UF Herbert Wertheim College of Engineering
UNIVERSITY of FLORIDA

Development of an Accelerated Test Method for Determination of Delayed Ettringite Formation (DEF)

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Background on DEF

What <u>general</u> conditions are needed for DEF to occur?

- Concrete must experience exposure to high temperatures during curing period
  - Greater than > 70ºC
- Concrete must have a high sulfate to aluminate ratio
- Concrete must have exposure to moisture
  - *All concrete*

K. Folliard – Course CE 397 (2008)

Background on DEF

Recommendations to avoid DEF from ACI 201.2 **R16** Table 6.2.2.2

Max. Concrete Temperature, °F	Prevention Required
T ≤ 158°F (70°C)	No prevention required
158°F < T ≤ 185°F (70°C< T ≤ 85°C)	One of the following approaches: 1. Type II or Type V low-alkali portland cement having a Blaine fineness of 430 m²/kg or less. 2. Portland cement with 1-day ASTM C109 mortar strength of 2,850 psi or less. 3. Any ASTM C150 portland cement in combination with the following proportions of pozzolan or slag cement: a. At least 25% Class F fly ash b. At least 35% Class C fly ash c. At least 35% slag cement d. At least 5% silica fume with at least 25% slag cement e. At least 5% silica fume with at least 20% Class F fly ash f. At least 10% metakaolin
T > 185°F (85°C)	The internal concrete temperature should not exceed 185°F (85°C) under any circumstances.

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Revisiting ACI 201.2

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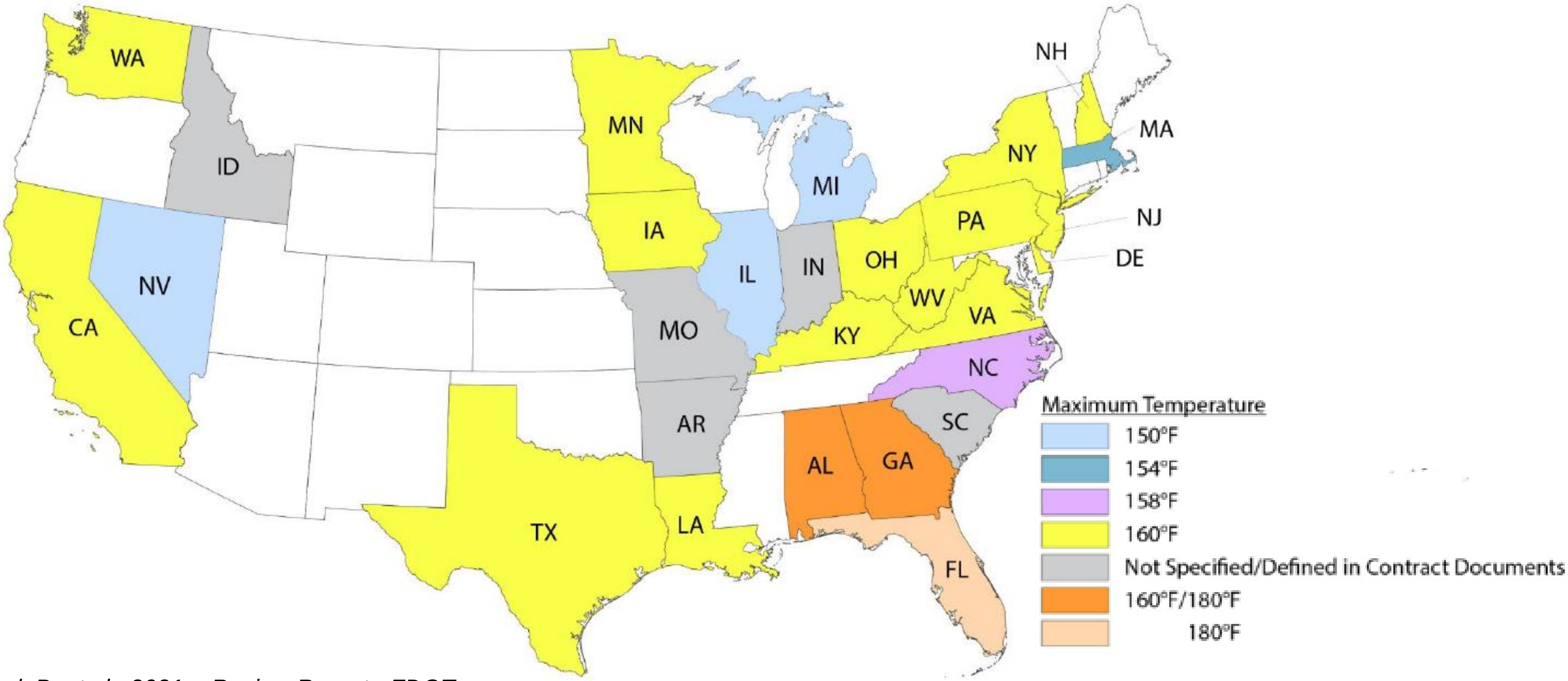
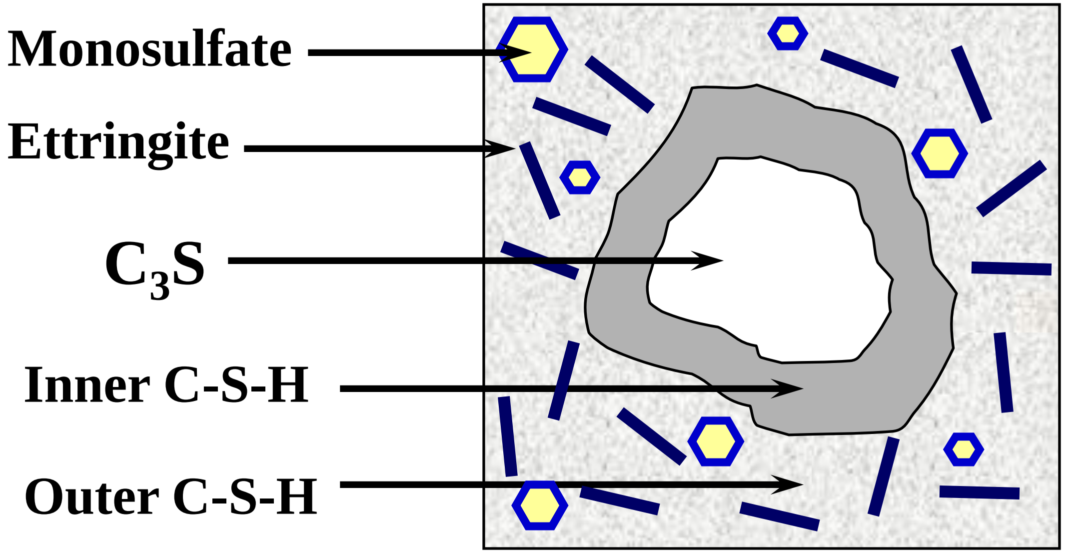
Mass Concrete Specifications



Clark B. et al., 2021 - Review Report - FDOT

Background on DEF - Folliard

Normal Concrete Cured low Temps

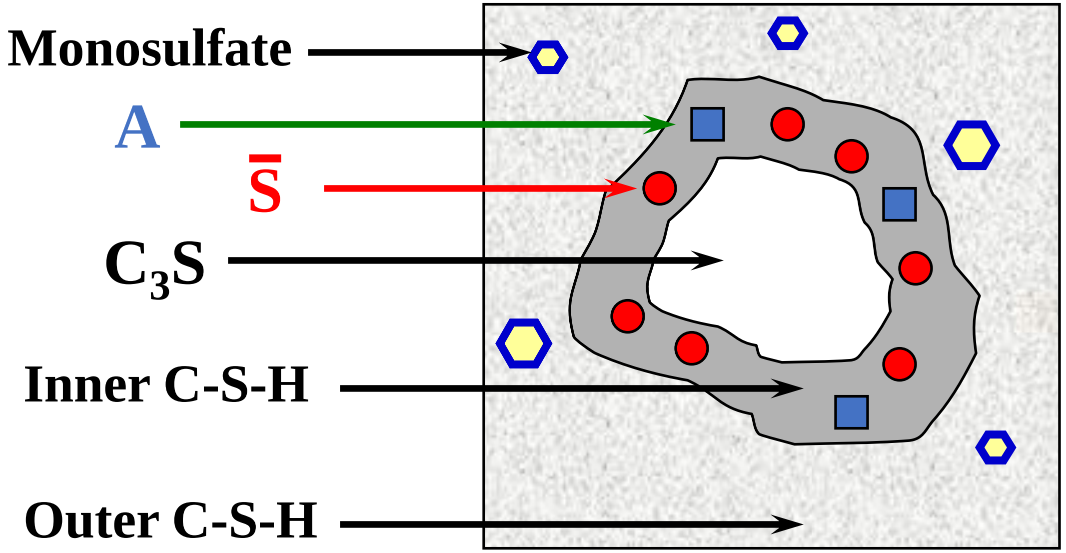


Ettringite and form formed as part of the outer hydration products

K. Folliard – Course CE 397 (2008)

Background on DEF - Folliard

Concrete Cured at Temps greater than 70^{o}C

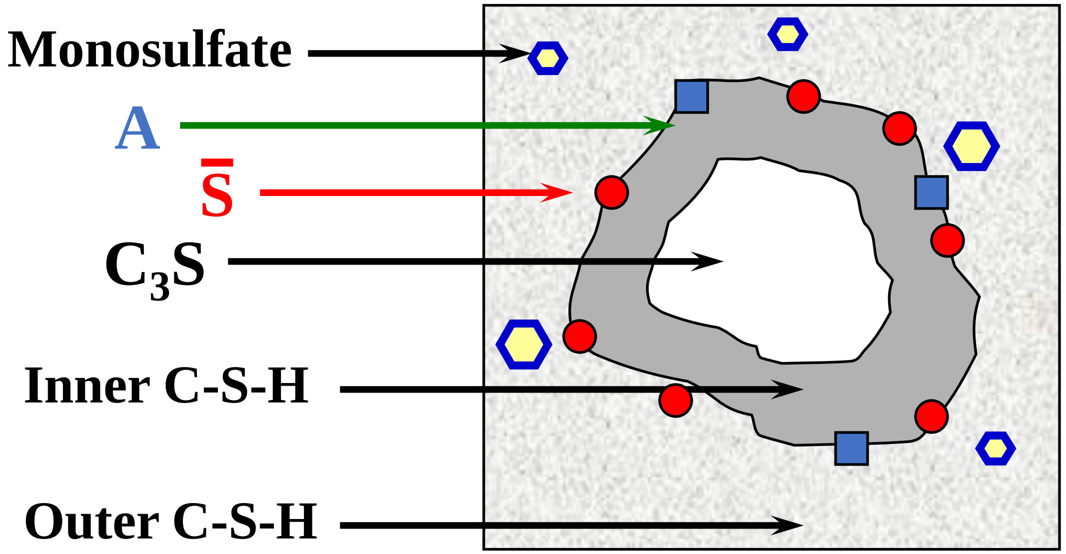


Dissolution of ettringite resulting in both sulfate and alumina being encapsulated in the rapidly forming inner C-S-H

K. Folliard – Course CE 397 (2008)

Background on DEF - Folliard

Subsequent storage in water at 20°C

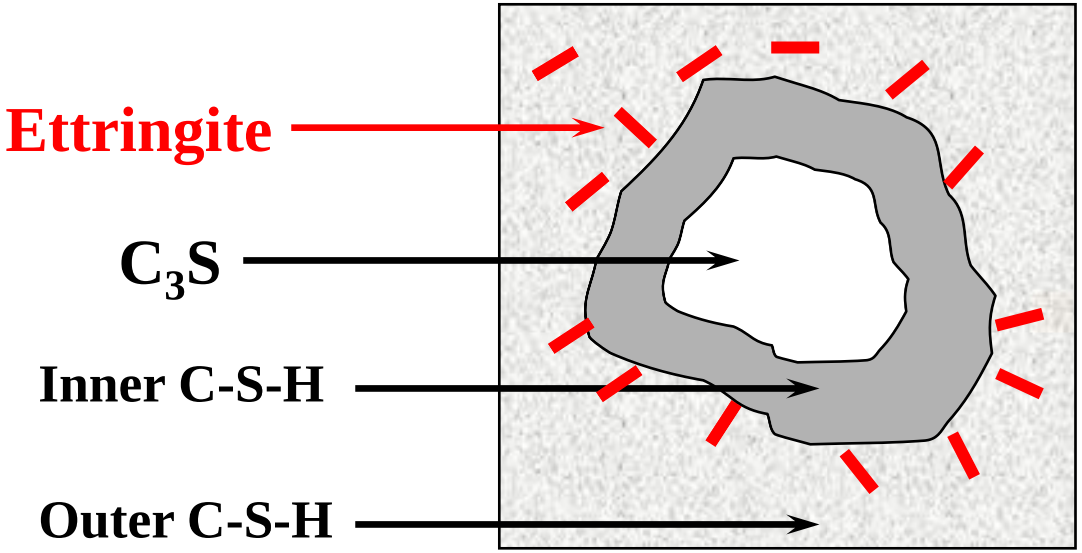


Sulfate and alumina slowly released from the inner C-S-H

K. Folliard – Course CE 397 (2008)

Background on DEF - Folliard

Storage in water at 20^{o}C – Long Term Curing

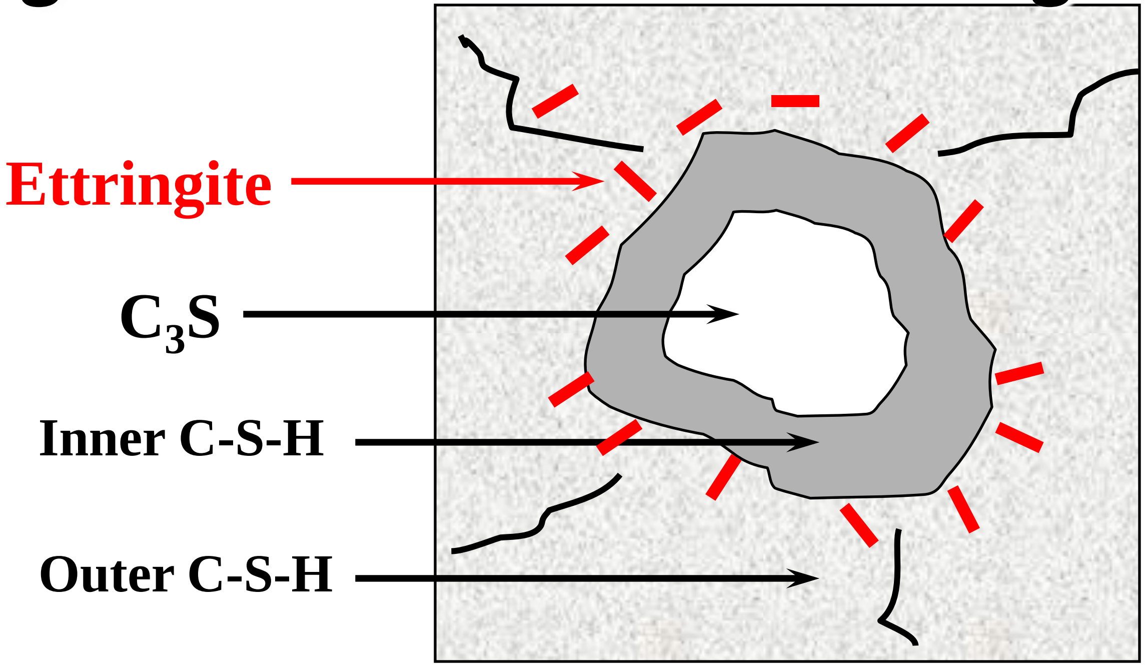


Ettringite formed in the fine pores of the outer C-S-H

K. Folliard – Course CE 397 (2008)

Background on DEF - Folliard

Storage in water at 20°C – Long Term Curing

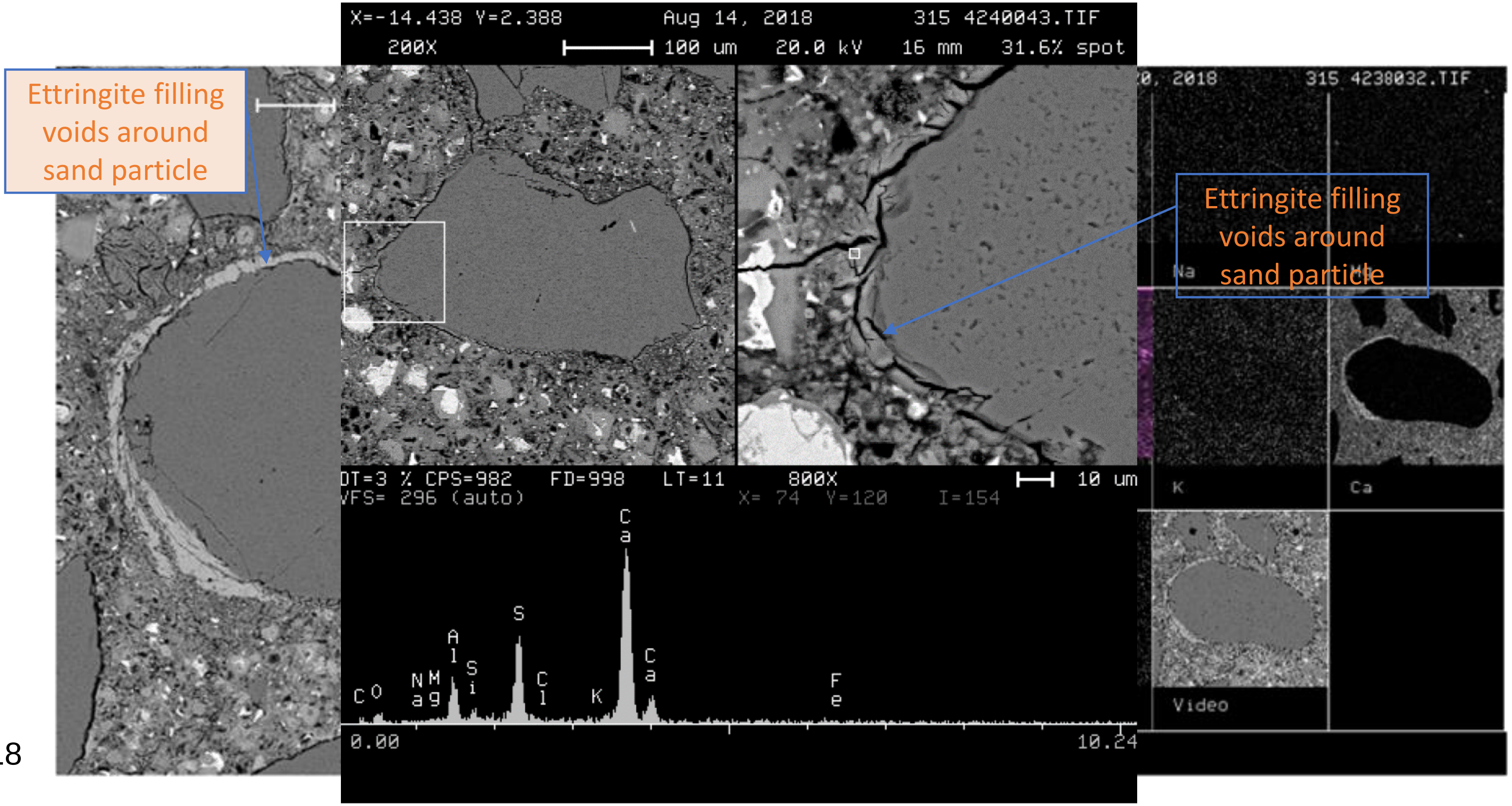


Under certain conditions this leads to expansion of the cement paste

K. Folliard – Course CE 397 (2008)

Background on DEF

Microcopy on Concrete with DEF



Ferraro et al., 2018

Predicting DEF

"Prediction is difficult, especially about the future"

- Niels Bohr (Jan Skalny)

Historical Work – Precast Industry

Skalny & Locher

Internal Sulfate attack and European Pre-cast Railroad Ties

Factors contributing to internal sulfate attack

- t_v – length of precuring time
- t_a – rate of temperature rise
- t_d – maximum curing temperature*

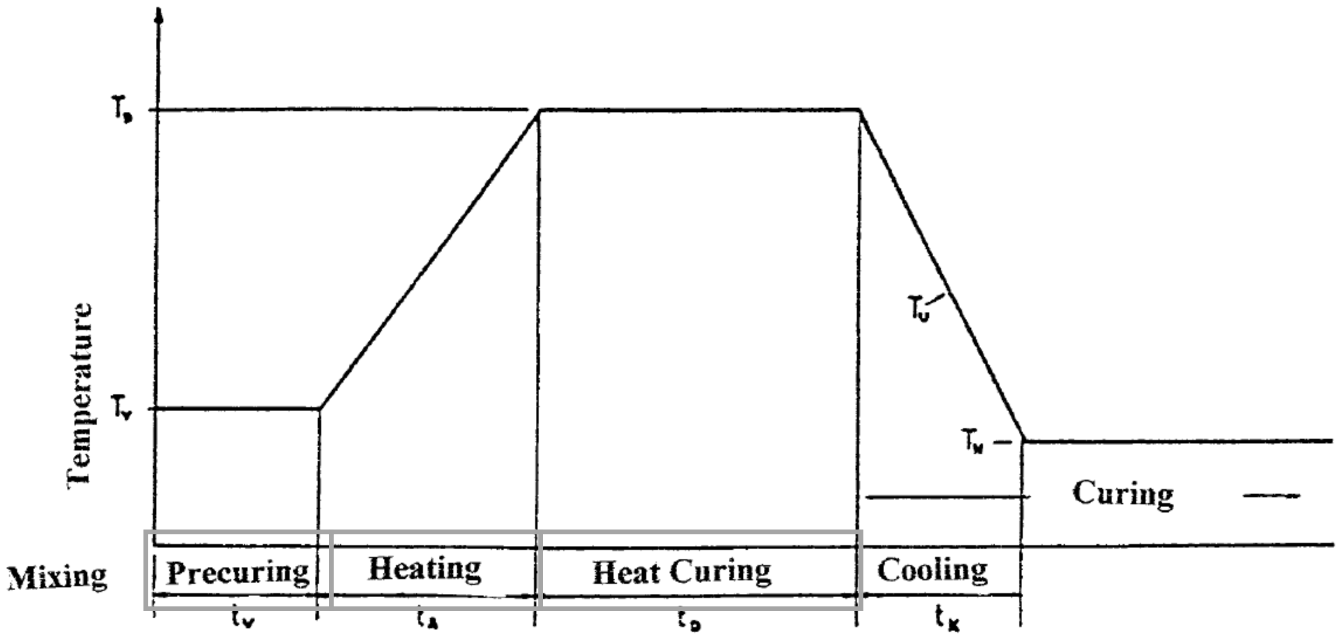


FIG 1—*Schematic Representation of the Heat Treatment Procedure.*

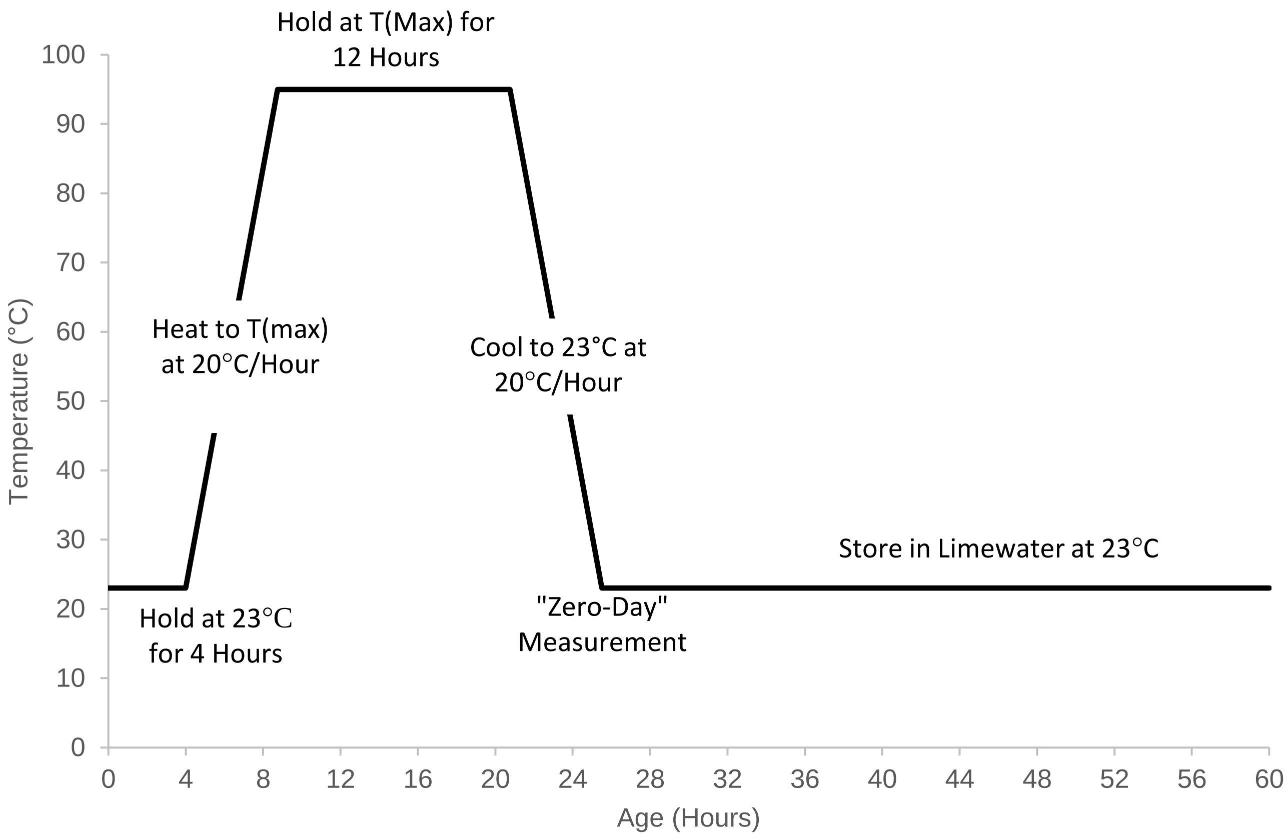
Historical Work – Precast Industry

Kelham’s work

“The Effect of Cement Composition and Fineness on Expansion Associated with Delayed Ettringite Formation”

- Developed in 1996 and based on cycles used in curing precast elements

- Maximum curing temperatures of
  70°C (158°F)
  80°C (176°F)
  90°C (194°F)
- Greatest expansions at 90°C



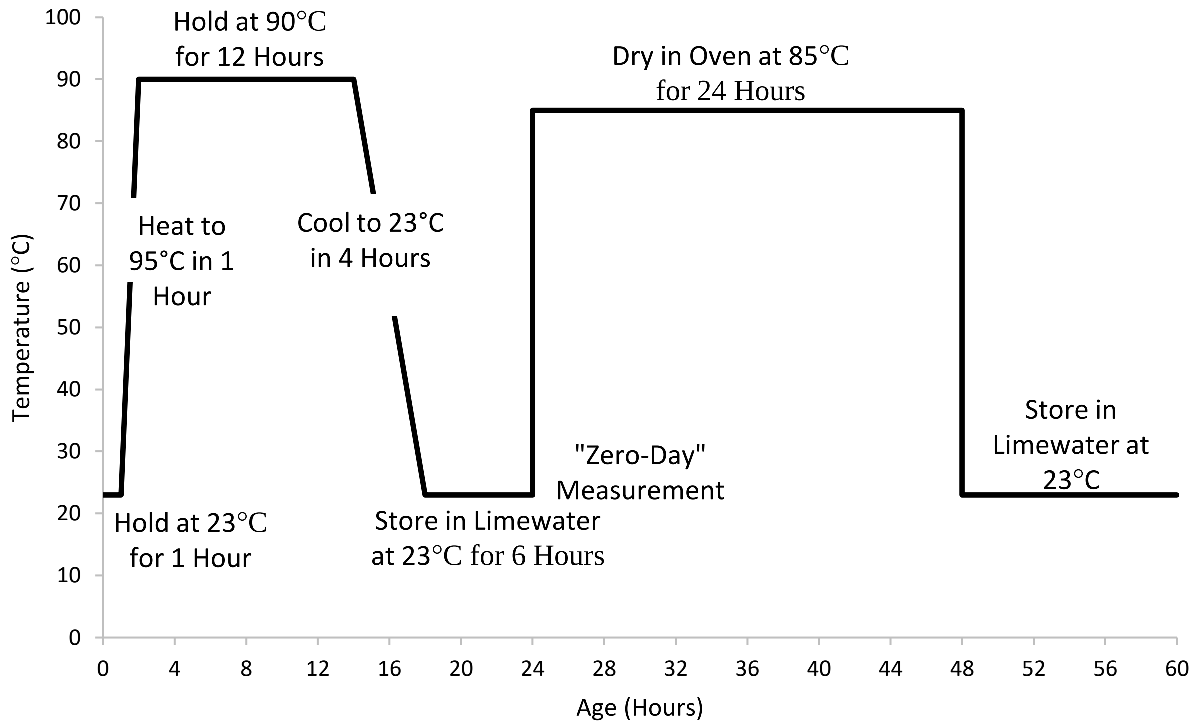
Kelham, S. (1996). *Cement and Concrete composites*, *18*(3), 171-179.

Historical Work – Precast Industry

Fu’s work

Delayed Ettringite Formation in Portland Cement Products

- Developed in 1996 and based on cycles used in curing precast elements
- Maximum curing temperatures of
    - 80°C (176°F)
    - 90°C (194°F)
- Dry oven portion to induce micro cracking to speed up the onset of DEF



Fu, Y. (1996). *Delayed ettringite formation in Portland cement products*. University of Ottawa (Canada).

Historical Work

Internal Sulfate Attack Publications between 1989 and 2012

The majority of published research sulfate to alumina ($\bar{S}$ / A) is important in DEF formation

Some work also looks at the Alkali content

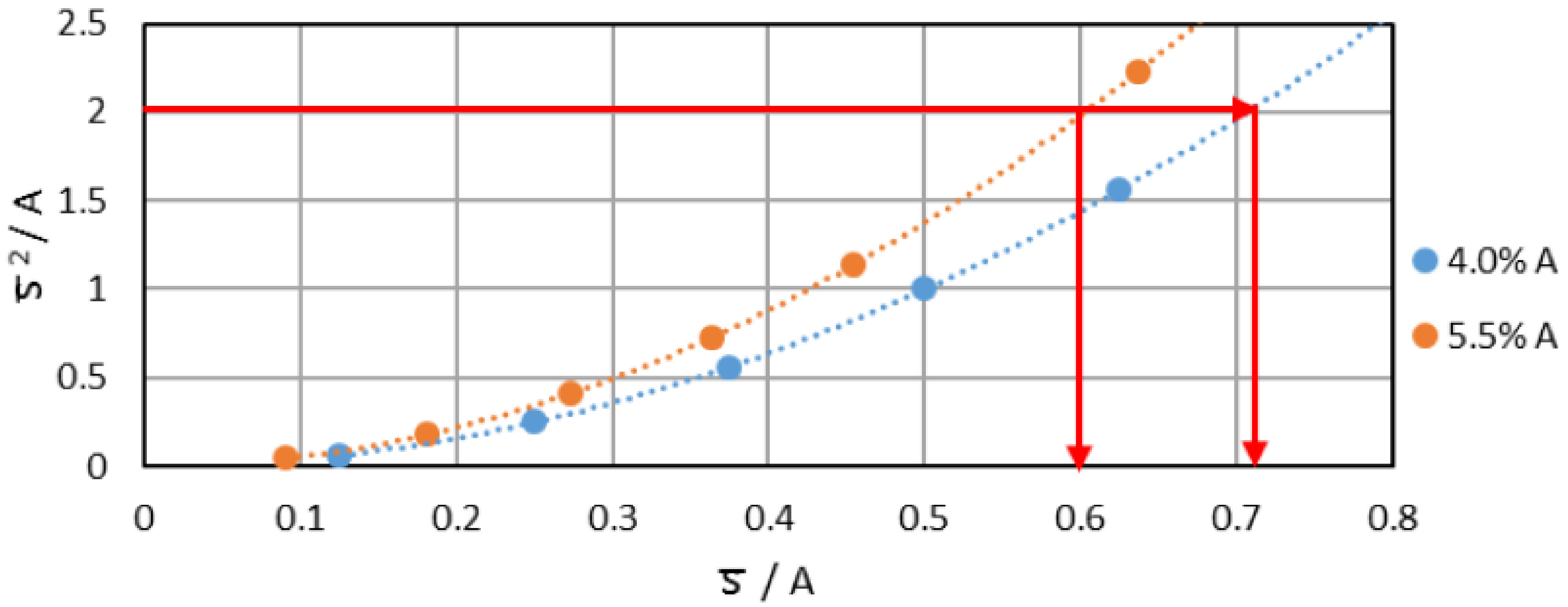
1989 - Heinz et al.	$(SO_3)^2 / Al_2O_3)_{wt.} < 2.0$
1992 - Day	$(SO_3/Al_2O_3)_{wt.} < 0.67$
1993 - Lawrence	$SO_3 < 3.6\%_{wt.}$ $MgO < 1.6\%_{wt.}$ $Na_2O_e < 0.8\%_{wt.}$
1995 - Lawrence	Expansion (@ 800 days) = $9.51 + 0.304 \times SO_3 + 0.00085 \times SSA + 1.728 \times Na_2O_{eq.} - 0.162 \times CaO - 0.040 \times C_3A$
1996 - Kelham	Expansion = $0.00474 \times SSA + 0.0768 \times MgO + 0.217 \times C_3A + 0.0942 \times C_3S + 1.267 \times Na_2O_{eq.} - 0.737 \times ABS(SO_3 - 3.7 -1.02 \times Na_2O_{eq.}) - 10.1$
1999 - Heinz	$SO_3 < 3.0\%_{wt.}$ $(SO_3 / Al_2O_3)_{wt.} < 0.45$ $Na_2O_e < 1.0\%_{wt.}$
2002 - Zhang et al.	$(SO_3 / Al_2O_3)_{mol.\ ratio.} < 0.8\%$ DEF Index [(SO3/Al2O3)m x [(SO3 + C3A)wt. / 10] x √(Na2Oeq)] < 1.1
2012 - Pavoine et al.	Expansion (@ 490 days) = 0.243 + 0.135s + 0.116a + 0.072b + 0.068t + 0.055sa + 0.031sb + 0.041st + 0.031ab 0.012at 0.015sat(%) Expansion (@ 1700days) = 0.314 + 0.144s + 0.127a + 0.099b + 0.050t + 0.052sa + 0.024sb + 0.034st + 0.021ab 0.030at 0.017sat (%)

Background on DEF

Day 1992 [17] **$\bar{S}$ / A** < 0.70

Heinz et al. 1999 [18] **$\bar{S}$ / A** < 0.45

Zhang et al. 2002 [19] **$\bar{S}$ / A** < 0.80% (molar ratio)



Historical Work – Mass Concrete "Field Study"

"The Green Book"

- International RILEM TC 186-ISA Workshop on Internal Sulfate Attack and Delayed Ettringite Formation (4–6 September 2002, Villars, Switzerland)

- K. Scrivener & J. P. Skalny

- One of the first studies on mass concrete in bridges
- Divet, L., and Pavione, A., ***Delayed Ettringite Formation in Massive Concrete Structure:***, an Account of Some Studies of Degraded Bridges,, 98-126.

"The initial cracking of the structure was attributed to a severe temperature differential between the outer concrete strata and the internal core. Upon the initiation of the initial cracking, it was determined that the presence of moisture and sulfate from the environment, rather than internal sulfate attack or DEF, contributed to years of deterioration"

Ferraro (2009). PhD Dissertation, University of Florida

Historical Work – Mass Concrete "Field Study" - USA

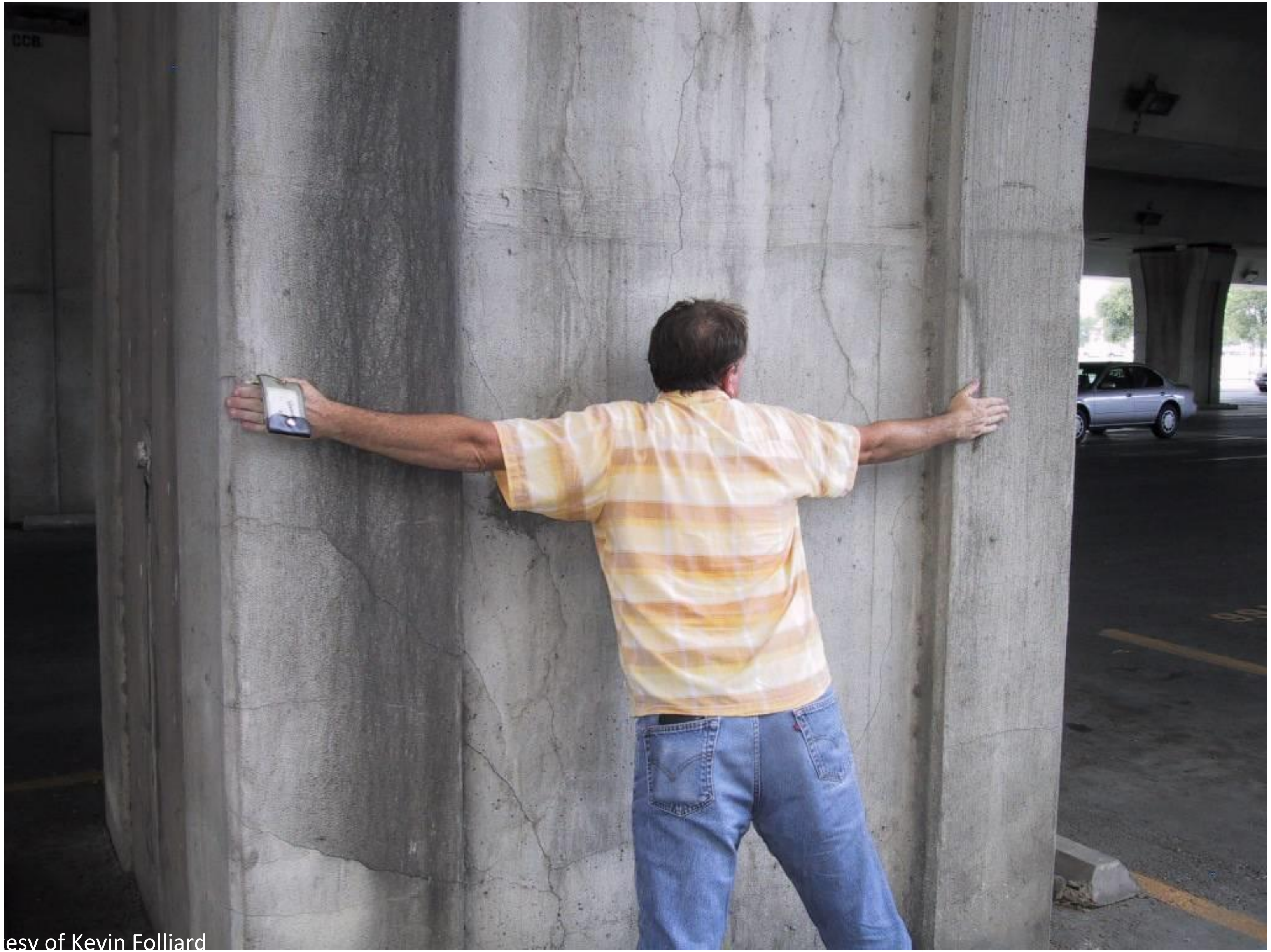


Photo from Riding and Folliard

Behavior of Mass Concrete

- Mass Concrete often stays as an elevated temperature for long periods of time
- Historical DEF testing does not consider this
- 12 hour soak times

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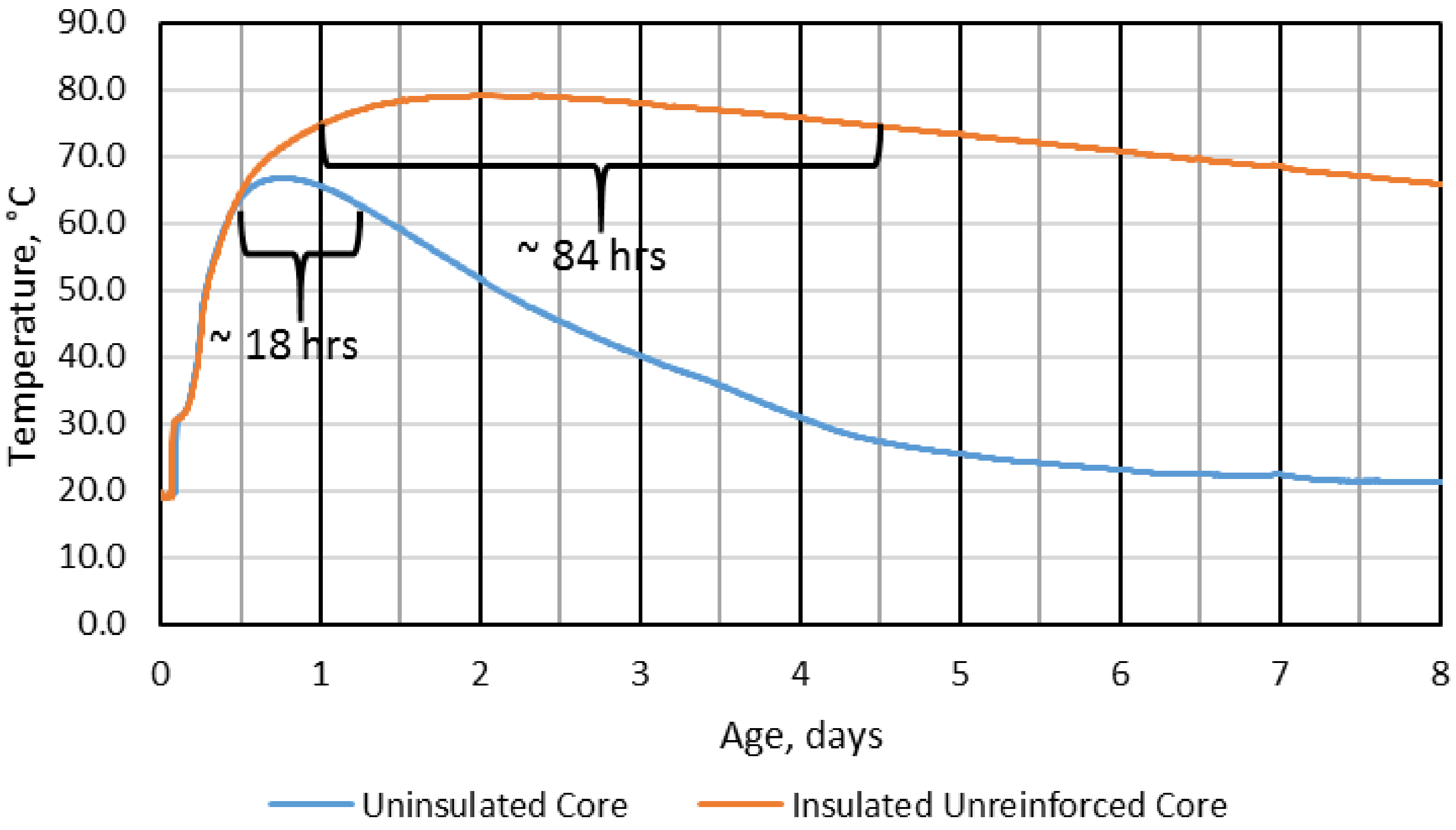
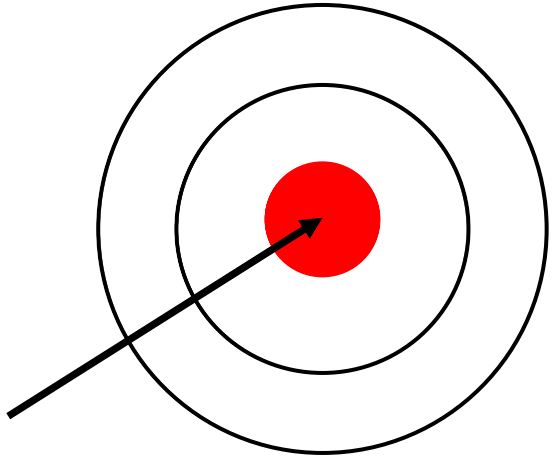


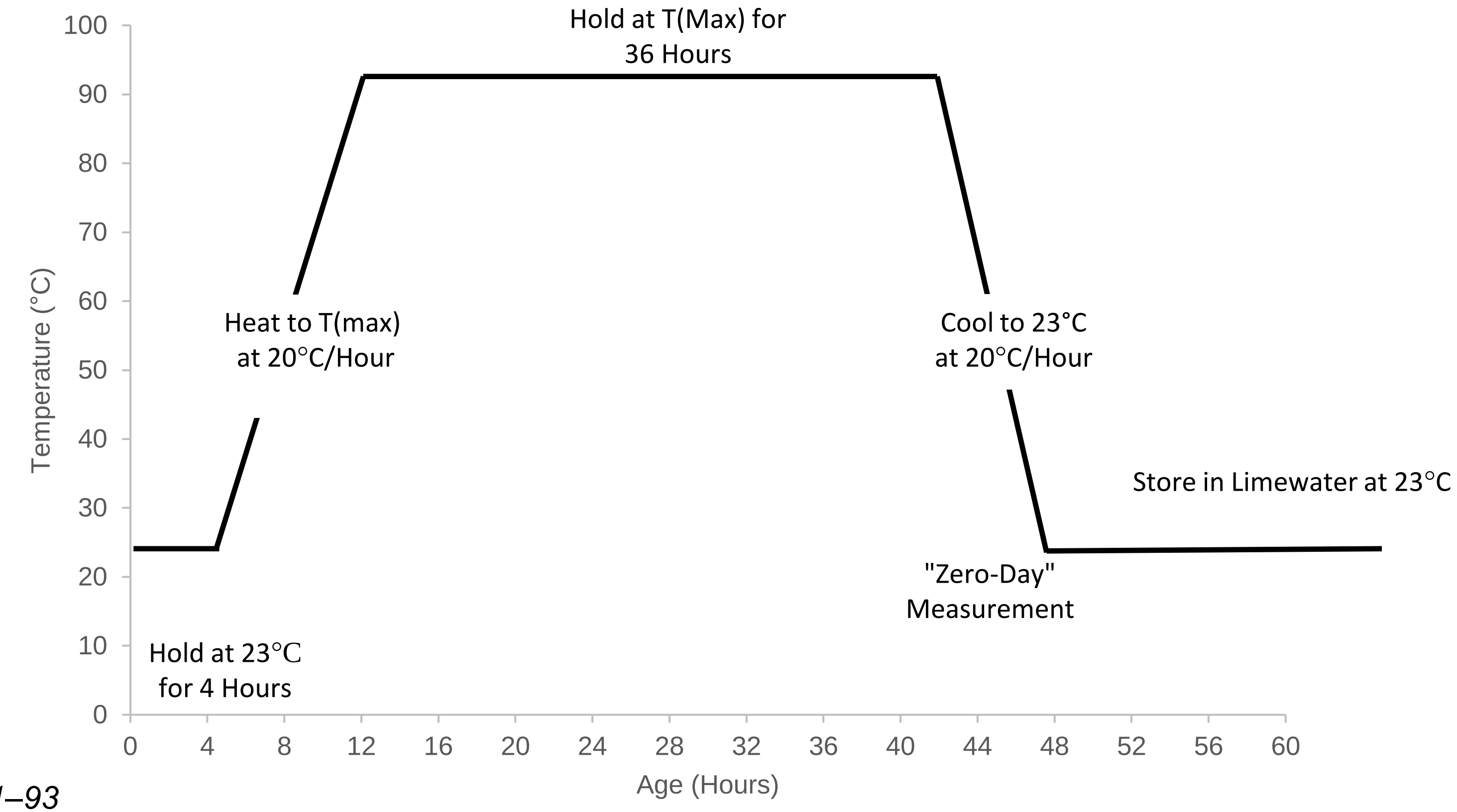
"New" Contributions - 2015 – 2022

- What is the threshold temperature for delayed ettringite formation (DEF) in portland cement with Concrete (OPC)?
- What is the threshold temperature for when combined with supplementary cementitious materials?
- What should be the maximum allowable specified temperature for mass concrete during hydration to avoid DEF?
- Can we hit the "bullseye"?



New Method – “Ferraro Method”

- Evaluating the effect of longer “soak times” on the potential for DEF
- Massive concrete structures



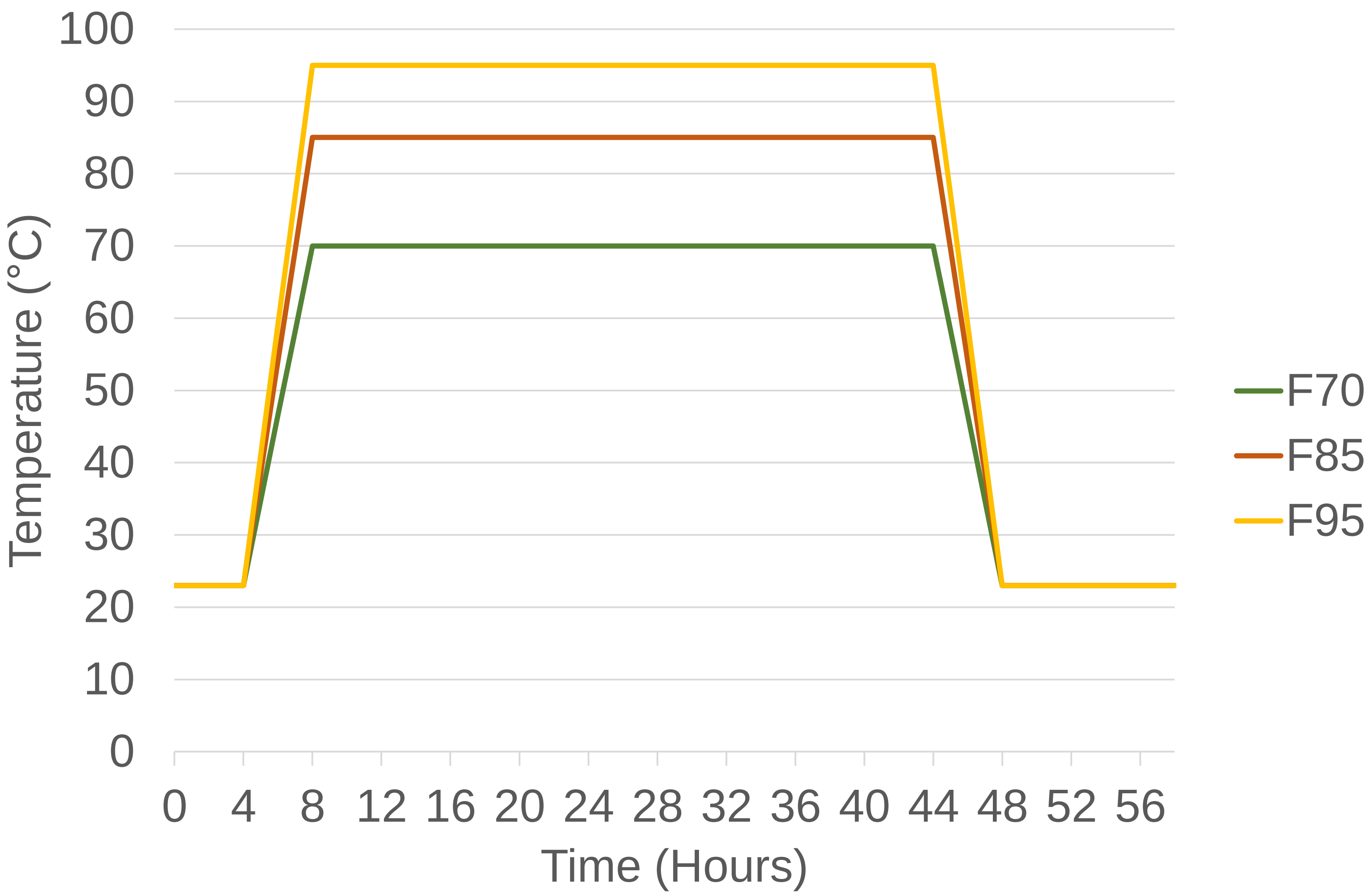
Paris, et al.. (2022) ACEM 11(1): 81–93
Ferraro (2018) EPRI Final report 3002007577

New Method – "Ferraro Method"

Peak temperatures of

- 70°C (158°F)
- 85°C (185°F)
- 95°C (203°F)

48-hour curing profile before long-term storage in limewater

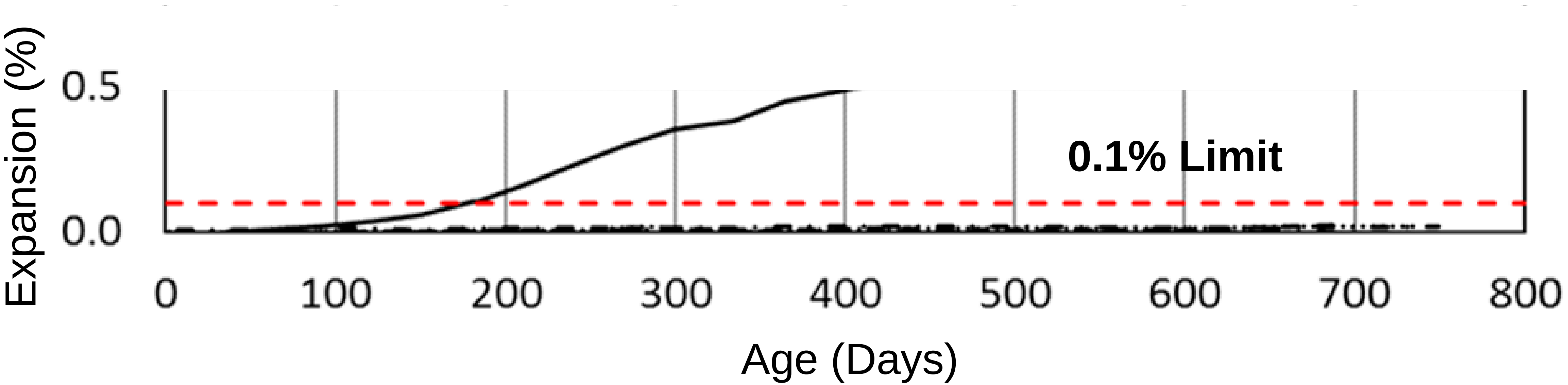


Paris, et al.. (2022) ACEM 11(1): 81–93
Ferraro (2018) EPRI Final report 3002007577

DEF and Instability

How much expansion is needed to “fail” a concrete or mortar?

- No uniformity
- ASTM C1293 (a much less aggressive ASR test: exposure for up to 24 months)
- ASTM C1260 – 0.10% expansion
- 0.1% limit for this work



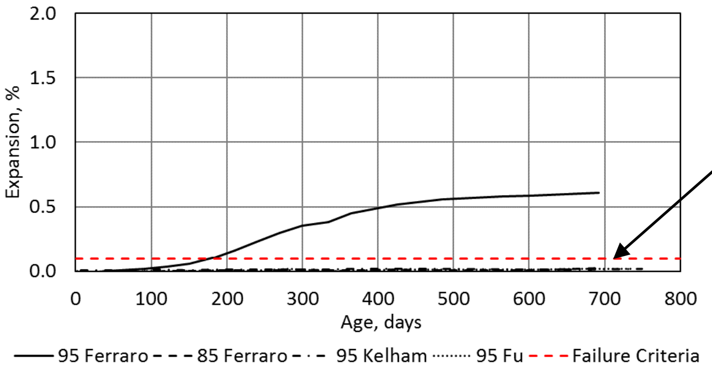
If its good enough for ASR…

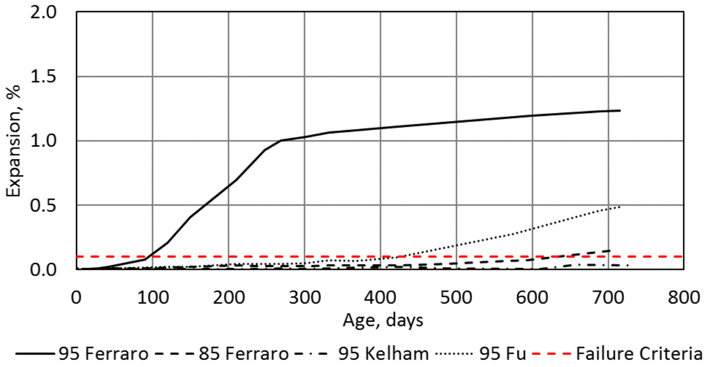
Paris, et al.. (2022) ACEM 11(1): 81–93
Ferraro (2018) EPRI Final report 3002007577

Testing and Results – Mortar Tests

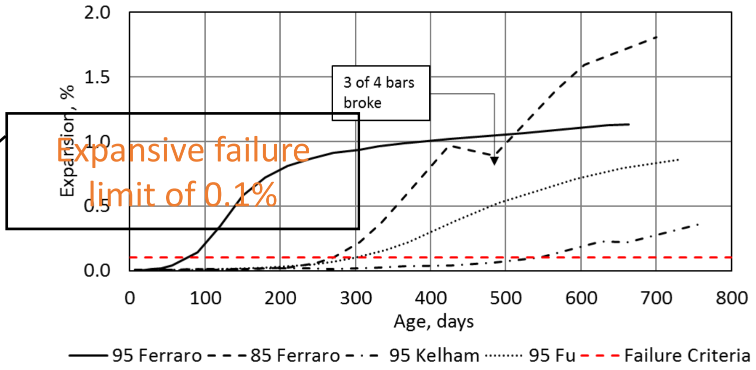
- Investigated mortars with Kelham and Fu methods at various maximum temperatures (85°C, 95°C)
- Proposed “Ferraro” method at same maximum temperatures, but exposure soak times (36 hours)
- Resulted in greatly accelerated results

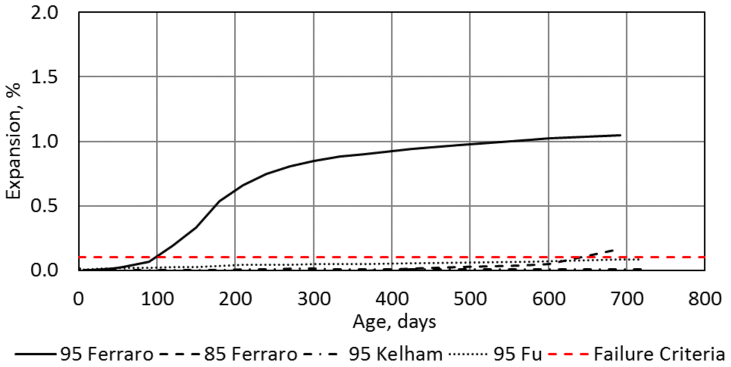
Type I/II Cements





Type III Cements

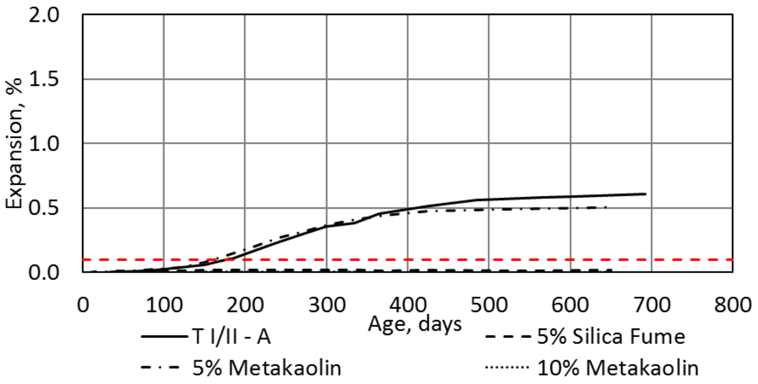


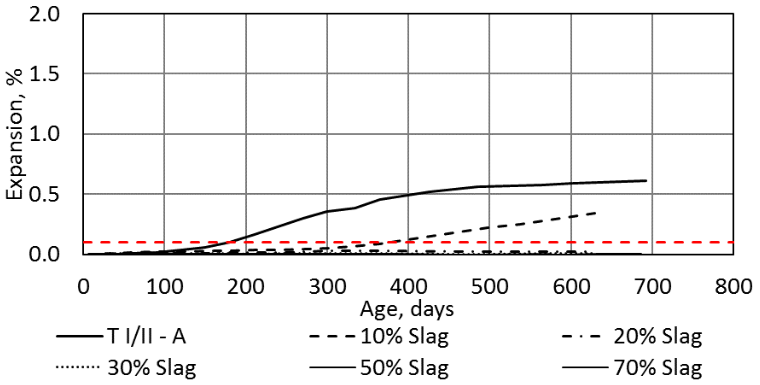


Testing and Results – Mortar Tests

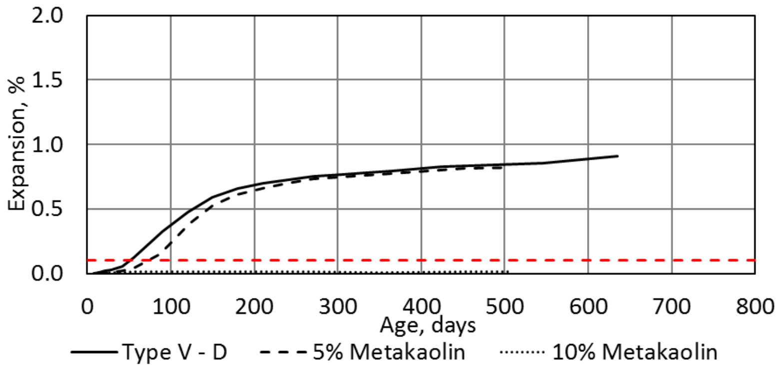
- Used new method with 95°C max. temperature to investigate 27 binary and ternary blends to assess mitigation

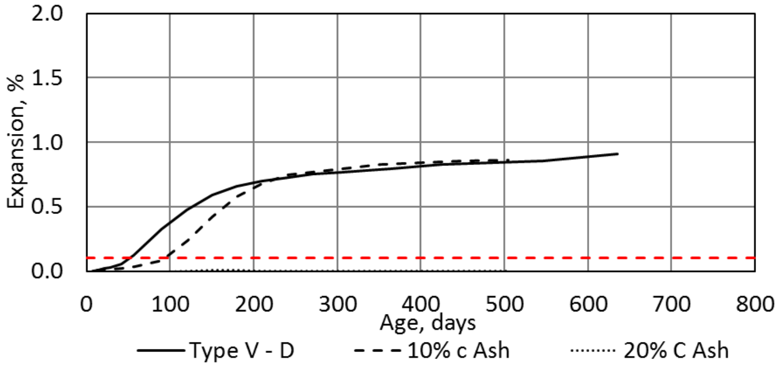
Type I/II Cement





Type V Cement





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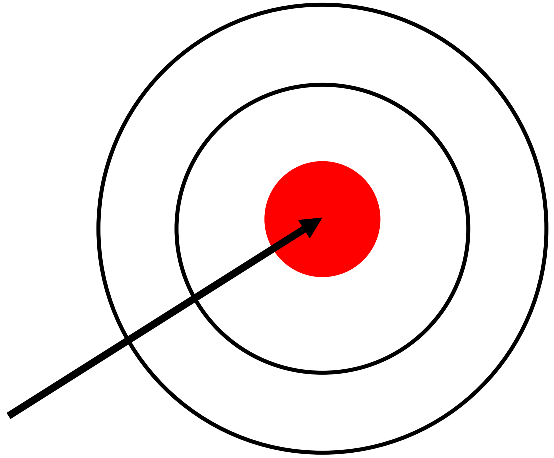
- What is the threshold temperature for delayed ettringite formation (DEF) in portland cement with Concrete (OPC)? **70°C**
- What is the threshold temperature for when combined with supplementary cementitious materials? **85°C**
- What should be the maximum allowable specified temperature for mass concrete during hydration to avoid DEF? **85°C**
- Can we hit the “bullseye“? **Yes**
- What if the bullseye moves?
  - Limestone Cements?
  - Coal ash? *(bottom ash)*
  - Alternative Pozzolans?



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Acknowledgements

- Electric Power and Research Institute
- Advanced Nuclear Technology: Mass Concrete Modeling and Thermal Control: Investigation of Delayed Ettringite Formation and Thermal Cracking in Massive Concrete Structures. EPRI, Palo Alto, CA: 2018. 3002007577. – David Scott
- FDOT Contract# BED31-977-04
  Effects of Composition and Temperature Control Measures on Mass Concrete Durability – Rodrigo Antunes and David Cerlanek
- Kyle Riding – University of Florida

Thank You

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