Mechanical And Computational Evaluation Of Cementitious Materials With Hollow Glass Microsphere Inclusions

Zachary J. Wallace

University of Mississippi

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MECHANICAL AND COMPUTATIONAL EVALUATION OF CEMENTITIOUS
MATERIALS WITH HOLLOW GLASS MICROSPHERE INCLUSIONS

A Thesis
presented in partial fulfillment of requirements
for the degree of Masters of Science
in the Department of Mechanical Engineering
The University of Mississippi

By
Zachary J. Wallace

May 2017
ABSTRACT

The effect of volumetric replacement of cementitious materials by an inert filler known as hollow glass microspheres on elastic properties is evaluated in this study through a combined computational and experimental approach. This approach considered the variation in properties of type I Portland cement for 0, 5, 10, and 15% volume fractions of two kind of glass microspheres; one was high density with high crushing strength, and the other with a relative lower density. Using a suite of software called Virtual Cement and Concrete Testing Laboratory (VCCTL), the microstructural details were determined for 80% degree of hydration of the cement mixture for these different volume fractions. The VCCTL-generated three dimensional heterogeneous microstructures with various micro-constituents, such as the C-S-H gel, tricalcium silicate, and other cement byproducts, were explicitly modeled in ABAQUS© commercial finite element code. The representative volume element (RVE) in ABAQUS was a 100x100x100 \( \mu \text{m}^3 \) cube. Since the symmetry as well as the anisotropy of the cementitious material system is not fully established, iso-strain based boundary conditions were applied to this RVE towards determining various elements of the elastic tensor. The computationally calculated elastic moduli compared reasonably well with data from quasi-static compression tests for various volume fractions of the glass microspheres.
LIST OF ABBREVIATIONS AND SYMBOLS

\( \beta \)  Shape parameter

\( \eta \)  Scale parameter

\( t \)  Normalization around the mean

\( R_o \)  Outside radius

\( R_i \)  Inner radius

\( \rho_{gs} \)  Hollow glass microsphere true density

\( \Delta p \)  Change in pressure

\( D \)  Measured diameter of microsphere

\( t_w \)  Wall thickness

\( \sigma \)  Stress

\( E \)  Young’s modulus

\( \varepsilon \)  Strain

\( V_o \)  Initial volume

\( k \)  Effective bulk modulus

\( \nu \)  Poisson’s ratio
\phi \quad \text{Porosity}

K \quad \text{Bulk Modulus}

G \quad \text{Shear Modulus}
ACKNOWLEDGMENTS

The author from The University of Mississippi, acknowledges the support by the U.S. Army Research Office under a cooperative agreement award contract No. W911NF-11-2-0043 (Program Manager: Dr. Joseph Myers) and the U.S. Army Engineering Research and Development Center’s Military Engineering Basic/Applied “MMFP” Research Program. Additionally, discussions with several researchers at Engineering Research and Development Center, Vicksburg, MS, and U. S. Army Research Laboratory are gratefully acknowledged.

Chemical analysis of cement used in experimental testing was provided by Michael S. Hammer, Manager of Technical Services at Buzzi Unicem USA. This testing was invaluable to the process and is acknowledged gratefully.

Particle size distribution for the 3M glass microspheres was provided by Rob Hunter, Applications Development Specialist, from the Advanced Materials Division Laboratory at 3M. This data was critical for development of the microstructure, therefore they are gratefully acknowledged.

SEM imagery was provided by Vijayasankar Raman, Research Scientist, from the Nation Center for Natural Products Research School of Pharmacy. This imagery lead to the particle size distribution for the FM glass microspheres and eventually wall-thickness determination. Dr. Raman and the research center are acknowledged gratefully.
Furthermore, valuable attributions provided by Matthew Nelms, Damian Stoddard, Matthew Lowe, and Dr. Rajendran, all from the Mechanical Engineering Department at the University of Mississippi, are acknowledged.
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I. INTRODUCTION

One area that the scientific community has been exploring for a while is replacement of a percentage of cement paste with alternative materials that is lighter and cheaper with enhanced material properties. Additives often considered include: fly ash, cut metallic fibers, and graphite fibers. Glass microspheres, in particular, have been used in a very limited amount of studies involving cementitious materials, as compared to other inert fillers, and will thus be considered for this study.

Cement is a binder used mainly for construction purposes. Concrete is formed when this cement binder has fine or course aggregates added to its mixture. There are two types of cement, hydraulic and non-hydraulic. For this work, only hydraulic is taken into consideration. The difference being a non-hydraulic cement will not cure in wet conditions and relies upon carbon dioxide in the air. Hydraulic cement cures due to reaction between the dry ingredients and the water. Hydraulic cements are made with several silicates and oxides. The cement used for this study is a Portland Type 1 cement, and is viewed as one of the most common types of cement generally used throughout the world. The process of making this cement powder begins by heating limestone and other materials to a high temperature and after a chemical process called calcination, a resulting substance called a “clinker” is then pulverized into a powder to make Portland cement. Adding an aggregate and water to this cement powder will create common concrete. For this study, the focus will be the mechanical properties for cement past
Tricalcium Silicate (C₃S) is typically responsible for the strength early in the curing process as well as the initial set of the cement paste. As the percentage of this compound increases, so does the early age strength. The Young’s modulus for Tricalcium Silicate is 135 GPa.

Dicalcium Silicate (C₂S) takes longer to contribute to the cement and will not be effective until past 7 days of curing. The Young’s modulus used is 130 GPa. [1] Tricalcium aluminate (C₃A) is a main cause for the large amount of heat expended in the early stage of curing. With the C₂S and C₃S, this compound contributes to the strength increase early on due to the heat of hydration. The Young’s modulus was found to be 138.7 GPa through computational and nanoindentation [2]. Tetracalcium Aluminoferite (C₄AF) has a slight contribution to strength gain, and is a cause of the color of the gray cement. The Young’s Modulus for tetracalcium alumniferrite is taken to be 125 GPa [2]. Portlandite (CH) has average Young’s Modulus of 60 GPa and a Poisson’s ratio of .23 which is calculated for an isotropic polycrystalline aggregate [3], which would be expected in this form to exist in the presented mixes.

Calcium silicate hydrates (CSH Gel) makes up a larger percentage of the cement, and thus it is expected to influence the mechanical properties of cement mixtures, in general. Unfortunately, determining mechanical properties for CSH experimentally is very difficult and previous research has shown that the experimental values and literature values do not agree for this constituent [4]. The large discrepancy between theoretical and experimental values are often up to five times larger, with the greater being theoretical. Therefore, a value for Young’s Modulus of 25 GPa, which comes from experimental [4], is taken for this parameter. All other constituents have their values for Poisson ratios and Young’s modulus. used in the ABAQUS® model, are from the research group mainly involved in the creation of VCCTL [5].
Glass microspheres are hollow glass spheres (HGS) that are very lightweight, usually used in compositions with epoxies, urethanes, and polyester resins. They mainly provide benefits in weight reduction, reduction in shrinkage, and water resistance. Using these spheres in conjunction with a cement paste should provide the resulting matrix composition with some benefits. They could help fill many voids left during the curing process of cement as well as not interfering with the curing process itself. The drying shrinkage of cement could possibly be reduced by these glass spheres as well. The spheres effectively would limit the number of pores that the moisture can evaporate from, thus limiting the cement shrinkage. The HGS could also possibly provide a decrease in density with no loss of strength to cement mixtures. These microspheres have a maximum working pressure (isostatic crushing strength) and consistent particle size distribution which is advantageous towards the computational modeling of this cementitious material.

Current applications are currently limited to particular fields, with one being off shore drilling. An example of these glass spheres being used is described as a development for “lightweight cement slurry using hollow glass bubbles to reduce density to as low as that for water or less” [6]. These bubble slurries were mainly used to reduce the density below normal conventional low-density cements but higher strength than the usual low-density cements is needed. There are also patent documentations describing the use of glass microspheres for reducing densities while having a higher strength than with other cementing compositions that reduce density as well [7]. The applications for these bubble slurries are for deep ocean drilling, where the cement is used as a binder between the pipe and the surrounding substrate. The cement in this use provides insulation and holds the pipe in place for use.
An example of inert fillers is limestone, which is used to reduce the amount of cement powder. In 2004, the ASTM International passed a standard in which the incorporation of up to 5% mass fraction of limestone and will not affect the performance of the Portland cement [8], even higher with lower water to cement ratios (<.45). A report shows that up to 10% mass replacement of cement by limestone powder only has a reduction in 28-day strength of 6-7% [9].

Another example of an inert filler is the Class F fly ash. Torkittikul and Chaipanich in 2010 explored the use of ceramic wastes as aggregate within Portland cement and fly ash concretes and found that the already viable combination of concrete increased in compressive strength up to 100% with increasing ceramic waste content. They also found that fly ash slump is very similar to Portland Cement concrete with a difference of only 10 mm [10].

Lee et al. from Kunsan National University found through testing of class F fly ash that fluidity increases as particle size distribution becomes wider [11]. Naik of the University of Wisconsin-Milwaukee found that the optimum water to cementitious ratio of cements including Class C or Class F fly ash ranged from 0.35 to 0.6 for their investigation [12]. Bentz et al. observed a percolation-type relationship between yield stress and cement particle density and that plastic viscosities were linear functions of either total particle surface area or total particle density concerning cement and fly ash mixes [13].

Chindaprasirt’s team found that replacement of Portland cement by original fly ash increased porosity but decreased average pore size, while a finer Class F fly ash showed decreased porosity and average pore size compared to the original cement [14].

Lam’s team from Hong Kong Polytechnic University explored the hydration of cement with fly ash replacements and found that high volume fly-ash pastes underwent a lower degree of
fly ash reaction, and in some pastes more than 80% of the fly ash remained unreacted after 90-days of curing. Lam found that the effect was more pronounced in lower water-to-binder ratios, so those cement mixes suffered less losses in strength [15].

Waste glass comes from recycled beverage bottles, windows, dishware, and any other glass material thrown away. Worldwide around 14 million tons of this glass is disposed of each year [16]. Waste glass has also been studied thoroughly in previous research studies mostly yielding positive results. One of the disadvantages with waste glass is an alkali-silica reaction described in detail by Topcu and Canbaz [17].

Shi and Zheng [18] used waste glasses as their entire portion of aggregates in concrete mixes. They found that the concrete mixtures with the smallest particle sizes (38 µm) of the waste glass, had the highest strength, while their largest particle (150 µm) mix designs show below average results. Furthermore, they cite studies, published in 1995, indicating that at 90 days, the glass content increases the cement paste up to 25%. Overall conclusions by Shi and Zheng are the use of waste glass as cement replacement or aggregate is a valid concept without compromising on cost. The microspheres in this study have the benefit of the waste glass properties while having a simple structure, which is beneficial during modeling.

The main objective of this thesis is to computationally model the effect of volume percentage of additive like HGS on the mechanical properties of cementitious materials. Taking advantage of a fully developed suit of a software known as Virtual Cement and Concrete Testing Laboratory VCCTL at the National Institute of Science and Technology (NIST), the resulting microstructures of cement due to the hydration process for a water to cement ratio of 0.4 were determined. The evolved microstructures from VCCTL were explicitly modeled in the commercial finite element code, ABAQUS® to determine the elastic tensor. In the finite element
modeling, a representative volume element (RVE) approach with appropriate boundary conditions was employed to establish the effects of glass microsphere type additives on weight reduction as well as on mechanical properties. The computationally determined properties, such as Young’s modulus and Poisson’s ratio for 80% degree of hydration were compared with values obtained from experimental data for different HGS volume fractions. Section II describes cement as well as the HGS characterization in terms of both chemical composition and mechanical properties of individual constituents. This section also provides the microstructural aspects of the microspheres. The Section III provides details of experimental test methods and sample preparations. The experimental results are presented in Section IV. The results and discussions in Section V focused on the RVE based modeling efforts using the three dimensional ABAQUS® code. A comparison study between experimental data and computationally obtained elastic properties is presented in Section VI. Section VII provides summary and conclusions.
II. MATERIAL CHARACTERIZATION

II-A Cement

The type I Portland cement used in experimental and computational analysis was purchased from Buzzi Unicem USA. Buzzi also supplied a chemical analysis for a sample of cement powder sent to their testing facilities. Using these constituents, the base microstructure was designed using VCCTL. This structure is the base microstructure design in which all computational mix designs were developed from. The exact results of the completed chemical analysis are used to build this base hydrated microstructure. The makeup of this fully hydrated microstructure is shown in Table 1.

Table 1: Type I Portland Cement Constituents as provided from the VCCTL fully hydrated microstructure. The composition shown is typical for a type I cement.

<table>
<thead>
<tr>
<th>Constituents</th>
<th>%</th>
<th>E (Gpa)</th>
<th>ρ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saturated Porosity</td>
<td>21.53</td>
<td>1</td>
<td>0.49</td>
</tr>
<tr>
<td>C3S (Alite)</td>
<td>4.03</td>
<td>145</td>
<td>0.314</td>
</tr>
<tr>
<td>C2S (Belite)</td>
<td>1.89</td>
<td>130</td>
<td>0.314</td>
</tr>
<tr>
<td>C3A (Tricalcium Aluminate)</td>
<td>0.12</td>
<td>138.7</td>
<td>0.314</td>
</tr>
<tr>
<td>C4AF (Calcium Aluminoferite)</td>
<td>3.91</td>
<td>125</td>
<td>0.314</td>
</tr>
<tr>
<td>Gypsum</td>
<td>0.00</td>
<td>45.7</td>
<td>0.33</td>
</tr>
<tr>
<td>Portlandite (CH)</td>
<td>15.19</td>
<td>42.3</td>
<td>0.315</td>
</tr>
<tr>
<td>CSH gel</td>
<td>40.52</td>
<td>18</td>
<td>0.25</td>
</tr>
<tr>
<td>Hydrogarnet (C3AH6)</td>
<td>3.63</td>
<td>18</td>
<td>0.25</td>
</tr>
<tr>
<td>AFT (generic ettringite)</td>
<td>2.27</td>
<td>18</td>
<td>0.25</td>
</tr>
<tr>
<td>AFT (with Fe substitution)</td>
<td>3.12</td>
<td>18</td>
<td>0.315</td>
</tr>
<tr>
<td>AFm (monosulfate)</td>
<td>2.65</td>
<td>42.3</td>
<td>0.315</td>
</tr>
<tr>
<td>Fe(OH3)</td>
<td>0.77</td>
<td>18</td>
<td>0.25</td>
</tr>
<tr>
<td>Gypsum Absorbed by CSH</td>
<td>0.36</td>
<td>35</td>
<td>0.32</td>
</tr>
</tbody>
</table>
II-B Hollow Glass Microspheres

One of the two types of glass microspheres used in this study come from a company named Freeman Manufacturing (FM) and Supply, and are listed as Mia 65 Glass Bubbles. The properties of these glass spheres include a true density of .19 g/cm$^3$ and an averaged particle size of 72 microns with a particles size distribution between 5 and 150 microns. The isostatic crushing strength of these spheres is 500 psi. These properties are provided by the manufacturer and are shown on the technical data sheet which was provided with the product. An initial optical microscopy was performed to determine characteristics of the particle size distribution of these glass microspheres early in the research process. Three images were taken using an in-house optical microscope to get an initial estimation of the sphere dimensions. These images are shown in Figure 1. Measurements of clearly defined spherical images are also shown in these images.

With the outside diameters of a selection of spheres known, a STAT-17 approach towards determining the Weibull probability density function shape is then implemented. STAT-17 is a composites materials handbook originally developed by the Department of Defense for the statistical analysis of composite materials for aerospace applications.

A two parameter Weibull function is determined from 40+ of the FM glass microspheres measured from microscopy. Equation 1 is the Weibull function. Where $\beta$ is the shape parameter (slope), $\eta$ is scale parameter, and $t$ is the normalization around the mean. An example of the particle density functions is shown in Figure 2.
**Figure 1:** Optical Microscopy images of FM Glass Spheres taken from the structures lab at the University of Mississippi. Diameters of the spheres were measured with this process.

\[
f(t) = \frac{\beta}{\eta} * \left(\frac{t}{\eta}\right)^{\beta-1}e^{-\left(\frac{t}{\eta}\right)^{\beta}}
\]  

(1)
Figure 2: The normalized particle diameter found through Weibull function and STAT-17 analysis. This normalization is in relation to the measured average wall diameter of the FM glass microspheres.

These dimensions were further refined with the use of SEM imagery, provided by the Natural Products Center at the University of Mississippi, taken by Vijayasankar Raman Ph. D., and are shown in Figure 3. These high-resolution images enable the outer wall dimensions to be much clearer visually, thus allowing for a much more accurate dimensional analysis of the glass spheres.
Figure 3: SEM of the FM glass microspheres taken by the Natural Products Center located at the University of Mississippi Pharmacy School. These images, as well as the others in Appendix A, provide an outside wall diameter used in the STAT-17 analysis. Both images have scales of 20 µm and x650 zoomed in.

A second set of glass microspheres were obtained from 3M™. A true density value of .56 (g/cc) and average micron size of 36 are associated with these spheres. These values as well as a particle size distribution (PSD), shown in Figure 4, comes directly from 3M™ itself and therefore the need for a STAT-17 analysis and SEM imagery were not necessary.

Figure 4: Particle Size Distribution of 3M™ Glass Spheres as provided by 3M™. The microspheres were tested with a LS Particle Size Analyzer, providing a volume percentage as a function of particle diameter (µm).
The 3M™ glass spheres have an isostatic crushing strength of 10,000 psi. 3M™ provides many other types of glass microspheres with varying properties. A comparison of isostatic crushing strengths for different types of glass spheres is shown in Figure 5. In this study, the H50 EPX was selected for the testing.

![Figure 5: 3M™ Glass Microsphere product isostatic collapse resistance comparisons. The spheres used in this study are highlighted in the red oval.](image)

Using these diameters and densities, the wall thickness of the microspheres is then calculated for both types of glass spheres. The wall thickness is necessary for when determining elastic properties of these microspheres for computational testing. The equation for relation between the inner and outer radius of the glass microsphere is shown as Equation 2:

\[
R_i = \sqrt[3]{R_o^3 - \frac{R_o^3 \rho g_s}{2.54}}
\]

(2)
Where \( R_0 \) is the outside radius, \( \rho_{gs} \) is the true density of the glass microsphere as determined by a gas pycnometer from the manufacturer. And 2.54 is the density of solid glass. Using the inside and outside radius, the wall thickness is then easily calculated.

The wall thickness is then used to calculate the stress on the material if a small differential change in pressure is applied. This is shown in Equation 3 below:

\[
\sigma = \frac{\Delta p D}{4t}
\]  

(3)

Where the differential pressure is \( p \), \( D \) is the diameter of the sphere and \( t \) is the wall thickness. Using this stress value the strain is calculated by:

\[
\varepsilon = 3 \left( \frac{\sigma}{E} \right) (1 - \nu)
\]  

(4)

The change in volume is calculated by applying the strain to the original volume, which was found through the relation:

\[
V_o = \frac{\pi D^3}{6}
\]  

(5)

Where \( V_o \) is the original volume and \( D \) still refers to the diameter of the spheres. The change in volume is found by simply multiplying this original volume by the calculated strain. Using this change in volume, \( \Delta V_o \), the effective bulk modulus of the sphere is determined by:

\[
\frac{pV_o}{\Delta V_o} = \bar{k}
\]  

(6)
Where $p$ is the differential pressure, applied earlier in Equation 3, and $k$ is the effective bulk modulus. There has been some work done experimentally to determine these mechanical properties of different types of hollow glass microspheres [19-26]. However, this value for the present study needs it to be the effective bulk modulus due to how the microstructures are built in VCCTL. The spheres are modeled as solid in ABAQUS® but they are in reality, thin hollow shells. This is reason for the use of effective bulk modulus from experimental results.
III. EXPERIMENTAL METHOD

III-A Mix Design

The mechanical properties of the Type I Portland cement with and without both sets of glass microspheres have been obtained through the experimental testing of cylindrical cement samples. These samples were made from single use plastic molds with dimensions of 76.2 mm in diameter and 152.4 mm in height, in accordance with ASTM standard C192 [27]. A water to cement ratio of 0.4 was chosen for all samples, so as to have enough workability, while also not significantly separate the microspheres from cement as mixing occurs. The samples were made in seven separate batches with the first four consisting of the cement-only mixture and a 5, 10, and 15\% volume fraction of the glass microspheres which came from the Freeman Manufacturing Company (FM). The last three batches of mixes consisted of a 5, 10, and 15\% volume fraction of the glass microspheres from 3M™.

The preparation method for each mix and both batches was exactly the same. Before batching of all the mix designs, every component of the mixes was measured out ahead of time. This was to help to complete the batches all in the same day, which benefits the mechanical testing process. The mix designs showing the mass and volume fractions for each mix are in Table 2, while the actual masses during the mixing process are in Table 3.
Table 2: Mix design percentages used in determining the amount of mass for each batch. The FM and 3M™ will have slightly different mass values because of the difference in their densities.

<table>
<thead>
<tr>
<th>Mix #</th>
<th>Mass Percentage</th>
<th>Volume Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cement</td>
<td>Glass Spheres</td>
</tr>
<tr>
<td>Cement</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>Glass 5VF</td>
<td>99.16</td>
<td>0.84</td>
</tr>
<tr>
<td>Glass 10VF</td>
<td>98.25</td>
<td>1.75</td>
</tr>
<tr>
<td>Glass 15VF</td>
<td>97.25</td>
<td>2.75</td>
</tr>
</tbody>
</table>

Table 3: Masses for each mixing component for batches made for all testing.

<table>
<thead>
<tr>
<th>Inert Filler</th>
<th>Cement (kg)</th>
<th>Water (kg)</th>
<th>Glass Spheres (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>None</td>
<td>14.52</td>
<td>5.81</td>
<td>0</td>
</tr>
<tr>
<td>3M™ Glass 5VF</td>
<td>14.32</td>
<td>5.73</td>
<td>.27</td>
</tr>
<tr>
<td>3M™ Glass 10VF</td>
<td>13.99</td>
<td>5.59</td>
<td>.53</td>
</tr>
<tr>
<td>3M™ Glass 15VF</td>
<td>13.73</td>
<td>5.49</td>
<td>.79</td>
</tr>
<tr>
<td>FM Glass 5VF</td>
<td>14.42</td>
<td>5.77</td>
<td>.11</td>
</tr>
<tr>
<td>FM Glass 10VF</td>
<td>14.33</td>
<td>5.73</td>
<td>.19</td>
</tr>
<tr>
<td>FM Glass 15VF</td>
<td>14.23</td>
<td>5.69</td>
<td>.29</td>
</tr>
</tbody>
</table>
III-B Sample Preparation

Each set of samples were then mixed in a concrete mixer located in the University of Mississippi materials lab then vibrated the cylinders as per ASTM standard C192. After 24 hours, the plastic molds were removed from now solid cylinder samples as per ASTM C192. An example of these plastic molds is shown in Figure 6.

![Plastic Cylinder Mold](image)

**Figure 6:** Plastic Cylinder Mold for cement testing. The diameter is 3 inches and the height is 6 inches. These were removed after 24 hours of cure time.

Once each cylinder was removed from the plastic molds the samples were then placed in a water storage container following ASTM C511-13 [28] until time of compressive and elastic testing. An example of a typical sample that just had its removal of the plastic mold is shown in Figure 7.
Figure 7: An example of a cement cylinder sample directly after removal from the plastic mold. Dimensions were measured and the mass was recorded immediately before testing.

These containers consist of a five gallon buckets with saturated lime water in line with requirements of ASTM C511. The samples were kept in these containers until immediately before the mechanical testing for elastic modulus and ultimate compressive strength determination.
III-C Mechanical Testing

The compressive strengths of each set of samples were determined by compressing the samples using an 810 Material Test System. Once the samples were placed, and aligned in the MTS machine, the load was then increased until failure for each sample. An example of a typical failure shown in Figure 8 represents a Type 1 failure which is defined by ASTM C39 as “reasonably well-formed cones on both ends with less than 1 inch of cracking through caps” [29].

![Type 1 cone failure](image)

**Figure 8**: Type 1 cone failure, typical of all failures for the mix designs tested. Further images and descriptions are shown in Appendix B.

All mixtures failed in similar fashion and can be described as Type 1 failures. Further evaluations of these failures are shown in Appendix B. The loading capacity of the MTS machine and load cell is 110 kips shown in Figure 9. The loading rate for the MTS machine is .001 unit/sec and is set to a maximum of 1.8-inch compression displacement. A data acquisition system (DAQ)
is used for recording of the load as function of time and to record the strain values as the load is increased to 40% of the maximum load, and this was done three times for modulus determination. The sampling rate for the load data and the strain data is 1000 Hz. This DAQ is shown in Figure 10, and is a NI PXIe-1073. A simple LabView code was implemented to record this data. Two linear displacement sensors, one in the vertical and one in the horizontal direction, on a compressometer sent the displacement values to the DAQ to be recorded. The compressometer, seen in Figure 11, follows the ASTM standards for quasi-static compression testing.

Figure 9: A 810 MTS used for the quasi-static testing of all cement samples.
**Figure 10:** NI DAQ system, with two data inputs for vertical and radial displacement measurements. Another channel was taken from the 810 MTS, which provided a voltage for the current load on the system.

**Figure 11:** Compressometer with two linear displacement sensors, one in the horizontal position and the other in the radial direction. These were measured at a 1000 Hz sampling rate.

Initially the displacement voltages recorded were difficult to decipher, an example of this recorded data is shown in Figure 12A, plotted through a simple Matlab code. Several moving averages were implemented through Matlab and the resulting data is much clearer and shown in
Figure 12B. The compressometer itself was validated by having Delrin cut to the exact dimensions as the cement samples were. The Delrin was then tested just as the cement samples would have been. The calculated modulus from the data was within 10% (1.9 GPa) of the known modulus (2 GPa) for this exact composition of Delrin.

Figure 12: A) Represents the raw displacement voltages as a function of time for the vertical position. B) Various moving averages used to clear up the signal for better strain accuracy.
IV. EXPERIMENTAL RESULTS AND DISCUSSION

The moduli for all batches, tested at 14, 21 and 28 days were calculated from the data acquired by LabView. The calculated Young’s moduli for both the FM and 3M™ glass mixtures are shown in Figure 13. Each bar on the figure represents an averaged value for five cement samples with a standard deviation of 1. The modulus for these cement samples remains relatively between 8-12 GPa for all mix designs and cure times. There also appears to be no consistent trend in relation to the glass microsphere inclusions, although there are variances between the magnitudes themselves.

![Figure 13: A) FM and B) 3M™ Young’s Modulus averages for each testing date with standard deviations for each date and mix design.](image)
The compressive strengths for all the samples are shown in Figure 14. As before each bar represents an averaged value of five samples with a standard deviation of 1. These are found by using the ultimate load values, which were taken at failure after the modulus testing. The FM and 3M™ each have an outlier for the 5VF batch. Outside of this value, the compressive strengths remain between 30 and 40 MPa which is widely acceptable for Type I cements [24]. There is no noticeable increase or decrease of strength as the percentage of glass inclusions increase.
Figure 14: A) FM and B) 3M™ compressive testing results, with the maximum load used in calculating the compressive strengths.

The modulus of resilience is found by using the calculated compressive strengths and moduli through Equation 7. As previous data, these values have a standard deviation of 1 but this deviation is much larger than other data sets. Other than the outliers, the values appear to be centered on 50kPa with no loss of MOR as the glass replacement increases for both types of
microspheres, shown in Figure 15. Where $\sigma$ is the compressive strength, and $E$ is the modulus, for a solid glass. There is no decrease in this property as the inert fillers are increased in the mix designs.

$$MOR = \frac{\sigma^2}{2E}$$

(7)

**Figure 15:** A) FM and B) 3M™ Modulus of Resilience for the averaged values, a relation between the moduli and the compressive strengths.
Next the specific strengths (SS) is now shown and similar to previous figures, comes with a standard deviation of 1. This is a relationship between the calculated compressive strength and density of the material is shown in equation 8. Where \( \sigma \) is the compressive strength and \( \rho \) is the density for each sample. The results of this relation are given in Figure 16. These values show that there is no relative increase or decrease as the glass microsphere amounts are increased. Maintaining the same mechanical property values while having a decrease in the density is invaluable towards structural materials.

\[
SS = \frac{\sigma}{\rho}
\]  

(8)
Figure 16: A) FM and B) 3M™ specific strengths calculated through the relation between densities and compressive strengths.

The mass of the samples was measured immediately after removal from curing and before quasi-static testing as in accordance with ASTM C469 [30]. The reductions in mass for the FM glass mixtures are 1.6, 6.4, and 8.5 % respectively while the 3M™ mixtures reductions are 3.7, 7.9, and 11 %. This is significantly advantageous to have these reductions with no
decrease in the previously mentioned mechanical properties. Figure 17 shows these averaged mass values for each testing batch. No standard deviation was needed due to the extremely small difference in mass between samples of the same batch, less than 1%.

**Figure 17:** A) FM and B) 3M™ Masses of the samples measured immediately after removal from curing area, and right before quasi-static testing.
Density can be expected to decrease as well, similar to the mass and this is shown in Figure 18. The FM mixtures were reduced 3.4, 7.7, and 9.8 % respectively while the 3M™ reduced 3.7, 8.7 and 12.3 %. As before with the mass comparisons, this is significant when the mechanical properties remain relatively constant up to the highest percentage of microspheres, but reduction in mass properties occur.

**Figure 18:** A) FM and B) 3M™ averaged values for each testing date for all mix designs.
Specific strength, determined by equation 8, is a relation between the compressive strength and the density of the material. Overall the specific strengths between the mix designs remains within 30%, this goes down to 15% if the outliers are excluded. This means while the samples are lighter in density, they still have a compressive strength of the same ratio as the cement paste. Comparing the cement paste and both FM and 3M™ at 15%, the specific strengths are either the same or higher in favor of the glass inclusion mixes. Increasing the percentages of glass in further research would benefit the analysis for this relationship.

The modulus of resilience, as shown in equation 7, is calculated through the relations of the compressive strength and modulus of the sample. As with the specific strength, excluding the outliers, the data shows consistent values. Also, the cement paste has a similar or smaller MOR than either of the 15% inclusion samples. The fully cured samples with 3M™ glass are all almost twice as large than the cement paste in their data set. Again, a larger data set with an increased amount of percentages of glass inclusions would beneficial.

The mass of any cementitious material based structure is one of the most important factors to account for. In this case, the materials clearly show a minimal change in mechanical properties. However, the mass and densities clearly are affected by the glass microspheres. With this inert filler, structures would have significant decrease in the weight of the cement and thereby reducing the weight of many structural materials. This benefits many different aspects of the process. Such as, structures having many more options to be built on if they are 10% or more lighter than normally designed. Another benefit being a reduction in cement reduces prices of procurement to construction sites, whether it be through purchasing, shipping, manufacturing or any combination thereof.
V. COMPUTATIONAL DEVELOPMENT, RESULTS, AND DISCUSSION

V-A VCCTL

The known constituents of the Portland cement used in experimental testing were used to create a unique cement mix in VCCTL. Table 1, from chapter 2, shows the base cement composition. This base composition was hydrated through VCCTL and became the control of pure cement paste. The next six mix designs used this same base cement composition, but with inclusions of glass. VCCTL took the glass as an inert filler, which replaces cement in the composition, and lowering the amount of water needed by a slight amount. These seven microstructures are the building blocks for the ABAQUS® analysis shown in the next major section.

The microstructure images for a Type 1 Portland Cement are shown in Figure 19. These images are a 2-D cross section of a 100 x 100 x 100 micron cube surface. The views of each image are in the planes of xy, xz and yz, respectively. These figures produced by VCCTL show only the surfaces of each of the planes. This output, of a hydrated microstructure, was found to be in a Microsoft Word formatted file, which is then ported to ABAQUS® through an in-house developed Matlab code. This transition from VCCTL to ABAQUS® is discussed in the next section of the chapter.
Figure 19: A) XY B) XZ C) YZ 2-D Portland cement microstructure developed with VCCTL and viewed through three different planes. Each color represents a different constituent.
V-B ABAQUS® MODEL GENERATION

The word file produced by VCCTL has all constituents listed individually in a single 1 x 1,000,000 list. Each number in the list represents a different chemical composition throughout the representative volume element (RVE). The micro-structure voxel information from NIST software VCCTL for a given particle distribution is imported into the general purpose ABAQUS® finite element code using an in-house developed Matlab® code and modeled as a micro-scale representative volume element (RVE) using continuum hexahedral elements to generate RVEs 100x100x100 microns in dimension. Figure 20 is the resulting RVE microstructure for a US Type I Portland cement, and Portland cement with a 5 % volume replacement of the FM glass microspheres. While, Figure 21 is the 3D spatial locations in the RVE of the microspheres for a 15% volumetric replacement for the FM (A) and 3M™ (B) glass inclusions.

**Figure 20:** A) Type I Portland Cement and B) FM Glass 5 % RVE representations of the microstructure created through the VCCTL hydration process. The ISBC are applied to the RVE’s, for all mix designs.
Figure 21: A) FM and B) 3M™ 15% spatial locations and particle size variations of the glass microspheres in their RVE’s.

The computational analysis includes the import of the batch specific chemistry for Portland Type I cement, used in the experimental investigation, into VCCTL to generate Representative Volume Elements (RVEs) for cementitious microstructures at various levels of microsphere inclusion. RVE-based Iso-Strain Boundary Conditions (ISBC) [31] have been employed to numerically approximate the elastic constants of the composite cement pastes. An example of these ISBC applied in ABAQUS® is shown in Figure 22, for a strain applied in the principal X-direction. The surface reactions are shown for the x, y and z directions. The strain is also applied to the other principle directions for their separate surface reactions. With these values, the elastic constants are calculated and shown in the results chapter.
Figure 22: ISBC on the Portland Cement RVE, representing the surface reaction forces from the .01 strain applied, in this case, the principle X-direction.
V-C HASHIN APPROXIMATION

The microstructures for a given particle distribution of various constituents are explicitly modeled in the finite element code ABAQUS® as a 100 micron cube RVE. The effective Young’s Modulus was 24 GPa and Poisson’s ratio was 0.33 for the RVE which represented the control cement of pure cement. Similar values have been found in open literature using other models [5]. This is much higher than the experimental data, so an approximation has been developed for softening the constituents in the model to agree with experimental testing. A Hashin pore approximation model [32] has been used to predict the effect of included porosity on the quasi-statically determined elastic modulus.

The moduli of the constituents are softened by using a microstructure provided by Hashin’s model of composite-sphere assemblage, with a derived approximation from Ramakrishnan:

\[
\frac{E}{E_s} = \frac{(1-\phi)^2}{(1+2\phi-3v_s\phi)} \quad (9)
\]

\[
\nu = \frac{(4v_s+3\phi-7v_s\phi)}{4(1+2\phi-3v_s\phi)} \quad (10)
\]

Where, \(E_s\) is the Young’s modulus of the constituent as a solid particle, \(v_s\) is the Poisson’s ratio, and \(\phi\) is the porosity. Both \(E\) and \(\nu\) are the resulting parameters that are solved for, as porosity is varied. These new parameters are then implemented in ABAQUS® for elastic constant determination.
VI. COMPUTATIONAL RESULTS

The first step in validating the results from the F RVE model is determining how varied microstructures were. So, using VCCTL, the FM glass microstructure with a 15% of glass inclusion was created nine times. These microstructures are equivalent in their porosity and constituent percentages but with different orientations. Each of these structures were implemented in ABAQUS® with the results shown in Figure 23. The orientation of the constituents shows to have a minimal effect on the overall elastic constants. For the FM glass at 15% inclusions, there is a less than five percent difference between the lowest and highest calculated modulus.

![Graph showing modulus values for different microstructures](image)

**Figure 23:** Random configuration of the constituents for FM Glass of 15% mix design. Each microstructure was developed and hydrated through VCCTL, with all the same percentage of constituents but the configuration for each structure is unique.
In Figure 24 the resulting moduli are shown for each mix design for a 20–30% porosity range. Each data point represents an RVE for each particular mix design. To determine the modulus at each porosity increment, the Hashin softening effect is used for all constituents except the glass spheres. These values are implemented in ABAQUS® and the RVE has each surface locked in position except for a primary direction, \((X_1, Y_1, \text{or } Z_1)\). A strain of .1 is then applied and the resulting surface reactions are computed for each direction. This means running the model three times for each RVE of each mix design for each porosity amount shown in the figure. Approximately 1.5 hours is the amount of time it normally took for each RVE to finish. Therefore, Figure 24 represents over 300 hours of computational time.

**Figure 24:** Modulus for all mix designs as a function of porosity, calculated through the ISBC analysis through ABAQUS®. These values are within 10% of the experimentally calculated data.
The range of 20-30 % porosity was chosen because of the experimental moduli. Using a very simplified volumetric summation for Young’s modulus, the effects of microscale porosity are shown in Figure 25 for a pure cement paste microstructure. The modulus as a function of porosity was found by summing the moduli each element in the RVE, and dividing out by the total number of elements.

Running the full model for each mix design for all porosity ranges would have taken ten times as longer, but this quick summation of elements provided a range to focus on computationally.

Figure 25: Volumetric approximation of modulus for a Type I Portland cement. This is a simple summation of modulus for all the elements in the RVE and averaged out based on the volume of this RVE itself and plotted as a function of Hashin porosity. Most experimental results fell within the 25-40% porosity range.

The non-uniform distribution in Figure 23, which shows the resulting moduli for several FM 15 % glass inclusion microstructures, is minimal. The magnitude differences seen are well within 10%, thus providing validation of the model. The values for the Young’s modulus, which are shown in Figure 24, show magnitudes higher than the cement paste for 3M™, while the FM
samples have lower moduli than the cement paste. As the Hashin porosity increases, the moduli for all cement mix designs decrease at the same rate. This Hashin approximation generally is accurate up to ~15%; with the 25% estimated from experimental data, there will be some variances to the actual values. Future work would include an interactive pore model, which takes into account this accuracy issue.

Discrepancies between the computational results and experimental could be attributed to the amount of strain applied. In the compression testing, the strain was taken to failure, while the ISBC analysis only applies a small amount of strain in comparison.

The shear conditions were not applied due to the isotropic nature of the results from the ISBC. The modulus determined through those boundary conditions were relatively similar (<5%) in magnitude for all the principle directions, leading to the isotropic assumption.

This analysis process is much faster than the experimental testing. With batching, mixing and testing, the process takes at least 28 days from beginning to end as well as many man hours. The ISBC in conjunction with VCCTL provides a quicker result with a good deal of accuracy. Computational evaluation of a single mix design would take approximately 3 hours from beginning to end.
VII. COMBINED DISCUSSION

The experimentally calculated Young’s modulus for the pure cement paste control samples are within 15% of all other mix designs, from Figure 13. If the two outliers, FM 5 and 3M™ 5 on day 14 and 21 respectively, are excluded then all samples have moduli within 10%. This consistency leads to the validity of the mixing process and that the elastic constant in this case is still dominated by the cement paste. Also, significant changes in the moduli over time are not expected until +30 days into curing [22]. The modulus is also affected by the degradation of the cement powder [33] used in the mixing process, but since all samples used the same powder this can be neglected as contributing factors to the mechanical properties. Having a consistent modulus throughout all samples benefits later calculations such as the modulus of resilience. The modulus of resilience relies on the compressive strength as well. Using the Young’s Modulus and Poisson’s ratio, both the shear and bulk moduli are easily found through basic equations. The ISBC show the same modulus for each principal direction which allows the use of these relations; the shear modulus \( G \) through Equation 11 and bulk \( K \) through Equation 12. However, since the Poisson’s ratio changes minimally as does Young’s modulus, the bulk and shear follow this trend. These values are shown in Table 4 the experimental at 28 day testing and computational at 25% porosity, and the Poisson’s ratio is taken as .3 for all.

\[
G = \frac{E}{2(1+v)} \tag{11}
\]

\[
K = \frac{E}{3(1-2v)} \tag{12}
\]
Table 4: Bulk and Shear modulus for the experimental and computational data results. The experimental values use E values on the 28 day cure time, while the computational uses the 25% porosity value for its calculations.

<table>
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<td>Bulk (GPa)</td>
<td>Shear (GPa)</td>
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Compressive strength is the one of the most essential mechanical properties for cementitious compositions. Excluding outliers, all samples exhibited compressive strengths between 30 and 35 MPa, which is typical of most Type I Portland Cements. Glass inclusions showed no decrease in the compressive strength as their amount increased. This further validates that the glass spheres have minimal or no effect on the mechanical properties of the material composition up to 15% inclusion consistent with the results shown by Nemes et al [26].
VIII. CONCLUSIONS

Through a combined experimental and computational effort, the elastic properties have been determined for cementitious materials with small inclusions of hollow glass microspheres. The amount of these inclusions in the type I Portland cement were 0, 5, 10, and 15% by volume. The two types of hollow glass microspheres showed no distinct differences mechanically, but both reduced the total mass of the material. The use of VCCTL was to create a valid microstructure representing the cement tested experimentally. Testing from Buzzi Unicem provides a chemically accurate percentage of constituents to create the unique VCCTL microstructure. These were explicitly modeled in the ABAQUS® finite element code under ISBC to determine the elastic properties.

The quasi-static compression testing followed ASTM standards for determining the compressive strength modulus of cylindrical samples. These property values were well within accepted magnitudes for pure cement pastes. No mechanical effects were seen from the inclusions of the glass spheres. However, these glass spheres produce a reduction of the materials’ mass and density by up to 10% with no observable changes in compressive strength or Young’s modulus. The amount of the inert fillers in this case should be tested at higher percentages in any future work to observe when this filler will start to affect the cement paste.
The computationally calculated elastic moduli compared reasonably well with data from quasi-static compression tests for various volume fractions of the glass microspheres. Differences between the types of glass inclusions themselves were minimal. The main trend seen is that the 3M™ glass inclusions all increase the modulus of the cement RVE while the FM glass inclusions are all below the pure cement RVE. This mainly can be attributed to their modulus in comparison with the calcium salts, which dominate the percentage of the microstructure. FM have a lower modulus than these salts while 3M™ have higher values.

Future work will include dynamic loading conditions involving high strain rates involving Split Hopkinson Pressure Bar. Blast panels could also be made for testing. These additional tests will give more insight into how the hollow glass microspheres can affect cementitious materials. More work could also include taking samples for high resolution SEM imagery, as this can provide valuable information on porosity. These images would validate the mixing process, ensuring the glass spheres are equally distributed among the sample.


LIST OF APPENDICES
APPENDIX A: SEM IMAGERY
APPENDIX B: CEMENT SAMPLES
14 Day 3M™ Glass 10VF Sample 5 ~30948lbs, Pores appear to have been filled with water which released upon failure. These pores contribute to the larger porosity overall and the Hashin softening method developed, corrected for this in the computational results.
This is a comparison of a typical failure seen throughout the experimentation. The difference is that these pictures are taken with and without a flash on the camera. Immediately is it seen that the one with the flash used highlights failure points. This also shows that this powdery substance which typically was present at failure regions is the cement itself and not glass microspheres, further showing this is still matrix dominated and this type failure is not due to the glass microsphere. Pics are 28 day Cement1 Sample 1 ~38000lbs
21 day FM Glass 5VF Sample 5 ~30000lbs, portions in which hydration had not occurred are more often in the lower sections of the cylinders. This cylinder was noted to have a particularly flat surface when compared to the other specimens.
This cylinder was damaged during the strain loading phase of modulus determination. It is possible if failed early due to this ~18000lbs the failure is also shown. It appears to have failed where previous damage had already occurred. Twenty one day Cement sample 3
A typical failure of the cement cylinder specimens is shown here. A cone shape is seen along the top pointing down. A larger cone holds the top up with a large failure crack down the middle. The missing cement sheared off dramatically. 21 day Cement1 sample 5 ~40000lbs

This figure shows an untypical failure with fractures occurring vertically throughout the specimen. The failure curve shows multiple fractures before ultimate failure. 21 day FM Glass 5VF Sample2 ~ 36000lbs
This figure shows another typical cone failure seen throughout testing for all specimen types. ~45000lbs
21 day FM Glass 5VF Sample 4.

Typical failure, the top cone fell off. Main failure crack split down the middle portions. Lighter colored powdered substance among failure region, previously noted as showing up in the cement only samples as well. 14 day FM Glass 15VF Sample 1 ~45000lbs
Another cone failure for 14 day FM Glass 10VF ~41800lbs. Most cement remained in place during failure, cone shapes are larger than in some and appears to have failed largely in shear.

Failure around 23000lbs, 14 day FM Glass 10VF Sample 2. Upper cone is larger, this occurrence rarely happened during testing of all cylinders. Both this and above figure have glass spheres in the same amount, failed in similar fashions, and have the same curing time, but very dramatically different in ultimate strength.
Another typical cone failure. Small groove made by demolding is seen in the before pictures. The groove runs vertically the full length of each specimen. Throughout testing it was seen to have zero effect on the structural integrity of the cylinder.
VITA

Zachary Joseph Wallace

2038 Cold Springs Road · Sardis, MS 38666 · (662)609-8188 · zjwallace87@gmail.com

EDUCATION

B.S. Mechanical Engineering, University of Mississippi, May 2013

M.S. Mechanical Engineering, University of Mississippi, May 2017

Thesis: Mechanical and Computational Evaluation of Cementitious Materials with Hollow Glass Microsphere Inclusions

TEACHING EXPERIENCE

Teaching Assistant, 2015-2017

University of Mississippi

Courses: Materials Lab, Fluid Dynamics and Lab

Research Engineer, 2013-2015

NCPA, SOAIR

Acoustics