Understanding Material Behavior by Integrating Numerical Simulation with 3D Microstructural Imaging

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ABSTRACT: In this work we seek to improve our understanding of micromechanical phenomena by coupling 3D images of specimens subjected to damage with discrete element computational models. Through this close coupling we might quantify properties such as cement-aggregate interface, the spatial distribution of aggregates, and other features that elude simple measurement. Results show that by producing computational models in which aggregate locations match exactly with real specimens, we are not only able to simulate the damage and fracture patterns observed in the real specimens, but also match the bulk load-deformation and fracture response. Further simulations predicted a relationship between strength and aggregate fraction that was confirmed by further experiments. We view this work as an important step in a movement towards more physical-based modeling.

1 INTRODUCTION

Quantitative microstructure-property relationships for heterogeneous materials have been hampered by the difficulties in not only characterizing complex microstructure, but also representing that complex structure in a predictive model. The usual tools of planer cracks, penny-shaped cracks, and spherical inclusions can provide useful insight into damage and fracture behavior, however, they can fall short as honest descriptors of real heterogeneous microstructure. The work described here is aimed at developing new ways to measure the physical microstructure of damage and fracture, and use those measurements to create realistic numerical models. Our approach is to identify key microstructural features in 3D tomographic images, and create lattice-type finite element models that match the measured microstructure (Landis and Bolander 2009). Below we provide an overview of the 3D imaging, the micromechanical experiments, and preliminary simulations results.

2 EXPERIMENTS

2.1 X-ray microtomography

X-ray Microtomography (XMT) is an image acquisition method that produces a 3-dimensional map of an objects x-ray absorption through the mathematical reconstruction of a series of 2-dimensional radiographic images taken over many different rotation angles. The technique is perhaps best known for its use in medical imaging, where it is referred to as a CAT-scan. As the different phases in a material typically exhibit characteristic x-ray absorption, the 3-dimensional map may be interpreted as a material phase map. XMT has gained relatively wide usage for a wide range of materials applications (Stock 2008), and its use is growing thanks to the increasing availability of both laboratory scale instruments and synchrotron-based facilities.

In the work described here, we exploit the technique to examine the internal structure of the material in three dimensions at relatively high spatial resolution.
2.2 Specimens

The specimens used here were prepared using a high early strength (ASTM Type III) portland cement, very fine silica sand (pass #80 sieve), small glass bead aggregates, and water. 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. Micrographs of etched and unetched bead aggregates are shown in Fig. 1. The mix proportion was 1: 0.64: 0.23: 0.45, by weight cement:fine sand:glass beads:water. Additionally, a set of specimens without glass beads was prepared to investigate properties of the cement matrix. The material was mixed with a benchtop rotary mixer, and cured in wet conditions for seven days. The small cylindrical test specimens were extracted from the bulk material using a 5.5 mm inside diameter diamond coring bit. Resulting cores were then cut to a nominal 4 mm length.

![Figure 1. Micrographs of smooth and etched glass bead aggregates.](image1)

2.3 Experimental Protocol

An experimental protocol was established such that changes in internal structure could be tracked from undamaged to damaged states. A custom load frame was constructed and is illustrated in Fig. 2. The load frame allows us to make 3D tomographic images of specimens under load, while simultaneously monitoring load and deformation information. The first tomographic scan is made of the specimen mounted in the load frame prior to any load being...
applied. Once the first scan is complete a load is applied and the second scan is made with the specimen under load. The nominal plan for each specimen was to have a scan at: 0%, 50%, and 90% of peak load as well as a post fracture scan. Since the exact strength of each individual specimen was not known a priori, these percentages were approximate.

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Experiments were conducted at beamline 5BMC at the Advanced Photon Source (APS) at Argonne National Laboratory, US. The x-ray source was a monochromatic beam at an energy of 30 keV. Tomographic scans consisted of 1200 projection images taken over 180 degrees. Due to the image acquisition and data transfer times, a single scan took about 2.5 hours to complete. Tomographic reconstruction was done using a filtered back-projection algorithm resulting in a three-dimensional image 1299 x 1299 x 920 voxels. A typical cross sectional “slice” image along with a 3D rendering are shown in Fig. 3. The slice image shown is a cross section perpendicular to the axis of loading. In these images, grayscale corresponds to x-ray absorption, so brighter regions correspond roughly to denser phases, and vice versa. The images illustrate the material heterogeneity. The uniformly gray circular objects are the glass bead aggregates. Black objects inside the specimen are pore spaces, while the white objects are either un reacted cement grains (fine white speckles), or fine sand grains that contain some metal oxides that absorb more x-rays.

A typical scan sequence is illustrated in Figure 4, where a series of slice image oriented parallel to the axis of load are shown, highlighting the splitting nature of failure. Fig. 4(a) shows an undamaged specimen. The flat regions above and below the specimen are the steel loading platens. In the image of Fig. 4(b), which was taken at roughly 97% of peak load, a fine crack has formed through the center of the specimen. It should be noted that this crack does not extend through the entire length of the specimen. In the image of Fig. 4(c), taken after peak load, the crack has opened, and has branched near the top and bottom platens. It can be seen in Fig. 4 (b) and 4(c) how the aggregate particles can deflect and redirect the cracks.

3 LATTICE FORMULATION & SIMULATION

Lattice representations of heterogeneous materials have been used for a number of years, emerging from work in statistical physics (Herrmann and Roux 1990). The philosophy of the approach used here is that statistical variations and disorder can be explicitly represented in a way that resembles the actual material.

In the approach applied here, lattice topology is based on the Delaunay tessellation of nodal points within the specimen domain. The dual Voronoi tessellation defines the elastic and
fracture properties of the lattice elements (Berton and Bolander 2006). This discretization involves a three-phase representation of the material meso-structure as follows: hardened cement paste, aggregates, and cement-aggregate interface.

Figure 5 shows the simulated fracture pattern for a split-cylinder without aggregate inclusions. Fracture initiates in the central region of the specimen, where the tensile stress is quasi-uniform, and propagates toward the loading platens. Qualitatively, the general pattern of fracture is consistent with that observed in tomographic scans. Specifically, while the mesh was relatively coarse, we obtained qualitative agreement in the physical features such as crack branching and crack distribution.

4. Examination of Specimens with Spherical Inclusions

Of particular interest in random heterogeneous materials such as concrete is the properties of the cement-aggregate interface. Presented here are the first steps of a process that will enable us to characterize interfaces in situ. A model specimen was developed in which the aggregate particles were spherical beads. This simplification was done to facilitate meshing of the lattice model, and allows us to remove geometric variations in aggregates from the list of experimental variables. The selected aggregates were nominal 0.5 mm spheres made of soda lime glass. In order to vary the cement-aggregate interface bond strength, both smooth (as purchased) and etched beads were used. The specimens were etched using a very dilute HF acid. An example of a tomographic scan of such a specimen is shown in Figure 5, where the spherical aggregates appear as a relatively uniform gray level in the image.

As the primary objective of this work was to match numerical to real specimens, we developed an image processing routine that allowed us to isolate the individual

Figure 4. Sequence of split cylinder fracture. (a) undamaged specimen, (b) initiation of splitting fracture at close to peak load, (c) post-peak fracture showing multiple crack branches, and (d) schematic illustration of viewing planes relative to specimen.

Figure 5. Example simulation of damage progression in cement paste specimen (a) - (c) along with final crack pattern in actual paste specimen (d).
MATCHED REAL AND “VIRTUAL” SPECIMENS

In addition to the qualitative assessments that can be made from the images, we can also employ 3D digital image analysis methods to extract quantitative measurements. Relevant to the work reported here are efforts to create a matched set of real and “virtual” specimens for subsequent study. To do this, the spherical aggregates in the tomographic scans were isolated and their coordinates using a variance filter as illustrated in Fig. 6. Traditional intensity-based segmentation does not work because the voxel intensities of the glass aggregates are in the same range as the cement hydrates. However, because of the relative homogeneity of the glass beads, one can simply determine the variance of all voxels in a region. In regions where the variance is high we can assume we are in among cement hydrates and fines. Where the variance is low, we can assume are in either aggregates or voids. To distinguish bead aggregates from voids, we simply return to the original grayscale image. The process is illustrated in Fig. 6 for a single slice, although in practice the process is run for the entire volume.

![Figure 6. Process used to isolate glass bead aggregates. Original slice image (a), variance of voxels (b), regions identified as aggregates (c).](image)

Once the aggregates are located in the real specimen, the coordinates of the centroids relative to an overall specimen reference frame can be calculated and used to create a companion virtual specimen as shown in Fig. 7. Here a virtual mesh is generated based on the measured aggregate centroids and diameters. Mesh properties can then include separate aggregate and paste phases, as well as an interface phase.

![Figure 7. 3D rendering of aggregate locations in real specimen (a), and a cutaway mesh of the corresponding virtual specimen.](image)
Matching the real and “virtual” specimens allows us to examine things that are otherwise difficult to measure. For example, the nature of the tomographic scans only allows us to examine the specimen at discrete points in time while the load is held static. In the virtual specimens we can also examine dynamic phenomenon and subtle increments in crack growth (Asahina et al 2011).

4.1 Virtual Experiments

Using volumes generated from matched real specimens, simulations on virtual split cylinder specimens were run. Details of the mesh properties are presented in Asahina et al (2011). Images illustrating the simulations are shown in Fig. 8, where virtual specimens are compared qualitatively with real specimens. The real specimens shown had unetched aggregates, and a volume fraction of about 10%. The images highlight the ability of the discrete element model to capture physical fracture phenomena. Specifically, the role of the aggregates in redirecting cracks and branching cracks can be clearly observed. It should be noted that to save meshing time, aggregates on the extreme outer edges are not meshed. Clearly the fineness of the mesh in the paste phase affects the tortuosity of the cracking, but with continued improvements in computational efficiencies, we likely see numerical solutions that converge on the patterns of the real specimen.

Figure 8. Qualitative comparison of post peak damage patterns in real and virtual specimens.
5 CONCLUSIONS

We have presented some preliminary work on the development of numerical models of random heterogeneous composites that are meshed based on a direct correspondence between features of the numerical specimen and the real specimen. The work is presented as a first step in micromechanical characterizations of complex cement matrices and interfaces. Through the use of matched specimens we are able to capture realistic damage patterns. While not shown here, simulations can also capture load-deformation behavior with good accuracy.

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7 REFERENCES