Material Characterization of Ultra-High Performance Fiber Reinforced Concrete at Elevated Temperatures

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Material Characterization of Ultra-High Performance Fiber Reinforced Concrete at Elevated Temperatures

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Bachelors of Science in Civil Engineering, University of Connecticut, 2010

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Master of Science Thesis

Material Characterization of Ultra-High Performance Fiber Reinforced Concrete at Elevated Temperatures

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# TABLE OF CONTENTS

Approval Page .................................................................................................................. ii
Acknowledgements ........................................................................................................... iii
Table of Contents .............................................................................................................. iv
List of Figures ..................................................................................................................... vi
List of Tables ...................................................................................................................... ix
Chapter One – Background ............................................................................................. 1
  1.1 Introduction .............................................................................................................. 1
  1.2 Literature Review .................................................................................................... 2
    1.2.1 Ultra-High Performance Fiber Reinforced Performance Concrete .......... 2
    1.2.1.1 Fiber Reinforced Concrete ............................................................. 2
    1.2.1.2 Ultra-High Performance Concrete .............................................. 7
    1.2.2 Chemical Change of Concrete at Elevated Temperature ....................... 14
    1.2.3 Mechanical Properties of High Strength Concrete under Elevated Temperatures .................................................................................................................. 17
    1.2.4 Mechanical Properties of Ultra-High Strength Concrete at Elevated Temperatures .................................................................................................................. 22
    1.2.5 Thermal Properties of Normal Strength Concrete and High Strength Concrete ....................................................................................................................... 26
    1.2.6 Thermal Properties of Ultra-High Performance Concrete ....................... 29
    1.2.7 Discussions and Conclusions ........................................................................... 29
  1.3 Report Structure ...................................................................................................... 30
Chapter Two – Materials and Methods ........................................................................... 31
  2.1 Introduction .............................................................................................................. 31
  2.2 Materials ................................................................................................................ 31
    2.2.1 Ductal AF ................................................................................................. 32
    2.2.2 House Mix ............................................................................................... 33
  2.3 Methods .................................................................................................................. 34
    2.3.1 Mixing Procedure .................................................................................... 34
      2.3.1.1 Ductal Mixing Procedure ......................................................... 35
      2.3.1.2 House Mixing Procedure ......................................................... 38
    2.3.2 Casting Process ....................................................................................... 40
    2.3.3 Specimen Preparation ............................................................................. 41
  2.4 Heat Testing ............................................................................................................ 46
  2.5 Compressive Strength and Elastic Modulus Tests .................................................. 49
  2.6 DSC/TGA .............................................................................................................. 51
LIST OF FIGURES

Figure 1.1 – Early Patents Submitted by R. D. Weakly and Meischke-Smith.......................3
Figure 1.2 – Pull Out Load for Deformed Wire vs. Smooth Wire (left) and Hooked Fibers vs. Smooth Fibers (right).................................................................5
Figure 1.3 – Comparison of Typical Stress–Strain Response in Tension of HPFRCC with Conventional FRCC.................................................................6
Figure 1.4 – Void Left by Melting Polypropylene Fibers in HSC Matrix .........................7
Figure 1.5 – Schematic of Packing Model of UHPC ...........................................................9
Figure 1.6 – Typical Makeup of UHPC in Comparison to Normal Strength Concrete ......9
Figure 1.7 – Grading Curves of Raw Materials and Mixture.............................................10
Figure 1.8 – Pozzolanic Reaction with Silica Fume ..........................................................11
Figure 1.9 – Plot of Pore Pressure vs. Tensile Strength as a Function of Temperature .....18
Figure 1.10 – Spalling Mechanisms .................................................................................19
Figure 1.11 – Previous Research on High Performance/ High Strength Concrete vs. Temperature ........................................................................................................21
Figure 1.12 – Explosive Spalling in Concrete Due to Fire Exposure ...............................23
Figure 1.13 – Collective Results on Compressive Strength vs. Temperature from Various UHPFRC’s .........................................................................................24
Figure 1.14 – Collective Results on Elastic Modulus vs. Temperature from Various UHPFRC’s .........................................................................................25
Figure 1.15 – Tensile Strength vs. Temperature for Hot and Residual Case .......................25
Figure 2.1 – Ductal Mix Fibers .........................................................................................34
Figure 2.2 – House Mix Fibers .......................................................................................34
Figure 2.3 – Eirich Adjustable Frequency High Shear Mixer ............................................35
Figure 2.4 – Ductal Mix Series – Mix with Water Added ..................................................37
Figure 2.5 – Ductal Mix Series – Fluid Mix ........................................................................37
Figure 2.6 – Ductal Mix Series – Fibers Added to Fluid Mix ...........................................37
Figure 2.7 – House Mix Series – Fume and Sands Premixing ..........................................39
Figure 2.8 – House Mix Series – Premix with Cement and Silica Powder Added ............39
Figure 2.9 – House Mix Series – Mix with Water Added ................................................39
Figure 2.10 – House Mix Series – Turn over ....................................................................39
Figure 2.11 – House Mix Series – Fluid Mix ....................................................................40
Figure 2.12 – House Mix Series – Fluid Mix with Fibers Added ......................................40
Figure 2.13 – Steam Box .................................................................................................41
Figure 2.14 – Masonry Wet Saw .....................................................................................42
Figure 2.15 – Cylinder End Grind with Lid Opened ..........................................................43
Figure 2.16 – Fine Cylinder Grinder ................................................................................44
Figure 2.17 – Dialitometer ...............................................................................................44
Figure 2.18 – Specimen Stages - Raw (left), Saw Cut (middle left), End Grinder Ground (middle right), Finished Specimen (right) ..........................................45
Figure 2.19 – Fully prepared and Finished Specimen ..................................................45
Figure 2.20 – Preparation of DSC/TGA Specimens ..........................................................46
Figure 2.21 – Thermocouple Placement in Dummy Specimen ..........................................47
Figure 2.22 – Fisher Scientific Isotemp Oven Model 630G – 200ºC Drying Oven ............48
Figure 2.22 – Fisher Scientific Isotemp Muffle Furnace 1125ºC ........................................48
Figure 2.24 – Metal Cage Used for Fast Heating Trials ...................................................49
Figure 2.25 – SATEC 1780kN Load Frame with MTS Controller ....................................50
Figure 2.26 – LVDTs in Holder for Strain Calculation ......................................................51
Figure 2.27 – Netzsch STA 449 F3 Jupiter Simultaneous TGA/DSC ..................................52
Figure 3.1 – Metal Cage and Concrete Debris in Oven ...................................................54
Figure 3.2 – Intact and Pulverized Specimens .................................................................54
Figure 3.3 – Destroyed Concrete Specimen from 300ºC HF Series ..................................55
Figure 3.4 – HF Series Color Change with Temperature of Exposure (Left to Right: 90ºC, 135ºC, 200ºC, 300ºC, 400ºC, 500ºC, 600ºC, 700ºC) ........................................57
Figure 3.5 – Dark Failed Surface of 600ºC DS Specimen ................................................57
Figure 3.6 – DS Series Color Change with Temperature of Exposure (Left to Right: 90ºC, 135ºC, 200ºC, 300ºC, 400ºC, 500ºC, 600ºC, 700ºC, 800ºC, 900ºC) ..................................58
Figure 3.7 – Sample Stress vs. Strain Curve Showing Pre-Elastic Hardening ....................61
Figure 3.8 – Sample Strain Corrected Stress-Strain Curve for Corrected Strains ..........62
Figure 3.9 – Strain Normalized Average Stress vs. Strain Curve with Individual Series Included .................................................................................................................64
Figure 3.10 – Average Residual Stress-Strain Curves for DS Series without Adjusted Strains ..................................................................................................................67
Figure 3.11 – Average Residual Stress-Strain Curves for DS Series with Adjusted Strains ..................................................................................................................68
Figure 3.12 – Comparison between Model Code and 400ºC DS Strain Adjusted Series ..................................................................................................................68
Figure 3.13 – Average Residual Stress-Strain Curves for HS Series without Adjusted Strains ..................................................................................................................70
Figure 3.14 – Average Residual Stress-Strain Curves for HS Series with Adjusted Strains ..................................................................................................................70
Figure 3.15 – Comparison between Model Code and 400ºC HS Strain Adjusted Series ..................................................................................................................71
Figure 3.16 – Average Residual Stress-Strain Curves for HF Series without Adjusted Strains ..................................................................................................................72
Figure 3.17 – Average Residual Stress-Strain Curves for HF Series with Adjusted Strains ..................................................................................................................72
Figure 3.18 – Comparison between Model Code and 300ºC HF Strain Adjusted Series
Figure 3.19 – Residual Compressive Strength of DS Series with Increasing Temperature
Figure 3.20 – Residual Compressive Strength of HS Series with Increasing Temperature
Figure 3.21 – Residual Compressive Strength of HF Series with Increasing Temperature
Figure 3.22 – Spalling on the Ends of 300ºC HF Series
Figure 3.23 – Residual Compressive Strength of HF Series vs. HS Series
Figure 3.24 – Residual Compressive Strength of HF Series vs. HS Series
Figure 3.25 – Experimental Results vs. Existing Literature on UHPC Compressive Strength at High Temperatures based on Graph in [107]
Figure 3.26 – Experimental Results vs. Existing Literature on UHPC, HSC, and Normal Strength Concrete for Compressive Strength at High Temperatures based on Graph in [106]
Figure 3.27 – Experimental Results vs. Equation 3.5 Predictions
Figure 3.28 – Equation 3.5 Predictions vs. Existing Literature on UHPC Compressive Strength at High Temperatures based on Graph in [107]
Figure 3.29 – Residual Elastic Modulus of DS Series with Increasing Temperature
Figure 3.30 – Residual Elastic Modulus of HS Series with Increasing Temperature
Figure 3.31 – Residual Elastic Modulus of HF Series with Increasing Temperature
Figure 3.32 – Residual Elastic Modulus of HF Series vs. HS Series
Figure 3.33 – Residual Elastic Modulus of DS Series vs. HS Series
Figure 3.34 – Experimental Results vs. Existing Literature on UHPC Elastic Modulus at High Temperatures based on Graph in [107]
Figure 3.35 – Experimental Results vs. Existing Literature on Normal and High Strength Concrete Elastic Modulus at High Temperatures based on Graph in [133]
Figure 3.36 – Experimental Results vs. Equation 3.6 Predictions
Figure 3.37 – Equation 3.6 Predictions vs. Existing Literature on UHPC Compressive Strength at High Temperatures based on Graph in [107]
Figure 3.38 – Compressive Strains vs. Temperature
Figure 3.39 – Model Code with Predicted Values for Control HS Series
Figure 3.40 – Model Code with Predicted Values for 300ºC HF Series
Figure 3.41 – Model Code with Predicted Values for 800ºC DS Series
Figure 3.42 – Thermo Gravimetric Analysis: Stepwise Mass Loss of Ductal Mix
Figure 3.43 – Differential Scanning Calorimetry Plot for Ductal Mix
Figure 3.44 – Thermo Gravimetric Analysis: Stepwise Mass loss of House Mix
Figure 3.45 – Differential Scanning Calorimetry Plot for House mix
# LIST OF TABLES

Table 1.1 – Characteristic Durability Values for UHPC, HPC, Normal Strength Concrete .................................................................................................................................................................................. 8
Table 1.2 – Chemical Change in Concrete with Temperature ................................................................................................................................. 14-15
Table 2.1 – Ductal Manufacturer’s Supplied Material Characteristics ......................................................................................................................... 32
Table 2.2 – Ductal Mix Proportions ......................................................................................................................................................................................... 33
Table 2.3 – House Mix Proportions ....................................................................................................................................................................................... 34
Table 2.4 – Ductal Mixing Procedure .................................................................................................................................................................................. 36
Table 2.5 – House Mixing Procedure .............................................................................................................................................................................. 39
Table 2.6 – Process of End Preparation for Concrete Cylinders ............................................................................................................................ 42
Table 3.1 – Color Change in Concrete with Respect to Temperature of Exposure ........................................................................................................... 56
Table 3.2 – Color change of DS and HS series with Respect to Temperature ......................................................................................................... 56
Table 3.3 – DS Series Strain and Stress Values at Onset of Elastic Region .............................................................................................................. 66
Table 3.4 – HS Series Strain and Stress Values at Onset of Elastic Region ........................................................................................................... 69
Table 3.5 – HF Series Strain and Stress Values at Onset of Elastic Region ......................................................................................................... 71
Table 3.6 – Residual Compressive Strength Data from DS Series with Increasing Temperature ............................................................................................ 75
Table 3.7 – Residual Compressive Strength Data from HS Series with Increasing Temperature .............................................................................................. 77
Table 3.8 – Residual Compressive Strength Data from HF Series with Increasing Temperature .............................................................................................. 78-79
Table 3.9 – Residual Compressive Strength Data from HF Series vs. HS Series with Increasing Temperature ........................................................................ 80
Table 3.10 – Residual Compressive Strength Data from DS Series vs. HS Series with Increasing Temperature .................................................................................... 81
Table 3.11 – Residual Elastic Modulus Data from DS Series with Increasing Temperature ............................................................................................................................ 87
Table 3.12 – Residual Elastic Modulus Data from HS Series with Increasing Temperature ............................................................................................................................ 89
Table 3.13 – Residual Elastic Modulus Data from HF Series with Increasing Temperature ............................................................................................................................ 90
Table 3.14 – Residual Elastic Modulus Data from HF Series vs. HS Series with Increasing Temperature ............................................................................................................................ 91
Table 3.15 – Residual Elastic Modulus Data from DS Series vs. HS Series with Increasing Temperature ............................................................................................................................ 92
Table 3.16 – Compressive Strains for DS, HS, and HF Series ......................................................................................................................................................... 97
Chapter One – Background

1.1 Introduction

Ultra-High Performance Fiber Reinforced Concrete (UHPFRC) is a relatively new form of high strength, high performance concrete. UHPFRC utilizes an optimized particle size distribution to achieve a high density, low air content concrete capable of compressive strength in excess of 206MPa (30ksi). This high packing density results in low permeability and superior durability over normal and high strength concretes. While this concrete is typically very brittle, the addition of fibers allows for higher ductility and energy dissipation capacity.

While concrete is typically considered to be resistant against fire and high temperatures, the low permeability of UHPFRC may result in failure by explosive spalling at high temperatures. Explosive spalling is the process of concrete explosively breaking away as a result of elevated temperatures. Thermal spalling is widely accepted to be caused by the combination of two phenomena; first being the unequal thermal expansion between the concrete core and surface, and the second being the increase of vapor pressure caused by the evaporation of free and bound water trapped in the cementitious matrix. Spalling reduces the cross section area of critical sections and may expose conventional steel reinforcement to high temperatures. The use of steel and polypropylene fibers has shown to mitigate the problem of spalling at high temperatures.

The thermal resistance of concrete is complex with many factors needing consideration. The three main effects are the chemical degradation of the cementitious matrix, thermal gradients caused by rapid heating, and the vapor pressure effects causing spalling. These effects themselves are reliant on the tensile strength, permeability of the concrete, thermal conductivity,
thermal diffusivity, and coefficient of thermal expansion. The non-homogeneous nature of concrete makes these difficult parameters to measure. This is especially true as several of these are both temperature dependent and non-linear in nature.

The focus of this research is to investigate the residual mechanical properties of ultra-high performance fiber reinforced concretes at elevated temperatures. Heating will be performed using two heating regimes in order to separate the effects of chemical and mechanical degradation on the residual mechanical properties of the concrete. Chemical degradation is defined as the temperature induced phase change and dehydration of the constituent parts of the concrete. Mechanical degradation is defined as the damage sustained by concrete at high temperatures as a result of unequal thermal gradients and vapor pressure. The residual properties will be measured by heating the concrete samples to a prescribed temperature and then returning it to room temperature to perform compressive testing.

1.2 Literature Review

1.2.1 Ultra-High Performance Fiber Reinforced Performance Concrete

Ultra-high performance fiber reinforced concretes are the latest advancement in high strength and high performance concretes. These composites utilize a low water to binder ratio and an engineered packing density to achieve improved strength and durability over high and ultra-high strength concretes. This composite is the joining of two technologies; fiber reinforced concrete and ultra-high performance concrete.

1.2.1.1 Fiber Reinforced Concrete

Fiber reinforced concretes consist of many of the same constituent parts as normal and high strength concrete, however they also contain a percentage by mass or volume of fibers.
These fibers may be any combination of steel, polypropylene, glass, carbon, or organic fibers. They can be deformed in order to gain better adhesion to concrete or they can be straight. Fibers have been used as reinforcement for thousands of years beginning with straw or horsehair being added to sunbaked bricks in ancient Egypt. [1-2] However, the first patent for the modern style of fiber reinforced concrete was issued in 1874 to A. Berard. His patent called for irregularly shaped pieces of iron to be added to the concrete mix as an artificial stone. [3]

Between 1874 and the late 1940’s, numerous other patents were filed for various types of fiber reinforcement in concrete. These would range from hoops of wire interconnected to increase cohesion [4], to short distorted wires added to the mix. [4-6] Two examples of these patents are shown in Figure 1.1. The first modern fiber reinforcement was patented in 1943 by G. Constantinesco. The fibers described in the patent had a wide range of shapes and lengths. Like his predecessors, Constantinesco used the wires to increase the crack resistance and energy absorption. He suggested these fibers be used to reinforce structures subjected to large dynamic loads such as army tanks and air raid bunkers. [4,6]

![Figure 1.1 – Early Patents Submitted by R. D. Weakly and Meischke-Smith](image-url)
By the early 1970’s, fiber reinforced concrete had come into use in construction. The first fiber reinforced structures had been built and a wide variety of fibers types were being used and researched including steel, polypropylene, nylon, glass, carbon, and organic fibers, etc. [2,4,6] The development of new fibers in conjunction with increases in concrete strength and durability has led to fiber reinforcements being used to enhance the brittle structure of high and ultra-high strength concrete matrices. [6-10] The addition of steel fibers is also used to increase the fractural toughness and energy dissipation capacity of the concrete. This makes fiber reinforced composites good for seismic, blast or projectile loading. [11-14] Additionally, polypropylene fibers have been used in conjunction with steel fibers to produce composites which are more resistant to fire damage. This is type of fiber reinforcement is particularly necessary for very dense concretes. [15-19]

Fibers as a concrete reinforcement obtain their strength from several areas; physical/chemical adhesion between the matrix and fiber, mechanical bond due to deformed fibers, fiber to fiber interlock in the form of tangling, and friction which is more dependent on confinement. [20] The pull out test is used to examine the resistance of the fiber to being pulled out of the concrete matrix. The larger the pull out load, the more resistant the fibers are to applied forces. [20] Fibers which are deformed in some way such as hooked, twisted, or indented will have higher pull out strengths than smooth circular wire. This is due to the fiber needing to be deformed in order to pull it from the concrete. This is produces high pull out loads compared with the purely physical and chemical adhesion of the smooth round wire. [20] Figure 1.2 shows the relation of deformed fibers and hooked wires to smooth wires. These tests show that the hooked wires provide better resistance to pull out than the smooth or deformed wires. The
greased tests were done in order to determine the effect of the physical bond of the fiber to the concrete itself.

This pull out strength is especially useful in increasing the post-cracking strength which increases the ductility of an otherwise brittle material. An idealized model of the failure and stress strain curve of a fiber reinforced concrete versus a high performance fiber reinforced concrete in tension can be seen in Figure 1.3. This shows the post crack strain continuing out well beyond the first crack which would typically rupture and fail a plain concrete specimen. This post cracking behavior increases the tensile and shear strength of the concrete even without mild steel reinforcement. [21]. It has also been shown that the strength of the concrete can have an effect on the post crack response of the concrete. This is a mutually beneficial situation as the discontinuous reinforcement helps to prevent crack propagation and catastrophic failure for high and ultra-high strength concrete. [7,8,20,22]
Polypropylene fibers function in a different fashion. In most cases, these fibers are not added to concrete in order to increase the tensile strength, but to act as a sacrificial material. They are typically added to increase the fire resistance of high density or low permeability concretes. [15-19,23-24] Polypropylene fibers have a melting point of roughly 165°C. [28] When the concrete heats up past this point, the fibers themselves melt and disperse into the surrounding matrix. This leaves an artificial pore space roughly the size of the fiber. Figure 1.4 shows a Scanning Electron Microscopy image of the residual melt material and void left by the melted fiber. [24] This newly introduced void space helps reduce the vapor pressure from the dehydrating concrete and evaporating water. This reduces the tendency of dense concretes to spall or explode when heated quickly. [15-19,23-24]. In low density concrete, the pressure build up is generally not as severe due to the high pore volume in the concrete.
1.2.1.2 Ultra-High Performance Concrete

Ultra-high performance concrete (UHPC) is a relatively new composite which has been developed in the past several decades. Ultra-high performance concretes typically have compressive strengths of at least 150 MPa (22 ksi). [25-32] It is important to note that the term Ultra-High Performance Concrete does not necessarily indicate that high strength is the only requirement for this designation. UHPC also must have superior durability to gain the distinction. Table 1.1 contains a comparison of various durability factors of normal strength, high performance, and ultra-high performance concretes. [31]
This increase in strength and durability is owed to the engineered packing density and low water to binder ratios. Figure 1.5 shows a schematic of the highly refined packing density used for UHPC. [31] Water to cement or water to binder ratios (w/c) of less than 0.24 are common for these high end composites. [25, 28, 30, 32] This is only possible with the use of high range water reducing admixtures. [34] Figure 1.6 shows a diagram of the typical particle make up of UHPC as compared with normal strength concrete. The ultra-high performance concrete typically will have a high paste content than normal strength or high strength concretes. [30]
The first instances of UHPC came in the late 1960’s and early 1970’s. These techniques focused on applying high pressure and heat prior to and during the concrete setting process. While these techniques were able to produce concretes with compressive strengths in excess of 480 MPa (70ksi), they were very difficult to replicate for field use. The next steps for UHPC came in the early 1980’s with the advent of Densified Powder Concretes and Micro Defect Free concrete. These composites were produced using a highly refined packing matrix and superplastisizers or polymers in order to achieve strengths of 150 to 250 MPa (22 to 36ksi). [7,25,27,32,35]

Over the next several years, there were many more advancements in UHPC and UHPFRC’s. The most notable came in 1995 with the development of Reactive Powder Concrete (RPC). RPCs combined a variety of methods in order to produce extraordinarily high compressive strength and fracture energies. They eliminated coarse aggregates and enhanced the packing density in order to increase the homogeneity of the mix. Combining these techniques in addition with heat and pressure curing, RPC’s with compressive strengths upwards of 800MPa (117ksi) were produced. These high end composites achieved their strength from the use of steel.
aggregate as a replacer for the siliceous aggregates. Even without heat and pressure treating, RPCs were capable of producing composites with compressive strengths over 200MPa (29ksi). In order to reduce the extremely brittle matrix of RPC, long steel fibers were introduced to increase the tensile strength and ductility. [32] Reactive powder concrete would go on to be the basis of the commercial product Ductal by Lafarge and the source of information for many future researchers in the quest to refine the concrete matrix. [27,29]

The dry mixes of most modern UHPCs contain many of the same components; Portland cement, silica fume, glass powder, and fine sands. [9-10,30,32-34] Barring the use of nanoparticles, silica fume is the smallest particle in the premix with an average size of roughly 0.1µm. [36] Glass powder, also called silica powder or ground silica, is the second smallest, with a size range of 1-5µm. [27] Cement contains a large range of particle sizes, from 1 to 100µm. [37] The largest particle of the matrix is the sands. These can range 150 to 1000µm. [29,33] Some researchers have experimented with use of coarse aggregate in UHPC’s with grading up to 12.7 mm (½ inch). [38-39] A typical grading curve of UHPC is shown in Figure 1.7. This overall gradation curve is overlaid with the individual grade curves for each constituent part. [40]

![Figure 1.7 – Grading Curves of Raw Materials and Mixture](image)
As mentioned previously, silica fumes are the smallest part of the concrete ranging in size from 0.01 to 1 μm. [36] Silica fume use holds two purposes; as a pozzolanic material and as a mechanical filler. The pozzolanic nature of silica fume causes a secondary hydration reaction to produce additional calcium silicate hydrates (C-S-H) from the calcium hydroxide in concrete and the silica fume being added. A visualization of this can be seen in Figure 1.8. [41]

![Figure 1.8 – Pozzolanic Reaction with Silica Fume](image)

In cement chemist’s notation, this reaction is characterized by the following formula: [42]

\[ 2S + 3CH + 5H \rightarrow C_3S_2H_8 \]  

Where 

- **S**: SiO\(_2\)  
- **CH**: Ca(OH)\(_2\)  
- **H**: H\(_2\)O  
- **C**: CaO

The products of this reaction allow the concrete to be more resilient against chemical attack than those without silica fume. This is owed to the decreased permeability caused by further
production of C-S-H. The extra C-S-Hs in the matrix also provide additional strength to the concrete. [27,36]

Silica fume is significantly smaller than any other particle in the mix. Its small size means that the silica fume will fill space otherwise held by pore water or air voids. This works to strengthen the concrete and increase the durability. [27,36] Silica fume, due to its round particle shape also increases the fluidity of the mix. [32] However, it is costly and difficult to obtain in some areas. In order to combat this problem, research has been done using industrial waste materials such as Ground Granulated Blast Furnace Slag (GGBFS), Fly Ash (FA), and other natural and processed pozzolan. These materials are used as either silica fume or cement replacements. This is due to the fact that when placed in combination with calcium hydroxide or another alkali activator, they may undergo pozzolanic reactions converting them into cement-like C-S-H. [43-46]

Quartz powder/glass powder is the next smallest particle in the UHPC mix. With a particle size ranging from 1 to 5\(\mu\)m, it acts as an effective mechanical filler to occupy the spaces between the larger cement particles and the finer sand particles. The additional packing of the quartz powder is found to increase the strength of the concrete. [30,33] While quartz powder may undergo a pozzolanic reaction, it requires the special curing conditions to do so. [47-48]

The most important part of any concrete is the cement or cementitious material. Beginning with work on reactive powder concretes, researchers working with UHPC have been using cements with a low Tricalcium Aluminate content (C\(_3\)A) content to provide better compressive strength and workability. [28,30,32-33] While C\(_3\)A contributes to high early strength in concrete, the rapid hydration generates excessive heat during hydration. It is also
deleterious to the workability of the mix due to its high water demand. The rapid hydration decreases the viscosity and can make the mix stiff and unworkable. [30,32-33]

Aggregates make up the remainder of the concrete dry materials. They can be any number of materials; silica sand/gravel, basalt, carbonaceous, or in some cases steel. While many researchers utilize sands of less than 1mm particle size, research has been done to reintroduce coarse aggregates up to 16 mm. These are the largest particles in the UHPC composition. [32-33,38-39]

Due to the very low w/c ratio of UHPC, conventional water reducing agents are not sufficient to cause fluidity. [34,49] This led to the development of a new breed of water reducing admixtures. Instead of using only electrostatic repulsion to disperse water, as original water reducing admixtures did, these admixtures have a long polymer backbone with many side branches in order to aid dispersion. In addition to electrostatic repulsion, the new class of superplasticizer uses steric hindrance to further disperse water and cement particles. Electrostatic repulsion works by the plasticizing admixture adsorbing to the cement particle and neutralizing the static cohesion between them to aid dispersion. Steric hindrance involves the overlapping of this surround admixture and further repelling adjacent cement particles in that fashion. [48]

The marriage of fiber reinforced concrete with the ultra-high performance concrete utilizes the strength and adhesion of the UHPC and the ductility and energy absorption of fiber reinforced concrete. These two build off the strengths of one another; the ultra-high performance concrete increases the pull out load of fibers making for a more ductile, energy absorbent structure and the fibers create a higher ductility in an otherwise very brittle matrix.
1.2.2 Chemical Change of Concrete at Elevated Temperature

An area of technical knowledge often overlooked is material behavior under elevated temperatures. Most research done, has not worked to separate the influence of chemical degradation and mechanical damage to the concrete. The chemical deterioration with high temperatures has also been examined separately but rarely with respect to mechanical strength. Table 1.2 shows a summary of these results. [50-56,59,61-62] The chemical degradation occurs by the dehydration of the C-S-H products as temperatures increase. The most hydrous start at the beginning of the list (11.3Å tobermorite, jennite, C-S-H I, C-S-H gel) and those located at the end are completely anhydrous (wollastonite, larnite).

<table>
<thead>
<tr>
<th>Chemical or Physical Change</th>
<th>100</th>
<th>200</th>
<th>300</th>
<th>400</th>
<th>500</th>
<th>600</th>
<th>700</th>
<th>800</th>
<th>900</th>
<th>&gt;900</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Evaporable Water driven off</td>
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<td>30-105°C</td>
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<tr>
<td>Decomposition of Gypsum</td>
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<td>110-170°C</td>
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<tr>
<td>Decomposition of Ettringite</td>
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<td>110-170°C</td>
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<tr>
<td>C-S-H I dehydration to Larnite</td>
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<td>120-500°C</td>
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<tr>
<td>C-S-H Gel dehydrates</td>
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<td>200-300°C</td>
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<tr>
<td>Degradation of Metajennite to disoriented phase</td>
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<td>350-450°C</td>
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<tr>
<td>Dehydroxylation of Portlandite</td>
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<td>450-550°C</td>
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<tr>
<td>Phase change from α-phase to β-phase quartz</td>
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<td></td>
<td>573°C</td>
</tr>
<tr>
<td>Dehydration of Hillebrandite to Larnite</td>
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<td></td>
<td>490-665°C</td>
</tr>
<tr>
<td>Dehydration of 9.3Å Tobermorite to Low-T Wollastonite</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>700-810°C</td>
</tr>
<tr>
<td>Dehydration of Xonotlite to Low-T Wollastonite</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>775-800°C</td>
</tr>
<tr>
<td>Phase change from β phase quartz tridymite</td>
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<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>872°C</td>
</tr>
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Table 1.2 – Chemical Change in Concrete with Temperature (continued)

<table>
<thead>
<tr>
<th></th>
<th>100</th>
<th>200</th>
<th>300</th>
<th>400</th>
<th>500</th>
<th>600</th>
<th>700</th>
<th>800</th>
<th>900</th>
<th>&gt;900</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-S-H II dehydration to Larnite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>650-900ºC</td>
</tr>
<tr>
<td>Disordered Jennite to Larnite and Wollastonite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>800-900ºC</td>
</tr>
<tr>
<td>Decarbonation of CaCO3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>700-900ºC</td>
</tr>
<tr>
<td>Reorientation of Low-T Wollastonite to High-T Wollastonite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1125ºC</td>
</tr>
</tbody>
</table>

Research has indicated that the temperature dependence of the chemical phases at under 300ºC is influenced by the ratio of silicon dioxide to calcium oxide in the mix. Additionally, the composition is dependent on the pressure and temperature while the concrete is setting as well. This research suggests that for the range of 150-300ºC three separate C-S-H phases can exist given the ratio of Si/Ca and the pressure applied. [28,32,47-48,55-57]

Tobermorite is known to exist in 3 separate phases. These phases are distinct from one another by the differences in their basal spacing and hydrate content. At around 20ºC, the calcium layers in tobermorite are at 14Å spacing. This form is aptly referred to as 14Å tobermorite. After heating to 60ºC, this form dehydrates and compresses so the basal spacing decreases to 11.3Å. With continued heating to between 200 and 300ºC, further dehydration results in the formation of 9.3Å tobermorite. [57-59] However, there is an anomalous form of tobermorite which does not reduce in spacing when the final dehydration occurs. [59] All forms of tobermorite undergo dehydroxylization at temperatures between 700 and 800ºC to low-t wollastonite. [51-57,59]

With heating, jennite breaks down into a metajennite form between 70 and 90ºC. Metajennite is stable up to 350ºC when it begins to decompose into a disordered form. This form maintains until roughly 835ºC when it finally degrades to larnite and wollastonite. [55]
Additional reactions occur with C-S-H (I) and C-S-H (II). C-S-H (I) is similar in form to 14Å tobermorite and is typically seen in steam cured concretes with highly reactive silica added. Some research suggests that C-S-H (I) dehydrates at a low temperature range from 120°C to 500°C to larnite. However, other research shows that it maintains its chemically bound water until 800°C. The formation of C-S-H (II) has eluded research. It is similar in structure to jennite and limited research has shown that it dehydrates to larnite between 650 and 900°C. Most research has shown, by 900°C, all C-S-Hs have lost all hydroxyl and bound water and transformed to wollastonite or larnite.

In UHPC, the application of heat and pressure greater than 200°C and 1bar at the time of setting results in the formation of xonotlite. Xonotlite is a better organized C-S-H and retains its chemical stability at higher temperatures than tobermorite. Where tobermorite undergoes several dehydrations and reorientations from 20 to 800°C, xonotlite only undergoes one at around 775°C. The presence of xonotlite is theorized to be part of the reason behind the excellent mechanical properties of heat and pressure treated reactive powder concretes and UHPCs.

At 573°C, the quartz present undergoes an α to β phase change. In the quartz mineral, this phase change is a reversible second order phase change. As the temperature increases, the tetrahedral chains of quartz molecules elongate and reorient. When the temperature decreases, the chains of quartz buckle and return to their original orientation. With this phase change comes a significant positive change in volume which would be visible as radial cracking around the perimeter of the silica particles in heated specimens. There is a second, less reported, phase change which silica undergoes at high temperatures. At approximately 872°C, silica
exhibits a phase change from $\beta$ phase quartz to tridymite. However, this phase change is thought to be difficult to produce at atmospheric pressure without the aid of alkali chlorides. [62]

### 1.2.3 Mechanical Properties of High Strength Concrete at Elevated Temperatures

As early as the late 1920’s, engineers have understood the need for investigation of heat loading on concrete structures. When subjected to elevated temperatures, concrete will deteriorate both mechanically and chemically. [15-19,63-81] The mechanical degradation at lower temperatures is typically attributed to the vaporizing of unbound water in the pore structure which may cause spalling. At higher temperatures, the C-S-H products and Calcium Hydroxide (portlandite) dehydrates resulting in decreased strength. [63] Additionally, the incompatibility between the thermal expansion of the aggregate and paste has shown to be an issue as well as other thermal strains. [53,80-81]

In recent years, research has investigated the effect of elevated temperatures on high strength concrete. A problem in high strength and high performance concretes (HSC and HPC) is spalling. Spalling occurs when pressure from within the concrete exceeds its tensile strength. This causes the surface of the concrete to break off. Tensile strength is a major contributing factor to the spalling resistance of concrete. [18-19,76-78,82] Figure 1.9 shows a stress based representation of this failure. The trend of the tensile strength of concrete versus temperature is plotted alongside the pore pressure caused by vaporizing water and thermal gradients within the specimen. When the pore pressures exceed the tensile strength, spalling will occur. [82] This pore pressure can also be generated by chemical intrusion, or by freezing water in the pore spaces.
Spalling caused by heat is found to be influenced by a number of factors including heating rate, the porosity, the strength of concrete, and the presence of fibers. In unreinforced concrete, it typically begins between 300-400°C. [70] Research has shown that higher mechanical strength concretes exhibit lower spalling resistance. This is caused by HSC/HPC’s dense matrix and low permeability. [63-72] Figure 1.10 shows the two assumed mechanisms behind thermal spalling. The first assumption shows the vapor barrier forming and leading to hydraulic pressure exceeding the tensile strength of the concrete which leads to spalling. The second assumption shows the confined expansion leading to the tensile strength being exceeded and causing failure. [71-72,74]
To alleviate this, researchers began adding steel and polypropylene fibers to the mix. Polypropylene fibers were added because of their low melting point (165°C) [16,72-73]. When heated, the polypropylene fibers melt and increase permeability. [15-16,72-73] This reduces the pore pressure and thereby reduces the risk of spalling. [15-17,72-73] Research into the influence of polypropylene fibers on residual mechanical properties is varied. Some research indicates the presence of polypropylene fibers has a negative influence on the residual mechanical properties. In these studies, the residual strength of concrete using polypropylene fibers alone is lower compared to the concrete using polypropylene fibers in combination with steel fibers. [15,18] Other research suggests that the presence of polypropylene fibers does not significantly affect the residual mechanical properties of heated concrete. [17,79]
Steel fibers are also used to increase the resistance to spalling. They accomplish this by increasing the tensile resistance of the concrete. Depending on its porosity, steel fibers are only useful in preventing spalling below 600ºC. The increased tensile strength leads to an increase in fracture energy which can result in spalling far more violent than had the concrete failed without fiber reinforcement. [18,77]

It has been shown that combining both steel and polypropylene fibers yields the best results with regard to fire resistance. The steel fibers being responsible for increasing the tensile strength to help handle the thermal stresses and the polypropylene fibers to help mitigate the vapor pressure build up inside the matrix. [78] These are not the only ways to prevent spalling. Controlling the reinforcement, the cover of the concrete, the moisture condition of the concrete, and the use of air entraining admixtures can also reduce the effects of spalling in concrete systems. [73]

Beyond the risk of spalling, the mechanical properties deteriorate with temperature. Numerous studies have been done on the effects of high temperature versus the residual and in-situ mechanical properties of high strength concrete. [17-19,65,67-68,73,75-91] The mechanical properties most often addressed include residual compressive strength, flexural strength, tensile strength, porosity, and permeability. The residual bending strength and tensile strength both typically follow the same downward trend with heating. [17-18,65,81,83-84,87-90] However, research on the residual compressive strength and elastic modulus exposed to high temperature shows varied results. There are two general schools of thought: trials in which the compressive strength and elastic modulus increase or remain constant up to a point between 200-400ºC and decreases rapidly afterwards [15,76,80-81,84,91], and trials where the residual strength and elastic modulus decrease with any heating applied. [17,65,69,75-76,80,83-84,86-88]
shows some research done on relative strength of HPC/HSC versus temperature. [92] Some sources suggest that the increase in strength is caused by a phase transformation in the concrete itself from poorly organized C-S-H products to better organized C-S-H products. [85] There is limited information available about the testing of the Poisson ratio versus heat, however the research that has been done as shown that as the temperature increases the Poisson ratio increases as well. [75,84]

Most existing research suggests that as the temperature increases, the permeability and the porosity increases as well. [67,75,84,91,93-94] This increase in permeability and porosity is attributed to the increase in micro-cracking caused by pressure, thermal expansion, and chemical

Figure 1.11 – Previous Research on High Performance/High Strength Concrete vs. Temperature [92]
dehydration. [67,91,93-95]. In the case of concretes with polypropylene fibers, the fibers
themselves serve to increase the permeability and porosity of the concrete when exposed to high
temperatures. [18,72,91,93-94]

1.2.4 Mechanical Properties of Ultra-High Strength Concrete at Elevated
Temperatures

The same issues which plague HPC/HSC at high temperatures are more prevalent with
UHPC. With its further refined matrix and optimized packing density, UHPC is far more brittle
and less permeable than normal and high strength concretes. [10,30,32,96-98] While the low
permeability is beneficial for the durability of UHPC, it is detrimental when it is exposed to high
temperatures. Similar to HPC/HSC, UHPC’s low permeability leads to a vapor barrier in
concrete exposed to elevated temperatures. [27,97,100] Figure 1.12 shows the process of spalling
as caused by a vapor barrier. As the concrete heats, C-S-H are dehydrated and pore water are
vaporized and pushed further into the matrix where they encounter lower temperatures and
condense. At a certain point, the fully saturated zone increases to such pressure that it exceeds
the tensile strength of the concrete and breaks away. [24]

As the UHPC packing density is higher, the spalling problem tends to be more severe and
occur at lower temperatures (200-300°C) than HPC/HSC. [100] Again, polypropylene and steel
fibers have been used to mitigate this problem. The use of both fiber types is shown to be an
optimal solution as it provides ductility as well as the thermal resistance required to withstand
high temperatures. [24,99-100,103-104] In UHPC, the use of either fiber alone was found to be
an inadequate solution as it does not mitigate the spalling problem. Using only polypropylene
fibers was not as effective as it was for HPC/HSC [103] and the use of steel fibers alone merely delays the onset of spalling. [102-103]

Figure 1.13 – Explosive Spalling in Concrete Due to Fire Exposure. [24]

Due to the relatively new nature of UHPC, there is only a small amount of research on the topic of its mechanical properties exposed to elevated temperatures. However, there have been some investigations in the compressive strength of ultra-high performance fiber reinforced concrete with respect to heat. These have shown what is commonly seen with regards to other types of concrete at high temperatures. Some studies have shown that as the temperature
increases, the compressive strength decreases. This decrease typically occurs slowly before 400°C then more quickly after. [100,105,107] However, some other research has shown that with an increase in temperature the residual compressive strength of UHPFRC may either remain relatively the same or increase up to 20% from room temperature up to 500°C before decreasing at higher temperatures. Similar results were shown for specimens under loading, however, these specimens lost strength at 100°C before gaining most of it back by 500°C. After 500°C, the compressive strength again decreases. [106-107] Tensile strength for both the hot tested and residual strength were found to generally decrease with an increase in temperature. [103-104] The elastic modulus was shown to decrease with an increase in temperature as well. [107] Results from residual compressive strength, elastic modulus and tensile strength with respect to temperature can be found in Figures 1.13-1.15. [103,107] It must be noted that these tests are typically run with both steel and polypropylene fibers. [100,105-107] Without fibers, specimens typically do not survive past 400°C due to the effects of spalling. [97,100,107] These tests were also completed at a wide variety of heating rates using different material compositions.
Figure 1.14 – Collective Results on Elastic Modulus vs. Temperature from Various UHPFRC’s [107]

Figure 1.15 – Tensile Strength vs. Temperature for Hot and Residual Case [103]
1.2.5 Thermal Properties of Normal Strength Concrete and High Strength Concrete

In order to construct a reliable finite element model for heat flow through a solid, several parameters are required. These are thermal conductivity, coefficient of expansion, and heat capacity. The problem with testing thermal properties of concrete is the very nature of the cementitious composite. Since it is a non-homogenous solid, concrete thermal properties are influenced by the thermal properties of their constituent parts. For example, the thermal conductivity will depend on the thermal conductivity of the cement paste, aggregate, air entrained, and the pore water. Changing volume of any of these in the mix can alter the thermal properties of the concrete. [108-113]

The thermal conductivity is defined as the heat flux across a unit cross section divided by the temperature gradient between the two surfaces. [114] There are many different methods for computing the thermal conductivity of concrete. The most commonly used is ASTM C177: Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus. This method uses a flat plate heater sandwiched in between two specimens of similar known thickness to be tested simultaneously. The specimens and the heater are encased on the ends by a series of insulating guards. The temperature across the sample and the power to the heater are recorded and used to calculate the thermal conductivity. [115]

The value for thermal conductivity of normal and high strength concrete at room temperature can vary from 0.27 W/m·°C to 3.18 W/m·°C for normal strength concretes [112] and roughly 1.99 W/m·°C [116] for high strength concretes. Concrete with carbonate based aggregate tended to have lower thermal conductivities than those with siliceous aggregates. [117]
Some researchers have gone so far as to derive theoretical equations to account for the difference in the thermal conductivity of the constituent parts of the concrete. [108,112,118-120] ACI Committee 122 has prepared a table containing various concretes, aggregates, and their thermal conductivity. [112]

In addition to the mineralogical makeup of the concrete, it has been shown that the temperature and moisture content can change the value as well. [89,103,112,118,120] The results for thermal conductivity with respect the temperature are varied. Some research indicated that with an increase in temperature the thermal conductivity of normal strength concrete generally decreased through the entire range of temperature tested. [117] However, some research has showed that with an increase in temperature the opposite occurs as either a general increase or as a decrease followed by an increase. [89,118,120] Research has shown that for cement mortar, thermal conductivity increases with increasing temperature. This was attributed to the dilated pore structure which would allow for an increase in radiative and convective heat transfer, though this was merely a theoretical analysis by the researchers. [118] This temperature dependence required a different method for calculating the value. Therefore, some researchers have begun using ASTM C1113: Standard Test Method for Thermal Conductivity of Refractories by Hot Wire (Platinum Resistance Thermometer Technique). This method has proven more suitable to use in high temperature environments since the use of elaborate apparatuses has been reduced to a heating wire and a series of thermocouples. [122]

The coefficient of thermal expansion is defined as the fractional change in dimension per unit change in temperature while the stress on the material is kept constant. [123] Empirical equations have been derived in order to predict the coefficient of thermal expansion of concrete based on the influence of its ingredients. [109,116] Studies have shown that the CTE of concrete
containing siliceous gravel tends to be higher than that of limestone gravel. There is a large accumulation of results for the coefficient of thermal expansion of various types of concrete containing different aggregates and fiber compositions. [88-89,109-110,116] The results from existing literature are varied. Some research tested fiber-reinforced concretes and showed that the coefficient of thermal expansion decreases with heat up to around 500°C after which it begins to climb again. This was attributed to the chemical breakdown of the C-S-H products. [88-89] Other research tested siliceous vs. carbonate aggregate based concretes with and without fibers. These studies showed the coefficient of thermal expansion to increase slowly over the range of 20°C to 700°C then the two fiber mixes flatten out while the non-fiber carbonate mix increased dramatically. This was attributed to the dolomite dissociating in the carbonate aggregate and the fibers constraining the expansion at high temperatures. [116]

The term heat capacity is defined as the amount of heat required to raise the temperature of one gram of a substance one Centigrade. Some research has shown that the heat capacity of concrete spikes at very specific areas where chemical reactions of phase transformations occur in the micro structure. In concrete with siliceous aggregate, these peaks in the heat capacity occur between room temperature and 100°C where evaporable water is released, 400°C and 500°C where portlandite begins to deteriorate, and 550°C and 600°C where silica quartz under goes the α-β phase shift. For carbonaceous aggregate concrete, the peaks occur in the range of 20°C-100°C, 400°C-500°C, and 750°C-800°C where calcium carbonate degrades into carbon dioxide and lime.[117] Other research has suggested that the specific heat of concrete increases up to 500°C then decreases slowly afterwards. [89]
1.2.6 Thermal Properties of Ultra-High Performance Concrete

There has been limited research done on the thermal properties of UHPC with respect to increasing temperature. There have, however, been studies on the thermal conductivity and specific heat of UHPFRC with respect to temperature. Some research has shown that the thermal conductivity decreases throughout the test period [103] while other research shows thermal conductivity decreasing to 400ºC then recovering slightly through 600ºC. [106] Research on specific heat is shown to reflect the microstructural breakdown of UHPC at high temperatures. These changes are similar to those of normal strength concrete. [106] On the other hand other research shows a peak in the specific heat at 200ºC then a decrease afterwards to 600ºC.

Some research has been done regarding the coefficient of thermal expansion of UHPC/UHPFRC for unheated specimens. These tests were conducted against the type of curing regiment used and results measured $13.93 \times 10^{-6}/^\circ C (7.74 \times 10^{-6}/^\circ F)$ for 28 day air cured specimens and $14.76 \times 10^{-6}/^\circ C (8.20 \times 10^{-6}/^\circ F)$ for 7 day steam cured specimens [126] and $14.94 \times 10^{-6}/^\circ C (8.30 \times 10^{-6}/^\circ F)$ from the other. [10] Another group using fiber optic sensors to test the coefficient of thermal expansion measured the constant ranging from $11.76 \times 10^{-6}/^\circ C$ to $12.33 \times 10^{-6}/^\circ C$. [127]

1.2.7 Discussions and Conclusions

There has been very limited research completed which works to separate the mechanical and chemical degradation of concrete with increase in temperature. As stated above, the degradation is often investigated without separating the two mechanisms of strength loss; mechanical and chemical. Further investigation using thermochemical analysis such as Differential Scanning Calorimetry (DSC), Thermo Gravimetric Analysis (TGA), and X-Ray
Diffraction (XRD) is necessary in order to determine the dehydrations and phase changed resulting in the strength loss at each phase.

A great deal of work has been done on the strength of normal and high strength concretes with regards to temperature. These results have been investigated with respect to any number of different parameters in the concrete mix. However, due to the relatively new nature of UHPC, the amount of research done on residual strength is limited. This research is further hindered by the problem of spalling which forces users to either adopt a slow heating rate or to use various fibers or fiber cocktails. Additional investigations are needed in order to understand the process by which ultra-high performance fiber reinforced concrete loses mechanical strength at high temperatures.

1.3 Report Structure

Chapter Two of this report will contain the materials used, the mixes prepared, the heating regiments used, the material analysis carried out and the mechanical tests performed.

Chapter Three presents the observations and results. This chapter will outline the data from experiments performed as well as stress vs. strain curves, residual mechanical property trends, and thermochemical analysis using Differential Scanning Calorimetry and Thermo Gravimetric analysis due to heating. These results will be compared and analyzed here to existing research and model codes to produce predictions based on the findings of this study.

Chapter Four will present the conclusions derived based on this research as well as a comparison to existing trends in published literature.
Chapter Two - Materials and Methods

2.1 Introduction

This chapter will discuss the materials and methods used throughout the course of this experimentation. It will cover all mix designs and mixing procedures as well as loading rates for both compressive tests and heating series. Additionally, it will cover all equipment and instrumentation used to carry out the tests and preparation of all specimens.

2.2 Materials

During the course of this research, two separate ultra-high performance fiber reinforced concretes were tested. The first was a commercially available UHPFRC, Ductal AF® by Lafarge. This product was developed by three European companies, Bouygues-Lafarge-Rhodia working off research on reactive powder concretes. This material advertises compressive strength of over 250MPa, flexural strength of over 40MPa, and improved fire resistance with the advent of their Ductal AF mix.[103] Material mechanical properties supplied by Lafarge are available in Table 2.1 This Ductal AF mix was used through the course of one half of this experimentation.
Table 2.1 – Ductal Manufacturers Supplied Material Characteristics [128]

<table>
<thead>
<tr>
<th></th>
<th>Ductal w/ Metallic Fibers</th>
<th>Ductal w/ Polypropylene and Metallic Fibers</th>
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</tr>
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<tr>
<td></td>
<td>TT*</td>
<td>No TT</td>
<td>TT</td>
</tr>
<tr>
<td>Density (kg/m³)</td>
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<td>2500</td>
<td>2350</td>
</tr>
<tr>
<td>Compressive Strength (MPa)</td>
<td>150-200</td>
<td>150-180</td>
<td>140-160</td>
</tr>
<tr>
<td>Elastic Limit In Tension (MPa)</td>
<td>9-10</td>
<td>7-9</td>
<td>8-9</td>
</tr>
<tr>
<td>Post Cracking Direct Tensile Strength w=0.3mm (MPa)</td>
<td>7-10</td>
<td>6-9</td>
<td>6-9</td>
</tr>
<tr>
<td>Equivalent Flexural Strength (MPa)</td>
<td>20-40</td>
<td>15-30</td>
<td>15-30</td>
</tr>
<tr>
<td>Young’s Modulus (GPa)</td>
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<td>45-55</td>
<td>35-45</td>
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<tr>
<td>Poisson’s Coefficient</td>
<td>0.2</td>
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</tr>
<tr>
<td>Shrinkage (mm/m)</td>
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<td>0.6-0.8</td>
<td>0.7-0.9</td>
</tr>
<tr>
<td>Creep</td>
<td>0.2-0.4</td>
<td>0.8-1.0</td>
<td>0.2-0.4</td>
</tr>
<tr>
<td>Thermal Dilation (µm/m/ºC)</td>
<td>12</td>
<td>12</td>
<td>12</td>
</tr>
</tbody>
</table>

* TT and No TT = thermally treated and not thermally treated

1kg/m³ = 1.69 lb/yd³  1 MPa = 145 psi  1 GPA = 145ksi

The second mix was a mix developed at University of Michigan Ann Arbor. This mix reports compressive strengths of at least 150MPa without the need for any exotic treating regiments. [30] The mixture used in this experimentation departed from these mix proportions. These changes were discussed with the authors and settled with some trial batches before research began.

### 2.2.1 Ductal AF®

The product Ductal is a proprietary product which was graciously provided for this testing by Lafarge North America. The research effort was supported with the Ductal premix, superplasticizer, steel fibers, and the mix design. The exact makeup of the premix is a trade secret so it is not available for discussion. The mix proportions used for this testing can be found in Table 2.2. The mix contains 2% steel fibers by volume. These steel fibers are 0.16mm.
(0.0063in) in diameter and 13mm (0.511in) in length. The fibers are slightly deformed by a bend at the midsection. They can be seen in Figure 2.1. These fibers are made of high strength steel with a tensile yield strength of roughly 2600kN (377 ksi). The deviation from the given mix proportions involved removing the polypropylene fibers in order to avoid contamination of slow heated specimens and chemical analysis at temperatures over 200°C.

<table>
<thead>
<tr>
<th>Material</th>
<th>Mass of material for One Cubic Meter (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Premix</td>
<td>2194</td>
</tr>
<tr>
<td>Water</td>
<td>150</td>
</tr>
<tr>
<td>Polypropylene</td>
<td>5</td>
</tr>
<tr>
<td>Superplasticizer One</td>
<td>30</td>
</tr>
<tr>
<td>Steel Fibers</td>
<td>150</td>
</tr>
</tbody>
</table>

### 2.2.2 House Mix

The mix referred to as the House mix was developed at the University of Michigan Ann Arbor by Drs. Kay Wille and Antonie Naaman. This mix was designed in order to produce an ultra-high performance concrete without the need for elaborate curing conditions which are not readily producible in the field. The mix uses a Portland type I cement with a low C₃A content and a silica fume with low carbon content. [30]

This mix was modified in order to reduce the w/c ratio and increase the fiber content. The fiber content of 2.5% is higher than that of Ductal but lower than other existing mixes. The lower w/c ratio was obtained by the use of two different superplasticizers. The first superplasticizer referred to as “SPL A”, was a polycarboxalate ether based water reducer. The second water reducer, referred to as “SPL B” was a modified phosphonate based compound. The steel fibers used were straight high strength steel fibers with a tensile strength of 2600kN (377 ksi). These
fibers were 0.18mm (0.0071 in) in diameter and roughly 13mm (0.51 in) long. These can be seen in Figure 2.2. The complete mix proportions used for the House mix can be seen in Table 2.3.

Table 2.3 – House Mix Proportions

<table>
<thead>
<tr>
<th>Material</th>
<th>Mix Design (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Type I Cement</td>
<td>196.25</td>
</tr>
<tr>
<td>Silica Fume</td>
<td>222.00</td>
</tr>
<tr>
<td>Silica Powder</td>
<td>222.00</td>
</tr>
<tr>
<td>Fine Sand</td>
<td>245.27</td>
</tr>
<tr>
<td>Coarse Sand</td>
<td>572.30</td>
</tr>
<tr>
<td>Water</td>
<td>155.22</td>
</tr>
<tr>
<td>Superplasticizer One</td>
<td>23.98</td>
</tr>
<tr>
<td>Superplasticizer Two</td>
<td>7.99</td>
</tr>
<tr>
<td>Steel Fibers</td>
<td>196.25</td>
</tr>
</tbody>
</table>

2.3 Methods

2.3.1 Mixing Procedure

All mixes were prepared using an Eirich Variable Intensity High Shear Mixer as seen in Figure 2.3. This mixer was intensity controlled rather than speed controlled so any mixing directions will be given in terms of Hertz rather than RPM. The bowl and mixing rotor were controlled independently to allow for different shears in the mix. No mixes larger than 6.6 liters were used to avoid spilling when the mixing crosshead was lifted after the mix had completed. All materials added to the mixer, with the exception of silica fume and sand had to be added
while the crosshead was closed. This was done to avoid bending the mixing rotor while closing the crosshead.

![Figure 2.3 – Eirich Adjustable Frequency High Shear Mixer](image)

**2.3.1.1 Ductal Mix Procedure**

The mixing procedure for Ductal was taken from a combination of resources. The first source used was the mixing guidelines provided by Lafarge. However, these guidelines were based on a cubic foot or cubic meter mix. As the mixes used in the course of this study were anywhere between three and six liters, the exact amount of time and how certain ingredients were added was changed. The manufacturer’s mixing procedure was supplemented with the process used by the Michigan Department of Transportation during its research on Ductal. [126] The mixing frequencies and times used can be found in Table 2.4. These times would vary based on the lab conditions at the time of the mix. Due to the fact that the lab is not climate controlled, the temperature and humidity of the lab was subject to change based on the time of year and the equipment in use during mixing.
Table 2.4 – Ductal Mixing Procedure

<table>
<thead>
<tr>
<th>Action</th>
<th>Time (minutes)</th>
<th>Mixer Intensity (rotor speed/pan speed)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix premix to break up agglomerations</td>
<td>&gt;2</td>
<td>15/15</td>
</tr>
<tr>
<td>Add water + ½ SPL A</td>
<td>3 – 5</td>
<td>15/15</td>
</tr>
<tr>
<td>Mix</td>
<td>8 – 10 (until mix begins to turn)</td>
<td>20/20</td>
</tr>
<tr>
<td>Add rest of SPL A</td>
<td>1 – 2</td>
<td>20/20</td>
</tr>
<tr>
<td>Mix</td>
<td>4</td>
<td>20/20</td>
</tr>
<tr>
<td>If mix as not turned increase speed, else skip this step</td>
<td>Until mix has turned completely</td>
<td>25 – 35/25 – 35</td>
</tr>
<tr>
<td>Stop mix</td>
<td>0.5</td>
<td>0/0</td>
</tr>
<tr>
<td>Start mix and add fibers</td>
<td>1</td>
<td>15/15</td>
</tr>
<tr>
<td>Allow to mix</td>
<td>2</td>
<td>15/15</td>
</tr>
<tr>
<td>Stop mix</td>
<td></td>
<td>0/0</td>
</tr>
</tbody>
</table>

The mix began with the dry premix being added to the mixer and run on low frequency for at least two minutes or until any large agglomerations were broken up and well mixed. The agglomerations were likely due to compacting and consolidating in the premix bag during shipping or storage. After the large chunks were broken up, the mix water and one half of the SPL A was mixed together and added slowly to the mix over three to five minutes.

At this point, the frequency of the mixer was increased and allowed to mix for eight to ten minutes or until the mix began to turn over. The sides were also periodically scrapped with a long spatula to pull any moist mix off the walls and back into the mix.

After approximately ten minutes had elapsed, the rest of the SPL A was added and allowed to mix for another four minutes. On some occasions, the mix would not become sufficiently fluid during this time. If this case arose, the mix frequency was increased further and allowed to mix until it became fluid. Once the mix became fluid, the mixer was stopped to allow air bubbles to rise out of the mix for thirty to forty-five seconds. The mixer was then restarted at low frequencies and the fibers were added in a sprinkling fashion into the mix over one minute.
The mix then continued for an additional minute before ending. Pictures of the progression of the mix can be seen in Figures 2.4-2.6.

In addition to the mix with fibers, a second small batch was mixed without steel or polypropylene fibers. This was mixed in the same fashion but the steps for adding fibers were left out. The purpose of this test was to create a small sample of concrete to undergo thermochemical analysis.
2.3.1.2 House Mix Procedure

The House mixing procedure is similar to that of the Ductal mixing procedure with the addition of several steps and changes to the times and the preparation of the premix. To prepare the premix, the silica fume and sands were mixed at low frequencies for three to five minutes. This served the purpose of both mixing the sand and silica fumes, but also allowed for some additional grinding of the silica fume to form a better packing density. The silica powder and cement were then added to the mix as well and allowed to mix at low intensity until the mix was uniform. This process was typically run for at least five minutes while the water and superplasticizers were measured.

After the premix was prepared, the water was combined with third of the SPL A and SPL B and was added over three minutes. The intensity of the mixing was increased and the mix continued for five to eight minutes or until the mix began to turn over. The sides were periodically scraped with a spatula to ensure no material remained unincorporated. The rest of the two SPL’s were then added to the mix over one minute. Mixing continued until the mix was completely turned over and sufficiently fluid. The mix intensity was then reduced to low and the fibers were added over one minute in a sprinkling fashion. The mix continued for another minute at low intensity before it was stopped. A table containing mix times and intensities can be found in Table 2.5. Pictures of the mixing process are shown in Figures 2.7-2.12.
### Table 2.5 – House Mixing Procedure

<table>
<thead>
<tr>
<th>Action</th>
<th>Time (minutes)</th>
<th>Mixer Intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix Silica Fume and Sands</td>
<td>3 – 5</td>
<td>15/15</td>
</tr>
<tr>
<td>Add cement and silica powder</td>
<td>3 – 5</td>
<td>15/15</td>
</tr>
<tr>
<td>Add water and 1/3 of SPL A and SPL B</td>
<td>3 – 5</td>
<td>15/15</td>
</tr>
<tr>
<td>Increase speed and allow to mix</td>
<td>5 – 7</td>
<td>20/20</td>
</tr>
<tr>
<td>Add the rest of SPL A&amp;B</td>
<td>1</td>
<td>20/20</td>
</tr>
<tr>
<td>Allow to mix</td>
<td>4 or until has turned over</td>
<td>20/20</td>
</tr>
<tr>
<td>If mix as not turned increase speed, else skip this step</td>
<td>Until mix has turned completely</td>
<td>25 – 45/25 – 45</td>
</tr>
<tr>
<td>Stop mix</td>
<td>0.5</td>
<td>0/0</td>
</tr>
<tr>
<td>Start mix and add fibers</td>
<td>1</td>
<td>15/15</td>
</tr>
<tr>
<td>Allow to mix</td>
<td>2</td>
<td>15/15</td>
</tr>
<tr>
<td>Stop mix</td>
<td></td>
<td>0/0</td>
</tr>
</tbody>
</table>

Figure 2.7 – House Mix Series
Fume and Sands Premixing

Figure 2.8 – House Mix Series
Premix with Cement and Silica Powder Added

Figure 2.9 – House Mix Series
Mix with Water Added

Figure 2.10 – House Mix Series
Turn Over
An additional mix was prepared without fibers. The purpose of mix was to create a small sample of the House mix to undergo thermochemical analysis.

### 2.3.2 Casting Process

Mixes were cast into lightly oiled three inch metal molds. Light vibratory compaction was used to draw additional air out of the mix. This was done by way of a 60Hz variable amplitude vibration table. Specimens were slowly cast in a circular pattern in order to aid the circular distribution of the fibers. Once each specimen was filled, it remained on the table for roughly 45 seconds before being removed. This was done to reduce the risk of over consolidation or fiber segregation while still allowing some air to be evacuated from the mix.

Specimens prepared for the DSC/TGA were cast into lightly oiled two inch plastic cylinder molds. They were also allowed to remain on the table for longer to further reduce the air content of the concrete.

Specimens were covered with a plastic sheet for between twenty-four and forty-eight hours prior to being removed from the molds and loaded into a steam box for curing. The steam box was heated to 90°C and 95% humidity in order to accelerate the curing process of the
concrete. The specimens remained in the steam box for roughly forty-four to forty-eight hours prior to removal and preparation. After removal from the steam box, specimens were allowed to cool to room temperature. Pictures of the steam box can be seen in Figure 2.13.

![Figure 2.13 – Steam Box](image)

### 2.3.3 Specimen Preparation

In order for specimens to be suitable for compressive testing, the specimen ends must be within a certain degree of parallel to one another. While previous research has used 1° of end planeness as the cut off for appropriately plane specimens [10], this research adopted a more stringent standard for testing. In order for cylinders to useable in compressive tests, the ends could be no more than 0.2° out of plane. This level of precision was obtained by using a three stage end preparation procedure. The approximate level of end planeness for each step can be seen in Table 2.5.
Table 2.6 – Process of End Preparation for Concrete Cylinders

<table>
<thead>
<tr>
<th>Order of Application</th>
<th>Method of Preparation</th>
<th>Approximate End Planeness</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Masonry Saw</td>
<td>$0.6 - \geq 1^\circ$</td>
</tr>
<tr>
<td>2</td>
<td>End Grinder</td>
<td>0.1-0.3$^\circ$</td>
</tr>
<tr>
<td>3</td>
<td>Fine Grinder</td>
<td>$\leq 0.2^\circ$</td>
</tr>
</tbody>
</table>

The first step of end preparation was to remove the rough unfinished top of the cylinders. This was accomplished by using the Clipper Supermatic wet masonry saw seen in Figure 2.14. This step removed part of the cylinder where large amounts of air accumulated during vibration.

![Masonry Wet Saw](image)

Due to the flexible nature of the saw blade, the saw cut was not a reliable way to produce a clean, parallel end. In order to achieve this, a mechanized diamond plated end grinding apparatus produced by Diam-END, shown in Figures 2.15, was used. Specimens were loaded into the holders on the machine and secured using a rubber wheel. Each run of the machine removed 4.5mm of the surface. In some cases, the saw cut end was more out of plane than could be removed in one run, so multiple runs through the end grinder were required. After one side of
the specimen was complete, it was reversed and the other end was ground. As the bottom of the specimen did not contain the rough unprepared surface of the top, the bottom of the specimens did not require cutting using the saw. The end grinder was capable of grinding up to four specimens per run.

![Figure 2.15 – Cylinder End Grinder with Lid Opened](image)

The final stage of preparation was used to finely grind each end of the specimen to smooth out any rough spots and loose fibers. A METlab METPOL-1V grinding machine shown in Figure 2.16 was used with an 80 or 120 grit grinding pad. First, the specimen was lightly ground on each side to remove protruding fibers and ridges caused by the end grinder. Afterwards, the specimen was dried and the ends were measured using the dilatometric device shown in Figure 2.17.
The specimen was placed on the rotating platen and measured against the dilatometer in order to obtain the location of a high point and a low point. If the specimen was not within the prescribed 0.2º, the high point was marked with a wax pencil and a grid was drawn. The specimen was reground focusing more on the high side until the grid was worn away. It was then measured again on the dilatometer. This process was repeated until the specimen was under 0.2º out of plane. At this point the center of the specimen was measured against the perimeter to ensure it was less than the highest point on the perimeter of the specimen. A picture of the progression of end preparation and the final prepared specimen can be seen in Figures 2.18 and 2.19. The right shows an uncut and unground specimen, the right middle shows a specimen which has been cut, the left middle shows as a specimen which has been ground on the end grinder and the far left shows a finished specimen ready for testing.
Specimen identification was done by way of a four part name. A typical name is shown below with the description of the naming convention:

155-12-d1-4

This name denotes that the specimen was cast on Julian date 155 (155th day) of the year 2012 (12), where it was the first Ductal (d1) mix cast and the fourth specimen in that mix (4). This
naming convention was used for all specimens cast for heating and compressive tests. Had the mix been a House mix, the letter “d” would be replaced with an “h”.

Specimens for the thermochemical analysis using the DSC/TGA were prepared in a different fashion. These specimens were cut into thin slices approximately three quarters of an inch to one inch thick using the masonry saw. These pieces were then broken up with a hammer and ground lightly using a large ceramic mortar and pestle as can be seen in Figure 2.20. The right most specimen is the first stage which is to cut the piece, the second stage shows the broken large chunks, and the third shows the ground concrete that will be used for testing.

![Figure 2.20 – Preparation of DSC/TGA Specimens](image)

2.4 Heat Testing

The heating tests were designed to separate the chemical and mechanical damage to the concrete done by various levels of heating. Two heating regiments were used; the first was a slow heating regiment designed to inflict chemical damage but not mechanical damage and will be referred to as the “S” series. The second was a fast heating regiment designed to increase the
temperature as quickly as possible and then remain at the target temperature long enough to allow enough time for the chemical degradation to take place while also inflicting mechanical damage. This will be referred to as the “F” series. Ductal and House mix specimens from the S series will be denoted “DS” and “HS”, respectively. Similarly, Ductal and House mix specimens from the F series will be referred to as “DF” and “HF”.

The S series used a very slow heating rate of 0.1ºC/min. The purpose of this heating rate was to ensure the specimens heated uniformly and avoid any severe thermal gradients. In order to ensure this, a dummy specimen was cast with two Type K high temperature thermocouples. The heating rate was checked with the dummy specimen to ensure the temperature difference between the surface the core never exceeded 15ºC. These thermocouples were placed as shown in Figure 2.21.

Prior to being heated, the S series specimens underwent a 28 day drying period at 135ºC to remove as much evaporable water as possible. The oven used in drying the specimens, seen in Figure 2.22, was a Fisher Scientific Isotemp Oven Model 630G drying oven. Three specimens from each mix were not dried and were used as the control for both heating regiments.
After the drying period, another three specimens were put aside and served to show the difference between the control specimens and those which had been dried. The rest of the specimens were heated in two ovens. Some specimens were heated in a Fisher Scientific Isotemp Muffle Furnace capable of achieving 1125ºC and others were heated in a separate high temperature furnace capable of reaching 1700ºC. The Fisher Scientific Isotemp Muffle Furnace can be seen in Figures 2.23. The second high temperature furnace was taken out of commission and was not available for photos.

The S series heated three specimens to the target temperature and then cooled at the same rate. The temperatures were chosen in 100ºC increments starting at 200ºC and increasing to 700ºC for the House mix specimens and 900ºC for the Ductal specimens.

The F series used the same Fisher Scientific Isotemp Muffle Furnace as the S series. Additionally, a steel mesh cage with lid was used to contain the debris caused by any explosive
spalling and decrease the probability of damage to the oven. The metal cage can be seen in Figure 2.24. This fast heating rate ran from room temperature to the target temperature at a rate of 14°C/min. This was designed to mimic the guidelines for a test fire as closely as possible within the capabilities of the furnaces used. [19,129]

Three specimens were heated to the target temperature and remained there for four hours. The extra time was to allow the temperature inside the specimens to equilibrate to the external temperature. After the four hours had elapsed, the heating units were shut off and the oven returned to room temperature at a natural pace. The temperatures were chosen in one hundred degree increments starting at 200°C and increasing to 800°C. This fast heating test was designed to be run for both the house mix and the ductal mix.

2.5 Compressive Strength and Elastic Modulus

Following the heating for each series, specimens underwent mechanical testing for both residual elastic modulus and residual compressive strength. The residual properties are defined as the properties of specimens which underwent heating and were then returned to room

Figure 2.24 – Metal Cage Used for Fast Heating Trials
temperature for testing. These tests were run using a Satec 1780 kN (400 kip) load frame with a MTS controller which can be seen in Figure 2.25. In order to accurately measure the strain of the specimen, a series of three Linear Variable Differential Transformers (LVDTs) were used in a custom rig.

![Figure 2.25 – Load Frame and MTS Controller](image)

The compression tests were displacement controlled and conducted at a rate of 0.5 mm/min. While the ASTM C39 standard recommends a force controlled load rate of 0.25 MPa/sec (35 psi/sec), this loading rate was found to be too slow for the testing of UHPC. Therefore, sources suggest using a loading rate of 1.05MPa/sec (150psi/sec). [10] However, preliminary tests with this method were incapable of capturing of any post rupture behavior. This lead to the utilization of the displacement controlled loading rate. For most trials this resulted in a compressive failure within several minutes of testing.

The elastic modulus was measured as the slope of the stress versus strain (stress-strain) curve. In order to calculate this strain, a custom rig was developed. This rig, seen in Figure 2.26,
employed three LVDTs at third points around the specimen. The LVDTs were held at a distance away from the specimen for two reasons; to prevent damage to the LVDTs in case the specimen failed explosively, and to allow the LVDTs to be downward facing which would prevent them from becoming wedged between the load platens. This rig was held in place by thumb screws which were located half way in between the holder arms. The thumb screws were ground to a point in order to prevent the specimen failure from bending the screws. The point was made as close to the centerline of the screw as possible. This was done for ease in calculating the initial distances for strain calculations. The initial lengths were taken by measuring the distance between outer diameters of the screws and subtracting half of each screw’s diameter. Once all lengths had been recorded, the average was taken for the LVDT between two adjacent screws. Readings from the LVDTs and the load frame were captured and recorded to a data file at a rate of 51.2 Hz. From this data, a stress-strain plot for each specimen was produced.

![Figure 2.26 – LDVTs in Holder for Strain Calculations](image)

2.6 DSC/TGA

Differential Scanning Calorimetry and Thermo Gravimetric Analysis (DSC/TGA) are tools used to perform the thermochemical analysis on the concrete specimens. This testing
determines the change in the structure of a material with increasing temperature. The DSC uses a high temperature furnace and several high temperature thermocouples to very accurately measure the amount of heat required to increase the temperature of a sample as a function of temperature. Since many materials show variations in heat capacity with material and phase changes, this data is very useful in detecting chemical or other microstructural changes in materials. These changes will appear on the DSC output as an increase or decrease in required voltage depending on the transitions’ enthalpy.

The TGA uses a high temperature furnace in conjunction with a microgram balance to measure the change in mass as a function of temperature. This is useful for determining the amount of mass lost from a material with temperature. In concrete, this allows researchers to observe the temperature ranges at which certain decompositions occur.

Testing was carried out for both the House and Ductal mixes using a Netzsch STA 449 F3 Jupiter Simultaneous TGA/DSC which can be seen in Figure 2.27. This machine performs both measurements simultaneously. The crushed and ground sample was first massed and loaded into the special alumina holder. It was then loaded into the machine and heated in an air atmosphere (80% nitrogen, 20% oxygen) up to 1000°C at a rate of 40°C/min. Once at 1000°C, the specimens remained for one hour before cooling to room temperature.

Figure 2.27 – Netzsch STA 449F3 Jupiter Simultaneous TGA/DSC
Chapter Three – Results and Observations

3.1 Introduction

This chapter will contain experimental data and observations resulting from the experiments detailed in the previous chapter. This will include all observations from during the experimentation, data preparation and analysis, stress vs. strain data, residual compressive strengths, residual elastic moduli, residual compressive strains, mass change with temperature, and chemical change data.

3.2 Heating

3.2.1 Spalling

It was understood from the onset of this experimentation that the F series would lead to some spalling due to the absence of polypropylene fibers. However, the nature of this spalling was more severe than initially anticipated. One HF specimen heated to 300ºC experienced explosive spalling roughly three hours into the test. During this spalling, the energy released was far greater than anticipated resulting in the heavy metal cage being knocked off the specimens and the ovens being covered in pulverized concrete. Figures showing the oven after the explosion, the specimens remaining intact, and the pulverized specimen are found in Figures 3.1 and 3.2. This led to the remainder of the fast heating experimentation being put on hold while a safety investigation was launched. It was decided that continued testing of the F series would not be wise. Therefore the only the F series data is two HF temperature ranges containing the 200ºC and the remaining two 300ºC specimens.
The specimens in the 300°C series which did survive heating experienced some surface spalling on the ends and corners, while the third specimen exploded, failing completely. It separated into three large disk shaped sections and many more small particles and fibers. The failed surface showed fibers protruding in a circular fashion which indicates that the pouring method used was successful in distributing the fibers as intended. The remains of failed
specimen can be seen in Figure 3.3. The remnants suggest that the increase in vapor pressure inside the concrete caused extensive micro cracking in the matrix. This matrix cracking propagated through large regions of the concrete until the specimen failed completely.

![Destroyed Concrete Specimen from 300°C HF Series](image)

**Figure 3.3 – Destroyed Concrete Specimen from 300°C HF Series**

### 3.2.2 Material and Color Change

During the course of the S series of experimentation, a change was noted in the nature of the concrete being heated. With an increase in temperature, there was a change in color which was characteristic across both mixes. Concrete has been shown in the past to exhibit color change at high temperatures. [55,130-132] Table 3.1 shows existing descriptions of color change with temperature.
Table 3.1 – Color Change in Concrete with Respect to Temperature of Exposure [131-132]

<table>
<thead>
<tr>
<th>Temperature (ºC)</th>
<th>Color Change</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient (20ºC)</td>
<td>Yellow</td>
</tr>
<tr>
<td>232-250ºC</td>
<td>Faint Pink</td>
</tr>
<tr>
<td>300ºC</td>
<td>Distinct Pink</td>
</tr>
<tr>
<td>300-600ºC</td>
<td>Pink changing to red or black</td>
</tr>
<tr>
<td>600-900ºC</td>
<td>Red or black</td>
</tr>
<tr>
<td>900ºC</td>
<td>Gray or Buff</td>
</tr>
<tr>
<td>1000ºC</td>
<td>Buff</td>
</tr>
</tbody>
</table>

The two mixes carried out were examined using a Pantone Color Match System.

Swatches of each color were compared to a representative sample of the concrete at each temperature and a label was found using Adobe Photoshop image editing software. Tables of the color change with regard to temperature for each mix can be found in Table 3.2. Concrete from the literature is reported as being a yellow at the ambient temperature. The concrete tested here started at a cool gray 6 for the HS series and a cool gray 8 for the DS series.

Table 3.2 – Color change of Ductal and House mix with Respect to Temperature of Exposure

<table>
<thead>
<tr>
<th>Temperature (ºC)</th>
<th>DS Color (Pantone CMS)</th>
<th>HS Color (Pantone CMS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>Cool Gray 8</td>
<td>Cool Gray 6</td>
</tr>
<tr>
<td>135</td>
<td>Cool Gray 9</td>
<td>Cool Gray 8</td>
</tr>
<tr>
<td>200</td>
<td>Cool Gray 9</td>
<td>Cool Gray 7</td>
</tr>
<tr>
<td>300</td>
<td>Cool Gray 9</td>
<td>Cool Gray 9</td>
</tr>
<tr>
<td>400</td>
<td>Cool Gray 9</td>
<td>Warm Gray</td>
</tr>
<tr>
<td>500</td>
<td>Warm Gray 8</td>
<td>Cool Gray 11</td>
</tr>
<tr>
<td>600</td>
<td>Cool Gray 8</td>
<td>Cool Gray 11</td>
</tr>
<tr>
<td>700</td>
<td>Cool Gray 7</td>
<td>Cool Gray 9</td>
</tr>
<tr>
<td>800</td>
<td>Warm Gray 6</td>
<td>N/a</td>
</tr>
<tr>
<td>900</td>
<td>Dark Taupe</td>
<td>N/a</td>
</tr>
</tbody>
</table>

The HS series varied in shades of gray starting at a light hue gray (cool gray 6) and darkening with temperature applied. At 400ºC, the concrete adopted a more reddish tone (warm gray) but returned to a blue grey hue again by 500ºC. The general trend with the HS series showed that the concrete started light and then became darker up to 500 and 600ºC after which
point the color became pale again. Figure 3.4 shows the progression from left to right of the color change in the HS series from the 90°C control specimen to the 700°C maximum.

The DS series varied less up to 300°C. In this range, there was little change from the cool gray 8/9 region. This could be attributed to the inherently dark color of the Ductal product. While Ductal’s color didn’t necessarily change for specimens heated to 400°C, the coloring became less uniform and gave more of a burnt look than those at other temperature series. Similar to the HS series and in literature, the concrete became very dark at 500°C. At 600°C, the specimen’s core remained very dark while the outside of the specimen acquired a lighter tone similar to the 700°C specimens. This can be seen on the failed surface of the specimen shown in Figure 3.5.
After 800°C, the specimens again acquired a reddish hue and by 900°C had become a dark taupe color. This color is also commonly referred to as buff. Figure 3.6 shows the progression in color change for the DS series from the control specimen at 90°C on the left to the 900°C specimen on the right.

![Figure 3.6 – DS Series Color Change with Temperature of Exposure (Left to Right: 90°C, 135°C, 200°C, 300°C, 400°C, 500°C, 600°C, 700°C, 800°C, 900°C)](image)

The change in color is widely attributed to the oxidation of iron hydroxides in the aggregates and cement paste. This produces hematite around 350 to 400°C which gives the concrete the brownish burnt look in both mixes. The final change to a pale color is reported to be caused by the breakdown of calcareous constituent parts of the paste and aggregate. [55] The reddish colors (dark taupe for the 900°C) at the high end of the heating cycle are reported to be further oxidation of other iron containing compounds in the concrete. [131-132]

In addition to the concrete itself changing color, at very high temperature ranges the material itself began to be affected. As the temperature of exposure increased, the sound produced when each specimen was struck with a metal rod became very ceramic and hollow in nature. While the control specimens sounded dull when struck, specimens heated to between 135 and 800°C produced a sharp ringing noise. While this noise was not readily distinguishable between specimens, some variation was evident between high and low temperatures. By 900°C, the specimens became so deteriorated that when struck, the sound produced was very dull.
The fibers also showed signs of deterioration from heating. While it was not evident below 500°C, afterwards, the fibers became increasingly brittle and weak. While at low temperatures, the steel fibers would take numerous flexes to break, the fibers at high temperatures broke with very little effort. This was especially true for temperature ranges above 800°C, where the fibers more resembled specks of carbon dust on the failed surface rather than a solid fiber. This could be attributed to the slow cooling time which resulted in fibers not going through a strengthening phase of cooling.

3.3 Data Collection and Preparation

During this study, most of the data collected came in the form of load and displacement data from the load frame and LVDTs. This data was used to produce a series of stress-strain curves to determine the residual stress-strain curves, residual elastic modulus, and residual compressive strength. Data was recording using the MTS controller and output into a data file on a host computer.

Load and displacement data were manipulated to produce stress-strain curves and to calculate the residual elastic moduli and compressive strengths. There were three types of stress-strain curves produced. The first was a standard stress-strain plot for the data as obtained from the machine, the second is the strain corrected stress-strain plot, and the third is the average stress-strain for each temperature series.

The standard stress-strain plot was produced using the refining loading data and displacements obtained from the three LVDTs. The average strain for each specimen was computed by equation 3.1:
Where:
\[ \varepsilon_{\text{avg}} = \frac{(d_{i1} - d_{o1}) + (d_{i2} - d_{o2}) + (d_{i3} - d_{o3})}{L_1 + L_2 + L_3} \]  
(3.1)

\[ \varepsilon_{\text{avg}}: \text{Average Strain} \]
\[ d_{i1}: \text{Displacement of LVDT 1 at a point } i \]
\[ d_{o1}: \text{Initial reading on LVDT 1} \]
\[ d_{i2}: \text{Displacement of LVDT 2 at a point } i \]
\[ d_{o2}: \text{Initial reading on LVDT 2} \]
\[ d_{i3}: \text{Displacement of LVDT 3 at a point } i \]
\[ d_{o3}: \text{Initial reading on LVDT 3} \]
\[ L_1: \text{Initial distance across which the strain is being measured from LVDT 1.} \]
\[ L_2: \text{Initial distance across which the strain is being measured from LVDT 2.} \]
\[ L_3: \text{Initial distance across which the strain is being measured from LVDT 3.} \]

This method allowed for redundancy in the specimen as it took three points around the specimens exterior to calculation the strains, as opposed to other setups which only use one LVDT. This helped to account for the possibility of the setup rotating during the test. This way the average of the three LVDTs still produced an accurate strain. This average strain was produced for every load point on record.

The load data was translated to stresses through use of the stress definition of:
\[ \sigma = \frac{F}{A} \]  
(3.2)

Where: \( \sigma \): Stress
\( F \): Load
\( A \): Cross sectional Area of the Specimen

As the specimens were 7.62cm (3in), the cross sectional area was calculated to be roughly 45.60cm\(^2\) (7.07in\(^2\)). Like the average strains, the stress was computed for each data point.

These stress-strain curves were plotted and the linear portion of the elastic region was located. This was done by using linear regression to find the most linear region. The lower and higher strain portions of the elastic slope were removed until the R\(^2\) value for linear regression was greater than 0.995. It became necessary to trim both ends, especially at higher temperatures, where there was a period of non-linearity at the beginning of each test. This non-linearity would
sometimes extend up to 65MPa (5.8ksi) of stress applied in the specimen. Therefore it was necessary to remove this portion to obtain a more accurate elastic modulus for each specimen. This hardening, which will be referred to in this study as pre-elastic hardening, can be seen in an example of the stress-strain curve for the 600°C HS specimen shown in Figure 3.7. The slope of the trend line was used to find the elastic modulus of the concrete specimen.

![Figure 3.7 – Sample Stress vs. Strain Curve Showing Pre-Elastic Hardening](image)

The strain corrected stress-strain curve was produced using the stress-strain curve, the elastic modulus and the y-intercept of the unadjusted curve. The strain corrected graphs were used to produce a relationship in which the elastic modulus intercepted the y-axis at zero. This would imply that strain is zero when no load is applied. In order to accomplish this, the strain value was offset by a strain adjustment coefficient. This coefficient was equaled to the quotient of the y-intercept and the elastic modulus. This value moved the stress-strain curve to neglect any initial hardening of the specimen or error in the strain measurements themselves. The adjusted strain was calculated with equation 3.3:

\[
\text{Adjusted Strain} = \frac{\text{Y-intercept}}{\text{Elastic Modulus}}
\]
\[ \varepsilon_{adj} = \varepsilon_o + \left( \frac{y}{E} \right) \] 

(3.3)

Where:
\( \varepsilon_{adj} \): Adjusted Strain
\( \varepsilon_o \): Measured strain
\( y \): y-intercept of linear portion of elastic region of stress vs. strain curve
\( E \): Elastic Modulus

Once this plot was produced, all values before the linear portion of the stress-strain curve began were removed and a 0,0 coordinate was placed to ensure the slope intersected the origin. A sample of the plot for the 300°C DS series is shown in Figure 3.8. Here, a comparison between the initial stress-strain plot and the corrected strain-strain plot are given.

![Figure 3.8 – Sample Strain Corrected Stress-Strain Curve for Corrected Strains.](image)

The adjusted plots were compared with the 2010 Fédération Internationale du Béton (fib) Model Code provisions for the non-linear stress-strain relationship of concrete. In order prepare for this comparison, each test was non-dimensionalized by dividing each stress value by the...
compressive strength of the corresponding test. Equation 3.4 is a reproduction of Equation 7.2-10 from the *fib* 2010 Model Code. [133]

\[
\frac{\sigma_c}{f_{cm}} = \frac{k\eta - \eta^2}{1 + (k - 2)\eta} \quad (3.4)
\]

Where:

- \(\eta\): Strain at given value
- \(\varepsilon_c\): Strain at maximum stress
- \(\varepsilon_{c1}\): Strain at given value
- \(k\): \(k = \frac{1.05f_{cm}\varepsilon_{c1}}{f_{cm}}\)
- \(f_{cm}\): Compressive strength of concrete
- \(E_{cm}\): Elastic Modulus of Concrete
- \(\sigma_c\): Stress at a given strain

This equation predicts the non-linear stress-strain behavior of concrete. Equation 3.4 predicts a stress-strain curve using three experimental parameters; the elastic modulus, the compressive strength, and the corresponding strain. This stress-strain curve is valid for the range of zero to the maximum strain of the specimen.

The final series of stress-strain curves produced were curves which had been adjusted to common strain values. Due to the fact that the MTS controller recorded values in time-based rather than displacement-based increments, the strain values were not at intervals which would allow for a meaningful average of the stress-strain graphs of a given temperature series. In order for an average curve to be produced, stresses and strains needed to be adjusted to a common set of strain values. These curves are referred to as the strain normalized series.

The imputing process was done by first parsing through the array of stresses and strains of a given series to determine the approximate location of a known strain. The strain values before and after that point were located. This same process was used to find the stresses corresponding to the strains found. Once these stresses and strains were found, linear interpolation was performed to find the corresponding stress at the known strain value. The
graph produced used strains which would be common for all series and would allow an average curve to be formed for each temperature range. After the stress-strain values for each specimen were normalized with respect to strain, the three specimens in each series were averaged together to obtain a representative stress-strain curve for that temperature series. These graphs were produced for each mix and each temperature series within that mix. The same process was followed for the strain adjusted curves. Once each individual specimen reached total failure, the values were no longer considered in the averaging process. For this reason some of the average curves become disjoint after the maximum stress. An example of this strain normalized stress-strain plot for the 200°C HF series can be found in Figure 3.9.

![Strain Normalized Stress vs. Strain 200°C HF Series](image)

Figure 3.9 – Strain Normalized Average Stress vs. Strain Curve with Individual Series Included

The final plot was a series which depicts the average stress-strain curve for each temperature series tests. Six of these plots were produced in Figures 3.10-3.15. These plots depict the residual stress-strain behavior of the mixes at each temperature range tested.
3.4 Stress-Strain Data

All specimens reported in this section were heated to the prescribed temperature, held for either two or four hours depending on the series and then cooled to room temperature. Afterwards, specimens were compressive tested until the specimen failed or the load had dropped to less than one quarter of the maximum load. Data was recorded in the form of load values and displacement data from the LVDT. In some cases, the failure occurred very quickly with little ductility, while in others, the test continued for several minutes. There were some cases where the test continued to run, but the data had to be truncated due to some sudden change in the specimen causing the LVDTs to no longer produce meaningful data. This occurred in several cases and resulted in the strain decreasing with applied load. These values were removed for the purpose of calculation and evaluation.

3.4.1 DS Series

The results for the DS series without strain adjustment are shown in Figure 3.10. In this series, control specimens up through those heated to 200°C showed a linear elastic region through the majority of the curve. After 200°C, pre-elastic hardening was shown before the onset of the elastic region. The effect became more prominent with higher temperature of exposure. Data showing the stress and strain values at the onset of the elastic region for the DS series are shown in Table 3.3. These strains were found using the method described in Section 3.3. While this method was not exact, it did provide an estimate of the onset location of the elastic region for the stress-strain curves. The data showed the strain at the onset of the elastic region increased from 0mε for the control specimens to 6.90mε at 700°C. For the 800°C and 900°C series, the values decreased rather than increased. This could be attributed to the extensive mechanical damage of the concrete matrix and heavy cracking observed.
Many specimens in the low temperature ranges (90ºC-200ºC) failed very quickly with little load carried after the maximum stress was reached. Cracking typically began as early as 30MPa before failure initiating. However, once the specimen reached the maximum stress, failure occurred shortly afterward. This behavior became less prevalent with the increase in temperature. At higher temperatures, these specimens typically exhibited higher ductility and energy dissipation. The higher temperatures also showed reduced slopes of the stress-strain curves. For specimens heated to 800ºC and 900ºC, the linear region of the stress-strain curve became difficult to define due to the presence of extensive pre-elastic hardening.

Table 3.3 – DS Series Strain and Stress Values at Onset of Elastic Region

<table>
<thead>
<tr>
<th>Temp (ºC)</th>
<th>Strain at onset of elastic region (mm/mm)</th>
<th>Stress at onset of elastic region (MPa) [ksi]</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>no offset</td>
<td>N/a</td>
</tr>
<tr>
<td>135</td>
<td>no offset</td>
<td>N/a</td>
</tr>
<tr>
<td>200</td>
<td>0.000664</td>
<td>30.10 [4.37]</td>
</tr>
<tr>
<td>300</td>
<td>0.002078</td>
<td>65.06 [9.44]</td>
</tr>
<tr>
<td>400</td>
<td>0.002448</td>
<td>64.01 [9.28]</td>
</tr>
<tr>
<td>500</td>
<td>0.002996</td>
<td>62.03 [9.00]</td>
</tr>
<tr>
<td>600</td>
<td>0.005394</td>
<td>68.30 [9.91]</td>
</tr>
<tr>
<td>700</td>
<td>0.006902</td>
<td>43.07 [6.25]</td>
</tr>
<tr>
<td>800</td>
<td>0.000322</td>
<td>2.00 [0.29]</td>
</tr>
<tr>
<td>900</td>
<td>0.000716</td>
<td>2.01 [0.29]</td>
</tr>
</tbody>
</table>

The strain adjusted DS series is located in Figure 3.11. This shows each individual temperature series with any initial non-linearity removed prior to the elastic region. These curves represent an idealized stress-strain curve.

The *fib* Model Code shows good agreement with the majority of the DS series temperature ranges. However, the presence of fibers led to a strain softening period in some series. This was particularly true in the more ductile, high temperature series. The strain softening made the code curve inadequate shortly after the specimens’ initial rupture. Figure 3.12 shows the 400ºC DS series curve with the Model Code overlaid. Here, it was shown that while
the Model Code doesn’t follow the linear nature of the elastic portion of the curve, the behavior up until rupture was reasonably accurate. However, shortly after the rupture occurs, the Model Code no longer was capable of predicting the behavior due to the large ductility of the specimens in this range.

Figure 3.10 – Average Residual Stress-Strain Curves for DS Series without Adjusted Strains
3.4.2 HS Series

The results for the HS series without strain adjustment are shown in Figure 3.13. The pre-elastic hardening was found in specimens heated beyond the drying temperature of 135°C and
increased through the end of the series. Table 3.4 shows the stress and strain values at the onset of the elastic region. The onset strains increased from 0m$\varepsilon$ to 9.25m$\varepsilon$ by 700ºC. This implies that as the temperature increases, the specimens must compress more before entering the elastic strain portion of the stress-strain curve.

While the control specimens for the HS series showed a prolonged strain softening period, the 135ºC and 200ºC series did not. Specimens from the control group held load out to nearly 12.6m$\varepsilon$ whereas 135ºC and 200ºC failed complete after 7.0m$\varepsilon$ and 6.1m$\varepsilon$, respectively. By 300ºC, the ductility and accompanying strain softening returned and continued through the end of the test range.

The strain adjusted HS series can be found in Figure 3.14. These values were compared with the Model Code. The code predicted values model the stress-strain curve well for the linear elastic region. In specimens with long strain softening regions, once the maximum load was reached, the code could no longer adequately predict the concrete’s behavior. Figure 3.15 shows an example of this comparison.

Table 3.4 – HS Series Strain and Stress Values at Onset of Elastic Region

<table>
<thead>
<tr>
<th>Temp (ºC)</th>
<th>Strain at onset of elastic region (mm/mm or (in/in))</th>
<th>Stress at onset of elastic region (MPa) [ksi]</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>no offset</td>
<td>N/a</td>
</tr>
<tr>
<td>135</td>
<td>no offset</td>
<td>N/a</td>
</tr>
<tr>
<td>200</td>
<td>0.000296</td>
<td>14.78 [2.14]</td>
</tr>
<tr>
<td>300</td>
<td>0.001438</td>
<td>40.05 [5.81]</td>
</tr>
<tr>
<td>400</td>
<td>0.003312</td>
<td>69.38 [10.06]</td>
</tr>
<tr>
<td>500</td>
<td>0.004822</td>
<td>63.97 [9.28]</td>
</tr>
<tr>
<td>600</td>
<td>0.008160</td>
<td>60.06 [8.71]</td>
</tr>
<tr>
<td>700</td>
<td>0.009254</td>
<td>28.02 [4.06]</td>
</tr>
</tbody>
</table>
Figure 3.13 – Average Residual Stress-Strain Curves for HS Series without Adjusted Strains

Figure 3.14 – Average Residual Stress-Strain Curves for HS Series with Adjusted Strains
3.4.3 HF Series

The results for the HF series without strain adjustment are shown in Figure 3.16. This series shows the onset of pre-elastic hardening delayed to the 300ºC specimens. Values for the pre-elastic hardening can be found in Table 3.5. Only the 300ºC series showed any pre-elastic hardening. The strain value at onset of linear-elastic behavior for the HF specimens was 0.4mm.

The strain adjusted stress strain curves can be found in Figure 3.17. These individual series were again compared to the Model Code and all were comparable to the strain adjusted test data. While only the control showed a long strain softening period, even with limited post-rupture strains, the code was still incapable of capturing the post-rupture behavior. The Model Code comparison can be seen in Figure 3.18.

<table>
<thead>
<tr>
<th>Temp (ºC)</th>
<th>Strain at onset of elastic region (mm/mm or (in/in))</th>
<th>Stress at onset of elastic region (MPa) [ksi]</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>no offset</td>
<td>N/a</td>
</tr>
<tr>
<td>200</td>
<td>no offset</td>
<td>N/a</td>
</tr>
<tr>
<td>300</td>
<td>0.000464</td>
<td>21.22 [3.08]</td>
</tr>
</tbody>
</table>
Figure 3.16 - Average Residual Stress-Strain Curves for HF Series without Adjusted Strains

Figure 3.17 – Average Residual Stress-Strain Curves for HF Series with Adjusted Strains
3.4.4 Comparisons

Each of the series showed some commonalities in their stress-strain curves. All series tested showed that for specimens heated to at least drying temperature at 135°C, no pre-elastic hardening would occur. However, despite the fact that both HS and DS series begin their hardening phase at 135°C, they do so at different values. At 135°C, the HS series has a pre-elastic strain which is 55% lower than the DS series. This changes as the HS series then increases faster than the DS series. This holds true out to 700°C where the HS series shows a pre-elastic strain which is 134% of the DS series.

Unlike the DS and HS series, the HF series showed the onset of pre-elastic hardening delayed until 300°C. Even at this temperature, the pre-elastic strain was far less than either DS or HS specimens showed at the same temperature. The pre-elastic strain for HF was only 32% of the value of HS series and 22% of the DS series at 300°C, respectively.
3.5 Residual Compressive Strength

The residual compressive strength is considered to be the maximum compressive strength obtained following heating to the prescribed temperature and returning to ambient temperature. The value presented is the average of the three tests heated to a given temperature.

3.5.1 DS Series

The DS series graph for residual compressive strength with respect to temperature can be found in Figure 3.19 and corresponding data can be found in Table 3.6. This graph is presented as set of three experimental values with an average curve overlaid.

![Residual Compressive Strength vs. Temperature DS Series](image)

Figure 3.19 – Residual Compressive Strength of DS Series with Increasing Temperature
### Table 3.6 – Residual Compressive Strength Data from DS Series with Increasing Temperature

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$f'_c$(MPa)</th>
<th>Average (MPa)</th>
<th>Standard Deviation</th>
<th>$f'_c$(ksi)</th>
<th>Average (ksi)</th>
<th>Standard Deviation</th>
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<tr>
<td>90</td>
<td>229.39</td>
<td>232.59</td>
<td>2.95</td>
<td>33.27</td>
<td>33.73</td>
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<td>10.53</td>
<td>0.72</td>
<td>1.64</td>
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<td>1.0</td>
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<td>900</td>
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<td>1.64</td>
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</tr>
<tr>
<td>900</td>
<td>10.37</td>
<td></td>
<td></td>
<td>1.50</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The tests showed a marked increase in compressive strength in specimens from the 135°C and 200°C. These series increased to 132.6% and 143.5% of the control value, respectively. After this increase, the compressive strength decreased to roughly the control. Up to 500°C, the compressive strength remained at 92.6% of the control series. Beyond 500°C, the residual strength decreased dramatically from 92.6% at 500°C to 37.2% at 700°C. After 700°C, the
residual compressive strength dropped to less than 15% of the control. At 900°C, the compressive strength was just 4.5% of the control.

It should be noted that at temperatures less than 700°C, the DS series still showed a residual compressive strength of roughly 86.61MPa (12.56ksi). However, by 800°C, that value decreased to only 24.74MPa (3.59ksi).

### 3.5.2 HS Series

The HS series graph for residual compressive strength with respect to temperature can be found in Figure 3.20 and the corresponding data can be found in Table 3.7. This contains a plot of the three individual test results with an average curve overlaid.
Table 3.7 – Residual Compressive Strength Data from HS Series with Increasing Temperature

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$f'_c$(MPa)</th>
<th>Average (MPa)</th>
<th>Standard Deviation</th>
<th>$f'_c$(ksi)</th>
<th>Average (ksi)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
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<td>206.10</td>
<td>16.44</td>
<td>28.46</td>
<td>29.89</td>
<td>2.38</td>
</tr>
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<td>196.99</td>
<td>205.40</td>
<td>16.25</td>
<td>28.34</td>
<td>29.76</td>
<td>2.32</td>
</tr>
<tr>
<td>90</td>
<td>225.08</td>
<td>223.28</td>
<td>12.91</td>
<td>32.64</td>
<td>34.40</td>
<td>2.14</td>
</tr>
<tr>
<td>135</td>
<td>270.23</td>
<td>258.84</td>
<td>12.75</td>
<td>39.19</td>
<td>41.19</td>
<td>2.17</td>
</tr>
<tr>
<td>135</td>
<td>261.38</td>
<td>258.93</td>
<td>12.73</td>
<td>37.91</td>
<td>39.37</td>
<td>1.86</td>
</tr>
<tr>
<td>135</td>
<td>244.91</td>
<td>245.53</td>
<td>12.73</td>
<td>35.52</td>
<td>37.12</td>
<td>1.86</td>
</tr>
<tr>
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<td>266.84</td>
<td>270.52</td>
<td>10.87</td>
<td>38.70</td>
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<td>10.87</td>
<td>38.00</td>
<td>39.66</td>
<td>1.58</td>
</tr>
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<td>41.01</td>
<td>42.92</td>
<td>1.58</td>
</tr>
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<td>48.31</td>
<td>50.00</td>
<td>3.64</td>
</tr>
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<td>25.11</td>
<td>43.93</td>
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<td>3.64</td>
</tr>
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<td>350.62</td>
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<td>51.16</td>
<td>52.96</td>
<td>3.64</td>
</tr>
<tr>
<td>400</td>
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<td>4.68</td>
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<td>205.22</td>
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<td>250.24</td>
<td>9.72</td>
<td>36.31</td>
<td>37.35</td>
<td>1.14</td>
</tr>
<tr>
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<td>213.22</td>
<td>205.22</td>
<td>9.72</td>
<td>30.92</td>
<td>32.08</td>
<td>1.14</td>
</tr>
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<td>28.20</td>
<td>29.34</td>
<td>1.01</td>
</tr>
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<td>208.04</td>
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<td>0.73</td>
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<td>21.04</td>
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<td>143.47</td>
<td>6.97</td>
<td>20.81</td>
<td>20.81</td>
<td>1.01</td>
</tr>
<tr>
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<td>156.26</td>
<td>6.97</td>
<td>22.66</td>
<td>22.66</td>
<td>1.01</td>
</tr>
<tr>
<td>700</td>
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<td>74.76</td>
<td>5.04</td>
<td>10.96</td>
<td>10.96</td>
<td>0.73</td>
</tr>
<tr>
<td>700</td>
<td>79.34</td>
<td>79.34</td>
<td>5.04</td>
<td>11.51</td>
<td>11.51</td>
<td>0.73</td>
</tr>
<tr>
<td>700</td>
<td>69.36</td>
<td>69.36</td>
<td>5.04</td>
<td>10.06</td>
<td>10.06</td>
<td>0.73</td>
</tr>
</tbody>
</table>

The HS series showed an increase in residual compressive strength for temperature ranges up to 300°C. Specimens which had only undergone the drying treatment increased in strength to 125.6% while specimens heated to 200°C increased further to 131.3% of the control. There was a drastic increase at 300°C to 159.9% of the control. However, this large increase in compressive strength was short lived and by 400°C, the residual compressive strength had dropped to 116.6% of the control values. In terms of stresses, this decrease corresponded to an 89.3MPa reduction in compressive strength from the 300°C average of 329.58MPa to 240.28MPa. The 500°C series continued this downward trend, showing 99.6% of the control. After this point, the strength decreased quickly to 71.9% at 600°C and to 36.3% at 700°C.
3.5.3 HF Series

The HF series plot for the residual compressive strength with respect to temperature can be found in Figure 3.21 and the corresponding data can be found in Table 3.8. This graph is a plot of the three individual test results with an average curve overlaid for the control and the 200°C range, however due to the spalling failure of one of the 300°C specimens, there are only two results from that series.

![Figure 3.21 – Residual Compressive Strength of HF Series with Increasing Temperature](image)

**Table 3.8 – Residual Compressive Strength Data from HF Series with Increasing Temperature**

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$f'_c$(MPa)</th>
<th>Average (MPa)</th>
<th>Standard Deviation</th>
<th>$f'_c$(ksi)</th>
<th>Average (ksi)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>196.22</td>
<td>206.10</td>
<td>16.44</td>
<td>28.460</td>
<td>28.571</td>
<td>2.38</td>
</tr>
<tr>
<td>90</td>
<td>196.99</td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>90</td>
<td>225.08</td>
<td>233.36</td>
<td>5.85</td>
<td>32.645</td>
<td>33.281</td>
<td>0.85</td>
</tr>
<tr>
<td>200</td>
<td>240.08</td>
<td></td>
<td></td>
<td>34.821</td>
<td></td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>229.47</td>
<td></td>
<td></td>
<td>33.281</td>
<td>33.436</td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>230.53</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 3.8 – Residual Compressive Strength Data from HF Series with Increasing Temperature (continued)

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$f'_c$(MPa)</th>
<th>Average (MPa)</th>
<th>Standard Deviation</th>
<th>$f'_c$(ksi)</th>
<th>Average (ksi)</th>
<th>Standard Deviation</th>
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<tbody>
<tr>
<td>300</td>
<td>262.62</td>
<td>270.27</td>
<td>10.81</td>
<td>38.09</td>
<td>39.20</td>
<td>1.57</td>
</tr>
<tr>
<td>300</td>
<td>277.91</td>
<td></td>
<td></td>
<td>40.31</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

While the data from the HF heating series is limited, it can still be seen that the compressive strength increased over the control. Specimens heated to 200°C showed an increase of 13.2% and specimens heated to 300°C exhibited an increase of 31.1% over the control specimens. This increase in compressive strength for the 300°C specimens occurred with the mild spalling on the ends seen in Figure 3.2 and Figure 3.22.

![Figure 3.22 – Spalling on the Ends of 300°C HF Series.](image)

### 3.5.4 Comparisons

The residual compressive strength versus temperature plots presented in this section were normalized with respect to the compressive strength in order to show the relative changes in compressive strength as opposed to the absolute changes. This was done by dividing the compressive strength of each series by the compressive strength of the control series.
The HS and HF series are compared in Figure 3.23. While, these two series were both the House mix, they were heated at different rates. The HF series showed a compressive strength 18% lower strength than the HS series when tested at 200ºC. This disparity increased for specimens heated at 300ºC. In this range, the difference between the two increased to 28.8%. Experimental data from these two series showing residual relative compressive strengths can be found in Table 3.9.

Table 3.9 – Residual Compressive Strength Data from HF Series vs. HS Series with Increasing Temperature

<table>
<thead>
<tr>
<th>Temp (ºC)</th>
<th>HS</th>
<th>HF</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>1.000</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>200</td>
<td>1.313</td>
<td>1.132</td>
<td>0.180</td>
</tr>
<tr>
<td>300</td>
<td>1.599</td>
<td>1.311</td>
<td>0.288</td>
</tr>
<tr>
<td>400</td>
<td>1.166</td>
<td></td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>0.996</td>
<td></td>
<td></td>
</tr>
<tr>
<td>600</td>
<td>0.719</td>
<td></td>
<td></td>
</tr>
<tr>
<td>700</td>
<td>0.363</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 3.23 – Residual Compressive Strength of HF Series vs. HS Series

Comparisons between the DS and the HS series can be found in Figure 3.24. The primary difference between these two series was the shift of the maximum compressive strength. As reported previously, the DS series showed an increase in compressive strength up to 200ºC while the house series showed the increase through 300ºC. At 200ºC, the compressive strength of the DS series was only 12.2% more than the HS series. However by 300ºC, the two exchanged places and the HS series’ compressive strength was almost 60% larger than the DS. This can be partially attributed to the fact that, the DS series had already begun to lose strength.
After this initial disparity, the residual compressive strengths of the two series showed reasonably close results with only a 5% difference from one another at 700ºC. Experimental data from these two series showing residual normalized compressive strengths can be found in Table 3.10.

Table 3.10 – Residual Compressive Strength Data from DS Series vs. HS Series with Increasing Temperature

<table>
<thead>
<tr>
<th>Temp (ºC)</th>
<th>HS</th>
<th>DS</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>1.000</td>
<td>1.000</td>
<td>0.000</td>
</tr>
<tr>
<td>135</td>
<td>1.256</td>
<td>1.326</td>
<td>-0.070</td>
</tr>
<tr>
<td>200</td>
<td>1.313</td>
<td>1.435</td>
<td>-0.122</td>
</tr>
<tr>
<td>300</td>
<td>1.599</td>
<td>1.002</td>
<td>0.597</td>
</tr>
<tr>
<td>400</td>
<td>1.166</td>
<td>0.986</td>
<td>0.180</td>
</tr>
<tr>
<td>500</td>
<td>0.996</td>
<td>0.926</td>
<td>0.070</td>
</tr>
<tr>
<td>600</td>
<td>0.719</td>
<td>0.688</td>
<td>0.031</td>
</tr>
<tr>
<td>700</td>
<td>0.363</td>
<td>0.372</td>
<td>-0.010</td>
</tr>
<tr>
<td>800</td>
<td>0.106</td>
<td>-0.106</td>
<td></td>
</tr>
<tr>
<td>900</td>
<td>0.045</td>
<td>-0.045</td>
<td></td>
</tr>
</tbody>
</table>

Figure 3.24 – Residual Compressive Strength of HF Series vs. HS Series

Comparisons between the residual compressive strength of the DS, HS, and HF tests and existing literature on UHPC at elevated temperatures can be found in Figure 3.25. [107] The research conducted in this study differed from the existing research on the topic. Many of the studies conducted either decreased with any heat applied or increased slightly to a certain temperature then decreased afterwards. There were two separate cases where the literature shows an increase in strength up to roughly 500ºC and 600 ºC. However, this was not shown to the same extent as the research conducted in this investigation. One series from the existing literature showed an increase to roughly 110% of the initial values, however, this does not match the results of any tests in this investigation. After 500ºC, the research performed here falls within the
general trend of other research found in literature. It is important to note, that these tests were conducted at a variety of heating rates with material compositions. The purpose of this graph is merely for comparison with other compressive strength results of UHPC and UHPFRC at high temperatures.

![Figure 3.25](image)

**Figure 3.25 – Experimental Results vs. Existing Literature on UHPC Compressive Strength at High Temperatures Based on Graph in [107].**

Figure 3.26 shows the comparison of the experimental results with literature results regarding normal and high strength concrete. [106] Existing literature showed a slow steady loss of strength for normal strength concrete. The high strength concrete shown decreased from baseline, but gained some of this strength back before finally decreasing out to failure. The UHPC exhibited similar behavior to the high strength concrete, however, during it returned to almost 100% of the original value. These results differed from the test data due to the lack of a well-defined increase in strength at temperatures below 300ºC. As with the comparison made
above, there is little information about the testing rate and compositions of these concretes. The purpose of this plot is to compare experimental results with the results of other research in existing literature for normal and high strength concretes.

Figure 3.26 – Experimental Results vs. Existing Literature on UHPC, HSC, and Normal Strength Concrete for Compressive Strength at High Temperatures Based on Graph in [106].

In order to model the behavior recorded in this study, a series of equations were derived to approximately predict the residual compressive strength versus temperature plot. This series of linear equations can be seen in Equation 3.5:

\[
\frac{f'_c(T)}{f'_c(90^\circ C)} = \begin{cases} 
-6.45 \times 10^{-5}T + 1.006 & 90 < T \leq 400 \\
-2.20 \times 10^{-3}T + 1.860 & 400 \leq T \leq 800 \\
-6.00 \times 10^{-4}T + 0.580 & 800 \leq T \leq 900 
\end{cases}
\]

Where:
- \( f'_c(T) \): Residual Compressive Strength at temperature \( T \)
- \( f'_c(90^\circ C) \): Residual Compressive Strength of control at 90°C
- \( T \): Temperature in °C
Similar to the comparisons between series, these are relative values which have been normalized with respect to the compressive strength. This allows for a more representative comparison between changes of the experimental series and the proposed predictions. These equations were modeled after the behavior of the residual compressive strength with increasing temperature. A comparison between the experimental data and the predictions of these equations is seen in Figure 3.27.

![Experimental Data vs. Predictions of Equation 3.5](image)

**Figure 3.27 – Experimental Results vs. Equation 3.5 predictions**

While the experimental data showed an increase up to 200°C in the DS series and 300°C in the HS and HF series, these increases may not necessarily occur in every situation. Therefore, a linear relationship negating these features was chosen. This was done in order to safely predict the residual compressive strength of concrete after heat loading. Equation 3.5 was also compared to the existing research shown in Figures 3.25 in Figures 3.28.
Figure 3.28 – Equation 3.5 Predictions vs. Existing Literature on UHPC Compressive Strength at High Temperatures Based on Graph in [107].

This graph shows reasonable agreement between the equation 3.5 predictions and the existing literature on UHPC at elevated temperatures. While there are variations between this data and the predictions, the general trend approximately fits the data shown.

### 3.6 Residual Elastic Modulus

The residual elastic modulus is considered to be the slope of the linear portion of the elastic region of the stress-strain curve following heating to the prescribed temperature and returning to ambient temperature. The value presented is the average of the three tests heated to a given temperature.
3.6.1 DS Series

The DS series plot for residual elastic modulus with respect to temperature can be found in Figure 3.29 with corresponding data located in Table 3.11. This shows a plot of three individual test results with an average curve overlaid.

Results from the residual elastic modulus tests showed that, similar to the residual compressive stress results, the elastic modulus increased with increasing temperature. However, this increase was less pronounced that of the residual compressive strength. The DS series showed a maximum of a 5.5% increase in the residual elastic modulus for specimens in the 135ºC DS series. The maximum residual elastic modulus occurred in this series with a value of 56.78GPa. After this maximum, the elastic modulus decreased to 84.7% and 56.2% of the control values by 300ºC and 500ºC, respectively. By 800ºC and 900ºC, the residual elastic modulus decreased to only 4.1% and 2.7% of the control value, respectively.
Table 3.11 – Residual Elastic Modulus Data from DS Series with Increasing Temperature

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$E$ (GPa)</th>
<th>Average (GPa)</th>
<th>Standard Deviation</th>
<th>$E$ (ksi)</th>
<th>Average (ksi)</th>
<th>Standard Deviation</th>
</tr>
</thead>
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<td>2.88</td>
<td>8275.96</td>
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</tr>
<tr>
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<td>7480.53</td>
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</tr>
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<td>8356.97</td>
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</tr>
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<td>54.18</td>
<td>4.68</td>
<td>7175.15</td>
<td>7858.26</td>
<td>2.03</td>
</tr>
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<td>45.56</td>
<td>1.22</td>
<td>6810.59</td>
<td>6608.33</td>
<td>3.02</td>
</tr>
<tr>
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<td>46.96</td>
<td>45.56</td>
<td>1.22</td>
<td>6527.33</td>
<td>6487.08</td>
<td></td>
</tr>
<tr>
<td>300</td>
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<td>45.56</td>
<td>1.22</td>
<td>6527.33</td>
<td>6608.33</td>
<td>3.02</td>
</tr>
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<td>300</td>
<td>44.73</td>
<td>45.56</td>
<td>1.22</td>
<td>6487.08</td>
<td>6608.33</td>
<td>3.02</td>
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<td>4657.89</td>
<td>4382.66</td>
<td>0.52</td>
</tr>
<tr>
<td>600</td>
<td>20.87</td>
<td>21.46</td>
<td>0.63</td>
<td>3027.08</td>
<td>3100.76</td>
<td>3112.27</td>
</tr>
<tr>
<td>600</td>
<td>21.38</td>
<td>21.46</td>
<td>0.63</td>
<td>3100.76</td>
<td>3112.27</td>
<td>3112.27</td>
</tr>
<tr>
<td>600</td>
<td>22.13</td>
<td>21.46</td>
<td>0.63</td>
<td>3208.96</td>
<td>3112.27</td>
<td>3112.27</td>
</tr>
<tr>
<td>700</td>
<td>9.91</td>
<td>10.68</td>
<td>0.80</td>
<td>1436.63</td>
<td>1525.22</td>
<td>0.32</td>
</tr>
<tr>
<td>700</td>
<td>10.64</td>
<td>10.68</td>
<td>0.80</td>
<td>1549.40</td>
<td>1525.22</td>
<td>0.32</td>
</tr>
<tr>
<td>700</td>
<td>11.50</td>
<td>10.68</td>
<td>0.80</td>
<td>1647.34</td>
<td>1647.34</td>
<td></td>
</tr>
<tr>
<td>800</td>
<td>2.34</td>
<td>2.22</td>
<td>0.15</td>
<td>350.64</td>
<td>329.38</td>
<td>0.92</td>
</tr>
<tr>
<td>800</td>
<td>2.27</td>
<td>2.22</td>
<td>0.15</td>
<td>322.40</td>
<td>322.40</td>
<td>0.92</td>
</tr>
<tr>
<td>800</td>
<td>2.06</td>
<td>2.22</td>
<td>0.15</td>
<td>298.05</td>
<td>298.05</td>
<td></td>
</tr>
<tr>
<td>900</td>
<td>1.32</td>
<td>1.48</td>
<td>0.28</td>
<td>191.45</td>
<td>260.78</td>
<td>214.41</td>
</tr>
<tr>
<td>900</td>
<td>1.80</td>
<td>1.48</td>
<td>0.28</td>
<td>260.78</td>
<td>260.78</td>
<td>214.41</td>
</tr>
<tr>
<td>900</td>
<td>1.32</td>
<td>1.48</td>
<td>0.28</td>
<td>191.01</td>
<td>191.01</td>
<td></td>
</tr>
</tbody>
</table>

3.6.2 HS Series

The HS series graph for residual elastic modulus with respect to temperature can be found in Figure 3.30 and the corresponding data can be found in Table 3.12. This graph is a plot of the three individual test results with an average curve overlaid.
The HS series showed an increase in the elastic modulus with temperature up through 300°C. In this range, the maximum occurred at 135°C and was a 9.2% increase over the control value corresponding to an average residual elastic modulus of 52.07GPa. The elastic modulus remained at approximately this level though 200°C. By 300°C, it decreased to a value of 103.6% of the control.

After this increase, the residual elastic modulus decreased rapidly to 76.3% by 400°C and 39.9% of the initial value by 600°C. This decrease continued out to 700°C where the residual elastic modulus was calculated to have only 14.8% of the control.

Figure 3.30 – Residual Elastic Modulus of HS Series with Increasing Temperature
Table 3.12 – Residual Elastic Modulus Data from HS Series with Increasing Temperature

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>E (GPa)</th>
<th>Average (GPa)</th>
<th>Standard Deviation</th>
<th>E (ksi)</th>
<th>Average (ksi)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>44.25</td>
<td>47.68</td>
<td>3.84</td>
<td>47680.67</td>
<td>6915.50</td>
<td>557.26</td>
</tr>
<tr>
<td>90</td>
<td>46.97</td>
<td>52.07</td>
<td>1.05</td>
<td>49411.33</td>
<td>7552.74</td>
<td>152.40</td>
</tr>
<tr>
<td>135</td>
<td>52.83</td>
<td>52.04</td>
<td>2.27</td>
<td>6417.34</td>
<td>7547.85</td>
<td>329.27</td>
</tr>
<tr>
<td>300</td>
<td>46.51</td>
<td>49.41</td>
<td>2.66</td>
<td>7662.78</td>
<td>7166.51</td>
<td>385.84</td>
</tr>
<tr>
<td>400</td>
<td>37.01</td>
<td>36.39</td>
<td>0.73</td>
<td>7177.05</td>
<td>5277.34</td>
<td>106.16</td>
</tr>
<tr>
<td>500</td>
<td>23.35</td>
<td>26.66</td>
<td>2.87</td>
<td>6745.56</td>
<td>3866.03</td>
<td>416.47</td>
</tr>
<tr>
<td>600</td>
<td>18.32</td>
<td>19.02</td>
<td>1.56</td>
<td>5367.41</td>
<td>2758.67</td>
<td>226.64</td>
</tr>
<tr>
<td>700</td>
<td>7.06</td>
<td>7.06</td>
<td>0.061</td>
<td>3385.91</td>
<td>1024.60</td>
<td>8.96</td>
</tr>
</tbody>
</table>

3.6.3 HF Series

The HF series graph for residual elastic modulus with respect to temperature can be found in Figure 3.31 and the corresponding data can be found in Table 3.13. This graph is a plot of the three individual test results with an average curve overlaid.

While the data available for the HF series is limited, some information can be gleaned from the data present. Temperatures up to 300°C did not have an effect on the residual elastic modulus of the concrete. The tests only showed a fluctuation of ±1% from the control specimens.
up through 300ºC. However, due to the limited nature of this series, it cannot be determined whether this pattern would hold for higher temperatures.

![Residual Elastic Modulus vs. Temperature HF Series](image)

**Figure 3.31 – Residual Elastic Modulus of HF Series with Increasing Temperature**

**Table 3.13 – Residual Elastic Modulus Data from HF Series with Increasing Temperature**

<table>
<thead>
<tr>
<th>Temp (ºC)</th>
<th>$E$ (MPa)</th>
<th>Average (MPa)</th>
<th>Standard Deviation</th>
<th>$E$ (ksi)</th>
<th>Average (ksi)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>44.25</td>
<td>47.68</td>
<td>3.84</td>
<td>6417.34</td>
<td>6915.50</td>
<td>557.26</td>
</tr>
<tr>
<td>90</td>
<td>46.97</td>
<td></td>
<td></td>
<td>6811.84</td>
<td></td>
<td></td>
</tr>
<tr>
<td>90</td>
<td>51.83</td>
<td></td>
<td></td>
<td>7517.31</td>
<td></td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>48.38</td>
<td>47.55</td>
<td>1.27</td>
<td>7177.05</td>
<td>6896.35</td>
<td>183.87</td>
</tr>
<tr>
<td>200</td>
<td>46.09</td>
<td></td>
<td></td>
<td>6684.77</td>
<td></td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>48.17</td>
<td></td>
<td></td>
<td>6986.88</td>
<td></td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>49.73</td>
<td>48.40</td>
<td>1.88</td>
<td>7212.62</td>
<td>7019.52</td>
<td>273.08</td>
</tr>
<tr>
<td>300</td>
<td>47.07</td>
<td></td>
<td></td>
<td>6826.42</td>
<td></td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>44.25</td>
<td></td>
<td></td>
<td>6417.34</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### 3.6.4 Comparisons

The residual elastic modulus versus temperature plots presented in this section were normalized with respect to the elastic modulus order to show the relative changes in elastic
modulus as opposed to the absolute changes. This was done by dividing the elastic modulus of each temperature series by that of the control value.

The HS and HF series are compared in Figure 3.32. Specimens from the HF series showed a residual elastic modulus which was 9.2% less than those in the HS series at 200ºC. By 300ºC, this disparity decreases to only 2.1%. Experimental data from these comparisons can be seen in Table 3.14.

![Figure 3.32 – Residual Elastic Modulus of HF Series vs. HS Series](image)

Table 3.14 – Residual Elastic Modulus Data from HF Series vs. HS Series with Increasing Temperature

<table>
<thead>
<tr>
<th>Temp (ºC)</th>
<th>HS</th>
<th>HF</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>1.000</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>200</td>
<td>1.091</td>
<td>0.997</td>
<td>0.09421</td>
</tr>
<tr>
<td>300</td>
<td>1.036</td>
<td>1.015</td>
<td>0.02125</td>
</tr>
<tr>
<td>400</td>
<td>0.763</td>
<td></td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>0.559</td>
<td></td>
<td></td>
</tr>
<tr>
<td>600</td>
<td>0.399</td>
<td></td>
<td></td>
</tr>
<tr>
<td>700</td>
<td>0.148</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comparisons between the residual elastic modulus of DS and HS series can be found in Figure 3.33. The difference between these two series was the increased range of the elevated elastic modulus. In the DS series, the increase in the elastic modulus only occurred for specimens heated to 135ºC before returning to approximately the control value. However, for the HS series, the elastic modulus remained at least 103% of the control until 300ºC. In this range, there was a difference between the HS and DS series of 19%. At higher temperatures, the two series become
more similar with only a 5% fluctuation seen at 700°C. Experimental data from these comparisons can be seen in Table 3.15.

![Figure 3.33 – Residual Elastic Modulus of DS Series vs. HS Series](image)

### Table 3.15 – Residual Elastic Modulus Data DS Series vs. HS Series with Increasing Temperature

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>HS</th>
<th>DS</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>1.000</td>
<td>1.000</td>
<td>0.000</td>
</tr>
<tr>
<td>135</td>
<td>1.092</td>
<td>1.055</td>
<td>0.037</td>
</tr>
<tr>
<td>200</td>
<td>1.091</td>
<td>1.007</td>
<td>0.085</td>
</tr>
<tr>
<td>300</td>
<td>1.036</td>
<td>0.847</td>
<td>0.190</td>
</tr>
<tr>
<td>400</td>
<td>0.763</td>
<td>0.723</td>
<td>0.040</td>
</tr>
<tr>
<td>500</td>
<td>0.559</td>
<td>0.562</td>
<td>-0.003</td>
</tr>
<tr>
<td>600</td>
<td>0.399</td>
<td>0.399</td>
<td>0.000</td>
</tr>
<tr>
<td>700</td>
<td>0.148</td>
<td>0.199</td>
<td>-0.050</td>
</tr>
<tr>
<td>800</td>
<td>0.041</td>
<td>0.041</td>
<td>-0.041</td>
</tr>
<tr>
<td>900</td>
<td>0.027</td>
<td>0.027</td>
<td>-0.027</td>
</tr>
</tbody>
</table>

A comparison between the residual elastic modulus results from this experimentation and the current literature on ultra-high performance concrete can be found in Figure 3.34. [107] Here, existing tests on the elastic modulus of various commercially available UHPCs are plotted with respect to temperature. These results varied from the data obtained from this research by the lack of an increase in elastic modulus before 300°C. The existing literature showed a purely downward trend in elastic modulus with increase in temperature. This information is only for a comparison to other existing research. The methods and materials used in these other studies may or may not match with those used in this research.
Figure 3.34 – Experimental Results vs. Existing Literature on UHPC elastic modulus at High Temperatures Based on Graph in [107].

Figure 3.35 shows a comparison of the residual elastic modulus of a normal and high strength concrete with the research conducted in this study. [133] Literature reported a similar trend to the existing research on ultra-high performance concrete. Data on normal and high strength concrete showed a downward trend at with any heat applied. This varied from the experimental results presented in this study by the lack of an increase in elastic modulus with temperature up to 300°C. This information is only for a comparison to other existing research. The methods and materials used in these other studies may or may not match with those used in this research.
In order to model the behavior recorded in this study, a series of equations were derived to approximate the residual elastic modulus versus temperature plot. This series of linear equations can be seen in Equation 3.6:

\[
\frac{E(T)}{E(90^\circ C)} = \begin{cases} 
1 & 90 \leq T \leq 200 \\
-1.60 \times 10^{-3} T + 1.32 & 200 \leq T \leq 800 \\
-2.00 \times 10^{-4} T + 0.20 & 800 \leq T \leq 900
\end{cases} 
\]

Where: 
\( E(T) \): Residual Elastic Modulus at temperature \( T \) 
\( E(90^\circ C) \): Residual Elastic Modulus of control at 90\(^\circ\)C 
\( T \): Temperature in \(^\circ\)C

These equations were modeled after the behavior of the residual elastic modulus with increasing temperature. As with the comparisons, these equations produce a non-dimensional value which has been normalized to the elastic modulus of the control. A comparison between the experimental data and the predictions of these equations is seen in Figure 3.36.
While the residual elastic modulus increased up to 200°C in the DS series and 300°C in the HS and HF series, these increases may not necessarily occur in every situation. Additionally, existing research did not show these increases. Therefore, in order to ensure a safe design for concrete at elevated temperatures, a linear relationship negating these features was chosen.

Equation 3.6 was also compared to the existing research shown in Figure 3.34 in Figure 3.37.
This graph shows reasonable agreement between the equation 3.6 predictions and the existing literature on UHPC at elevated temperatures. Even though there are variations between this data and the predictions, the general trend is in an appropriate region and direction.

### 3.7 Residual Compressive Strains

For the purposes of this research, the residual compressive strains are defined as the strain when the compressive strength of the specimen is reached. In order to make an accurate comparison the compressive strains in this section have been made non-dimensional by normalizing them with respect to the compressive strain of the control. A plot of residual compressive strains for all series is shown in Figure 3.38 along with corresponding data in Table 3.16. Each series was produced by plotting the compressive strain for the average of each temperature series. Additionally, the linear regression plotted will be used to predict the compressive strains with increasing temperature. This prediction is shown in Equation 3.7:
\[
\frac{\varepsilon_{cm}(T)}{\varepsilon_{cm}(90^\circ C)} = 2.68 \times 10^{-3}T + 0.778
\]  

(3.7)

Where:

- \(\varepsilon_{cm}(T)\): Compressive Strain at temperature T
- \(\varepsilon_{cm}(90^\circ C)\): Compressive Strain of control at 90°C
- T: Temperature in °C

Table 3.16 – Compressive Strains for DS, HS, and HF Series

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>DS</th>
<th>HS</th>
<th>HF</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>0.004606</td>
<td>0.004726</td>
<td>0.004726</td>
<td>0.004686</td>
</tr>
<tr>
<td>135</td>
<td>0.005825</td>
<td>0.005383</td>
<td>n/a</td>
<td>0.005604</td>
</tr>
<tr>
<td>200</td>
<td>0.006612</td>
<td>0.005473</td>
<td>0.005641</td>
<td>0.005909</td>
</tr>
<tr>
<td>300</td>
<td>0.006003</td>
<td>0.008209</td>
<td>0.00576</td>
<td>0.006657</td>
</tr>
<tr>
<td>400</td>
<td>0.006918</td>
<td>0.008603</td>
<td>n/a</td>
<td>0.007761</td>
</tr>
<tr>
<td>500</td>
<td>0.009521</td>
<td>0.010653</td>
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<td>0.010087</td>
</tr>
<tr>
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<td>0.013164</td>
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<td>0.011737</td>
</tr>
<tr>
<td>700</td>
<td>0.011369</td>
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<td>n/a</td>
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<tr>
<td>800</td>
<td>0.014</td>
<td>n/a</td>
<td>n/a</td>
<td>0.014</td>
</tr>
<tr>
<td>900</td>
<td>0.014142</td>
<td>n/a</td>
<td>n/a</td>
<td>0.014142</td>
</tr>
</tbody>
</table>

Figure 3.38 – Compressive Strains vs. Temperature
3.8 Model Code Implementation

The *fib* Model Code was compared to each of the strain adjusted stress-strain curves. These results are shown in the Stress-Strain Curves section of this chapter. These comparisons showed the Model Code was capable of predicting the stress-strain behavior of each series up to rupture given the residual compressive strength, elastic modulus, and compressive strain values of that temperature and mix series. However, after rupture the Model Code was no longer adequate.

The Model Code, with the aid of relationships presented in equations 3.5-3.7, can also be used to produce an approximate stress-strain relationship given only the control values for the compressive strength, elastic modulus, and compressive strain. Three examples for this are presented using the control data for the DS, HS, and HF series. These Model Code plots were graphed against the original stress-strain data and strain corrected stress-strain data for that mix and temperature range. These are presented in Figure 3.39-3.41.

![Figure 3.39 – Mode Code with Predicted Values for Control HS Series](image-url)
Figure 3.40 – Model Code with Predicted Values for 300°C HF Series

Figure 3.41 – Model Code with Predicted Values for 800°C DS Series
These figures show that while the Model Code predictions with equations 3.5-7 implemented do not match the experimental data exactly, the comparison is reasonable. The curves did not fit as well as when the data from each corresponding test was used. A possible cause for the discrepancy between the predicted and the experimental compressive strain was due to the variability in the residual compressive strain data shown above in Figure 3.38. For all series, the compressive strain is within 27% of the experimental values.

The Model Code curves also varied by their inability to capture the pre-elastic hardening phase. This is seen in higher temperature series, where the pre-elastic hardening phenomenon was more noticeable. An example of this is seen above in Figure 3.41.

3.9 DSC/TGA

The DSC/TGA was run from room temperature to 1000ºC at a ramp rate of 40ºC/min in an air atmosphere containing 80% nitrogen and 20% oxygen. Once the samples were heated to the maximum temperature, they remained there for one hour before the furnace in the unit was powered off and the specimens returned to room temperature.

3.9.1 Ductal

The Thermo Gravimetric Analysis of Ductal showed a total mass loss of 5.22%. A stepwise breakdown of the mass loss over specific ranges is seen in Figure 3.42. Here, the percentage mass loss is plotted against increasing temperature. Specific dehydrations are referenced from Table 1.2.
The span of 20 to 200ºC showed a loss of 2.14% of the initial mass. During this range, the evaporable water is driven off and the C-S-H gel is dehydrated. Between 200ºC and 305ºC, the plot showed a loss of 0.93% of the initial mass. It is likely that this loss in mass was due to the dehydration of 11.3 Å tobermorite to 9.3Å tobermorite.

Another sharp decrease was visible between 400 and 500ºC. Over this range, the decomposition of portlandite begins and progresses until it is completely decomposed around 550ºC. Additionally, hillebrandite dehydrates into larnite between 490ºC and 665ºC. The range of 400 to 665ºC accounted for a mass loss of 1.3%. Between 600 and 700ºC, hillebrandite finishes decomposing to larnite. Additionally, the disorganized jennite and 9.3Å tobermorite release carbon dioxide during this range. Between 700 and 810ºC, 9.3Å tobermorite and...
disordered jennite decomposes to low-t wollastonite. In the range of 650 and 900°C, C-S-H (II) decomposes into larnite and wollastonite. The ratio of larnite to wollastonite formed is closely tied with the silica ratio in the concrete. At 900°C, the disordered jennite remnants break down into larnite.

The differential scanning calorimetry plot, shown in Figure 3.43 serves a similar purpose. This plot shows more of a change in thermal properties rather than a specific decomposition or liberation of mass. This process plots the change in specific heat as a function of temperature. The specific heat of a material changes as the material itself changes. Therefore, this makes the DSC a useful tool in determining the change in microstructure of dehydrating concrete.

Figure 3.43 – Differential Scanning Calorimetry Plot for Ductal Mix
The decomposition of concrete is mostly characterized by water loss. Therefore, the DSC plot shows many of the same features as the TGA. However, in some cases, there are mineralogical shifts which decrease strength in addition to dehydration reactions. The most noted of these is the transition from $\alpha$-phase to $\beta$-phase of quartz at roughly 573°C. This was clearly seen by the peak on the DSC plot at roughly 561°C. There is another peak around roughly 858°C. Despite research which indicates that producing tridymite is difficult to achieve without reagents, there is very little associated mass loss with this reaction. It is, therefore, possible that this represented the transition of the $\beta$-phase quartz to Tridymite.

### 3.9.2 House Mix

The Thermo Gravimetric Analysis of the House mix showed a total mass loss of 6.02%. This represents a larger mass loss than the Ductal mix. A stepwise breakdown of the mass loss over specific ranges is seen in Figure 3.44. Here, the percentage mass loss is plotted against increasing temperature. Many of the same reactions that occurred in Ductal are seen here in the House TGA. The differences are the amount of overall mass lost from each section. Certain regions where reactions or decompositions are expected to take place are examined. These points of interest are referenced from Table 1.2.
Figure 3.44 – Thermo Gravimetric Analysis: Stepwise Mass Loss of House Mix

The temperature range from 20ºC to 200ºC showed a loss of 1.38%. Over this range, the evaporable water is driven off and C-S-H gel is dehydrated. The 200ºC to 305ºC temperature range showed a loss of 0.94% which likely corresponded to the dehydration of 11.3Å tobermorite to 9.3Å tobermorite.

The 300ºC to 450ºC range in the Ductal TGA showed little activity while the House mix TGA showed a large mass loss. This likely corresponds to the transformation of metajennite into the disordered jennite phase. This mass loss corresponds to a 1.42% decrease in mass of the specimen.

Between 450ºC and 600ºC, the TGA showed a gradual decrease which corresponds to the dehydration of the portlandite. However, the reaction between 490ºC and 665ºC may also show the decomposition of hillebrandite to larnite. The total mass loss between 450ºC and 665ºC was
1.41%. Another mass loss occurred between 650°C to 700°C. This range likely corresponds to the beginning of a final decomposition of C-S-H(II) into larnite and wollastonite as well as disordered jennite and 9.3 Å tobermorite releasing carbon dioxide. The mass loss across between 650°C and 900°C was 1.15%.

The DSC plot for the House mix is shown in Figure 3.45. This plot reinforces the assumptions made based on the TGA study. The transition to 9.3 Å tobermorite is more visible in the DSC plot than in the TGA. The transition of metastable jennite to the disordered jennite phase is visible as a strong reaction in the 350°C to 450°C range. The recrystallization of disordered jennite and 9.3 Å tobermorite is visible after 780°C. The same quartz phase transition of α-phase to β-phase quartz is seen at 564°C. However, the phase change to tridymite is no longer visible.

![Figure 3.45 – Differential Scanning Calorimetry Plot for House Mix](image-url)
Chapter Four – Conclusions

4.1 Summary

Ultra-high performance fiber reinforced concrete is a relatively new composite which has only recently begun to be implemented in the construction world. The research on this material with respect to fire is limited. This research was designed to increase the knowledge on the effects of high temperatures on two different ultra-high performance fiber reinforced concrete mixes by separately examining the chemical degradation and the residual mechanical properties.

Two concrete mixes were tested for residual mechanical strength and thermochemical degradation at elevated temperatures. Changes in mechanical behavior were determined through examination of the residual elastic modulus, residual compressive strength, and the residual stress-strain behavior. Data from the compressive tests were used to produce average stress-strain curves as well as the residual elastic modulus and compressive strength for each temperature series. This data provided the basis for a semi-empirical model of the temperature dependent stress-strain behavior for UHPC using the *fib* Model Code..

Thermochemical analyses were performed using DSC and TGA measurements. The DSC measured changes in specific heat of the specimens in order to track phase changes and chemical dehydrations/degradations. The TGA was used to monitor mass loss in order to determine dehydrations which occurred at elevated temperatures. This data was then coupled with literature resources in order to approximately identify the chemical reactions taking place in the concrete specimens.

4.2 Observations

Throughout the course of the thermal and destructive testing, it was found that:
1. The color and material texture of specimens exposed to higher temperatures than 300°C visibly changed. The failed concrete material at temperatures over 800°C more closely resembled sand than fractured concrete and fibers were little more than black dots on the failed specimens surface.

2. Series under fast heating (HF) specimens heated to 300°C exhibited spalling behavior despite the presence of steel fibers.

3. Stress-strain curves for series heated beyond 200°C showed pre-elastic hardening. While the cause could not be substantiated with other existing research, the phenomenon appears in both mixes tested.

4. All strain corrected series matched the Model Code approximately up to fracture. The Model Code was incapable of capturing the strain softening behavior of the more ductile series.

5. Specimens heated to higher temperatures tended to show greater ductility and a longer strain softening region than the lower temperature specimens. This held true for both the DS and the HS series.

6. The residual compressive strength for slowly heated specimens increased to 59.9% at 300°C for HS specimens and 43.5% at 200°C for DS specimens over the control. The compressive strength for both series reduced to approximately the control around 500°C before decrease afterwards. This differed from existing research on both UHPCs and normal strength/high strength concretes due to the lack of an increase before 500°C. This was possibly due to the heating rate examining only the chemical and material degradation.
7. The HS series showed a larger increase in residual compressive strength than the DS or HF series did. The HS series showed a 59.9% increase over the control series, while DS and HF showed a 43.5% and 31.1% increase, respectively.

8. While the residual compressive strength at 500ºC was still within 10% of the control value for both HS and DS series, the elastic modulus had reduced by over 44.9% and 43.8%, respectively.

9. The residual elastic modulus increased over the control series for both the HS and DS series up to 300ºC and 135ºC, respectively. However, the increase was less notable than the increase seen in the residual compressive strength increasing only a maximum of 9.2% for the HS series and 5.5% for the DS series.

10. The residual elastic modulus for HS series maintained an increase for specimens up to 300ºC, while the DS series showed maintained the increase to 200ºC.

11. The HF series showed very little change in elastic modulus, fluctuating only ±1% over the three temperature ranges tested.

12. The TGA for the House series showed a larger mass loss than that of the Ductal specimens.

13. Both the residual compressive strength and elastic modulus were predicted using semi-empirical relationships. This facilitated the prediction of the residual stress-strain behavior of UHPFRC at elevated temperatures with a focus on the chemical degradation only.

4.3 Conclusions

The following conclusions were drawn based on the observations of the mechanical and thermochemical tests:
1. As in normal strength concretes, the color of concrete above critical temperature ranges can be used to determine the level of damage to a concrete as the first noticeable color changes occur around the point when the residual compressive strength begins to decrease.

2. The pre-elastic strain hardening affect appears to be influenced by temperature. These pre-elastic strains may make behavior unpredictable when comparing to a model code without some strain based curve adjustments. However, the cause and mechanisms behind them are not well understood and require additional research to understand completely.

3. DS series specimens showed a decrease in the residual elastic modulus resulting in a softer matrix after 200°C. This same change occurred in HS series specimens after 300°C. This was likely caused by the chemical deterioration and dehydration in the matrix.

4. Specimens which were oven dried showed better compressive strength than those which were not. This is likely caused by the removal of incompressible water from the matrix allowing the specimen to compress without having to withstand the tensile forces caused by compressing water on the matrix during loading.

5. There was an increase in residual compressive strength for the range of 135°C to 200°C for the DS series and 135°C to 300°C for the HS series. The reasoning behind these low heat gains was unclear in both this research and in the literature. This area of knowledge requires additional investigation into microstructural changes of the UHPFRC at these temperature ranges. The increase in strength may be a result of the decomposition of the C-S-H gel at roughly 180°C.
6. Strength losses in Ductal after 200°C are likely due to the decomposition of 11.3Å tobermorite into 9.3Å tobermorite. Strength loss after 500°C is likely the result of microcracking caused by the swelling of the silica sands due to the α-β phase transition as well as the decomposition of the portlandite into lime and water. The strength loss at after 700°C is likely due to the final dehydrations and recrystallizations from the 9.3Å tobermorite, jennite, and C-S-H (II) into larnite and wollastonite.

7. Strength losses after 300°C in House specimens are likely due to the decomposition of metajennite into the disordered jennite phase. The strength losses after 500°C are likely caused by the further development of a more disordered jennite phase in addition to the α-β phase transition as well as the decomposition of the portlandite into lime and water.

8. The difference between residual compressive strength in the HS and the HF series is likely attributed to the vapor pressure damage and thermal gradient damage caused by rapid heating. This most likely led to failure through the extensive microcracking due to the increase in internal pressure caused by temperature variations and vapor pressure within the UHPFRC’s dense pore structure. While further HF and DF tests beyond 300°C were not conducted, due to the failure of specimens at this point, it is reasonable to assume that further heating to higher temperatures at the same rate would result in more spalled specimens which could not be compressive tested.

9. Using the *fib* Model Code and equations 3.5-3.7, stress-strain curves can be produced with the only input being the temperature of exposure, the compressive strength, elastic modulus, and compressive strain as inputs. While these curves do not fit exactly to the experimental data, the results are within 27% difference for the
compressive strain values. The Model Code predictions also vary for specimens which have extensive pre-elastic hardening. Specimens at higher temperatures would require strain-based curve corrections to match the curve more closely. These predictions and the proposed equations allow for a stress-strain curve to be implemented into a finite element software for analysis.
References


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