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THE ALKALI-SILICA REACTION; A STUDY OF REACTIVE AGGREGATES AND PRODUCTION OF EXPANSIVE CONCRETE SPECIMENS

By

D. ROBB SPARKS

B.S., University of Colorado, 2016

A thesis submitted to the

Faculty of the Graduate School of the

University of Colorado in partial fulfillment

Of the requirement for the degree of

Master of Science

Department of Civil and Environmental Engineering

2016

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This thesis entitled:

The Alkali-Silica Reaction; A Study of Reactive Aggregates and

Production of Expansive Concrete Specimens

written by Donnie Robb Sparks

has been approved by the Department of Civil and Environmental Engineering

_____

Prof. Victor Saouma

_____

Prof. Mija Hubler

_____

Prof. Yunping Xi

Date_____

The final copy of this thesis has been examined by the signatories, and we find that both the content and the form meet acceptable presentation standards of scholarly work in the above mentioned discipline.

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Sparks, Donnie Robb (M.S., Civil, Environmental and Architectural Engineering)

The Alkali-Silica Reaction; A Study of Reactive Aggregates and

Production of Expansive Concrete Specimens

Thesis directed by Professor Victor Saouma

As part of an ongoing research effort to evaluate the risk represented by potential shear strength degradation of reinforced concrete containment vessels in US nuclear power plants due to the alkali silica reaction (ASR), a number of reactive concrete test specimens are produced. The ASR expansion potential of reactive aggregates sourced from two concrete suppliers drawing materials from quarries along the Gunnison River in Colorado is evaluated. A testing program for evaluating the ASR activity of concrete produced from said aggregates is introduced. Five reactive concrete mix designs are generated and subjected to this testing program with the objective of producing a highly expansive concrete with mechanical properties suitable to experimental use. These candidate concrete mixes are produced with two cements of varying alkalinity and some have their alkalinity further boosted through the addition of sodium hydroxide. A control concrete design is also developed which relies on lithium nitrate to prevent ASR expansion. Concrete prisms are cured either immersed in 1M $NaOH_{(aq)}$ at 80°C or uncovered in a fog room at 21°C and relative humidity greater than 90%. A testing apparatus capable of destructively fracturing test specimens in shear (simulating static earthquake load) while simultaneously applying a perpendicular force (representing the weight of the structure above) is prepared. Finally, a program for curing test specimens is presented which is intended to accelerate ASR expansion my minimizing alkali leaching.

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DEDICATION

To my beloved wife

Ellen Sparks

And to all of my family

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ACKNOWLEDGEMENTS

My sincerest gratitude and respect are extended to my advisor, Professor Victor E. Saouma who provided both the vision for this research project and countless hours of council, guidance, and supervision. His vast experience, conscientious advice, and benevolent criticism have helped me grow as both a researcher and as a person.

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Additional thanks are due to Doug Wolf of Whitewater Building Materials of Grand Junction, Colorado generously arranged the donation of several batches of sand and gravel over the course of the project. Sam

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Rendon and Tony Spafford of Brady Trucking Inc., of Vernal Utah coordinated for donation of transportation services. Holcim of Hagerstown, Maryland generously donated cement for project use. Finally, Bill Hart of Grace Concrete Products coordinated for donation of lithium nitrate admixture. To all of them, and everyone else associated with this project, please accept my most heartfelt thanks.

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The views and opinions expressed in this thesis are the author's own and do not reflect the views of the Nuclear Regulatory Commission or any agency of United States Government.

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CHAPTER 1

INTRODUCTION

1.1 Motivations

The alkali-aggregate reaction has long been recognized as a cause of concrete degradation (Stanton, 1940). In the past few years, ASR-induced damage has been observed in nuclear power plant (NPP) containment vessels such as that at the Seabrook NPP (Saouma & Hariri-Ardebili, 2014).

Operating licenses for nuclear power plants in the United States are issued for 40 years to which may be added an unlimited number of 20-year extensions (NUREG-980, 2013). Several US NPPs have received such extensions, prolonging structural service life to 60 years and further extensions may be granted (U.S. Energy Information Administration, 2016). Of course, it is of critical concern that concrete containment vessels can be relied upon throughout the extension period.

While the effects of ASR on compressive strength, tensile strength, and elastic modulus of concrete have been examined, its influence on shear strength is not yet well established. Characterization of the effects of ASR degradation on the ability of an NPP containment vessel to resist transverse earthquake loading is of pressing concern. Such a study requires production of ASR-degraded concrete specimens which are reasonably representative of containment vessel walls.

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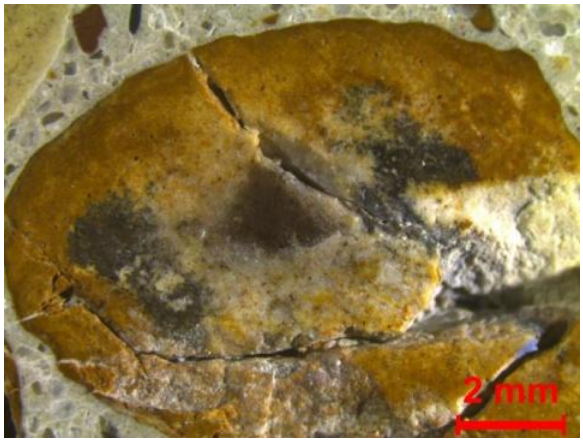

Figure 1 – ASR damage in chert coarse aggregate.

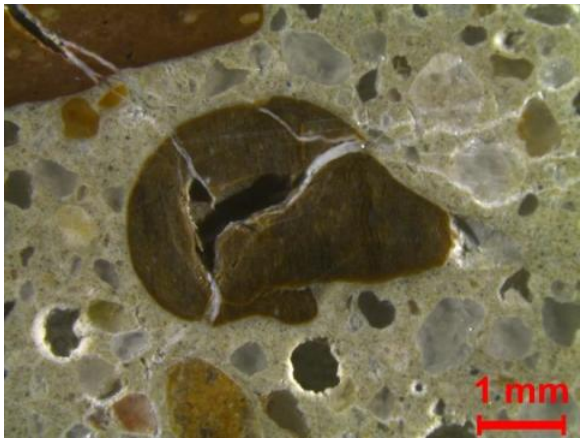

Figure 2 – Chert aggregate partially consumed by ASR

1.2 Research Objectives

This study is part of an ongoing research effort to quantify the risk of earthquake damage to NPP containment vessels degraded by ASR. Samples of a model concrete that have undergone ASR expansion are to be tested in biaxial shear and the results contrasted with similar control specimens to which an admixture has been added to prevent ASR.

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The specific goal of this thesis is the production of a reactive concrete mix, whose propensity for ASR expansion is well-documented. A source of reactive aggregates is identified and the expansive potential of these aggregates evaluated. Suitable test specimens, some of which are reinforced, are constructed. A shear test apparatus is refurbished and assembled for use in the upcoming test program. Conditions under which reactive samples are to be stored to achieve ASR expansion targets is proposed. Finally, the potential of lithium nitrate admixture for halting ASR reactivity for purpose of constructing control specimens is evaluated.

1.3 Thesis Organization

This thesis is comprised of seven chapters and two appendices. This chapter presents factors motivating the project as well as the specific objectives of the thesis. Subsequent chapters are organized by task and the actions described therein are not necessarily presented chronologically.

CHAPTER 2 presents a selection of background information and a literature review of prior research on the topic. Included is a brief description of two standard ASTM testing methods which were used in modified form during this study as well as a summary of criticism of these and other commonly accepted ASR tests.

CHAPTER 3 discusses a prototype NPP containment vessel how it is scaled down to a model system. The apparatus to be used for shear testing experimental specimens is discussed, as well as the process of refurbishing, assembling, and preparing the apparatus for use. The design and production of shear specimens is discussed in detail, to include design and construction of reinforcement, and formwork. A plan for casting experimental specimens is presented along with an outline of required materials. Finally, a recommended plan for storing specimens such that ASR expansion will be maximized is presented.

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CHAPTER 4 discusses the process of identifying a supply of reactive aggregates. A test procedure to evaluate the reactivity of aggregates using standard mortar bar tests is described in detail. Samples of aggregate from two suppliers are subjected to the test program and the results tabulated. A supplier is selected and factors motivating the decision discussed.

CHAPTER 5 outlines an experimental program to test aggregates prior to casting and to evaluate the ASR reactivity of concrete mix designs. Standardized ASTM test procedures are preferred when appropriate, but necessary modifications to these procedures are discussed when appropriate. Included are procedures for use of admixtures to further boost or retard ASR.

CHAPTER 6 represents the bulk of this thesis. Five candidate concrete mixes are presented and subjected to the test program proposed in CHAPTER 5. Mix design is an iterative process, with the results of testing each mix informing subsequent designs. The result of this testing is adoption of a reactive concrete mix suitable for production of ASR-reactive experimental specimens.

CHAPTER 7 contains conclusions and recommendations for future research as well as some personal observations.

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CHAPTER 2

LITERATURE SURVEY

2.1 Introduction

The alkali-silica reaction (ASR) was first identified by Stanton (1940) as a source of long-term concrete degradation. ASR occurs when alkali constituents of cement, admixtures, or the environment react with amorphous, disordered or poorly crystallized silica present in aggregates in a sufficiently moist environment. The product of the reaction is a hygroscopic silica gel which combines with water and expands. If a sufficient quantity of alkali and reactive silica are present, the reaction causes formation of gel-filled microcracks and bulk expansion of the concrete.

$$xSiO_2 + yNa(K)OH \rightarrow Na(K)_ySi_xO_z$$

$$Na(K)_ySi_xO_z + wH_2O \rightarrow Na(K)_ySi_xO_z \cdot wH_2O$$

The three necessary components for ASR expansion are: (1) reactive minerals in the aggregate, (2) sufficient alkali in the pore solution, either from cement or the environment (3) sufficient water or humidity to hydrate resulting gels (Hobbs, 1988).

The rate at which the reaction proceeds is strongly dependent on temperature. Larive (1998) proposed a model for reaction kinetics based the results of extensive experimentation. Larive's model suggests a given concrete may reach a degree of expansion requiring over 6 years when stored at low temperature (7 °C) in less than a year when stored at higher temperature (38°C). This result corresponds to a general observation that dams built in hotter climates seem to suffer from ASR at earlier ages than those built in

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colder climates or higher elevations in accordance with a constitutive model proposed by Saouma & Perotti (2006). Numerical modelling of ASR has also been addressed by Saouma (2013).

ASR has been observed in many dams and bridges worldwide. Some of the affected structures are the Hanshin Expressway in Japan (Clark, 1989), Fontana Dam in the U.S. (Wagner & Newell, 1995), Mactaquac Dam in Canada (Gilks & Curtis, 2003), Canning Dam in Australia (Shayan, Wark, & Moulds, 2000) and in Iran (Jabarooti & Golabtoonchi, 2003). Unfortunately, repair of degraded structures is often not possible.

2.2 Effects of ASR on Mechanical Properties of Concrete

The influence of ASR-induced expansion on the mechanical properties of concrete has been the subject of numerous studies (Swamy & Al-Asali, 1988) (Monette, 1997) (Larive, 1998) (Ahmed, Burley, Rigden, & Abu-Tair, 2003) (Multon, 2004). These studies involved a range of natural aggregates, and in the case of Swamy & El-Asali, an artificial aggregate comprised of amorphous fused silica. Water-to-cement ratios varied between 0.30 and 0.61 while alkali content ranged between 0.40% and 2.25% as Na_2O. Storage conditions were typically 38°C and high relative humidity or immersed in water. None of the cited studies took measures explicitly intended to prevent alkali leaching.

A recent statistical analysis of data sets produced by the cited authors, along with several others, by Esposito et. al. (2016) reveals a complex relationship between compressive strength and the unrestrained expansion of concrete prisms under laboratory conditions. For low to moderate expansions ($\varepsilon < 0.05\%$), compressive strength increases to a peak roughly 15% higher than that of unaffected concrete, though experimental data exhibits significant scatter depending on test conditions. At greater expansions, compressive strength declines approximately linearly with respect to prism elongation to a minimum of

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around 46% of its original value at extreme elongation ($\varepsilon < 2.50\%$). Swamy and El-Asali caution that "compressive strength is not a good indicator of the initiation or progress of ASR."

The elastic modulus of ASR-degraded concrete tends to decline significantly as expansion increases. For small expansions ($\varepsilon < 0.03\%$) stiffness is either unchanged or marginally increased. At higher expansions ($\varepsilon < 0.10\%$), elastic modulus declines almost linearly with increasing expansion until stiffness is degraded about 90%. Esposito feels that elastic modulus degradation provides a better indication of ASR progress than does compressive strength.

Tensile strength as measured by a splitting tensile test is also reduced by ASR, but without the strengthening at small expansions. Rather, tensile strength declines nonlinearly to about 64% of its original value at high expansions ($\varepsilon \approx 0.5\%$). Greater expansions have little effect on tensile strength, according to Esposito's analysis.

The effects of ASR expansion on shear strength are a subject of ongoing research. Some studies have concluded that ASR has a negligible effect on the shear strength (Bach, Thorsen, & Nielsen, 1993). Other researchers have observed a slight reduction in shear capacity when considering reinforced structural members suffering ASR degradation (den Uijl & Kaptijn, 2003) (Nakamura, Watanabe, & Koga, 2008). Still other researchers have reported an increase in shear strength, perhaps due to the prestressing effect of ASR expansion (Ahmed, Burley, & Rigden, 1998). In any case, no widely accepted theoretical model connecting cylinder compressive strength or tensile splitting strength to the shear strength of ASR-degraded concrete is known to the author.

2.3 Standard ASTM Testing Procedures for ASR

Many standard tests have been proposed worldwide for characterizing ASR. However, most of them are variations of the ASTM standards. Two standard methods of evaluating the ASR reactivity of

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aggregates are ASTM C1567 and C1293. Briefly, ASTM C1567 details a procedure for evaluating the potential for aggregates to produce deleterious expansion by measuring the expansion of 1"x1"x10" mortar bars produced from prescribed quantities of water and cement combined with graded fine aggregates or crushed coarse aggregates. Mortar bars are stored at 80°C while immersed in 1M aqueous NaOH solution in sealed containers. Elongation greater than 0.1% measured 16 days after casting is considered a positive result for ASR.

The ASTM C1293 procedure measures the elongation of 4"x4"x10" concrete prisms. The concrete mix is a prescribed design with predetermined water to cement (w/c) ratio, quantities of fine aggregate and graded coarse aggregate. Alkali content is boosted through addition of NaOH to bring the alkali content to 1.25% by mass of cement. Concrete bars are stored at 38°C in sealed containers suspended on grates above water. Elongation greater than 0.04% measured one year after casting is considered a positive result for deleterious ASR expansivity.

2.4 Suitability of Concrete Prisms Tests for ASR

Considering the objective of this thesis is to design, mix, cast, and cure large concrete specimens affected by ASR, it is appropriate to devote attention to the doctoral thesis of Lindgård (2013), who has conducted the most extensive evaluation of the effects of test conditions on the expansion of concrete prisms to date. Lindgård's exhaustive testing has revealed that concrete prisms produced from the same concrete mix can exhibit substantially variable expansion depending on test conditions.

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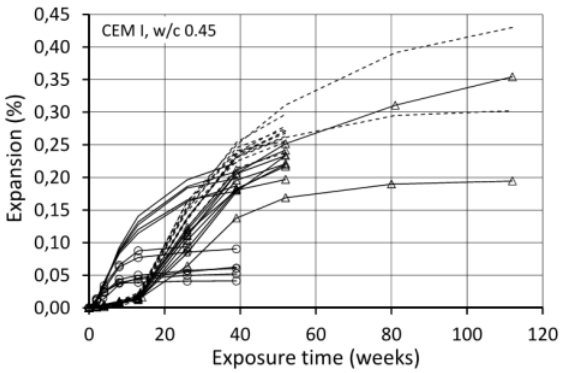

Figure 3 - Expansion vs time curves for the same concrete mix exposed to various test conditions, from (Lindgård, et al., 2013)

Lindgård prepared a large number of concrete prisms using the same reactive mix design and subjected them to a variety of curing conditions. Measuring the expansion of these prisms with a length comparometer, he found a startling degree of variation, displayed in Figure 3. Lindgård identifies the following principle causes of this variability.

1. Leaching of alkali (lost alkali reduces expansion)
2. Internal moisture content (desiccation reduces expansion)
3. Temperature (lower temperature reduce expansion rate, but not ultimate expansion)
4. Alkali content of cement (high alkalinity forestalls the effects of leaching)
5. Diffusion rate (impermeable concrete tends to expand less)

Of the effects above, Lindgård finds that alkali leaching in the first weeks of exposure has the greatest affect on concrete prism expansion. Three to 20% of the alkali initially present in the concrete of standard 4”x4”x10” prisms is lost to leaching in the first four weeks of exposure and up to 50% is lost after one year. Alkali loss in the first four weeks is well-correlated with ultimate expansion.

Further confounding the issue is Lindgård’s finding that the factors above are not independent. Higher temperatures accelerate the ASR reaction, but for samples stored in sealed containers such as prescribed by

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ASTM C1293, higher temperatures also accelerate alkali leaching. Hot water evaporating from bottom of the sample container condenses on the concrete surface, where it leaches alkali and transports it to the bottom of the container. However, samples stored in low-humidity environments can undergo shrinkage which obscures any ASR-induced expansion.

One successful strategy for controlling alkali leaching is to cover samples in cloth soaked with high-pH solution. Lindgård measured the alkali lost by samples wrapped in cloth soaked in either 1M NaOH solution (pH 14.2) or 0.1M NaOH solution (pH 13.2). Samples exposed to 1M solution were found to take up a small amount of NaOH from the soaking water and exhibited expansion 25% greater than corresponding unwrapped samples after 39 weeks, and 3.5 times more than samples wrapped in deionized water. However, the 0.1M solution proved ineffective at mitigating alkali loss and had a negligible effect on sample expansion.

2.5 Influence of High Alkalinity

Alkali is present in cement as sulfates and clinker constituents. While the effects of high alkalinity on ASR are described above, alkalinity also affects hydration rate. High alkalinity suppresses calcium concentrations due the common ion effect. This subsequently promotes dissolution of tricalcium aluminate (C_3A). Together these effects accelerate the rate of hydration and thus strength development at early ages (Jawed & Skalny, 1978). This accelerated strengthening effect is short-lived, and high alkalinity is generally thought to depress strength at 28 days and later (Gebhardt, 1995) (Burrows, 1999). Juenger and Jennings (2001) confirm these observations, finding that substitution of aqueous 1M NaOH solution for ordinary water results in elevated heat generation and rapid hydration for the first few hours after mixing. Heat generation and hydration rate is depressed thereafter. They further note that high alkalinity appears to promote shrinkage-related cracking.

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CHAPTER 3

TEST PROGRAM

3.1 Model System

Shear specimens produced as part of this thesis are intended to model a portion of the containment structure of a nuclear power plant and will be tested under biaxial shear, simulating lateral earthquake loading. The prototype containment vessel is taken to be a cylindrical structure with dimensions as described in Table 1 below. These values are considered reasonably representative of such containment vessels, though not precise to any particular NPP. The model scale factor is selected such that the height of the experimental shear specimen is equal to the wall thickness of the model system.

Table 1 - Prototype containment vessel dimensions

Inner radius (ft)	63
Wall thickness (ft)	4.5
Wall height (ft)	122
Foundation thickness (ft)	10
Grade level (ft above foundation base)	56

Table 2 - Model containment vessel dimensions

Scale Factor	0.56
Inner radius (ft)	35
Wall thickness (ft)	2.5
Wall height (ft)	68
Foundation thickness (ft)	5.6
Grade level (ft above foundation)	31

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The rendered drawings below provide an indication of prototype vs. model scale and the orientation of experimental shear specimens. The portion of the prototype structure modeled by the shear specimen is taken from a location just above grade level. It is subject to a vertical load from the overbearing structure and to a lateral load from earthquake forces.

Figure 4 - Prototype system (right) with model system (left)

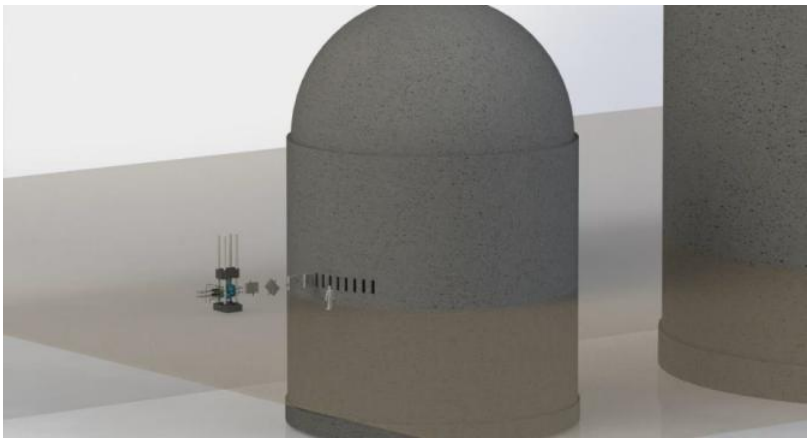

Figure 5 - Model system showing eight experimental specimens taken just above grade level

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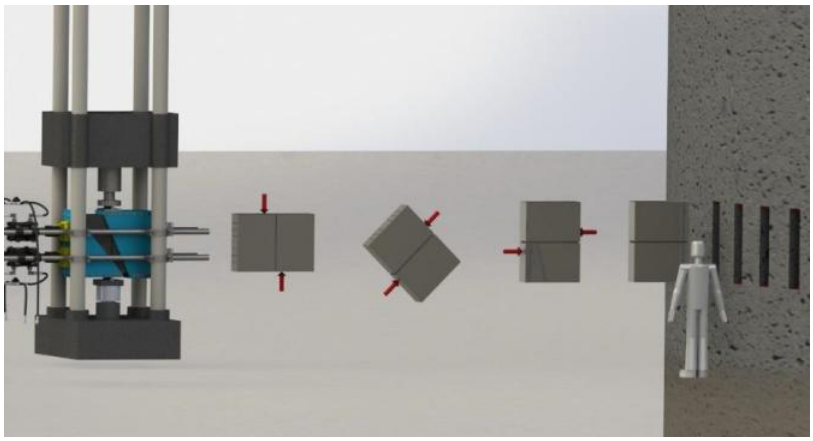

Figure 6 - Specimens are rotated 180 degrees about circumferential direction

The prototype system is reinforced in the axial and circumferential directions. Under lateral earthquake loading, the axial bars are engaged in dowel action but the circumferential bars are not. This reinforcement is duplicated in the experimental samples and is discussed further in Section 3.3. The lateral earthquake load and vertical overburden load are simulated in the laboratory by a vertical hydraulic test apparatus to which are fit horizontal actuators, discussed in greater detail in Section 3.2 below.

3.2 Shear Test Apparatus

Samples are tested using a hydraulic press produced by MTS which is capable of producing 1 million pounds of force. The vertical force produced by the test machine is transferred to either end of the shear sample by means of a heavy steel cage, which is assembled around the sample. This cage is in turn bolted to steel end plates which are cast into the sample. To each sample end plate is welded 30 1/2" steel shear studs which transfer force to the concrete specimen. This vertical force in the lab simulates lateral static earthquake load on the prototype structure.

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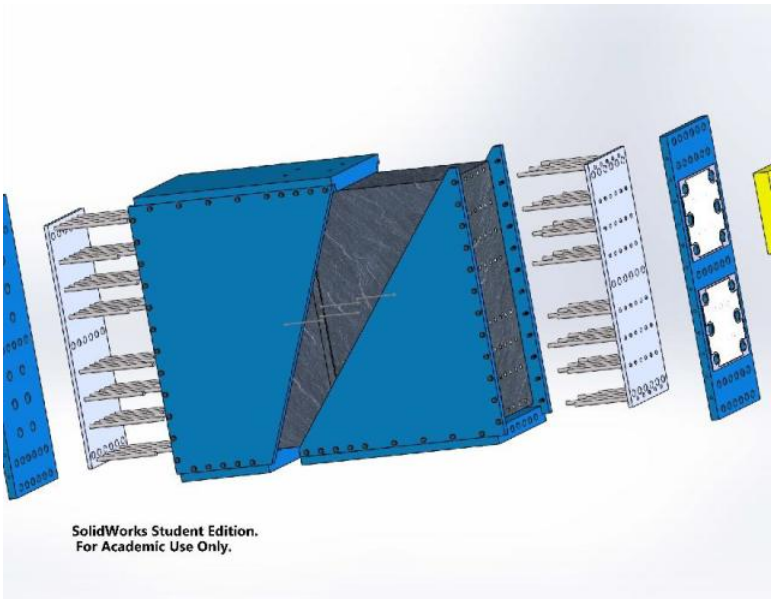

Figure 7 - Exploded rendering of shear specimen with sample end plates and steel cage

Figure 8 - Sample end plates with shear studs

Normal to the shear force is a pair of hydraulic actuators which apply compressive load to the sample. These actuators are fixed to cradles which transfer force to the sample end plates by means of two pivoting yokes bolted to the steel cage. This horizontal force in the lab simulates the vertical load of overbearing structure in the prototype structure.

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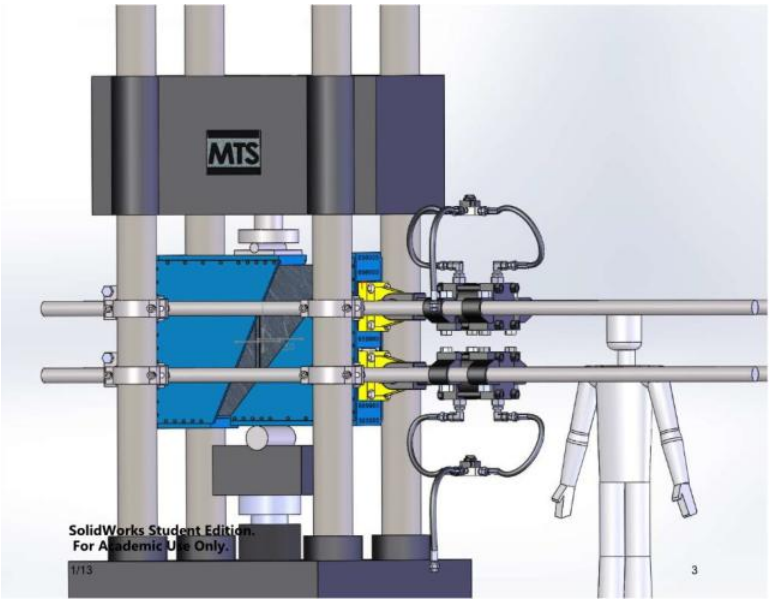

Figure 9 - Rendering of test apparatus. Note how the blue specimen cage transfers the vertical force of the press to shear load on specimen, while horizontal actuators apply a normal force.

Sixteen shear specimens are to be tested, of which 12 are ASR-damaged concrete and 4 are control samples unaffected by ASR. Nine of the ASR samples and two of the control samples are reinforced. The remainder are not reinforced. For each shear sample, two 4"x8" compression test cylinders are to be cast, which will help monitor the effects of ASR expansion on concrete strength. More information on the number and type of specimens cast is presented in Section 3.5.

The testing apparatus was constructed as part of a previous study but had been left unused for several years. Each component was retrieved from storage, cleaned, and checked for serviceability. Many components were shipped to Zimmerman Metals, of Denver for industrial cleaning, painting and, in some instances, replacement. After inventorying and dry-fitting components, apparatus assembly began on January 15th, 2016. The assembly procedure is briefly outlined below.

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Figure 10 - Components of the shear test apparatus were cleaned and painted prior to assembly.

Figure 11 - Bushing clamps are installed on the vertical columns of the MTS hydraulic press.

The four reaction bars are installed in bushing clamps which bear against the vertical columns of the million-pound press. It is critical that the reaction bars are parallel with the ground and with each other. It proved quite a challenge to align these components properly. Several methods were attempted, but it ultimately proved most effective to simply measure bushing clamp locations with a tape measure and use a spirit level to verify bars are level with the ground.

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Figure 12 - Reaction bars are lifted with an overhead crane and pushed into bushing clamps by hand.

Horizontal actuators bear against a set of clevis brackets which articulate through rods which are themselves held by rod clamps. In Figure 13 below, the yellow parts are the clevis brackets and the unpainted parts are the rod clamps. Installing this assembly is difficult. The rods must be held loosely between two rod clamps and the two brackets installed simultaneously over two reaction bars. The rod brackets are a tight fit and must be pried apart with wooden wedges to fit over the reaction bars. Also visible in Figure 13 is a blue backing plate, which is part of the specimen cage. It is being dry-fit, but would ordinarily be mounted to the specimen and the entire cage/specimen assembly mounted to the apparatus as a unit.

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Figure 13 – With reaction bars in place, rod clamps and clevis brackets are installed.

On the actuator side of the assembly, clamps are slid over the reaction bars and bolted into place. Actuators are suspended from the overhead crane and carefully mounted to the clamps. In Figure 14 below, one may notice that the lower actuator is installed 'upside down' in order to allow its weight to bear vertically on its clamp. This also allows more clearance for hydraulic lines.

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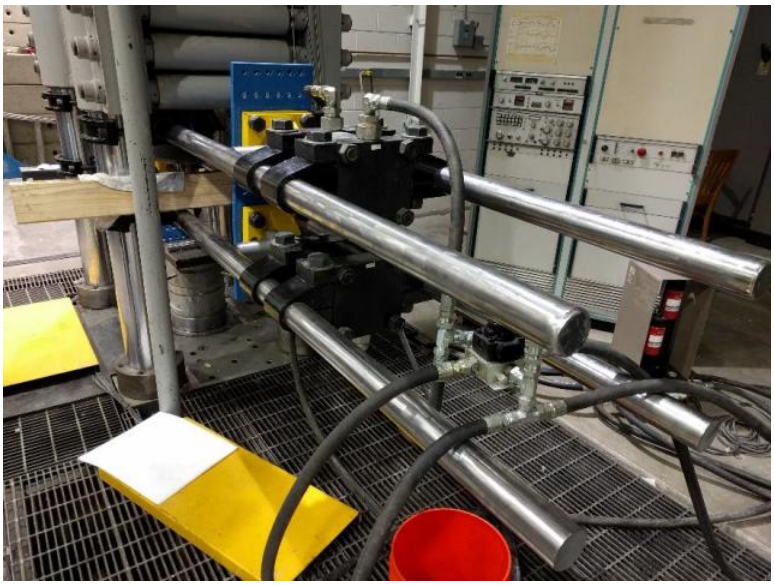

Figure 14 - Actuators are mounted to clamps which are themselves bolted to reaction bars.

Figure 15 – Specimen cage being dry fit to check tolerances.

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3.3 Reinforcement

Experimental shear specimens are cast using steel endplates with shear studs to transfer the vertical force applied by the press to shear force in the concrete specimen. Reinforcement is provided in two directions, corresponding to the axial and circumferential directions in the prototype system. Axial reinforcement (shown as red bars in Figure 18) aids in resisting shear forces via dowel action. Azimuthal reinforcement (shown as blue bars in Figure 18) is not engaged by shear forces.

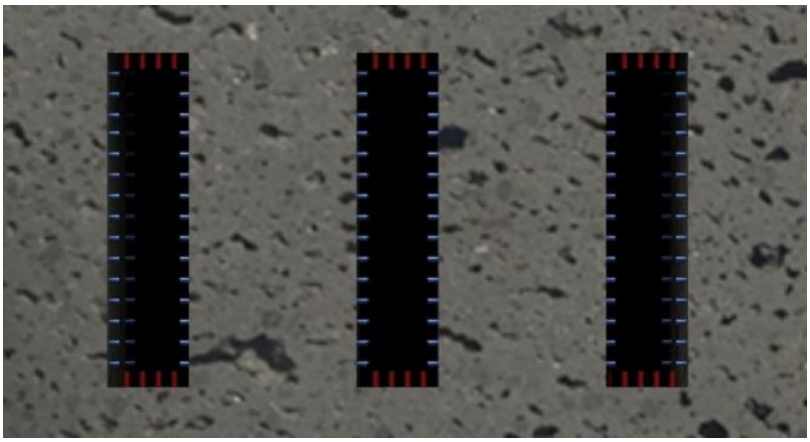

Figure 16 - Reinforcement in prototype system is oriented in the axial (red) and circumferential (blue) directions

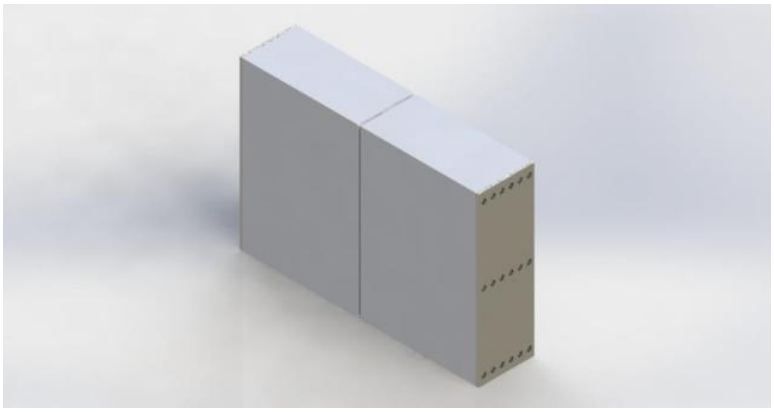

Figure 17 - Shear specimen showing concrete and end plates. Note scoring line coincides with shear plane.

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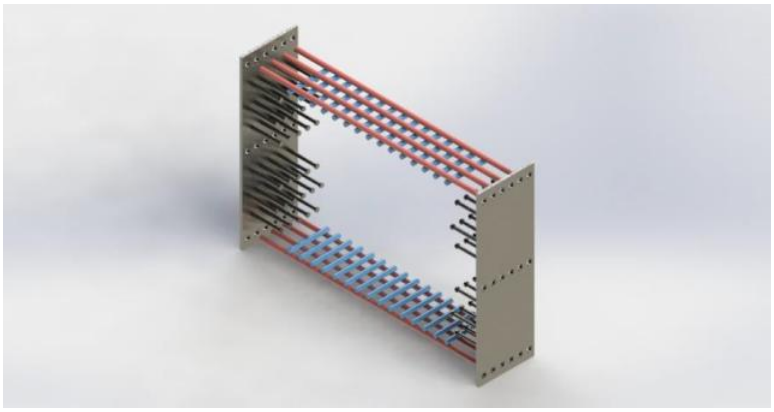

Figure 18 – Schematic sketch of shear specimen with concrete hidden. Red bars correspond to axial reinforcement and resist shear by dowel action. Blue bars correspond to the circumferential reinforcement and are not engaged in shear.

Unfortunately, reinforcement details of the prototype structure are not publicly available. Thus ratios must be selected such that the resulting experimental specimens are both constructible and approximate typical NPP containment vessel reinforcement. Various reinforcement ratios were considered and the number and size of bars required to for each is presented in Table 3 and Table 4 below. In the interest of constructability, it was decided to allow only two layers of reinforcement in each direction. Considering that samples are cast using 3/4" coarse aggregate (see Section 4.5), clear spacing between bars can be no less than 1" per ACI 211.1. It was decided to use a reinforcement ratio of 0.5% per layer (1% total) in each direction with #6 bars in the axial direction and #7 bars in the circumferential. Eleven of the sixteen shear specimens are to be reinforced (see Section 3.5). Thus 242 short #7 bars and 88 longer #6 bars are required.

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Table 3 - Circumferential reinforcement ratio selection

Sample Dimensions (in^2)		Lyy	30	Concrete Area, (in^2)
		Lxx	42	1260
Bar Number	Steel area per bar A_{bar} (in)	Reinforcement ratio, ρ	Required steel area, A_s (in^2)	Number of bars required per layer
5	0.31	0.2%	2.52	9
		0.5%	6.30	21
		1.0%	12.60	41
6	0.44	0.2%	2.52	6
		0.5%	6.30	15
		1.0%	12.60	29
7	0.6	0.2%	2.52	5
		0.5%	6.30	**11**
		1.0%	12.60	21
8	0.79	0.2%	2.52	4
		0.5%	6.30	8
		1.0%	12.60	16

Table 4 - Axial reinforcement ratio selection

Sample Dimensions (in^2)		Lzz	10	Concrete Area (in^2)
		Lyy	30	300
Bar Number	Steel area per bar, A_{bar} (in)	Reinforcement ratio, ρ	Required steel area, A_s (in^2)	Number of bars required per layer
4	0.2	0.2%	0.6	3
		0.5%	1.5	8
		1.0%	3	15
5	0.31	0.2%	0.6	2
		0.5%	1.5	5
		1.0%	3	10
6	0.44	0.2%	0.6	2
		0.5%	1.5	**4**
		1.0%	3	7
7	0.6	0.2%	0.6	1
		0.5%	1.5	3
		1.0%	3	5

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Insufficient length is available for either the axial or circumferential steel to develop its full tensile strength. Considering the large strains anticipated due to ASR expansion, it is necessary to provide for some type of anchorage at the bar terminations. A number of options were considered, including hooks and threaded terminations. Unfortunately, the standard hook size for a #7 bar is 10.5" with a minimum bend diameter of 7". Considering that these bars are only 8" long, attempting to use standard hooks would deform the circumferential reinforcement geometry to an extent that it would bear little resemblance to the prototype structure. Furthermore, the minimum development length even with hooks is 19" which exceeds the out-to-out thickness of the sample (10").

Ultimately, it was decided to weld axial bars to the sample end plates, and weld circumferential bars to the axial bars at each intersection. While welding rebar is not typically best practice, no other practical option exists for developing tensile strength in such a confined volume as the shear samples. The sample end plates provide development for the axial bars, while the axial bars themselves act as hooks for the circumferential bars. While certainly not ideal, this solution allows for at least some tensile development without drastically altering the reinforcement scheme of the prototype system.

Table 5 - Reinforcement plan

(All dimensions in inches)	**Bar Number**	**Bar Diameter**	**Bar Length**	**Number of bars per layer**	**Bar spacing (center to center)**	**ρ_{actual}**	**Total bars required**
Circumferential Reinforcement	7	0.875	8	11	2.813	0.52%	242
Axial Reinforcement	6	0.75	42	4	2.083	0.59%	88

To facilitate rapid construction of the sixteen required rebar cages, a wooden jig was built to hold the loose bars during the welding process. The desired locations of the bars were computed and carefully laid

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out on a piece of plywood. Sixty small wooden blocks sized to fit in the areas between the steel bars with 1/32” clearance were then cut. These wooden blocks were then affixed to the plywood base using wood glue and 18-gauge brad nails.

Figure 19 - Cutting wooden blocks for the welding jig.

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Figure 20 - Wooden blocks ready for installation in the jig.

Figure 21 - Joining wooden blocks to jig base with wood glue and steel brad nails.

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Steel bars were primarily sourced from stock on-hand in the structures laboratory. All were cut to size using a steel saw and ground to final dimension with a bench grinder. This proved to be a time-consuming process, as a total of 330 bars are required.

Figure 22 - Cutting reinforcing bar

Figure 23 - Rebar layout and welding using the wooden jig.

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The jig allowed rapid layout and welding of the rebar cages. The jig also provided a simple way to verify that all bars were cut to proper length. Any long or short bars would not fit properly into the jig and could be ground down or replaced.

Bars were MIG-welded to one another at each intersection. Care was taken to make small welds in order to minimize the size of heat-affected regions in the substrate bars.

Figure 24 - Rebar cages ready to be welded to sample end plates.

Completed cages were then connected to the sample end plates by tack-welding the #6 bars to the plates.

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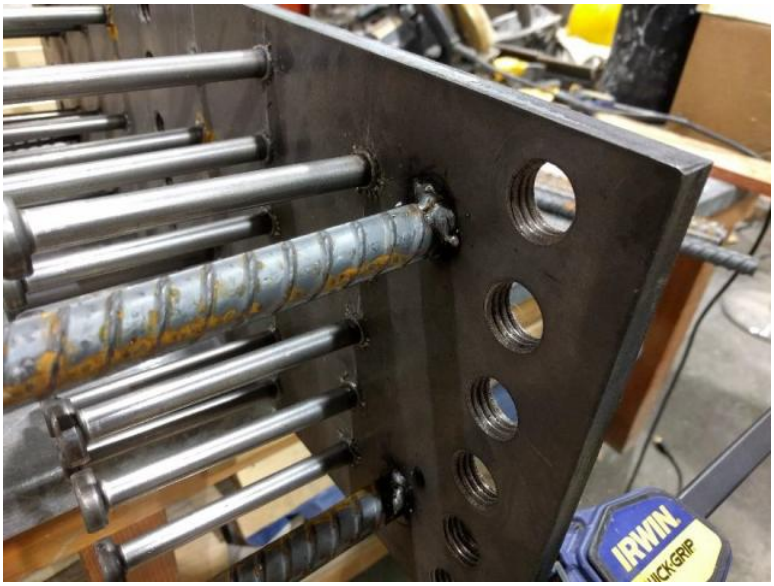

Figure 25 - MIG-welded joint between #6 rebar and sample end plates.

3.4 Formwork

Specimens will be cast horizontally, in order to better facilitate concrete penetration between closely-spaced shear studs. Thus a simple form was designed using 21/32" oriented-strand board and 2x4 studs. The top brace of the stud was elevated somewhat from the top surface of the concrete to allow a trowel to pass under during finishing. Corners are strengthened with steel brackets and the entire assembly is joined with deck screws. The form rests on its 2x4 braces, which allow it to be moved via forklift without extra blocking.

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Figure 26 – Example shear specimen formwork

Two options for formwork material were considered. Either a small number of reusable forms could be constructed using more durable (but expensive) *Plyform*, or a larger number of single-use forms could be constructed using less-expensive oriented strand board (OSB). Since the large volume of concrete required necessitates that casting would be performed at the laboratory of research partner Fall Line Testing and Inspection, in Denver, it was decided to adopt a compressed casting schedule to minimize impact on Fall Line business operations. Therefore, it was decided to construct single-use formwork.

Seventeen forms were built using OSB and 2"x4" studs, one for a dummy sample and sixteen for the experimental shear specimens. The upper brace visible in Figure 26 is built to float above the surface of the concrete to allow a trowel finish to be applied. Formwork for additional block and prism specimens (discussed in Section 3.5) were produced in a similar fashion. To mitigate water absorption by the wood from the fresh concrete, the inside of each form was given two coats of oil-based primer.

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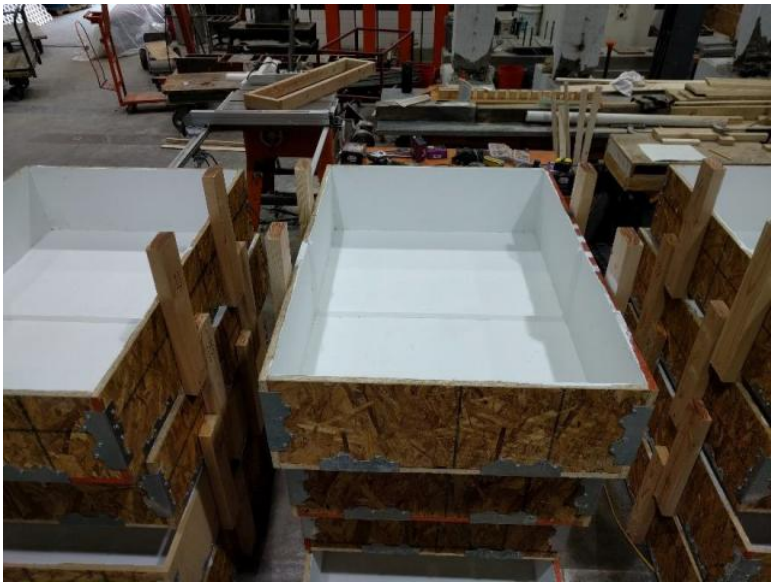

Figure 27 - Assembled and painted formwork without upper brace. Seventeen forms were built in total.

3.5 Casting Plan

A listing of concrete specimens to be produced is provided in Table 6. The most significant effort is preparation of the 16 shear samples, which are 42”x30”x10” prisms, discussed in greater detail in section 3.3. Additionally, 15 cubical specimens measuring 14”x14”x14” referred to as “blocks” will be produced. These blocks will allow extraction of cores from the center of their centers, thereby avoiding any undesirable surface effects such as alkali leaching. A third type of sample is intended for a wedge splitting test. A significant number of cylinders will be necessary to measure compressive strength and tensile splitting strength. Finally, a small number of 4”x4”x12” prisms will be produced which will allow monitoring of expansion using a demountable strain gauge (DEMEC). In total, 6.3 cubic yards of concrete will be cast, which is a significant quantity to produce without the benefit of a commercial facility.

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Table 6- Sample requirements

Required Concrete Volume		
	Number	Volume yd^3
Reactive		
Shear specimens with rebar	9	2.43
Shear specimens without rebar	3	0.81
Wedge splitting test	3	0.02
Cylinder, 4”x8”	36	0.08
Cylinder, 6”x12”	12	0.09
Blocks without rebar	6	0.35
Blocks with rebar	3	0.18
Prism 4”x4”x12”	6	0.06
Wastage factor	15.0%	
Total	**4.54**	
Non-Reactive		
Shear specimens with rebar	2	0.54
Shear specimens without rebar	2	0.54
Wedge splitting test	3	0.02
Cylinder, 4”x8”	12	0.03
Cylinder, 6”x12”	4	0.03
Blocks without rebar	3	0.18
Blocks with rebar	3	0.18
Prism 4”x4”x10”	3	0.03
Wastage factor	15.0%	
Total	**1.73**	
Grand Total (yd^3)	**6.27**	

Casting of experimental samples takes place at the Fall Line laboratory on April 25th and 27th. Concrete mixing is achieved using a three-cubic yard mixer provided by Fall Line. The mixer is charged using a mobile batch plant. The batch plant is a trailer equipped with two hoppers for coarse and fine aggregate and a water tank. The hoppers may be emptied onto a built-in conveyor which charges a large 3-yard mixer.

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Each hopper and tank is equipped with load cells to monitor weight change as aggregates are loaded into the mixer. The hoppers can be filled using a skid-steer loader.

Figure 28 - The three-cubic yard mixer at Fall Line

Figure 29 - Close view of mobile batch plant at Fall Line. Note load cells for weighing hopper contents.

Concrete will be transported from the mixer to the formwork using a forklift-borne hopper. Concrete will be shoveled or troweled into forms, where it will be vibrated or tamped into place. Shear specimens and blocks will receive a trowel finish while cylinders will be capped.

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3.6 Aggregate Supply

Casting the required volume of concrete requires a significant supply of material, summarized in Table 7. Both reactive and nonreactive specimens are cast using aggregate supplied by Whitewater Building Materials of Grand Junction, Colorado (refer to CHAPTER 4 for a full description of the aggregate selection process).

Table 7 - Estimate of materials required based on Mix 5R and Mix 5NR.

Material	**lbs**	**kg**
Portland Cement, Type 1, Holcim	4,200	2,500
Fine Aggregate: Manufactured Sand	10,100	6,100
Coarse Aggregate: 3/4" Crushed Rock	8,600	5,300
Admixtures		**Unit**
NaOH(s) Doping Additive (kg)		12.2
Lithium Nitrate Additive (L)		34.5

Aggregates were delivered to the Fall Line laboratory on February 1st, 2016. Using a conveyor and skid-steer loader, the aggregates are formed into piles, conditioned (wet slightly with a hose), and mixed. Each pile is then covered with plastic sheet. Aggregates are tested to determine relative density, bulk density, absorbance, specific gravity, and fineness modulus as described in Section 5.2. Two days prior to casting, piles are mixed and conditioned again. On the morning of each casting, moisture content is measured again and concrete mix adjusted accordingly.

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Figure 30 - Offloading aggregate

Figure 31 - Mixing fine aggregate and forming into pile.

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3.7 ASR Development Conditions

After curing for 24 hours in the Fall Line laboratory, all samples are to be transported to the CU structures lab and demolded. Specimens will be fit with DEMEC measurement points by gluing with epoxy adhesive. The precise positioning of these measurement points will be the subject of a future work. Extension of shear samples must be measured in three axes, while extension of smaller 4"x4"x12" samples are measured along one axis only.

The greatest expansion observed by Lindgård was among concrete prisms exposed to a solution of 1.5M $NaOH_{(aq)}$ one day after casting (Lindgård, et al., 2013). This behavior aligns with results observed in this study (see CHAPTER 6). Reactive concrete prisms stored immersed in 1.0M $NaOH_{(aq)}$ at 80°C exhibited dramatically greater expansion than those stored in a fog room at high humidity and 21°C. It is probable that both lower temperatures and increased alkali leaching contributed to the reduced expansion of the fog room specimens.

Therefore, experimental samples should be stored at the highest practical temperature, which for the renovated CU fog room is 38°C. Exposure to high humidity in the absence of high pH soaking solution is to be avoided as it contributes to alkali leaching and significantly reduces both rate of expansion and ultimate expansion. Reactive samples must be protected from alkali leaching by provision of a high-pH surface environment. Furthermore, relative humidity should be maintained above 90%. This is intended both to reduce loss of the protective film of alkali solution and to resist shrinkage where the alkali solution is not fully covering the specimens.

Reactive samples are to be wrapped in burlap fabric and exposed to a continual flow of 1.5M $NaOH_{(aq)}$ solution. The solution will be circulated by a sprinkler system devised by fellow researcher David Graff. The solution is caught in drip pans below the samples and recirculated by means of a pump. The pH of this solution should be monitored at least weekly, and maintained at 14.2 (1.5M NaOH). Lindgård has shown

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that a wash solution of 13.2 (0.1M NaOH) is no more effective at preventing alkali leaching than ordinary tap water, thus care should be taken to replenish the hydroxide concentration of the wash water as it is depleted by the dissolution of atmospheric CO_2 and by decay of cellulose fibers in the burlap fabric.

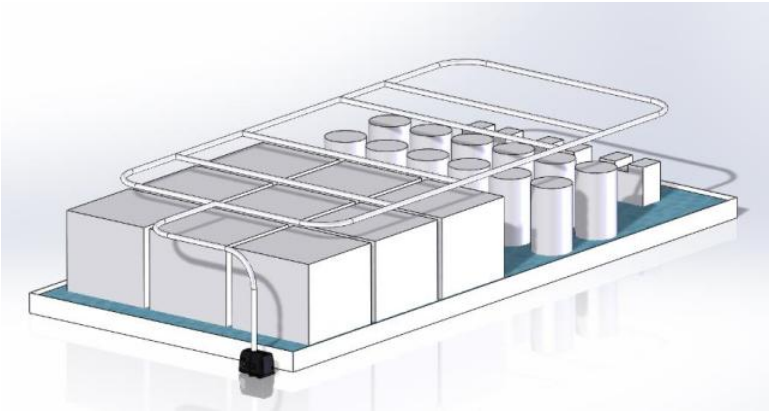

Figure 32 - Schematic of reactive blocks and cylinders in drip pan underneath caustic sprinkler system.

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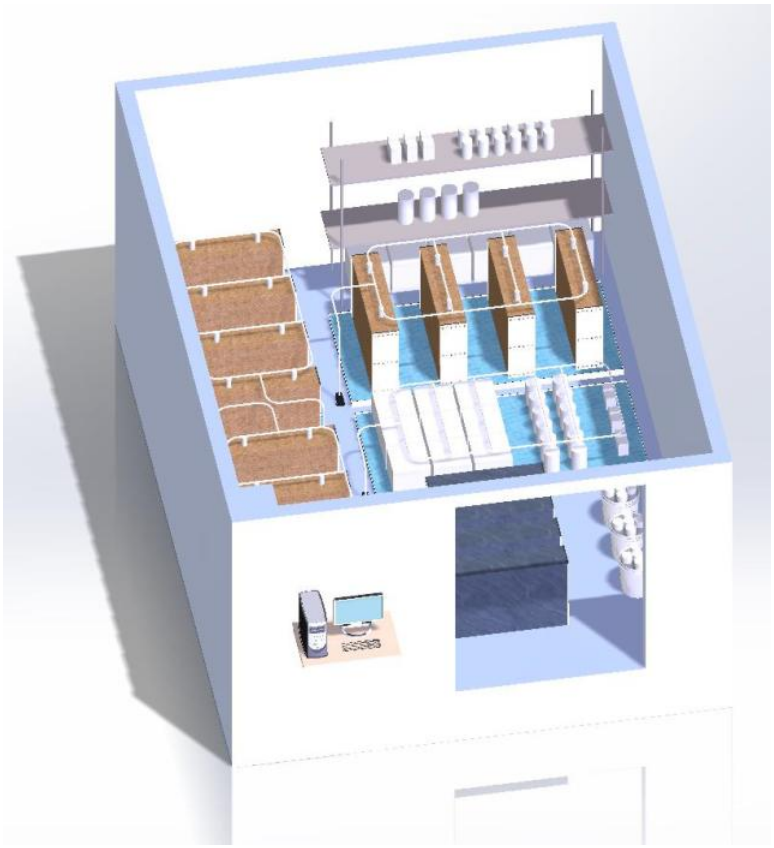

Figure 33 - Rendering of sample arrangement in CU fog room. Note drip pans and sprinklers under reactive specimens.

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CHAPTER 4

IDENTIFICATION OF REACTIVE AGGREGATES

4.1 Introduction

Two potential sources of reactive aggregates were brought to the project's attention:

1. Whitewater Building Materials
2. Grand Junction Ready Mix

Both suppliers obtain aggregate from quarries along the Gunnison River in western Colorado, near Grand Junction.

On January 21, 2015, both sites were visited, and 5-gallon bucket of each aggregate type (3/4, 3/8 and sand) were brought for initial testing. Whitewater samples were obtained directly from storage bins.

Figure 34 - Whitewater Building Materials, Whitewater, Colorado

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Figure 35 - 3/4" Gravel storage bin at Whitewater Building Materials

Grand Junction ready mix had prepared samples of sand and 3/4” gravel, while the 3/8” gravel was pulled from a storage bin.

Figure 36 - Grand Junction Ready Mix, Grand Junction, Colorado

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Figure 37 - 3/8" Gravel storage bin at Grand Junction Ready Mix

ASTM describes two standard test methods to assess aggregate reactivity: C1293 and C1567. The former lasting about 52 weeks, the second was selected as it tests the potential for aggregate reactivity in only 2 weeks.

4.2 Overview of ASTM C1567 mortar bar test

ASTM 1567 identifies the presence of ASR by measuring the elongation of three 1" X 1" X 10" mortar bars after storage in a solution of 1N aqueous sodium hydroxide at 80˚C for fourteen days. Expansion beyond 0.1% is considered sufficient to identify problematic expansion due to ASR.

The mortar mix must be prepared according to ASTM C305, using a precise mix of gradations as described in Table 8.

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Table 8 - Proportions of aggregate by size and mass required for 3 mortar bars

Sieve Size		Mass %	Mass (g)
Passing	Retained on		
4.75 mm (No. 4)	2.36 mm (No. 8)	10	99.0
2.36 mm (No. 8)	1.18mm (No. 16)	25	247.5
1.18mm (No. 16)	600 µm (No. 30)	25	247.5
600 µm (No. 30)	300 µm (No. 50)	25	247.5
300 µm (No. 50)	150 µm (No. 100)	15	148.5

Cement and water are proportioned according to the relative density of the aggregate.

Table 9 - Cement and water required for 3 mortar bars.

Cement (g)	Water (mL)
440.0 g	207

Bars are cured for 24 ± 2 hours in a fog room with relative humidity of at least 50% and temperature maintained at 23 ± 2˚C. An initial length measurement is taken after curing is completed. Bars are then submerged in water at 23 ˚C and the bath is transferred to an oven at 80.0 ± 2 ˚C for a further 24 ± 2 hours.

The bath is then removed from the oven and a zero length measurement is taken. Each bar is removed from the bath, dried, and measured within 15 ± 5 seconds. Bars are then transferred to aqueous solution of 1N NaOH at 80.0 ± 2 ˚C and replaced in the oven within at most ten minutes from the time the water bath was removed. Samples are stored in this manner for a further 14 days. During this time, at least three interim measurements are taken, each at the same time of day and following a procedure similar to that of the zero measurement. The final measurement is taken 14 days after the zero reading (16 days after casting).

The difference between the zero comparometer reading and the 14-day reading is calculated to within 0.001%. Expansions more than 0.10% indicate potentially deleterious expansion.

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4.3 Narrative of Aggregate Testing

The distribution of grades required by ASTM 1567 was obtained by sieving sand samples directly. However, coarse aggregates were crushed using a machine in the University materials laboratory. After crushing, all samples were sieved using a mechanical shaker and calibrated sieves at the Fall Line laboratory. Even with the mechanical shaker, it is easy to overload the sieves. Each sieve-shaking cycle takes 7 minutes and 30 seconds, and 10-12 cycles were required to obtain the required mass of each grade.

Figure 38 - Aggregate crusher in use at University of Colorado materials laboratory

Figure 39 - Mechanical shaker with sieves at Fall Line laboratory

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After each sieve-shaking cycle, the contents of each sieve were emptied into steel bowls. Each was then washed in water and carefully decanted. At least three separate washes were used for coarser grades, while finer grades typically required a fourth wash.

Figure 40 - The contents of each sieve were separated into steel bowls.

Figure 41 - Each grade was washed three to four times in tap water and decanted.

Washed material was then transferred into drying trays and allowed to dry in an oven at 230˚ F overnight.

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Figure 42 – Dana Schwartz of Fall Line placing washed material in drying oven.

Dried aggregate and cement was then weighed using a laboratory balance. Water was measured with a graduated cylinder.

Figure 43 - Balance used to weigh dried aggregate

Steel molds were prepared by cleaning and oiling prior to assembly. Two release agents were tried. One was a silicon-based spray lubricant that performed poorly. A generous coat of '*3 In 1'* oil proved more effective. Slight corrosion was noticed on the molds (visible Figure 44), although they remained smooth to the touch. While we initially thought that such minor corrosion would have a negligible effect, it made

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the cured mortar bars quite difficult to extract. This was corrected by mechanically polishing the bars using a hand drill with cotton wheel and abrasive compound.

Figure 44 - Oiled molds

Figure 45 - Assembled molds ready for mortar

Mortar was mixed according to ASTM C305. Water was placed in mixer bowl and entire quantity of cement was added. Water and cement were then mixed at low speed (140 ±5 rpm) for 30 seconds. Graded aggregate was then added slowly the next 30 seconds. The mixer was then switched to medium speed (285±10 rpm) for 30 seconds. Then the mixer was switched off and the mortar scraped down from the

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paddle and bowl edges. After sitting 90 seconds, the mixer was switched back on at medium speed and mixing continued for a further 60 seconds.

Figure 46 – Mixer with measured cement, water, and aggregate

Figure 47 - Time was kept with a stopwatch during mixing

After mixing was complete, one person half-filled each mold with mortar while a second person tamped. Special care was required to ensure no void remained near the measuring studs visible in Figure 45. The molds were then filled with a second layer of mortar and tamped again. Finally, each mold was struck using a wetted magnesium trowel. ASTM calls for this process to be complete within two minutes and fifteen seconds from completion of mixing. This proved difficult to achieve, even with the two-person

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method described. Filling and tamping all three molds took us between two minutes, thirty seconds and two minutes, forty-five seconds consistently.

Figure 48 - Tamping mortar into molds

Figure 49 - Mortar bars placed in curing room and covered with plastic

Molds were then placed in the Fall Line curing room and covered with a plastic sheet. After curing for 24 $\pm$ 2 hours, the mortar bars were extracted from the molds. As mentioned previously, this proved to be a difficult task. Any corrosion on the mold caused the mortar bars to stick, even though the molds felt smooth to the touch. One bar molded from Grand Junction Ready Mix sand and two bars of Grand Junction Ready Mix 3/8" gravel broke, requiring that new sets of three bars be molded for each of these samples.

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Figure 50 – This mortar bar broke during extraction

Once extracted from their molds, an initial reading of each bar was taken using a length comparometer. The comparometer was zeroed before the first measurement and after between each group of three bars. Bars were then placed in a water bath at 23˚C and the bath was placed in an oven at 80 ˚C for another 24 ± 2 hours. The following day, a zero reading was taken. Each bar was removed from the water bath, quickly dried with a towel (ensuring no water remained on the measuring studs) and measured on the length comparometer. It was not difficult to complete each measurement well within the 15 ± 5 seconds permitted by ASTM C1567.

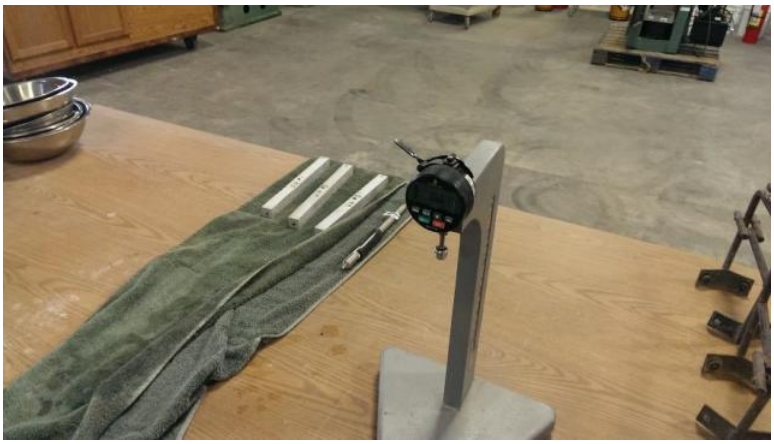

Figure 51 - Initial readings were taken before bars were placed in water bath.

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After zero readings were taken, bars were placed in a solution of 1N aqueous sodium hydroxide and returned to the oven. This solution was prepared in advance, stored in a sealed plastic bin, and kept in the oven until the zero readings were complete.

Figure 52 - After zero readings, bars were placed in 1N aqueous NaOH.

Over the next 14 days, four measurements were taken following a similar procedure to the zero reading. The determination of ASR reactivity was obtained by determining the average expansion percentage 14 days after the zero reading (16 days after casting). Additional measurements were taken roughly once per week after the 14-day mark to evaluate continued expansion over a longer term.

4.4 Test Results

All samples expanded well beyond the threshold of 0.10% at 16 days after casting established by ASTM as indicative of ASR. Whitewater 3/4" gravel exhibited the greatest expansion at 1.02%, over ten times the threshold value. Whitewater 3/8” gravel proved least expansive at 0.68%. The expansion of Grand Junction Ready-Mix samples fell between these extremes.

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Table 10 – Summary of ASTM C1567 initial test results

Percent expansion, 16 days after casting		
	Whitewater Building Materials	Grand Junction Ready Mix
Sand	0.69	0.98
3/8"	0.68	0.74
3/4"	1.02	0.77
Average	0.80	0.83

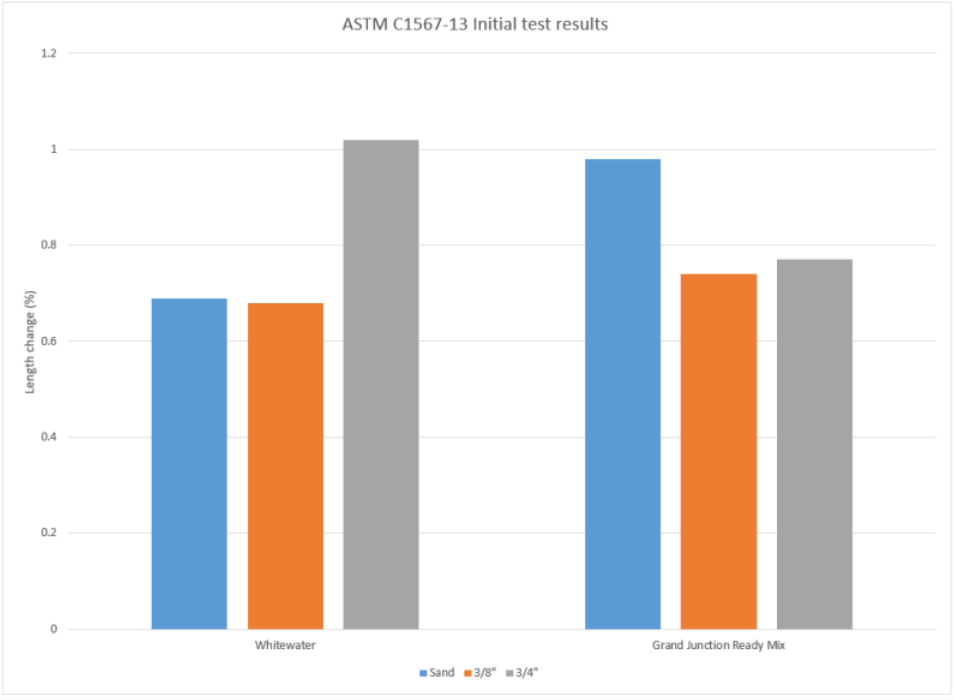

Figure 53- Summary of 16-day ASTM C1567 results

All samples continued to expand after the 16-day measurement used to establish reactivity. Expansion of each sample vs. time is plotted in Figure 54 - Figure 59 below. Note that expansion during the first ten days is rapid for all samples, followed by a long period of expansion at a reduced rate. Expansion typically

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begins slowing around 100 days, and most samples reach their maximum elongation after approximately 150 days.

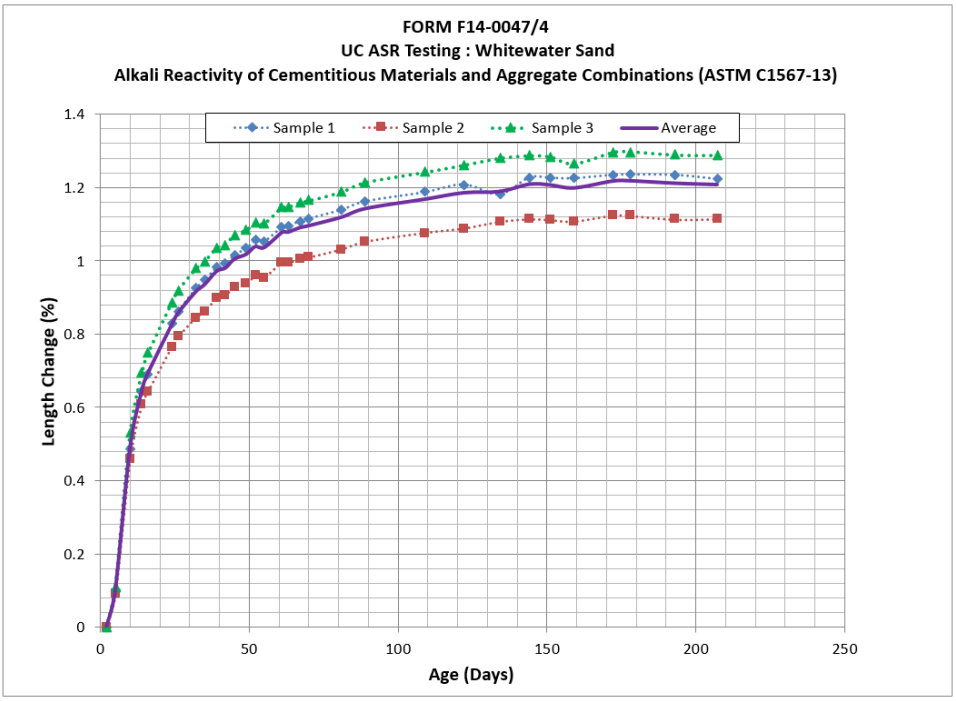

Figure 54 - Mortar bar expansion vs. time, Whitewater sand

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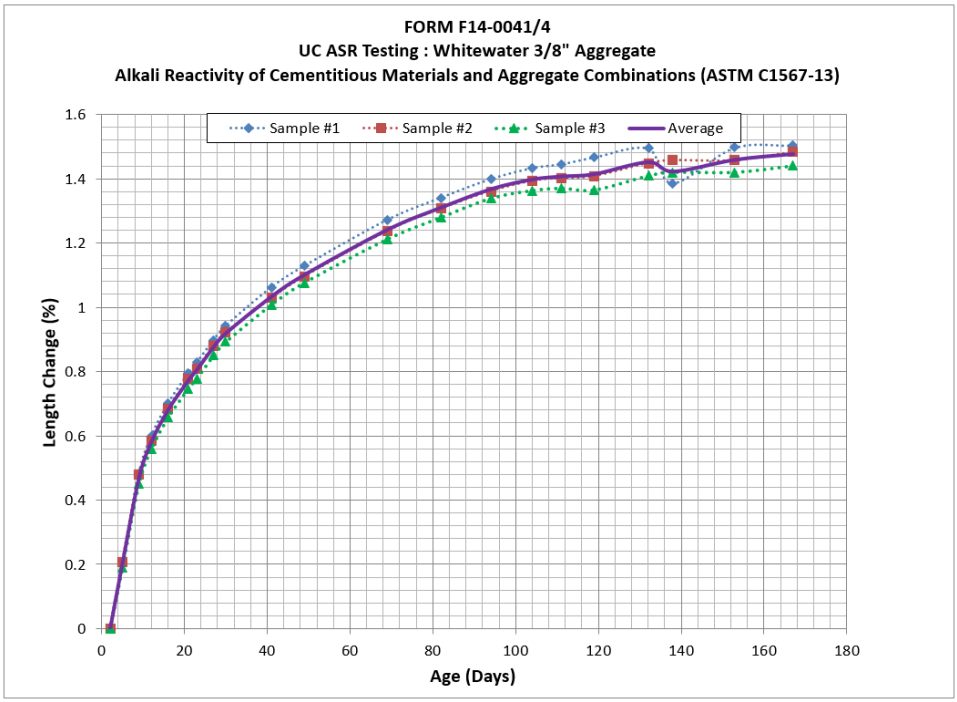

Figure 55 - Mortar bar expansion vs. time, Whitewater 3/8" aggregate

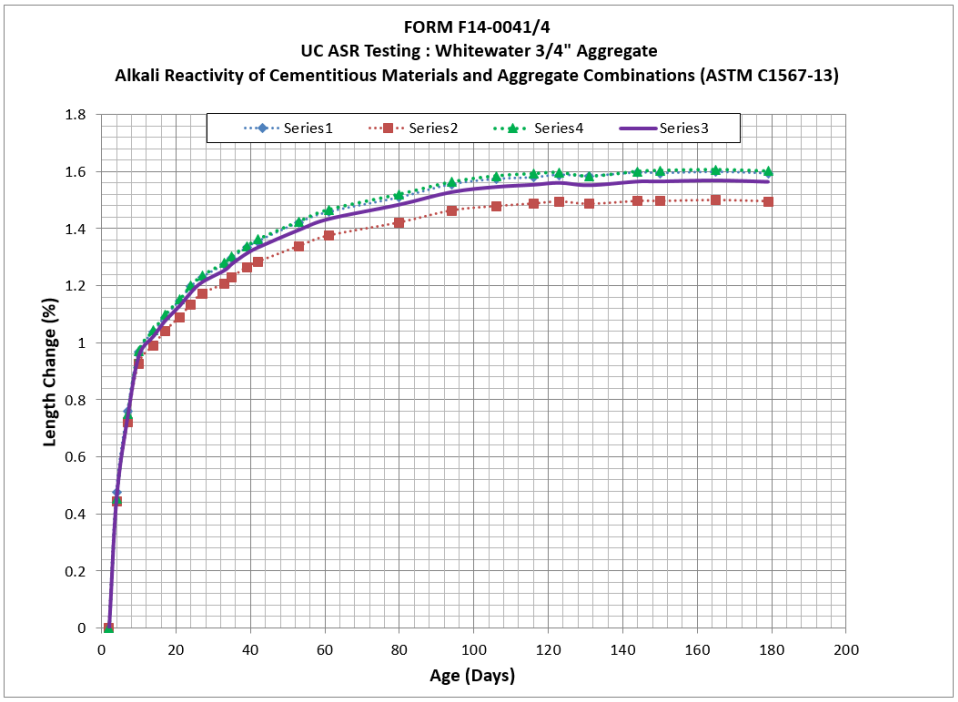

Figure 56 - Mortar bar expansion vs. time, Whitewater 3/4" aggregate

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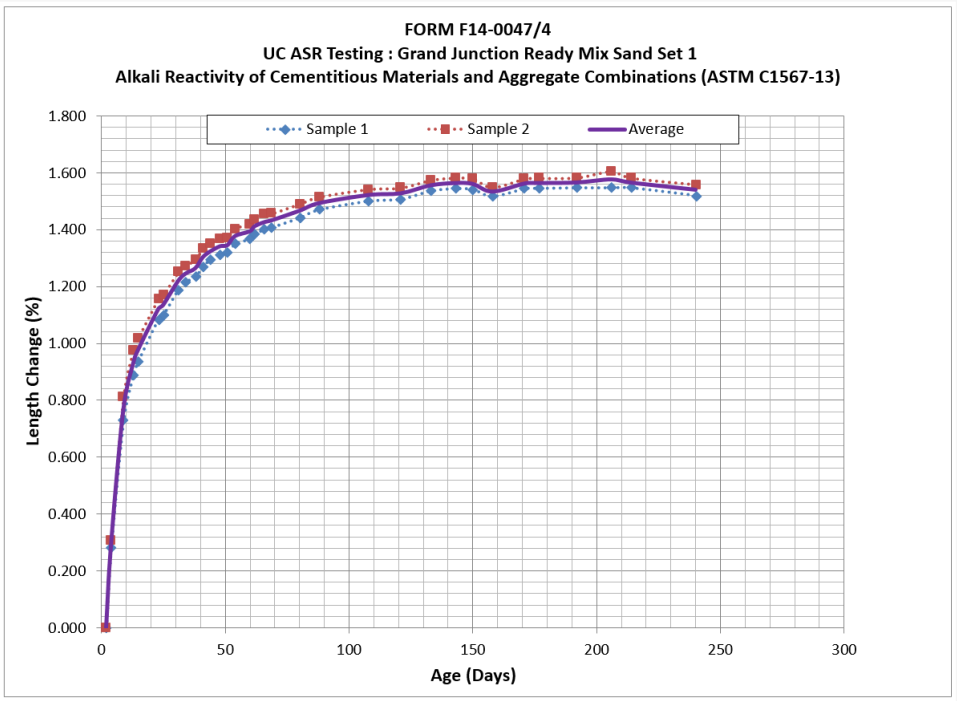

Figure 57 - Mortar bar expansion vs. time, Grand Junction Ready Mix sand

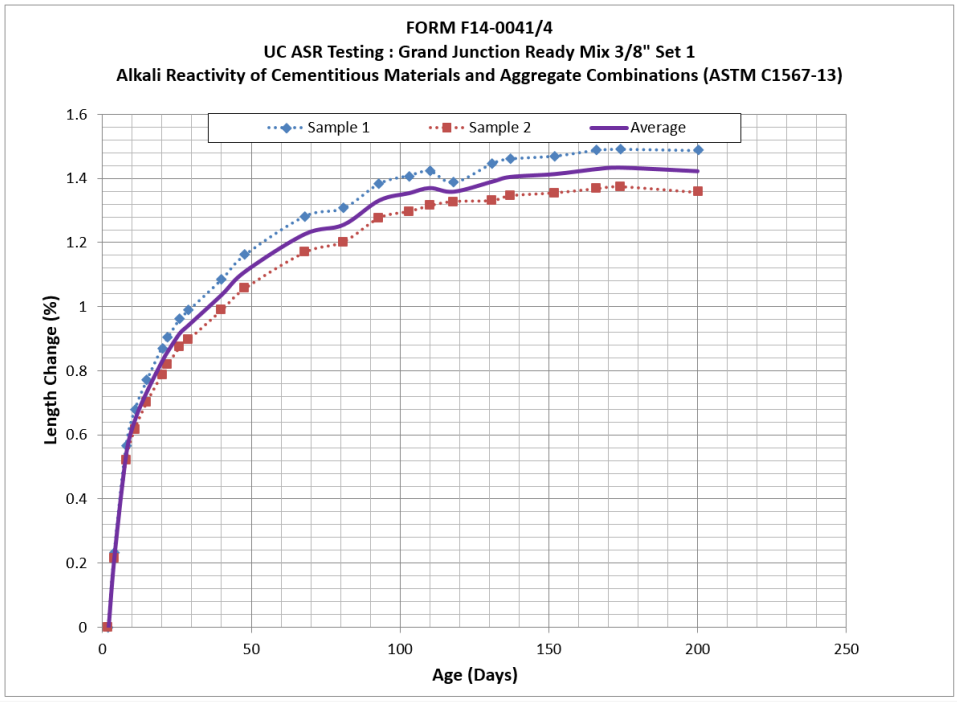

Figure 58 - Mortar bar expansion vs. time, Grand Junction Ready Mix 3/8" aggregate

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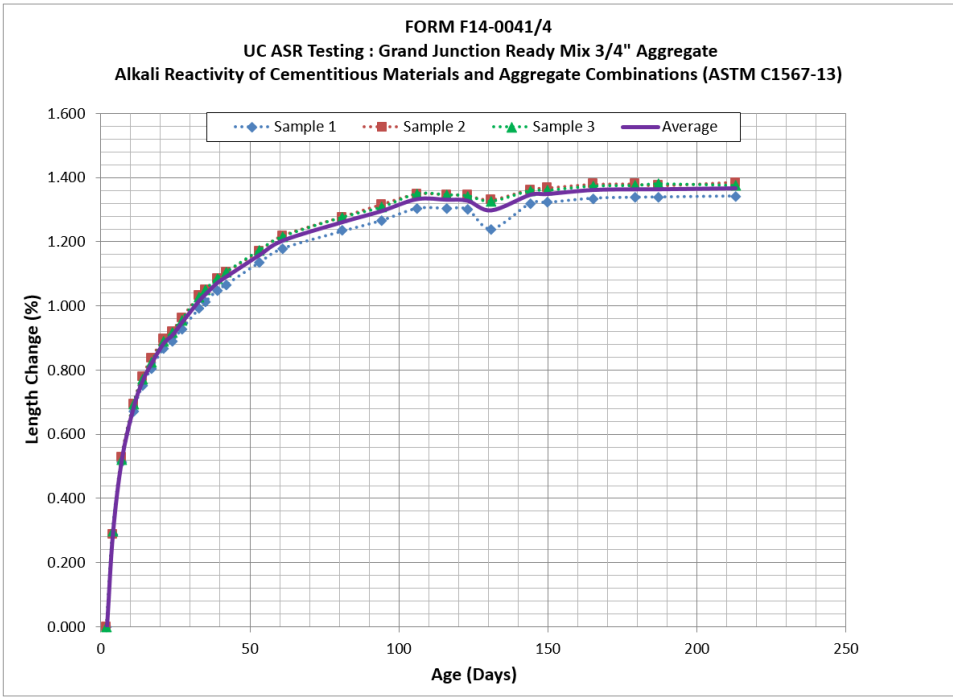

Figure 59 - Mortar bar expansion vs. time, Grand Junction 3/4" aggregate

4.5 Conclusion

Considering the reactivity data presented above, it is evident that both Whitewater and Grand Junction Ready Mix products produce significant expansion. Whitewater sand and 3/4" were selected for production of experimental concrete due both to the reactivity of the aggregates and the generous support Whitewater Building Materials staff.

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CHAPTER 5

CONCRETE TESTING PROGRAM

5.1 Introduction

Evaluation of candidate concrete mixes requires the adoption of a test regimen that adequately characterizes the mix in a reasonable period of time and allows for the mix to be reproduced despite small variances in aggregate or cement. Preference was given to standardized test procedures that can be easily duplicated.

Each shipment of Whitewater aggregate was subjected to the tests in Table 11 before use in candidate concrete mixes. All tests except C566 were conducted as soon as practical after receiving aggregate. Moisture was not measured until immediately before casting.

Table 11 - Aggregate testing program

Test	Standard
Coarse aggregate relative density	ASTM C127
Fine aggregate relative density	ASTM C128
Coarse aggregate bulk density	ASTM C29
Fineness modulus / gradation	ASTM C136
Moisture content	ASTM C566

Candidate concrete mixes were subjected to the tests in Table 12 below. The most important of these are C173, C39, and the ASR test. The remainder of the tests are taken the sake of completeness and to monitor against any unintended environmental effects.

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Table 12 – Concrete mix testing program

Test	Standard
Slump	ASTM C173
Unit Weight	ASTM C138
Air Content	ASTM C231
Temperature	ASTM C1064
Compressive Strength	ASTM C39
ASR Expansion	N/A

Each of these tests procedures is summarized briefly in the following sections.

5.2 Aggregate tests

5.2.1 Coarse aggregate relative density

Relative density (specific gravity) of coarse aggregate is readily evaluated following ASTM test procedure C127. A sample of washed coarse aggregate is soaked in water for 24 ± 4 hours. After soaking, the sample is spread onto a towel and rolled against the cloth until visible surface water film is removed. Once the sample has reached saturated surface-dry (SSD) conditions it is weighed on a laboratory scale. The sample is then immersed in 23˚ C water and weighed again. The sample is then dried overnight in an oven at 110˚ C and weighed a third time.

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Figure 60 - Soaked coarse aggregate is spread on a towel for drying to saturated surface-dry condition.

Figure 61 – Close up of coarse aggregate at saturated surface-dry condition

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Figure 62 - Coarse aggregate at SSD is weighed

Figure 63 - Aggregate is immersed and weighed a second time. This apparatus allows the same scale to be used for both weighings.

The results of C127 for the first sample shipment of aggregate are as follows.

Table 13 - Whitewater 3/4" (Coarse Aggregate) Specific Gravity

Oven Dry Bulk Specific Gravity	2.604
SSD Bulk Specific Gravity	2.641
Apparent Bulk Specific Gravity	2.705
Absorption (%)	1.433

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5.2.2 Fine aggregate relative density

The relative density of fine aggregate is found by following ASTM C128. Similar to C127 described above, a sample of fine aggregate is first soaked for 24 ± 4 hours. It is then spread on a tray and allowed to dry at room temperature until it just reaches saturated surface-dry condition. Surface moisture is tested by filling a standard cone mold to overflowing and lightly tamping 25 times. The tamper is held 5 mm above the heaped aggregate surface and allowed to fall freely under the influence of gravity. SSD condition is reached when the molded aggregate slumps slightly when the cone mold is removed.

Figure 64 - Filling cone mold to check surface moisture of fine aggregate.

Figure 65 - Saturated surface-dry conditions are reached when sample slumps slightly when cone mold is removed. This sample does not slump and is still too wet.

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A 500 mL volumetric flask is weighed empty and weighed again filled to the line with water at 23 ˚C. A portion of the water is removed and a weighed sample of 500 g of fine aggregate at SSD is added. The flask with aggregate is again filled to the line with water and gently swirled to remove all trapped air. Finally, the de-aerated flask with water and aggregate is weighed.

Figure 66 - A 500g sample of fine aggregate at SSD is weighed

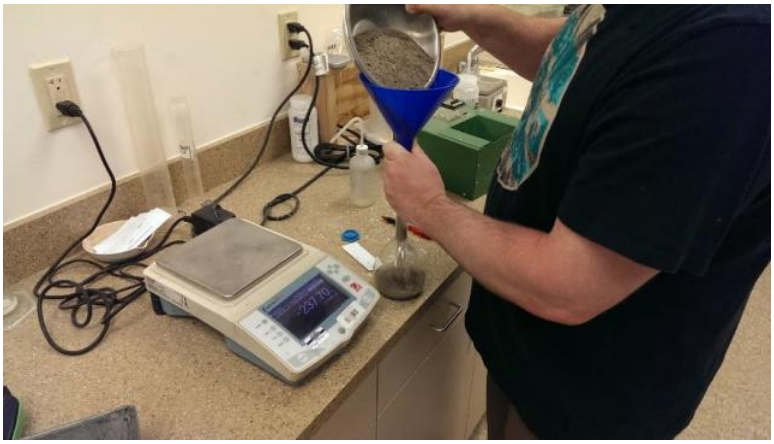

Figure 67 - Adding fine aggregate to volumetric flask.

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Figure 68 - The volumetric flask is rotated to remove trapped air.

The results of ASTM C128 are summarized below.

Table 14 - Whitewater Sand (Fine Aggregate) Specific Gravity

Oven Dry Bulk Specific Gravity	2.583
SSD Bulk Specific Gravity	2.623
Apparent Bulk Specific Gravity	2.690
Absorption (%)	1.551

5.2.3 Coarse aggregate bulk density

Bulk density of coarse aggregate is measured by following ASTM C29. A sample of coarse aggregate is shoveled into a 1/2 ft^3 measure (a bucket-like steel container of known volume). The measure is filled in three lifts. Each lift is levelled by hand and rodded 25 times using a cylindrical steel tamping rod 5/8 inch in diameter and 24 inches long.

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Figure 69 – Coarse aggregate is shoveled into the measure in three lifts.

Figure 70 - Each lift is rodded 25 times.

Results of ASTM C29 testing are summarized below.

Table 15 – Whitewater 3/4" (Coarse Aggregate) Bulk Density

Bulk Specific Gravity	2.641
Bulk Density (pcf)	100.9
Void (%)	39%

5.2.4 Sieve analysis and fineness modulus

A sieve analysis as described in ASTM C136 permits determination of the fineness modulus as well as the grade classification of the aggregate. The procedure for both fine and coarse aggregate is similar. A

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sample of about 1500 g of aggregate is weighed and washed. After washing, it is oven-dried at 110 ± 5 ˚C before being weighed again. The sample is then divided into portions of about 300 g each and each portion is introduced separately to the sieve stack. Fine aggregate is separated using 3/8 inch, #4, #8, #16, #30, #50, #100, and #200 sieves. Coarse aggregate is separated using 1 inch, 3/4 inch, 1/2 inch, 3/8 inch and #4 sieves. The sieves are mechanically shaken for 7 minutes using a "Sally Mae" sieve shaker which vibrates, taps, and rotates the sieve stack automatically. The material retained on each sieve is removed and combined with the corresponding retained material from the other portion runs.

Figure 71- Washing aggregate in preparation for sieve analysis

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Figure 72 Sieve stack ready for coarse aggregate analysis

Figure 73 - Cleaning a screen during coarse aggregate sieve analysis

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Table 16 – Whitewater Sand (Fine Aggregate) Sieve Analysis

Sieve Size	Percent Retained	Percent Passing
3/8"	0.0	100.0
#4	2.8	97.2
#8	15.3	84.7
#16	31.0	69.0
#30	40.6	59.4
#50	67.8	32.2
#100	91.1	8.9
#200	98.3	1.7
Fineness Modulus		2.5

Table 17 - Whitewater 3/4" (Coarse Aggregate) Sieve Analysis

Sieve Size	Percent Retained	Percent Passing
1"	0.0	100.0
3/4"	6.0	94.0
1/2"	56.4	43.6
3/8"	77.8	22.2
#4	97.9	2.1

5.3 Mixing Procedure

As an illustrative example, we consider that a 2 ft^3 sample of concrete must be prepared in accordance with ASTM C192.

The concrete mixer is prepared by buttering with a sufficient quantity of water, cement and sand to thoroughly coat the inside surface of the mixer. The ratios of water, cement, and sand, are chosen to approximately match the proportions of the concrete mixture. The butter mixture is rotated for about a minute and discarded.

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Figure 74 - Buttering the mixer

Figure 75 - Discarding butter

The mixer is loaded by first adding about 1/3 of the water and all of the coarse aggregate. Rotation is then started and the entire quantities of by fine aggregate and cement are added, along with remaining water. Mixing continues for 3 minutes. Rotation is then halted and the concrete allowed to rest for 3 minutes. The mixer is capped during rest to minimize moisture loss. The mixer is restarted and mixing continues for a final 2 minutes. The freshly-mixed concrete is then discharged into a wetted wheel barrow.

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Figure 76 - Charging the mixer

Figure 77 - Mixer is capped to prevent moisture loss during rest period

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Figure 78 - Discharging freshly-mixed concrete

5.4 Freshly-mixed concrete tests

5.4.1 Temperature

The temperature of freshly-mixed concrete is measured in accordance with ASTM C1064. A digital thermometer with remote probe is used. Immediately after discharging the fresh concrete into the wheelbarrow, the temperature probe is placed in the concrete such that its tip is 3 inches below the concrete surface but not in contact with the walls or base of the wheelbarrow. The temperature is read to the nearest 1 ˚F between 2 and 5 minutes after probe placement.

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Figure 79 - Placing temperature probe

5.4.2 Slump

Slump is measured following the method of ASTM C143. A tapered cylindrical mold is wetted and placed small-end up on a flat surface. While standing on the mold handles, the tester fills the mold with freshly-mixed concrete in three roughly equal-volume layers. Each layer is rodded 25 times. While rodding the topmost layer, extra concrete is heaped about the opening to ensure the mold remains filled. After rodding, the top of the mold is struck off using the tamping rod.

Figure 80 – The slump mold is filled in three layers, each tamped 25 times.

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After the mold is struck off, spilled concrete is cleared away from the base of the mold. The tester then removes his feet from the mold handles and lifts the mold in one smooth motion taking 5 ± 2 seconds to lift the mold 12 inches above the concrete. The entire process from filling to mold removal is completed in 2.5 minutes or less.

Figure 81 - The slump mold is lifted in one smooth motion.

Slump is measured by placing the mold next to the concrete and laying the tamping rod across the top of the mold. The distance between the bottom of the tamping rod and the displaced original center of the concrete specimen.

Figure 82 - Measuring slump

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5.4.3 Air content and unit weight

Air content of freshly-mixed concrete is determined using an air meter in accordance with ASTM C173. The unit weight is measured in accordance with ASTM C138 using the air meter bowl, which is of known volume. The inside of the air meter bowl is wetted slightly and weighed. It is then filled with freshly-mixed concrete in two equal layers. Each layer is rodded 25 times and the container is tapped 10-15 times with a rubber mallet after each rodding step. The top surface of the wet concrete is struck off and any excess concrete wiped away. The air meter bowl with concrete is weighed.

Figure 83 – The air meter bowl is filled in two layers, each rodded 25 times.

Figure 84 - After filling, the air meter bowl is struck off. Note the mallet used for tapping the bowl after each layer is rodded.

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The top portion of the air meter is wetted and installed on the measuring bowl. A small quantity of water and isopropyl alcohol is added through the fill port of the air meter, just until it begins to run out the weep hole. The fill port and weep hole are closed and the meter is tilted and rolled to allow trapped air to escape the concrete. Air content is read using the dial pressure gage and comparing with a table provided by the air meter manufacturer.

Figure 85 - The top portion of the air meter is installed.

Figure 86 - A small quantity of water and isopropyl alcohol is added just until it begins to run out the weep hole.

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5.5 Cured-concrete tests

5.5.1 Compressive Strength

Compressive strength of concrete is evaluated in accordance with ASTM C39. Six standard 4-inch diameter, 8-inch long cylinders are prepared after completion of the freshly-mixed concrete tests described in sections 5.4.1 - 5.4.3. Concrete is scooped into plastic molds in two lifts and each lift rodded 25 times per layer with a 3/8" rod. Cylinders are permitted to rest for 30 minutes before capping in order to evaluate bleed. After capping, cylinders are placed in a fog room to cure for 24 -48 hours. After initial cure, molds are removed and the cylinders replaced in the fog room.

Figure 87 - Filling compression test cylinder molds

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Figure 88 - Striking off cylinder molds

Figure 89 - Cylinders were allowed to rest 30 minutes before capping. Notice minimal bleeding.

Two of the cylinders are destructively tested for compression strength after curing for 8 days and two more tested after 28 days. The final two cylinders are reserved for potential future testing.

Compression testing begins with measurement of cylinder diameter and length using calipers. The average of three measurements is accepted. Cylinders are then weighed using a laboratory scale. These measurements permit calculation of circular area, cylinder volume, and concrete density.

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Figure 90 - Measuring cylinder diameter

Cylinders are then capped with either sulfur mortar or unbonded rubber end caps. Generally, sulfur mortar is preferred, but both methods are acceptable. Mix 1 was tested using sulfur end caps, but all subsequent mixes were tested with unbonded rubber caps.

Figure 91 - Installing sulfur mortar end caps

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Figure 92 - Compression test apparatus

The cylinders are then mounted in a compression testing machine and tested to destruction. The load at failure is recorded and strength in force per unit area calculated.

Figure 93 - Fractured cylinder after testing

5.5.2 ASR Expansion

The accelerated mortar bar tests described above (ASTM C1567) are valid for establishing aggregate reactivity. However, that test is performed using crushed aggregate mortar. In order to characterize the expansion of a given concrete mix, a modified version of ASTM C1293 was adopted.

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ASTM C1293 is intended to evaluate the efficacy of fly ash or pozzolans in controlling ASR when reactive aggregates are used. This standard specifies measuring the elongation of 4"x4"x10" concrete prisms that have been cured at 38 ± 2 °C while suspended above water in an enclosed container. C1293 requires use of a standard concrete mix (420 ± 10 kg/m^3 cementitious materials, water-to-cement ratio of 0.42-0.45 by mass, coarse aggregate content of 0.70 ± 0.02 of its dry-rodded bulk density, and adjustment of total alkalinity to 1.25% as Na_2O by doping with NaOH. Measurements are taken periodically up to 24 months after casting.

Measuring pozzolan efficacy is not relevant to the current study. In order to obtain a characteristic value representing the expansivity of a given concrete mix, ASTM C1293 is modified in the following ways.

1. Instead of testing a standard concrete mix, variation is permitted to allow testing of candidate mixes as-designed.
2. Aggregates are not grade-separated and oversize coarse aggregate is not crushed; all materials are employed as-delivered.
3. Two groups of samples are produced. The first is subjected to the storage environment specified in C1567. Specifically, curing temperature is increased to 80°C and samples are immersed in 1N aqueous NaOH.
4. The second group of samples is stored in a fog room at >90% relative humidity and 22 ± 2°C

Specimens are cast using carbon steel molds of standard dimensions. Before casting, the molds are cleaned and scoured using coarse steel wool. Each surface of the mold is coated with a generous layer of "3-in-1" oil, which acts as both a release agent and as a protectant for the steel. The molds embed two studs in the long axis of each sample, which allows length measurement using the same comparometer described in section 4.3.

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Figure 94 - 4-inch square prism molds ready for casting

Molds are filled in two layers. Each layer is rodded at least 25 times. Special care is taken to ensure that concrete is well placed below the measurement stud. Filled molds are placed in a fog room at 21˚C and permitted to cure 24 hours. All samples are then demolded.

Figure 95 - 4 inch prisms are placed in fog room to cure 24 hours after casting.

Typically, half of the samples are returned to the fog room where they are stored uncovered in the fog room at 21˚C for the duration of the study. An important variation from this procedure occurred with Mix 2R, which will be discussed in more depth below.

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The other half of the samples are placed into tap water baths ambient temperature after demolding. The water baths are then placed in an oven at 80 ˚C for a further 24 hours. 48 hours after casting, specimens are removed from water baths and placed in 1 aqueous NaOH solution at 85 ˚C.

Figure 96 - Demolding a concrete prism.

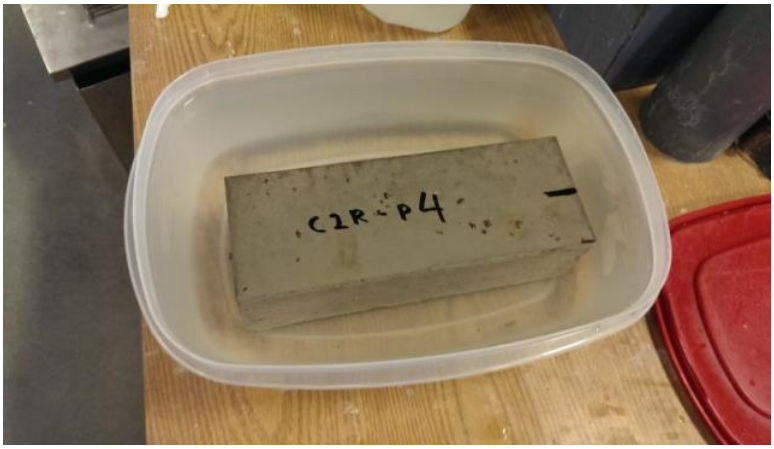

Figure 97 - Concrete prism in container with 1N NaOH.

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Figure 98 – Taking length comparometer readings of a concrete prism.

Length measurements are taken with a comparometer approximately weekly 4 days for a period of two weeks. Subsequent measurements are taken approximately weekly.

5.6 Doping the Cement with Alkali

Adapted from: MCPT Test and its Round Robin Evaluation. Federal Highway Administration. Solicitation No. DTFH61-08-R-00010. Principal Investigator – Dr. Prasad Rangaraju, Clemson University

5.6.1 Reason

In order to accelerate the reaction, the concrete mix must have an Na_2O_{eq} of 1.25-1.6%. The higher value was recommended by Experts from the Danish Technical University and is equivalent to 5-6 kg/m^3.

Part of the Na_2O_{eq} will be provided by the cement whose alkalinity is provided in the Mill Report, and the rest must be provided by doping the water with Sodium Hydroxide.

5.6.2 Procedure

Let us assume that Type I Portland Cement having an alkali content of 0.53 ± 0.1% Na_2O_{eq} (as supplied to us by the Ash Grove Cement Company of Midlothian, TX) should be used and that the alkali content of the concrete should be further boosted to 1.25% by weight of cement by adding adequate reagent grade NaOH to the mix water in order to achieve approximately 1M NaOH, such that the hydroxyl ion concentration in the pore solution is similar to that of the external soak solution (1M NaOH). Also, the impact of alkali

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leaching from the concrete test specimens during the initial storage of test specimens in water for 1 day is minimized.

The alkali content of concrete is calculated based only on the mass of the cement and not that of the supplementary cementitious materials. This assumes that the alkali content of the supplementary cementitious materials is not greater than 4% by mass of the supplementary cementitious material.

> *Example Calculation for determining the amount of NaOH to be added to the mixing water to increase the alkali content of the cement from 0.90% to 1.25%.*

Cementitious Materials content of 1 m^3 of concrete	= 365 kg
Cement Content of Concrete	= 365 kg
Amount of Alkali in the Concrete	= 365 kg x 0.53%
	= 1.93 kg
Specified Amount of Alkali in Concrete	= 365 kg x 1.25%
	= 4.56 kg
Amount of Alkali to be added to Concrete	= 4.56 kg – 1.93 kg
	= 2.63 kg

The 2.63 kg of alkali (i.e. the difference) is the amount of alkali, expressed as Na_2Oequivalent, to be added to the mix water.

The conversion factor to convert Na_2O equivalent to NaOH is 1.291, derived as follows:

$Na_2O + H_2O \rightarrow 2\ NaOH$
1 mole of $Na_2O \rightarrow$ 2 moles of NaOH
61.98 grams/mole of $Na_2O \rightarrow$ 2 x 39.997 grams/mole of NaOH

Therefore, 2 x 39.997 / 61.98 = 1.291.

Therefore, NaOH required to achieve a total alkali content of 1.25% of Na_2O in 1 m^3 of concrete = 1.291 x 2.63 = 3.39 kg/m^3

5.7 Mitigation of ASR by Lithium Nitrate

5.7.1 Control concrete

In order to evaluate the degradation of concrete shear strength due to ASR, it is necessary to generate control specimens that are as similar as possible to the ASR-affected (experimental) specimens, with the single exception that the experimental concrete has experienced ASR while the control concrete has not. Ideally, these reactive and nonreactive concretes should be generated from the same materials (or as similar

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as practical) and exhibit the same mechanical properties absent the effects of the reaction itself. The following strategies were considered for generating a control concrete.

1. Substitute nonreactive aggregate
2. Employ an ASR-mitigating admixture
    a. Fly ash / pozzolan
    b. Lithium nitrate

The use of a nonreactive aggregate for control concrete was rejected due to concerns that doing so would produce unacceptable variation in mechanical properties between the reactive and control specimens. Addition of fly ash was also rejected despite its proven efficacy at mitigating ASR. According to conversations with Whitewater Building Materials, approximately 25% replacement of cement with fly ash has proven effective at limiting reactivity of their aggregates. It was felt by the researcher that such a modification to mix design might require reduction of water-to-cement ratio to maintain desired strength. This change in turn would likely necessitate the inclusion of a water reducing agent to maintain workability. The end result is a control concrete that varies significantly from the experimental concrete in both composition and mechanical properties.

Lithium nitrate is an alternative to fly ash for control of ASR. Its effectiveness is widely accepted, though it is less commonly used for construction than fly ash due to its increased price. The advantage of lithium for production of a control concrete is that it is dosed as an aqueous solution that that replaces a portion of the mix water and is effective in small quantities. This allows the control concrete to be nearly identical to the experimental concrete.

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5.7.2 Procedure

A commercially-prepared lithium nitrate admixture supplied by Grace Concrete Products called *RASIR* was selected. This is a 30% solution of lithium nitrate. Dosing is calculated per manufacturer's recommendation.

$$Lithium\ admixture\ dose\ \left(\frac{L}{m^3};\frac{gal}{yd^3}\right) = \frac{\alpha * \beta * \gamma}{100}$$

where

$$\alpha = Cement\ content\ of\ concrete\ \left(\frac{kg}{m^3};\frac{lb}{yd^3}\right)$$

$$\beta = Alkali\ content\ of\ cement\ in\ \%\ as\ Na_2O$$

$$\delta = Coefficient\ \left(4.6\ for\frac{L}{m^3};\ 0.55\ for\frac{gal}{yd^3}\right)$$

Because the lithium nitrate solution is a liquid, mix water is reduced proportional to the amount of *RASIR added.*

$$w = w_0 - 0.84 * \left(LiNO_3\ admixture\ volume\ (L;gal)\right)$$

where

$$w = adjusted\ concrete\ mix\ water\ volume\ (L;gal)$$

$$w_0 = original\ concrete\ mix\ water\ volume\ (L;gal)$$

Example Calculation for determining the amount of Grace RASIR and adjusted water content of a control concrete mix.

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Alkali content of cement	= 0.91 % as Na_2O
Cement content of Concrete	= 365 kg/m^3
δ Coefficient	=4.6
$LiNO_3$ Dose	=15.3 L/m^3
Water content of concrete	=208 L/m^3
Water reduction factor	=0.84
Adjusted water content	= 195 L/m^3

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CHAPTER 6

CONCRETE MIX DESIGN

Concrete mix design plays a significant role in the progression of the alkali silica reaction. Even a concrete mixed with highly reactive aggregates may exhibit slow or small expansion due to inadequate alkali content to attack reactive minerals, insufficient water to maintain ionic mobility and hydrate ASR gels, or excessively high or low permeability.

It is important to note that no effort was made to precisely duplicate the concrete used in the Seabrook reactor containment vessel. The authors have access to neither the aggregates nor cement that were convenient to its builders. Furthermore, construction concretes are never designed with the intention of magnifying ASR effects; these are exclusively deleterious and unintended. Therefore, responsible designers avoid reactive aggregates or include pozzolans to mitigate their effects if such aggregate is unavoidable.

However, the purpose of this study is to deliberately create a concrete which will exhibit rapid, vigorous expansion. Therefore, best practices for producing durable concrete must be deliberately avoided. The best that can be hoped is to produce a model concrete that is reasonably similar to a construction material.

6.1 Concrete mix objectives

It is believed that prototype concretes were designed to have a compressive cylinder strength of at least 4000 psi, with actual strength likely closer to 4,500 psi. The model concrete must have sufficient

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workability to be vibrated between the closely-spaced shear studs of the sample end plates is also required. A slump of at least 5" is considered sufficient. Finally, the expansion target for our reactive samples is 0.5%. This is an ambitious target, and will require some experimentation to achieve.

There are a number of factors which may be adjusted to increase expansion while maintaining workability and strength. The most important of these is the alkali content of the cement. Sufficient alkali must be provided to fully activate reactive aggregate minerals. This may be achieved by selecting cements with high natural alkalinity and by artificially boosting alkalinity through addition of sodium hydroxide.

Considering that experimental samples must reach their expansion target in six months or less, it is probable that the bulk of expansion will be due to the reactivity of the fine aggregates. Indeed, the role of the coarse aggregates may be quite small. Thus increasing the ratio of fine aggregate to coarse aggregate may reasonably be expected to increase expansion. However, it is unrealistic to boost sand content excessively without producing more of a model mortar than a concrete.

Both very low and high air content can inhibit expansion. Excessively high air content allows a large pore volume for expanding ASR gels to fill without inducing gross strain. Low air content, such as is present in high-performance concrete occurs with low water-to-cement ratios. The lack of water in such concrete can limit ionic mobility and hydration of ASR gel. Thus there is likely an ideal w/c ratio that we hope to find by experimentation.

Table 18 – Concrete mix objectives

Compressive Strength	4,500 psi	31.0 MPa
Slump	4.5-6.5 "	11-14 cm
Expansion	0.5%	
Air Content	Less than 3%	

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Several mix designs were considered and tested until objectives for strength, workability, and expansion were met.

6.2 Concrete mix design 1

The first mix was designed by following ACI 211.1, Chapter 6 with the intent meeting the objectives for mechanical properties described in Table 18. The first mix would also form a baseline against which future refinements could be compared. Recall that admixtures such as water-reducing agents or retarders were avoided in order to minimize the number of variables that might affect ASR expansion. Of particular concern was obtaining adequate workability to allow thorough penetration of concrete between the closely-spaced shear studs in our sample end plates.

Slump was therefore selected at 5 inches, one inch greater than ACI recommendations in Table 6.3.1 for beams, reinforced walls, and building columns. Maximum aggregate size is limited to 3/4 the clear distance between reinforcement members. Considering the 1.5 inches of clear space between shear studs in sample end plates, maximum aggregate size of 3/4 inches was selected. Water to cement ratio was estimated following Table 6.3.4 at 0.57, which is consistent with suggestions by researchers in Switzerland (EMPA, Holcim, and EPFL) that *w/c* should be no less than 0.45 in order to avoid inhibition of ASR by pore-space desiccation during hydration.

Table 19 – Mix 1, Reactive concrete design

Material	lbs/yd3	kg/m3
Portland Cement, Type 1	614	365
Fine Aggregate: Manufactured Sand	1187	705
Coarse Aggregate: 3/4" Crushed Rock	1771	1052
Water	350	208
w/c	0.57	0.57

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6.2.1 Test results

A 1 ft^3 sample of concrete Mix 1 was prepared on June 11, 2015 and subject to the battery of freshly-mixed concrete tests described above in section 5.4. The results of these tests are summarized below.

Table 20 - Freshly-mixed concrete testing results: Mix 1

Temperature of freshly-mixed concrete (°F)	77
Ambient temperature (°F)	75
Slump (in)	6.25
Air Content (%)	0.7%
Unit Weight (lbs/ft^3)	147.6

Reviewing these results, we find that this mix exceeds the target slump value by 1.25", but easily meets the desired air content. The higher-than-anticipated slump is deemed acceptable, since a higher slump provides acceptable workability, provided no bleed occurs. Mix 1 was found to bleed very slightly (refer to Figure 89 below), less than is typically considered problematic according to Dana Schwartz.

Compression testing was conducted on June 19th, 2015 and on July 9th, 2015, 8- and 28-days after casting, respectively. The 28-day cylinder strength of Mix 1 is nearly ideal.

Table 21 - Compressive strength of Mix 1

Age (Days)	Strength (psi)
8	4170
28	4430

Expansion was measured using the modified version of ASTM C1293 described in section 5.5.2, with the exception that no samples from Mix 1 were stored in the fog room. All Mix 1 samples were kept immersed in 1 M NaOH and stored in an oven at 80C. Results are summarized in Figure 99 below.

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Table 22 - Elongation of 4x4x10 prisms, Mix 1, Reactive

Curing Conditions	Elongation	Age
80°C, 1M NaOH	0.247%	65 days

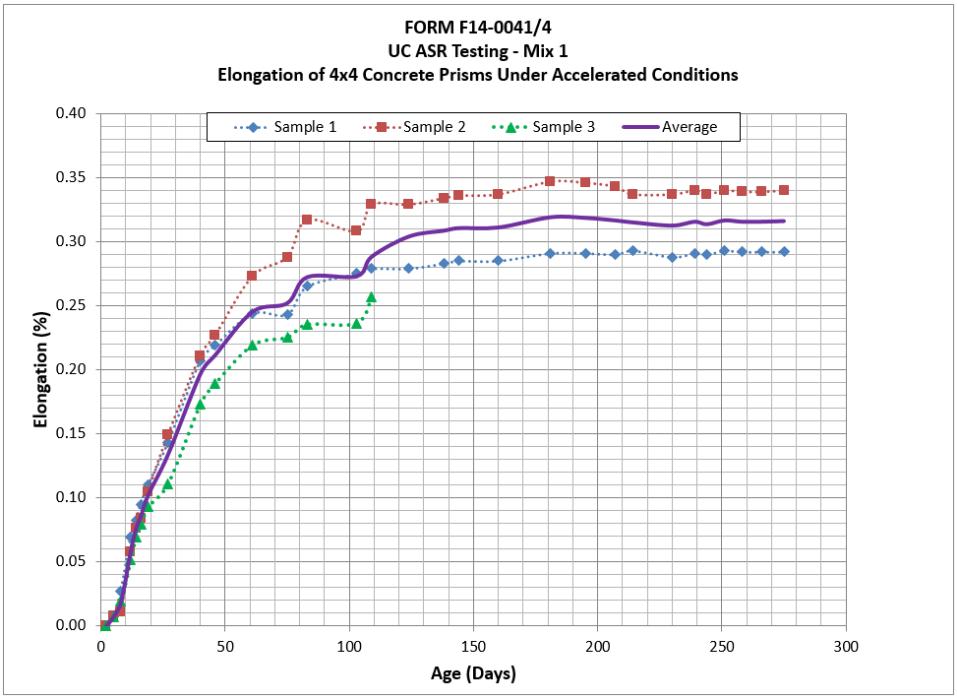

Figure 99 - ASR expansion of Mix 1

6.2.2 Discussion

Contrasting Figure 99 with Figure 54 suggests that concrete prisms are much less expansive than mortar bars mixed using the same aggregates. Mix 1 concrete bars expanded only 0.27% after 100 days while mortar bars cast with the same fine aggregate expanded roughly 1.15% after 100 days under the same curing conditions. Ultimate elongation of Mix 1 is 0.32%, reached after about 180 days.

Subsequent mix designs will focus on improving reactivity while maintaining acceptable mechanical properties.

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6.3 Mix design 2, reactive

The goal of the second reactive mix design (called hereafter Mix 2R) is to evaluate the potential for increasing ASR expansion by boosting alkalinity. The cement used for the first mix was provided by Midlothian of Ash Grove, Texas and has alkalinity of 0.45% as Na_2O. For mix 2R, a different cement provided by Holcim of Hagerstown, Maryland with alkalinity of 0.91% as Na_2O was used. The alkalinity of this mix was further increased to 1.25% as Na_2O by addition of sodium hydroxide as described in section 5.6 above. Mix 2 is similar in all other ways to mix 1 above.

Table 23 - Mix 2, Reactive concrete design

Material	**lbs/yd³**	**kg/m³**
Portland Cement, Type 1, Holcim	614	365
Fine Aggregate: Manufactured Sand	1,205	716
Coarse Aggregate: 3/4" Crushed Rock	1,753	1,041
Water	350	208
w/c	0.57	0.57
Admixtures	**kg/yd³**	**kg/m³**
NaOH(s) Doping Additive	1.22	1.6

6.3.1 Test results

A 2.5 ft^3 batch of concrete mix 2R was produced on August 31, 2015. The results of testing on the freshly-mixed concrete are summarized below.

Table 24 - Freshly mixed concrete testing results: Mix 2 Reactive

Property	Value
Slump	8.3 in
Air content	2.1%
Unit weight	145.7 pcf
Wet-concrete temperature	83.4 °F
Ambient temperature	78.2 °F

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Note that the slump of mix 2R is excessively high. Some variance in properties is to be expected when changing a major component like cement. The air content of this mix is higher than ideal.

The compressive cylinder strength of mix 2R was tested by on September 8th, 2015, and again on September 25th, 2015.

Table 25 –Compressive strength of Mix 2, Reactive

Property	Value
Cylinder strength at 8 days	3,920 psi
Cylinder strength at 28 days	4,760 psi

Note that the compressive strength of mix 2R after 28 days of curing is quite close to our target value.

Unlike previous the elongation test, prisms of mix 2R, were divided into two groups and were stored under different conditions. Immediately after casting, molds with fresh concrete were placed in a fog room to cure 24 hours. Samples were then demolded and placed into water baths which were themselves moved to an 80°C oven. Twenty-four hours later, all samples were transferred to 1M NaOH at 80°C and returned to the oven. Eight days after casting, two bars were removed from the oven and the NaOH soak containers and placed in the fog room 21°C. The remaining two bars were left undisturbed in the oven at 80°C and immersed in 1M NaOH.

Table 26 - Elongation of 4x4x10 prisms, Mix 2 Reactive

Curing Conditions	Elongation	Age
80°C, 1M NaOH	0.478%	65 days
21°C, Fog Room	0.191%	65 days

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Note that mix 2R is significantly more reactive than mix 1. However, reducing cure temperature to near-ambient greatly retards expansion. Even after many months, samples stored in the fog room reached only 0.251% elongation, while those in the oven reached 0.688%.

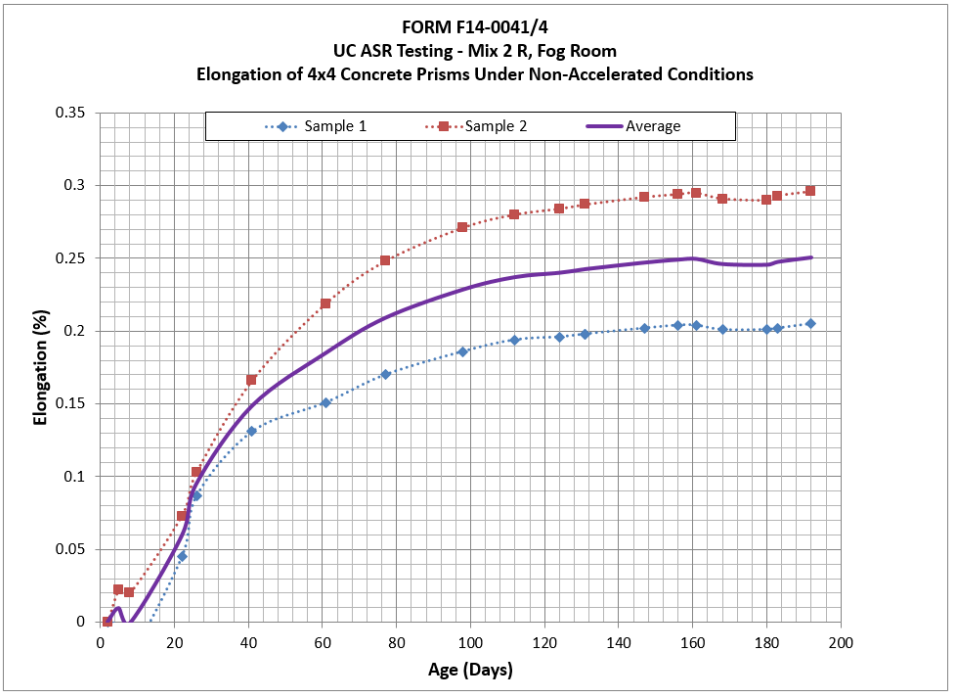

Figure 100 - Elongation of Mix 2 Reactive, non-accelerated conditions

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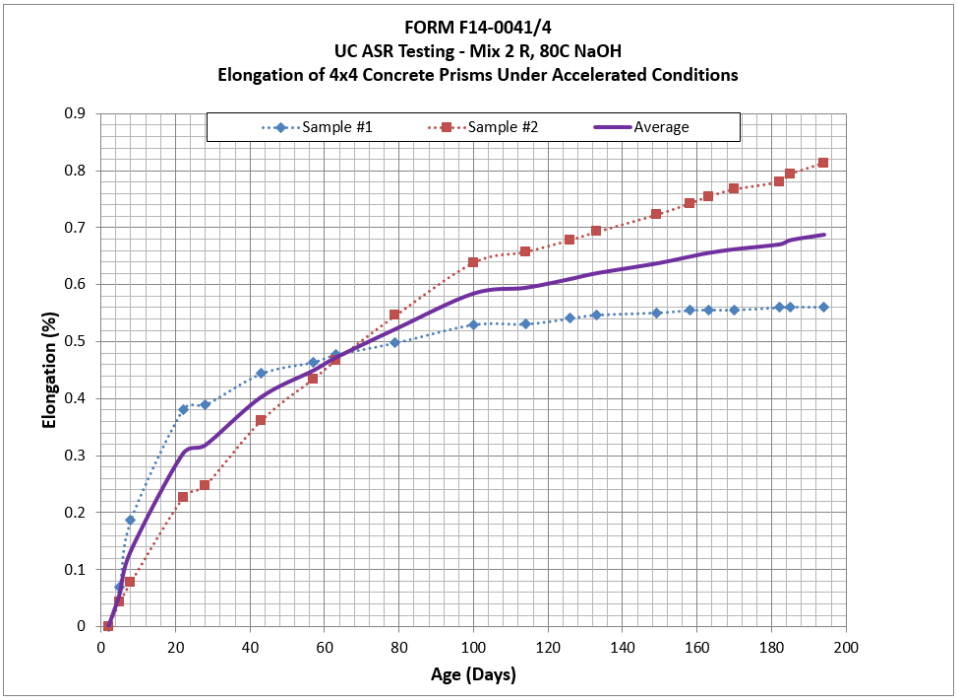

Figure 101 - Elongation of Mix 2 Reactive, accelerated conditions

6.3.2 Discussion

The slump of mix 2R is higher than the target and should be reduced. Air content is also slightly high. Elongation of bars stored at 80 ˚C exceeded target values of 0.5% after about 70 days, but those stored at 21 ˚C did not. Since it is difficult to anticipate how the larger shear samples will expand, subsequent designs will attempt to further increase expansion.

It is important to note that measures taken to boost reactivity in subsequent designs were successful only for those bars stored at high temperature and immersed in NaOH. Samples of later, more reactive mixes which were stored in the fog room underperformed those of mix 2R. It appears that the initial 6 days these unaccelerated mix 2R samples spent soaking in 1M NaOH had a lasting effect on expansion. This idea is developed in more detail below.

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6.4 Mix design 2, nonreactive

It is beneficial produce a control concrete mix with composition as similar as possible to reactive concrete with the single exception that the control mix does not undergo ASR expansion. To evaluate the efficacy of using aqueous lithium nitrate to prevent ASR expansion in an otherwise reactive mix, a nonreactive version of mix 2 was produced (hereafter called mix 2NR). A 30% solution of lithium nitrate manufactured by Grace Concrete Products called *RASIR* was used to produce nonreactive concrete.

Table 27 - Mix 2, Nonreactive concrete design

Material	**lbs/yd³**	**kg/m³**
Portland Cement, Type 1, Holcim	614	365
Fine Aggregate: Manufactured Sand	1,227	729
Coarse Aggregate: 3/4" Crushed Rock	1,786	1,786
Water	329	195
w/c	0.57	0.54
Admixtures	**L/yd³**	**L/m³**
Lithium Nitrate Additive	11.7	15.3

6.4.1 Test results

A 2 ft^3 batch of concrete Mix 2NR was produced on September 1st, 2015.

Table 28 - Freshly mixed concrete testing results: Mix 2NR

Property	**Value**
Slump	7.0 in
Air content	1.7%
Unit weight	145.4 pcf
Wet-concrete temperature	81.6 °F
Ambient temperature	82.1 °F

The slump of Mix 2NR is one inch lower than that of mix 2R. However, considering that it is rarely practical to attempt to control slump to better than ±1 inch, this may be a result of statistical uncertainty.

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The compressive cylinder strength of mix 2NR was tested on September 8th, 2015, and again on September 25th, 2015.

Table 29 –Compressive strength of Mix 2NR

Property	Value
Cylinder strength at 7 days	4,160 psi
Cylinder strength at 27 days	5,030 psi

The compressive strength of the nonreactive version of Mix 2NR is somewhat higher than that of Mix 1. It may be that the water-to-cement ratios of the control and experimental mixes must differ in order to achieve similar strengths.

All samples of Mix 2NR were cured in a fog room and were never soaked in 1M NaOH. Because this mix is only intended for use as a control, no effort was made to accelerate ASR in samples produced from it. The goal of the nonreactive mix is to limit expansion to nearly zero.

Table 30 - Elongation of 4x4x10 prisms, Mix 2NR

Curing Conditions	Elongation	Age
21°C, Fog Room	0.006%	65 days

Mix 2NR exhibits very little expansion, as desired.

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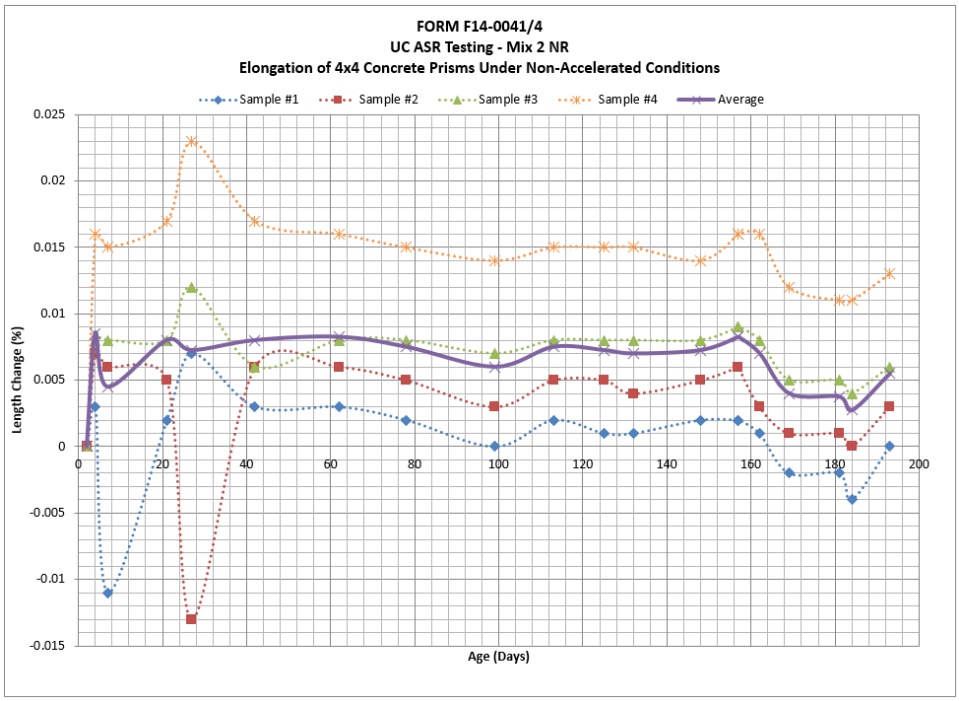

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Figure 102 - Elongation of Mix 2NR, non-accelerated conditions

6.4.2 Discussion

Lithium nitrate is ideal for the purpose of generating a control concrete. Expansion of mix 2NR is essentially zero. Simultaneously, mechanical properties were altered only slightly. Contrasting results of mix 2R and 2NR, we find that slump declined 16%, strength increased 6%, and expansion declined 97%. It is doubtful that any other method of ASR control, such as fly ash, kaolinite, or other pozzolans could outperform $LiNO_3$ in terms of halting ASR expansion without drastically influencing mechanical properties.

6.5 Mix design 3,

Increasing the alkalinity of mix 2R relative to mix 1 corresponded to an increase in ASR expansion. The objective of Mix 3 is to further increase reactivity without adversely affecting other properties. This is achieved by adjusting the following parameters.

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1. Because fine aggregate exposes a much greater surface area to caustic pore water solution than an equal mass of coarse aggregate, it is reasonable to assume that sand is the primary driver of elongation within the timescale of the study. Thus, increasing the proportion of sand to gravel in the mixture should increase gel formation and elongation. For Mix 3, a volume proportion of about 34.5% fine aggregate to total concrete was used.
2. Increasing the amount of fine aggregate in the mix should increase expansion, provided sufficient alkalinity is available to drive the reaction. Thus, alkalinity of the reactive mix is increased to 1.6% as $Na_2O_{(s)}$, which corresponds to 5.84 kg/m^3.
3. Water-to-cement ratio was reduced to about 0.50. The intent of this change is to reduce slump and offset any strength reduction due to increasing the content of fine aggregate.

Making these adjustments to the prior reactive concrete mix yields the following design.

Table 31 - Mix 3 concrete design

Material	**lbs/yd^3**	**kg/m^3**
Portland Cement, Type 1, Holcim	614	365
Fine Aggregate: Manufactured Sand	1,525	906
Coarse Aggregate: 3/4" Crushed Rock	1536	912
Water	310	184
w/c	.50	.50
Admixtures	**kg/yd^3**	**kg/m^3**
NaOH(s) Doping Additive	2.48	3.25

6.5.1 Test results

On November 16th, 2015 a 1 ft^3 sample of Mix 3 was prepared at Fall Line Testing.

Aggregate moisture is measured the morning before mixing a test batch and the mix is adjusted accordingly. Unfortunately, I made a mistake in my calculations which caused the as-mixed water content to be less than the 310 lbs/yd^3 specified above.

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Table 32 - Mix 3 test batch actual weights

Volume of test batch (ft^3)	1.0
Material	**lbs**
Portland Cement, Type 1, Holcim	22.7
Fine Aggregate: Manufactured Sand	58.2
Coarse Aggregate: 3/4" Crushed Rock	56.7
Water	10.0
Admixtures	**g**
NaOH(s) Doping Additive	92.03

Considering the observed moisture content of the fine aggregate pile on the day of mixing was 4.46 % and that of the coarse aggregate pile was 1.10%, one may note that adding 12.95 lbs of water would produce the design water content, but only 10.0 lbs were added to the test batch.

The combination of adding much more sand than Mix 2R coupled with lower-than-design water content caused Mix 3R to have an unacceptably low slump.

Table 33 - Freshly mixed concrete testing results: Mix 3

Property	Value
Slump	2.5 in
Air content	2.8%
Wet-concrete temperature	68 °F
Ambient temperature	65 °F

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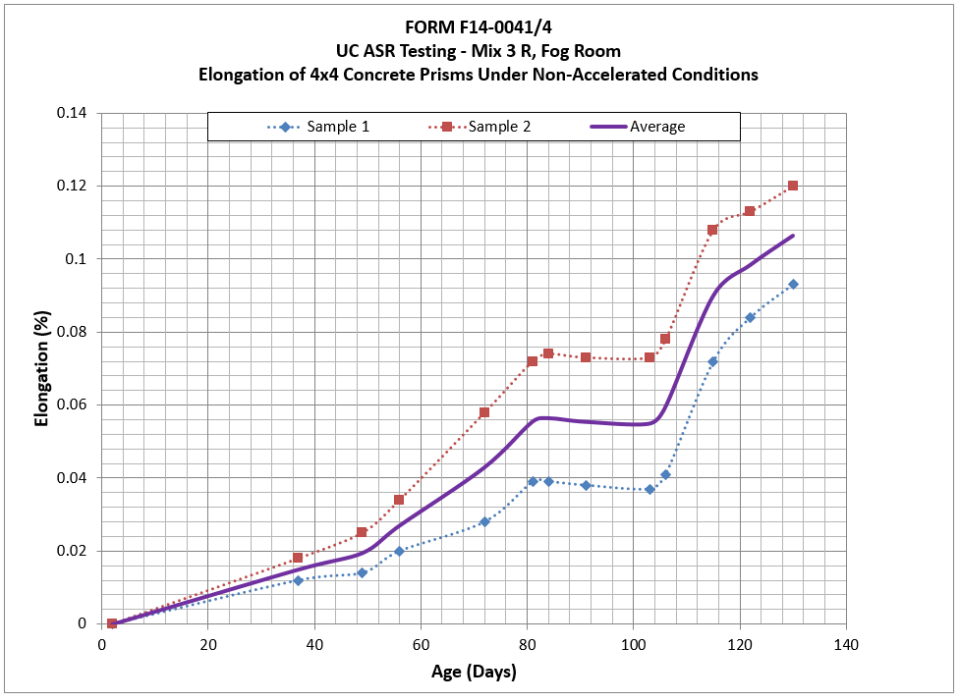

Figure 103 - Elongation of Mix 3, under nonaccelerated conditions

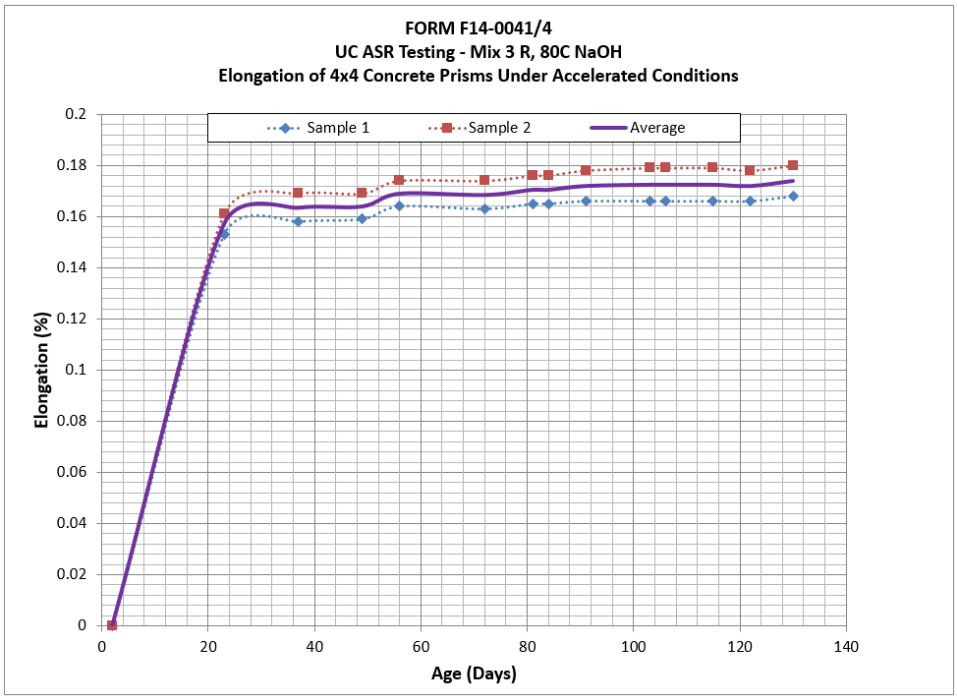

Figure 104 - Elongation of Mix 3 under accelerated conditions

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6.5.2 Discussion

Mix 3 was rejected based both on its unacceptably low slump and on my mistake mixing the test batch properly. While the elongation data may be useful for developing a sense for the effects of fog-room conditioning vs conditioning in hot sodium hydroxide solution.

6.6 Mix design 4

The objective for Mix 4 were to correct the mistakes which caused Mix 3 to exhibit unacceptably low slump, while retaining the high sand and alkali content. Comparison of the as-mixed content of Mix 2R to that of Mix 3 suggested that the increased proportion of fine aggregate to coarse in Mix 3 contributed somewhat to slump reduction. Therefore, the water content of Mix 4 is between those of Mix 2 and Mix 3.

Table 34 – Mix 4 concrete design

Material	**lbs/yd^3**	**kg/m^3**
Portland Cement, Type 1, Holcim	636	378
Fine Aggregate: Manufactured Sand	1,585	941
Coarse Aggregate: 3/4" Crushed Rock	1362	809
Water	350	208
w/c	.55	.55
Admixtures	**kg/yd^3**	**kg/m^3**
NaOH(s) Doping Additive	2.57	3.37

6.6.1 Test results

A test batch of Mix 4, reactive was produced on December 21, 2015 at Fall Line Testing.

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Table 35 - Freshly mixed concrete testing results: Mix 4

Property	Value
Slump	4.5 in
Air content	2.7%
Unit weight	144.7 pcf
Wet-concrete temperature	69.8 °F
Ambient temperature	64.2 °F

Note that slump is within just within the target range, providing sufficient workability to vibrate concrete between the closely-spaced shear studs of the sample end plates. The air content is slightly higher than ideal, closer to that of Mix 3 than Mix 2R. This may be an unavoidable consequence of increased sand content.

Two 4x8" cylinders were tested to failure in compression 7 days after casting and four more after 28 days. While the 7-day results were acceptable, the 28 day results were slightly below the target of 4000 psi. Of particular concern, two of the 28-day cylinders failed at less than 3800 psi. Mix 4 must be rejected on the basis of strength.

Table 36 - Compressive strength of Mix 4

Property	Value
Cylinder strength at 7 days	3500 psi
Cylinder strength at 27 days	3958 psi

Similar to previous cases, two prisms were conditioned in hot 1M $NaOH_{(aq)}$ and two prisms in a fog room at ambient temperature. Plotting the expansion curves allows interpolation of the 65-day elongation figures.

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Table 37 - Elongation of 4x4x10 prisms, Mix 4

Curing Conditions	Elongation	Age
80°C, 1M NaOH	0.587%	65 days
20°C, Fog Room	0.030%	65 days

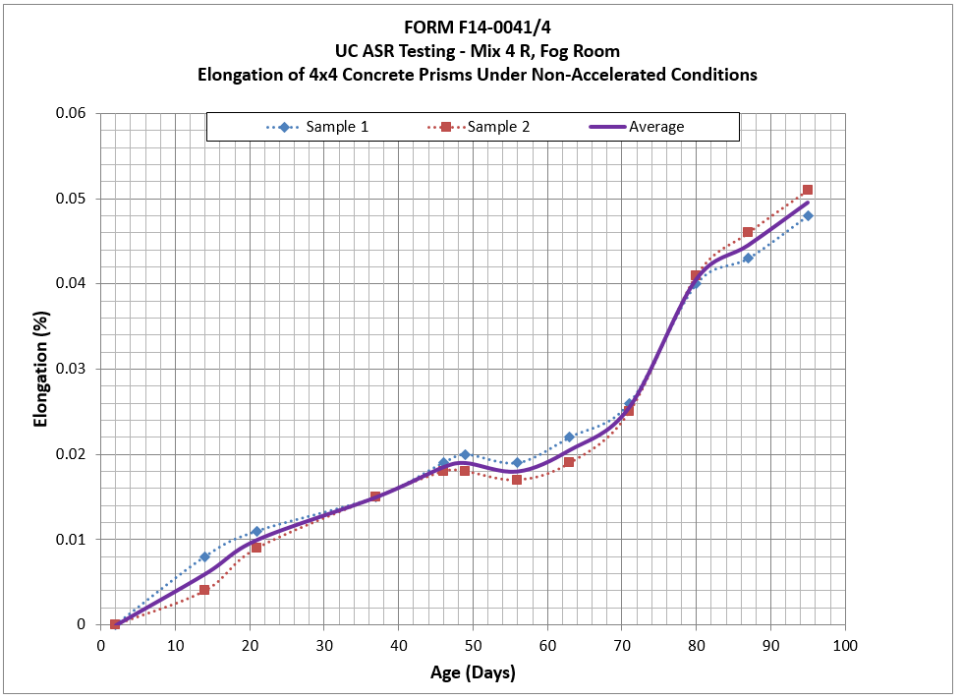

Figure 105 - Elongation of Mix 4, under nonaccelerated conditions. Note the dip between 50 days and 70 days. During this time period only one of three Hydrofogger units were operational.

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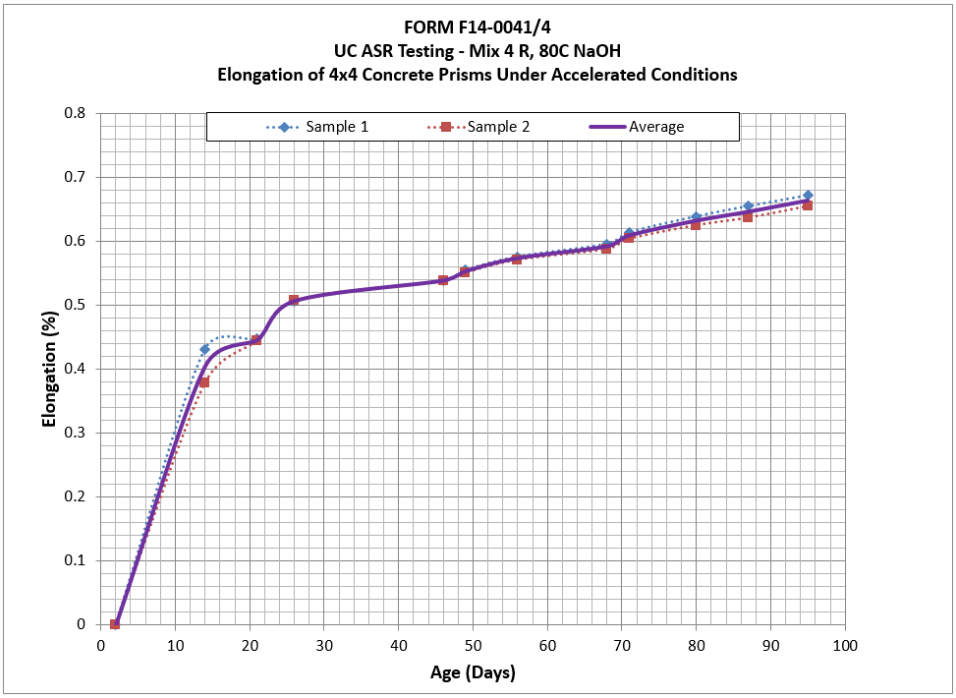

Figure 106 - Elongation of Mix 4 under accelerated conditions

6.6.2 Discussion

Note that, under accelerated conditions, expansion of Mix 4 exceeds that of Mix 2R and exceeds the target value of 0.5% after 65 days. This suggests that the provision of additional sand and alkali are effective at boosting reactivity.

However, elongation of specimens conditioned in the fog room is significantly retarded in comparison to Mix 2. I suspect that the unexpectedly slow expansion may be due to partial drying of the specimens in the fog room. Normally, the fog room at Fall Line uses three Hydrofogger units to maintain humidity, however, only one unit was found to be operational on when measurements were taken on December 23rd. It is conceivable that humidity may have dropped over the holidays, allowing some degree of shrinkage to occur. Once all three Hydrofogger units were brought back online on January 4th, expansion accelerated and returned to trend.

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6.7 Mix design 5

Considering the failure of Mix 4 to reach strength targets after 28 days, a fifth mix was designed with the objective of increasing strength without altering other properties. A new w/c ratio was interpolated between tabulated values and the measured results of Mix 4, resulting in a decrease of 0.02. Likewise, water content was also interpolated to maintain workability, resulting in an increase of 3 lbs/yd^3. The resulting design is tabulated in Table 38

Table 38 – Mix 5 concrete design

Material	**lbs/yd^3**	**kg/m^3**
Portland Cement, Type 1, Holcim	666	396
Fine Aggregate: Manufactured Sand	1,552	922
Coarse Aggregate: 3/4" Crushed Rock	1,362	809
Water	353	210
w/c	.53	.53
Admixtures	**kg/yd^3**	**kg/m^3**
NaOH(s) Doping Additive	2.69	3.52

6.7.1 Test results

A two cubic foot batch of Mix 5 was produced on January 22, 2016 at Fall Line Testing and subjected to the usual testing program.

Table 39 - Freshly mixed concrete testing results: Mix 5

Property	Value
Slump	6.5 in
Air content	1.7%
Unit weight	146.4 pcf
Wet-concrete temperature	68.7 °F
Ambient temperature	66.2 °F

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All initial test results are positive. While the slump is at the high end of acceptability, no bleeding was evident in cylinders or prism molds

The compressive strength of mix 5 was tested on January 29th, 2016 and again on February 29th, 2016. Its 28-day strength is higher than our target value of 4500 psi. However, it is common for construction concretes to exceed their rated strength and it is not unreasonable to consider a strength of 5100 psi to be representative of a construction concrete rated at 4000 psi.

Table 40- Compressive strength of Mix 5

Property	Value
Cylinder strength at 7 days	3700 psi
Cylinder strength at 27 days	5100 psi

Similar to previous cases, two prisms were conditioned in hot 1M $NaOH_{(aq)}$ and two prisms in a fog room at ambient temperature.

Table 41-Elongation of 4x4x10 prisms, Mix 5

Curing Conditions	Elongation	Age
80°C, 1M NaOH	0.590%	65 days
21°C, Fog Room	0.033%	65 days

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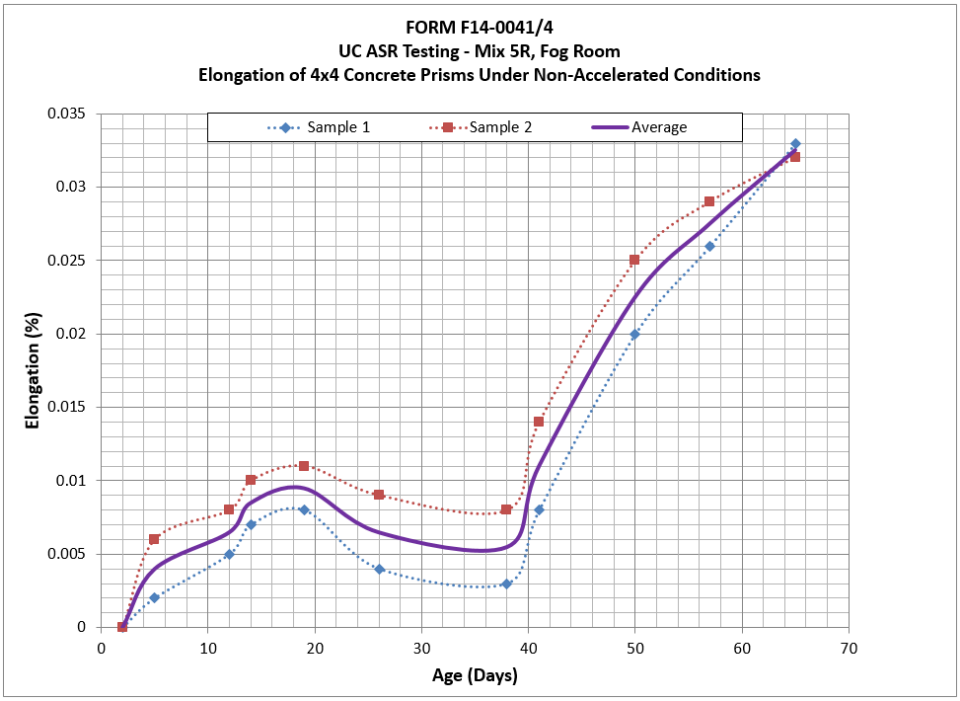

Figure 107 - Elongation of Mix 5, under nonaccelerated conditions

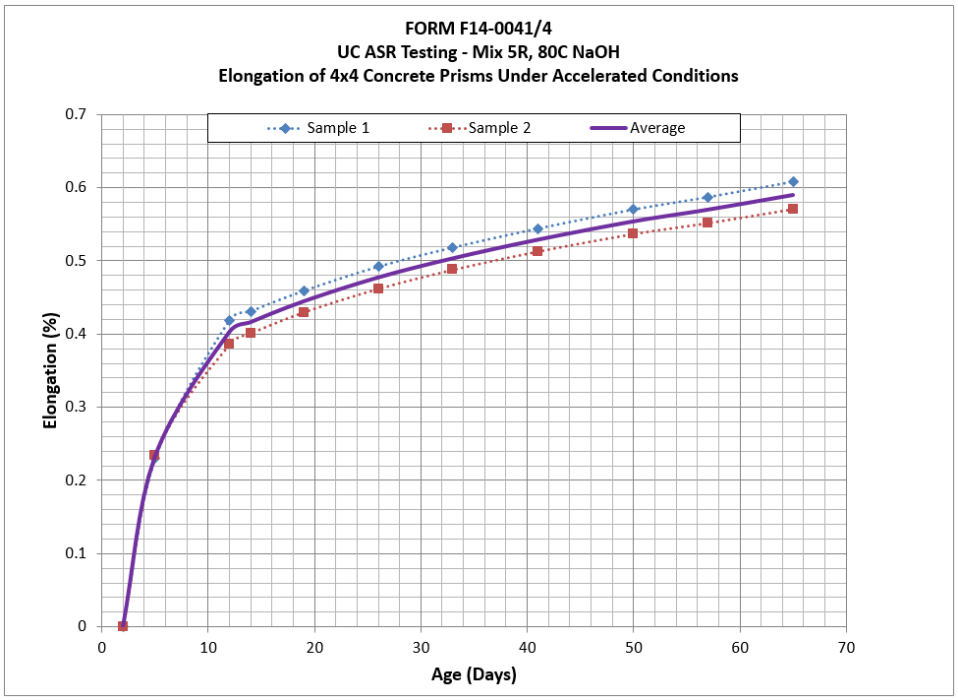

Figure 108 - Elongation of Mix 5 under accelerated conditions

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6.7.2 Discussion

Continuing a pattern first observed in Mix 4, the accelerated samples readily exceed the expansion target within a relatively short period of time. However, the non-accelerated samples significantly underperform.

As remarked above in the discussion for Mix 4, the result of the Hydrofogger failure is evident as a dip in elongation between age of 20 days and 40 days in Figure 107. In fact, not only did the drying effect of lower humidity arrest the expansion of Mix 5, these samples shrank somewhat during the drying period. This serves as a reminder of the critical nature of moisture control in quantification of ASR.

Concrete mix design 5 meets all design objectives outlined in Section 6.1. Slump and strength are within the target range. Expansion when stored in 1M NaOH and 80°C is rapid and exceeds the target of 0.5% after 65 days. However, expansion is greatly reduced when stored uncovered in a high-humidity fog room at 21°C. It is reasonable to assume that this is due in part to the reduced temperature, but also due to alkali leaching. Samples in the fog room are subjected to a continuous flow of condensation which collects on the samples and drips onto the floor.

6.1 Temperature Effects

As noted in Section 3.7, experimental shear specimens are to be stored in the fog room in the structures laboratory of the University of Colorado. This facility is capable of maintaining a temperature of 38°C. This temperature is midway between that of the fog room at Fall Line Testing & Inspection (21 °C) and the oven (80° C). Experimental shear specimens are to be protected from the effects of alkali leaching and drying shrinkage by provision of a steady sprinkling of aqueous 1.5M NaOH. However, it remains to be established how the change in storage temperature might affect expansion.

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The temperature dependence of ASR can be estimated using Larive's kinetic model (Larive, 1998), which described concrete expansion as a function of both absolute temperature (T) and time (t).

$$\varepsilon(t,T) = \frac{1 - e^{\left(\frac{1}{\tau_C(T)}\right)}}{1 + e^{\left(\frac{1-\tau_L(T)}{\tau_C(T)}\right)}}$$

The two parameters τ_L and τ_C are the latency and characteristic times of the sigmoidal strain function, respectively. Each of these parameters may be calculated for some given temperature if they are known for some other temperature (T_0) as follows (Ulm, Coussy, Li, & Larive, 2000):

$$\tau_C(T) = \tau_C(T_0)e^{\left(U_C\left(\frac{1}{T}-\frac{1}{T_0}\right)\right)}$$

$$\tau_L(T) = \tau_L(T_0)e^{\left(U_L\left(\frac{1}{T}-\frac{1}{T_0}\right)\right)}$$

The values of $\tau_C(T_0)$ and $\tau_L(T_0)$ may be obtained from curve fitting experimental data. Note that reducing storage temperature increases the time for concrete to achieve maximum strain, but does not reduce maximum strain. However, reducing pore humidity does alter maximum strain. Thus, this conversion is only valid for scenarios in which humidity is unchanging.

The most reasonable data set to use for this purpose is that obtained using concrete mix 5 under accelerated conditions (Figure 108). The accelerated prisms were stored immersed in 1M NaOH, and thus experienced nearly infinite pore humidity. Experimental shear specimens will be stored under a constant flow of 1.5M NaOH, and therefore also remain very nearly saturated. Furthermore, the caustic wash solution prevents alkali leaching in both scenarios.

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Results of curve fitting generously provided by Dr. M. Hariri-Ardebili of the University of Colorado are presented below. The result is latency time $\tau_L(353\,K) \approx 0$ and characteristic time $\tau_c(353\,K) = 7.7\,days$ with acceptable goodness-of-fit $R^2 = 0.9$.

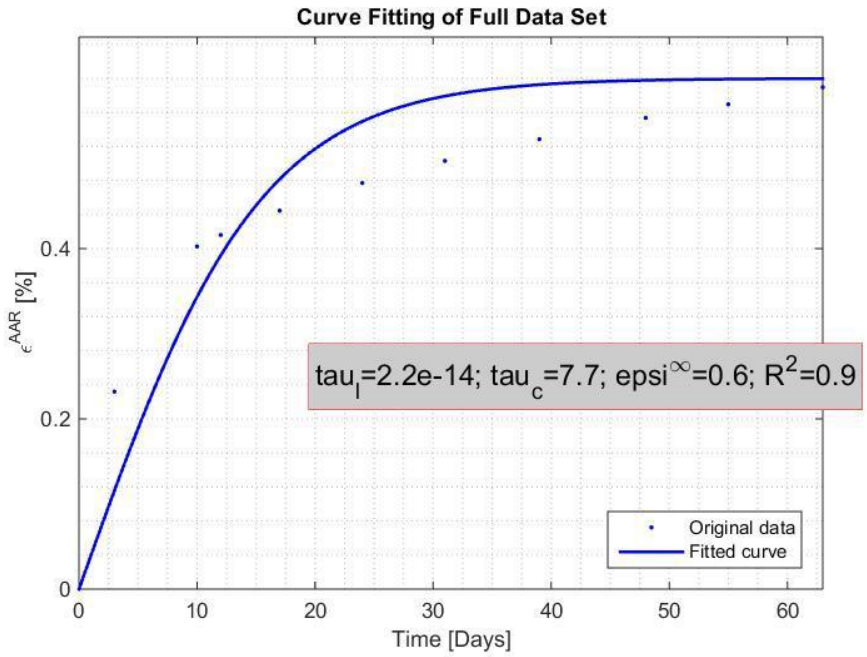

Figure 109 - Curve fitting data from concrete mix 5 (80C, 1M NaOH) to Larive's kinetic model.

Using these values, characteristic times at 38°C may be obtained: $\tau_L(311\,K) \approx 0$ and $\tau_c(353\,K) = 60.8\,days$. The resulting expansion curve is presented in Figure 110. Observe that reducing storage temperature does not alter maximum strain, but does extend the time required to reach it. Inspection of this plot reveals that approximately 145 days are required to achieve the target expansion of 0.5%. This is acceptable, as it is quite likely the target value of 0.5% expansion will be reached within the 6-month period permitted by the overall project schedule.

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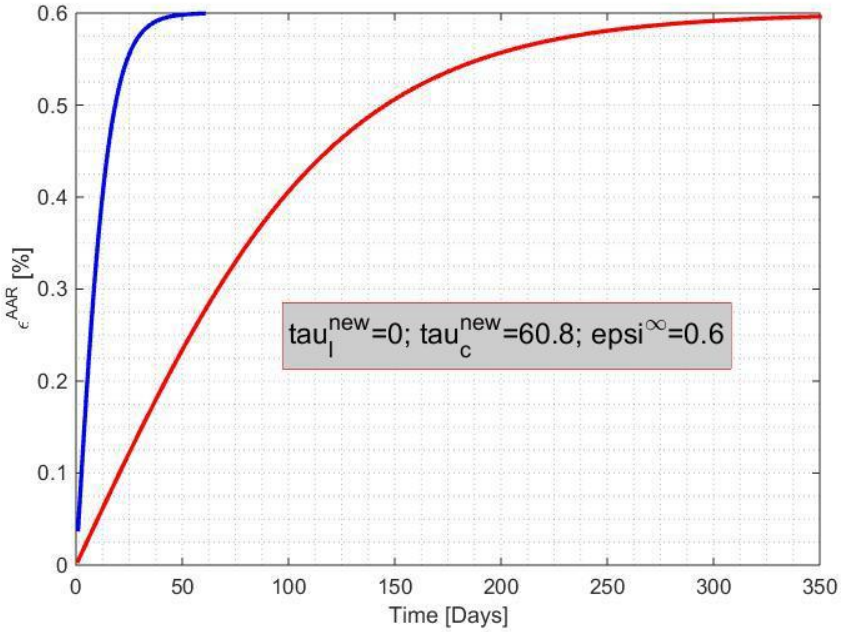

Figure 110 - Expansion vs time for 80C (blue line) and 38C (red line)

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CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORKS

7.1 Conclusions

This thesis describes the reactivity of aggregates sourced from quarries along the Gunnison River in western Colorado and supplied by Whitewater Building Materials in Grand Junction, Colorado. Sand from this source was found to produce expansion of 0.68% while crushed 3/4" gravel produced expansion of 1.02% measured sixteen days after casting by ASTM 1567. Mortar bar expansions continued for approximately 150 days, reaching maximum elongations of 1.22% and 1.57%, respectively.

A test program suitable for characterizing the expansion potential of these aggregates and concrete produced from them has been outlined. Concrete bars 4"x4"x10" were produced and either immersed in 1M $NaOH_{(aq)}$ at 80°C or stored uncovered in a fog room at >90% relative humidity and 21˚C. Samples kept immersed in NaOH were observed to expand vigorously, while those kept in the fog room expanded much less. Control specimens produced with a 30% solution of $LiNO_3$ were found to exhibit negligible expansion

Five concrete mixes were devised and tested. Of these, mix 5 was found to have properties suitable for production of further experimental samples. Twenty-eight-day compressive strength is 5100 psi and slump is 6.5 inches. Expansion after 65 days is 0.59% for samples held immersed in 1M NaOH at 80°C and 0.033% at 65 days for those stored uncovered in a fog room. Fitting experimental data to Larive's model of ASR expansion reveals that concrete produced from mix 5 and held saturated under caustic wash will reach 0.5% expansion after roughly 145 days.

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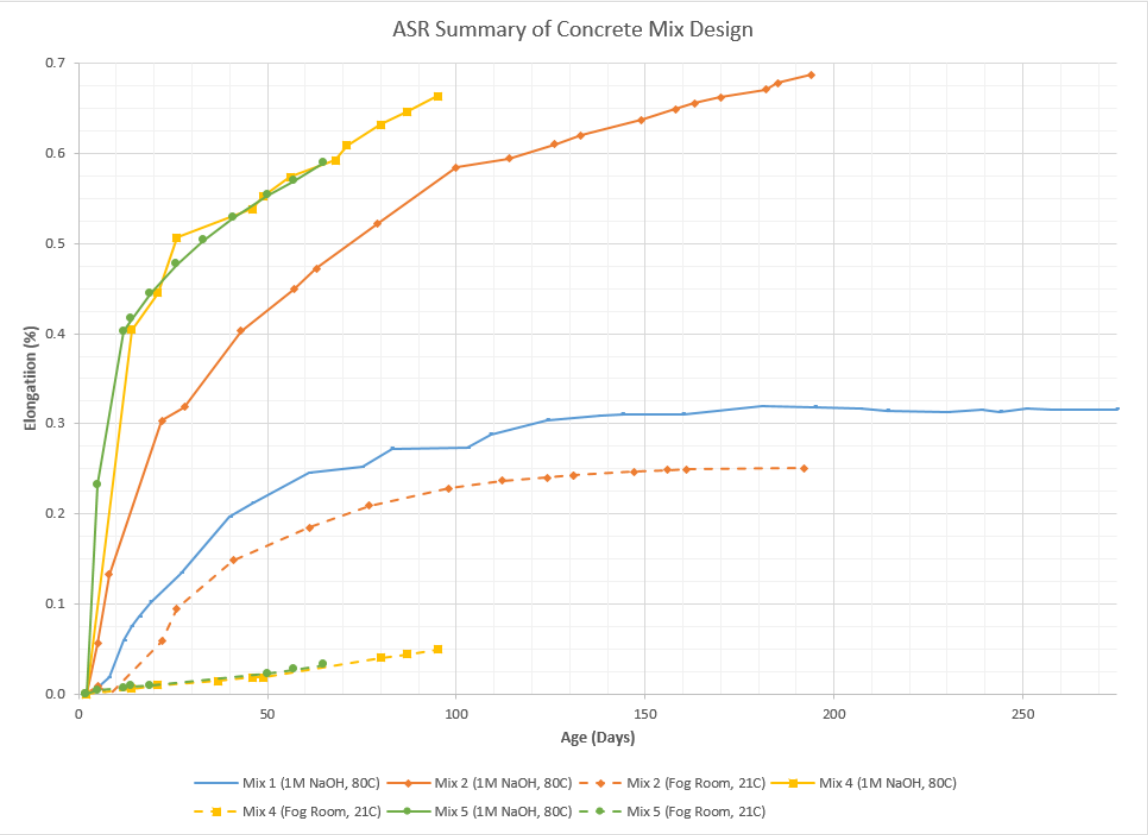

Figure 111 - Summary of concrete prism expansion testing

The results of concrete prism expansion testing are summarized in Figure 111. Solid lines indicate prisms stored immersed in 1M NaOH at 80°C while dashed lines indicate prisms stored uncovered in a fog room at 21°C and relative humidity over 90%. The color of each line indicates the concrete mix from which the prisms were cast. Note that data points corresponding to the period during which the fog room humidifiers were not operating properly are omitted in Figure 111.

The first mix design (blue line), generated with 0.53% alkali cement exhibited moderate expansion. Increasing alkalinity to 1.25% for mix 2R, increased both the rate and ultimate expansion, as seen in orange line. Further boosting alkalinity to 1.6% and increasing the proportion of sand (mix 4 and 5) resulted in still faster expansion (yellow and green solid lines), although the effect on ultimate expansion is not

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evident. It is also not clear the extent to which the expansion increase of mix 4 and mix 5 is due the increased alkalinity versus mix 2R or the increased sand content.

Samples kept in the fog room (dashed lines) exhibited significantly reduced expansion versus those kept in more aggressive conditions. However, it is especially enlightening to contrast the expansion of mix 2R (orange dashed line) to those of mixes 4 and 5 (yellow and green dashed lines). Recall that prisms of mix 2R were treated in a non-standard manner. These prisms were immersed in 1M NaOH for eight days after casting, after which time they were removed to the fog room. Prisms of mix 4 and 5 were never exposed to NaOH and were returned to the fog room immediately after demolding. The result of this discrepancy is that mix 2R prisms expanded significantly more than those of mix 4 and 5, despite having lower initial alkalinity and greater proportion of sand. Evidently, this initial treatment had a lasting effect on expansion potential. This observation underscores the importance of early treatment (called pre-treatment by Lindgård).

7.2 Recommendation for future work

Samples should be cast as prescribed in Section 3.5 and stored as recommended in Section 3.7. It is recommended to take expansion measurements twice weekly for the first four weeks of storage, as the majority of elongation was observed to take place in the first month. Subsequent measurement should be taken weekly for the next six months or until elongation is observed to plateau.

At the termination of the ASR development period, shear samples should be mounted to the biaxial shear test apparatus installed on the 'million-pound' press and tested to failure. Concurrent with shear sample testing, it would be advisable to take select two of the 4"x8" cylinders to test in compression. This provides a measure of compressive strength degradation for comparison to shear strength degradation,

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which may allow for characterization of prototype shear strength based on compressive testing of core samples.

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APPENDIX A

PETROGRAPHIC STUDY

The following report details the results of a petrographic study kindly conducted by Bent Grelk, Kurt Kielsgaard, and Antonio Barbosa of the Technical University of Denmark. Specimens provided for petrographic analysis include mortar bars made from Whitewater sand and crushed 3/4" gravel per the procedure outlined in Section 4.2 and a concrete prism produced from mix 2R per the procedure outlined in Section 5.5.2 and stored in 1M NaOH at 80°C.

Transmittal letter

To Professor Victor E. Saouma (University of Colorado Boulder)

Re. Petrographic analysis of potential ASR reactive concrete prism and mortar bars

From Chief Consultant Bent Grelk (Grelk Consult and Technical University of Denmark) December 29th , 2015

Associate Professor Kurt Kielsgaard Hansen (Technical University of Denmark)

PhD-student Ricardo Antonio Barbosa (Technical University of Denmark)

Introduction

The purpose of the petrographic analysis is to:

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- verify whether the longitudinal expansion measured on the received concrete prism and mortar bars are caused by alkali-silica reaction (ASR)
- determine which rock types that may be reacting in the concrete sample and in the mortar bars

The petrographic analysis is conducted on thin sections prepared from one of concrete prism and from two of the mortar bars. The authors have been informed by our colleagues at the University of Colorado Boulder that one of the mortar bars was cast with potentially ASR reactive fine aggregate (sand) and the second mortar bar was cast with potentially crushed ASR reactive coarse aggregate.

By the naked eye, both mortar-bars had visually more cracks than the concrete prism. To the authors knowledge all the received samples have been exposed to a NaOH solution at 80 degree Celsius in accordance with ASTM C1260 "Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method)".

The authors are not aware of the exposure time of the samples in the NaOH solution. Additionally, the authors are not aware of the concrete mix used for the samples and the rock types used in the concrete mix.

Preparation of thin sections

The thin sections were prepared from a slice of the concrete prism and a slice of each of the mortar bars.

The thin sections are made by 1) vacuum impregnation of the slices cut from the samples with an epoxy resin containing a fluorescent dye, and 2) the impregnated slices are mounted on glass plates and grinded and polished to a thickness of 0.02 mm. The thin sections are examined in a polarizing optical microscope using plane polarized light, crossed polarized light and blue transmitted light with a yellow blocking filter (fluorescent mode). The vacuum impregnation of the samples with fluorescent dye causes all voids and cavities to be filled with fluorescent epoxy. By transmitting blue light trough the thin section in the

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microscope, the fluorescent epoxy in the various porosities will emit a yellow light that makes voids, cavities and cracks easy to identify.

The thin sections are examined using a polarization microscope in accordance with the Danish test method "TI-B 5 (87) Structure analysis of hardened concrete" and ASTM C856-04 "Standard Practice for Petrographic Examination of Hardened Concrete".

Results of the petrographic analysis

The petrographic analysis verifies that the measured longitudinal expansion in the concrete prism and in the mortar bars is caused by ASR. In all samples there is on-going harmful ASR. The harmful reaction is linked to a reactive mix of porous and semi porous flint like grains, metamorphic, sedimentary and magmatic rock types. The metamorphic and sedimentary rock types consist typically of areas with reactive microcrystalline quartz. Since many different rock types are reacting in the concrete prism and in the mortar bars, the reactive rock types will not be distinguished in this report.

The ASR reactions are observed in both the fine aggregate fraction and in the coarse aggregate fraction. For the coarse aggregate fraction, reactions are mainly seen in the mortar-bar where the coarse aggregate fraction has been crushed. In thin sections prepared from the concrete prism only few reactions in the coarse aggregate have been observed. In our opinion there may be a potential for further expansion in the concrete prism, since mostly the fine aggregate fraction is reacting. However, by the petrographic analysis it is not possible to give a quantitative evaluation of the rate and extend of the reaction – only a rough qualitative evaluation.

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Photographic documentation

The following photographic documentation shows a representative selection of the reactive rock types in the samples. The photographic documentation shows different harmful reactive rock types including crack formation and ASR gel formation. The presented photos are taken in different light configurations which give the reader a better opportunity to identify the reactive rock typesGenerally, on the following fluorescent light photos the on-going ASR reactive rock types are marked with a red circle. The ASR induced cracks in the cement paste are locally marked with an arrow.

Final remark

The petrographic analysis is a powerful tool to reveal on-going harmful ASR.

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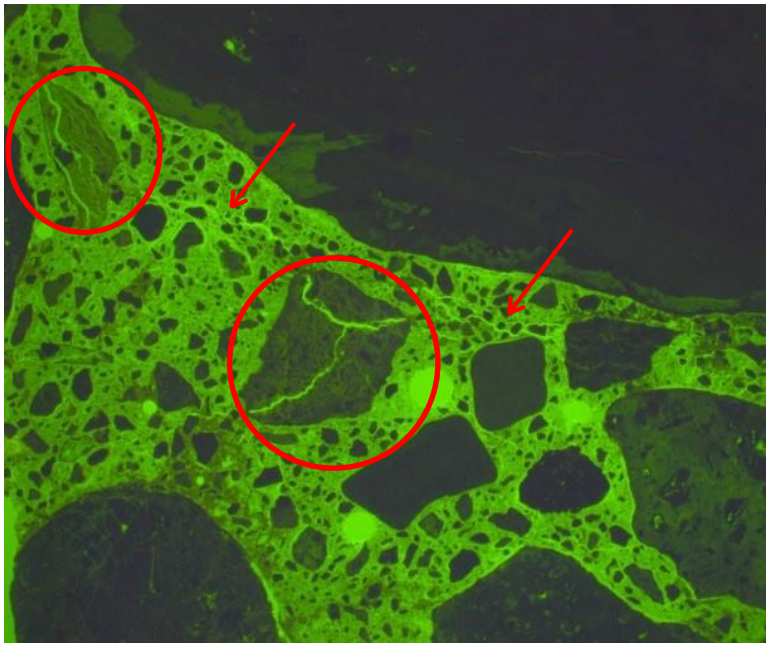

Figure 1: Concrete sample. Fluorescent light. Magnification: x12.5.

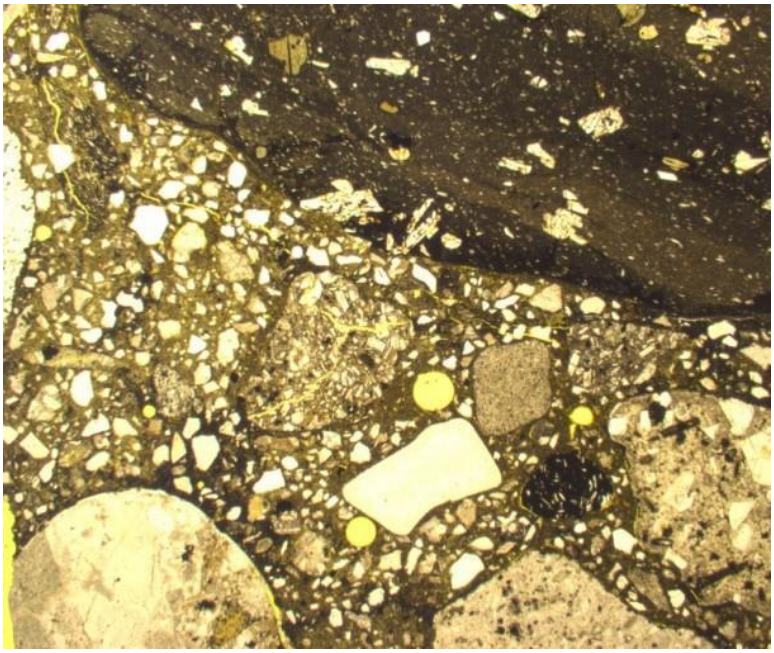

Figure 2: Concrete sample – the same area as in Figure 1. Plane polarized light. Magnification x12.5.

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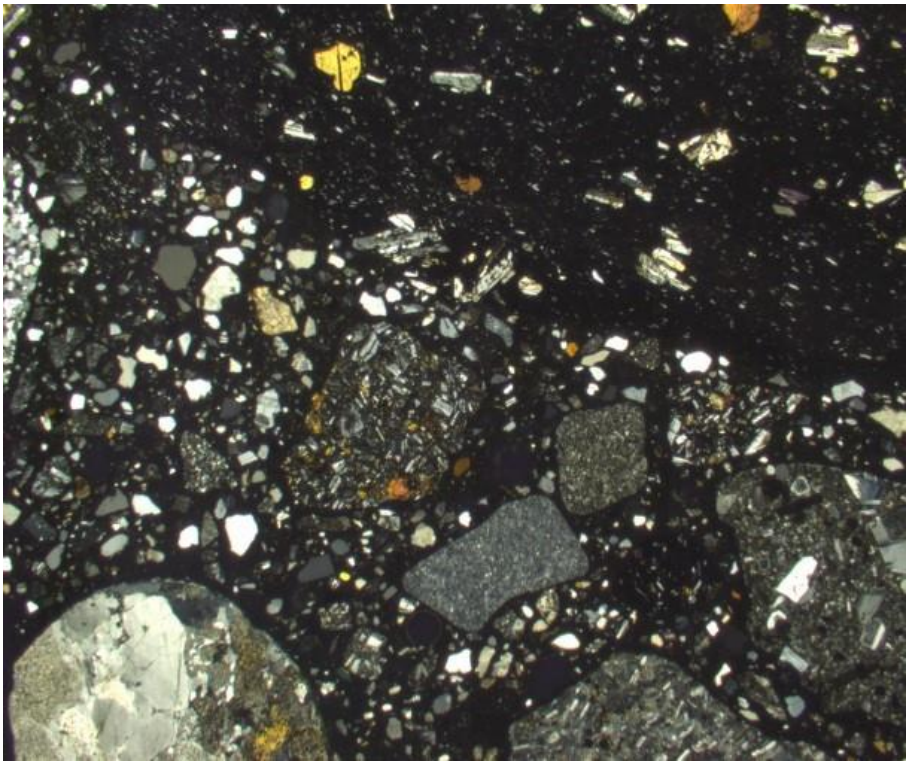

Figure 3: Concrete sample – the same area as in Figure 1 and 2. Cross polarized light. Magnification: x12.5

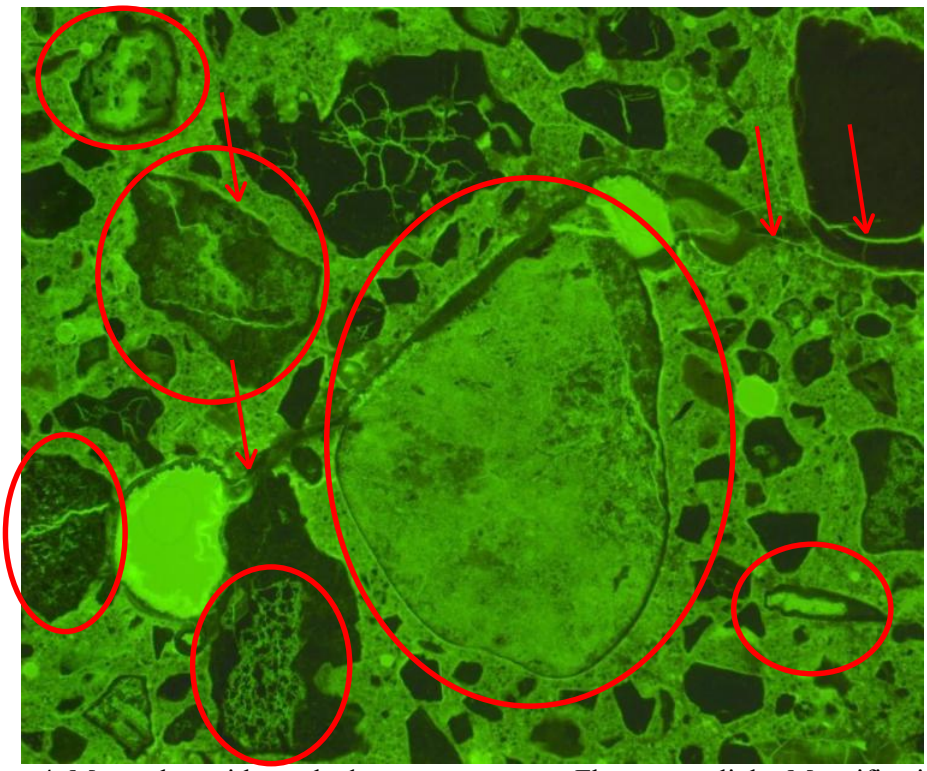

Figure 4: Mortar-bar with crushed coarse aggregate: Fluorescent light. Magnification: x25..

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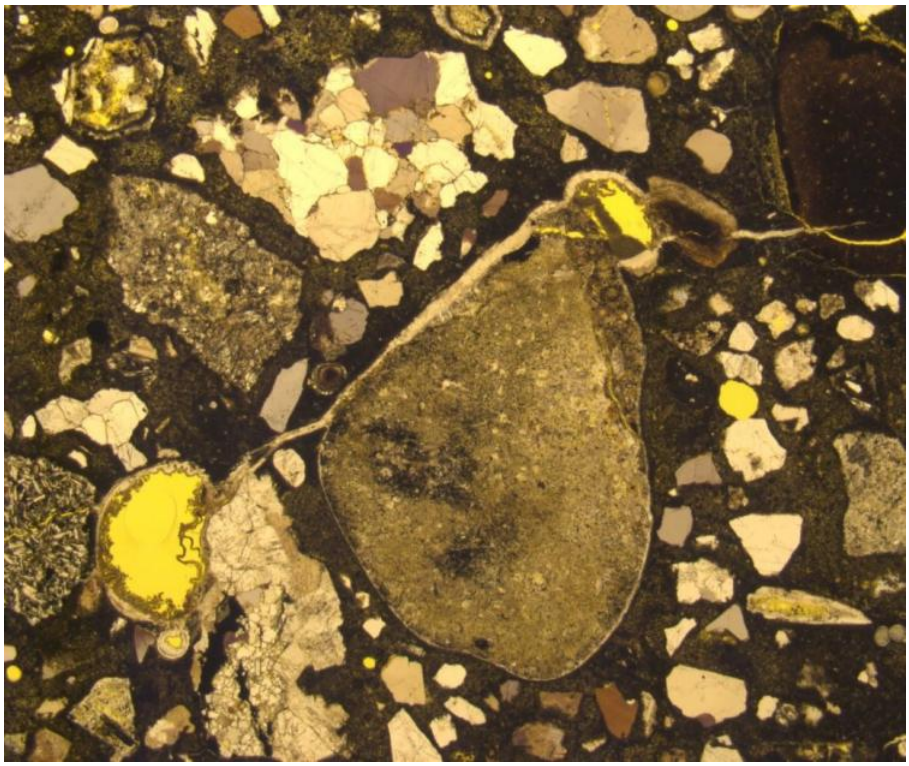

Figure 5: Mortar-bar with crushed coarse aggregate – the same area as in Figure 4. Plane polarized light.

Magnification: x25.

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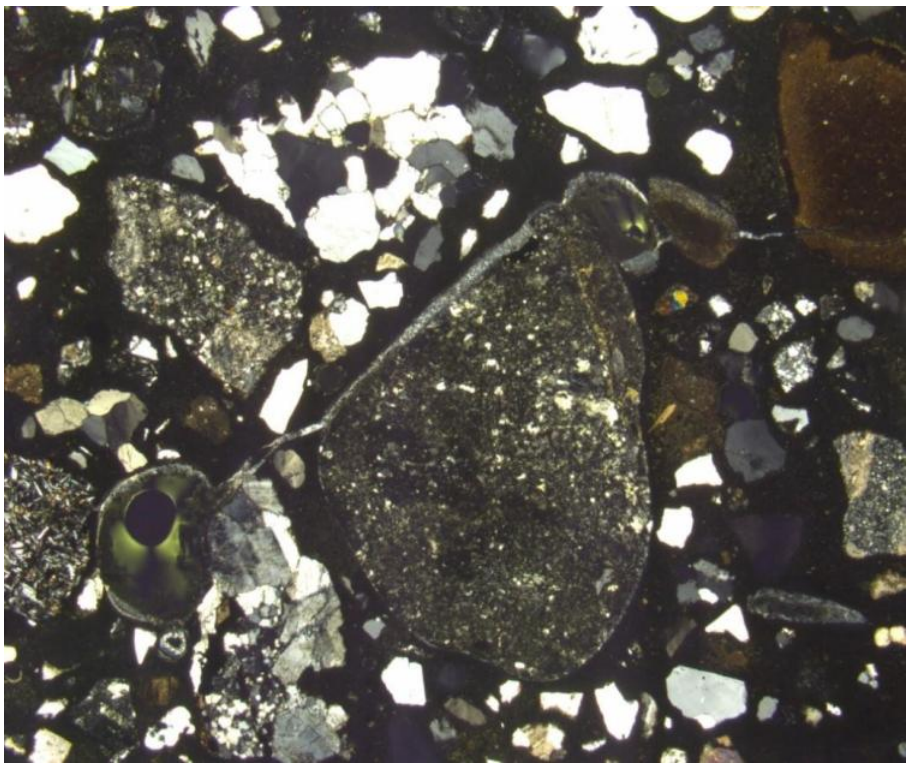

Figure 6: Mortar-bar with crushed coarse aggregate – same area as in Figure 4 and 5. Cross polarized light.

Magnification: x25.

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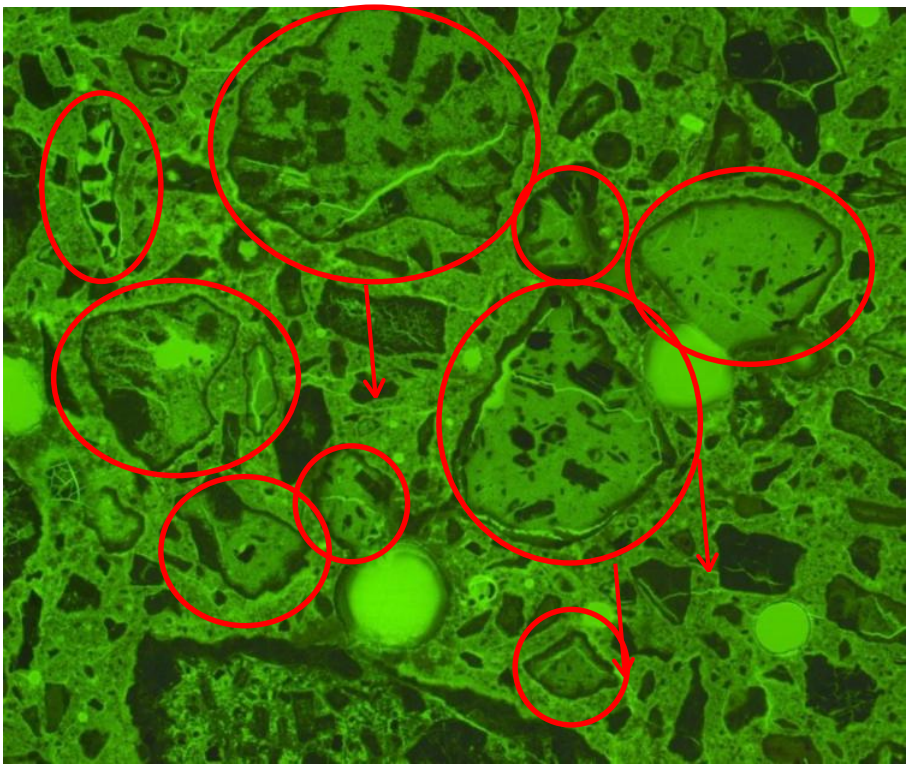

Figure 7: Mortar-bar with fine aggregate. Fluorescent light. Magnification: x25.

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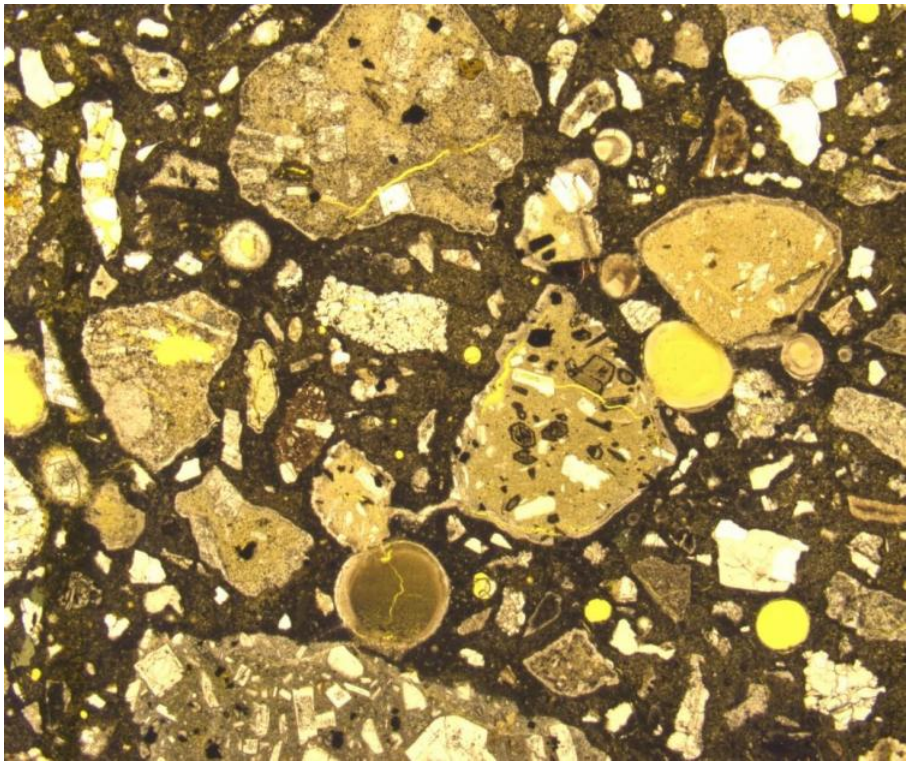

Figure 8: Mortar-bar with fine aggregate – the same area as in Figure 7. Plane polarized light. Magnification: x25.

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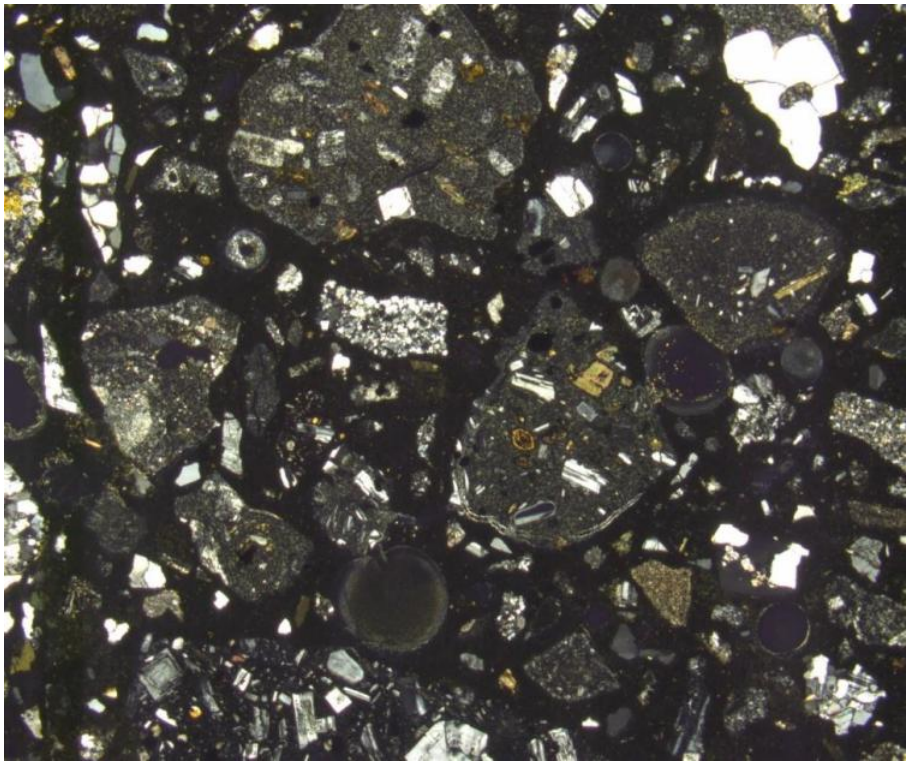

Figure 9: Mortar-bar with fine aggregate – the same area as in Figure 7 and 8. Cross polarized light. Magnification: x25.

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APPENDIX B

FORENSIC ASR TESTING SURVEY

The following table summarizes the result of a survey of several proposed forensic tests for ASR reviewed as part of this study. The reviewed test procedures proposed methods of ascertaining the extent to which ASR has progressed in a core sample obtained from a field structure. Rather than summarize each in turn, the key points of each proposed method are presented in tabular form for easier comparison. The reader is cautioned that this summary is based on preliminary, draft recommendations. Special thanks to the communicators of these test methods: Alain Sellier & Stéphan Multon, R. P. Martin, M. A. Bérubé, Andreas Leeman & Christine Mertz, Cyrille Dunant, and Jonathan G. M. Wood.

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Method Name	1 - Toulouse / Sellier	2 - LCPC	3 - Canada - Berube
Summary	Measures length change of mortar bars made from recovered gravel and sand.	Measures length and weight change of core cylinders, includes final drying phase.	Measures expansion and weight change of core cylinders with emphasis on controlling for non-ASR effects.
Sample Type	Mortar bars made from recycled aggregates	100mm diameter x 250mm cylindrical core	4" or 6" diameter cylinderical core
Time to Results	~200 days	~ 1 year	>1 year
Results Obtained	Residual expansion	Swelling potential and residual free expansion	Swelling poential and residual free expansion
Provides estimate of expansion rate?	Yes, but calculation incomplete	No	No
Provides estimate of historical expansion?	Yes, but calculation incomplete	No (though commentary suggests method)	No
Provides estimate of time to ultimate expansion?	Yes, but calculation incomplete	No	No
Provides estimate of magnitude of ultimate expansion?	Yes, but calculation incomplete	Broad classification only	Broad classification only
Variables Measured	Length	Length	Length, diameter, weight, temperature
Weight Measured?	No	No	Yes
How is weight data used?	N/A	N/A	-Determine end of preconditioning phase -Obtain corrected axial expansion

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Method Name	5 - Swiss-Leeman/Metz	6 - Swiss-Dunant	7 - UK-Wood
Summary	Measures expansion and weight change of core cylinders with emphasis on comparison of relative rates of expansion throughout test.	Measures length change of core cylinder with emphasis on comparison of elongation with results of electron microscopy.	Measures expansion and weights of core cylinders with emphasis on replicating field conditions.
Sample Type	Cylindrical core	Cylindrical core	70mm diameter X 200mm cylindrical core
Time to Results	~ 1 year	?	> 1 year
Results Obtained	Residual expansion	Residual expansion	Potential for further expansion
Provides estimate of expansion rate?	No	No	Unclear. No data interpretation provided.
Provides estimate of historical expansion?	Broad classification only	No	Unclear. No data interpretation provided.
Provides estimate of time to ultimate expansion?	No	No	Unclear. No data interpretation provided.
Provides estimate of magnitude of ultimate expansion?	Broad classification only	Yes, assumed equal to core expansion in lab	Probably. No data interpretation provided.
Variables Measured	Length, diameter, weight	Length	Length, weights of sample, free water, steel plate.
Weight Measured?	Yes	No	Yes
How is weight data used?	Determine end of preconditioning phase	N/A	Unclear. No calculations provided

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Method Name	1 - Toulouse / Sellier	2 - LCPC	3 - Canada - Berube
Sample Handling	Not relevant - samples decomposed to recover aggregates	After coring, cylinders washed in clean water, wiped, and stored in sealed plastic bags.	Cores should be taken 1 - 3 inches from exposed surface. Skin effects include high microcracking, low alkali due to leaching, or high alkali due to evaporative concentration.
		Drilling water introduces water which does not affect final expansion if test is begun quickly. Mass however, is increased.	After coring, cores should be sealed immediately by wrapping in heavy duty shrink wrap and storing in sealed polyethylene bags
		Care taken to avoid drying our sample before testing begins.	
Necessary to extract cores unaffected by ASR?	Yes	No	No
Special shipping instructions?	No mention	No mention	No mention
How important is sample orientation?	No mention	Expansion dependent on coring direction. Measuring both axial and transverse expansion sufficient to control.	Expansion dependent on directions of restraint. Note core direction and measure both axes.
Sample Conditioning	28 Days at 20C in sealed bags; 60 C and 95% RH for remainder	38C and 100% RH. Samples placed in containers on grills above water. Containers placed in reactor on larger grill above heated water. Drying phase: oven at 105 C	38C and >95% RH
Immersion?	No	No	No

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Method Name	5 - Swiss-Leeman/Metz	6 - Swiss-Dunant	7 - UK-Wood
Sample Handling	Samples should be immediately sealed in plastic bags after coring.	Samples should be wrapped in plastic, then sealed in aluminum foil after coring.	Samples should be rinsed, surface dried, and allowed to dry in shade for 1 hour before being wrapped plastic.
	Glueing pins onto cut samples may require a bit of drying be permitted.	Gluing pins onto cut samples with non-reactive mortar may require a bit of drying be permitted.	Dry-coring should be avoided as it heats and dries samples.
Necessary to extract cores unaffected by ASR?	No	No	No
Special shipping instructions?	No mention	No mention	No mention
How important is sample orientation?	No mention	No mention	Mentions high variability in expansion from cores in same structure.
Sample Conditioning	Conditioning Phase: 2 weeks at 20C in bucket with sample end in tap water Nonlinear phase : 30-60 days at 38C and 100% RH, sample wrapped in plastic film Linear phase: 9-12 months under same conditions as nonlinear Drying phase: Ambient temperature and 60-70% RH until initial weight achieved	38C and immersed in NaOH Concentration of NaOH should match concrete pore solution if possible If pore solution alkalinity unknown, use .320 m/L NaOH. Mention made of additional tests at 20C and 50C, but not discussed.	Ambient temperature (20C) with sample end in water Stored at 30 deg angle in sealed container
Immersion?	End Only	Yes	End Only

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Method Name	1 - Toulouse / Sellier	2 - LCPC	3 - Canada - Berube
Comments on Initial Expansion Phase	No commentary	Rapid initial expansion is due to water uptake in the core. Linked to capillary features (porosity, microcracking), and hydrophillic products (reaction gels).	Short Term variations are caused by: -Thermal expansion or contraction -Expansion due to release of restraining stress -Expansion due to moisture uptake -Free expansion of existing ASR gel due to stress relief -Expansion of existing ASR gel due to water sorption.
		Magnitude of initial expansion depends on moisture state, stress history, ASR history. Interpretation is difficult and omitted in LCPC.	Preconditioning lasts days or months. End of preconditioning characterized by points of inflection in mass and elongation curves.
		Rapid expansion phase assumed to be **8 weeks.**	Recommended to wait at least **one week** before taking zero measurements.
		Multon (2008) - believes that omitting first 8 weeks of data results in underestimate of expansion potential. He recommends including first 8 weeks data and correcting for temp and water uptake.	

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Method Name	5 - Swiss-Leeman/Metz	6 - Swiss-Dunant	7 - UK-Wood
Comments on Initial Expansion Phase	Phase 1, Conditioning, is performed at 20C with samples absorbing water by capillary action. Conditioning ends when constant mass reached. Usually **1-2 weeks**.	No commentary	Early part of test characterized by shrinkage recovery and water uptake. Shrinkage recovery swelling is reasonably uniform and stabilizes after a month. Compare to AAR gauge length expansions which are highly variable.
	The purpose of the conditioning phase is to ensure all samples have same initial conditions when Phase 2 starts.		Comparison of elongation to weight change can allow for compensation of
	During Phase 2, Nonlinear expansion, samples are wrapped in plastic film and stored above water in containers housed in larger reactor.		Notes that water absorbed during coring and rinsing may initiate expansion before Demec points fitted.

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Method Name	1 - Toulouse / Sellier	2 - LCPC	3 - Canada - Berube
Necessary to extract aggregates?	Yes	No	No
Role of Petrography	none	none	Observation of ASR gel assists in interpretation of expansion results.
Role of Microscopy	none	none	none
Questions	How does mortar bar expansion relate to concrete expansion?	Results of drying phase imply history of expansion. Does a quantitative relationship exist?	Pre-existing ASR gels expand during preconditioning phase due to moisture uptake. Any possibility of quantifying historic ASR by this measure?
Is a finite element calibration required?	Mentioned, not explained	No mention	No mention

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Method Name	5 - Swiss-Leeman/Metz	6 - Swiss-Dunant	7 - UK-Wood
Necessary to extract aggregates?	No	No	No
Role of Petrography	Mentioned in papers, but not test procedure	none	Recommended for verification of UK Chert All cylindrical ends should be retained after sawing for petrographic analysis
Role of Microscopy	none	Electron microscopy performed before and after test.	none
Questions	Can petrographic results be used to quantify past expansion?	What does degradation imply?	Expansion variability important for predicting damage. How does laboratory expansion variability relate to field expansion variability?
		Can electron microscopy be used to quantify past expansion?	Can meaningful data be obtained after only 1 year at 20C?
		Activation energy said to be obtained by additional testing at 20C and 50C. How?	
Is a finite element calibration required?	No mention	No mention	No mention

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