Micro-Mechanical Characterization of UHPC Stiffness Mechanisms: Towards a Better Understanding of Concrete Elastic Modulus

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Micro-Mechanical Characterization of UHPC Stiffness Mechanisms: Towards a Better Understanding of Concrete Elastic Modulus

A dissertation submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy in Civil Engineering

by

Charissa N. Puttbach
LeTourneau University
Bachelor of Science in Engineering (Civil Concentration), 2019

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University of Arkansas

This dissertation is approved for recommendation to the Graduate Council.

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Abstract

Current design code equations for estimating the elastic modulus of specialty concretes like ultra-high performance concrete are not all suitable. Developing empirical curves from exhaustive experimental testing is challenging for concretes that may have high levels of cement replacement with mineral admixtures and fibers at varying volume contents. As such, an alternative approach is taken that seeks to understand the constituent microscale stiffness mechanisms and relate the results to the macroscale. Micropillars are fabricated on several cement paste phases using a focused ion beam. A modified pico-indenter is used to determine the linear and non-linear behavior under micro-compression loading. Micropillar phases are determined by their chemical compositions, identified with an energy-dispersive X-ray. The compressive strength and stiffness of C-S-H, C-A-S-H, SF, CH, AFt/AFm, and a CH/C-S-H are provided in this study.

With the stiffness of individual phases calculated and statistical curves developed, a novel homogenization scheme is proposed to combine cement paste phases. Considering phase stiffnesses as springs (in amounts consistent with phase volume fractions determined by X-ray powder diffraction) allows for them to be combined in series and parallel. Monte Carlo arrangement simulations are run in MATLAB. The same approach is used to homogenize UHPC (fibers, fine aggregate, and cement paste), and the code output is verified through initial experimental testing of cylinders. Cement paste homogenization through spring arrangement simulations proves to be an adequate approach. The code output falls within a 30% error when compared to two different hardened cement paste mixes, potentially within a 10% error if the upper portion of the bimodal distribution is considered outliers. However, when the UHPC homogenization is evaluated, the code fails to provide a reasonable estimation of the results.
Acknowledgements

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List of Published Papers


C. Puttbach, G. Prinz and C. Murray, “Homogenization of cement paste and UHPC through Monte Carlo spring arrangement simulations,” ******* (Submitted – Chapter 4)
Chapter 1. Introduction

1.1. Background and Motivation

The modulus of elasticity of concrete is an important parameter for the design of concrete structures [1-2]. Empirical curves are provided in design codes for determining concrete stiffness based on measured strength [3-6], but the application of these equations (developed for normal, lightweight and high strength concrete [4, 7]) to specialty concretes is questionable. Variations in degree of hydration, curing condition, aggregate type, and mixture ratios can impact the mechanical properties of concrete [8-10], requiring that new concrete designs (i.e. self-consolidating, ultra-high performance, fiber-reinforced, etc.) have a new empirical relationship developed. Many specialty concretes use replacements for cement and aggregate (such as fly ash, silica fume, metakaolin, recycled concrete, glass, etc. [11-12]) and/or add fibers (made from steel, glass, synthetic materials, etc. [13]), which affect the stiffness of the interfacial transition zone (ITZ) and therefore the overall concrete stiffness [8, 14-16]. Thus, conducting enough empirical testing to accurately represent the entirety of material possibilities for a specialty concrete is extremely time-consuming. An equation that is suitable for a mixture made with portland cement as the binder may not be suitable for a mixture with a 30% replacement of cement with metakaolin and a 4% fiber volume fraction to control cracking [17].

As an alternative, one area of research has shifted to focus on studying micro- and nano-scale elastic modulus to better understand the controlling mechanisms for concrete [18-23]. Microscale testing protocols have been developed for other materials and are steadily being applied to concrete research [24-25]. Two common approaches for testing the microscale stiffness of materials like sapphire [26], bone [27], wood [28], and nickel [29-30] are nanoindentation and micro-specimen fabrication. Nanoindentation pushes an indenter tip into the
material surface until a specified deformation or maximum force is reached, usually performed in a grid pattern [31]. Micro-specimens (i.e. pillars, beams, and dog-bone tension members) are fabricated using a focused ion beam (FIB) [32-34]. Testing allows for the determination of additional mechanical properties, i.e. tensile and compressive strengths [5, 35]. Nanoindentation has been used in concrete research for a few decades, but micro-specimen testing has only recently been explored.

Of course, understanding the micro-mechanical behavior of concretes is only limitedly helpful unless it can be related to macroscale observations. Early attempts to upscale phase elastic modulus were limited by computational ability and technological capabilities [36-37]. In more recent years, efforts have attempted to implement several different homogenization methodologies such as hydration models, statistical/representative volume elements, and machine learning with varying levels of success [19, 38-42, 44]. Multi-scale homogenizations are necessary due to the complexity of the concrete microstructure [43-44]. At a minimum, cement paste phases need to be combined prior to modeling their overall interactions with fine aggregate, coarse aggregate, fibers, etc. [44]. There is a need for an easily implemented design tool that allows for concrete elastic modulus prediction.

1.2. Objectives and Research Approach

The objective of this study is to determine mechanistic relationships of UHPC stiffness through a multi-scale investigation. This work seeks to fill literature gaps and provide a foundation for future research. To begin, the adequacy and shortcomings of current design code elastic modulus calculations are evaluated to determine the types of specialty concretes most challenged by the empirical equation approach. This includes a detailed review of literature and an evaluation of data collected from many different studies. With the need to understand
fundamental phase behavior through experimental testing, a bottom-up approach is taken. This study seeks to apply a micro-compression methodology to cement paste phases, which has the benefit of both determining the stiffness in a uniaxial stress state and providing novel compressive strength data for phases. Micropillar compression testing has only recently been applied to concrete and, so far, has only been used to study C-S-H [8]. For an appropriate homogenization tool to be developed, more phases, such as ettringite and calcium hydroxide, need to be studied. Specimens are fabricated using a FIB on the surface of a polished concrete sample. Phase identification is accomplished using an energy-dispersive X-ray (EDX). Uniaxial compression loading is conducted using a modified pico-indenter, providing a load-displacement curve from where the elastic modulus and compressive strength can be determined.

Efforts in literature to upscale micro-mechanical data tend to be complex and may require an understanding of finite element software [4, 7], particularly for combining cement paste with aggregates and air voids. In this study, a novel approach to cement paste and bulk concrete homogenization is taken (Figure 1-1 and Error! Reference source not found.). By considering each phase as a spring with a specific stiffness connected in either series or parallel with the next phase, a homogenized stiffness can be calculated. Maintaining volume fractions of phases (determined through X-ray powder diffraction), the constituent elements of cement paste are combined in different arrangements. To ensure the randomness found in concrete microstructure, Monte Carlo simulations are run, producing a homogenized stiffness for cement paste. The same methodology is then applied to a mesoscale analysis of concrete (consisting of cement paste, aggregate, fibers, and a micro-cracking parameter). Results from the Monte Carlo spring
arrangement simulations provide an elastic modulus that can be compared to bulk concrete testing. Initial experimental verification using two UHPC mix designs is also conducted.

There are limitations that need to be considered when examining this study. The machines used for micro-mechanical investigations are complex and take training and practice to be used effectively. Compounding that issue, equipment like the SEM and FIB do not work quite as well with concrete as they do with conductive materials like steel. As a result, micro-pillar fabrication is challenging, and the final specimen geometries vary between specimens. Additionally, the use of a vacuum for concrete micro-mechanical testing is not ideal, as it may cause a collapse of crystals within the microstructure [23]. Finally, developing a ready to use design tool is complicated and a full experimental verification of its limitations is outside the scope of this project. Instead, a proposed novel homogenization approach is developed, and initial experimental testing is provided for proof-of-concept.

**Figure 1-1:** Flowchart of Research Tasks

There are limitations that need to be considered when examining this study. The machines used for micro-mechanical investigations are complex and take training and practice to be used effectively. Compounding that issue, equipment like the SEM and FIB do not work quite as well with concrete as they do with conductive materials like steel. As a result, micro-pillar fabrication is challenging, and the final specimen geometries vary between specimens. Additionally, the use of a vacuum for concrete micro-mechanical testing is not ideal, as it may cause a collapse of crystals within the microstructure [23]. Finally, developing a ready to use design tool is complicated and a full experimental verification of its limitations is outside the scope of this project. Instead, a proposed novel homogenization approach is developed, and initial experimental testing is provided for proof-of-concept.
Figure 1-2: Micro-Mechanical and Upscaling Approach for Cement Paste

Details on the methodologies and results are provided in the following chapters. They are presented as three published/submitted papers to delineate the findings of this study and aid future research. Chapter 2 focuses on an evaluation of current code elastic modulus equations. Chapter 3 studies the stiffness of UHPC on the microscale in a novel approach that allows both stiffness and compressive strength of phases to be determined without the confinement effects traditionally experienced in literature. Finally, Chapter 4 details an upscaling attempt using the micro-mechanical stiffness data so that its significance on the macroscale can be determined.

1.3. References


2.1. Introduction

In the design of concrete structures, deformation and serviceability calculations often require an accurate understanding of the concrete elastic stiffness [1-2]. For both prestressed and reinforced concrete components, early age crack control and calculation of prestress losses from surrounding material shrinkage and creep are only possible if the material stiffness is well approximated [3]. Additionally, to determine service-level deflections within structural components (beams, suspended slabs, etc.), an accurate understanding of material stiffness is required [4-5]. Material stiffness is also important for system level calculations, such as those required for slender columns in sway frames.

Because concrete is a mixture of several material components with time-dependent properties, many factors can affect the concrete elastic modulus, including aggregate type and properties, concrete density, presence of mineral admixtures, resulting porosity, curing and testing conditions, constituent material proportions, and characteristics of the interfacial transition zone (ITZ) between the aggregate and cement paste [4-6]. With each of the individual mixture components having a different elastic modulus, the resulting modulus of the final cured mixture is a result of the interaction between these components, affecting the stiffness homogeneity of the final material. For example, the porosity, size, shape, grading, and texture of coarse aggregate can affect the elastic modulus of the bulk concrete through the stiffness of the aggregate itself and the influence of these properties on micro-cracking in the ITZ [4]. The ITZ directly surrounding aggregates or fibers is usually weaker than the surrounding bulk cement-paste matrix and has direct effect on the concrete stiffness [4].
Chemical hydration reactions between the water and binder (traditionally portland cement) also form a variety of microstructural phases that add further complexity to concrete stiffness mechanisms. The interaction of calcium, sulfate, aluminate, and hydroxyl ions form crystalline microstructural phases such as calcium-silicate-hydrates, calcium hydroxide, ettringite, and monosulfoaluminate [4], which have differing stiffness properties. The type, amount, and distribution of these crystalline solids and voids in the cement paste matrix influence the resulting stiffness, strength, durability, and dimensional stability of the bulk concrete [4]. The addition of pozzolanic materials, such as fly ash, can produce a greater volume of strength-contributing crystalline phases in the cement paste and reduce the total volume of pores, which can increase the stiffness of the cement paste [4].

**Figure 2-1:** Empirical code equations for elastic modulus prediction commonly evaluated in literature

To simplify designs and prevent laborious experimental testing, design codes provide simplified empirical approaches to estimating concrete elastic modulus [4-5], based on information specified in design (i.e. concrete compressive strength and concrete unit weight) (see
Examples from design codes around the world include Eq. 2-8, where $E_c = \text{predicted elastic modulus (MPa)}$, $f'_c = \text{compressive strength (MPa)}$, $\alpha_E$ and $K_1 = \text{aggregate correction factors}$, and $\gamma_c = \text{concrete density (kg/m}^3\text{)}$.

**Table 2-1: References for code equations for elastic modulus evaluated in literature**

<table>
<thead>
<tr>
<th>Eq. No.</th>
<th>Reference</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-1a</td>
<td>ACI 318-14 [7]</td>
<td>Normal-strength</td>
</tr>
<tr>
<td>2-1b</td>
<td>ACI 318-99</td>
<td>Normal-strength</td>
</tr>
<tr>
<td>2-2</td>
<td>ACI 363R-10 [8]</td>
<td>$f'_c &lt; 83$ MPa</td>
</tr>
<tr>
<td>2-3a</td>
<td>CEB-FIP MC 90 [9]</td>
<td>$f'_c &lt; 80$ MPa</td>
</tr>
<tr>
<td>2-3b</td>
<td>CEB-FIP MC 90 [9]</td>
<td>High-strength</td>
</tr>
<tr>
<td>2-3c</td>
<td>fib MC 2010 [10]</td>
<td>$f'_c &lt; 80$ MPa</td>
</tr>
<tr>
<td>2-4</td>
<td>Eurocode 2 [11]</td>
<td>$12$ MPa $&lt; f'_c &lt; 90$ MPa</td>
</tr>
<tr>
<td>2-5</td>
<td>NS 3473-92 [12]</td>
<td>$25$ MPa $&lt; f'_c &lt; 85$ MPa</td>
</tr>
<tr>
<td>2-6</td>
<td>KCI 2007 [13]</td>
<td>Not stated</td>
</tr>
<tr>
<td>2-7</td>
<td>AASHTO LRFD [14]</td>
<td>$f'_c &lt; 105$ MPa</td>
</tr>
<tr>
<td>2-8</td>
<td>NBR 6118 [15]</td>
<td>$f'_c &lt; 50$ MPa</td>
</tr>
</tbody>
</table>

\[
E_c = 4700\sqrt{f'_c} \quad \text{Eq. 2-1a}
\]
\[
E_c = 4730\sqrt{f'_c} \quad \text{Eq. 2-1b}
\]
\[
E_c = 3320\sqrt{f'_c} + 6900 \quad \text{Eq. 2-2}
\]
\[
E_c = 21,500\left(\frac{f'_c}{10}\right)^{1/3} \quad \text{Eq. 2-3a}
\]
\[
E_c = 10,000(f'_c + 8)^{1/3} \quad \text{Eq. 2-3b}
\]
\[
E_c = \alpha_E 21,000\left(\frac{f'_c + 8}{10}\right)^{1/3} \quad \text{Eq. 2-3c}
\]
\[
E_c = 22,000\left(\frac{f'_c}{10}\right)^{1/3} \quad \text{Eq. 2-4}
\]
\[
E_c = 9500(f'_c)^{0.3} \quad \text{Eq. 2-5}
\]
\[ E_c = 8500\sqrt{f'_{c}} + 8 \quad \text{Eq. 2-6} \]
\[ E_c = K_1\gamma^{1.5}0.043\sqrt{f'_{c}} \quad \text{Eq. 2-7} \]
\[ E_c = \alpha_e 5600\sqrt{f'_{c}} \quad \text{Eq. 2-8} \]

However, studies have shown that the modulus of elasticity is less affected by concrete strength and more affected by localized damage at the ITZ and aggregate modulus [4-5, 16-20]. Porosity and testing parameters strongly affect both the compressive strength and elastic modulus, though the influence on the two can vary as strength and elastic modulus are not directly reliant on each other [4, 18]. For example, concrete modulus will increase but compressive strength will decrease when testing in dry conditions versus wet conditions [3-4].

Pauw [18] developed the empirical relationship provided in ACI 318 around sixty years ago when an increase in use of lightweight aggregate highlighted a need to reevaluate the relationships between strength, elastic modulus, and concrete weight [18]. Through the investigation, Pauw noted then that there was a poor statistical relationship between compressive strength and the elastic modulus and recommended a future reassessment of the role of compressive strength in estimating the elastic modulus [18]. Similar ideas have been stated throughout literature since then with many authors citing the need for correction factors for aggregate variations, mineral admixtures, and fiber content for certain specialty concretes like fiber-reinforced and eco-concretes [16-17, 20-23]. Since then, types of materials in concrete have become more varied, creating greater inaccuracies in current estimation calculations. Specialty concretes widely used today (i.e. self-consolidating concrete (SCC), high strength concrete (HSC), high performance concrete (HPC), fiber reinforced concrete (FRC), ultra-high performance concrete (UHPC), eco-concretes like recycled aggregate concretes, etc.) have replaced portland cement with mineral admixtures at various percentages, added materials such
as chemical admixtures and fibers, and adjusted coarse and fine aggregate ratios from the normal concrete mixtures on which the empirical relationship by Pauw [18] is based.

Most empirical equations published in design codes around the world were developed for normal strength concretes and are likely not applicable to all specialty concretes [4-5, 22]. Code equations for estimating the modulus of elasticity need further development before being applied to specialty concretes. For example, the ACI estimation assumes that the same absolute specific gravity applies to all mineral aggregates and that voids are the primary cause for weight differences [18]. This may not remain true for all specialty concretes, particularly since the elastic modulus is determined to be less sensitive to changes in compressive strength than small changes in weight [18].

This paper provides a review of specialty concretes and the applicability (or lack thereof) of existing elastic modulus equations through a detailed summary of the existing literature. By consolidating the existing literature related to concrete modulus approaches in this paper, a clear context and background is provided to both researchers and practitioners. Additionally, this paper offers evidence to support the observation in literature [4-5, 18, 20] that compressive strength alone is not a very good basis for elastic modulus estimations of all specialty concretes. The following sections detail individual specialty concrete types and summarize individual elastic modulus approaches. Next, a discussion related to concrete modulus estimations from the literature summary is provided, and conclusions are presented.

2.2. Self-Consolidating Concrete

Current American code standards require a minimum clear spacing between reinforcement in members to be 1 inch or a function of the nominal aggregate or bar size [7]. These minimum limits ensure that concrete can flow between bars and into forms without risking
honeycombing [7]. In heavily reinforced members, however, homogeneity during placement and compaction is still of concern [24] as large drops or blockage at reinforcement can cause segregation to occur [25]. Recognizing the concern and seeking to ease construction of concrete structures, researchers led by Prof. Okamura at the University of Tokyo developed a new category of specialty concrete called self-compacting concrete in the late 1980s [24, 26-28]. It has since become additionally known as self-consolidating concrete, particularly in North America [29]. SCC flows under its own weight around reinforcement and fills sharp corners of formwork without requiring any vibration [25]. The shear stresses and friction between aggregates in the cement paste (developed as the concrete flows through reinforcement) are decreased due to the viscosity of the paste, which prevents segregation from occurring [24]. The high flowability is created through a mixture of high volumes of paste, superplasticizers, low water to powder ratios, and the addition of mineral admixtures, which allows the concrete to flow without risk of segregation [4, 25]. Self-consolidating concrete is often used to limit construction time, noise, and labor and to create a better face finish in areas that may be hard to vibrate [25].

Self-consolidating concrete, as listed above, often contains more chemical and mineral admixtures as well as different proportions of constituent materials than normal strength concrete [4]. This calls the applicability of current design, testing practices and empirical equations into question. The elastic modulus can be affected by water-cement ratios (w/c), powder and water contents, admixture doses, particle size distributions, and aggregate volumes, all of which differ for SCC from normal strength concrete [25]. Several studies in recent literature have evaluated elastic predictions made by empirical code equations for SCCs made with various portland cement replacements, aggregates, and applications [2, 25, 30-31]. An overview of their work and
conclusions is provided here, followed by a larger evaluation using 100 SCC data points from literature.

Kuder et al. investigated high volume replacements of portland cement with fly ash and/or slag for both fresh and hardened concrete properties [31]. The modulus of elasticity values were compared with the ACI 318 (Eq. 2-1a) and CEB-FIP Model Code (Eq. 2-3b) estimations of the elastic modulus [31]. Compared to the ACI 318 empirical equation, the experimentally measured elastic modulus values were on average around twenty percent lower than the predictions provided by code equations [31]. As ACI 318 states in the commentary that actual values typically range between 80 and 120 percent from those predicted in their expression [7], the code equation was viewed as having a similar tendency to the experimental results [31]. Having a similar result as ACI 318, the European standard equation (Eq. 2-3b) was found to be adequate with a less than twenty percent error [31].

Seeking to evaluate a wider pool of code equations, Long et al. compared their measured elastic modulus results to AASHTO (Eq. 2-7), CEB-FIP MC (Eq. 2-3b), ACI 363 (Eq. 2-2), and ACI 318 (Eq. 2-1b) [2]. Sixteen mixtures were created using various binder contents and types, admixtures, sand to total aggregate ratios, and water-cement ratios [2]. Expected differences were seen in the specimens: high-range water reducer usage led to a low early elastic modulus, lower w/c ratios had a higher modulus, and the binder content did not have a significant impact [2]. The CEB-FIP Model Code overestimated the elastic modulus, while ACI 363 underestimated it, though almost all values were within a 20% error [2]. Of some interest, Long et al. also provided a list of suggested model coefficient corrections to better match their experimental results [2]. This is provided in Table 2-2. As seen, the CEB-FIP Model Code and
ACI 363 required a large adjustment to their coefficients to be accurate for their SCC mixture while the AASHTO curve was inherently fairly accurate [2].

**Table 2-2:** Suggested coefficient adjustments by Long *et al.* [2]

<table>
<thead>
<tr>
<th>Reference</th>
<th>Original Model Equation</th>
<th>Suggested Model Coefficients</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACI 318</td>
<td>$E_c = 4730\sqrt{f'_{c}}$</td>
<td>$E_c = 5180\sqrt{f'_{c}}$</td>
</tr>
<tr>
<td>ACI 363</td>
<td>$E_c = 3320\sqrt{f'_{c}} + 6900$</td>
<td>$E_c = 5080\sqrt{f'_{c}} + 640$</td>
</tr>
<tr>
<td>CEB-FIP Model Code</td>
<td>$E_c = 10,000(f'_{c} + 8)^{0.33}$</td>
<td>$E_c = 1,600(f'_{c} + 8)^{0.77}$</td>
</tr>
<tr>
<td>AASHTO</td>
<td>$E_c = K_1\gamma_{c}^{1.5} 0.043\sqrt{f'_{c}}$</td>
<td>$E_c = K_1\gamma_{c}^{1.5} 0.045\sqrt{f'_{c}}$</td>
</tr>
</tbody>
</table>

Seeking to further the applications of self-consolidating concrete, Floyd *et al.* [25] and Kim *et al.* [30] investigated the impact of lightweight coarse aggregate on the elastic modulus of SCC concrete. Between the two studies, expanded clay, shale, and manufactured lightweight aggregates made from rhyolite fine powder and wastes such as screening sludges were evaluated [25, 30]. Both studies compared the lightweight SCC results to the estimation provided by ACI 318 and determined it to be within a 20% error of the measured values [25, 30]. Floyd *et al.* also determined the expanded clay and shale to have a similar elastic modulus, indicating that large adjustments do not need to be made for aggregate type when considering one of these two [25].

Experimental and predicted elastic modulus values based on corresponding compressive strength tests are graphed from literature in Figure 2-2 for various empirical code equations. A perfect correlation between the measured and predicted elastic modulus values is indicated by the dark 45-degree line. Values above this line show a higher predicted elastic modulus than measured, while values below the line show an underestimation of elastic modulus by code equations. Predictions based on compressive strengths corresponding to measured elastic modulus values in literature are graphed along the y-axis, while the measured elastic modulus
values are along the x-axis. As seen, the majority of the data points fall within a 20% error, with some over- and under-estimations of the measured elastic modulus. The data points above the 20% error line (indicating an over-estimation by code equations) are largely from Omar et al. [32], which examined lightweight aggregate usage in self-consolidating concrete.

**Figure 2-2:** Experimentally measured elastic modulus compared to code predicted values based on measured compressive strengths in literature [2, 25, 30-35]

ACI 363 and CEB-FIP MC are primarily responsible for overestimations when lightweight aggregate is used in SCC, while ACI 318 and AASHTO provide smaller overestimations, in agreement with conclusions drawn by [25, 30]. From these data sets, it is concluded that empirical code equations are largely adequate for SCC. However, when using lightweight aggregate in SCC mixes, further evaluation may be necessary to prevent overestimations. Lightweight SCCs developed by [25, 30] found the ACI 318 prediction to be adequate for their mixes, while the data set from Omar et al. indicates overestimations for elastic modulus around or above
20% for all code equations evaluated [32]. As the density of the lightweight SCC has been accounted for already in the code equations that account for it, this may indicate the need for an aggregate factor based on the coarse aggregate material.

2.3. High-Strength Concrete

Similar to SCC mixes, high strength concretes use chemical and mineral admixtures to achieve the desired properties. High-strength concrete is defined as a concrete with a compressive strength of over 55 MPa at an age of 28 days [4]. As using portland cement alone can achieve around a compressive strength of 50 MPa, the chemical and mineral admixtures increase the compressive strength while maintaining the workability [4]. Since high strength concrete may contain cement paste that is as strong as or stronger than the aggregate, the elastic modulus of this concrete is more heavily influenced by aggregate type than a normal strength concrete mixture [22, 36]. Due to this variation from normal strength concrete, ACI 363 developed an equation (Eq. 2-2) to estimate the elastic modulus of high strength concrete [8]. Data from literature is used in Figure 2-3 to examine the adequacy of code equations and the equation proposed by Noguchi et al. (Eq. 2-9), which contains mineral admixture and aggregate correction factors [22].

Studies have analyzed the applicability of this code equation and others for variations in coarse aggregate types, such as limestone, granite, quartzite, sandstone, and crushed river gravel [6, 36]. In one study, Baalbaki et al. determined that empirical code equations are not applicable for all types of coarse aggregate for high-strength mixes [36]. ACI 363 (Eq. 2-2) predictions were generally within a 10 % error for the two types of limestone and granite and one type of quartzite evaluated, but provided significantly different values for the sandstone [16, 36] aggregates (see again Figure 2-3).
Figure 2-3 provides an analysis of measured and predicted elastic modulus values, with the dark 45-degree line representing a perfect correlation. As seen in the boxed data points, the sandstone aggregate elastic modulus values experimentally measured differ greatly from the predicted [16, 36]. CEB-FIP MC 90 (Eq. 2-3b) overestimated the elastic modulus by over 20% for sandstone and granite aggregate, while the Norwegian Standard 3473 (Eq. 2-5) only overestimated the modulus for the sandstone concretes [36]. For granite, quartzite, and limestone, the Norwegian Standard was found to be within a 15% error [36].

Figure 2-3: Comparison of experimentally measured and empirically predicted elastic modulus values for high-strength concrete with varying types of coarse aggregate from literature [16, 36-41]

In comparison with Baalbaki et al.’s results [16, 36], Mokhtarzadeh et al. found ACI 363 estimations to be within 20% error for all aggregate types and ACI 318 to overestimate the elastic modulus (within 40% error for limestone aggregate; within 25% error for round river gravel) [6]. The NS 3473 prediction slightly underestimated (within 25% error) the measured
values, and CEB-FIP Model Code had a good agreement (within 20% error) for low- and high-absorption limestone and crushed river gravel [6]. Mokhtarzadeh et al. specifically notes that sources for coarse aggregate vary greatly, making it difficult to effectively develop and use empirical equations with aggregate factors [6]. Noguchi et al. [22] strongly agrees with the assessment that even the ACI 363 equation may not be an adequate estimation for the high strength or other specialty concretes. In a statistical analysis of over 3000 tests, Noguchi et al. determined that lithological characteristics of aggregates and the presence of mineral admixtures play a decisive role in estimating the elastic modulus [22]. As a result, their proposed regression equation (Eq. 2-9) includes experimentally-obtained correction factors ($k_1$ and $k_2$) based on the type of aggregate and admixtures included [22]. For example, a concrete made from crushed limestone aggregate and having a replacement of portland cement with silica fume between 10 and 20% would have correction factors of $k_1=1.20$ and $k_2=0.913$ [22]. The other factors included are compressive strength in MPa ($f'_{c}$) and concrete density in kg/m$^3$ ($\gamma$).

$$E_c = k_1 k_2 1.486 \times 10^{-3} f'_{c}^{1/3} \gamma^2$$

Eq. 2-9

As seen in Figure 2-3, this equation seems to generally be within a 20% error, like the code equations evaluated. A notable exception is for the large underestimation of andesite coarse aggregate mixes made by Beunshausen et al. [37]. The use of andesite by Beunshausen et al. resulted in all code equations underestimating the measured elastic modulus by more than 20% [37].

From these studies, it can be concluded that the most applicable code equations are ACI 363 and NS 3473 for high strength concrete. CEB-FIP Model Code and ACI 318 generally show a similar tendency between predicted and measured values for elastic modulus, but often have a higher degree of error for aggregates like sandstone and limestone [6, 22]. However, errors
around 20% can be significant for high and ultra-high strength concretes. For a 60-MPa concrete having a predicted elastic modulus of approximately 32.6 GPa, the measured elastic modulus may be anywhere from 26.1 GPa to 39.1 GPa. This is a large range that may cause designers to notably over or underestimate the elastic modulus in the initial design phase.

2.4. High-Performance Concrete

While high-strength concrete exceeds a specific strength threshold (55 MPa), high-performance concrete additionally provides a high durability and workability. To obtain the high workability, high strength, and high durability found in high-performance concretes, supplementary cementitious materials and chemical admixtures are added to the mixture [4, 42]. Commonly added chemical admixtures include plasticizing, set-controlling, and air-entraining [4, 43]. Of particular interest, water-reducers and superplasticizers (also referred to as high-range water-reducing admixtures or HRWRAs) are commonly used to improve the dispersion of cement particles in water. The increased distribution during hydration can increase consistency (without risking excessive bleeding) or reduce the water requirement without affecting workability entraining [4, 44]. The use of plasticizing admixtures can also increase the compressive strength of the concrete, though adding too much to the concrete mix can delay the time of set entraining [4, 43]. Diamond additionally found water-reducing admixtures to promote a more homogeneous cement paste matrix [45].

Mineral admixtures added to specialty concretes are fine materials with cementitious or pozzolanic properties that can be used as a partial replacement of portland cement [44]. Common examples include fly ash, silica fume, metakaolin, rice husk ash and blast-furnace slag [19, 43-44]. Addition of pozzolans can influence the thickness and strength of the ITZ [46]. Some are highly reactive and increase the density of crystalline phases in the ITZ [44], reacting with the
calcium hydroxide to form strong crystalline phases similar to calcium-silicate-hydrates [46]. The fine materials themselves can additionally fill pores in the ITZ [46]. As ITZ properties are known to influence the overall stiffness, a denser and stronger ITZ can produce a higher bulk elastic modulus [19]. Silica fume has been found to reduce the thickness of the ITZ [46-47] and increase the overall strength, particularly at early ages [3]. Small replacements of portland cement with fly ash has increased the elastic modulus when compared to normal-strength concrete [3, 46]. As a result, use of admixtures can help produce the desired early age strength, permeability, toughness, heat of hydration, durability in severe environments, long-term mechanical properties and more [4]. Applications for HPC include long-span bridges, bridge decks, parking structures, and off-shore oil drilling platforms [4].

Evaluations of the elastic modulus of high-performance concretes often compare different types and volume fractions of pozzolan replacements of portland cement. Nassif et al. studied the impact of different amounts of silica fume, fly ash, and granulated blast furnace slag on increases in the elastic modulus of high-performance concretes [3]. The study showed that a 10% replacement with fly ash increased the elastic modulus by 6-17%, with higher replacements having little impact on the modulus [3]. Silica fume increased the early elastic modulus but had little impact on the modulus past 28 days [3]. When comparing the experimental mixtures with current code equations for conventional concrete, Nassif et al. determined that ACI 363 gave the most accurate modulus prediction for mixtures containing fly ash and silica fume [3]. For mixtures containing a low amount of fly ash, ACI 318 provided the best estimate as the mixture design is similar to conventional concrete [3]. High replacements with fly ash resulted in ACI 363 and ACI 318 predictions providing a higher error, though still less than 20% [3]. This is likely due to the variations in microstructure and ITZ from the normal-strength concrete mixes.
that were used to originally develop the empirical equations. The study by Nassif et al. concluded that the elastic modulus increases at a slower rate over time than the compressive strength when most mineral admixtures are used [3].

Issa et al. took a similar approach and evaluated mixtures with slag and fly ash as replacements for portland cement high-performance concretes [42]. The elastic moduli of cylindrical specimens were recorded and compared to the predicted values provided by ACI 318, ACI 363, and CEB-FIP/Eurocode [42]. The results from Issa et al. show that ACI 318 is the best estimate for the high-performance concrete mixtures evaluated, though the other codes had 15% error on average. Differences in replacement levels, coarse aggregate type and content, and curing conditions likely account for variation in results between studies.

A larger dataset from literature, including the compressive strength and elastic modulus values from the summarized studies [3, 42], is plotted in Figure 2-4. Predictions made by ACI 363 largely fall within a 20% error, with a tendency to underestimate the elastic modulus. Conversely, predictions by ACI 318 and Eurocode for the elastic modulus tend to overestimate. All ACI 318 estimations were within 25% of the experimentally measured values, while Eurocode values fell within a 40% error. From the conclusions drawn in literature and the data plotted in Figure 2-4, the reviewed studies show high-performance concrete to generally be adequately predicted by American code equations. The Eurocode tends to overestimate the elastic modulus to inadequate degree and should be used with hesitation.
2.5. Fiber-Reinforced Concrete

Fiber-reinforced concrete (FRC) is a specialty concrete developed to have an improved toughness against dynamic loads and improved tensile strength [50], particularly in mixture designs with higher compressive strengths that tend to be more brittle [51]. The presence of the fibers, which can be made of steel, glass, synthetic, or natural materials [52], helps distribute tensile and shear stresses by bridging across cracks [50]. As a result, smaller cracks form and are spread more evenly throughout the cement paste [50]. Fiber-reinforced concrete is used in a variety of applications, including slabs and floors, repair mortars, dam construction, composite decks, rehabilitation and retrofitting, overlays, shell structures, etc. [50, 52].

The addition of fibers into the concrete mixture adds additional variables to be considered in mixture proportioning (i.e. fiber strength properties, aspect ratios, and volume fractions) [53]. Proving that the compressive strength is not always influenced by the same factors as the elastic
modulus, Suksawang et al. determined that the fibers have little impact on compressive strength but a large impact on the elastic modulus [23], particularly if the fibers themselves have a large elastic modulus [54]. This is due to the fibers’ ability to provide strain-softening and strain-hardening properties and mixtures typically having a lower coarse-to-fine aggregate ratio than conventional concrete [23]. Studies [50, 53, 55-56] have shown that when the coarse to fine aggregate volume ratio (C/S) is greater than one, around a ten percent change from the elastic modulus of conventional concrete is expected [57]. However, if the C/S ratio is less than one, the elastic modulus is found to decrease with increasing fiber aspect ratios and volume fractions [57].

To better understand the impact of fiber volume fractions and C/S ratios on the elastic modulus and to evaluate current code equations, Suksawang et al. investigated five different types of fibers: steel, polypropylene, macro-polyolefin, polyvinyl alcohol (PVA) and basalt [23]. For all fibers, the elastic modulus was found to decrease an average of 20% when the C/S ratio was below one [23]. The results from their experimental testing were combined with over 400 data points from literature to compare with empirical code equations. The equations were evaluated based on a coefficient of variation from the data, which ranged from 24 to 31% [23]. The Norwegian Standard equation was determined to be the most accurate, while ACI 318 produced the highest coefficient of variation [23]. ACI 363 provided a similar estimation to the Norwegian Standard [23]. However, ACI 318 provided a similar curve shape for the elastic modulus when the C/S ratio was greater than one [23]. As a result, Suksawang et al. developed their own equation in the same form as the ACI 318 equation. The equation additionally accounts for the fiber volume fraction, \( V_f (\%) \), when C/S < 1 through a fiber volume correction factor, \( \lambda_{vf} \).
as provided in Eq. 2-10, 2-11, and 2-12 [23]. $E_c$ is the elastic modulus (MPa) and $f'_c$ is the compressive strength (MPa).

$$E_c = 4,700 \lambda_{V_f} \sqrt{f'_c} \quad \text{Eq. 2-10}$$

where: $\lambda_{V_f} = 1$ if $C/S > 1.0$ \quad \text{Eq. 2-11}$

and $\lambda_{V_f} = \frac{1+0.7V_f}{2}$ if $C/S < 1.0$ \quad \text{Eq. 2-12}$

Their equation provided a coefficient of variation of 15%, noticeably more accurate than the current empirical equations designed for conventional concrete [23].

**Table 2-3:** Coefficient of variations for code equations compared with FRC experimental results [21]

<table>
<thead>
<tr>
<th>Fiber Type</th>
<th>ACI 318 (Eq. 2-1a)</th>
<th>ACI 363 (Eq. 2-2)</th>
<th>CEB-FIP MC (Eq. 2-3a)</th>
<th>Suksawang et al. (Eq. 2-10)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel</td>
<td>10.7%</td>
<td>15.7%</td>
<td>18.0%</td>
<td>10.9%</td>
</tr>
<tr>
<td>Glass</td>
<td>12.4%</td>
<td>17.1%</td>
<td>15.7%</td>
<td>12.6%</td>
</tr>
<tr>
<td>Nylon</td>
<td>9.0%</td>
<td>13.8%</td>
<td>16.0%</td>
<td>10.0%</td>
</tr>
</tbody>
</table>

Using a nondestructive resonance frequency test, Hedjazi et al. performed a similar experiment with comparable conclusions [21]. Steel, glass, and nylon fibers added in various volume fractions between 0.1 and 1.5% were tested to determine the coefficients of variations for code equations for individual fiber types (Table 2-3) [21]. The equation proposed by Hedjazi et al. (Eq. 2-13) closely matches that of Suksawang et al., though the volume fraction factor (given as $\beta$ in Eq. 2-14 instead of $\lambda_{V_f}$ as in Eq. 2-10) additionally considers the type of fiber used in the concrete through two unit-less constants, $\alpha$ and $\gamma$, instead of only the volume fraction in percent, $V_f$ [21]. $E_c$ is the elastic modulus (MPa) and $f'_c$ is the compressive strength (MPa). The proposed equation is applicable for nylon, steel, and glass fibers, with values provided in their work for the
constants, and for volume fractions of fibers up to 1.5% (the maximum considered in their study) [21].

\[ E_c = 4700\beta\sqrt{f'_c} \quad \text{Eq. 2-13} \]

\[ \beta = \frac{0.9 + a^{\gamma - Vf\%}}{2} \quad \text{Eq. 2-14} \]

Hedjazi et al. concluded that each type of fiber affected the elastic modulus differently and agreed with other researchers that increase in fiber volume fraction led to a decrease in elastic modulus [21]. Like Suksawang et al., this study showed that current empirical code equations are not universally adequate for fiber-reinforced concrete.

The inadequacy of current code equations for predicting the elastic modulus when the C/S ratio is less than one is confirmed in Figure 2-5. Literature data from FRCs made with a C/S greater than one is shown in Figure 2-5a and a C/S less than one in Figure 2-5b. As seen, while values generally fall within a 20% error in the first graph, there is a notable shift toward overestimation in the second. Providing a fiber volume correction factor, as in Eq. 2-10, allowed predictions to fall within a 25% error range. Eq. 2-13 also contains a fiber volume correction factor, but it provides a much higher error than Eq. 2-10 when applied to data from literature. Hedjazi et al. developed the equation based on elastic modulus and compressive strength data taken at 44 days [21], rather than the usual 28 days, which may account for some of the error. Overall, FRC can generally be adequately predicted when C/S is greater than one. In mixes with low coarse aggregate fractions, inclusion of a fiber volume correction factor has been shown to prevent the large overestimations given by code equations.
Figure 2-5: A comparison of experimentally measured and empirically predicted elastic modulus values for fiber-reinforced concrete from literature [23, 50-51, 57-59]
2.6. Ultra-High Performance Concrete

Similar to fiber-reinforced concrete, ultra-high performance concrete (UHPC, also referred to as ultra-high performance fiber-reinforced concrete (UHPRFC) or reactive powder concrete (RPC) [60] usually contains fibers to help improve the properties of the concrete [61]. UHPC is characterized by excellent mechanical and durability properties, including a compressive strength greater than 150 MPa, a high tensile strength (around 30 MPa), and an improved ductility and fracture toughness [7, 60, 62]. Mixture proportions for UHPC often include a low w/c (less than 0.25) [62], fine quartz sand and/or quartz powder replacements, superplasticizers in high dosages, fibers, and supplementary cementitious materials in the binder [60]. Usually, 2-3 times more portland cement or binder is used in UHPC than in conventional normal strength concretes [54]. This high volume of fine particles results in a dense ITZ (or indifferentiable from cement paste [63]) [60]. However, the high volume of fines and binder tends to be very expensive, leading to the replacement of cement or fine sand with less expensive alternatives, such as natural-gradation sand or fly ash [63]. These replacements cause changes in the microstructure that affect mechanical properties and cause the concrete to differ even further from conventional concretes [63]. Applications for UHPC include precast-prestressed bridge girders, bridge decks [63], repairs and splices, blast-resistant structural members, and members in highly corrosive environments [54]. Overall, it is generally agreed that existing code equations for conventional concrete are not adequate for use with UHPC [61-65].

Focusing on the elastic modulus of UHPC, the water to binder ratio (w/b) and coarse aggregate content have been found to have the largest impact on the elastic modulus, the former decreasing the predicted elastic modulus and the latter increasing it, as seen in the regression
equation (Eq. 2-15) developed by Ouyang et al. [65]. It should be noted, however, that most UHPC mixtures do not contain any coarse aggregates.

\[ E_c = 45.57 - 126.32 \frac{W}{b} + 0.08M_c + 0.000M_{sf} - 0.0006M_{slag} + 0.013M_{sand} + 0.014M_{CA} + 0.008V_{fiber} \]  

Eq. 2-15

In Equation 15, the masses of cement, silica fume, slag, sand, and coarse aggregate are represented as \( M_c, M_{sf}, M_{slag}, M_{sand}, \) and \( M_{CA} \), respectively (kg/m\(^3\)), and the volume of steel fibers is represented as \( V_{fiber} \) (%) [65]. \( E_c \) is the predicted elastic modulus in GPa [65]. As this equation was developed based on the experimental data collected by Ouyang et al., it offers potential insight for predictions of UHPC elastic moduli, delineating the impact that each change in mixture proportioning has on the measured value.

Figure 2-6: Literature elastic modulus prediction equations developed for UHPC
Seeking to better understand the impact on elastic modulus, Dadmand et al. examined different shapes of fibers, such as micro-steel, crimped, hooked-end, and rounded crimped fibers (Figure 2-6), at two separate fiber contents to evaluate the adequacy of the code equations [61]. Testing of cylinders showed the presence of steel fibers to slightly increase the modulus of elasticity in the specimens [61]. The order of highest to lowest elastic modulus increase for 1 and 2% fiber contents was micro-steel (additional 3.4 and 13.8%, respectively), rounded crimped (additional 1.8 and 11.1%, respectively), crimped (additional 2.1 and 8.7%, respectively), and hooked-end fibers (additional 0.6 and 9.3%, respectively) [61]. Results from this experiment were also compared with equations and additional data from literature, as provided in Table 2-4 and Figure 2-6. Dadmand et al.’s study showed that the equation proposed by Haber et al. (Eq. 2-20) provided the smallest error for all types of steel fibers evaluated [61]. Kollmorgen et al.’s equation (Eq. 2-19) provided a significant overestimation in all specimens, while the other equations (Eq. 2-16, 2-17, and 2-18 where $E_c$ is the predicted elastic modulus in MPa and $f'_{c}$ is the measured compressive strength in MPa) provided much smaller overestimations [67].

**Table 2-4: References for equations from literature in Dadmand et al. [61]**

<table>
<thead>
<tr>
<th>Eq. No.</th>
<th>Reference</th>
<th>Note (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-16</td>
<td>Alsalman et al. 2017 [63]</td>
<td>$31 \leq f'_{c} \leq 235$</td>
</tr>
<tr>
<td>2-17</td>
<td>Graybeal and Stone 2012 [66]</td>
<td>$97 \leq f'_{c} \leq 179$</td>
</tr>
<tr>
<td>2-18</td>
<td>Graybeal et al. 2007 [64]</td>
<td>$126 \leq f'_{c} \leq 193$</td>
</tr>
<tr>
<td>2-19</td>
<td>Kollmorgen et al. 2004 [68]</td>
<td>$34 \leq f'_{c} \leq 207$</td>
</tr>
<tr>
<td>2-20</td>
<td>Haber et al. 2018 [67]</td>
<td>$65 \leq f'_{c} \leq 153$</td>
</tr>
</tbody>
</table>

$$E_c = 8010(f'_{c})^{0.36} \quad \text{Eq. 2-16}$$

$$E_c = 4069(f'_{c})^{0.5} \quad \text{Eq. 2-17}$$

$$E_c = 3840(f'_{c})^{0.5} \quad \text{Eq. 2-18}$$
\[ E_c = 11,800(f'_c)^{1/3.14} \quad \text{Eq. 2-19} \]
\[ E_c = 3755(f'_c)^{0.5} \quad \text{Eq. 2-20} \]

Encompassing a wider range of possible mixtures, Alsalman et al. evaluated 223 modulus values from across literature for UHPC and developed a relationship between the elastic modulus and compressive strength (Eq. 2-16) [63]. It was determined that Eq. 2-16 tends to overestimate the modulus of elasticity when using local materials instead of a UHPC premix, which can lead to larger deflections or prestress losses than expected in structures if used in design [63]. Local aggregate tends to have more soft particles and different minerals on the surface, which can create weak links in concrete and a less dense cement matrix as compared to a UHPC premix [63]. This highlights the need for UHPC design standards and a better understanding of its mechanical properties.

Seeking to further understand premixes, Allard et al. studied Cor-Tuf, a UHPC mixture developed by the United States Army Corps of Engineers (USACE) Engineer Research and Development Center (ERDC) [62]. Cor-Tuf was evaluated for the compressive strength and elastic modulus resulting from isothermal curing and submerged conditions, specifically to understand strength gain for full-scale placements [62]. ACI 318 (Eq. 2-1a) was determined to overestimate the modulus of elasticity for most compressive strengths [62]. Adjusting the constant in the ACI 318 equation to be 4139 instead of 4700 provided a significantly better estimation for their UHPC mixture [62].

As is shown through the summarized literature, the current code equations for conventional concrete are not universally suitable for use with UHPC. Data from literature in Figure 2-7 confirms this conclusion. ACI 318 and Eurocode 2 tend to overestimate the elastic modulus more than other code equations, but large portions of predictions made by all evaluated
code equations exceed a 20% error. Effort must be made to develop a widely applicable empirical equation (or equations if necessary) for estimating the elastic modulus. Significant progress has been made in this field already, as evidenced by the many experimental equations proposed in literature [63-64, 66-68], however, the high variability between UHPC mixtures make it difficult for one of the proposed equations to be applied to various UHPC mixes in practice.

![Graph comparing predicted and measured elastic modulus values](image)

**Figure 2-7:** Comparison of experimentally measured and empirically predicted elastic modulus values for ultra-high performance concrete from literature [61, 63-64, 69-71]

Increases in compressive strength in UHPC tend to increase the elastic modulus at a different rate than conventional concrete [63]. Alsalman *et al.* made progress towards providing an adequate estimation, but while the proposed equation (Eq. 2-16) captured 56% of the data points evaluated in their study within a 10% error, for their own experimental work it only included 31% of the data collected due to using local fine aggregates [63]. The complexity of the UHPC microstructure and composition is often not encompassed well in empirical equations in
current development [65]. To ensure safe and efficient structures, further development is required for UHPC elastic modulus estimations.

2.7. Eco-concrete

The construction industry is known to both produce a lot of waste and consume a high amount of non-renewable natural resources, resulting in a negative impact on the environment [72]. To lower environmental strain from the production of concrete, alternative concrete materials have been considered [54, 72-74]. Numerous waste and recycled alternatives have been examined throughout literature, from using sugar cane bagasse ash as a low carbon dioxide pozzolan [73] to aggregate replacement with waste tire rubber [74]. Additionally, alternative cements like calcium aluminum sulfate binders, alkali-activated binders, and calcium aluminate cements have gained interest as additives. In-depth experimental work is required for replacement of any materials with recycled or lower impact alternatives. Recognizing the need for extensive experimental testing to fully understand alternative materials, studies have examined the mechanical properties of a variety of eco-concretes and some have tested the applicability of current code equations in predicting the elastic modulus of those concretes [54, 72-74].

The extraction of aggregates can have a large environmental impact, from land use concerns and consumption of finite materials to carbon emissions from heavy equipment usage [74]. Alternative replacements (i.e. recycled aggregates and waste porcelain aggregates) were studied by Jacintho et al. for their suitability in concrete and their effects on mechanical properties [72]. Results showed that waste porcelain aggregates increased the elastic modulus, likely due to the adhesion of the non-polished surface with the paste [72]. A 30% replacement of basaltic aggregate with recycled aggregates led to a 5% increase in elastic modulus, while a 50%
replacement showed a slight decrease [72]. The measured elastic modulus values were compared with various codes [72]. As two of the equations require an aggregate factor \( (\alpha_E) \) and none is currently available for recycled aggregates and waste porcelain aggregates, the authors used the value for basaltic aggregates (1.20) [72]. Jacintho et al. concluded that the fib MC 2010 provided the best prediction of the elastic modulus, but both the fib MC 2010 and NBR 6118 overestimated several of the mixtures’ moduli by up to 14% [72]. On the other hand, the ACI 318 equation significantly underestimated the measured modulus by up to 78%, proving it to be an inadequate prediction [72] though likely not a safety or serviceability problem.

Liu et al. determined a similar inadequacy of current code equations in concrete containing alternative cement [54]. Currently, there is little research about the mechanical properties of alternative cements, especially the elastic modulus. In their study, an ultra-high performance geopolymer concrete (UHPGC) was investigated as a low-carbon and clinker-free alternative to portland cement [54]. The mixture contained silica fume and steel fibers, as is common with UHPC, and examined the influences of these material contents on mechanical and fracture properties of the concrete. From the data, a 22% increase in elastic modulus was found in concretes containing a 3% addition of steel fibers [54]. However, at a given compressive strength, the UHPGC had a lower elastic modulus than the portland cement UHPC specimens [54]. As a result, the examined empirical equations (Eq. 2-2, 2-5, 2-16, 2-17, 2-18) all overestimated the elastic modulus of UHPGC [54]. A new equation was proposed for the data of UHPGC (Eq. 2-21) [54], where \( E_c \) is the predicted elastic modulus (MPa) and \( f'c \) is the compressive strength (MPa).

\[
E_c = 2600\sqrt{f'c}
\]

Eq. 2-21
Variations in materials, sources, and processing have significant impacts on the compressive strength and elastic modulus of eco-concretes. Replacements of aggregates with recycled or waste materials may result in higher or lower density concretes and a stronger or weaker bond to the surrounding cement paste. For example, Jagadesh et al. studied sugar cane bagasse ash (SCBA) concretes, which have a lower density than normal strength concrete [73]. A higher elastic modulus than conventional concrete was found when testing processed SCBA, while an original SCBA (unprocessed) replacement resulted in a much lower elastic modulus [73]. This highlights challenges faced by the research community in creating standards and empirical prediction equations for eco-concretes as a lot of data is necessary before the effects of a specific material replacement can be statistically quantified. Cheng et al. reported that increased replacements of coarse aggregate with recycled aggregates greatly affected the elastic modulus and tensile strength of the concrete but had very little impact on the compressive strength [75]. Likely, measuring mechanical properties through non-destructive testing or testing labs will become increasingly more common for specialty projects using eco-concretes.

Data from literature representing a variety of coarse aggregate and binder replacements is shown in Figure 2-8. As seen, the variety of mixes largely provide higher elastic modulus values than that predicted by literature and current code equations. Most notably, the equation developed by Liu et al. [54] extremely underestimates the measured elastic modulus. This indicates that replacing portland cement with geo-polymer cement may have a large impact on the elastic modulus.

This section contains only a small sampling of eco-concretes currently being researched. For the studies reviewed, the current code equations are not adequate. There are likely examples of concrete made with alternative materials where the modulus of elasticity is estimated...
adequately, but it seems probable that there are many more examples where the inverse is true since these replacement materials often differ in properties dramatically from traditional concrete materials. Further investigation and development of new code standards and equations are required before these alternative concretes can be widely used in structural designs.

Figure 2-8: Comparison of measured and predicted elastic modulus values for various eco-concretes from literature [72-74, 76-79]

2.8. Discussion

Current empirical equations tend to predict similar elastic modulus values for a number of specialty concretes, including SCC, HSC, and HPC [25, 30, 36, 42]. While these mixtures can differ greatly from conventional concrete in terms of binder contents, aggregate volume fractions, and presence of admixtures, current literature evaluations generally show elastic modulus predictions to be within a 20% error of measured values [2, 42]. An exception is noted in Baalbaki et al.’s evaluation of HSC where code equations did not provide an adequate prediction for concretes made with sandstone aggregate [36]. While the 20% margin of error matches the one deemed acceptable by ACI for normal strength concrete, it is not always an
adequate prediction for designers, particularly when higher compressive strengths are used. In some situations, high strength concrete allows members to be more slender, requiring a calculated stiffness value to have a much lower potential error due to serviceability and buckling concerns. Comparatively, other types of specialty concretes do not even fall within a 20% error range and therefore require the development of an even more robust estimation than that currently provided in design codes. In particular, UHPC, FRC, and eco-concretes have been shown to require a higher complexity for empirical code equations than presently provided for conventional concretes [21, 23, 54].

For eco-concretes, the variety of aggregate and binder replacement options has created a large variance in concrete properties for researchers to explore. In an effort to sustainably recycle readily available wastes such as tires [74], geopolymers [54], porcelain [72], and sugar bagasse ash [73], researchers have to determine the adequacy of the material, replacement volume fractions, and effects on mechanical properties [72]. In addition, the suitability of current design code equations and standards needs to be evaluated. Varying levels of research have been conducted on potentially suitable recycled materials, but the number of potential replacement options leave this as a field open for further exploration. Prediction equations using an aggregate factor appear promising for aggregate replacements, though a characterization of new materials is required to accurately determine the corrective values [72].

For UHPCs and FRCs, one of the largest theoretical hurdles to overcome is characterizing fibers and determining optimum volume fractions. Progress has been made to develop elastic modulus prediction equations that incorporate the fiber volume fraction and material for FRC, particularly for low coarse to fine aggregate ratios that are most impacted by fibers [21, 23]. However, Dadmand et al. reports large errors in predictions for microfibers ($d_f <$
0.2mm, \( l_f < 13\text{mm} \) in literature [61], indicating the need for further development. Outside the scope of their study, Noushini et al. theorized that higher fiber volume fractions would decrease the elastic modulus of the concrete [52]. While a study by Yoo et al. confirmed this theory and found both a decrease in elastic modulus and compressive strength at a fiber content of 4% [80], Abbas et al. [81] and Kazemi et al. [82] found the compressive strength to increase up to at least fiber contents of 6% and 5%, respectively. Fibers can cause difficulties during consolidation [52, 81] and can affect mechanical properties differently based on their orientation [51] and degree of homogeneous distribution [80], leading to challenges in characterizations.

In specialty concretes like UHPC, the high binder volume fractions and lack of coarse aggregates in the mixture can have a large impact on the applicability of elastic modulus prediction equations developed for conventional concrete. The elastic modulus of coarse aggregates has a large impact on the overall stiffness of the bulk concrete. Without it, the main contributors to stiffness change slightly. Additionally, UHPC often has notable variability in terms of binder replacement rates of portland cement. Nassif et al. showed that the impact of binder replacements on the elastic modulus needs to be accounted for when over 10% of the portland cement is replaced [3], as is often found in concretes with very high compressive strengths.

Effort must focus on mixtures that differ greatly from conventional concretes, such as UHPC, FRC, and eco-concretes, as their behavior is less accurately characterized by current empirical code equations. Underlying reasons for these differences in behavior must be understood at a fundamental level so that as new materials continue to be developed, their properties can be quickly accounted for in the framework of existing equations and techniques.
Additionally, developing equations and models that are convenient for design engineers to use must be a continued focus of future efforts.

2.9. Conclusion

This paper provides a detailed review of current code equations for estimating the elastic modulus of specialty concretes to synthesize existing literature on the topic and to aid future research efforts. Conclusions from the review of relevant literature are as follows.

- Code equations for estimating the elastic modulus of concrete are generally based on empirical data for normal and high strength concretes, relating compressive strength to the elastic modulus.
- Mineral admixtures used in specialty concretes at high replacement rates, fiber use and dosage rate, and changes in coarse aggregate properties, all affect the accuracy of code-based elastic modulus predictions.
- Existing code equations for estimating concrete modulus do not adequately capture effects of aggregate type. Changes in aggregate type were shown to directly impact measured modulus values in the literature. Estimations for elastic modulus of normal-weight SCC are generally within 20% of measured values; however, some SCC mixes made with lightweight aggregate are consistently overestimated by code equations, even after accounting for unit weight. High strength concrete elastic modulus estimations are consistently within 27% of measured values with two notable exceptions in literature being for sandstone coarse aggregate (overestimated) and andesite coarse aggregate (underestimated).
- The existing FRC modulus equation proposed by Suksawang et al. (see again Eq. 2-10) shows good agreement with measured modulus values in the literature (within 23%).
Note however, that when the coarse-to-fine aggregate ratio is less than one, an adjustment factor is required to account for the fiber volume fraction.

- Existing UHPC and eco-concrete modulus equations relying only on compressive strength and unit weight varied greatly from measured values (errors of between -55% and 85% in the literature). Differences between estimated and measured values may be due to increased variability in material types and properties.

2.10. References


[8] ACI Committee 363, "Report on High-Strength Concrete (ACI 363-10)," American Concrete Institute, Farmington Hills, MI, 2010.


Chapter 3. Strength and Stiffness Characterization of Ultra High-Performance Concrete (UHPC) Cement Paste Phases Through In-Situ Micro-Mechanical Testing

3.1. Introduction

3.1.1. Background

Macroscale concrete material properties are influenced by the proportions and complex interactions of aggregates, sands, and hardened cement, which on its own involves complex microscale phases formed through hydration processes. Changes in these constitutive materials and their proportions affect the mechanical properties of the bulk concrete, including strength and stiffness (elastic modulus) [1-3]. Having an accurate understanding of material properties and the underlying interactions that contribute to behavior under loading in the bulk volumes is important for future material developments. Multiple microscale phases can be found in hardened cement paste, which are formed during the hydration reaction between portland cement, admixtures, and water. The needle-like crystalline structure of ettringite (AFT) interacts with the fibrous calcium silicate hydrates (C-S-H) and large prismatic calcium hydroxide (CH) crystals on the microscale to influence the bulk material strength and stiffness [4]. Adjustments to binder materials, water-cement ratios, curing, admixtures, pore content, etc. can affect the size, morphology, and distribution of phases [4-5]. This makes thoroughly understanding macroscale concrete behavior difficult without comprehensive knowledge of microscale interactions.

As such, a bottom-up approach considering individual constitutive contributions may provide a fundamental understanding of hardened properties. A common methodology for determining micro-mechanical properties is nanoindentation which involves applying a load to the surface of a polished sample using an indenter tip, usually a triangular prism-shaped Berkovich indenter, to infer elastic modulus and hardness values for the material [6-8].
Nanoindentation has been conducted on a variety of materials including concrete [9-10], lithium dislocate glass ceramics [11], shale [12], and rocks [13]. As an alternative to nanoindentation, micromechanical testing methods have been explored in literature to understand strength properties (i.e. compressive and tensile strength) more directly [6, 14-18].

A variety of micro-specimen shapes have been milled in literature using a focused ion beam (FIB), including cantilever beams [16, 18], pillars [14, 19-22], and dog-bone tensile specimens [23] on various materials. The FIB uses a finely focused gallium ion (Ga+) energy beam to etch the surface of a polished sample, allowing shapes to be milled on the nano- and micro-scale. The current and voltage can be adjusted to change the temperature effect, gallium ion implantation depth, sharpness of corners and speed of milling. FIB milling has been used on materials like wood cell walls [22], dental enamel [24-25], lamellar bone [21], zirconia [20], concrete [14], single crystalline silicon [26], nanocomposite exoskeleton [27], and gold [28].

Preliminary work has been conducted in the literature to characterize the behavior of hydration phases in hardened cement paste to better understand the mechanisms that control macroscale behavior of concrete. Shahrin et al. fabricated cement paste micropillars to study C-S-H phases [14-15]. The micropillars were fabricated with FIB milling and were loaded in uniaxial compression to determine the compressive strength, elastic modulus, and size effects [14-15]. Shahrin et al. examined the results from sixty-one specimens to establish general trends and a basis for future tests [15].

In this study, the initial loading stiffness and compressive strength of phases in hardened cement paste are characterized through extensive micro-mechanical testing. These phases include CH, ettringite and monosulfates (AFm/AFt), silica fume (SF), and calcium aluminate silicate hydrates (C-A-S-H). Fabrication and compression testing of micropillar geometries were
used to determine the intrinsic properties of CH, AFt/AFm, C-S-H, C-A-S-H and SF. An energy-dispersive X-ray (EDX) was used to identify the phase and uniaxial compression loading of micropillars was performed using a modified pico-indenter with a short flat end indenter tip. Fabrication and testing were conducted with ultra-high performance concrete (UHPC) due to the density of reaction products and relative homogeneity from a lack of coarse aggregate. Nanoindentation was also performed to compare property measurements with the micropillar compression testing.

3.1.2. Research Significance

Characterizing the inherent micro-mechanical properties of UHPC provides a basis for understanding the link between the strength of microstructural cement phases and the overall bulk behavior of the concrete [29-30]. The observed strength and stiffness of concrete are the result of specific microstructural configurations and properties, a relationship which is not currently fully understood. Effort has been made in literature to connect the micro- and macroscale behavior through computational modeling of meso-scale models. While progress is being made in developing an efficient upscaling scheme, the inherent mechanical properties (like compressive strength) of hydration phases and clinker need to be explored further.

3.2. Materials and Methods

3.2.1. Materials and Surface Preparation

The UHPC used in this research was developed for an elastic modulus study by Alsalman et al. [3]. Bulk sample UHPC-3 had an elastic modulus of 45.9 GPa and a compressive strength of 140.8 MPa [3]. The mix design contained Type I portland cement, a 20% by mass replacement with silica fume, and a water-to-binder ratio of 0.2. Steel fibers with a diameter of 0.2
millimeters and a length of 12.7 millimeters were incorporated at 4% by volume fraction [3].

Arkansas river sand (90% of the particles smaller than 1 mm) was used as fine aggregate [3].

Following similar approaches taken by [6, 14, 31], the sample preparation was as follows (Figure 3-1). A Buehler Isomet low speed saw was used to obtain a small rectangular prism sample (approximately 8mm x 16mm x 3mm) from the 75mm x 150mm cylinder used in [3]. To hold the sample more easily while polishing, protect skin against steel fibers, and prevent the concrete surfaces from crumbling, the sample is encased in a polymer resin by using the Buehler SIMPLIMET 4000 Mounting System, leaving one of the large surfaces exposed. A Buehler
Metaserv 2000 was then used to polish the 38-mm diameter polymer disk with multiple grit sizes ranging from 240 to 2000 for 15 minutes each to decrease the surface roughness. This produced a glass-like surface when visually inspected. The Buehler Isomet low-speed saw was used a second time to cut the sample down to 10mm x 10mm x 4mm, taking care to protect the polished face. This size was suitable for use in a scanning electron microscope (SEM). The sample was mounted to a standard SEM stub using double-sided carbon tape. To complete the sample preparation, samples were sputter-coated with platinum (10 nm thick) to increase the electrical conductivity for SEM imaging and to prevent charging of the specimen.

2.2 Micropillar Fabrication

Following the surface preparation, uniaxial compression members (micropillars) were fabricated to study the initial stiffness and strength of the constituent phases in UHPC using FIB. As the FIB is located within an SEM, a vacuum pressure was applied to the specimen, which can cause shrinkage cracks in concrete and potentially densify C-S-H phases [16]. To limit the effects, past studies have air dried their samples for a few weeks prior to fabrication with apparent success [15]. The same was done in the current study, with the polished sample air-drying for over six weeks.

A dual-beam FEG SEM and FIB workstation was combined with an annular milling method to create a series of concentric circles milled with decreasing diameters to form a micropillar shape, as used on a variety of materials by [14, 27-28, 32]. For the FIB used in this study, the sample was rotated within the SEM to a tilt of 52 degrees to position the beam perpendicular to the surface of the sample. The outer ring was milled to a diameter of 20 microns. This initial trench allowed the full length of the micropillar to be visible for a more accurate measurement of its height. The size of this circle also aided in the identification of the location of micropillars.
after fabrication, as in the small sample size makes locating the specimens for testing difficult.

**Figure 3-2**: Steps of the FIB fabrication procedure. Decreasing currents (from 1nA to 0.1nA) are used to etch concentric circles until a micropillar is fabricated within a trench.

The FIB voltage was set to 30kV. As seen in Figure 3-2, the outer ring was fabricated with a beam current of 1nA, with subsequent rings decreasing in current (0.5nA, 0.3nA, and 0.1nA). Decreasing the current results in sharper edges and a more refined shape. Additionally, Němeček *et al.* determined that using a low beam current to fabricate the final testing specimen limits temperature rise, tapering, and gallium ion (Ga⁺) implantation [6]. The surface of the specimen is affected less than 10nm deep when using a 0.1nA current, therefore having a minimal impact on mechanical properties [15, 24]. The total fabrication time per micropillar is
around 2 hours, not including the time needed to set up and close down the SEM and FIB for each session (40-60 mins).

The goal for each micro-pillar is to be representative of a homogenous phase (where possible) to characterize the inherent stiffness and compressive properties. Micropillars were fabricated at a variety of locations within the cement paste in order to provide properties for C-S-H, CH, AFt/AFm, C-A-S-H, and SF. As distinct cement phases are typically in the range of 100 nanometers to 10 microns in size (up to 3 microns for high density C-S-H) [15], fabricated specimens can only be a few microns in diameter at maximum to remain homogeneously representative. Therefore, this study followed the approach taken by Shahrin et al. [14-15] and sought to fabricate micropillars between one and two microns in diameter (Figure 3-3). Shahrin et al. concluded that while some size effects were noted in compressive strength results, elastic modulus calculations were not influenced by the size of the micropillars [15].

**Figure 3-3: Micropillar diagram with typical dimensions**

The FIB method resulted in a range of micropillar diameters relative to the exact desired dimensions. Note that the focused ion beam induces a taper on the sides of the micropillar, creating a smaller top diameter and larger bottom diameter [14, 19, 24, 33]. Taper angles in degrees, top and bottom diameters ($D_1$ and $D_2$, respectively) in µm, and micropillar heights ($H$)
in µm therefore need to be accurately recorded following milling. From these measured dimensions, other characteristics can be determined, such as the aspect ratio (Eq. 3-1). After removal from the SEM/FIB, samples were placed in SEM sample storage containers for protection until testing occurred.

\[
\text{Aspect Ratio} = \frac{H}{(D_1 + D_2)/2}
\]

Eq. 3-1

During fabrication, several challenges needed to be overcome. One of the main issues with using a SEM and FIB with concrete is the lack of conductivity of the material. This causes the image seen by the user to be fuzzy, glow, and/or move around, which can be detrimental to the FIB etching process (Figure 3-4a). The sample movement causes the material removal to shift out of place, causing the micropillar to not be centered in the trench. Small shifts can be overcome, but larger shifts may remove all material in the area, including the micropillar.
Sputter-coating the sample decreases sample charging, but additional measures were needed in the form of silver polish around the edges of the sample and SEM stub.

![Figure 3-4: Challenges with FIB fabrication: a) charging causing sample to move during fabrication; b) excessive removal of material leaving to a slender micropillar; c) material being removed at different rates; d) visible cracking on fabricated micropillar](image)

Additionally, the depth of material removed varies independent of user input settings. Setting the z-axis (depth) removal to 1 μm may remove 1.2 μm on one micropillar and 3.5 μm on another (Figure 3-4b). This is partly due to different phase materials being removed at different rates (Figure 3-4c), but also seems to be inherent to this FIB as similar issues have been reported when using steel. Finally, some micropillars show visible cracking after fabrication and are
unsuitable for testing (Figure 3-4d). This may be due to the micropillar being fabricated on a boundary between multiple phases or prior microcracking in that area.

3.2.2. **Phase Determination**

To determine the phase of each micropillar, an energy dispersive x-ray (EDX) analysis was conducted prior to testing. Each phase present in the cement paste contains different amounts of silicon, calcium, aluminum, sulfur and iron. From the concentrations of these elements, the constituent phase can be identified. A 12kV electron beam was used at a working distance of 5mm to determine the chemical composition of each micropillar. The EDX output provides a spectrum of the elements as well as their atomic percentages. Ratios such as Ca/Si and (Al+Fe)/Ca are calculated to help the identification; ranges and key identifiers from literature (Table 3-1) were used to aid in phase identification [34-37]. Tragardh *et al.* considered areas between the given ranges to represent a composite of two or more phases [34]. These ratios agree well with other designations in literature [38-42].

**Table 3-1:** Quantitative phase identification ranges for EDX analysis of micro-pillars

<table>
<thead>
<tr>
<th>Cement Paste Phase</th>
<th>Ratios for Identification from EDX Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-S-H</td>
<td>0.8 ≤ Ca/Si ≤ 2.5</td>
</tr>
<tr>
<td>C-A-S-H</td>
<td>0.8 ≤ Ca/Si ≤ 2.5</td>
</tr>
<tr>
<td>CH</td>
<td>Ca/Si ≥ 10</td>
</tr>
<tr>
<td>AFt/AFm</td>
<td>Ca/Si ≥ 4</td>
</tr>
<tr>
<td>SF</td>
<td>Ca/Si ≤ 0.5</td>
</tr>
<tr>
<td></td>
<td>(Al+Fe)/Ca ≤ 0.2</td>
</tr>
<tr>
<td></td>
<td>(Al+Fe)/Ca ≥ 0.2</td>
</tr>
<tr>
<td></td>
<td>(Al+Fe)/Ca ≤ 0.4</td>
</tr>
<tr>
<td></td>
<td>(Al+Fe)/Ca &gt; 0.4</td>
</tr>
</tbody>
</table>

3.2.3. **Micro-Compression Testing**

For the compression testing of the micropillars, the sample was placed in a TESCAN VEGA3 SEM with a modified Hysitron PI-88 pico-indenter (Figure 3-5). The Hysitron pico-
indenter is capable of measuring displacements and applied forces on the micro-level and is similar to the equipment used in prior studies [6, 14-16].

Figure 3-5: Micropillar compression testing using a PicoIndenter and a short flat-punch tip with loading protocol graph

To prevent contact between the surrounding material and the tip during testing, a 5-micron diameter tip was used. Cement paste micropillars were tested with a monotonic single cycle load profile to a maximum of 9mN at a rate of 0.01 mN/s, based on research conducted by Shahrin et al. [14-15].
Initially the maximum load was set at 2mN to directly follow [14], but failure did not occur before the maximum load was achieved in preliminary testing, so the value was increased to 9mN. The loading was applied in contact with drift correction using a short flat end tip, as used in [26, 33, 43]. The tip shape provided an even distribution of force across the surface of the micropillars. Failure was defined by a sudden increase in displacement of the tip during loading. Due to tapering, failure is most likely to occur in the top half of specimen where the smaller diameter leads to a higher stress. Therefore, the top diameter in μm \((D_1)\) is used in calculations of the compressive strength in MPa \((f_c)\) along with the maximum load carried by the pillar in mN \((F_Y)\) (Eq. 3-2) [22, 24, 27].

\[
f_c = \frac{4F_Y}{\pi D_1^2} \quad \text{Eq. 3-2}
\]

Using the force-displacement data collected during compression testing, stress and strain data was plotted using Eq. 3-3 and Eq. 3-4 [15, 27]. As the micro-pillars were fabricated on the concrete surface, the effect of indentation of the specimen into the underlying concrete needs to be considered. To account for this compliance of the bulk material, Sneddon et al.’s concept of an elastic half-space being acted on by perfectly rigid cylindrical punch has been adapted to apply to micro-compression testing in [15, 24, 27]. Therefore, the normalized engineering stress \((\sigma_n)\) (Eq. 3-3) and strain \((\varepsilon_n)\) (Eq. 3-4) were determined from the load-displacement data,

\[
\sigma_n = \left(\frac{4}{\pi D_1 D_2} \frac{1-\nu^2}{D_2 H}\right)F \quad \text{Eq. 3-3}
\]

\[
\varepsilon_n = \frac{\delta}{H} \quad \text{Eq. 3-4}
\]

where \(\nu\) is Poisson’s ratio, \(F\) is the force in mN recorded by the indenter, and \(\delta\) is the displacement at that force. For UHPC, Poisson’s ratio is generally between 0.21 and 0.25 as seen in [31, 44-47]. Nemecek et al. used a constant Poisson’s ratio for all phases after determining a range of 0.15-0.25 to have very little impact on the elastic modulus calculation [6, 16]. In this
study, 0.21 was used to determine the stress. Adjusting Poisson’s ratio between 0.18 and 0.26 had a negligible impact on the stiffness calculation. Once the stress-strain curve is developed, a linear line of best fit was added to the initial elastic portion of the curve (from 0 to 45% of the yield strength). The slope of this line was taken as the initial stiffness of the micropillar.

3.2.4. Nanoindentation

As elastic modulus data in literature for cement paste phases has primarily been reported from nanoindentation testing, the current study included this method for a general comparison after noting differences between stiffnesses provided by micro-compression and literature nanoindentation. Nanoindentation testing was conducted using a Berkovich tip to indent the surface of the hardened cement paste at a loading rate of 50 μN/s (Figure 3-6). The maximum load was held for one second before unloading. Seventy indents were recorded in a 7 by 10 array for each maximum load to provide a general bridge between the concrete sample used in this study and literature nanoindentation results. As in Wilson et al. and Bily et al., the spacing between indents was set at 10 μm [48-49]. Initial testing concluded that maximum loads of 750 μN and 1000 μN would result in indentation depths between 100 and 350 nm. This displacement range is commonly presented in literature as a distinct phase within the interaction volume [6, 50-52].

The Oliver-Pharr method was used to calculate the elastic modulus at each indentation [53-54]. The projected area of contact between the indenter tip and the surface of the material is calculated using Eq. 3-5 and 3-6 [53, 55]:

\[ A_c = \pi (\tan(\theta) h_c)^2 \]  
\[ h_c = h_{max} - 0.75 \frac{P_{max}}{S} \]

where \( A_c \) is the contact area in μm², \( \theta \) is the tip equivalent half-cone angle (70.32 degrees for an ideal Berkovich tip [55]), and \( h_c \) is the contact height in μm from Eq. 3-6.
Figure 3-6: Nanoindentation testing in an array using Berkovich tip. Loading diagram and sample graph shown.

The contact height is based on the maximum depth in μm ($h_{max}$), the maximum load in μN ($P_{max}$), and the initial slope of the unloading curve from the load-depth graph in MPa ($S$) (Figure 3-6).

The elastic modulus of the indent is determined from Eq. 3-8, which requires calculation of the reduced modulus, $M$, in Eq. 3-7:

$$M = \frac{\sqrt{\pi} \cdot S}{2\beta \sqrt{A}}$$  \hspace{1cm} \text{Eq. 3-7}

$$\frac{1}{M} = \frac{1-v_i^2}{E} + \frac{1-v^2}{E_i}$$  \hspace{1cm} \text{Eq. 3-8}

where $\beta$ is a geometric correction factor (1.034 for Berkovich tips [55-56]), $\nu_i$ is Poisson’s ratio for the indenter tip (0.07 [6, 57]), $E_i$ is the elastic modulus of the tip (1141 GPa [6, 57]), $\nu$ is Poisson’s ratio of the material (0.21), and $E$ is the elastic modulus of the material in MPa.

3.3. Results

A total of forty-six micropillars were fabricated, representing six hydration phases and micro-composites in the hardened cement paste. Upon visual inspection for surface cracks or
deformities, only thirty-five micropillars were deemed acceptable for compression testing. Initial loading stiffness and compressive strength data is compiled for micro-pillars by phase in Table 3-2, Figure 3-7, and Figure 3-8. The stiffness and compressive strength mean ($\mu$) and standard deviation ($\sigma$) for each phase is provided on the stress-strain graphs. Properties of individual micropillars are in Appendix A.

3.3.1. Micropillar Stiffness

AFt/AFm micro-pillars showed the highest stiffnesses with an average of 41.7 GPa. CH had a stiffness value around half of AFt/AFm at 19.8 GPa average, though the CH data is from only two micro-pillars and therefore may not accurately represent the intrinsic phase properties. One CH micro-pillar had a loading stiffness of 27.4 GPa, while the second one, which also had a lower Ca/Si ratio, was 12.2 GPa. Pure CH phase areas were difficult to identify and challenging to fabricate a micropillar on the exact location in the cement paste, likely due to the presence of silica fume in the concrete mixture [58]. Silica fume is known to consume CH and increase the amount of C-S-H in the hardened cement paste [40, 59-60]. Furthermore, Laugesen et al. reported the hexagonal plate structure of CH to exhibit directional dependency [61]; the exact orientation within the micropillars is unknown. As such, the CH data does not represent conclusive micropillar loading stiffness values and is provided here as a basis for future studies. Many nano-mechanical investigations have been performed on cement paste made with a portland cement binder which allows for easier testing of CH. However, notably less research in this field has been conducted with pastes containing binder replacements like silica fume and fly ash which are commonly used in UHPC. The micro-mechanical impact from binder adjustments needs to be investigated, though it may pose additional challenges to researchers such as not being able to easily quantify the properties of CH.
Table 3-2: Compressive strength and elastic modulus results by hydration phase

<table>
<thead>
<tr>
<th>Phase</th>
<th>No. of Specimens</th>
<th>$f_{c,avg}$ (MPa)</th>
<th>$E_{avg}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFt/AFm</td>
<td>4</td>
<td>2840 ± 760</td>
<td>41.7 ± 8.9</td>
</tr>
<tr>
<td>C-A-S-H</td>
<td>5</td>
<td>980 ± 515</td>
<td>9.4 ± 2.2</td>
</tr>
<tr>
<td>CH/C-S-H Composite</td>
<td>8</td>
<td>2100 ± 1100</td>
<td>21.6 ± 14.5</td>
</tr>
<tr>
<td>CH</td>
<td>2</td>
<td>800 ± 895</td>
<td>19.8 ± 10.8</td>
</tr>
<tr>
<td>C-S-H</td>
<td>7</td>
<td>1560 ± 600</td>
<td>10.6 ± 2.3</td>
</tr>
<tr>
<td>SF</td>
<td>9</td>
<td>1480 ± 530</td>
<td>12.3 ± 3.5</td>
</tr>
</tbody>
</table>

The loading stiffnesses of C-S-H, C-A-S-H, and SF ranged from 9.4 to 12.3 GPa (Table 3-2). The CH/C-S-H composite exhibited the highest standard deviation (21.6 ± 14.5 GPa) but no notable variations in chemical composition or failure modes were observed between the two distinct groupings (averages of 9.6 GPa and 33.6 GPa). No visible defects were observed on the micro-pillars with the lower stiffnesses prior to loading. Considering the directional dependency reported by Laugesen, the higher and lower stiffnesses may indicate different orientations of the CH crystal within the CH/C-S-H composite [61]. Additionally, Asgari et al. reported CH responses to vary based on whether a single layer was evaluated or a multi-layer; the former showing a brittle response to load and the latter behaving as a semi-ductile material [62]. Therefore, the amount of layers intermixing with C-S-H in the composite may also affect the loading elastic modulus and compressive strength behavior. According to Hu et al. the lower CH/C-S-H composite stiffness and strength values may also be indicative of a higher pore volume [63].
Figure 3-7: Micropillar stress-strain graphs by phase with average loading stiffness data plotted.
3.3.2. *Micropillar Compressive Strength*

Compressive strengths followed a similar trend to the stiffnesses; AFt/AFm micro-pillars had a notably higher average compressive strength than the other hydration phases (around 2840 MPa). SF and C-S-H had similar average compressive strengths (1480 and 1560 MPa respectively), while the CH/C-S-H composite was a little higher at 2100 MPa. C-A-S-H had an average failure stress of 980 MPa. The two CH micro-pillars failed at the lowest average stress, 800 MPa; once again, however, there is not enough data to determine the intrinsic property value for CH. While no defects were visible on the surface of the micro-pillars, it is unknown if any hidden nano-cracks influenced the failures. To the authors’ knowledge, this study is the first compressive strength evaluation of hydration phases beyond C-S-H. As such, the influence of CH crystal orientation on the compressive strength is unknown. Like the elastic modulus, the compressive strength of CH may have a dependency on crystal orientation.

3.3.3. *Failure Modes*

Failure modes were determined from video footage taken during loading and evaluation of post-failure SEM images. Partial and complete crushing were observed in many of the tests. In these failures, the micropillar material compressed either halfway or all the way to the substrate underneath before failure occurred. These failure modes have been observed in other micropillar studies [14, 24]. Partial or complete crushing was observed in 71.4% of the tests, while axial splitting and shear failures accounted for 14.3% and 14.3% respectively (Figure 3-9). AFt/AFm micro-pillars predominantly failed in axial splitting, proving to be more brittle than the other hydration phases tested (Figure 3-8 and Figure 3-9). The axial splitting failure occurred when a crack propagated from the top to the base of the micropillar, either in the center or on two sides of the pillar, in a similar manner to possible macroscopic concrete cylinder failures [64].
Figure 3-8: Micropillar stress-strain graphs by phase with average compressive strength data plotted
Five micro-pillars failed in shear, primarily at mid-height with slight buckling noticeable prior to failure. The aspect ratio of these micro-pillars was comparable to the micro-pillars exhibiting other failure modes, indicating that slenderness was not the only factor directing the shear behavior. One shear failure occurred at the base of the micro-pillar, likely due to an applied moment caused by a misalignment between the indenter tip and the micro-pillar top surface.

**Figure 3-9:** Post-compression testing micropillar failure modes

3.3.4. *Effect of Top Diameter and Aspect Ratio*

Evaluation of the elastic modulus and compressive strength values with respect to the top diameters and aspect ratios of the fabricated micro-pillars showed no distinct trends. However, this may be due to the limitation in size range of the micro-pillars, as size effects were not the focus of the current experiment. Most phases showed a general tendency towards a decreasing
compressive strength or stiffness with increasing top diameter, but not enough data was collected to draw definitive conclusions. Shahrin et al. reported size effects only on the compressive strength of C-S-H micro-pillars, with little discernable impact on the elastic modulus [15].

3.3.5. Nanoindentation Elastic Modulus

Nanoindentation was performed on the same sample as the micro-compression testing. One hundred and forty indents were measured for two maximum loads: 750 μN and 1000 μN. Of the seventy indents performed to a maximum load of 750 μN, twelve data points with unusual load-deformation behavior were removed due to premature fracture. The remaining indentation locations were analyzed using EDX to determine which phase the elastic modulus represented (Figure 3-10) [55, 65]. Five phases were identified, though only one indent was performed on AFt/AFm. Therefore, data is not reported for AFt/AFm. Similarly, only one indent represented unhydrated cement. C-S-H indents had an average elastic modulus of 17.6 GPa, while C-A-S-H, SF, and CH/C-S-H composites had average moduli of 12.5 GPa, 14.4 GPa, and 7.5 GPa, respectively. No indentations were conducted on CH.

Table 3-3: Elastic modulus results from nanoindentation testing at maximum loads of 750 μN and 100 μN

<table>
<thead>
<tr>
<th>Phase</th>
<th>Max Load (μN)</th>
<th>750</th>
<th>1000</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-A-S-H</td>
<td>12.0 ± 4.3</td>
<td>6.5 ± 1.7</td>
<td></td>
</tr>
<tr>
<td>CH/C-S-H Composite</td>
<td>7.5 ± 4.8</td>
<td>7.2 ± 1.0</td>
<td></td>
</tr>
<tr>
<td>C-S-H</td>
<td>17.6 ± 8.6</td>
<td>6.5 ± 1.8</td>
<td></td>
</tr>
<tr>
<td>SF</td>
<td>14.4 ± 8.4</td>
<td>5.7 ± 1.9</td>
<td></td>
</tr>
</tbody>
</table>

The average indentation depth for the maximum loads of 750 μN and 1000 μN were 250 nm and 350 nm, respectively. For the maximum load of 1000 μN, the average values for C-S-H, C-A-S-H, and SF were notably lower than for 750 μN (63%, 48%, and 61% less, respectively). The
average elastic modulus of the CH/C-S-H composite was similar at both maximum loads with only a 4% difference. For nanoindentation studies of a variety of materials where maximum loads under 100 mN are used, a decrease in elastic modulus with an increasing maximum load is observed, which may also be applicable to concrete.

3.4. Discussion

Micro-specimen testing has been used in the past decade to gather preliminary data on the compressive and tensile strength of hydration phases in concrete. Zhang et al. sought to determine the homogeneous compressive strength at the micrometer scale by conducting tests on cube shaped pillars 100µm by 100µm and 200µm by 200µm [66]. At this length scale, the pillars may represent multiple hydration phases and defects (such as pores) influence their behavior. The same may also be true for the studies conducted by Němeček et al., where cantilever micro-beams 20µm in length were loaded to evaluate the tensile properties of C-S-H and CH [6, 16]. While some micro-beams may represent a homogenous hydration phase, it is likely that many contain a variation of hydration phases, chemical compositions and/or defects due to the length of the specimens [14].

3.4.1. Comparison to Micropillar Results in Literature

Shahrin et al. conducted compression tests on C-S-H micro-pillars ranging from 0.5 to 2.5 µm in diameter [14-15] using a similar methodology as the current study. Initial stiffness results from monotonic loading for C-S-H micro-pillars provided a comparable average (8.8 GPa [14]) as compiled in the current study (10.6 GPa). However, in a later study by Shahrin et al. where unloading slopes of multi-cyclic tests were used to calculate the elastic modulus the average increased to 23.6 GPa [15]. The difference between the initial stiffness and the unloading elastic modulus in these studies is consistent with literature [67]. When determining the initial
micropillar stiffness, the behavior of the material under loading is measured in the same manner that macroscopic concrete cylinder testing is conducted [67]. By analyzing the unloading curves, the pure elastic recovery of the material is determined.

Comparing the C-S-H compressive strength data from the current study with that published by Shahrin et al. shows some dissimilarities. For pillars with nominal top diameters of approximately 1µm and 1.5µm, Shahrin et al. reported failure stresses of 704 ± 178.5 MPa and 660.5 ± 286.1 MPa respectively after loading the micropillars three times prior to failure [15]. Comparatively, an average failure strength of 1560 ± 600 MPa was reported in the present investigation where monotonic loading is used. Note that different indenter tip shapes were used (a short flat punch tip in this study vs. a spherconical tip in Shahrin et al. [15]) and variances in concrete mixture and curing conditions are present between the two investigations.

3.4.2. Comparison to Nanoindentation Results

![Graph showing elastic modulus comparison](image)

**Figure 3-10:** Average elastic modulus from nanoindentation testing at two maximum loads compared to micro-compression test results

A comparison of the micro-compression results with the experimental nanoindentation values (for 750 µN) shows that the latter provides a notably higher average stiffness for SF, C-S-
H, and C-A-S-H (17%, 66%, and 33% respectively). Conversely, CH/C-S-H composite nanoindentation values were 65% lower than micro-compression (see Figure 3-10).

Nanoindentation testing providing an elastic modulus value larger than micro-compression initial stiffness remains true when comparing to literature nanoindentation values (see Table 3-3). As seen in Table 3-3, there is disagreement between studies on the intrinsic elastic modulus of phases. For example, C-S-H micro-compression initial stiffness values are only 10% lower than the nanoindentation elastic modulus (for low-density C-S-H) in [68], but 70% lower than the nanoindentation values presented by [48]. Micro-compression of CH/C-S-H micropillars also exhibit significantly lower stiffness than nanoindentation, at an average of 56% less. Known limitations and test conditions for both methodologies likely explain the high variability between the two. For example, nanoindentation examines the unloading slopes for the elastic modulus calculations, while the current micro-compression study used the loading slopes to map nonlinear and failure behavior. Fundamentally, the initial stiffness measured in the micro-compression testing will differ from the elastic recovery measured by nanoindentation. The flat punch indenter tip (micro-compression) and triangular pyramid shaped Berkovich tip (nanoindentation) have varying geometries, which cause different stress fields during loading [26]. The Berkovich tip is sharp and plastically deforms the surface during loading, necessitating the unloading curves be analyzed to calculate elastic recovery. In contrast, the flat punch tip can elastically and plastically deform the micro-pillars, allowing an initial stiffness to be measured.

Looking more closely at each methodology, micro-compression testing is conducted in a uniaxial stress state with a limited interaction volume. Effects from the vacuum and FIB cannot be completely ignored as potentially influencing results, though Němeček et al. proved that by limiting the current (as done in the present study) FIB effects can be minimized [16]. The taper
angle of the FIB-milled columns additionally has been reported to cause an increase in the reported elastic modulus [67], though the values provided in the present study are lower than literature nanoindentation values.

**Table 3-4:** Elastic modulus (E) values from literature to compare to results from this study

<table>
<thead>
<tr>
<th>Phase</th>
<th>Test Method</th>
<th>$E_{\text{avg}}$ (GPa)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFt/AFm</td>
<td>NI</td>
<td>40.6</td>
<td>[50]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>17.4</td>
<td>[10]</td>
</tr>
<tr>
<td></td>
<td>MC</td>
<td>41.7</td>
<td>**</td>
</tr>
<tr>
<td>C-A-S-H</td>
<td>NI</td>
<td>26.2</td>
<td>[50]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>11.3</td>
<td>**</td>
</tr>
<tr>
<td></td>
<td>MC</td>
<td>9.4</td>
<td>**</td>
</tr>
<tr>
<td>CH</td>
<td>PF</td>
<td>52.2</td>
<td>[62]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>79.8</td>
<td>[48]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>47.7</td>
<td>[57]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>43.7</td>
<td>[6]</td>
</tr>
<tr>
<td></td>
<td>PF</td>
<td>33.5</td>
<td>[69]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>41.8</td>
<td>[50]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>25.4</td>
<td>[73]</td>
</tr>
<tr>
<td></td>
<td>MC</td>
<td>19.8</td>
<td>**</td>
</tr>
<tr>
<td>C-S-H</td>
<td>PF</td>
<td>69.4</td>
<td>[62]</td>
</tr>
<tr>
<td></td>
<td>MC</td>
<td>8.2</td>
<td>[14]</td>
</tr>
<tr>
<td></td>
<td>MC</td>
<td>23.6</td>
<td>[15]</td>
</tr>
<tr>
<td></td>
<td>PF</td>
<td>25.5</td>
<td>[69]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>27.0</td>
<td>[38]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 51.1; LD: 34.1*</td>
<td>[48]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 26.1; LD: 12.0*</td>
<td>[74]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 35.7; LD: 26.8*</td>
<td>[57]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 31.9; LD: 24.4*</td>
<td>[6]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 31.0; LD: 21.5*</td>
<td>[2]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 30.6; LD: 22.4*</td>
<td>[9]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 33.4; LD: 19.5*</td>
<td>[75]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 34.4; LD: 25.1*</td>
<td>[55]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>HD: 16.6; LD: 11.7*</td>
<td>[73]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>16.1</td>
<td>**</td>
</tr>
<tr>
<td></td>
<td>MC</td>
<td>10.6</td>
<td>**</td>
</tr>
<tr>
<td>CH/C-S-H</td>
<td>NI</td>
<td>47.5</td>
<td>[9]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>34.0</td>
<td>[38]</td>
</tr>
<tr>
<td></td>
<td>NI</td>
<td>6.7</td>
<td>**</td>
</tr>
<tr>
<td></td>
<td>MC</td>
<td>21.6</td>
<td>**</td>
</tr>
</tbody>
</table>

NI = nanoindentation; MC = micro-compression; PF = peak force AFM

* Reported as high density (HD) C-S-H and low density (LD) C-S-H

** Results from current study
Another known limitation to micro-compression testing is imperfect contact between the top diameter and tip during initial loading. Shahrin et al. reported some issues as evidenced by the dramatic upward curve as the sphericonical tip encountered the pillar, particularly with their 0.5-µm diameter pillars [15]. By using a flat end indenter tip and beginning the loading protocol in contact with the micro-pillar and correcting for drift, the current study did not observe such a sharp change in slope. Still, imperfect contact between the specimen and indenter may have had some influence on the elastic modulus.

In contrast, nanoindentation is performed in a triaxial stress state, influenced by the mechanical properties of the surrounding material. Most studies refer to the pioneering work of Oliver and Pharr [53], which is based on the elastic analysis of an ideal indenter shape indenting an infinitely homogeneous isotropic half-space with no surface roughness [69]. As hardened cement paste is a heterogeneous material of varying mechanical properties and defects, these initial assumptions complicate the interpretation of data gathered using nanoindentation [68]. The elastic modulus is calculated based on the contact area between the tip and sample surface, recorded stiffness from the initial unloading curve, and a geometric correction factor accounting for the non-symmetric indenter tip shape [53, 56]. Literature shows that errors in measuring the contact area [54, 70-71], using peak loads less than 100 mN [53, 72] (concrete is commonly tested to a maximum load of around 1-2mN [48-52]), and not first determining the critical indentation depth for a specific concrete mix [40, 68] can cause overestimations in elastic modulus testing.

As an alternative testing methodology, micro-compression experimentation is a novel way to determine the micro-mechanical properties of hardened cement paste. Němeček et al. found good agreement between the micro-beam bending and nanoindentation elastic modulus results.
(i.e. 39.2 GPa for CH in beam-bending and 39.0 GPa for CH in nanoindentation) [6], though the length of the cantilever beams indicate the presence of several hydration phases as likely. Further testing to expand sample sizes and evaluate potential influences is necessary for micro-specimen testing on hardened cement paste. The current study offers additional insight into applying micro-compression tests for concrete on multiple hydration phases.

3.5. Conclusions

This study applied an effective methodology to characterize the compressive strength and elastic modulus of phases in cement paste from a UHPC sample. Necessary data for the basis of future models delineating the strength and deformation behavior of hardened cement paste at various length scales is produced. The following conclusions are drawn:

- Micropillars fabricated using FIB and tested using PI-88 with monotonic loading allows initial stiffness and compressive strength to be mapped, providing a better understanding of individual phase behavior under loading.
- Loading stiffness values for five hydration phases and one composite were determined. AFt/AFm crystals produce the highest average initial stiffness at 41.7 GPa. Micropillars made of SF, C-S-H, and C-A-S-H exhibit notably lower average stiffnesses at 12.3 GPa, 10.6 GPa, and 9.4 GPa respectively. CH/C-S-H composite is additionally studied with a stiffness of 21.6 GPa.
- Similarly, AFt/AFm fails at a much higher strength (2840 MPa) than C-A-S-H, SF, and C-S-H which ranged between 980 MPa and 1560 MPa.
- The primary failure mode of hydration phases at the 1-3 µm length scale is partial or complete crushing of the micropillar, though AFt/AFm micropillars largely exhibit brittle failure through axial splitting through either the center or sidewalls.
Nanoindentation testing provides an unloading elastic modulus of the phases which is much higher than micro-compression loading stiffnesses. Dissimilarities in testing conditions, methodologies, and assumptions in the analysis technique likely account for the differences.

3.6. References


Chapter 4. Estimation of Cement Paste and UHPC Elastic Modulus through Measured Phase-Property Upscaling

4.1. Introduction

Current code equations fail to accurately predict the elastic modulus of specialty concretes because, generally, their only inputs are unit weight and cylinder compressive strength. These equations, such as in ACI 318 [1] and ACI 363 [2], are based on empirical testing of normal or high strength concrete and do not consider the impact of microstructure non-traditional mixture components such as fibers, present in specialty concretes [3-5]. Effort has been made in literature to develop new curves but these approaches are specific to certain combinations of mixture inputs [4, 6-9]. As an alternative approach, researchers have mapped micro-scale properties of concretes using methods like nanoindentation and micro-compression to gain a better understanding of the mechanisms that control stiffness [10-13]. Once the intrinsic micro-mechanical behavior is determined, it is necessary for a user-friendly design tool to be developed to provide insight for those working with bulk concrete. For any bottom-up approach, the complexity of concrete requires different scale levels to be considered. Concrete is regularly conceptualized in literature as having four levels (Figure 4-1) [14-15].

![Figure 4-1: Levels of concrete upscaling considered in literature](image_url)
Level one (around 100 nm to 10 μm) encompasses capillary porosity, high density (HD) calcium silicate hydrates (C-S-H) and low density (LD) C-S-H [14, 16]. Level two (around 10-100 μm) considers the homogenization of all cement paste phases such as calcium hydroxide (CH), ettringite and monosulfoaluminate (AFT/AFm), clinker, and C-S-H matrix [15-17]. Level three (around 100-1000 μm) includes mortar (made of sand, small voids, cement paste, and occasionally fibers). Level four (larger than 1000 μm) is the culmination of mortar, coarse aggregate, and large air voids [15].

Different approaches in literature have been used to incorporate micro-scale experimental data into macro-scale models with varying levels of success. Levels three and four are commonly evaluated through mesoscale modeling of a representative volume element (RVE) or a statistical volume element (SVE) [18-22]. The volume element sizes are typically chosen to be significantly smaller than structural components, but large enough to contain a representative sample of the constituent materials [23]. RVEs are used to model the effective homogenized properties and are sized to reflect the global response of the bulk material [24]. SVEs are smaller than RVEs, but still larger than the smallest characteristic microstructure [18, 24]. The apparent homogenized properties are modeled by SVEs as there is variability between elements of microstructural volume fractions, densities, sizes, and shapes [24-25]. Upscaling through volume elements necessitates the use of finite element software [18-19] and may require a high computational cost to effectively upscale the complexities found in concrete at multiple length scales [24-25]. Boundary conditions, particle interactions, particle shape effects, and potentially having to remove SVEs made entirely of voids are some other challenges faced by researchers [24, 26-29]. While it appears to be an effective approach for concrete upscaling, significant development is required before design engineers may find it a practical tool.
In this study, a novel approach to upscale micro scale experimental data into a meso- or macroscale model is presented. A preliminary programming code is developed to upscale micromechanical data through automated spring arrangement simulations. By combining phase elastic modulus values in series and parallel, the overall stiffness of a set of springs can be calculated to potentially represent homogenized cement paste. A similar approach is taken to combine the cement paste data with aggregate and fiber stiffnesses for an ultra-high performance concrete (UHPC) mix. UHPC was used to build these models due to its relative homogeneity compared to conventional concrete and for the density of its reaction products. Monte Carlo simulations are used to ensure that the randomness of concrete is captured and to limit the effects of any errors in micro-mechanical testing.

4.2. Code Development

The code requires the user to input the volume fractions of phases, aggregates, voids, and fibers to assign properties to a relevant number of springs. This allows the user to examine the effects of adjusting the amounts of each material on the elastic modulus and to tailor future mix designs to produce desired properties. The first half of the code upscales the elastic moduli of phases and voids (level 2) to determine the overall hardened cement paste stiffness. Micromechanical properties for the phases are taken from Chapter 2 and summarized in Table 4-1.

The void elastic modulus is taken as 0.00001 GPa. A value of zero could not be used because this would cause issues with converging on a real solution. Hain et al. reported a negligible influence when using pore stiffnesses between 0 N/mm$^2$ and 1000 N/mm$^2$, so using a non-zero value should not impact results [31]. The second half of the code upscales level 3: the hardened paste with steel fibers ($E = 210$ GPa) and fine aggregates ($E = 60$ GPa). Level four includes any coarse aggregates, which are not considered in this code or experimental
verification. A small addition of low stiffness springs to account for micro-cracking in the specimen are also included in the second half of the code \( (E = 0.0001 \text{ GPa}) \). Springs are combined in series and parallel.

![Image of micropillar and indenter tip](image)

**Figure 4-2:** Compression testing of micropillars to determine the loading stiffness of phases in cement paste from [30] with graph of hydrated silica fume (SF) micropillar results

**Table 4-1:** Mean phase elastic moduli and standard deviation from Chapter 3

<table>
<thead>
<tr>
<th>Phase</th>
<th>( E ) [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFt/AFm</td>
<td>41.7 ± 8.9</td>
</tr>
<tr>
<td>C-A-S-H</td>
<td>9.4 ± 2.2</td>
</tr>
<tr>
<td>CH/C-S-H</td>
<td>21.6 ± 14.5</td>
</tr>
<tr>
<td>CH</td>
<td>19.8 ± 10.8</td>
</tr>
<tr>
<td>C-S-H</td>
<td>10.6 ± 2.3</td>
</tr>
<tr>
<td>SF</td>
<td>12.3 ± 3.5</td>
</tr>
</tbody>
</table>

A flowchart of the programming code is provided in Figure 4-3. Based on the input volume fractions, 1000 springs are assigned a phase or void stiffness value, randomly chosen from within one standard deviation of the experiment mean [30]. For example, if the volume fraction of C-S-H is 53%, then 530 springs are assigned a random stiffness value between 8.3 GPa and 12.9 GPa. This accounts for variability across a concrete sample and decreases the influence of any potential testing errors in determining the intrinsic phase average modulus.
Figure 4-3: Code flowchart code upscaling using spring arrangement simulations
Once an array of 1000 stiffness values (representing 1000 springs) has been collected, the spring particle arrangements are created. A random number of springs between one and one hundred are selected to be combined in parallel until all 1000 springs have been grouped. For example, a simulation may combine the stiffness values into groups of 95, 2, 15, 64, etc. in parallel. The groups are then combined in series and the overall stiffness of the 1000 springs is calculated. By running numerous simulations, convergence can be achieved for a cement paste.

The same approach is used for the second half of the code. This time, 1000 springs are assigned stiffness values representing fibers, aggregate, cement paste (from level 2 upscaling output), and micro-cracking at the appropriate volume fractions based on the mix design. The cement paste inputs are taken from within one half of a standard deviation of the mean output from the first part of the code. The springs are once again randomly grouped in parallel and then combined in series with the other groups until an upscaled UHPC stiffness value is calculated.

![Figure 4-4: Convergence of Code](image_url)
Simulations are run until convergence is reached. Convergence of the code is evaluated in Figure 4-4. As seen, for a set of inputs, the code outputs stabilize after around 50,000 iterations.
simulations. Since not too much additional computational time is required and each run has an even lower variability, 100,000 simulations were used for comparison to experimental testing.

The distribution of elastic modulus values generated is also evaluated. As seen in Figure 4-5 three distinct bands of stiffness values are generated by the code. For the level two upscaling (Figure 4-5a), values above 100 GPa are considered outliers (Cutoff A). This removes around 12% of the simulations. Above this threshold lies unrealistic stiffness values for hardened cement, which typically is less than 45 GPa [32-38]. Similar consideration is given to level three UHPC upscaling (Figure 4-5b), with only stiffness values below 210 GPa (Cutoff B) considered (typical UHPC stiffness values are well below [29, 39-41]). The composite UHPC should not have a higher elastic modulus than the stiffest constituent element (steel fibers).

4.2.1. Experimental Verification

For experimental verification of the programmed code, hardened paste, mortar, and UHPC samples were created. The mixture proportions for each are provided in Table 4-2. The hardened paste mixes consisted of portland cement, silica fume, water, and a high range water reducer (HRWR). Two replacements of cement with silica fume (10% and 20%) were used. For the mortar mix, natural river sand (passing a No. 4 sieve with a fineness of 2.83) was added to the materials used in the hardened paste mixes. The UHPC samples included cement, silica fume, water, HRWR, sand, and steel fibers (0.2 mm diameter and 13 mm length). The amount of water was adjusted to reflect the water content of the fine aggregate.

For all samples, the portland cement and silica fume were first stirred together in a tabletop mixer for 3 minutes. During this time, the water and HRWR were combined in a separate bowl. Once the silica fume powder was well distributed in the portland cement, the water and admixture were added. After 4 minutes of mixing, the bottom and sides of the bowl
were manually scraped. It was noted that the powder tended to clump at the bottom of the mixer and so manual scraping was included for every 2 minutes of mixing. The paste was mixed for a total of 10 minutes (starting when the water and admixture were added). For the mortar mixture, the fine aggregate was added after 5 minutes of mixing for a total of 12 minutes. For the UHPC mixture, the fibers were added after 10 minutes of mixing with an additional 4 minutes added to ensure good dispersion. The fresh concrete was tested for air content, which ranged from 3.8 to 5.6 percent.

Table 4-2: Concrete mix design for experimental verification of spring arrangement simulations

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Binder (kg/m$^3$)</th>
<th>w/b</th>
<th>Silica fume (%)</th>
<th>Sand (kg/m$^3$)</th>
<th>Steel fiber (%)</th>
<th>HRWR (kg/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P10</td>
<td>1365</td>
<td>0.2</td>
<td>10</td>
<td>0</td>
<td>0</td>
<td>30.26</td>
</tr>
<tr>
<td>P20</td>
<td>1365</td>
<td>0.2</td>
<td>20</td>
<td>0</td>
<td>0</td>
<td>30.26</td>
</tr>
<tr>
<td>M20</td>
<td>1365</td>
<td>0.2</td>
<td>20</td>
<td>647</td>
<td>0</td>
<td>30.26</td>
</tr>
<tr>
<td>C10</td>
<td>1365</td>
<td>0.2</td>
<td>10</td>
<td>647</td>
<td>2</td>
<td>30.26</td>
</tr>
<tr>
<td>C20</td>
<td>1365</td>
<td>0.2</td>
<td>20</td>
<td>647</td>
<td>2</td>
<td>30.26</td>
</tr>
</tbody>
</table>

Due to the volume of the most suitable mixer available, three 2-inch cube and three 4-inch cylinder molds were used for each mixture. The specimens were covered with plastic after placement and consolidation. After 24 hours the specimens were demolded and placed in an environmental chamber with a relative humidity of 100% for 28 days. Prior to testing, the top and bottom of each cylinder was ground to ensure a flat surface for measurement of modulus of elasticity.

The cubes were loaded in compression to failure at a load rate of 145 psi/s [42]. The cylinders were also loaded at 145 psi/s [42] until 40% of the corresponding compression load was reached. Three loading and unloading cycles were performed and the second two cycles were used to calculate the elastic modulus. The use of a 4x8-inch cylinder allowed the stiffness
to be measured using an MOE compressometer ring for accurate results. All testing was performed in a servohydraulic testing machine using a linear variable differential transformer (LVDT) to measure the compression strain in the cylinder (Figure 4-6). This testing technique provided the best repeatability and accuracy.

![Figure 4-6: Bulk concrete compressive strength and elastic modulus testing setup in servohydraulic testing machine for a) compression testing of cubes and b) MOE testing of cylinders using a compressometer ring](image)

4.2.2. Phase Volume Fraction Inputs

To determine the volume fractions of hardened cement paste phases in the experimentally, an x-ray diffraction (XRD) analysis was conducted on the paste samples after MOE testing was performed. XRD is commonly used in literature to qualitatively compare several concrete mixtures or quantitatively determine volume fractions of phases [43-46]. It is a non-destructive technique that directs x-rays towards a powder sample and then measures the scattered intensity as a function of direction (Figure 4-7).

One of the challenges with using the XRD for concrete is that crystalline structures often show the highest intensities while semi-crystalline and amorphous structures have lower
intensities. In concrete, some of the phases, like calcium hydroxide, are crystalline and well mapped with a known chemical structure, while others, like C-S-H are amorphous and the exact chemical structure is known to vary [47-48]. The differences in intensities between amorphous and crystalline structures are overcome by comparing to a standard material through a relative intensity ratio (RIR) for a more accurate quantification of the sample [48].

To prepare a sample for XRD analysis, the hardened cement paste is ground using a mortar and pestle until it passes through a number 325 sieve (0.0017-inch nominal opening). XRD testing occurs 7 days after the MOE results are obtained. The powder sample is placed in the XRD and scanned. For this investigation, a 2θ range of 10 to 70 was used with a step size of 0.01 and a speed of 1.5 seconds. This led to an overall scan time of 2 hours and 29 minutes per sample. The results were analyzed using X’Pert High Score software to determine the background, find peaks, and match peaks with corresponding cement paste phase from database ID cards. The RIR value for each phase was pulled from the database to determine the quantitative volume fraction of each phase.

Figure 4-7: Diagram of XRD analysis and sample results with select peaks identified
4.3. Results and Discussion

4.3.1. Experimental inputs and verification

The compressive strength and modulus of elasticity results for the 2-inch cubes and 4-inch diameter cylinders, respectively, are provided in Table 4-3. Each result is an average of six cubes or cylinders, mixed and tested on two separate days. As anticipated, P10 and P20 exhibit the lowest stiffness; the addition of fibers and aggregate increases the elastic modulus.

In the specimens with a 20% replacement of portland cement with silica fume, a 24% increase in stiffness is observed between the hardened paste (P20) and UHPC (C20) samples. A similar increase in stiffness is seen between the specimens with a 10% replacement of portland cement with silica fume. The addition of fine aggregate to the paste mix (specimen M20) increases the elastic modulus by 17%. The results from the experimental testing are used as verification of the developed code. Fresh concrete air content tests indicate that the void volume content for the mixes range between 4 and 6 percent.

Table 4-3: Experimental results for concrete cubes and cylinders tested for compressive strength and elastic modulus respectively

<table>
<thead>
<tr>
<th>Mix</th>
<th>$f\prime c$ MPa [ksi]</th>
<th>E GPa [ksi]</th>
</tr>
</thead>
<tbody>
<tr>
<td>P10</td>
<td>83.2 [12.1]</td>
<td>30.8 [4.5]</td>
</tr>
<tr>
<td>P20</td>
<td>93.9 [13.6]</td>
<td>30.9 [4.5]</td>
</tr>
<tr>
<td>M20</td>
<td>110.2 [16.0]</td>
<td>36.8 [5.3]</td>
</tr>
<tr>
<td>C10</td>
<td>134.4 [19.4]</td>
<td>38.8 [5.6]</td>
</tr>
<tr>
<td>C20</td>
<td>155.1 [22.5]</td>
<td>40.6 [5.9]</td>
</tr>
</tbody>
</table>

The automated programming requires the input of phase volume fractions for the level two cement paste upscaling. Pieces of P10 and P20 were prepared for analysis with the XRD; results are provided in Table 4-4. Two scans with concrete taken from different areas are
averaged together for the results. The first scan was obtained 35 days after mixing and the second scan at 50 days.

Table 4-4: XRD phase volume fraction results

<table>
<thead>
<tr>
<th>Phase</th>
<th>Volume Fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>P10 Mix</td>
</tr>
<tr>
<td>AFt/AFm</td>
<td>10.5</td>
</tr>
<tr>
<td>C-A-S-H</td>
<td>7.0</td>
</tr>
<tr>
<td>CH</td>
<td>4.5</td>
</tr>
<tr>
<td>CH/C-S-H</td>
<td>2.5</td>
</tr>
<tr>
<td>C-S-H</td>
<td>67.0</td>
</tr>
<tr>
<td>SF</td>
<td>8.5</td>
</tr>
<tr>
<td>Total:</td>
<td>100.0</td>
</tr>
</tbody>
</table>

4.3.2. Level Two Upscaling Results

The phase volume fractions determined from the XRD analysis of the two paste mixes are used as inputs into the spring arrangement simulation code. The code is compared to the cylinder elastic modulus, which contains larger air voids than considered in a level two upscaling. Level two typically focuses on constituent elements smaller than 100 μm, which does not account for the size of air voids found in a hardened cement paste cylinder. However, as the experimental verification comes from a bulk hardened paste, the cylinder fresh air content is used.

The results for 3-7% void contents for cutoff A (stiffness values less than 100 GPa) is shown in Figure 4-8. For visual comparison, the cylinder experimental results are also shown as solid red line. Without removing any simulations, outputs from using the P10 and P20 XRD data show a level two upscaled elastic modulus of $43.3 \pm 40.7$ GPa and $41.4 \pm 38.7$ GPa respectively. By increasing the number of data points labeled as outliers in the cement paste homogenization (cutoff A), the elastic modulus and standard deviation decrease to $28.4 \pm 11.4$ GPa (Figure 4-8a) and $29.7 \pm 11.8$ GPa (Figure 4-8b). The elastic moduli removed are primarily from simulations...
where the maximum number of springs (100) are combined in a single group in parallel. However, not every grouping larger than 90 springs results in an unreasonably high value.

![Graph showing elastic modulus vs voids for cutoff A](image)

**Figure 4-8:** Code upscaled elastic modulus for cutoff A (typical distribution along 5% voids line) and in a) P20 and b) P10; Comparison to experimental results from cement paste cylinders

The spread of elastic modulus values generated while using cutoff A is shown in a probability density plot for the 5% void volume content. Adjusting the void volume fraction has a minimal impact on the standard deviation and spread of the simulation data. Therefore, the
curve shown is representative of the results for each void volume fraction. The experimental value lies within one standard deviation for the paste mixtures. Both the P10 and P20 graphs show good agreement between the code output and the experimental testing.

Cutoff A has an average error of around 8.3% (P20 mix) and 3.8% (P10 mix) error for the level two upscaling. As a result, spring arrangement simulations likely provide a fair estimation of cement paste elastic moduli and may be used as part of a design tool. Before the code can be relied upon, however, comparisons to other mixes with varying microstructures must be evaluated. The two paste mixes used to verify the code in this study ended up having similar microstructures and experimental results. A significantly different mix (such as the use of fly ash or metakaolin) should be compared in the future.

4.3.3. Level Three Upscaling Results

The code results for 3-7% void contents for cutoff B is shown in Figure 4-9 along with the measured experimental results. The probability plot for the simulations corresponding to the refinement is drawn along the 5% void volume fraction line. Again, the standard deviations and density curve are representative of each void volume fraction for that cutoff. Unlike with the cement paste upscaling, the experimental elastic moduli values do not lie within one standard deviation of the mean programming output.

As for level two upscaling, reducing the number of simulations by removing those considered to be outliers decreases the level three computational elastic modulus. A 47% decrease (from 148.7 ± 139.0 GPa to 78.9 ± 31.4 GPa) is observed in C20 (Figure 4-9a) when there is no cutoff versus when Cutoff B is used, respectively. The factors that lead to a high elastic modulus output for level 3 upscaling are not easily distinguished. Neither the presence of fiber springs nor the absence or presence of microcrack springs consistently led to an
unreasonably high simulation value. The group sizes in parallel similarly showed no notable impact. In its current form, the code seeks to uphold randomness in representing UHPC homogenizations. Adjustments to the approach of the spring arrangement simulations may create a code that fails to maintain randomness.

**Figure 4-9:** Code upscaled elastic modulus for cutoff B (typical distribution along 5% void line) in a) C20 and b) C10; Comparison to experimental results from UHPC cylinders
There was not as good agreement between experimental and model results when evaluating the code response for UHPC upscaling (combining 2% volume fraction of fibers, fine aggregate, cement paste, and micro-cracking parameter springs) compared to the cement paste model. Not only is the average error between the code outputs and experimental results over 100% (Figure 4-9), but the experimental results are also outside of the standard deviations. Only looking at results lower than cutoff B reduces the error from 265% to 108% when looking at the C20 mix but still provides a significant overestimation of the elastic modulus. As a result, the proposed spring arrangement simulations are not an adequate approach for upscaling of level three.

When a 0% fiber volume fraction is used, the computational modulus should be reduced by around 10% if following the experimental results (where \( E = 36.8 \) GPa for M20 and \( E = 40.6 \) GPa for C20). As the code output for mortar is reduced by 7.0%, indicating that the fiber stiffness may be accurately or close to accurately incorporated through the spring arrangement simulations. However, other homogenization schemes have shown the importance of fiber orientation [49], which may not be possible to account for in this developed code.

The mortar mix elastic modulus is also overestimated by the code (114%), showing that not even the combination of fine aggregate and cement paste can be accurately portrayed by the current spring arrangement simulations. However, the code does not account for a weaker interfacial transition zone (ITZ). As the elastic modulus of concrete is largely affected by the localized damage in the ITZ and the aggregate modulus [50-53], consideration of an ITZ decreases the error between the code and the experimental results. UHPC was chosen as the comparative mix for this initial code development as it is known to have a stronger ITZ than normal strength concrete mixes [54-55]. Additionally, the use of silica fume is known to reduce
the size of the ITZ more than other supplementary cementitious materials like fly ash and metakaolin [56-57]. Sorelli et al. reported an almost negligible ITZ zone around the fibers in their mix [58] and Petranova et al. observed no ITZ in their evaluation of UHPC using nanoindentation [17]. However, the size and strength of the ITZ around the fibers and aggregate are not evaluated for this concrete mix.

Without knowing the appropriate volume fraction and strength of the ITZ, it is not possible to completely integrate an ITZ parameter. A brief assessment with 45% of the cement paste volume fraction being considered an ITZ phase with a strength 40% less than the cement paste from the level two upscaling was conducted. This reduced the level three upscaled stiffness to 71.3 ± 29.3 GPa. This estimation continues to overestimate the elastic modulus of UHPC and fails to predict the experimental stiffness within one standard deviation. An increase of the micro-cracking volume fraction to 10% similarly failed to provide a reasonable comparison for the experimental results.

4.4. Conclusions

Spring arrangement simulations are used in a two-part upscaling scheme to upscale micro-mechanical properties of UHPC, verified through experimental testing. The results from this novel approach led to the following conclusions.

1. Monte Carlo simulations combining phase stiffnesses as springs in random groups of series and parallel are an effective way to maintain the randomness found in the microstructure of concrete.

2. Code convergence occurs when more than 10,000 simulations are run in this study.

3. Based on an initial evaluation, spring arrangement simulations can be used to determine the level 2 homogenization of cement paste. After refining the simulation results
averaged, the code estimates the elastic modulus within a 10% error of experimental data. For the P10 mix when 88% of the 100,000 simulations are averaged, the code predicts an elastic modulus within 4% of the measured experimental value.

4. For level 3 upscaling, the code fails to predict the elastic modulus within a reasonable error, indicating that spring arrangement simulations may not be an appropriate upscaling approach. The C20 code output produces a homogeneous elastic modulus value 108% higher than the measured results (when 11% of the simulations are considered outliers).

5. The spring arrangement simulations may fail to capture the effect of fibers and aggregate on concrete stiffness. The ITZ is not considered in the current spring arrangement simulations and should be evaluated in the future.

4.5. References

[1] ACI Committee 318, "Building Code Requirements for Structural Concrete (ACI 318-14)," American Concrete Institute, Farmington Hills, MI, 2014.


Chapter 5. Summary, Conclusions, Contributions, and Future Work

5.1. Summary and Conclusions

Design code equations predicting the elastic modulus of certain specialty concrete likes fiber-reinforced, ultra-high performance, and eco-concretes are not suitable. The presence of fibers and mixture adjustments from cement and aggregate replacements make it challenging for any empirical curve to accurately represent a variety of mixes consistently. New equations and improvements to existing equations have been made with varying levels of success, but with the number of factors affecting the elastic modulus an alternative approach would likely be more effective. Rather than relying heavily on the bulk compressive strength of concrete, the underlying mechanisms that control the elastic modulus need to be understood.

In this study, a micro-mechanical investigation sought to provide a better understanding of the elastic modulus of cement paste phases to inform a homogenization attempt. Six phases were identified using EDX and their compressive strength and elastic modulus were evaluated by fabricating and testing micropillars. Monotonic loading was used to map both the linear and nonlinear behavior of these phases. Micropillars primarily failed in partial or complete crushing, apart from AFt/AFm which failed in axial splitting. AFt/AFm additionally exhibited the highest elastic modulus as 41.7 GPa, while C-S-H, C-A-S-H, and SF micropillars were between 9 and 13 GPa. It was difficult to obtain consistent results for micro-pillars made from CH and a CH/C-S-H composite, potentially due to differences in orientations of CH crystals within the micropillar.

As micro-compression testing is a relatively novel methodology for concrete, additional testing was conducted using nanoindentation. Nanoindentation has been popular in literature for decades, but the results from this methodology may be influenced by testing conditions and...
underlying assumptions. Comparing the two approaches showed that nanoindentation tends to overestimate the stiffness of cement paste phases when compared to micro-compression data.

With key cement paste phases identified and their behavior understood, a novel two-step homogenization approach using spring arrangement simulations was attempted. Results from the code were compared with experimental testing of two different paste and UHPC mixes. When running 100,000 simulations, about 0.5% of the simulations were unquestionably outliers. However, the spread of data created a bimodal probability density plot and indicated that it may be worth analyzing the removal of about 12% of the simulations (cutoff A). Cutoff A provided an error of 4% and 9% compared to experimental testing of two hardened cement paste cylinder mixes. From these results, it can be concluded that spring arrangement simulations may provide a reasonable approach for homogenization of cement paste.

However, when the spring arrangement simulations were used to upscale the computational cement paste, fine aggregate, and fibers, the results were less positive. The code wildly overpredicted the elastic modulus of UHPC and a brief investigation of potential reasons (such as not considering the ITZ) failed to account for most of the overestimation. Therefore, currently it is not recommended to pursue spring arrangement simulations as a homogenization method when seeking to combine cement paste, aggregates, fibers, etc. (level three).

5.2. Contributions

The original contributions from this study are as follows:

1) Existing literature for elastic modulus prediction of specialty concretes are analyzed and summarized.

2) Protocol for fabrication and compression testing of micropillars using a FIB and PI-88 is developed.
3) Statistical relationships for the loading stiffness and compressive strength of phases within cement paste were developed. A comparison to nanoindentation results is conducted.

4) A basis for a future design tool to manipulate and/or predict the elastic modulus of concrete is provided. A novel upscaling methodology for cement paste homogenization was developed and preliminarily evaluated with two UHPC mixes.

5.3. Future Work

Recommendations for future research can be made based on the results in this study. There may be a benefit to continuing micro-compression testing of phases within the cement paste. This study found a decent spread in stiffness and compressive strength data for CH and CH/C-S-H composite phases. By increasing the number of micropillars tested, the mechanical properties can be better understood, and outliers identified. Additionally, micropillars should be fabricated on clinker and be used to investigate the size and stiffness of UHPC ITZ.

Regarding the cement paste homogenization, a formal addition of the ITZ to the level three code should be made once the properties are determined. A deeper investigation into what causes the overestimation of the UHPC upscaling may be beneficial, particularly if proper implementation of ITZ springs reduces the error significantly. It should be noted, however, that an informal adjustment of the code indicated that the addition of the ITZ may not make as big of an impact as necessary. In this case, alternative methods for level three homogenization (such as RVEs/SVEs) may be a more effective approach. The cement paste homogenization using spring arrangement simulations has potential, though, and should be experimentally verified through a large variety of concrete mixes. Future work should focus on determining if the code results are within an acceptable error even with large adjustments to cement and aggregate replacements.
### Appendix A. Micropillar Properties

<table>
<thead>
<tr>
<th>Pillar No.</th>
<th>D1 (μm)</th>
<th>D2 (μm)</th>
<th>H (μm)</th>
<th>Aspect Ratio</th>
<th>Taper angle (˚)</th>
<th>Phase</th>
<th>Ca/Si</th>
<th>fc (MPa)</th>
<th>E (GPa)</th>
<th>Failure Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1-8</td>
<td>1.50</td>
<td>1.91</td>
<td>2.94</td>
<td>1.7</td>
<td>4.0</td>
<td>AFt/AFm</td>
<td></td>
<td>7.32</td>
<td>2153</td>
<td>32.8 0.065</td>
</tr>
<tr>
<td>A2-29</td>
<td>1.35</td>
<td>2.15</td>
<td>3.51</td>
<td>2.0</td>
<td>6.5</td>
<td>AFt/AFm</td>
<td></td>
<td>22.53</td>
<td>2974</td>
<td>47.6 0.068</td>
</tr>
<tr>
<td>A2-30</td>
<td>1.42</td>
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<td>2.2</td>
<td>4.2</td>
<td>AFt/AFm</td>
<td></td>
<td>24.34</td>
<td>3850</td>
<td>50.9 0.080</td>
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<td>3.3</td>
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<td>2392</td>
<td>35.3 0.068</td>
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<td>A1-11</td>
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<td>1.93</td>
<td>3.70</td>
<td>2.2</td>
<td>4.3</td>
<td>C-A-S-H</td>
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<td>697</td>
<td>13.1</td>
<td>0.108</td>
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<td>1.97</td>
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<td>1.81</td>
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<td>4.1</td>
<td>C-A-S-H</td>
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<td>A2-22</td>
<td>1.46</td>
<td>2.00</td>
<td>2.77</td>
<td>1.6</td>
<td>5.6</td>
<td>CH/C-S-H</td>
<td>2.62</td>
<td>3245</td>
<td>13.5</td>
<td>0.137</td>
</tr>
<tr>
<td>A2-24</td>
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<td>2.20</td>
<td>3.35</td>
<td>1.9</td>
<td>6.8</td>
<td>CH/C-S-H</td>
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<td>2382</td>
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<td>A2-27</td>
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<td>3.8</td>
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<td>2.06</td>
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<td>1179</td>
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<td>A2-40</td>
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<td>2.22</td>
<td>5.00</td>
<td>2.6</td>
<td>3.2</td>
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<td>CH/C-S-H</td>
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<td>4.11</td>
<td>1.7</td>
<td>7.3</td>
<td>CH/C-S-H</td>
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<td>1.7</td>
<td>4.9</td>
<td>CH/C-S-H</td>
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</table>
Appendix B. MATLAB Homogenization Code

% Input Vf of phases (in percent)
Vf_AFT=9.5;
Vf_CASH=30.9;
Vf_CH=3.4;
Vf_CHCSH=3.5;
Vf_CSH=45.2;
Vf_Sc=7.5;

% Input Fibers Info
Vf_Fib=0; % percent of total
E_Fib=210; % GPa

% Input details of Fine Agg and Concrete
E_FAgg=60; % GPa
Density_FAgg=2.639*1000; % SG*1000 kg/m^3
Weight_FAgg=4.18; % kg
Vol_Concrete=0.0064; % m^3 or 0.226ft^3

% Void and Microcracking
Vf_Void=5; % percent of total
Vf_MCrack=1; % percent of total

%------------------------------------------------------------------------------------------------
% Calculate mean and std dev for each phase
CSH_Data = [6.9; 13.8; 9.1; 10.3; 11.3; 12.8; 9.9];
mean_CSH=mean(CSH_Data);
std_CSH=std(CSH_Data);

AFT_Data=[32.8;47.6;50.9;35.3];
mean_AFT=mean(AFT_Data);
std_AFT=std(AFT_Data);

CASH_Data=[7.9;13.1;9.5;8.8;7.8];
mean_CASH=mean(CASH_Data);
std_CASH=std(CASH_Data);

CHCSH_Data=[13.5;25;6.6;36.4;5.6;27.1;12.8;45.9];
mean_CHCSH=mean(CHCSH_Data);
std_CHCSH=std(CHCSH_Data);

CH_Data=[27.4;12.2];
mean_CH=mean(CH_Data);
std_CH=std(CH_Data);
SF_Data=[15.3;9.3;19;12.1;7.8;8.6;13.;12;13.1];
mean_SF=mean(SF_Data);
std_SF=std(SF_Data);

E_Void=0.00001; %GPa
E_MCrack=0.0001; %GPa

Amt_CSH=round(Vf_CSH*(1-Vf_Void/100)*10);
Amt_AFT=round(Vf_AFT*(1-Vf_Void/100)*10);
Amt_CASH=round(Vf_CASH*(1-Vf_Void/100)*10);
Amt_CHCSH=round(Vf_CHCSH*(1-Vf_Void/100)*10);
Amt_CH=round(Vf_CH*(1-Vf_Void/100)*10);
Amt_SF=round(Vf_SF*(1-Vf_Void/100)*10);

Vol_FAgg=Weight_FAgg/Density_FAgg; %m^3
Vf_FAgg=Vol_FAgg/Vol_Concrete*100;
Vf_CemPaste=100-Vf_Fib-Vf_FAgg-Vf_MCrack;

number_of_runsA=100000;

for q=1:number_of_runsA
    Num_AFT=(mean_AFT+std_AFT)+((mean_AFT-std_AFT)-(mean_AFT+std_AFT)).*rand(Amt_AFT,1);
    Num_CASH=(mean_CASH+std_CASH)+((mean_CASH-std_CASH)-(mean_CASH+std_CASH)).*rand(Amt_CASH,1);
    Num_CH=(mean_CH+std_CH)+((mean_CH-std_CH)-(mean_CH+std_CH)).*rand(Amt_CH,1);
end
Num_CHCSH=(mean_CHCSH+std_CHCSH)+((mean_CHCSH-std_CHCSH)-(mean_CHCSH+std_CHCSH)).*rand(Amt_CHCSH,1);
Num_CSH=(mean_CSH+std_CSH)+((mean_CSH-std_CSH)-(mean_CSH+std_CSH)).*rand(Amt_CSH,1);
Num_SF=(mean_SF+std_SF)+((mean_SF-std_SF)-(mean_SF+std_SF)).*rand(Amt_SF,1);
Num_Void=E_Void*1.05+(E_Void*0.95-E_Void*1.05).*rand(Amt_Void,1);

k=[Num_AFT;Num_CASH;Num_CH;Num_CHCSH;Num_CSH;Num_SF;Num_Void];

for o=1:11
    P=[randsample(k,90)];
    D=P;

    %Randomly create groups of springs in parallel
    number_of_runsB=1000;
    K=zeros(1,number_of_runsB);
    U=zeros(1,number_of_runsB);

    for n=1:number_of_runsB
        h=numel(D);
        j=1;

        while h > 6
            i=randi([1,h],1,1); %/(K1+...+Kf)
            c=[randsample(D,i)]; %Choose i values from the 10000 E values generated
            U(j)=1/sum(c)); %Group f values in parallel to be added in series with next iteration
            D=setdiff(D,c); %remove values from P and continue
            h=numel(D);
            j=j+1;
        end

        if numel(D)>0
            V=1/sum(D);
        else
            V=0;
        end

        K(n)=(sum(U)+V)^(-1); %Sum while loop and final parallel grouping; store in K matrix
    end

    %Average B number of iterations of spring combination
K_Avg_CemP(o)=mean(K);
K_Stdev_CemP(o)=std(K);

k=setdiff(k,P);

%Average A iterations of choosing values for springs and combining them
E_Avg_CemP(q)=mean(K_Avg_CemP);
E_Stdev_CemP(q)=std(K_Avg_CemP);

end

inRange=E_Avg_CemP<200;
L=E_Avg_CemP(inRange);
Avg_Stdev_E_CemP=std(E_Avg_CemP(inRange));
Avg_E_CemP=mean(E_Avg_CemP(inRange));

%%

%------------------------------------------------------------------------------------------------------------------------
%Homogenize fibers and cement paste

number_of_runsC=100000;

E_Avg_UHPC=zeros(1,number_of_runsC);
E_Stdev_UHPC=zeros(1,number_of_runsC);

M_Avg_UHPC=zeros(1,10);
M_Stdev_UHPC=zeros(1,10);

for u=1:number_of_runsC
    Num_CemP=(Avg_E_CemP+(0.5*Avg_Stdev_E_CemP)) + ((Avg_E_CemP-(0.5*Avg_Stdev_E_CemP)) - (Avg_E_CemP+(0.5*Avg_Stdev_E_CemP))).*rand(Amt_CemPaste,1);
    Num_FAgg=E_FAgg*1.1+(E_FAgg*0.9-E_FAgg*1.1).*rand(Amt_FAgg,1);
    Num_Fib=E_Fib*1.001+(E_Fib*0.999-E_Fib*1.001).*rand(Amt_Fib,1);
    Num_MCrack=E_MCrack*1.01+(E_MCrack*0.99-E_MCrack*1.01).*rand(Amt_MCrack,1);

    m=[Num_CemP;Num_FAgg;Num_Fib;Num_MCrack];

    for v=1:10
N=[randsample(m,95)];
A=N;

%--------------------------------------------------------------

% Randomly create groups of springs in parallel
number_of_runsD=1000;
M=zeros(1,number_of_runsD);
W=zeros(1,number_of_runsD);

for p=1:number_of_runsD
    g=numel(N);
e=1;

    while g > 6
        f=randi([1,g],1,1); %(1/(K1+...+Kf))
d=[randsample(N,f)]; %Choose f values from the 100 E values generated
    W(e)=1/sum(d)); %Group f values in parallel to be added in series with next iteration
    N=setdiff(N,d); %remove values from N and continue
    g=numel(N);
e=e+1;
end

    if numel(N)>0
        Y=1/sum(N);
    else
        Y=0;
    end

    M(p)=(sum(W)+Y)^(-1); %Sum while loop and final parallel grouping; store in K matrix
end

%M_Avg_UHPC(v)=mean(M);
%M_Stdev_UHPC(v)=std(M);

m=setdiff(m,A);

% Average A iterations of choosing values for springs and combining them
E_Avg_UHPC(u)=mean(M_Avg_UHPC);
E_Stdev_UHPC(u)=std(M_Avg_UHPC);
end

inRange=E_Avg_UHPC<700;
l=E_Avg_UHPC(inRange);
Avg_E_UHPC=mean(E_Avg_UHPC(inRange));
Avg_Stdev_E_UHPC=std(E_Avg_UHPC(inRange));