Study of shrinkage restraint effects at early-age in alkali-activated slag mortars

Fara Rifai, A. Darquennes, F. Benboudjema, B. Muzeau, L. Stefan

DE LA RECHERCHE À L'INDUSTRIE

STUDY OF SHRINKAGE RESTRAINT EFFECTS AT EARLY-AGE IN ALKALI-ACTIVATED SLAG MORTARS

Aveline DARQUENNES[†]
Farid BENBOUDJEMA[†]
Benoist MUZEAU[*]
Lavinia STEFAN[††]

[*]Den-Service d'Etude du Comportement des Radionucléides (SECR)
CEA, Université Paris-Saclay, F-91191, Gif-sur-Yvette, France

[†]LMT Cachan – ENS Cachan – Paris-Saclay University, Cachan, France

[††]AREVA NC, D&S, Technical Direction, La Défense, France

IC-FraMCoS 9 | Farah RIFAI[*]

MAY the 31rst, 2016

www.cea.fr

Outline

I. Context, Problems and Strategy

II. Thermo-Chemical Analysis
- Modelling Strategy
- Experimental Tests
- Numerical Model and Results

III. Mechanical Analysis
- Modelling Approach
- Autogenous Shrinkage
- Tensile Strength and Young’s Modulus
- Summary
- Behavior law
- Elastic Calculations
- Damage Model Results

IV. Conclusions and Perspectives

Outline

I. **Context, Problems and Strategy**

II. **Thermo-Chemical Analysis**
- Modelling Strategy
- Experimental Tests
- Numerical Model and Results

III. **Mechanical Analysis**
- Modelling Approach
- Autogenous Shrinkage
- Tensile Strength and Young's Modulus
- Summary
- Behavior law
- Elastic Calculations
- Damage Model Results

IV. **Conclusions and Perspectives**

INDUSTRIAL CONTEXT

Power Plant reactors dismantling
→ Metallic nuclear waste →
Disposal?

Alkali-activated slag (AAS) mortar
Chosen with respect to corrosion behavior

Dimensional Stability?

Durability?

PhD final objective:

Thermo-chemo-mechanical 3D Model → cracking risks during package lifecycle
(+ effects of waste corrosion)

(AAS) mortar → Need for experimental data for validation/determination of Hydration models and Mechanical behavior laws

ECOFRIENDLY MATERIAL

Application of recycled materials in civil engineering

- **1 ton of cement** produced = **1 ton of CO_2** released → searching for new solutions
- Volume of cement produced = **555** kilos/person/year (2013)
- Applications of industrial alkali-activated by-products : Blast furnace slag BFS (steel industry), Fly Ash (Carbon combustion) ..

Construction using alkali-activated materials due to shortage in Portland Cement in USSR

CO_2 emission (t)	calcination	blending	Silicate sol.	Total	Reduction
Portland Cement	1.000	0.020		1.020	0
Recycled slag	0.140	0.018	0.050	0.208	80%

CO_2 emissions reduction in blast furnace slag binders fabrication [Davidovids, 2014]

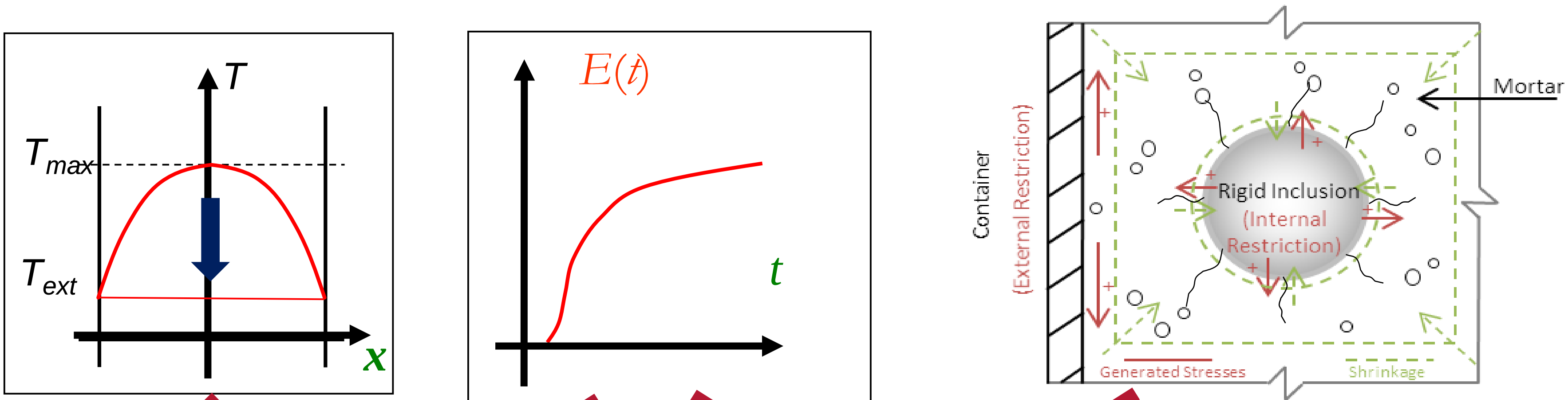
EARLY-AGE CRACKING RISK ORIGINS?

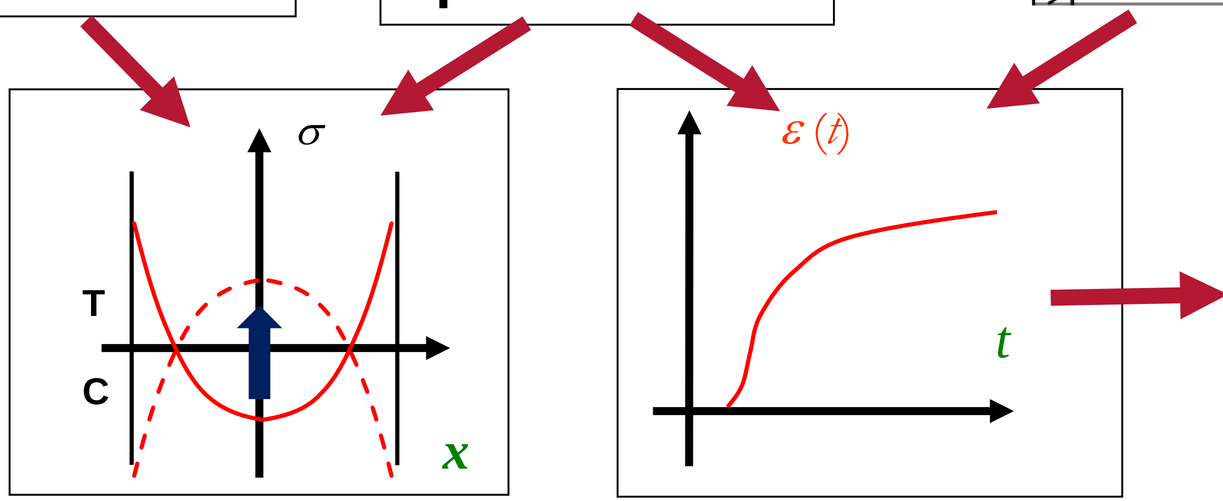
Stresses generated by <u>self-restraint</u>

Thermal Gradient

Stresses generated by <u>internal/external restraints</u>

Shrinkage restriction





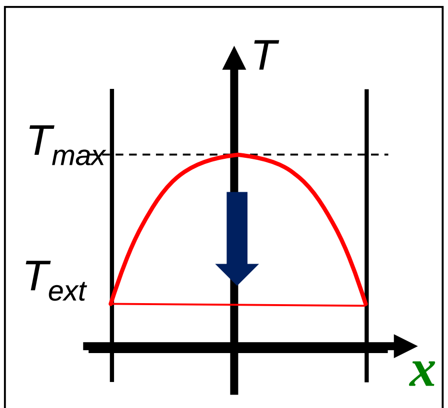
Fully-restricted :

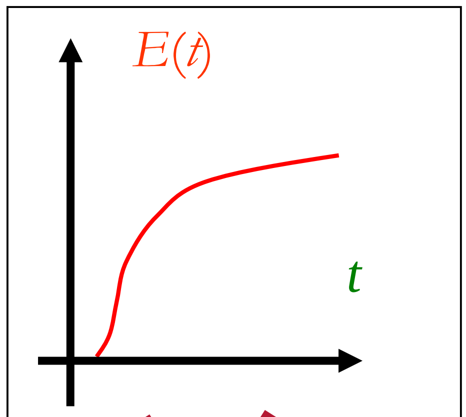
$$\dot{\sigma}(t) = E(t)\,\dot{\varepsilon}(t)$$

EARLY-AGE CRACKING RISK ORIGINS?

Stresses generated by <u>self-restraint</u>

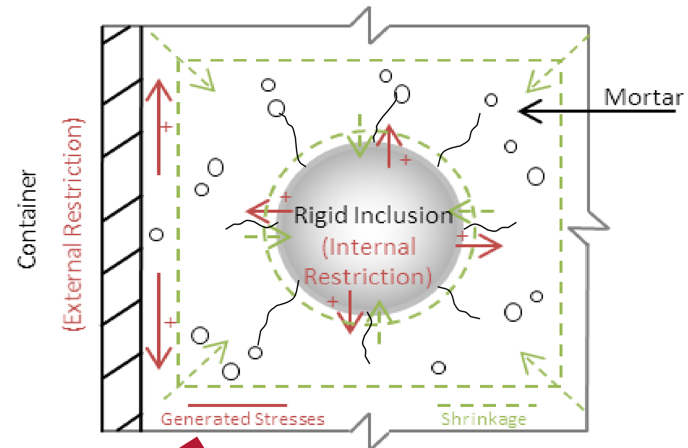
Thermal Gradient

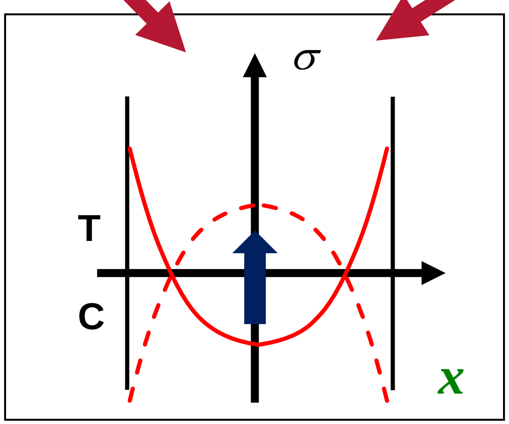


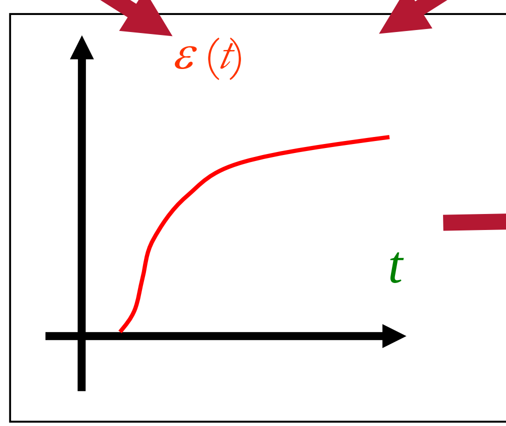


Stresses generated by <u>internal/external restraints</u>

Shrinkage restriction







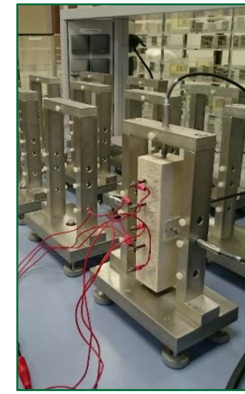
Fully-restricted :

$$\dot{\sigma}(t) = E(t)\,\dot{\varepsilon}(t)$$

MODELLING STRATEGY

« Classical » tests (identification)

« Electro-chemical/mechanical » coupled tests



Deformations within hydraulic binder matrix

$$\boldsymbol{\varepsilon(t) = \varepsilon^{e}(t) + \varepsilon^{th}(t) + \varepsilon^{au}(t) + \varepsilon^{bc}(t) + \varepsilon^{corr}(t)}$$

Study in autogenous conditions

$E(t)$ + ***restrictions***

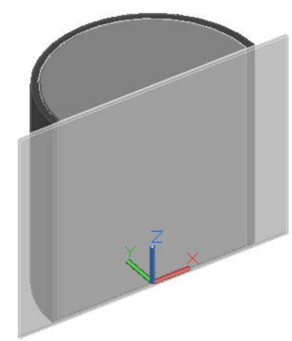
Stress generation $\boldsymbol{\sigma(t)}$

$\sigma(t) > f(t)??$

Material cracking??

Thermo-chemo mechanical modelling

Validation Tests



Instrumented mock-up (thermo-couples, DIC, Strain gauges)

Modelling of Scale 1 packages

Outline

I. Context, Problems and Strategy

II. **Thermo-Chemical Analysis**
- **Modelling Strategy**
- **Experimental Tests**
- **Numerical Model and Results**

III. Mechanical Analysis
- Modelling Approach
- Autogenous Shrinkage
- Tensile Strength and Young's Modulus
- Summary
- Behavior law
- Elastic Calculations
- Damage Model Results

IV. Conclusions and Perspectives

MODELLING STRATEGY

Thermo-Chemical Numerical Model

Heat Balance Equation + Hydration Heat Source

$$C\dot{T} = \nabla(k\nabla T) + L\dot{\xi}$$

C = Volumetric thermal heat capacity [J.C^{-1}.m^{-3}] - $C\ (T, t_e) = C$
k = Thermal conductivity [W.C^{-1}.m^{-1}] - $k\ (T, t_e) = k$
L = hydration latent heat

Hydration (Thermo-activation)

$$\dot{\xi} = \widetilde{A}(\xi)\exp\left(-\frac{E_a}{RT}\right)$$

[Ulm et Coussy, 1998]

- ✓ Chemical Affinity : polynomial function
- ✓ Thermo-activation : Ahrrenius' law
- ✓ **Time Correction (Maturity equivalent time)**

Hydration Reaction Advancement Degree

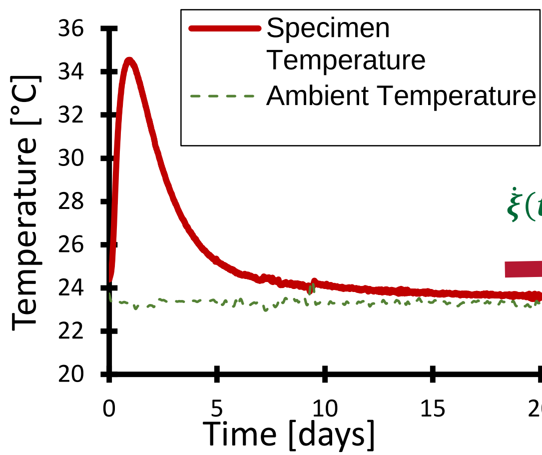
- ✓ Hydration reaction heat release :
  proportional to the advancement degree $\xi(t)$

$$\xi(t) = \frac{Q(t)}{Q_\infty}$$

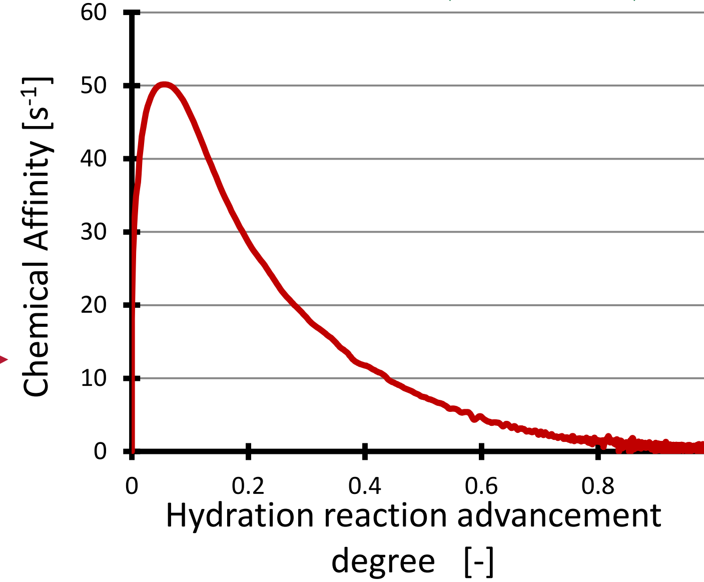
EXPERIMENTAL TESTS

Semi-adiabatic Calorimetry method
cylindrical specimen (Φ = 7 cm; h = 16 cm)

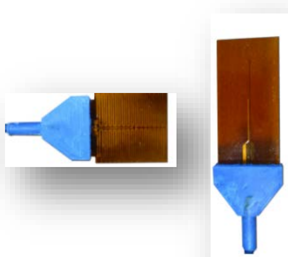
$$\dot{\xi}(t) = A(\xi(t))\exp\left(-\frac{E_a}{RT_{adiab}(t)}\right)$$



$$\dot{\xi}(t) = \frac{\dot{q}(t)}{L}$$



Hot wire/plane method

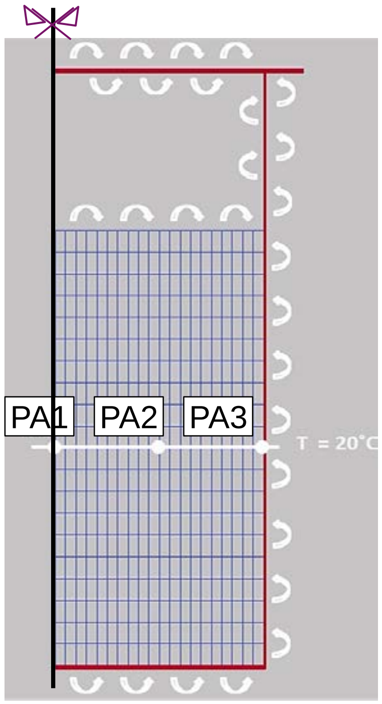


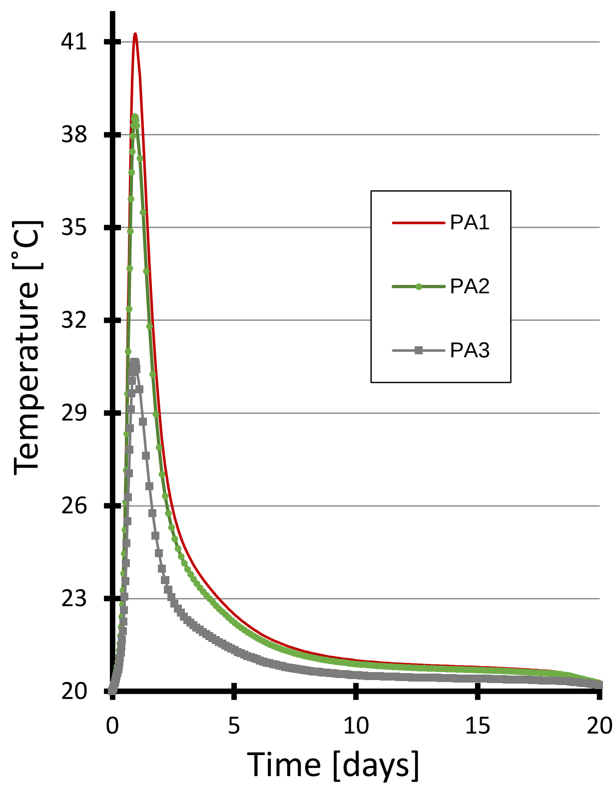
Thermal Conductivity (W/(m.K))	Thermal Effusivity ($W.s^{1/2}/(m^2.K)$)	Specific Heat (J/(kg.K))
2.642	1623	**457**

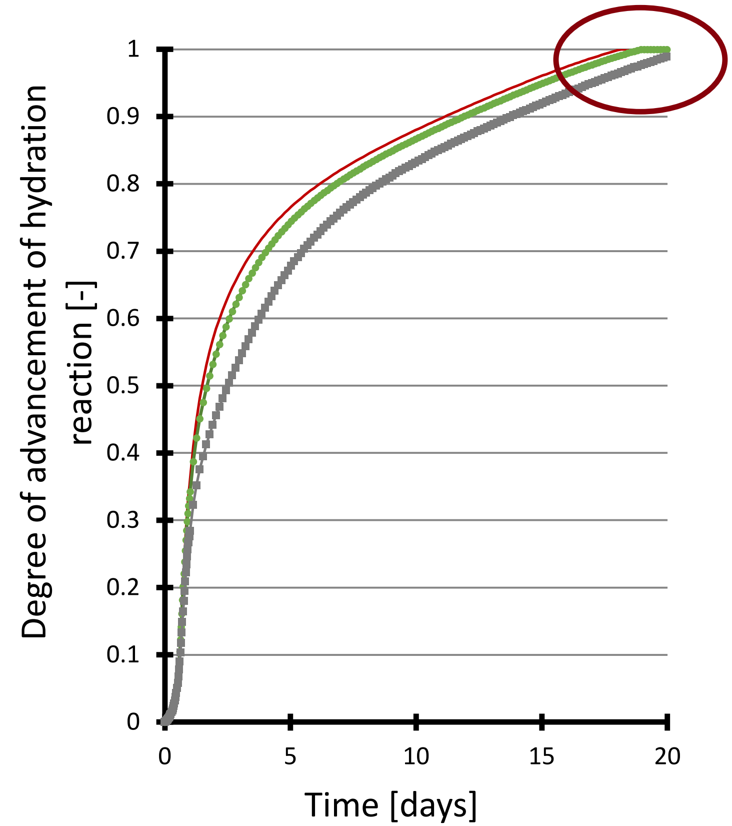
$$E = \sqrt{\lambda \rho C}$$

NUMERICAL MODEL AND RESULTS

$$\varphi = \varphi_{conv} + \varphi_{rad}$$







- ✓ Rise of 20°C in the center of the package
- ✓ Low thermal gradient → low cracking risk associated to thermal shrinkage self-restraint

Outline

I. Context, Problems and Strategy

II. Thermo-Chemical Analysis
- Modelling Strategy
- Experimental Tests
- Numerical Model and Results

III. Mechanical Analysis
- Modelling Approach
- Autogenous Shrinkage
- Tensile Strength and Young's Modulus
- Summary
- Behavior law
- Elastic Calculations
- Damage Model Results

IV. Conclusions and Perspectives

APPROPRIATE MODELLING APPROACH ??

Classical Approach
Hydration Reaction advancement degree

$$\bar{\xi} = \left\langle \frac{\xi - \xi_0}{\xi_\infty - \xi_0} \right\rangle_+$$

ξ_0 - Hydration degree at setting time

Eurocode Approach
Equivalent Time empirical model

$$t_e(t,T) = \int_0^t \exp\left[-\frac{E_a}{R}\left(\frac{1}{T} - \frac{1}{T_{ref}}\right)\right] \mathrm{d}t$$

T_{ref} - Reference temperature

Autogenous Shrinkage Evolution

$\dot{\varepsilon}_{ij}^{au} = \kappa \dot{\xi} \delta_{ij}$ For $\xi > \xi_0$

✓ κ = f (binder composition)

$$\varepsilon^{au}(t_e) = [\beta_c]^{n_{\varepsilon^{au}}} \cdot \varepsilon^{au}{}_{28}$$

✓ $\varepsilon^{au}{}_{28}$ = shrinkage at the age of 28 days

Mechanical Properties' Evolution

Young's Modulus

$$E(\xi) = E_\infty \, \bar{\xi}^{a_E}$$

Tensile Strength

$$f_t(\xi) = f_{t\infty} \, \bar{\xi}^{a_{f_t}}$$

[De Schutter et Taerwe, 1996]

$$\beta_c = \exp\left[s\left(1 - \sqrt{\frac{28}{\langle t_e - t_0\rangle_+}}\right)\right]$$

t_0 - setting time

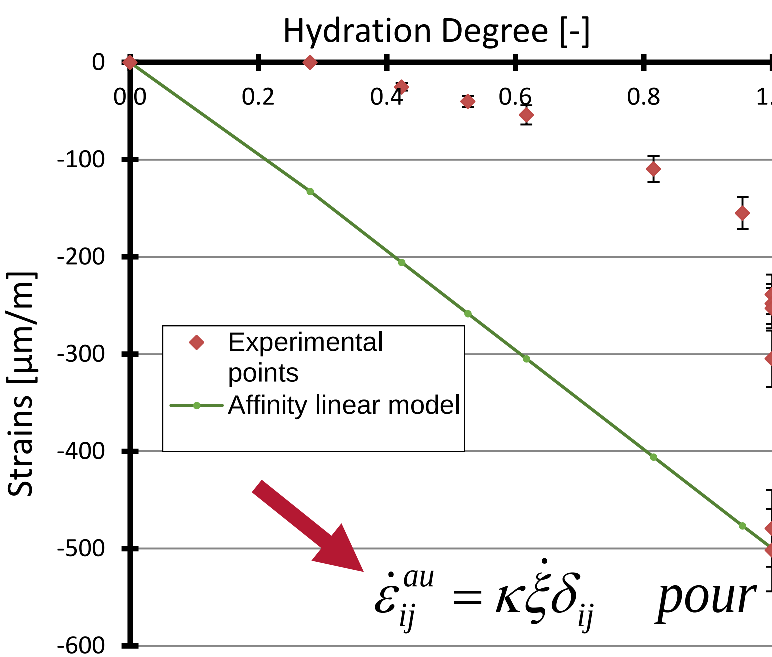
$$E(t_e) = [\beta_c]^{n_E} \cdot E_{28}$$

$$f_t(t_e) = [\beta_c]^{n_f} \cdot f_{t,28}$$

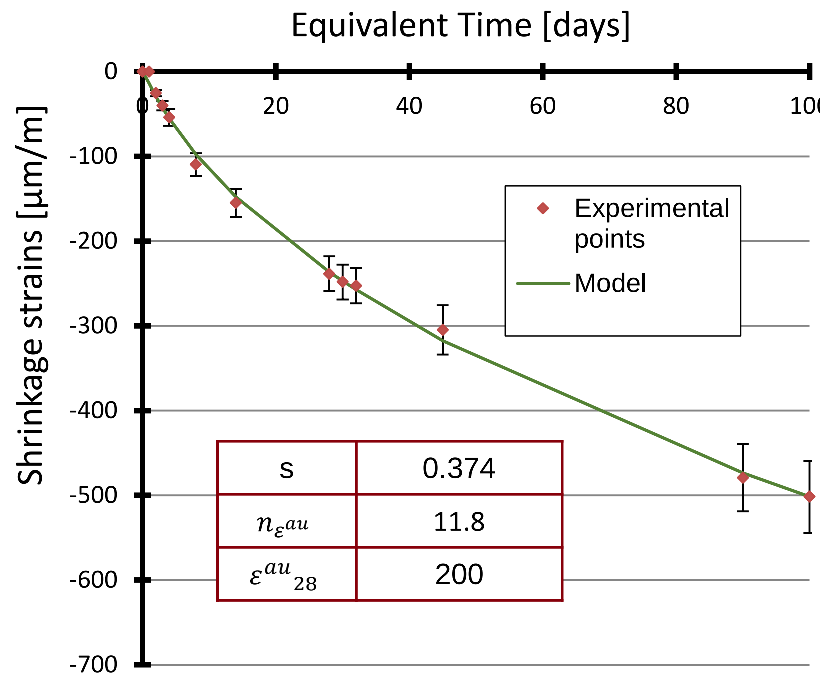
✓ $E_{28}, f_{t,28}$ = properties at the age of 28 days

AUTOGENOUS SHRINKAGE MEASUREMENTS

LVDT displacement monitoring on 3 duplicate samples 4x4x16 cm



$$\dot{\varepsilon}_{ij}^{au} = \kappa \dot{\xi} \delta_{ij} \quad pour \ \xi > \xi_0$$



$$\varepsilon^{au}(t_e) = [\beta_c]^{n_{\varepsilon^{au}}} \cdot \varepsilon^{au}{}_{28}$$

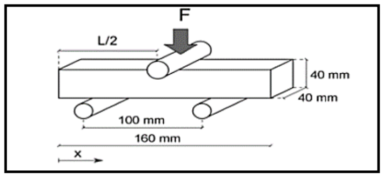
$$\beta_c = \exp\left[s \left(1 - \sqrt{\frac{28}{\langle t_e - t_0 \rangle_+}} \right) \right]$$

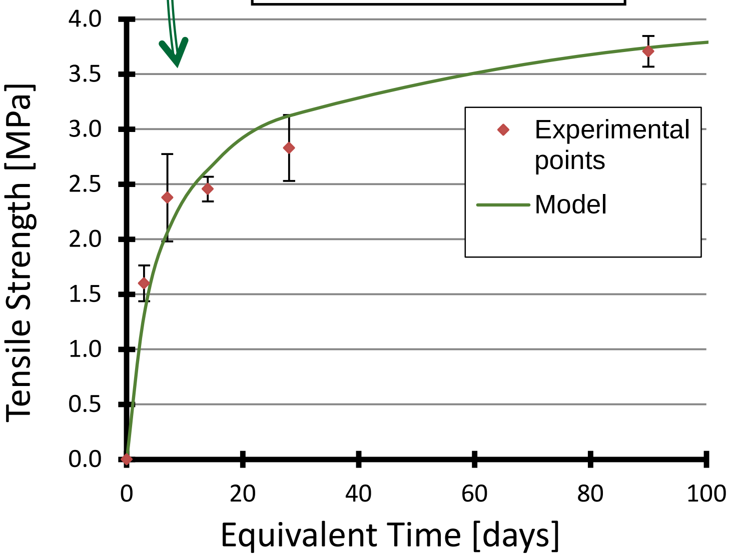
TENSILE STRENGTH AND YOUNG MODULUS EVOLUTION

3 points flexural test on 3 duplicate samples 4x4x16

$$f_t = \frac{f_{fl}}{1.6}$$

[EC2, 1992]

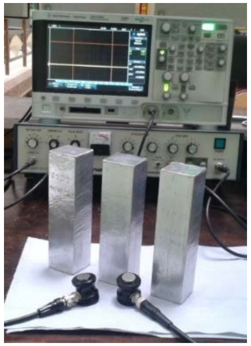




$$f_t(t_e) = [\beta_c]^{n_f} \cdot f_{t,28}$$

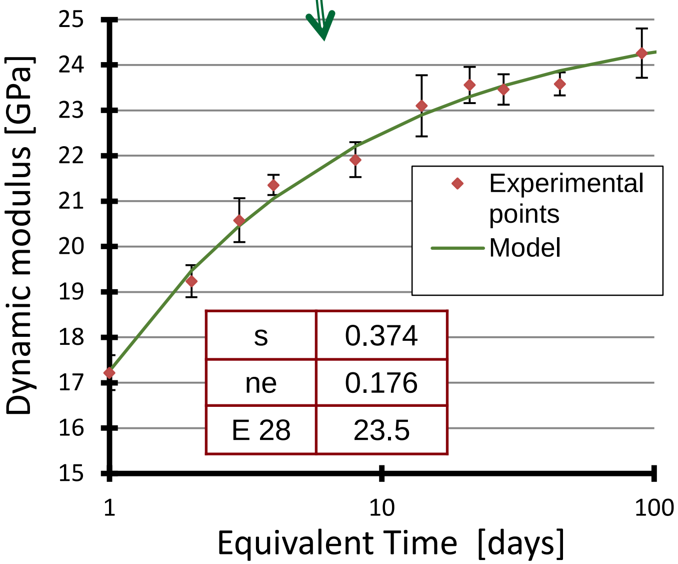
s	0.374
nft	1.09
Ft 28	3.13

Ultrasonic measurements on 3 duplicate samples 4x4x16



$$E_s = \frac{E_d}{1.2}$$

$$E(t_e) = [\beta_c]^{n_E} \cdot E_{28}$$



s	0.374
ne	0.176
E 28	23.5

MECHANICAL ANALYSIS PARAMETERS - SUMMARY

Basic creep relaxation effect taken into account in calculation tensile strength

[EC2, 1992]

Elastic strains tensor

$$\boldsymbol{\varepsilon}^{e}(\boldsymbol{t}) = \boldsymbol{\varepsilon}(\boldsymbol{t}) - (\boldsymbol{\varepsilon}^{th}(\boldsymbol{t}) + \boldsymbol{\varepsilon}^{au}(\boldsymbol{t}) + \boldsymbol{\varepsilon}^{bc}(\boldsymbol{t}))$$

$$\dot{\varepsilon}^{th} = \alpha_T \dot{T}$$

$$\varepsilon^{au}(t_e) = [\beta_c]^{n_{\varepsilon^{au}}} \cdot \varepsilon^{au}{}_{28}$$

Young Modulus and Tensile strength evolution

$$E(t_e) = [\beta_c]^{n_E} \cdot E_{28}\,; \qquad f_t(t_e) = [\beta_c]^{n_f} \cdot f_{t,28}\,; \qquad n_E = 0.176\,; \quad n_{f_t} = 1.09$$

Giving the Tensile strain threshold $\kappa_0 = \dfrac{f_t}{E}$

Effective stresses tensor

$$\dot{\bar{\sigma}} = E\,\dot{\boldsymbol{\varepsilon}}^{e} = E\,(\dot{\varepsilon} - \dot{\boldsymbol{\varepsilon}}^{au} - \dot{\boldsymbol{\varepsilon}}^{th} - \dot{\boldsymbol{\varepsilon}}^{bc})$$

Isotropic elastic damage model based on Mazars Model

$$\sigma = (1 - \boldsymbol{D})\bar{\sigma}$$

Apparent stresses tensor

MECHANICAL BEHAVIOR DAMAGE LAW

Mazars Damage Criterion

$$f = \hat{\varepsilon} - \kappa_0 \; ; \qquad \hat{\varepsilon} = \sqrt{\langle \varepsilon_{el} \rangle_+ : \langle \varepsilon_{el} \rangle_+}$$

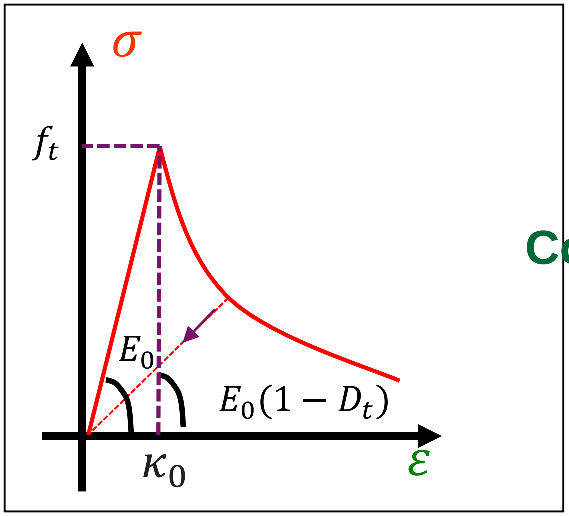
Equivalent tensile strain tensor

Damage Variable in tension $\quad \hat{\boldsymbol{\varepsilon}} \geq \boldsymbol{\kappa_0}$

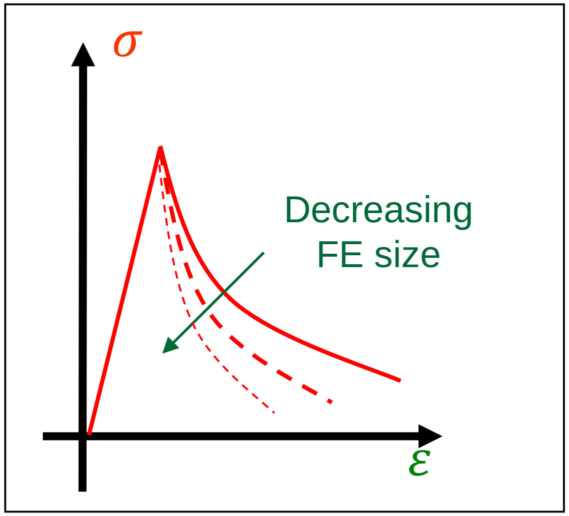
$$D = 1 - \frac{\kappa_0}{\hat{\varepsilon}}\left[(1 + A_t)\exp(-B_t\hat{\varepsilon}) - A_t \exp(-2B_t\hat{\varepsilon})\right]$$

Regularization using fracture energy

Dissipated energy density at failure



Constant B_t



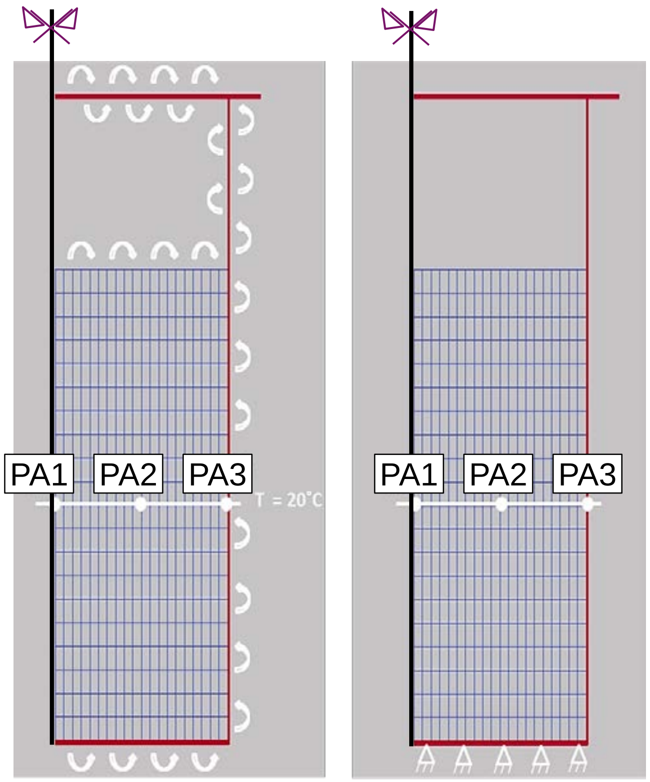
$$g_{ft}(te) = \frac{f_t(1 + A_t/2)}{B_t(te)}$$

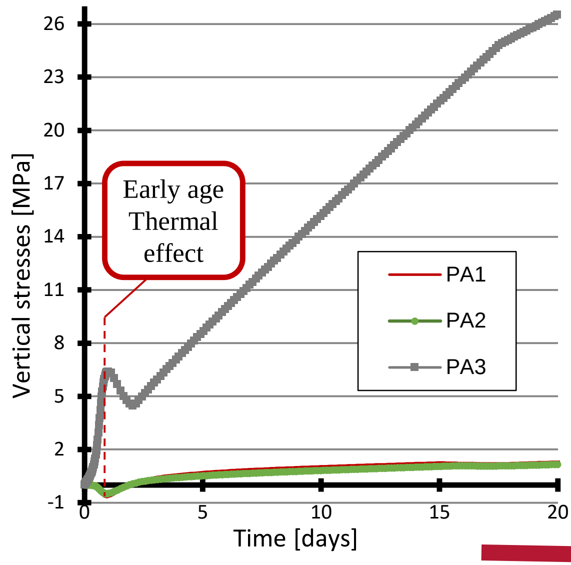
$$g_{ft}(te) = \frac{G_{ft}(te)}{l_c}$$

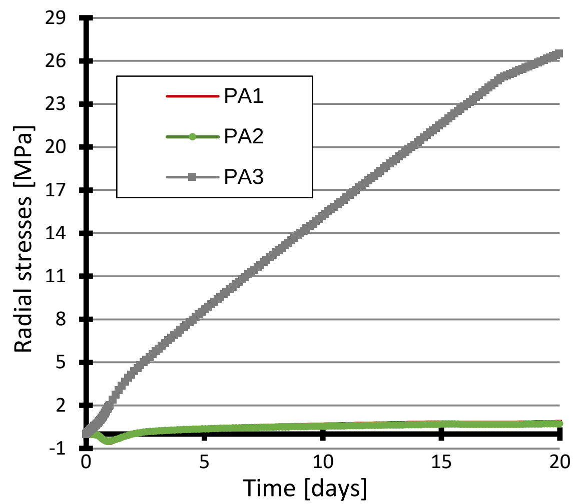
Finite Element characteristic length

[Hillerborg et al., 1976]

Geometric model with boundary conditions



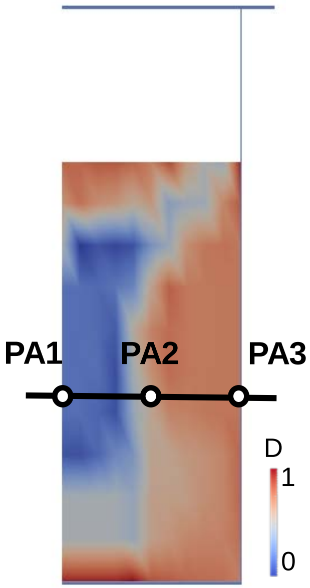




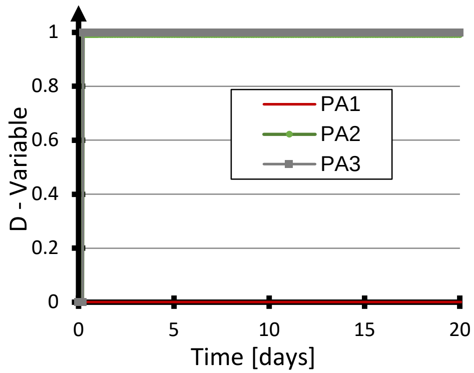
- ✓ Early age thermal effect results in surface tension, core compression
- ✓ Shrinkage restraint

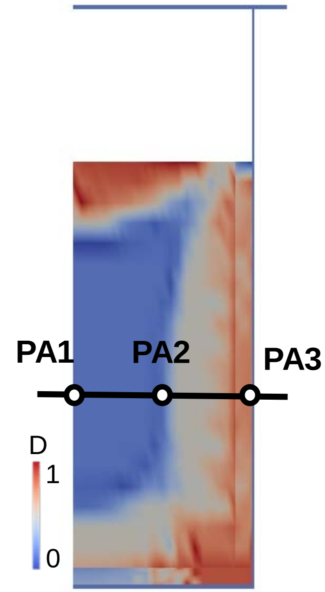
→High stresses at advanced ages

?? Steel/binder interface debonding

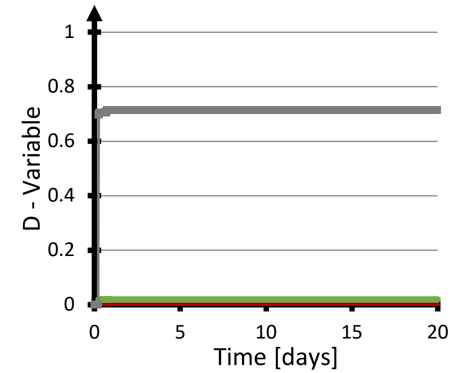


Original Geometry – without interfacial elements



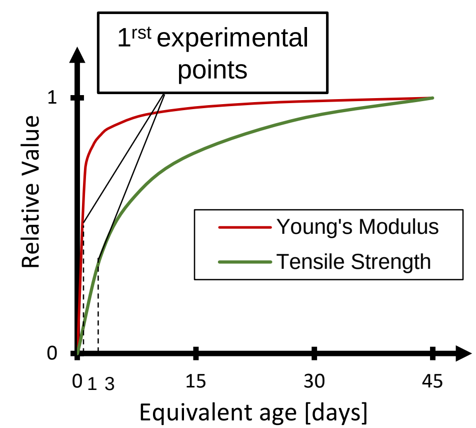


Interfacial elements – 5 cm ($f_t = f_t/3$)



Damage occurs at very early age – just after setting

Need to refine Tensile Resistance and Young's modulus development laws



$$\frac{n_{ft}}{n_E} \simeq 10$$

2-3 OPC binders

Outline

I. Context, Problems and Strategy

II. Thermo-Chemical Analysis
- Modelling Strategy
- Experimental Tests
- Numerical Model and Results

III. Mechanical Analysis
- Modelling Approach
- Autogenous Shrinkage
- Tensile Strength and Young's Modulus
- Summary
- Behavior law
- Elastic Calculations
- Damage Model Results

IV. Conclusions and Perspectives

CONCLUSIONS AND PERSPECTIVES

- Waste package : more sensitive to proper deformations restriction than temperature gradient

- Unexpected quick numerical damage → Dynamic to static E conversion
  Experimental points at early age
  Mechanical properties evolution
  Self-Healing??

- High radial stresses at mortar/container interface → Steel/Slag adhesion?
  Tensile creep ?

- High Autogenous shrinkage strains + significant evolution after 100 days
  → Internal relative humidity? → Phenomenological models

- Meso-scale Calculations → Rigid inclusion shrinkage restraint
  Thermal-equivalent corrosion strains
  Macro Properties → waste % in packages

Thank you for your attention !

Questions are Welcome ☺

F. RIFAI

Berkeley, May the 31rst 2016

Commissariat à l'énergie atomique et aux énergies alternatives
Centre de Saclay | 91191 Gif-sur-Yvette Cedex
T. +33 (0)1 69 08 98 53

Etablissement public à caractère industriel et commercial | RCS Paris B 775 685 019

Direction de l'Energie Nucléaire
Département de Physico-Chimie
Service d'Etude du Comportement des Radionucléides
LECBA