order to produce a relatively predictable stress state (i.e., less influence of friction at load platens than axial compression), in a simple-to-operate experimental apparatus. A custom load frame was used that allowed specimens to be scanned while under load (Landis et al. 2013). Load frames were capable of mon-
already identified, and they could be filled in to produce an image in which only the aggregates are highlighted, as shown in Fig. 1(c).

Once aggregate particles have been identified, we simply define the ITZ as the region of material in close proximity to the aggregate. An exact ITZ size is difficult to define (Ollivier et al. 1995), so for this work the ITZ was arbitrarily defined as the region within 100 μm of the aggregate surface. Thus, in the 3D image data, a voxel was considered to be part of the ITZ if it was within this 100-μm distance. The porosity of the ITZ region could then be analyzed by considering the pore space previously identified in the specimen. If these identified pore objects fall inside the ITZ region, then they are used to calculate the ITZ porosity, which is simply calculated as the fraction of ITZ voxels that are pores. An illustration of the ITZ regions in a specimen is presented in Fig. 2.

Once the ITZ region is identified, several characteristics can be evaluated. One item of interest, for example, was whether there was any directional bias in the ITZ porosity, possibly indicating an issue related to specimen casting. To answer this question, the ITZ of each aggregate was divided into segments relative to the axis of the cylinder. The porosity of each of the 18 regions in each ITZ was individually calculated, and this regional porosity was plotted against the orientation direction. There were no clear peaks in this plot, indicating that there was no directional bias in the ITZ.

Quantifying Effects of Flaws

In this section we consider how we might analyze the effects of different potential weak links in the material. Here we consider two possibilities: a weak link due to a critical flaw of arbitrary shape and location, and a weak link due to a critical ITZ region. For the former, we make a loose application of traditional fracture mechanics, whereas for the latter, we develop a technique based on the highly porous nature of the ITZ. Both techniques are developed with the available 3D X-ray CT image data in mind, and both techniques are formalizations of preliminary work (Gangsa et al. 2015).

Critical Stress Intensity Analysis

As a starting point for a stress intensity analysis, we consider a linear elastic fracture mechanics (LEFM) approach in which a stress intensity factor, $K$, is defined as the tip of an infinitely sharp crack (Broek 1986)

$$K = \beta \sigma \sqrt{a}$$

where $\sigma$ = far field stress; $a$ = length of the crack; and $\beta$ = a constant that is defined by local geometry. For our work we must deviate from this well-known relationship for several reasons. First, because of resolution limitations, we are not in a position to evaluate a flaw with an infinitely sharp tip. Although, if we recognize that in hydrated cement paste there is porosity at a nanoscale, this may not be such a concern because all flaws will be connected to such nanoscale porosity, which may for practical purposes be infinitely sharp. Second, and perhaps more importantly, the flaw geometry is typically not well represented by simple Euclidian objects. Hence a geometry parameter is not easily defined by analytical means.

Despite these limitations, we introduce here a quasi-stress intensity factor, $K_q$, that is intended to account for the stress amplification around a given flaw, as well as the geometry of the specimen relative to the flaw, but admittedly does not come with the analytical rigor of a conventional LEFM-based stress intensity factor. Evaluation of $K_q$ consists of two parts: the location of the flaw in the split cylinder and the orientation of the flaw. This can be written:

$$K_q = P \alpha \sqrt{A}$$

where $A$ = flaw area; $P$ = applied load; and $\alpha$ = principal tensile stress at the centroid of the flaw for a unit load. The 4th root of $A$ comes from dimensional analysis, and we note that $K_q$ in Eq. (2) has the same units as a traditional stress intensity factor, $K$ (e.g., MPa√m).

For each specimen a 3D image is created in which the intensity at any point is proportional to $\alpha$. A single 2D slice of this image is shown in Fig. 3. To create this image, an elastic medium is assumed to which we can apply the well-established stress field solution (Timoshenko and Goodier 1951). $\alpha$ is represented by a grayscale intensity ranging from zero (black) to the maximum (white), which has a value of $2/(\pi DL)$. $D$ = the cylinder’s diameter and $L$ = its axial length. Thus, the principal tensile stress for any point, and specifically at any flaw location, can be determined based on

Fig. 2. 3D rendering of isolated aggregates and their interfacial zone: (a) isolated bead aggregates inside specimen; (b) pores isolated around aggregate; ITZ pores are colored black in these renderings.
the intensity, $\alpha$, of the 3D image at that location, multiplied by the applied force, $P$.

To represent the effect of flaw orientation, we consider the principal direction associated with the principal stress at each point in the specimen. The assumption here is that a flaw of arbitrary shape has maximum effect in a plane normal to the principal direction, at a point in the flaw where the area in that plane is highest. This value can be found for each flaw by establishing a plane normal to the principal direction, and moving that plane through the flaw, as illustrated in Fig. 4. Eq. (2) is applied at the centroid of each plane as it is moved through the flaw. The quasi-stress intensity factor for the flaw is taken to be the maximum value of all possible positions of the plane in the flaw.

In an X-ray CT image for which flaws have been isolated, Eq. (2) can be applied to each flaw. It is assumed that the flaw that produces the highest $K_q$ is likely to be the critical flaw in the specimen.

**Critical ITZ Analysis**

The interfacial transition zone is frequently cited as the “weak link” in concrete (Buyukozturk and Wecharatana 1995; Alexander et al. 1999). Although most work intended to quantify relationships between ITZ properties and fracture strength has been done through computational modeling (Leite et al. 2004; Asahina et al. 2011; Königsberger et al. 2013a, b), a number of innovative experiments have helped elucidate the ITZ role in mechanical properties (Corr et al. 2007; Xiao et al. 2013).

In this work we took ITZ properties that we could extract from the X-ray CT data and sought to put them in a form that might be suitable as a predictive measure. To do this required several assumptions. First, we assume that the porosity in the ITZ is a proxy for the strength of the ITZ. Furthermore, we assume this is an inverse relationship. Second, we assume that the ITZ regions relevant to fracture strength are those that are nominally normal to the principal tensile stress.

Given this second assumption, a relevant ITZ region can be segmented for each aggregate particle. Because the ITZ has already been identified (Fig. 2), all that needs to be done is to isolate those regions of the ITZ that are subjected to tension. This was accomplished by inserting each aggregate on the principal tensile stress field of Fig. 3. We then run the principal direction axis through the aggregate, and remove all of the ITZ except the region within 22.5° (45° total sweep) of that axis. A 3D rendering of the resulting region is shown in Fig. 5. The porosity of the relevant ITZ is simply the volume of the pore space divided by the total volume of the region.

![Fig. 3. Illustration of the terms used to evaluate the quasi-stress intensity factor, $K_q$.](image)

![Fig. 4. Illustration of the terms used to evaluate the quasi-stress intensity factor, $K_q$: (a) 3D rendering of typical flaw; (b) flaw intersected with plane normal to principal direction](image)
Fig. 5, 3D rendering of aggregate particle with relevant segment of ITZ shown; section is defined such that the axis of the principal direction is centered on the relevant ITZ region.

Recognizing that the location of the relevant ITZ within the specimen is significant because of the varying stress field, we invoke our first assumption that the ITZ porosity is a proxy for strength. To complete our analysis, we use our map of principal tensile stress to estimate the stress acting across each of the relevant ITZ regions, and we define the critical ITZ region as the one that produces the highest combination of porosity and stress. Following a simple empirical relationship for porous materials (Lian et al. 2011), we can propose an ITZ strength, \( f_t \), of the form

\[
f_t = f_o(1 - p_i)
\]

where \( f_o = \) the strength in the absence of additional ITZ porosity; and \( p_i = \) the porosity of the region. This model suggests that the relevant ITZ ruptures when the principal stress, \( \sigma_1 \), reaches the ITZ strength, \( f_t \). Using the principal stress formulation in the preceding, and setting the applied load, \( P \), to its ultimate value, \( P_u \), Eq. (3) can be written as:

\[
\alpha P_u = f_o(1 - p_i)
\]

Experimentally, we can then estimate the baseline ITZ strength by solving this equation for \( f_o \):

\[
f_o = \frac{\alpha P_u}{(1 - p_i)}
\]

for all ITZ regions. Baseline ITZ strength, \( f_o \), is taken as the highest value obtained in all ITZ regions analyzed, because this is the region that would dictate failure.

We are presenting, of course, a very simplified version of the stress field in the relevant ITZ region. First of all, we are only considering the tensile stresses acting at the ITZ region. The shape of the ITZ will almost certainly lead to mixed-mode stress states, but implicit here is that the mixed-mode effects are small. Second, we are assuming that the relevant ITZ region that produces the highest \( f_o \) is the critical ITZ region, which is considered the “weak link” that precipitates fracture. As further detailed in the following, this assumption has limitations.

**Experimental Results**

**Critical Flaws Analysis Results**

We first present the results of a critical flaw analysis in the cement paste specimens. We start here because there is no ITZ present, so any critical flaw must be in the cement matrix. The parameters of Eq. (2) are presented in Table 1 along with the calculated values of the critical quasi-stress intensity factor, \( K_q \). That is, shown on the table are the results for the flaw–stress combination that produces the highest \( K_q \). Again, the assumption here is that, following LEFM theory, fracture occurs when a critical value of \( K_q \) is reached.

The results show reasonable consistency for \( K_q \) despite very different flaw areas. 3D renderings of two paste specimens are shown in Fig. 6. Fig. 6(a) illustrates a smaller amorphous flaw that is strategically (i.e., centrally) located, whereas Fig. 6(b) shows a much larger bubble-shaped flaw that is not quite as critically located. Nevertheless, both are identified as the critical flaw in the particular specimen.

We next turn our attention to the mortar specimens. Table 2 shows the flaw analysis for all mortar specimens, including sparse and dense aggregate fractions, and untreated and etched aggregate surfaces. Here the results are less consistent, with values of \( K_q \) varying considerably. However, it is interesting to note that there appears to be a ceiling of \( K_q \) values that does not exceed the values measured in the paste specimens. If we apply the mean \( K_q \) of the paste specimens (6.7 MPa·\( \sqrt{\text{mm}} \)) to the \( K_q \) values of Table 2, we see that two of the E10 specimens (E10-3 and E10-5) are very close, whereas one of the U10 specimens (U10-3) is not too far below.

One way to interpret this result is to propose that the fracture of the paste specimens is indeed governed by a critical \( K_q \), but this is not necessarily the case for the mortar specimens. The plot of Fig. 7 illustrates this by plotting all of the splitting force results against the flaw area. Note that these have been normalized as if they were all in the same central location (i.e., they all have the same geometry factor, \( \alpha \)). Hence the values plotted in the figure do not match exactly with the splitting forces shown in Tables 1 and 2. Also shown on the plot is the predicted splitting force for centrally located flaws of varying area. The plot shows that some of the specimens fall close to the predicted value, whereas others fall well below. A potential conclusion is that for the specimens that lie close to the line, a critical flaw governs splitting strength, whereas for the specimens that do not lie close to the line, there is another mechanism that governs splitting strength. In the next section, the effects of the ITZ properties are examined as a potential source of an alternate governing factor.

**Critical ITZ Analysis Results**

Using the methods described earlier, each bead aggregate in each mortar specimen was segmented, the ITZ aligned with the principal
stress direction was isolated, and its corresponding porosity calculated. Then, each of these tensile zones was analyzed using Eq. (5). As stated earlier, we assume the limit state is reached when one of the ITZ zones reaches its baseline strength (no porosity), \( f_o \). For each ITZ in a specimen, a value of \( f_o \) was calculated. The maximum value was taken to be the baseline interface strength for the specimen. Table 3 shows this value calculated for each specimen, along with some statistical measurements of all tensile ITZ regions.

Several observations can be made from this data. First, although the sample size is small, there appear to be differences among the baseline ITZ strengths of the different specimen types. The mean interface strength for the untreated aggregate specimens is 7.0 MPa, compared with 9.1 MPa for the etched aggregate specimens, confirming what we would expect in regard to interface strength. What is perhaps more interesting is that for the etched aggregate sample the two specimens that have the lowest interface strength (E10-3 and E10-5) were also the two that had critical stress intensities that approached the value of the paste specimens. This result could indicate that the full ITZ strength was never reached in these specimens because the specimen failed due to a critical flaw in the cement paste. Specimen U10-3 also fractured at a stress intensity factor close to the paste, but its interfacial strength is not so clearly different from the rest of the U specimens. Regardless, a preliminary conclusion partially supported by the data is that if a specimen fails by fracture due to a flaw in the cement paste, it will not reach a load level required to fail the specimen at the ITZ.

A second observation is that although some specimens seem to approach either a flaw-based stress intensity limit or an ITZ strength limit, others do neither. For example, the ultimate splitting strength of specimens U10-1 and E10-2 cannot be explained by

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**Table 2. Critical Flaw Analysis in Mortar Specimens**

<table>
<thead>
<tr>
<th>Specimen</th>
<th>( P_u ) (N)</th>
<th>( \alpha )</th>
<th>( A ) (mm(^2))</th>
<th>( K_q ) (MPa·(\sqrt{\text{mm}}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>U10-1</td>
<td>175</td>
<td>0.022</td>
<td>0.022</td>
<td>1.5</td>
</tr>
<tr>
<td>U10-2</td>
<td>420</td>
<td>0.010</td>
<td>0.532</td>
<td>3.6</td>
</tr>
<tr>
<td>U10-3</td>
<td>485</td>
<td>0.015</td>
<td>0.217</td>
<td>5.0</td>
</tr>
<tr>
<td>U50-1</td>
<td>275</td>
<td>0.013</td>
<td>0.155</td>
<td>2.2</td>
</tr>
<tr>
<td>U50-2</td>
<td>415</td>
<td>0.026</td>
<td>0.059</td>
<td>1.6</td>
</tr>
<tr>
<td>E10-1</td>
<td>415</td>
<td>0.010</td>
<td>0.217</td>
<td>4.4</td>
</tr>
<tr>
<td>E10-2</td>
<td>410</td>
<td>0.017</td>
<td>0.152</td>
<td>6.8</td>
</tr>
<tr>
<td>E10-3</td>
<td>375</td>
<td>0.024</td>
<td>0.319</td>
<td>4.2</td>
</tr>
<tr>
<td>E10-4</td>
<td>280</td>
<td>0.028</td>
<td>0.081</td>
<td>6.6</td>
</tr>
<tr>
<td>E10-5</td>
<td>220</td>
<td>0.031</td>
<td>0.851</td>
<td>1.8</td>
</tr>
<tr>
<td>E50-1</td>
<td>400</td>
<td>0.010</td>
<td>0.038</td>
<td>3.4</td>
</tr>
<tr>
<td>E50-2</td>
<td>385</td>
<td>0.018</td>
<td>0.056</td>
<td>5.0</td>
</tr>
</tbody>
</table>

**Table 3. ITZ Analysis in Mortar Specimens**

<table>
<thead>
<tr>
<th>Specimen</th>
<th>( P_u ) (N)</th>
<th>( f_o ) (MPa)</th>
<th>mean, ( \bar{p} )</th>
<th>Standard deviation, ( s_p )</th>
<th>max, ( p_{\text{max}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>U10-1</td>
<td>175</td>
<td>4.8</td>
<td>0.029</td>
<td>0.011</td>
<td>0.057</td>
</tr>
<tr>
<td>U10-2</td>
<td>420</td>
<td>8.6</td>
<td>0.037</td>
<td>0.012</td>
<td>0.062</td>
</tr>
<tr>
<td>U10-3</td>
<td>485</td>
<td>6.4</td>
<td>0.063</td>
<td>0.033</td>
<td>0.137</td>
</tr>
<tr>
<td>U50-1</td>
<td>275</td>
<td>7.5</td>
<td>0.038</td>
<td>0.028</td>
<td>0.140</td>
</tr>
<tr>
<td>U50-2</td>
<td>415</td>
<td>7.7</td>
<td>0.049</td>
<td>0.019</td>
<td>0.083</td>
</tr>
<tr>
<td>E10-1</td>
<td>415</td>
<td>11.2</td>
<td>0.036</td>
<td>0.019</td>
<td>0.080</td>
</tr>
<tr>
<td>E10-2</td>
<td>410</td>
<td>8.8</td>
<td>0.025</td>
<td>0.013</td>
<td>0.056</td>
</tr>
<tr>
<td>E10-3</td>
<td>375</td>
<td>6.2</td>
<td>0.047</td>
<td>0.014</td>
<td>0.085</td>
</tr>
<tr>
<td>E10-4</td>
<td>280</td>
<td>10.1</td>
<td>0.032</td>
<td>0.016</td>
<td>0.064</td>
</tr>
<tr>
<td>E10-5</td>
<td>220</td>
<td>6.3</td>
<td>0.030</td>
<td>0.017</td>
<td>0.082</td>
</tr>
<tr>
<td>E50-1</td>
<td>400</td>
<td>10.8</td>
<td>0.038</td>
<td>0.017</td>
<td>0.104</td>
</tr>
<tr>
<td>E50-2</td>
<td>385</td>
<td>10.6</td>
<td>0.041</td>
<td>0.015</td>
<td>0.092</td>
</tr>
</tbody>
</table>

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**Fig. 6.** 3D rendering of paste specimens: (a) P-1 and (b) P-4 along with their critical flaws, illustrating the different types of flaws that appear in the specimens.

**Fig. 7.** Plot illustrating analysis of critical flaws; the values plotted are shown for a centrally located flaw.

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either the ITZ strength or the critical flaw. It is possible, and perhaps even likely, that in these cases, microcracking prior to peak load forced a redistribution of stress leading to initially stable crack growth. In the macroscopic sense, every specimen failed in a brittle manner, suggesting a critical flaw. However, in some of these specimens, the fact that they did not quite reach a “critical state” would suggest some nonlinear fracture behavior.

Discussion

Two hypotheses were introduced in the introductory section. Both consider the ITZ as it relates to observed decline in specimen strength with increasing aggregate fraction. In order to answer these questions, we turn our attention to the rightmost three columns of Table 3. Presented there are the mean, standard deviation, and maximum value of all ITZ regions in a tensile zone. Although sample sizes are too small to be significant, we observe what could be important differences. First, the mean value of ITZ porosity is slightly higher in the U specimens: 0.043 for the U specimens compared with 0.036 for the E specimens. There may be a particle packing explanation for this: a rough surface may lend itself better to higher particle packing density as compared to a smooth surface. This is admittedly speculation.

A second observation, and one that relates to the proposed hypothesis, is that although there is little difference in mean porosities between sparse and dense specimens (e.g., E10 versus E50), the maximum measured values are higher in both the U50 and E50 specimens as compared to the U10 and E10, respectively. Presumably this stems from an ITZ porosity that follows a statistical distribution, and therefore a larger sample size (in this case more aggregates) leads to more values in the extremes. This would then support the hypothesis that the reason specimens get weaker with more aggregates is that the likelihood of a higher-porosity ITZ “weak link” region increases. The alternate hypothesis that the specimens with more aggregates are weaker because of higher probability of flaws in the cement paste is not supported by the data because none of the specimens tested reached what would be their critical stress intensity, and thus likely failed due to an alternate weak link such as an ITZ zone.

The second hypothesis regarding the observed indifference to split cylinder strength on interface strength is more challenging to address with the data. The results show slightly higher base (absence of porosity) ITZ strength in the etched aggregate specimens, as well as slightly lower mean interfacial porosities. However, the variation of ITZ porosity is fairly close among the two types. If, as accepted earlier, there is a dependence of split cylinder strength on weak ITZ zones that are more likely to appear with more aggregates, then it is plausible that any dependence on ITZ strength is lost in the noise of ITZ porosity variation. Although the baseline interfacial strength may play a role in material split cylinder strength, the effect is much smaller than the ITZ strength reductions due to extreme values in ITZ porosity that further reduce ITZ strength.

It should be noted that of course there are many limitations to the analysis here, and there are likely many alternate interpretations of the experimental results. First and foremost are the relatively small sample sizes of all specimen types. This is a difficult limitation due to limited access at a shared facility such as a synchrotron. We suggest, however, that if a larger sample size was possible, it is not clear that our results would be significantly altered. We base this suggestion on the relatively robust nature of the digital image analysis. In the image analysis, the primary source of uncertainty lies in the threshold chosen for image segmentation. Indeed, if we increase or decrease the threshold, we find that nearly all of our measured fracture parameters change. As the threshold increases, measured flaw area decreases, and subsequently \( K_q \) decreases. However, all the quantities vary in a way that their relative values do not change, meaning none of our conclusions would change. The threshold image intensity is based on a widely used technique (Otsu 1979), so it is likely that others would reach the same conclusions given the image data included here.

A second important limitation is the simple homogeneous material assumed for the principal stress analysis. Clearly the mismatches in stiffness among aggregates and the cement matrix lead to local stress variations that could be significant enough to alter the results determined here. A potential solution to the latter of these problems is to examine the experimental results through the lens of an appropriately tuned computational model that has the capability to quantify the local stress variations that arise from the heterogeneity. Such a model could then be used to explore numerous parametric variations that might converge to a better interpretation of the results.

Summary and Conclusions

The work presented in this paper suggests that 3D imaging techniques such as X-ray microtomography allow us to re-examine the role of defects in heterogeneous materials. In small cylindrical specimens, we were able to segment the material into paste and aggregates, and we were able to isolate voids in the paste and porosity in the ITZ. When these specimens were further tested in a split cylinder configuration, we could relate those measured flaws and ITZ regions to the ultimate splitting force required to fracture the specimen.

Results showed that a critical quasi-stress intensity factor, \( K_q \), could be determined based on a flaw with an area that is normal to the axis of principal tensile stress. The critical stress intensity factor was assumed to be the value of \( K_q \) at peak force. For the cement paste specimens, without aggregate, this value was reasonably consistent and indeed was found to apply to several of the mortar specimens. Similarly, the baseline strength of the ITZ was inferred by assuming a simple linear relationship between ITZ strength and ITZ porosity. In this case the relevant ITZ was defined as that oriented in the direction of the principal tensile stress at the location of the aggregate. Although not as consistent as the flaw in the paste, the data suggested a baseline ITZ strength that accounted for the failure of most of the mortar specimens. The fact that some specimen fracture strengths could not be explained by either ITZ or a critical flaw was attributed to potential prepeak microcracking (van Mier 2012) or some other nonlinear fracture phenomena.

Results supported the hypothesis that the observed decline in split cylinder strength with greater aggregate volumes is due to a higher probability of finding a high-porosity ITZ region that would serve as a weak link.

Among the implications of the work are that the results can be incorporated into discrete models such that the models can properly incorporate spatial variability. Specifically, it shows the interacting role of ITZ variability and void distribution relative to stress fields as the likely source of fracture initiation.

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Notation

The following symbols are used in this paper:

\[ A = \text{flaw area (mm}^2\text{)} \]
\[ D = \text{diameter of cylinder specimen (mm)} \]
\[ f_o = \text{strength of an ITZ in the absence of porosity} \]
\[ f_i = \text{strength of an ITZ in the presence of porosity} \]
\[ K_o = \text{quasi-stress intensity factor (MPa} \cdot \text{mm}) \]
\[ L = \text{length of cylinder specimen (mm)} \]
\[ P = \text{force applied to split cylinder (N)} \]
\[ P_{\text{max}} = \text{maximum porosity of relevant ITZ region} \]
\[ P_a = \text{ultimate force applied to particular split cylinder (N)} \]
\[ p_i = \text{porosity of relevant ITZ region} \]
\[ \sigma = \text{principal tensile stress for a unit force} \]
\[ \sigma_1 = \text{principal tensile stress (MPa)} \]
\[ \varepsilon = \text{strain} \]

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