
LAFARGE

From microstructure to macroproperties: strength and creep of cementitious materials

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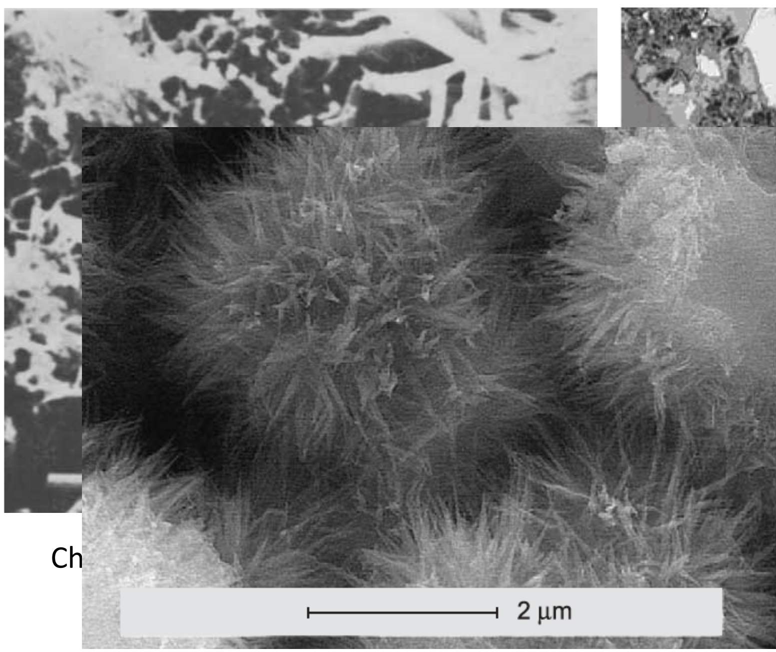
Motivation:

- cementitious materials are hierarchically organized
- (sub)micron-sized hydrate needles govern **creep and strength** of cementitious materials



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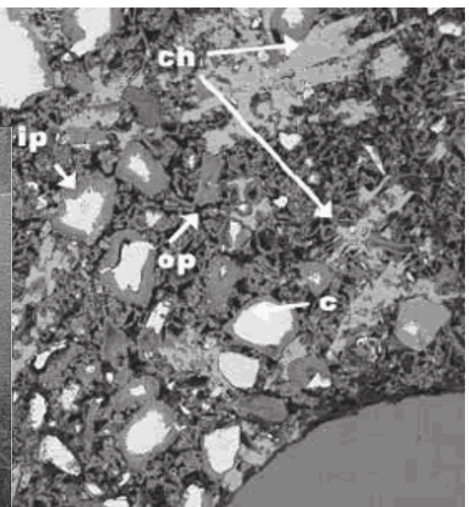
Tritthart & Häußler, CCR, 2003



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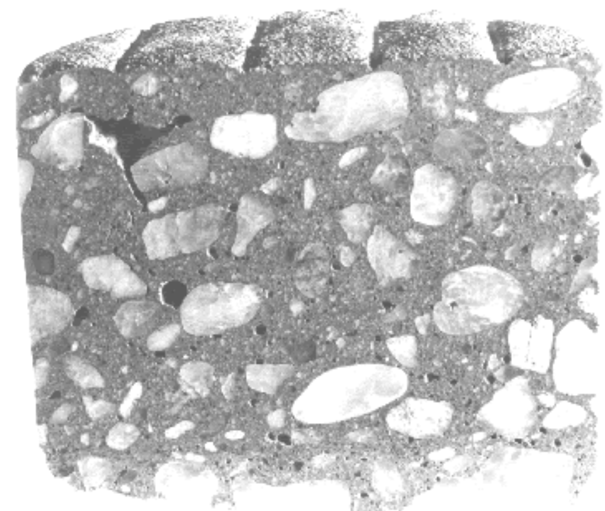

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Outline

Creep

1. Hourly-repeated, 3 min creep testing at early ages
2. Identification of hydrate creep function + validation

Strength

1. Elastic limit model for cement pastes

Early-age testing dilemma: coupling between creep and hydration

- duration of creep tests >> characteristic time of hydration
- Microstructural development ("aging") *during* creep test

Aim: decouple creep and hydration

- Characterize creep of specific ("non-aging") microstructures

Solution: new test protocol

- **3 minute creep tests**
  … referring to specific microstructures
- **hourly repeated**
  … successive creep tests refer to different microstructures

Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

Materials: OPC cement pastes

Raw materials

- CEM I 42.5 N
- distilled water

Compositions

- w/c = 0.42 / 0.45 / 0.50

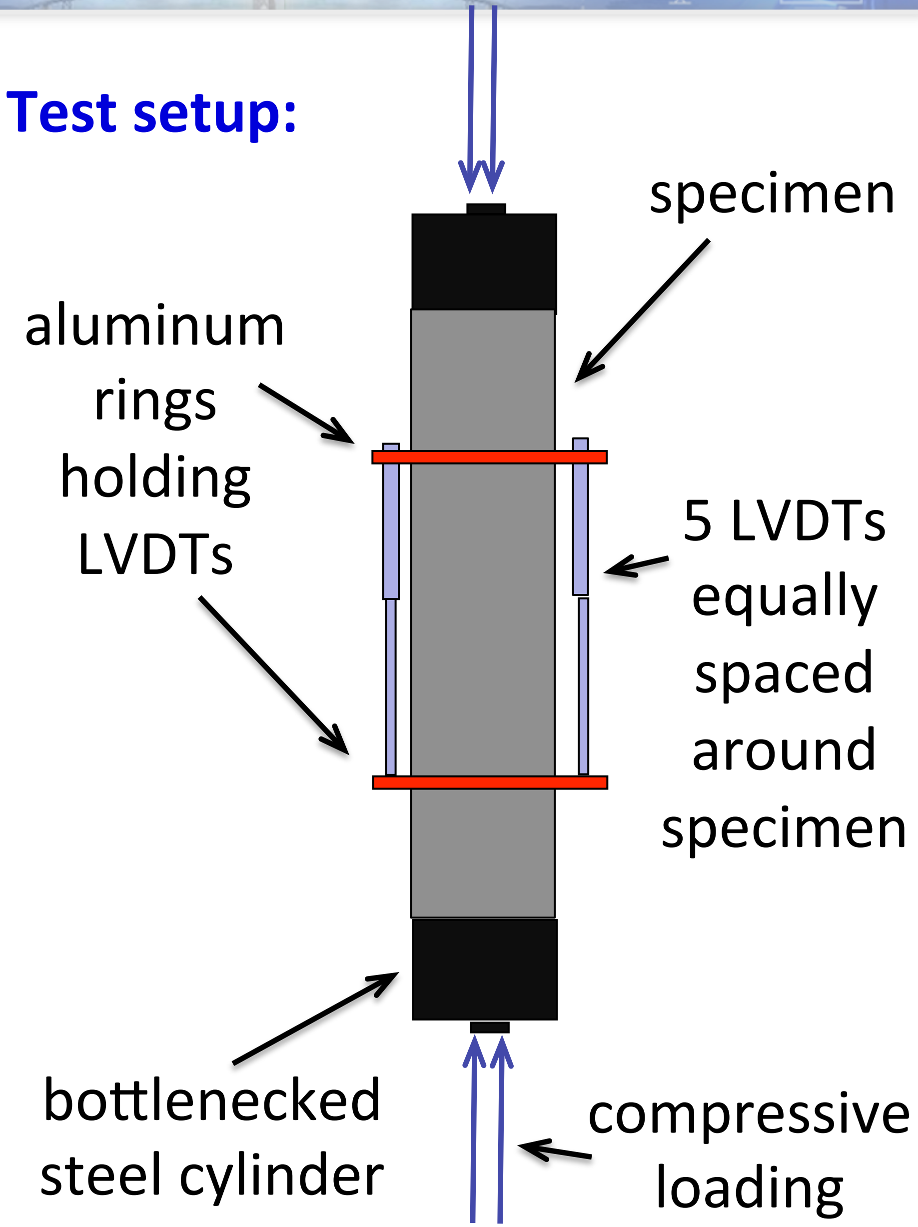
Cylindrical specimens

- diameter = 7 cm
- height = 30 cm

Curing conditions

- ambient air temp = 20 °C
- sealed (no drying)

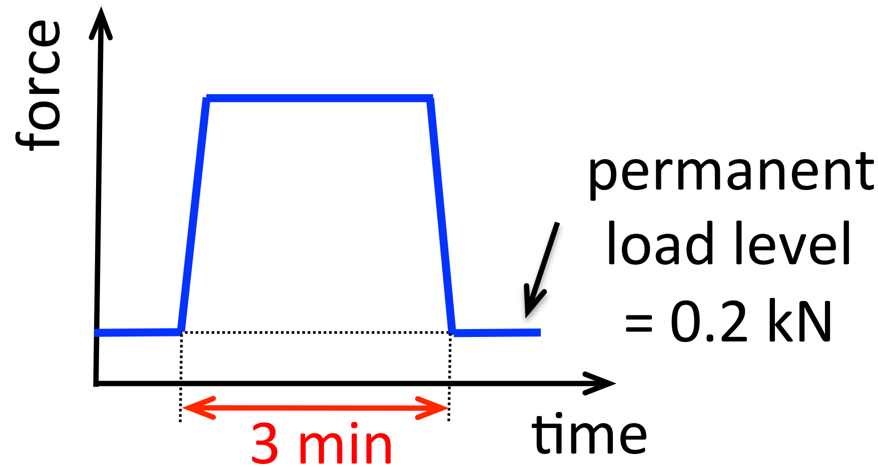
Test setup:



Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

Loading history:

- loading: …. 2 MPa/s
- unloading: 1 MPa/s



Hourly repeated:

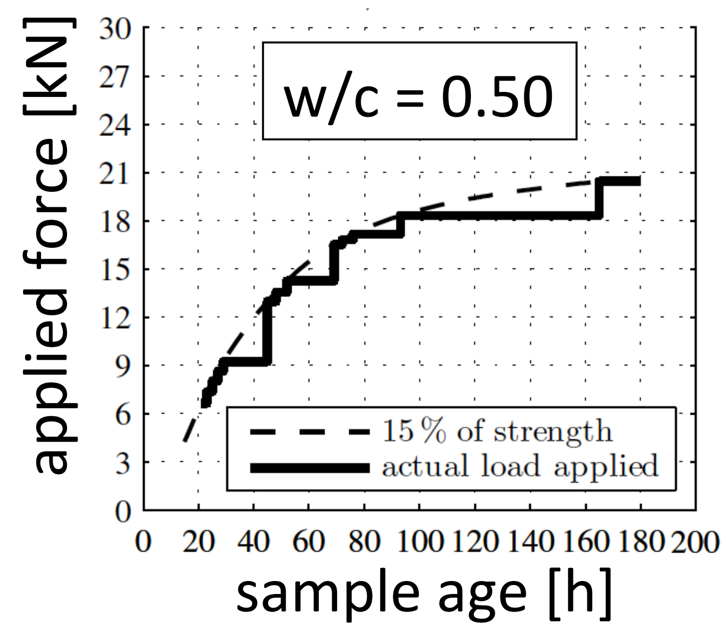
- From 21 hours after production up to 8 days

170 creep tests per specimen:

force level = max. 15 % of strength at time of testing, determined by

- calorimetry
- validated strength model

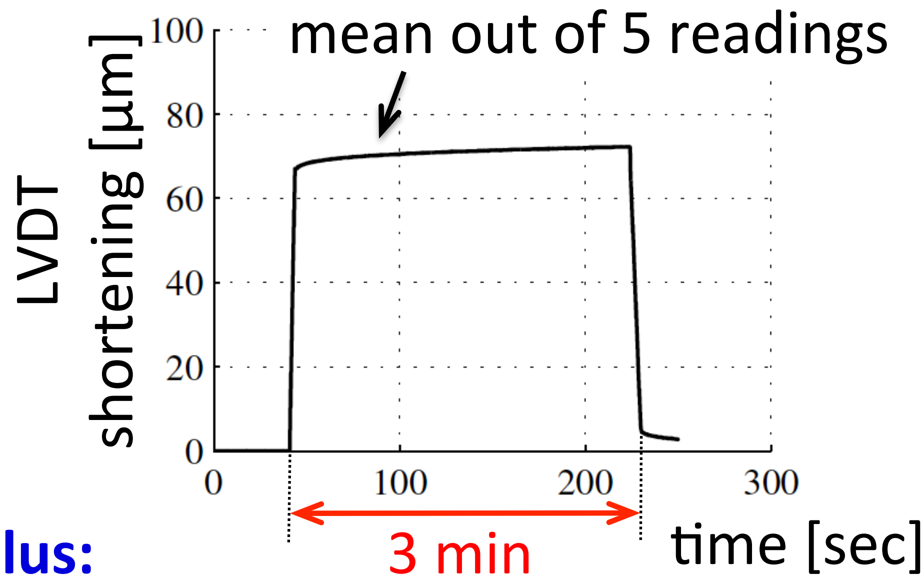
[Pichler Hellmich, CemConRes, 41, 2011]



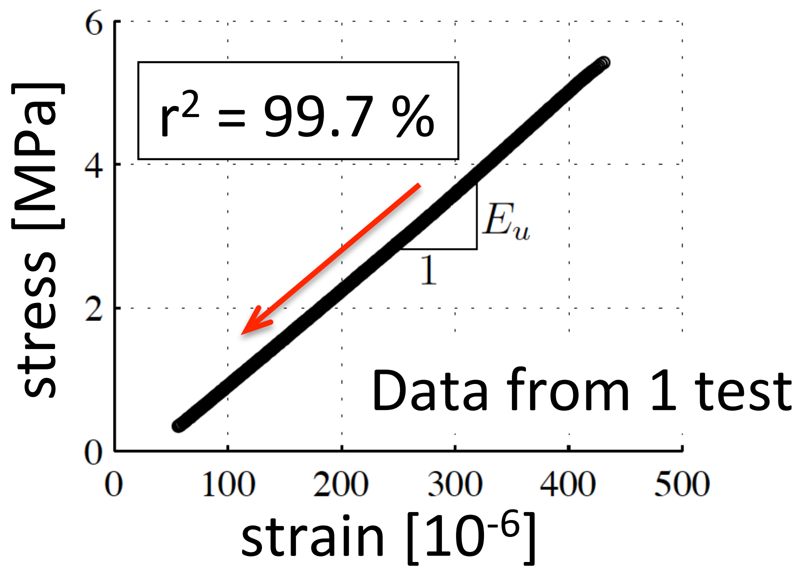
Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

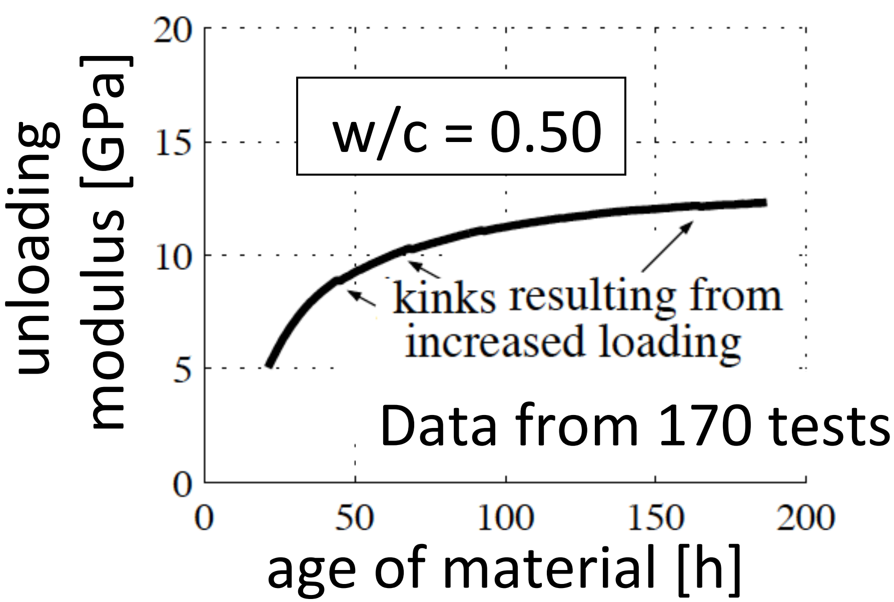
LVDT readings:

- small difference of 5 individual LVDT readings
  - ➢ very small eccentricity
  - ➢ mean value meaningful



Quantification of *unloading* modulus:





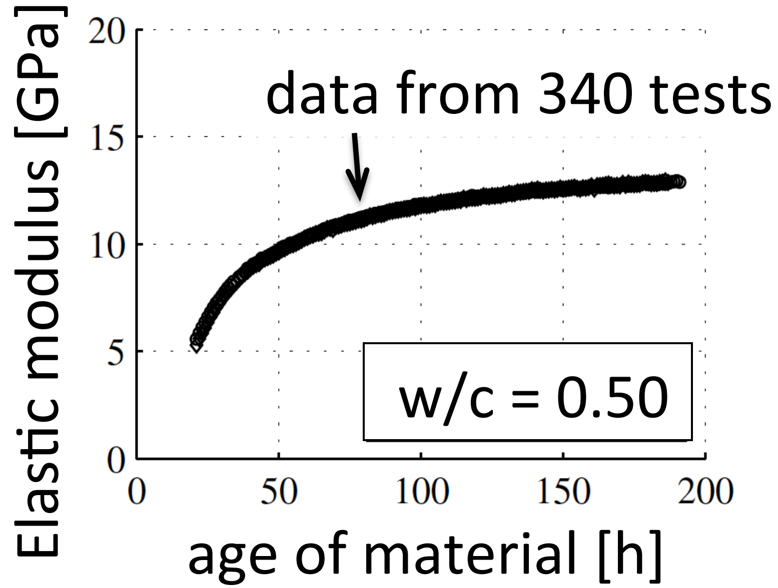
Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

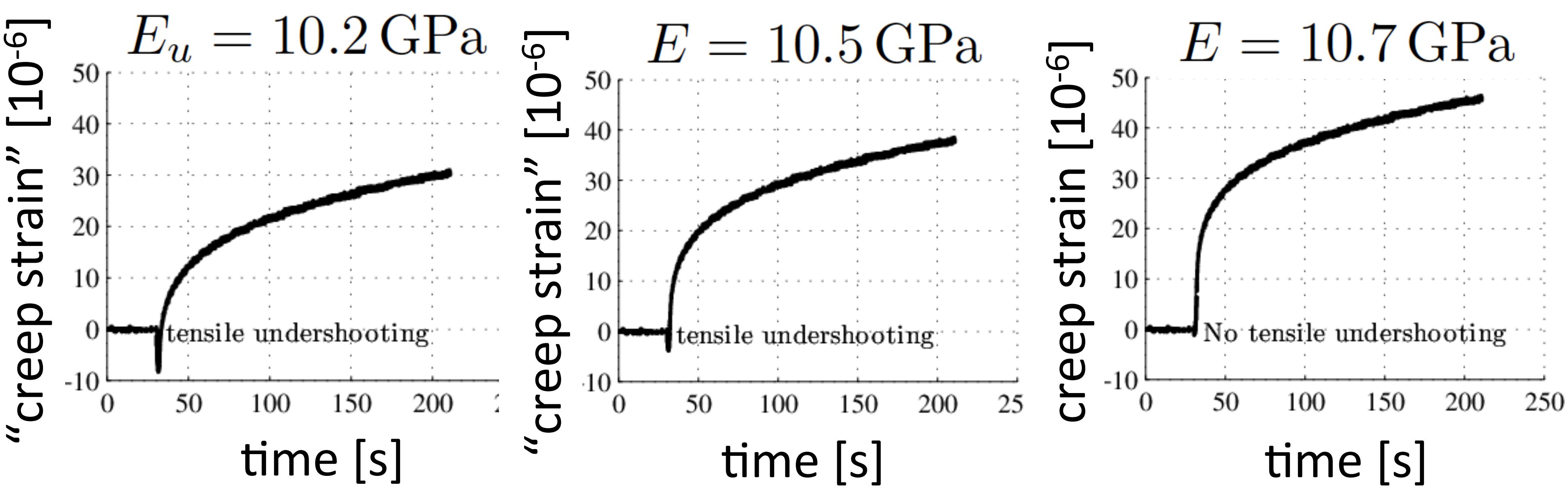
Quantification of *elastic* modulus:

- Quantify creep strains

$$\varepsilon_c(t) = \frac{\Delta\ell(t)}{\ell_0} - \frac{F(t)}{EA}$$

- Identify elastic modulus by avoiding *tensile* creep strains



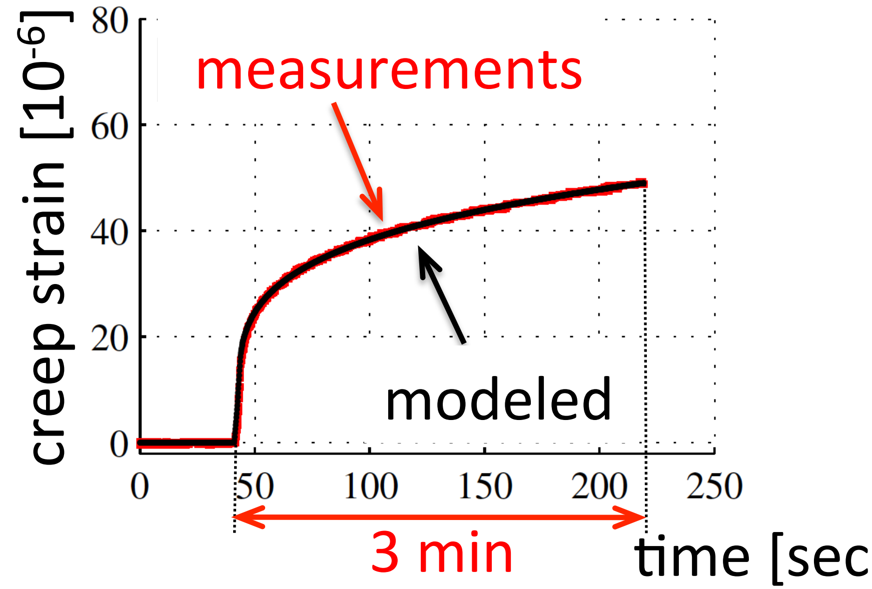


Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

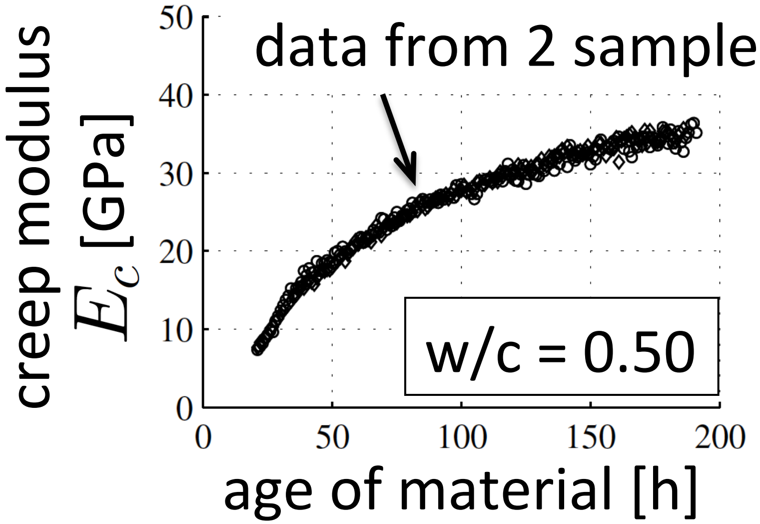
Creep strain evolution:

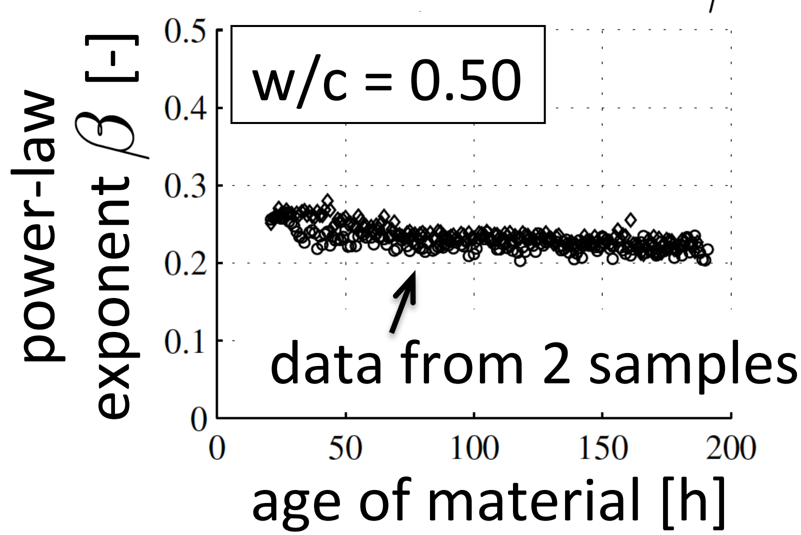
- fitting based on power law

$$\varepsilon_c(t) = \sum_{i=1}^{n} \frac{\Delta\sigma_i}{E_c} \left(\frac{t - t_i}{t_{ref}} \right)^{\beta}$$



Evolutions of creep modulus E_c and of power-law exponent β:

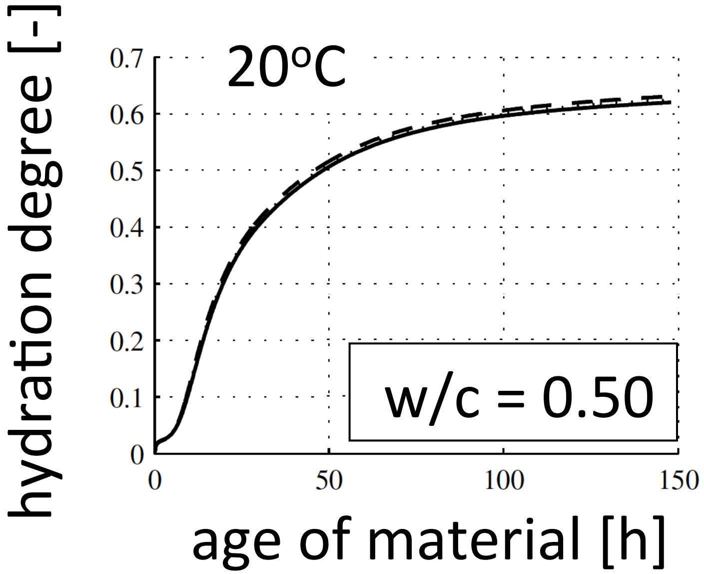




Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

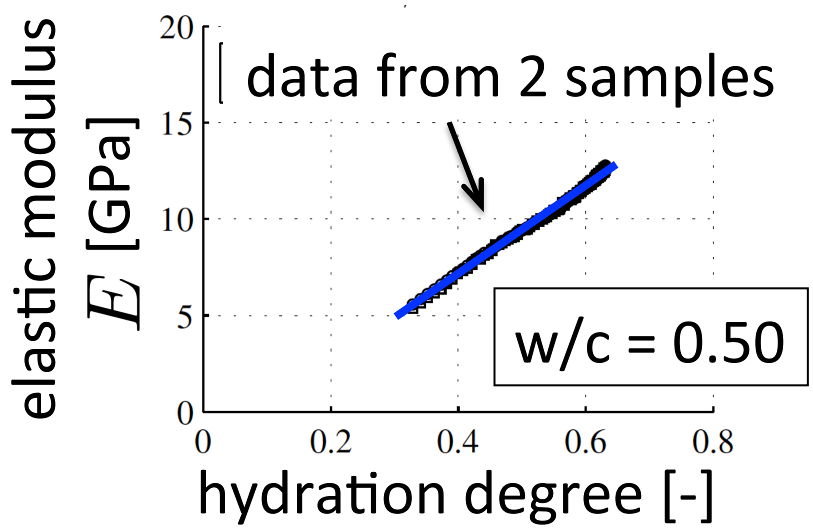
Hydration degree rather than material age

- calorimetry at 20°C



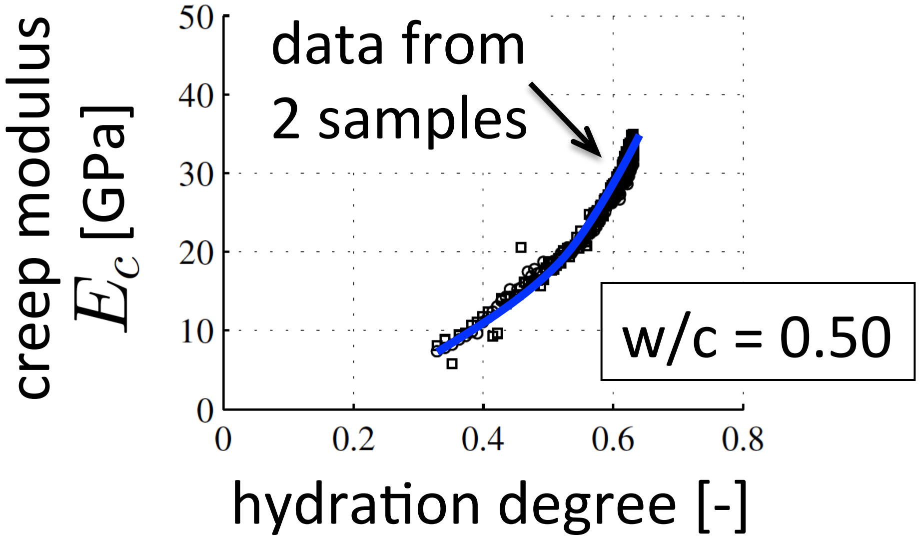
Evolution of elastic modulus E:

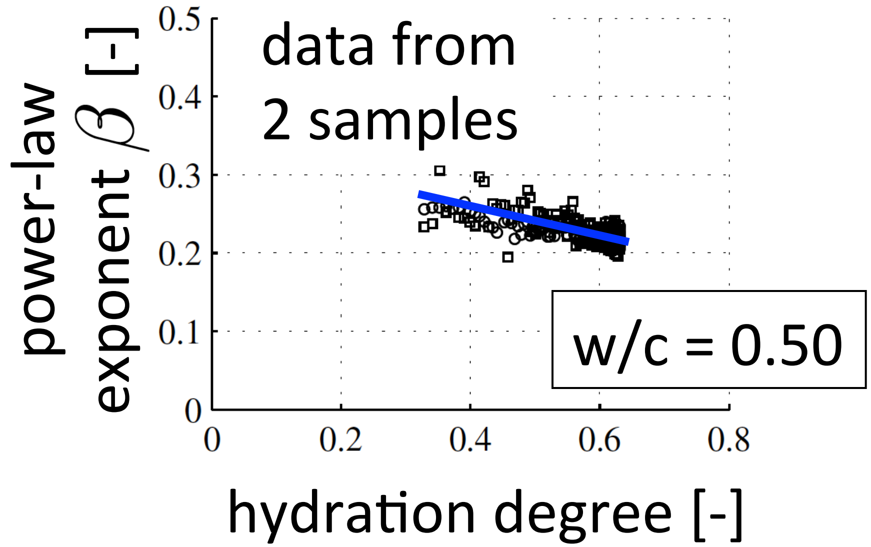
- Elastic modulus increases *linearly* with hydration degree



Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

Evolutions of creep modulus E_c and of power-law exponent β :





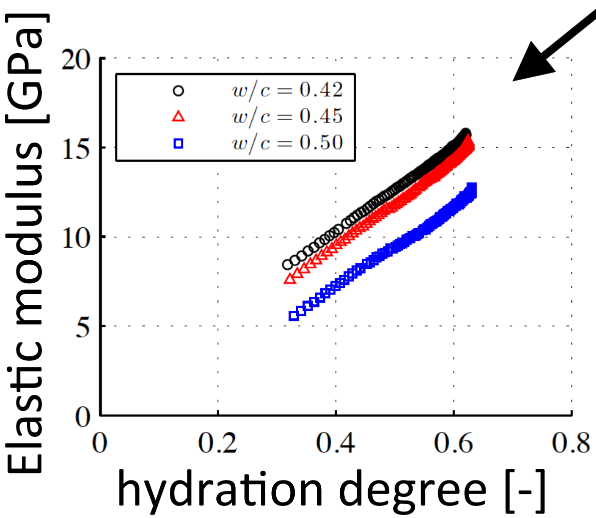
- Creep modulus increases *overlinearly* with hydration degree
- Power-law exponent decreases *linearly* with hydration degree

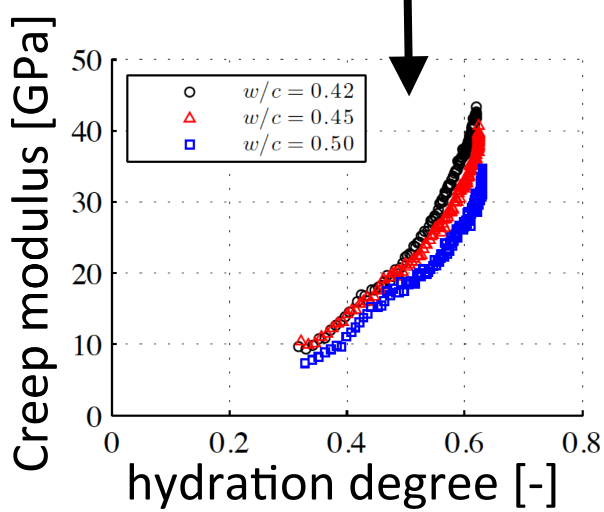
Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

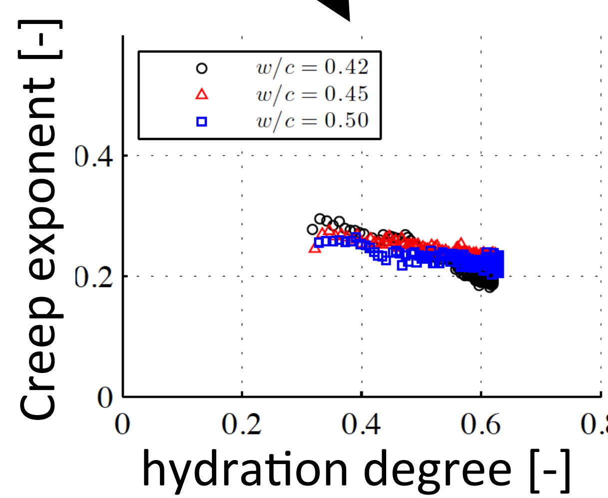
Summary of testing activities:

- 500 non-aging creep functions of cement pastes with w/c = { 0.42 , 0.45 , 0.50 }

$$J_{cp}(\xi, t-\tau) = \frac{1}{E_{cp}(\xi)} + \frac{1}{E_{c,cp}(\xi)} \left(\frac{t-\tau}{t_{ref}} \right)^{\beta_{cp}(\xi)}$$

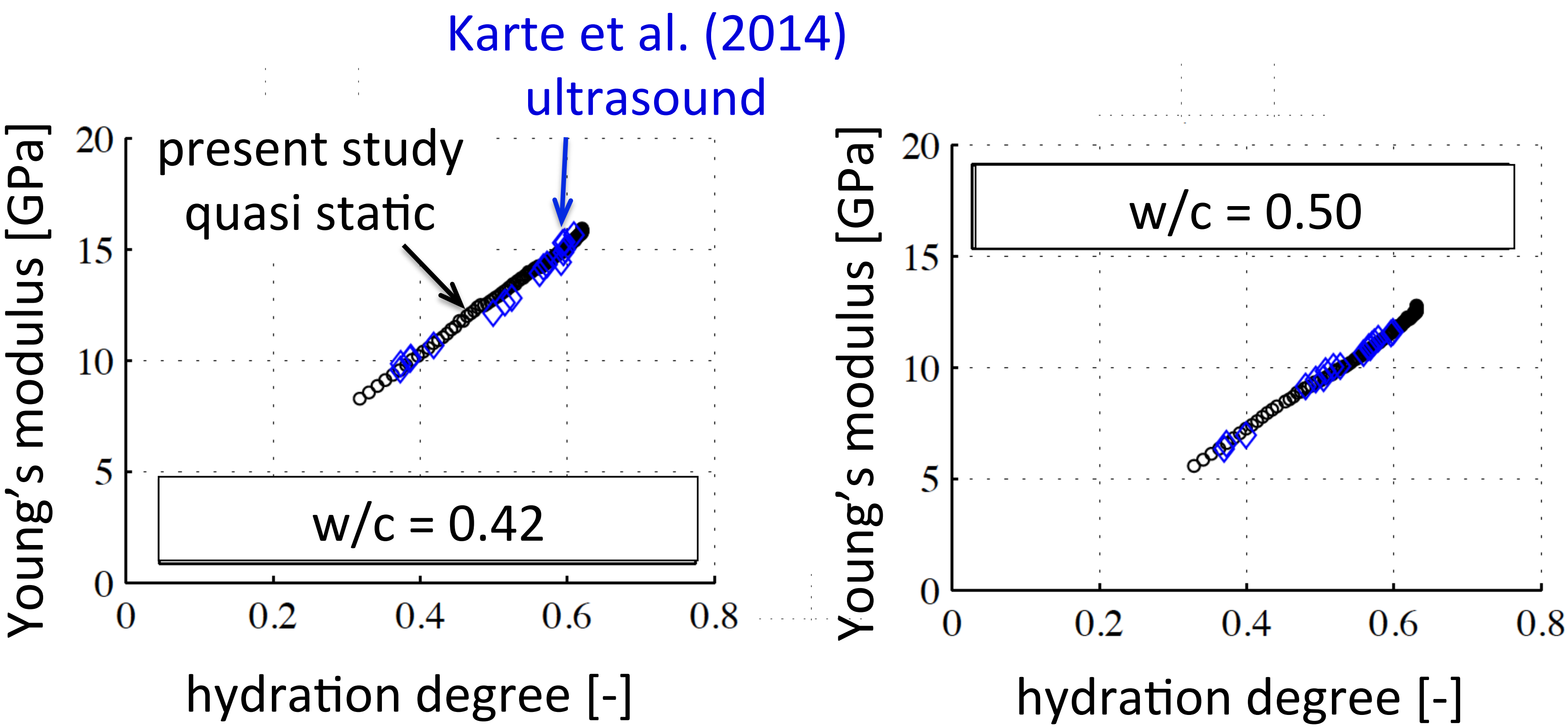






Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

Quasi-static Young's moduli vs. dynamic Young's moduli



Conclusion: Quasi-static Young's moduli = dynamic Young's moduli

Lit.: [Irfan-ul-Hassan et al., Cement & Concrete Research, 82: 36-49, 2016]

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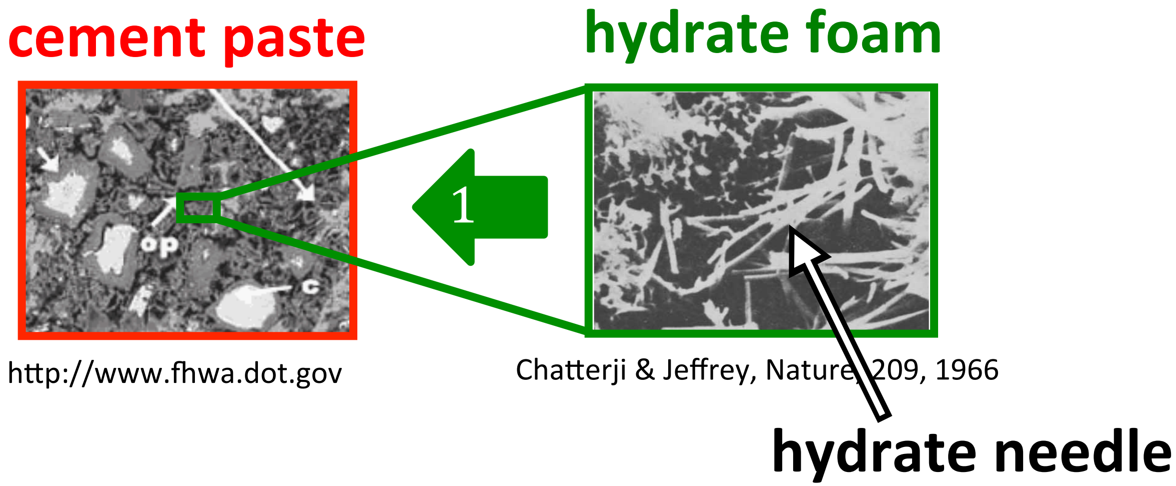
Multiscale exploitation of creep test data

1. Identify creep of micron-sized hydrate needles



Creep homogenization: Correspondence principle

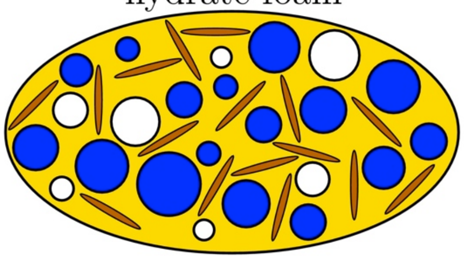
a. Laplace-Carson transformation of time-dependent problem
b. Quasi-elastic upscaling using continuum micromechanics, i.e. Eshelby-based homogenization schemes
c. Back-transformation to time space

Literature: [Königsberger et al., Journal of Engineering Mechanics, accepted]

1. Identification of creep of hydrate needles

- Phase volume fractions: Powers-Acker hydration model

hydrate foam

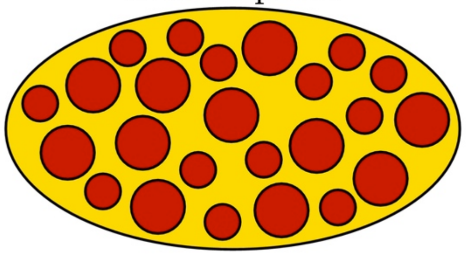


$$\tilde{f}_{hyd} = \frac{f_{hyd}}{1-f_{clin}} = \frac{43.15\,\xi}{20\,\xi + 63\,(w/c)}$$

$$\tilde{f}_{H_2O} = \frac{f_{H_2O}}{1-f_{clin}} = \frac{63\,(w/c) - 26.46\,\xi}{20\,\xi + 63\,(w/c)}$$

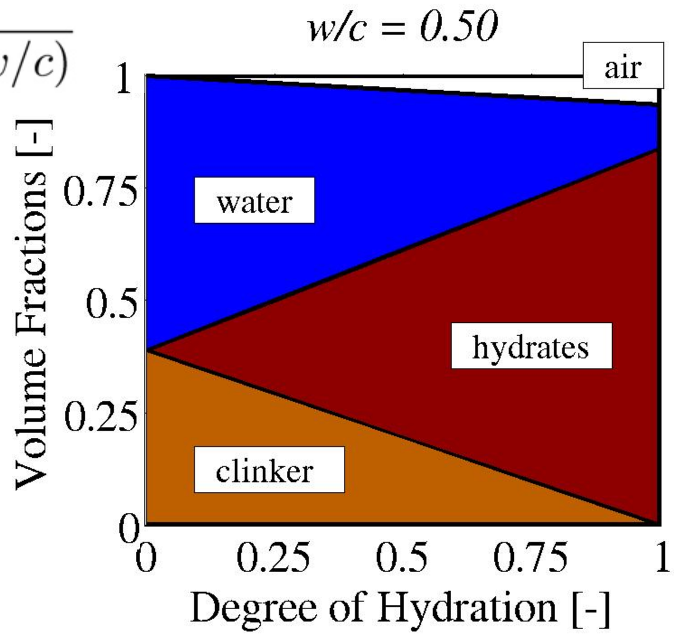
$$\tilde{f}_{air} = \frac{f_{air}}{1-f_{clin}} = \frac{3.31\,\xi}{20\,\xi + 63\,(w/c)}$$

cement paste



$$f_{clin} = \frac{20\,(1-\xi)}{20 + 63\,(w/c)}$$

$$f_{hf} = \frac{20\,\xi + 63\,(w/c)}{20 + 63\,(w/c)}$$



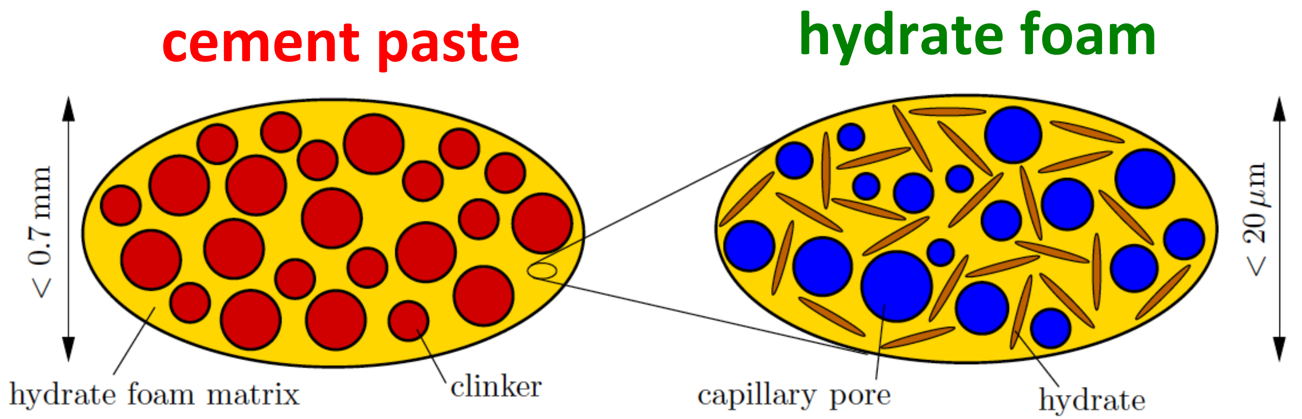
Powers, Brownyard, Res.Lab.Port.Cem.Ass.Bull, 22 101-992, 1948

Acker et al. in Concrete at Early Ages, ACI, 33-48, 1986

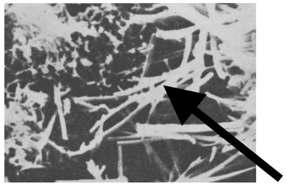
Literature: [Königsberger et al., Journal of Engineering Mechanics, accepted]

1. Identification of creep of hydrate needles

$$k_{cem} = 116.7\,\text{GPa}$$
$$\mu_{cem} = 53.80\,\text{GPa}$$



- Ansatz: **isochoric creep** of needle-shaped hydrates

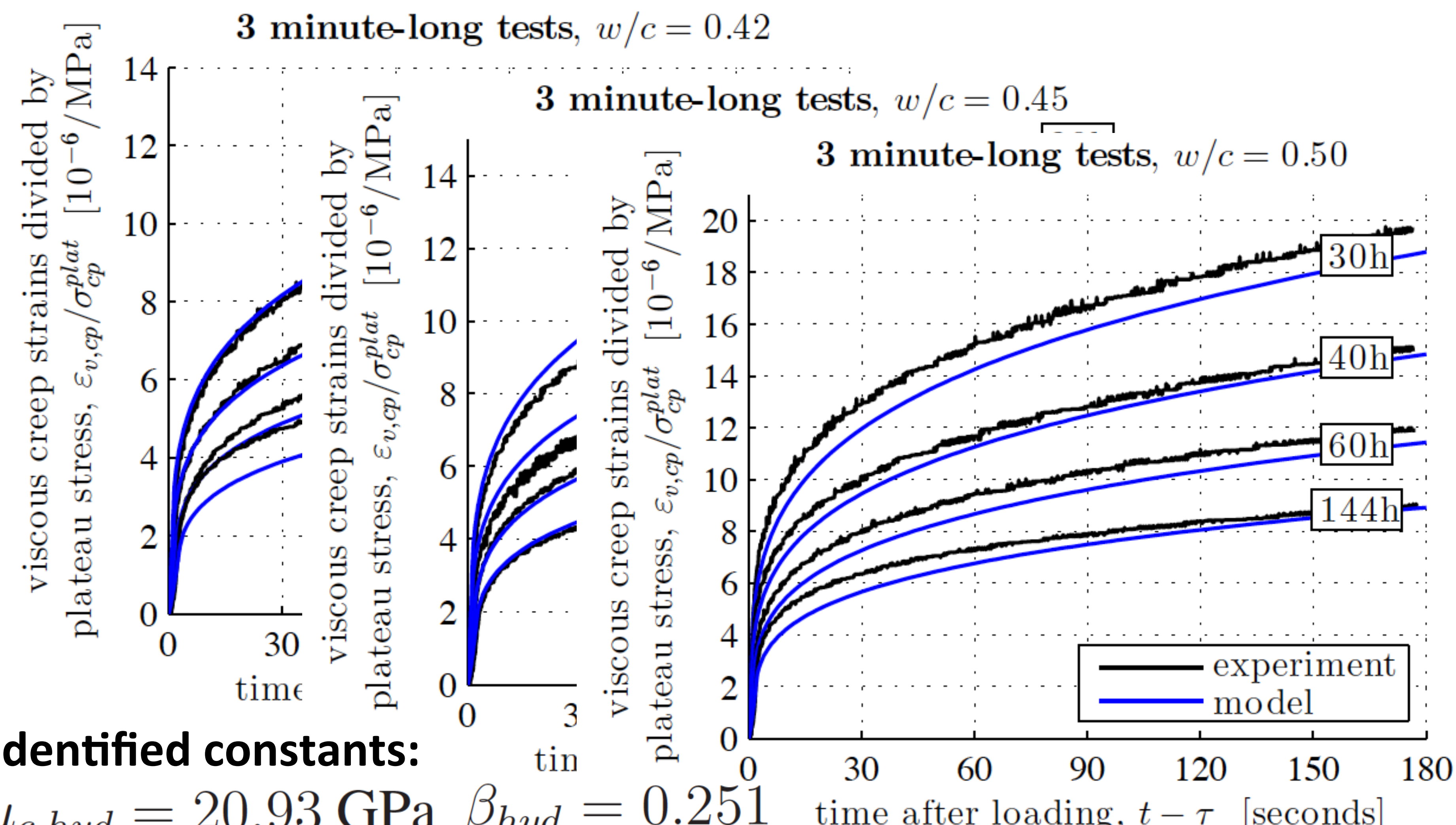


$$\mathbb{J}_{hyd}(t-\tau) = \frac{1}{3\,k_{hyd}}\mathbb{I}_{vol} + \frac{1}{2}\left[\frac{1}{\mu_{hyd}} + \frac{1}{\mu_{c,hyd}}\left(\frac{t-\tau}{t_{ref}}\right)^{\beta_{hyd}}\right]\mathbb{I}_{dev}$$

- Elastic properties: $\mu_{hyd} = 11.76\ \text{GPa}$ $k_{hyd} = 18.69\,\text{GPa}$
- Creep properties: $\mu_{c,hyd} = 20.93\,\text{GPa}$ $\beta_{hyd} = 0.251$

Literature: [Königsberger et al., Journal of Engineering Mechanics, accepted]

Experimental data vs. homogenization results: creep of cement paste



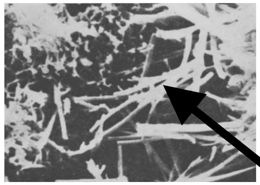
Identified constants:

$$\mu_{c,hyd} = 20.93\ \text{GPa} \quad \beta_{hyd} = 0.251$$

Literature: [Königsberger et al., Journal of Engineering Mechanics, accepted]

Is hydrate creep with constant Poisson's ratio more realistic ?

- Alternative ansatz for hydrate creep function


hydrate needle

$$\mathbb{J}_{hyd}(t-\tau) = \left[\frac{1-2\,\nu_{hyd}}{E_{hyd}} + \frac{1-2\,\nu_{hyd}}{E_{c,hyd}}\left(\frac{t-\tau}{t_{ref}}\right)^{\beta_{hyd}}\right]\mathbb{I}_{vol} + \left[\frac{1+\nu_{hyd}}{E_{hyd}} + \frac{1+\nu_{hyd}}{E_{c,hyd}}\left(\frac{t-\tau}{t_{ref}}\right)^{\beta_{hyd}}\right]\mathbb{I}_{dev}$$

- Identification results: isochoric creep vs. constant Poisson's ratio

assumption	$E_{c,hyd}$ [GPa]	β_{hyd}	ϵ [10^{-6}/MPa]
isochoric	62.8[a]	0.251	0.768
constant Poisson's ratio	62.4	0.250	0.765

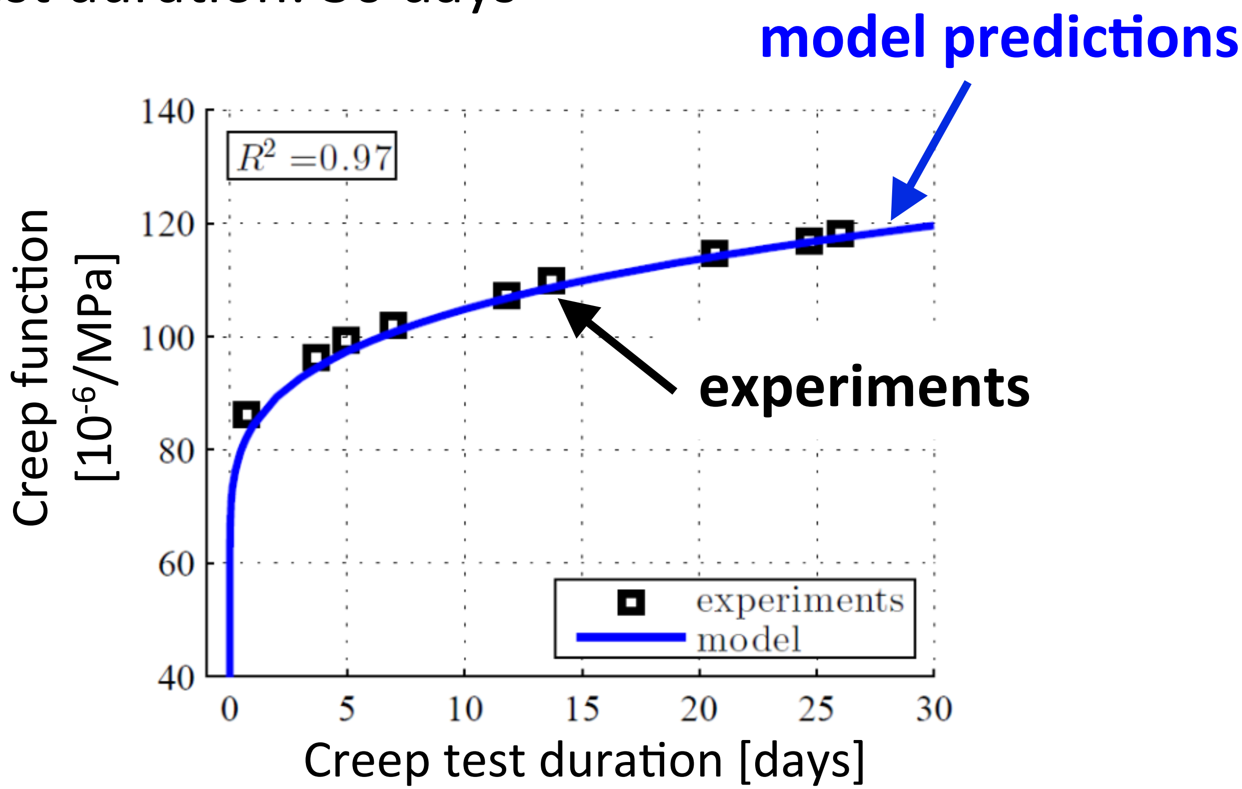
[a] $E_{c,hyd} = 3\,\mu_{c,hyd}$

- Isochoric creep *and* creep with constant Poisson's ratio deliver the same creep modulus and the same creep exponent

Literature: [Königsberger et al., Journal of Engineering Mechanics, accepted]

Model validation based on tests by Tamtsia and Beaudoin (2000)

- Composition of cement paste: w/c = 0.50
- Curing: 30 years under water -> hydration degree = 87 %
- Creep test duration: 30 days



Literature: [Königsberger et al., Journal of Engineering Mechanics, accepted]

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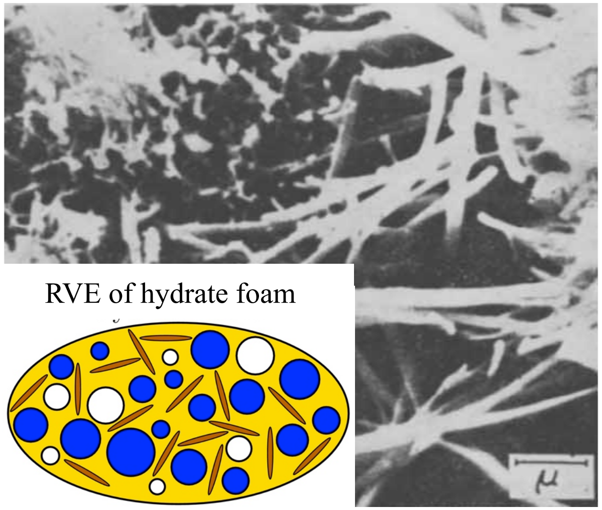
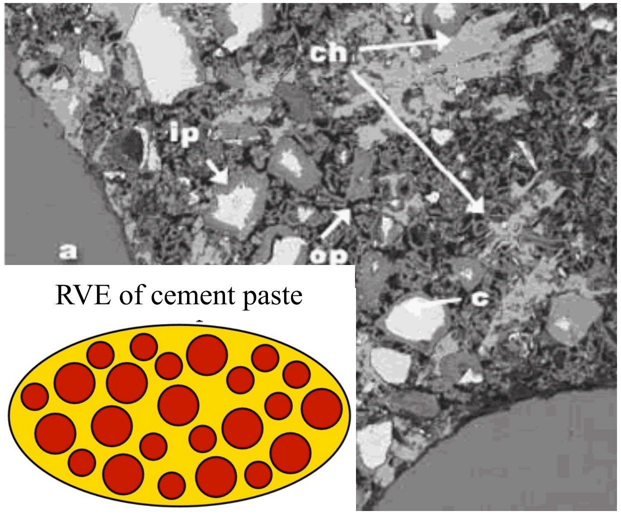
Multiscale modeling: continuum micromechanics

Material phases in scale-separated hierarchical organization



Chatterji and Jeffrey, Nature, 209, 1966



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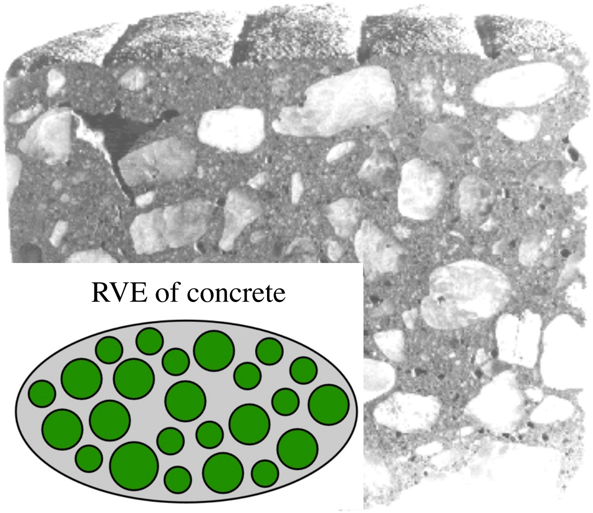

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Strength modeling idea:

- Cementitious materials are intact, if deviatoric stress peaks in hydrates < hydrate strength

Lit.: [Pichler & Hellmich, Cement and Concrete Research, 2011]

Multiscale modeling of strength of cement paste

- Cement paste is intact, if deviatoric stress peaks in hydrates < hydrate strength
- Microscopic hydrate failure = macroscopic material strength

$$\max_{\varphi,\vartheta} \overline{\overline{\sigma^{dev}_{hyd,\varphi,\vartheta}}} \leq \sigma^{dev}_{hyd,crit}$$

Scale transition to stress peaks:
via 2nd order stress averages

$$\overline{\overline{\sigma^{dev}_{hyd,\varphi,\vartheta}}} = \sqrt{\frac{-\mu^2_{hyd}}{\varphi_{hyd,\varphi,\vartheta}}\, \boldsymbol{\Sigma} : \frac{\partial[\mathbb{C}^{hom}]^{-1}}{\partial \mu_{\varphi,\vartheta}} : \boldsymbol{\Sigma}}$$

macroscopic stress ($\boldsymbol{\Sigma}$)

Identification of hydrate strength

- Nanoindentation testing on low-density C-S-H:
  - cohesion c = 50 MPa
  - angle of internal friction φ = 12°

Constantinides and Ulm, MIT Report (2006)
Sarris and Constantinides, CCC (2013)

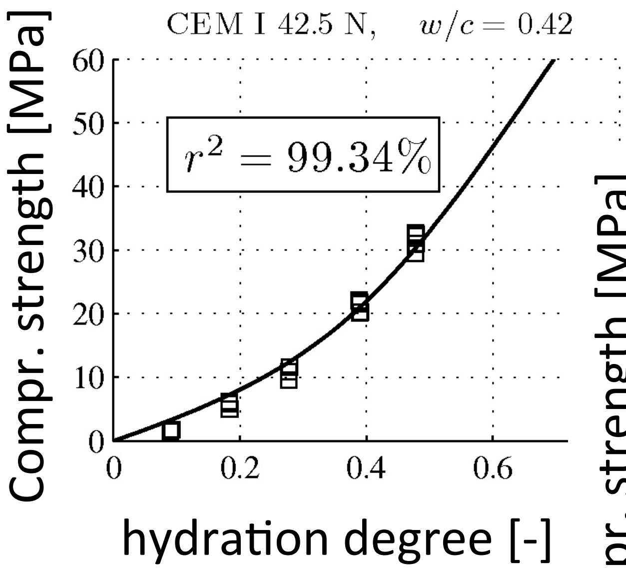
- This implies:
  - Uniaxial compressive strength of hydrates $f_{cu,hyd}$ = 123.5 MPa
  - von Mises-type deviatoric strength $\sigma^{dev}_{hyd,crit}$ = 71.3 MPa

Pichler et al. Concreep, (2013)

Lit.: [Pichler & Hellmich, Cement and Concrete Research, 2011]

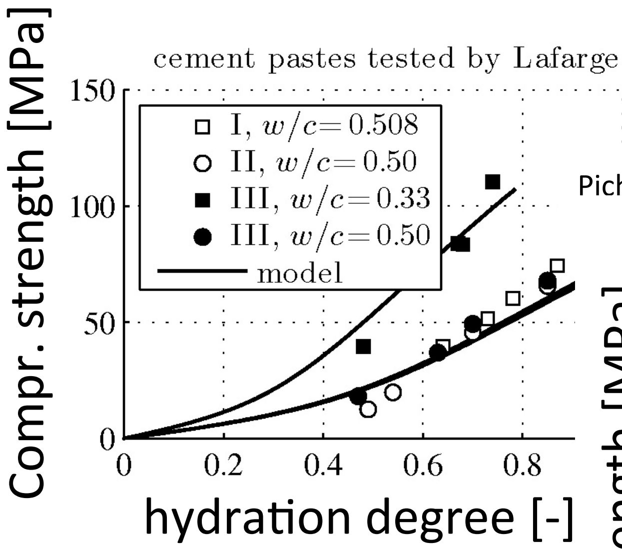
Model validation

Pichler et al., Concreep, (2013)



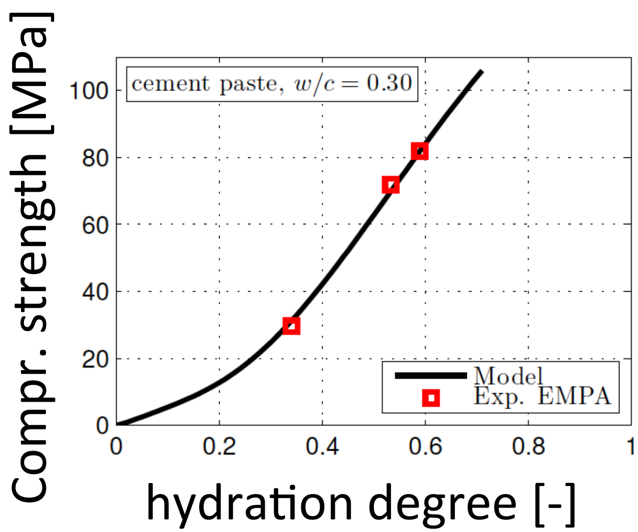
Model vs. tests performed in Vienna

Pichler et al., CCR (2013)



... at LCR

Pichler et al., CCR (2013)



... at EMPA

Wyrzykowski et al. COST action TU 1404

Same model performance for hydrate failure according to Mohr-Coulomb criterion

Literature: [Hlobil et al., Manuscript in preparation, 2016]