

SAOS as a predictive tool to assess the residual reactivity in recycled cementitious binders

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Schleibinger Geräte
Building Materials Testing Systems



OSTBAYERISCHE
TECHNISCHE HOCHSCHULE
REGENSBURG

Cement
Chemistry

& Particle
Interaction

Oscillatory
Rheology

Sustainability

Cement Chemistry & Particle Interaction

Origin of Cement Paste Cohesion

Cement composition: 55-65% of C_3S (15-25% of C_2S)

\nearrow
tricalcium silicate: model system for cement

C_3S (\approx cement) hydration:

1. C_3S grains dissolution and Calcium Silicate Hydrates (C-S-H) precipitation
2. Attractive electrostatic force development between C-S-H surfaces
3. C-S-H gel network formation (until cement setting)

$C=CaO$, $S=SiO_2$, $H=H_2O$

Taylor, H. F., Cement chemistry, 1997.

Jennings, CCR, 2000

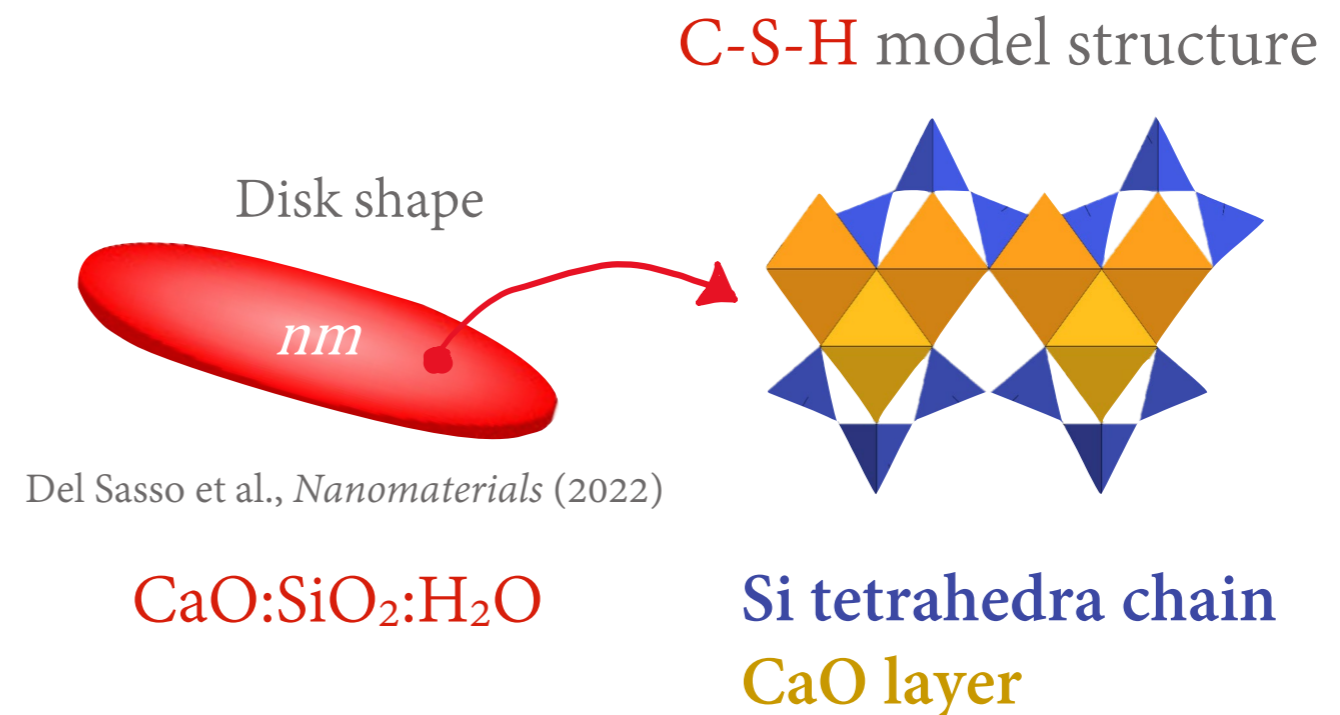
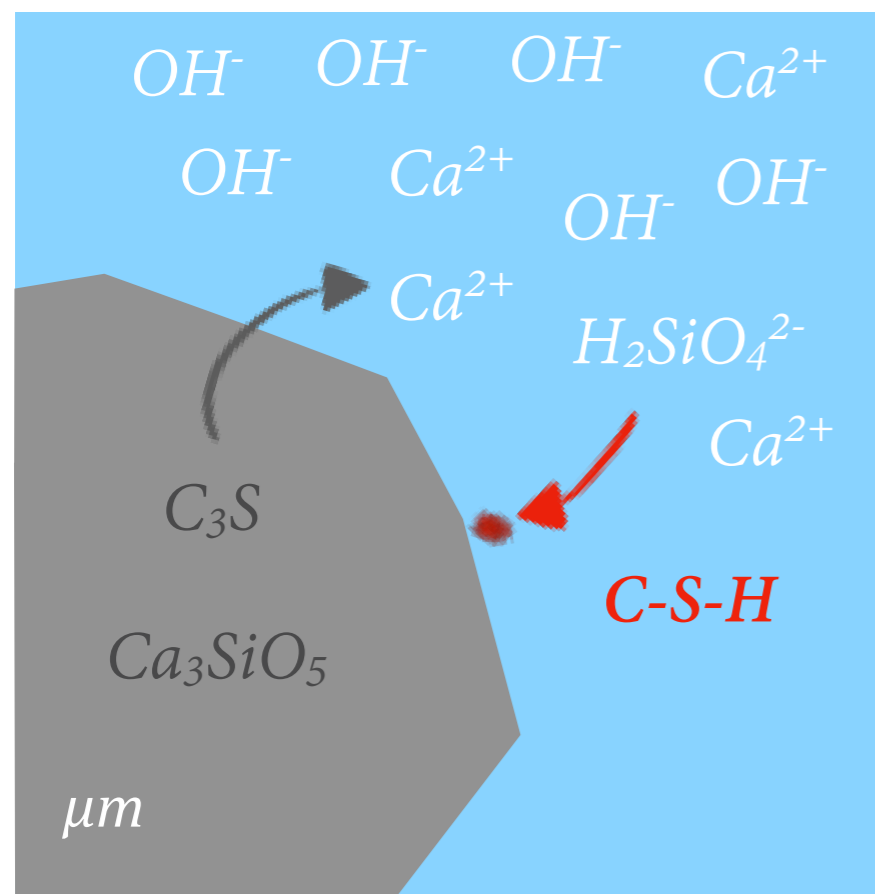
Jönsson et al. Langmuir, 2005

Thomas et al. CCR, 2006

Origin of Cement Paste Cohesion

Phase 1: C_3S dissolution & C-S-H precipitation

Tricalcium silicate Calcium Silicate Hydrate



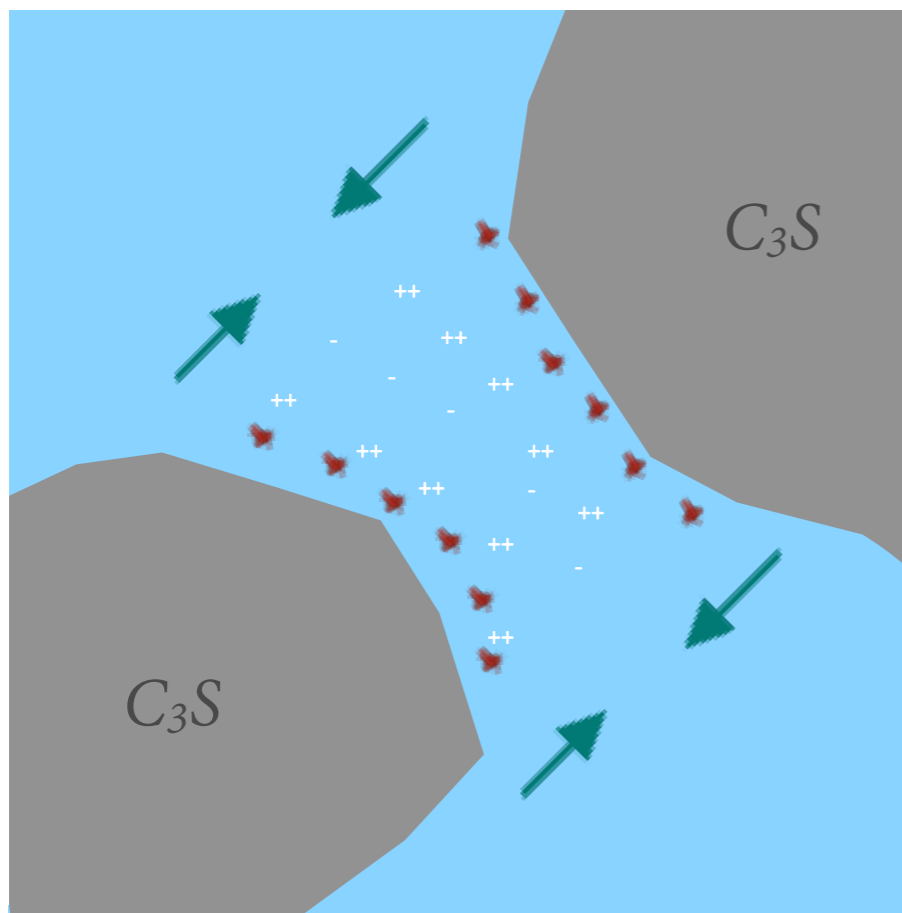
Immediately after contact with water the $[OH^-]$ increases resulting in a $pH > 12$

Origin of Cement Paste Cohesion

Phase 2: Attractive electrostatic force development between **C-S-H surfaces** ...and C_3S grains!

C-S-H model structure

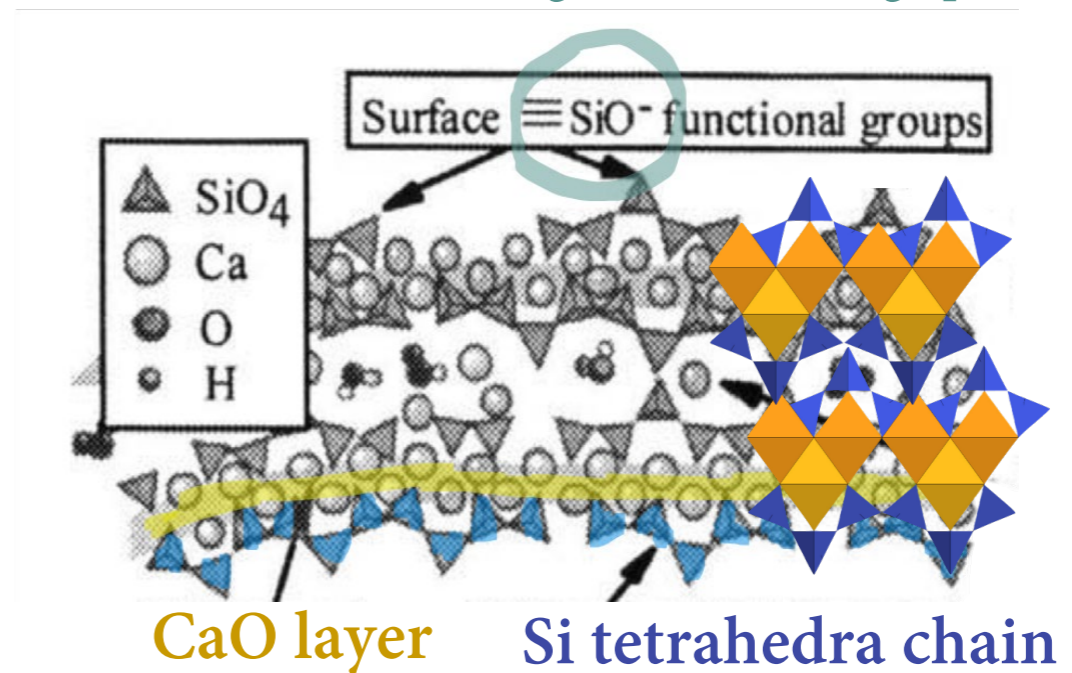
This negatively charged group forms after contact with water reacting with OH^- (at high pH)



Attractive force & cohesion
f(pore solution concentration)

Lesko et al., Ultramicroscopy, 2001

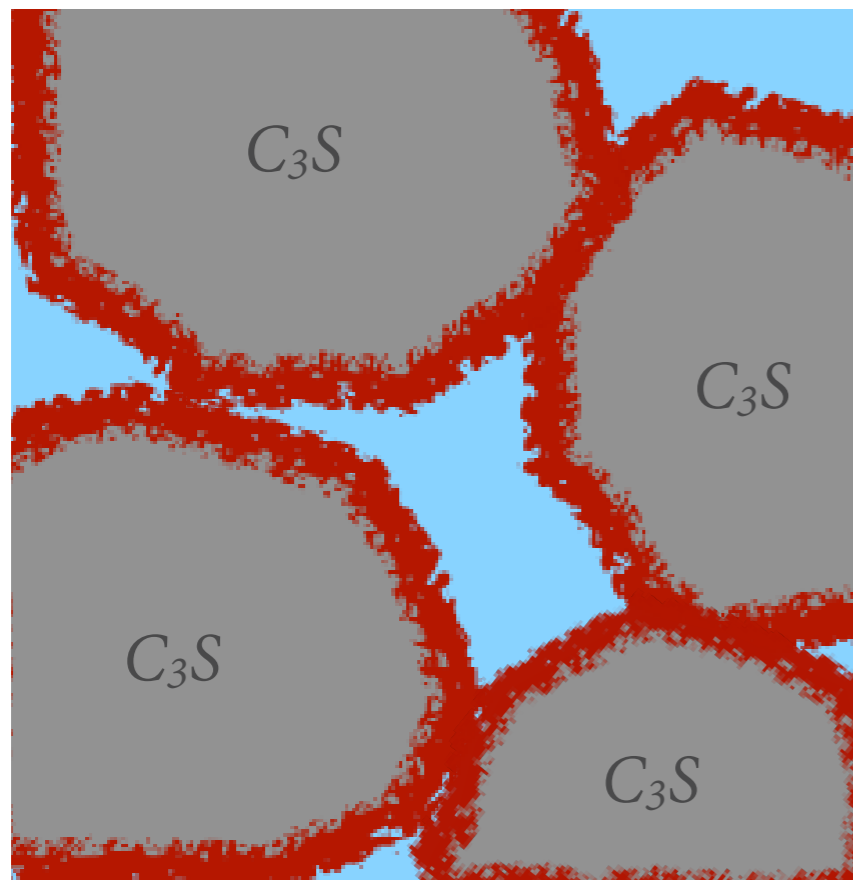
Jönsson et al. Langmuir, 2005



Iwaida et al. *Studies in Surface Science and Catalysis* (2001)

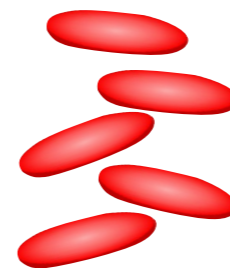
Origin of Cement Paste Cohesion

Phase 3: **C-S-H** gel network formation (until cement setting)



C-S-H gel:

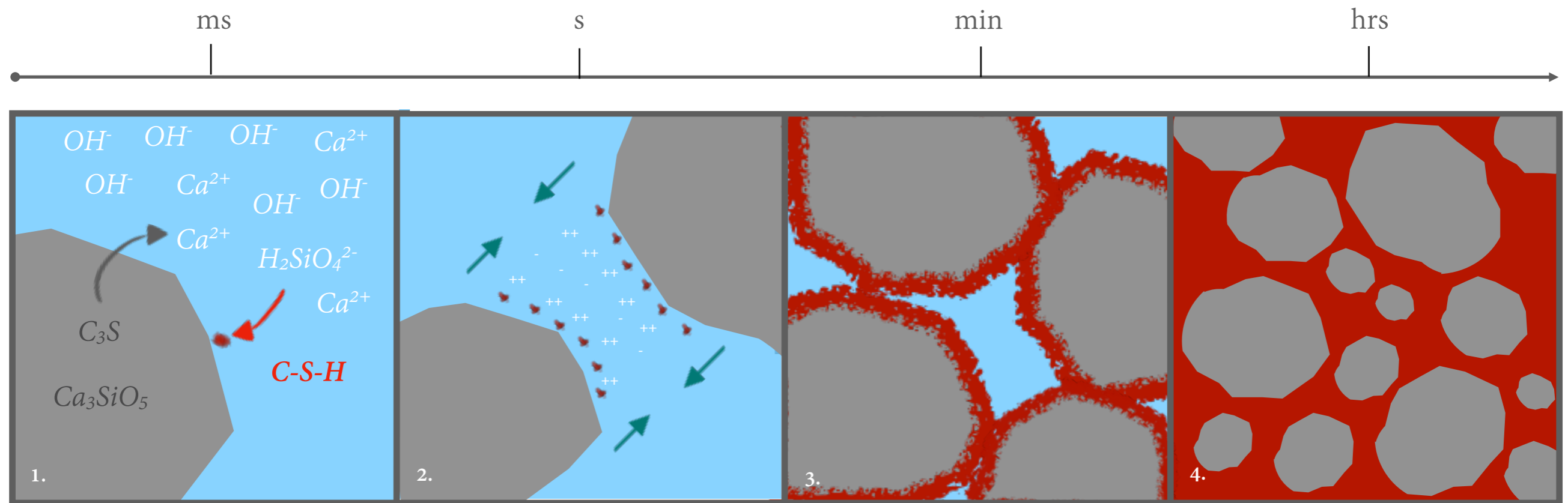
- nano crystalline/nano structure
- layered structure highly disordered at longer distances (μm)



Liberto et al. *Langmuir* (2022)
Del Sasso et al., *Nanomaterials* (2022)

Origin of Cement Paste Cohesion

Why does cement set?



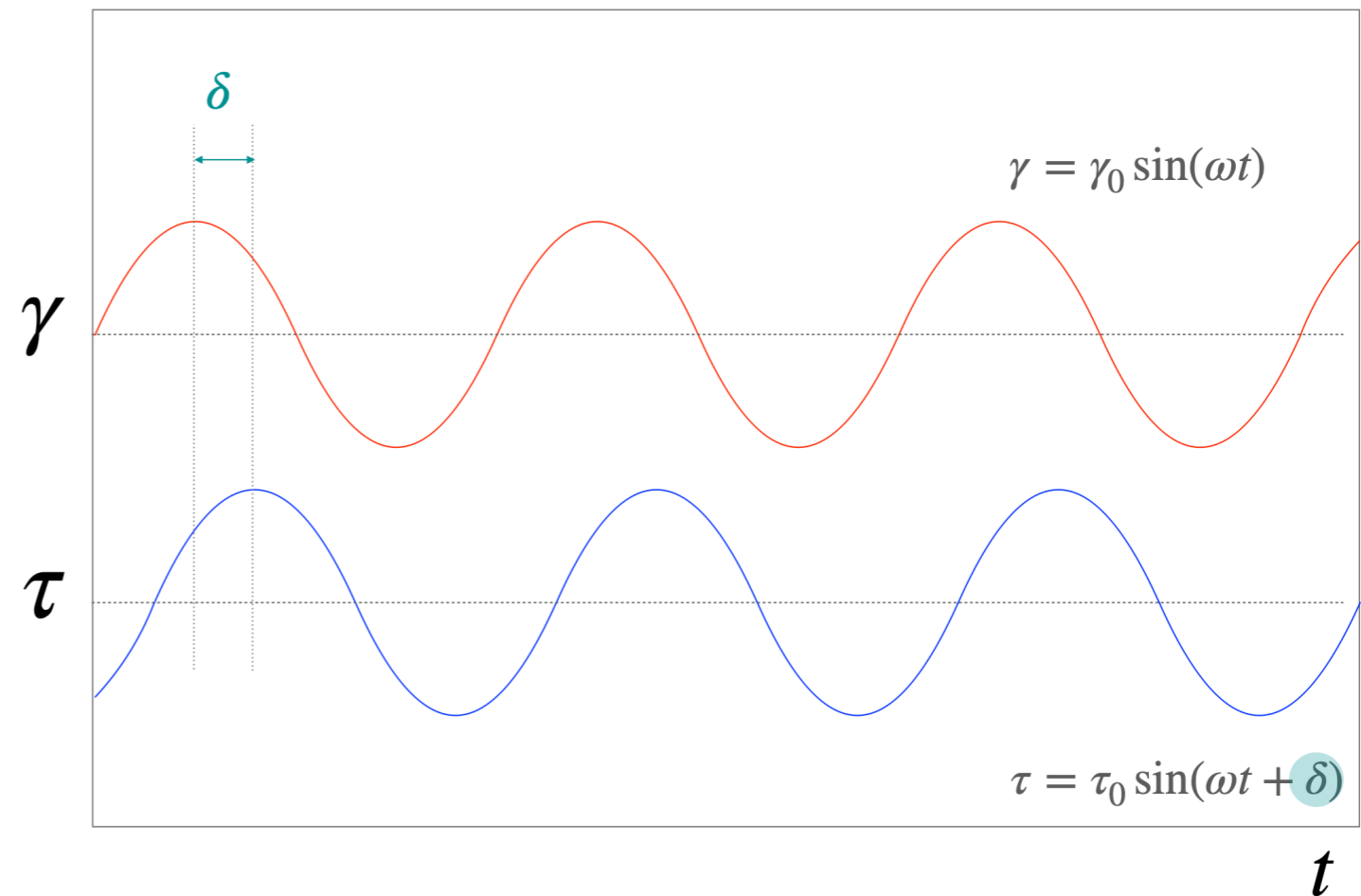
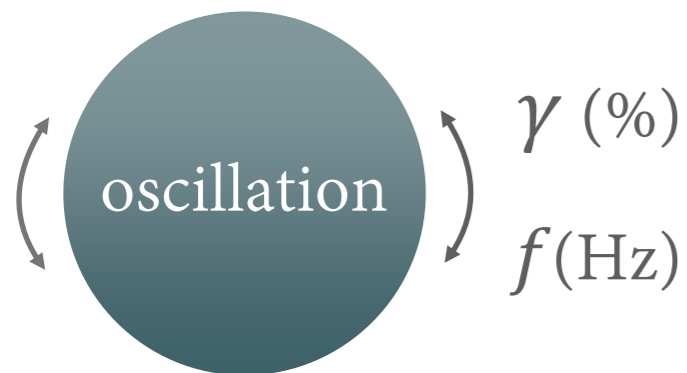
1. C_3S grain dissolution and **Calcium Silicate Hydrate (C-S-H)** precipitation
2. Attractive electrostatic force development between **C-S-H surfaces**
3. **C-S-H** gel network formation (until cement setting 4.)

C-S-H the "glue" of cement
After complete hydration
ca. 80% of the C_3S has been
dissolved

Cement
Chemistry
& Particle
Interaction

Oscillatory
Rheology

Oscillatory rheology



$$\delta = 0$$

purely elastic

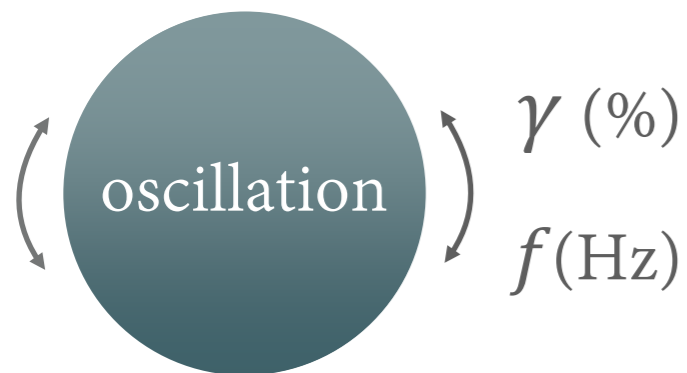
$$\delta = \pi/2$$

purely viscous

$$0 < \delta < \pi/2$$

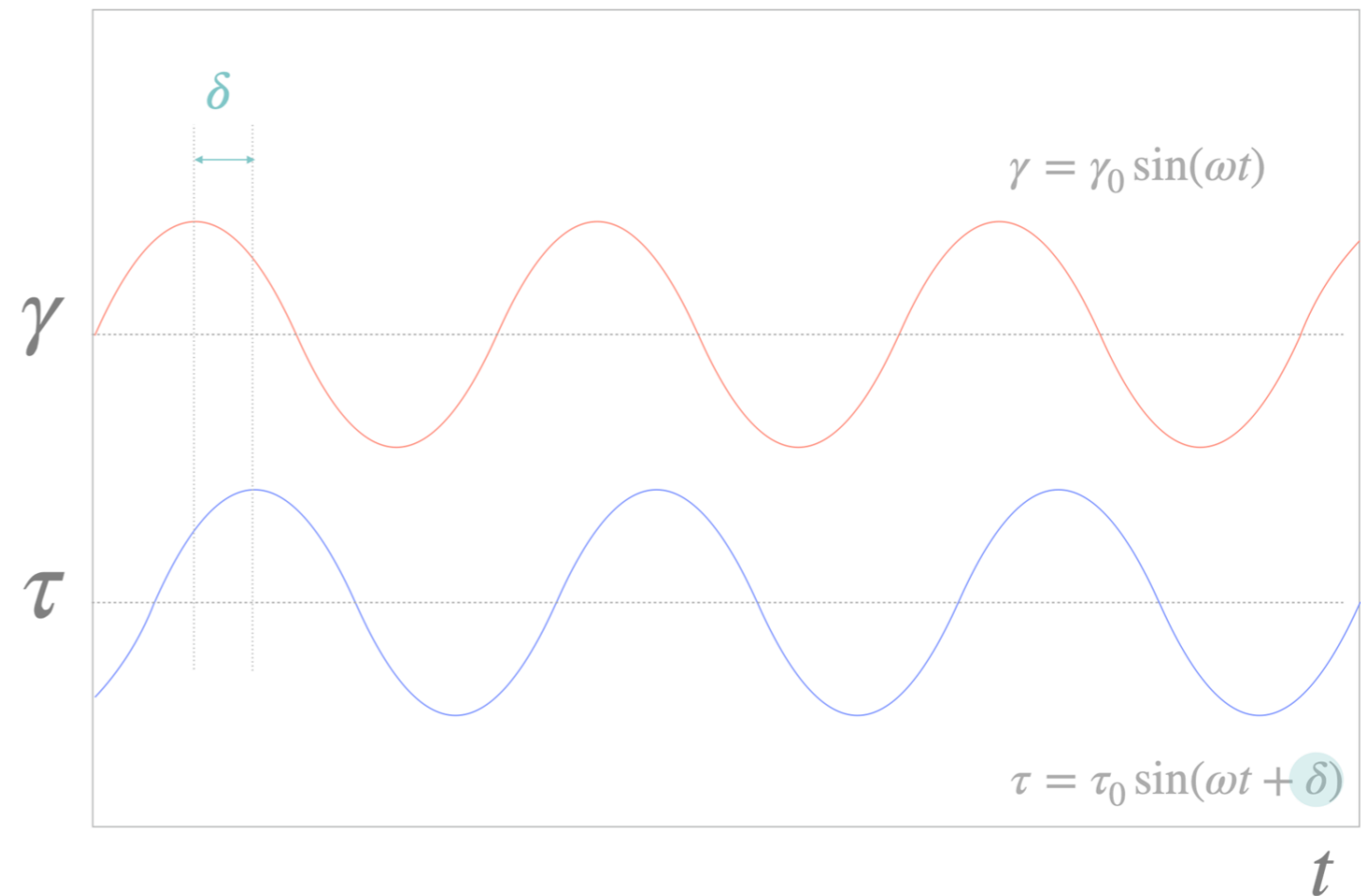
viscoelastic

Oscillatory rheology

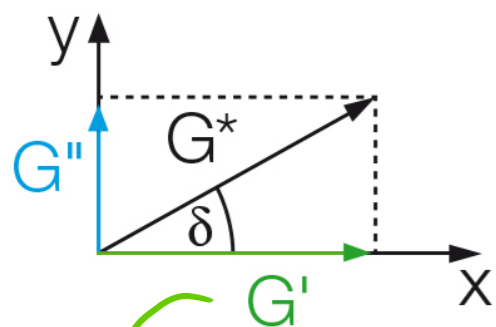


$$G^* = \tau^*(t) / \gamma^*(t)$$

$$G^* = G' + iG''$$



Viscous or loss modulus



Storage or elastic modulus

$$\delta = 0$$

purely elastic

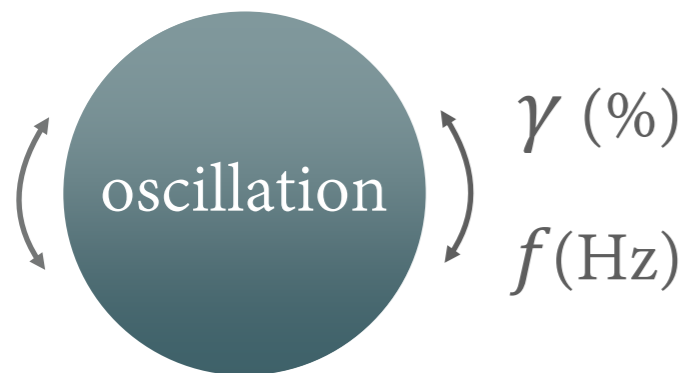
$$\delta = \pi/2$$

purely viscous

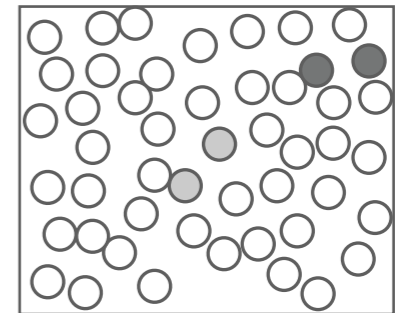
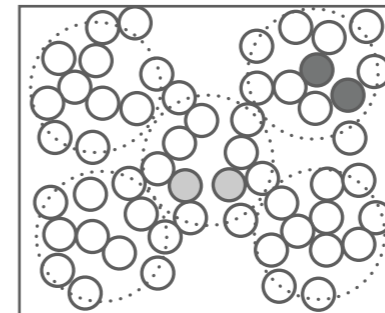
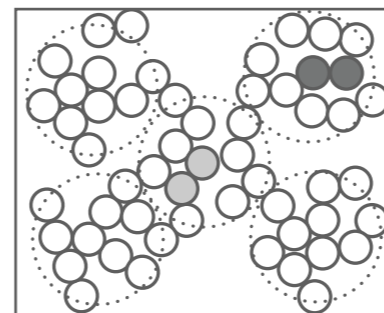
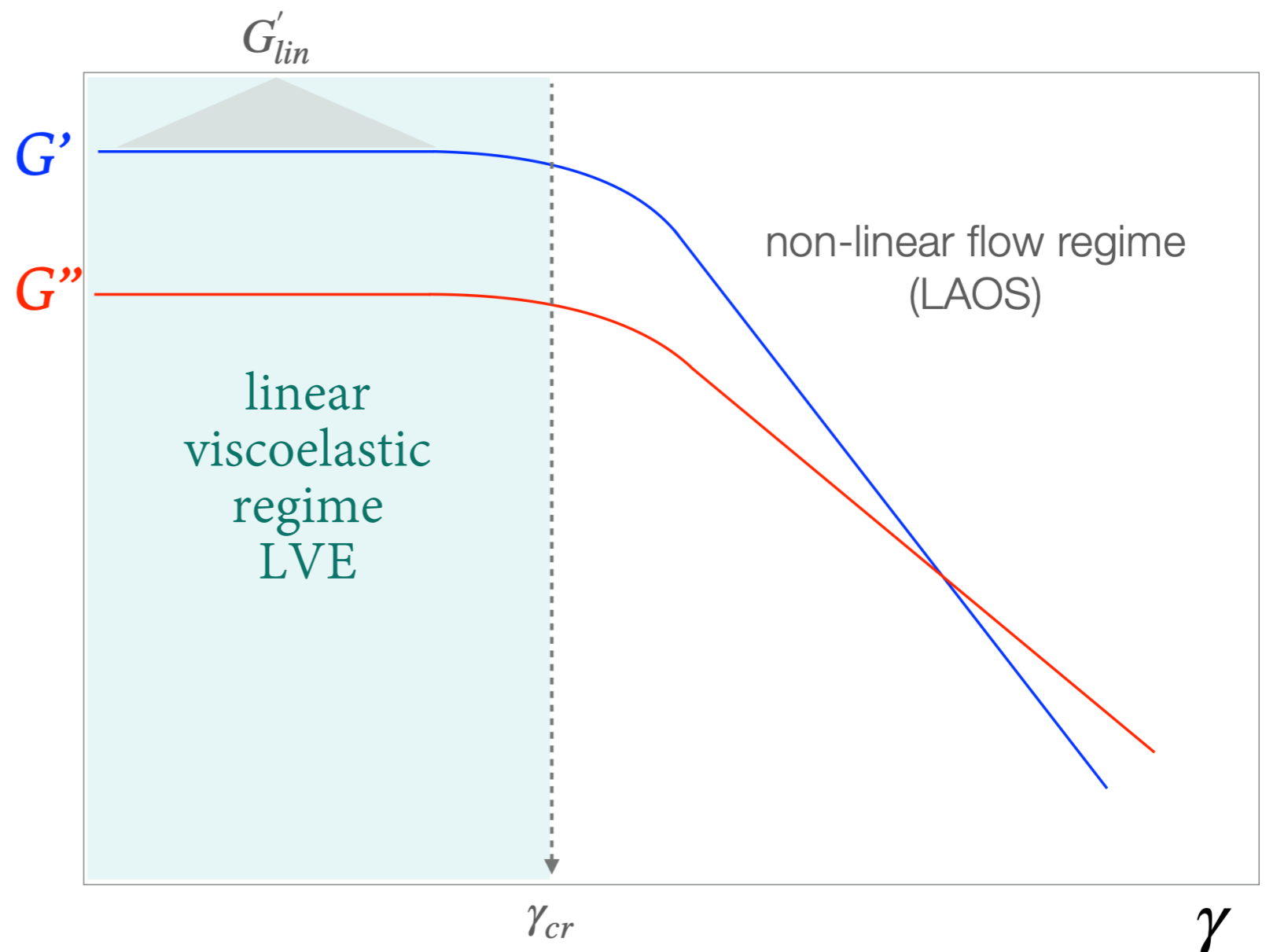
$$0 < \delta < \pi/2$$

viscoelastic

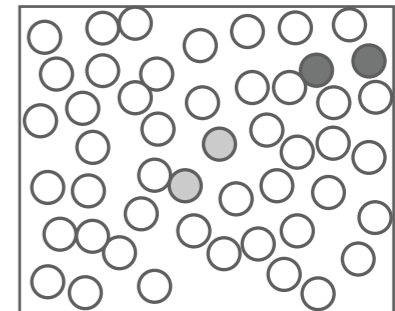
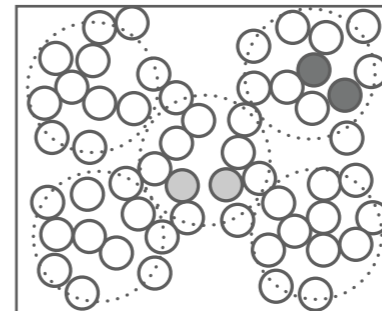
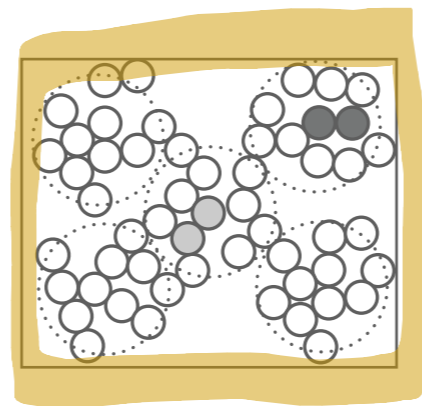
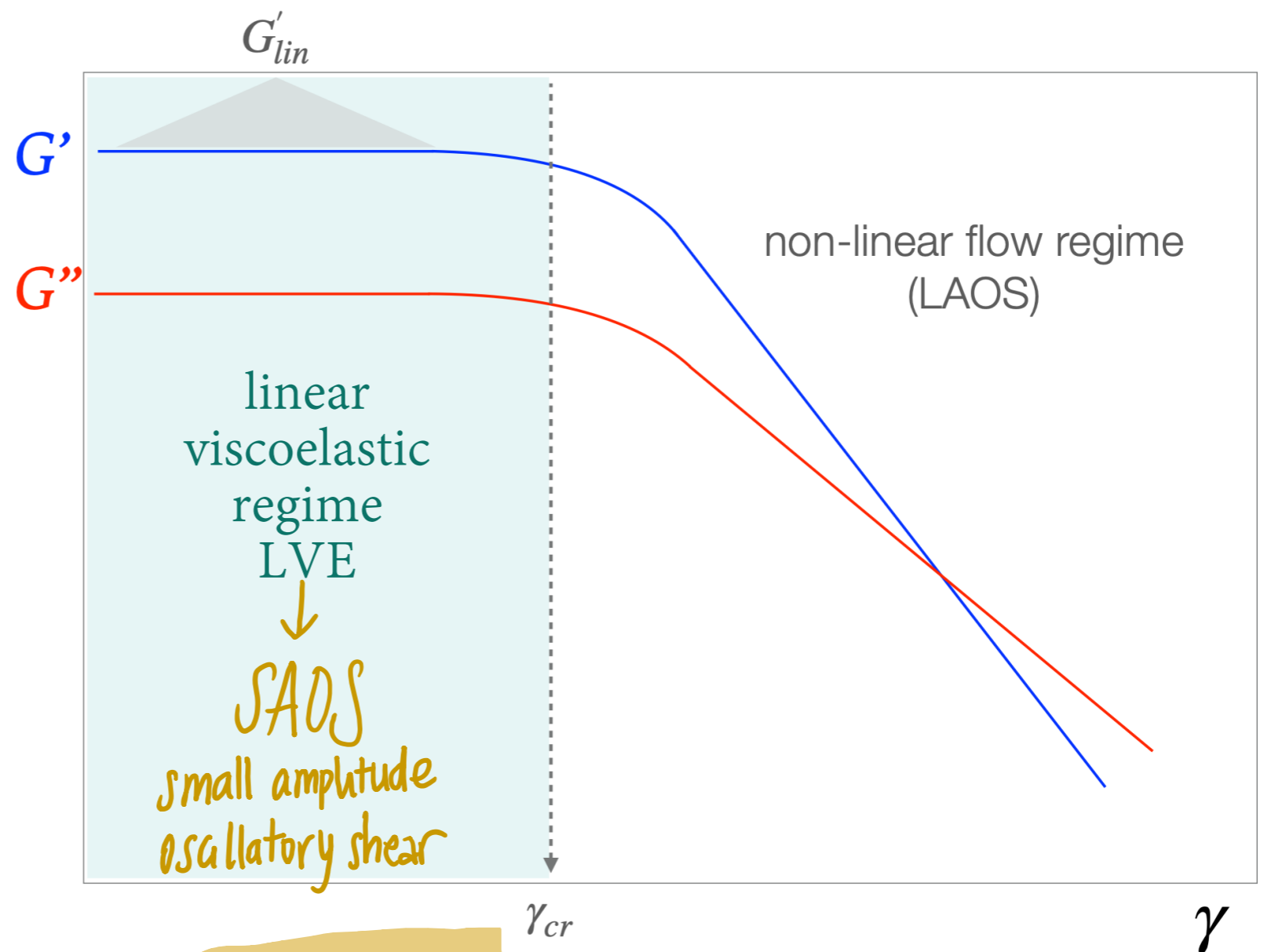
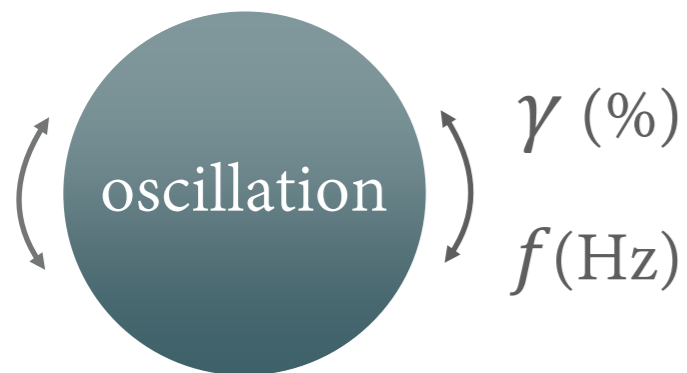
Oscillatory rheology



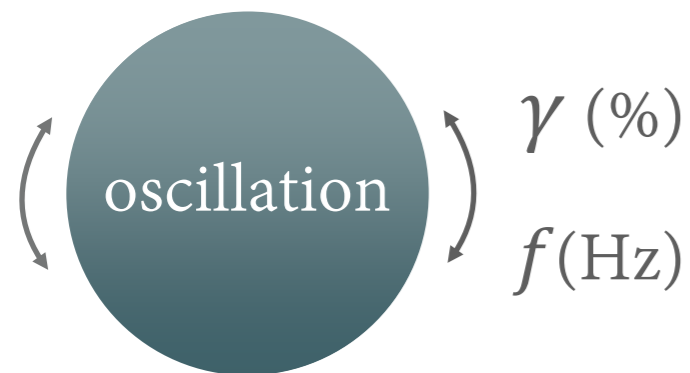
*Typical behavior for a
paste or gel*



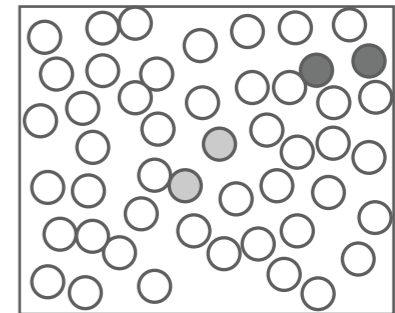
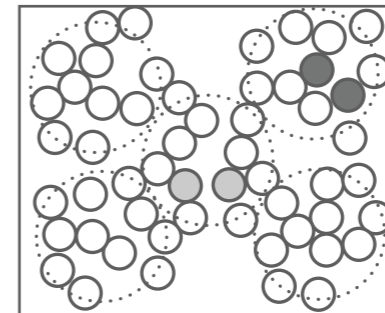
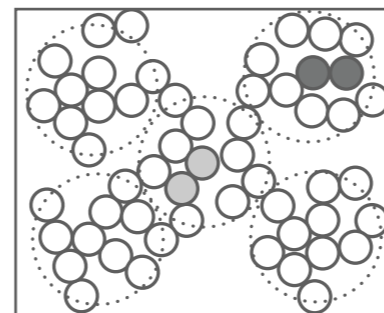
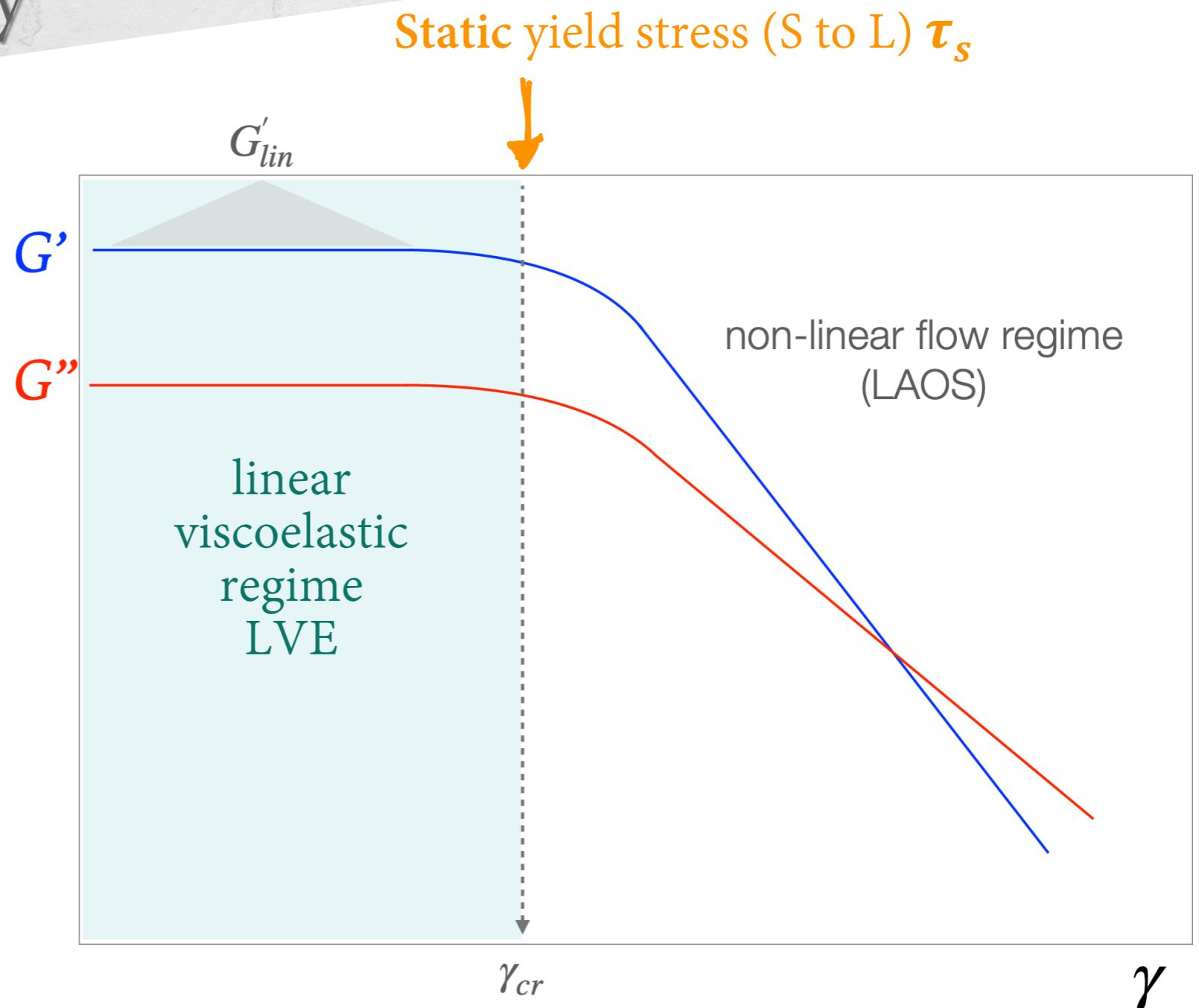
Oscillatory rheology



Oscillatory rheology

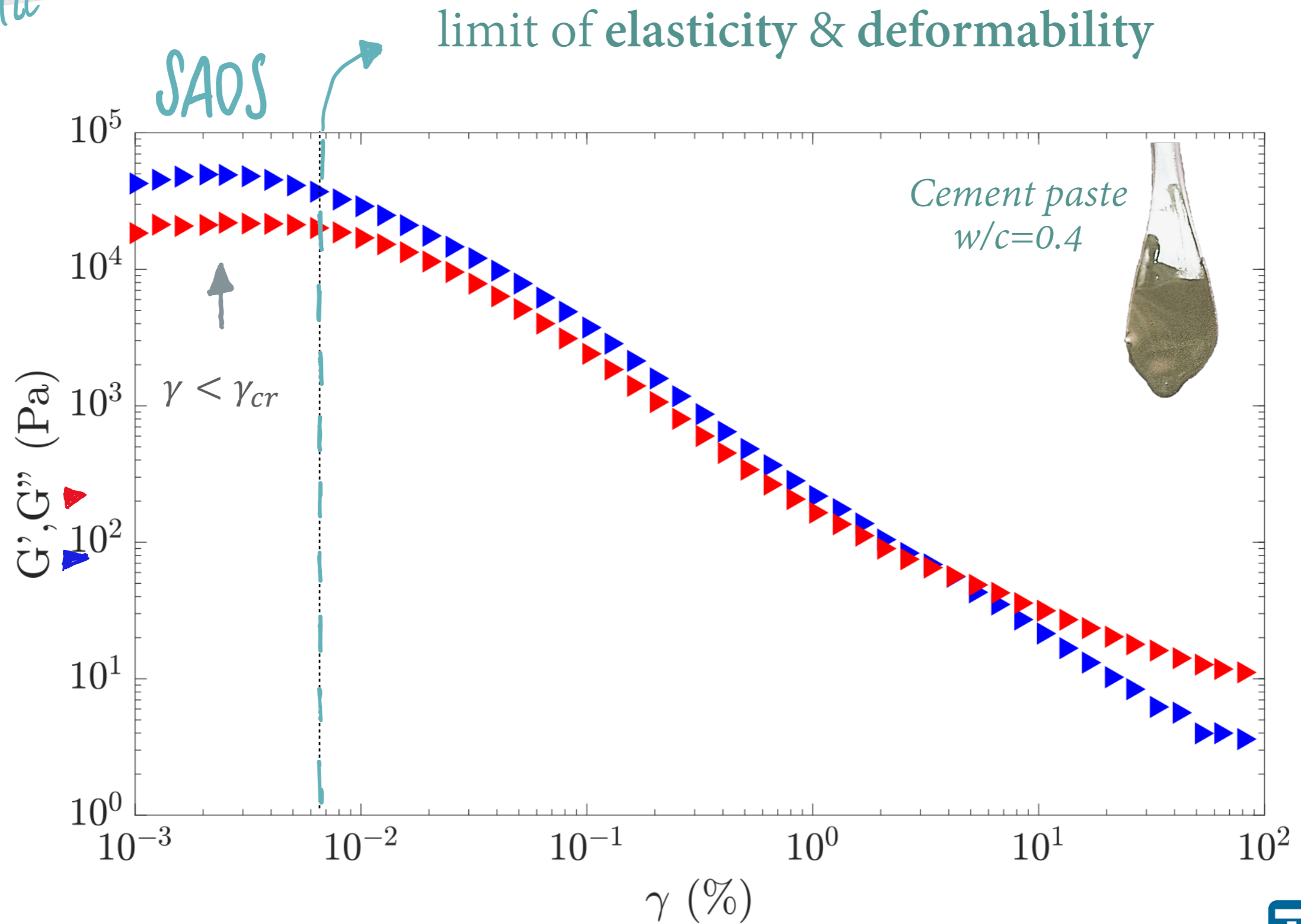


$$\tau_s = G'_{lin} \cdot \gamma_{cr}$$



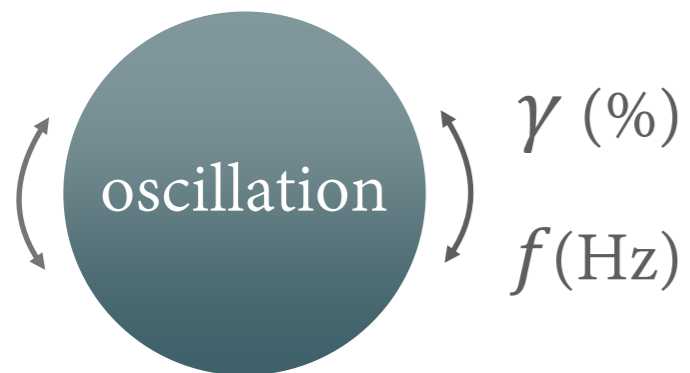
Oscillatory rheology

small



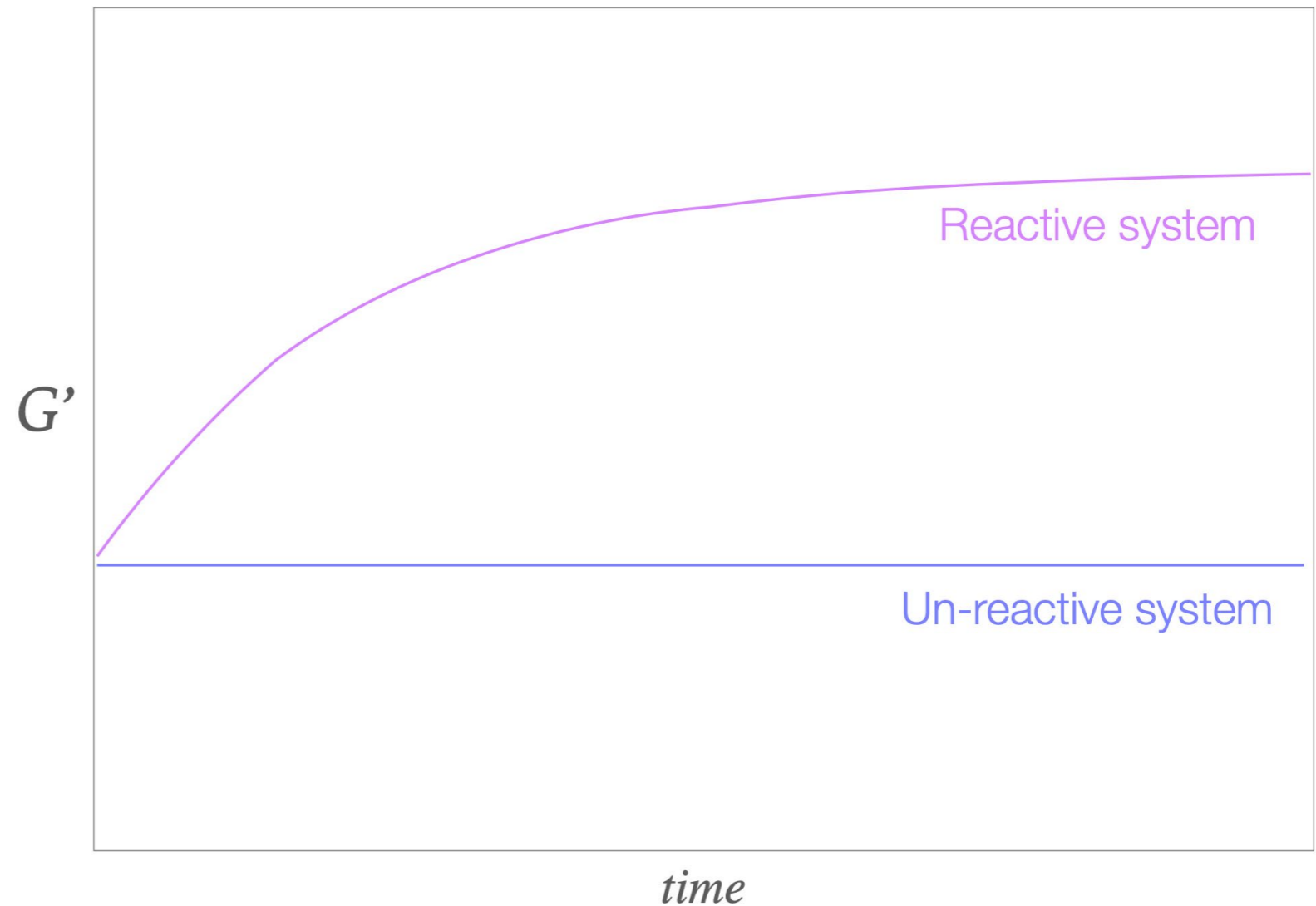
small oscillatory rheology

SAOS



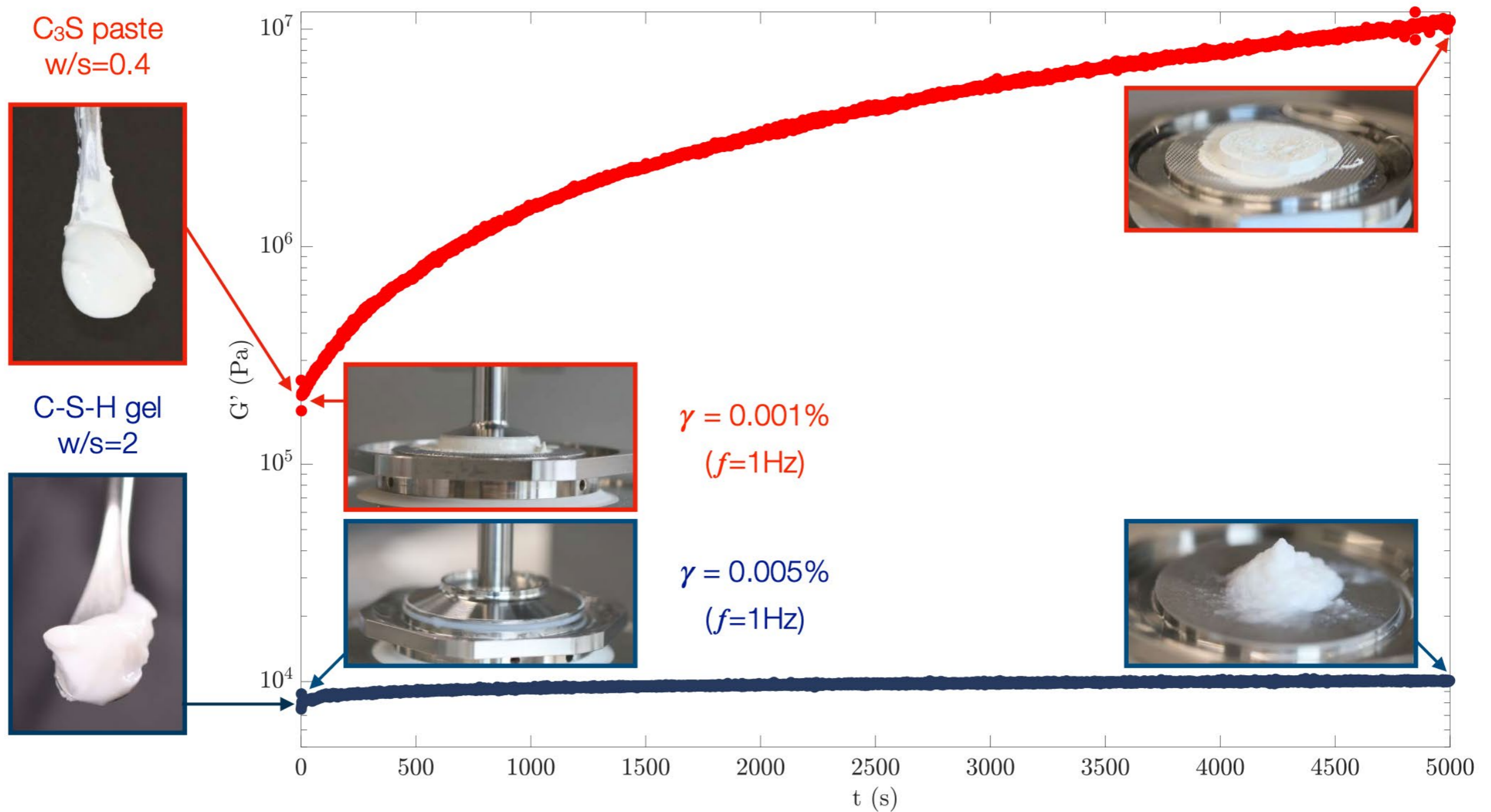
At a fix: $\gamma < \gamma_{cr}$

SAOS tracks the
cohesion (G')
development of a
paste at rest!



small oscillatory rheology

SAOS



Liberto et al. *Langmuir* (2022)

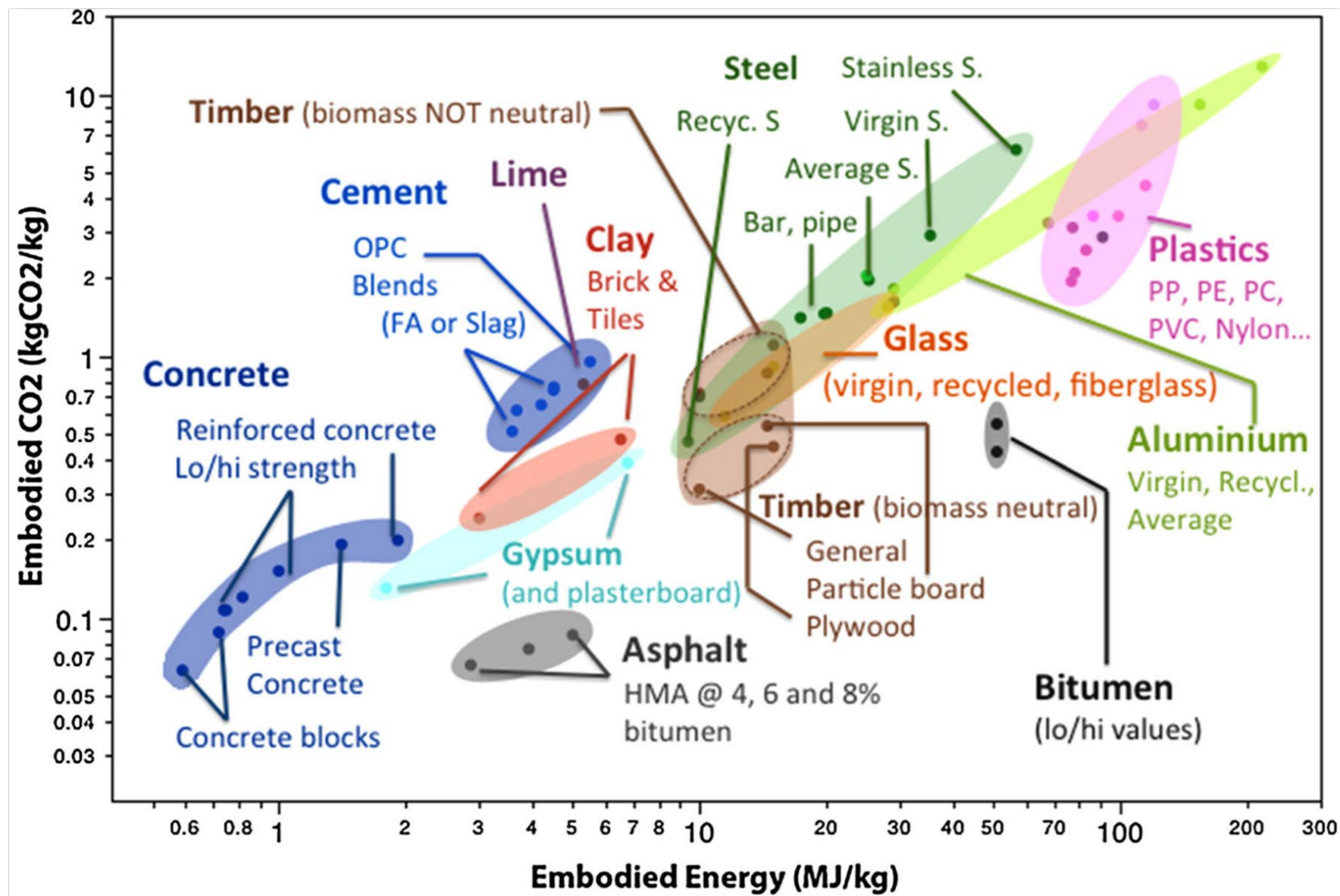
Cement
Chemistry.

& Particle
Interaction.

Rheology
SAOS

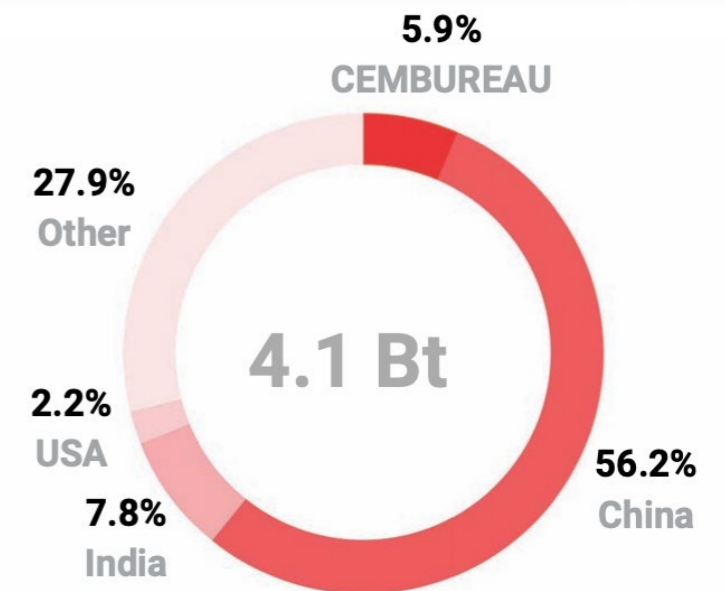
Sustainability

Cement sustainability



Barcelo et al. Mater Struct. (2014)

World cement production in 2019



Cembureau 2020 report

Recycled Concrete

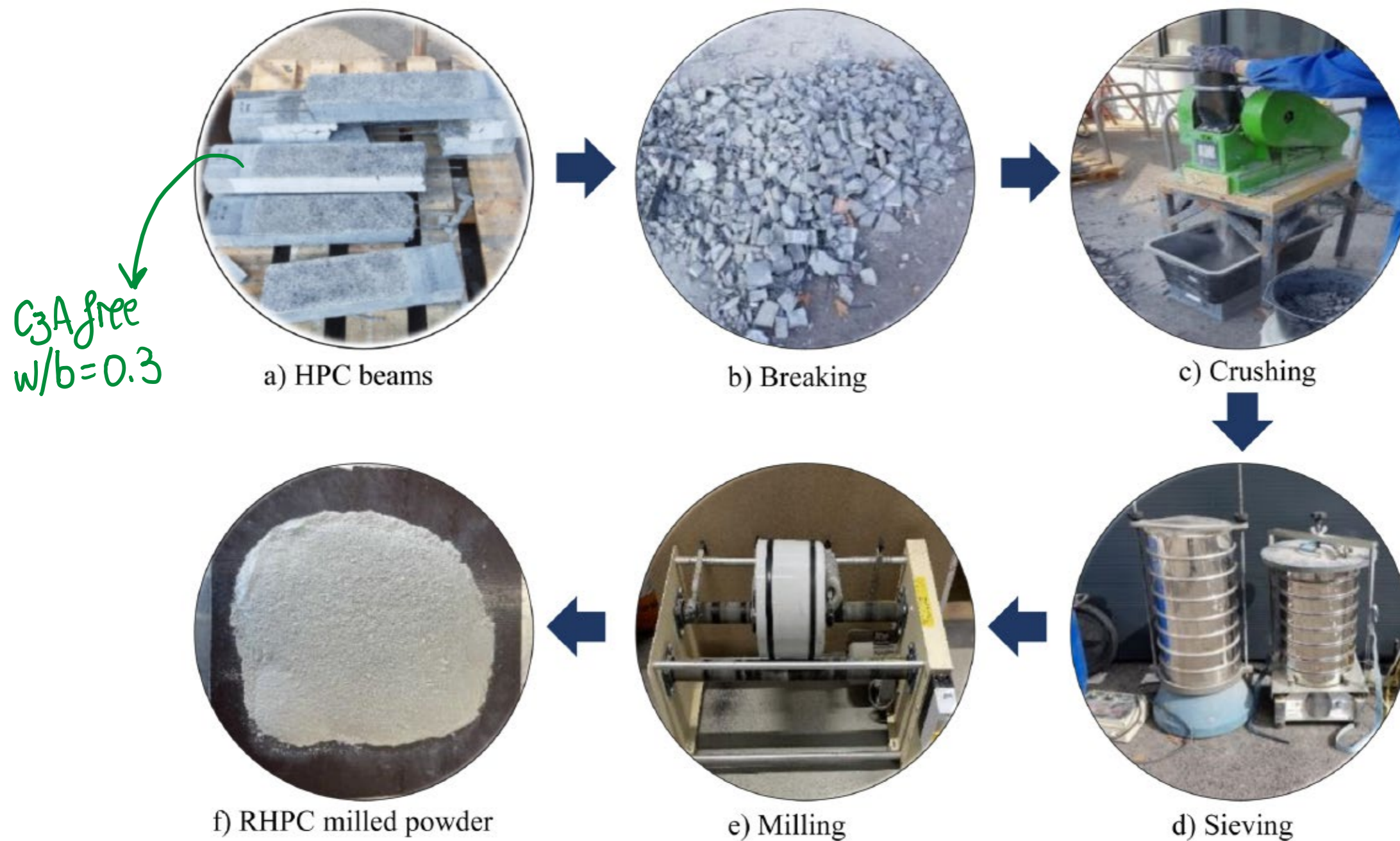


Fig. 1. Recycling process of high-performance concrete (HPC) to obtain mRHPC.

Daneshvar et al. *Case Studies in Construction Materials* (2022)

Recycled Concrete

PSD at different milling times:

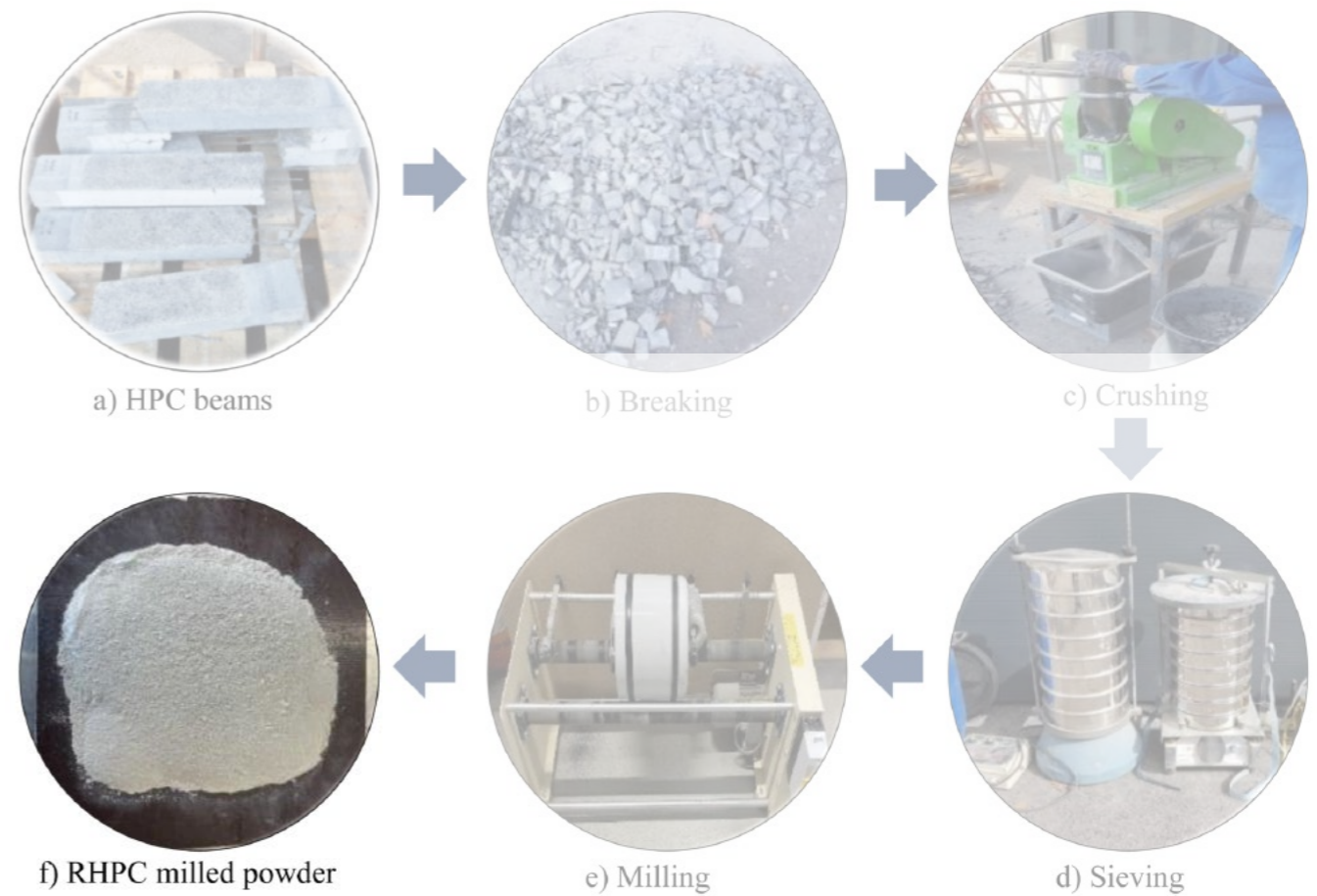
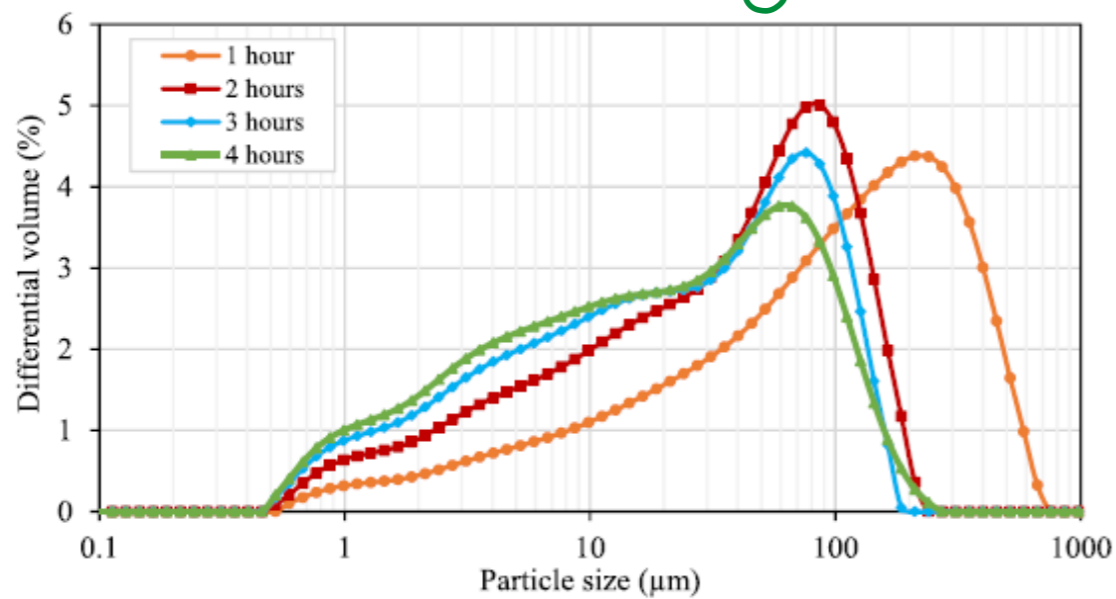


Fig. 1. Recycling process of high-performance concrete (HPC) to obtain mRHPC.

Recycled Concrete

PSD at different milling times:

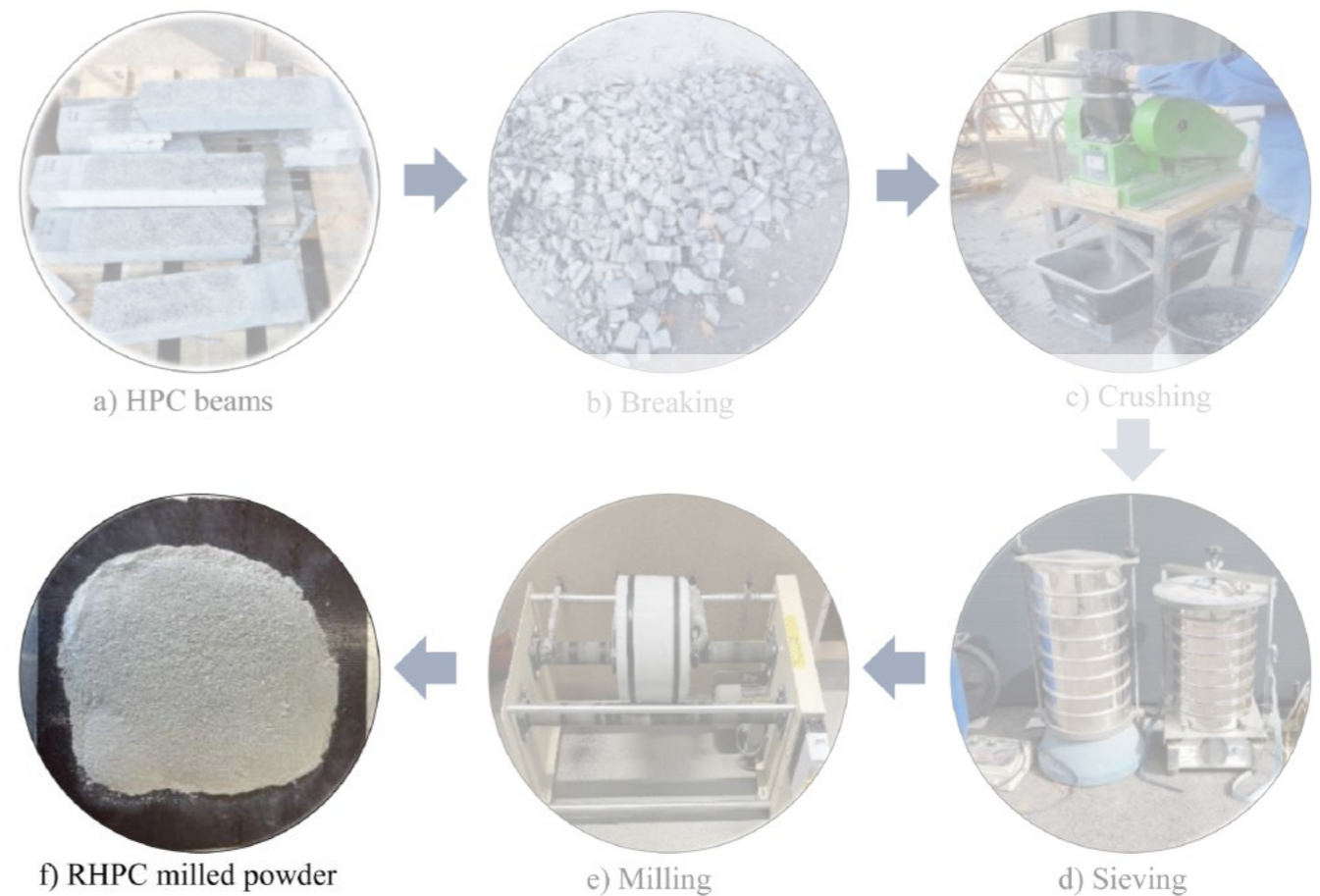
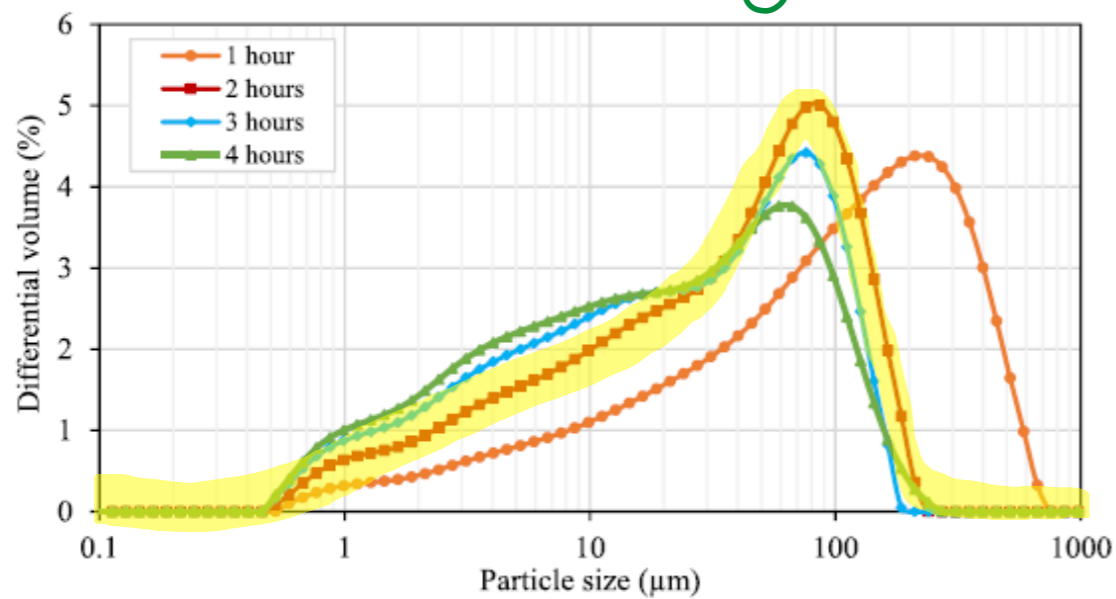
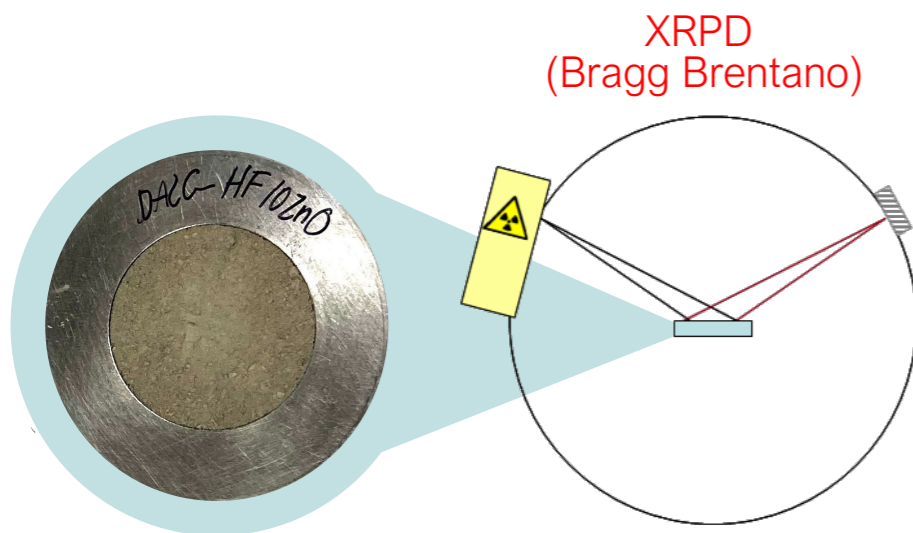


Fig. 1. Recycling process of high-performance concrete (HPC) to obtain mRHPC.

Recycled Concrete



Phase name	mRHPC (wt%)
C ₂ S	2.9 ± 1
C ₃ S	1.5 ± 1
C ₄ AF	2.1 ± 1

ca. 6.5 % of unreacted phases

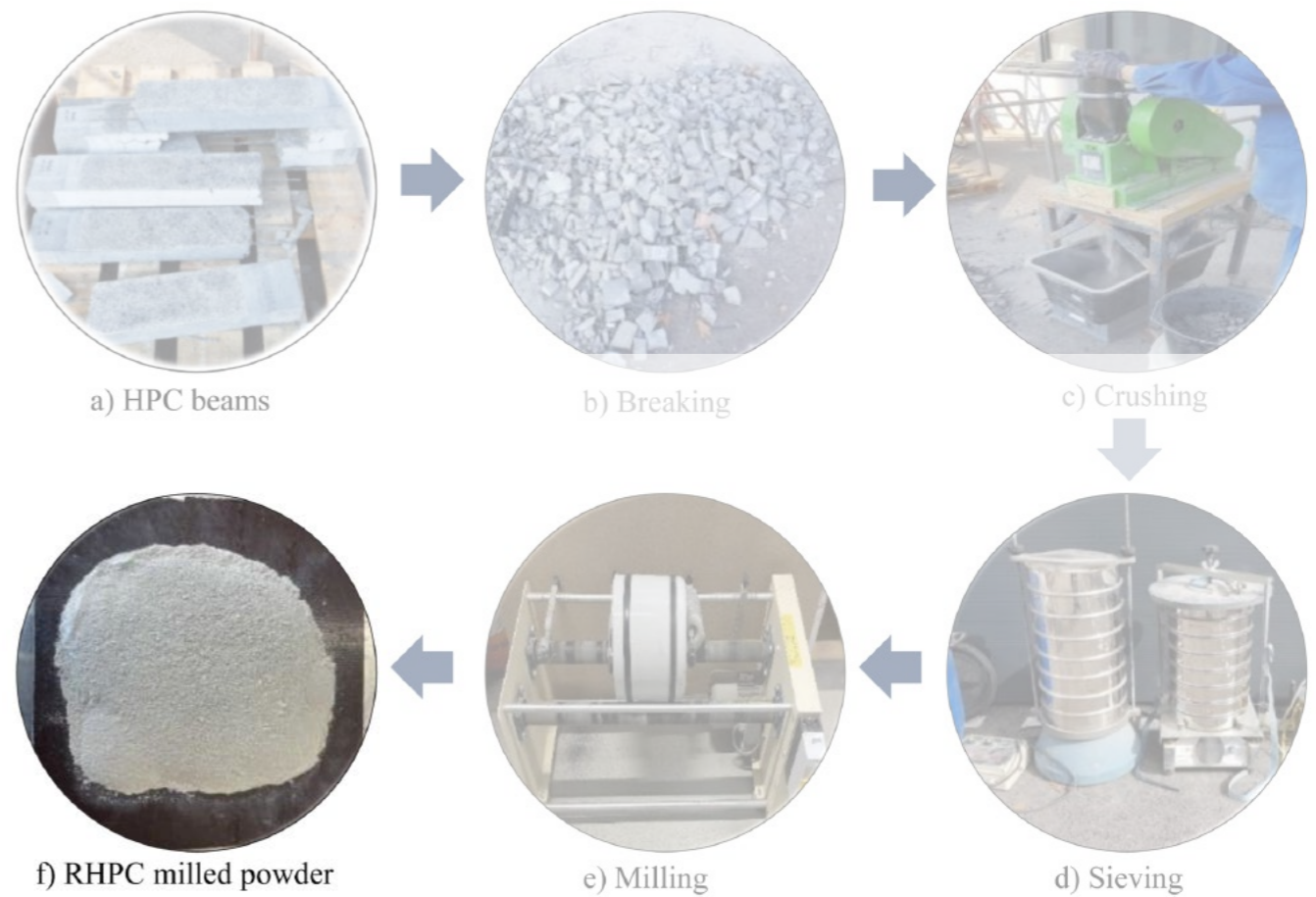
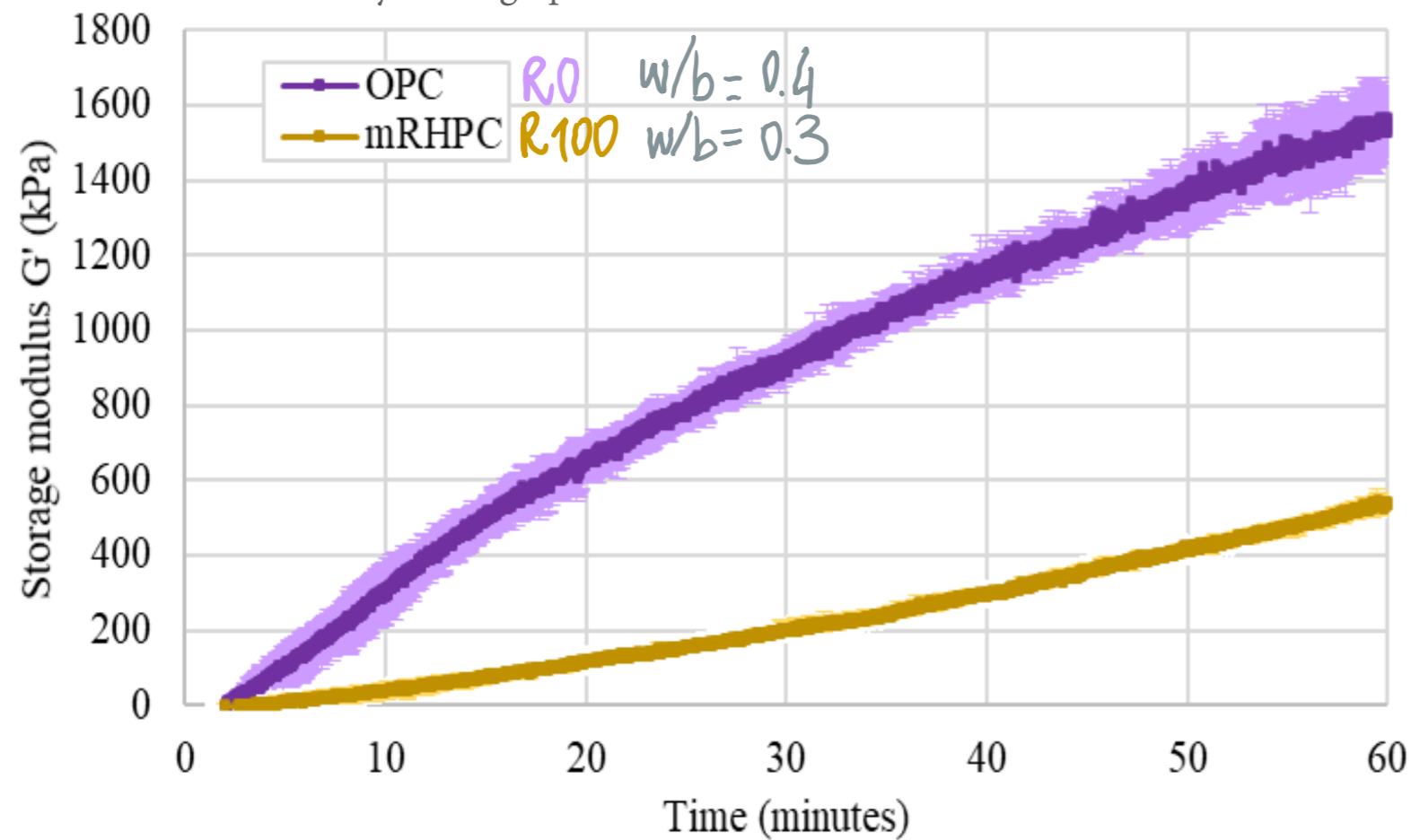


Fig. 1. Recycling process of high-performance concrete (HPC) to obtain mRHPC.

Recycled Concrete

OPC = ordinary Portland cement
RHPC = recycled high performance concrete



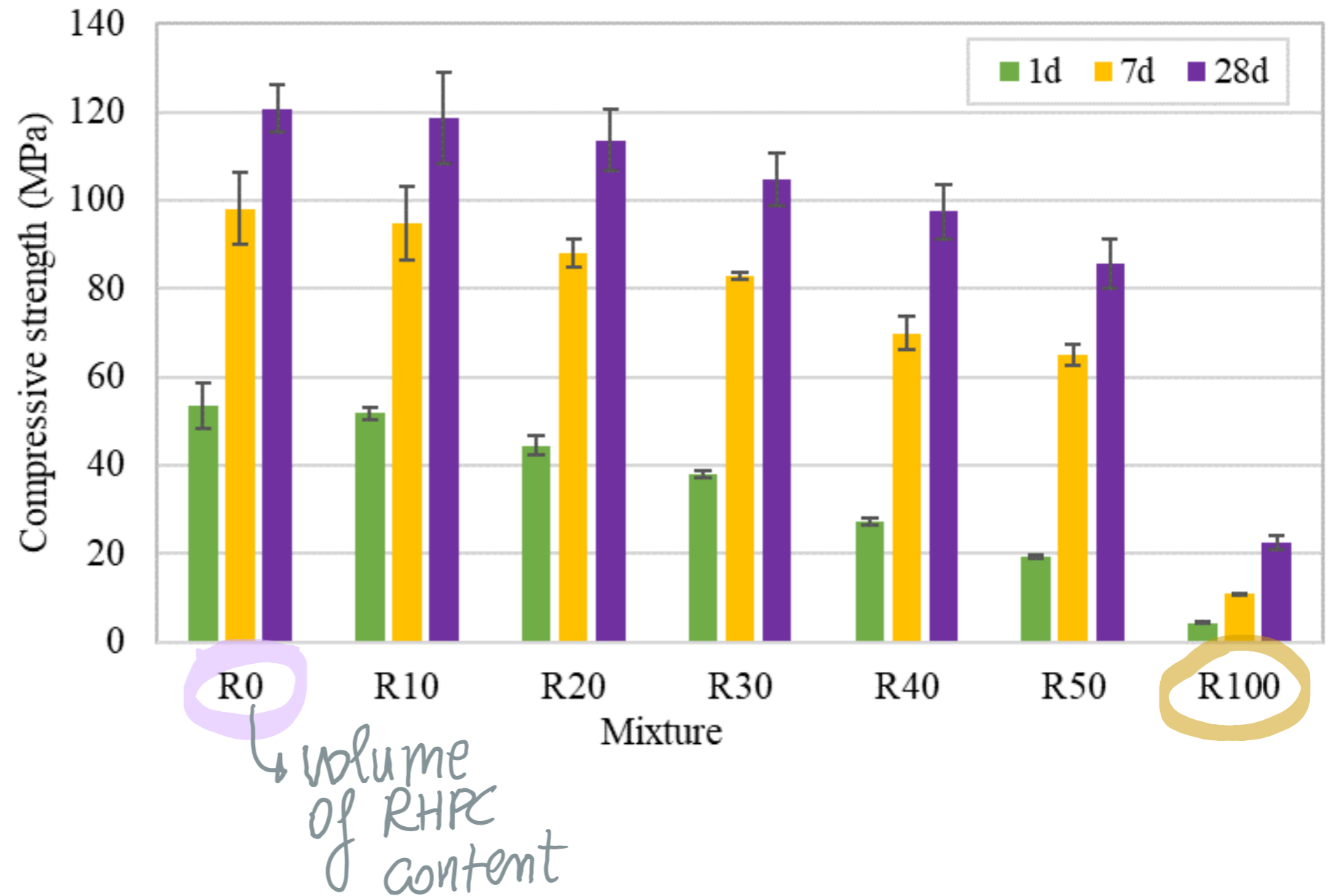
Phase name	OPC (wt%)	mRHPC (wt%)
C ₂ S	12.4 ± 1	2.9 ± 1
C ₃ S	66.1 ± 5	1.5 ± 1
C ₄ AF	12.6 ± 1	2.1 ± 1

Daneshvar et al. *Case Studies in Construction Materials* (2022)

Recycled Concrete



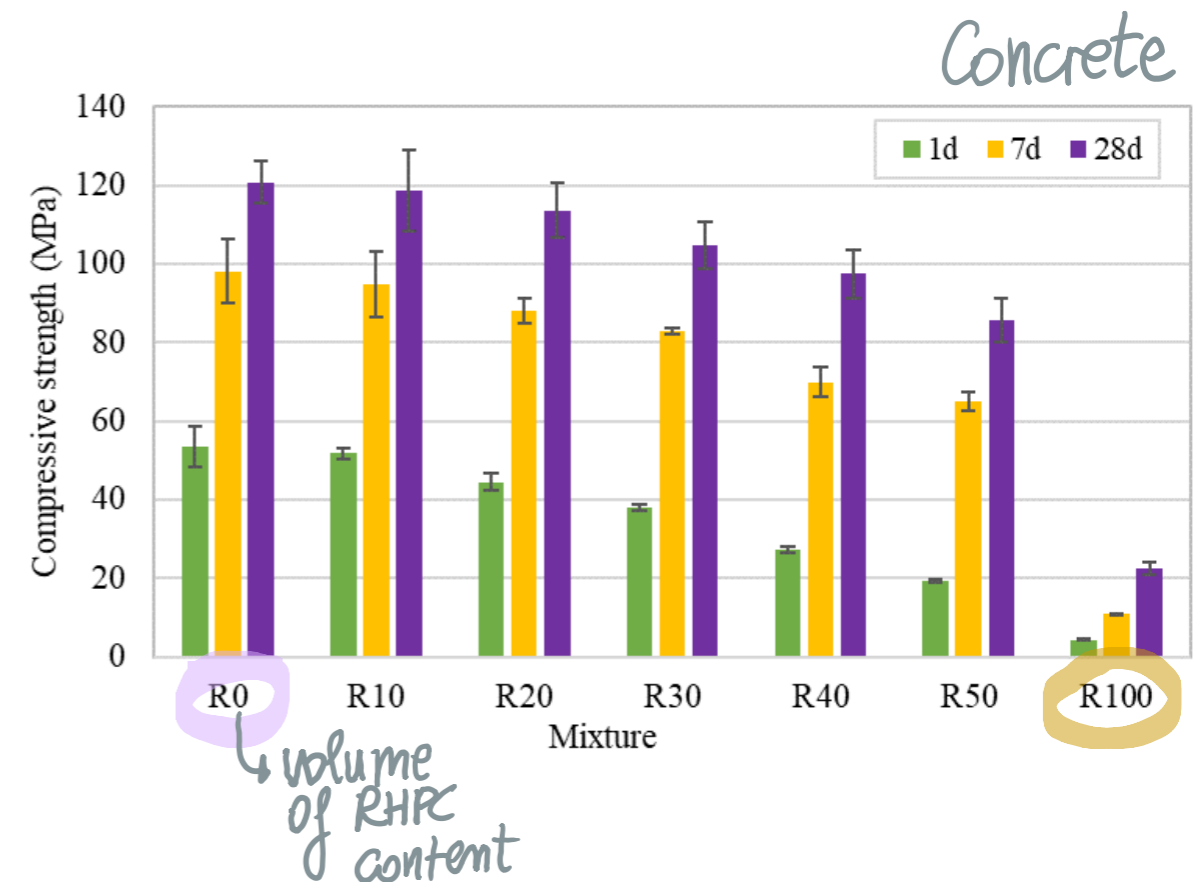
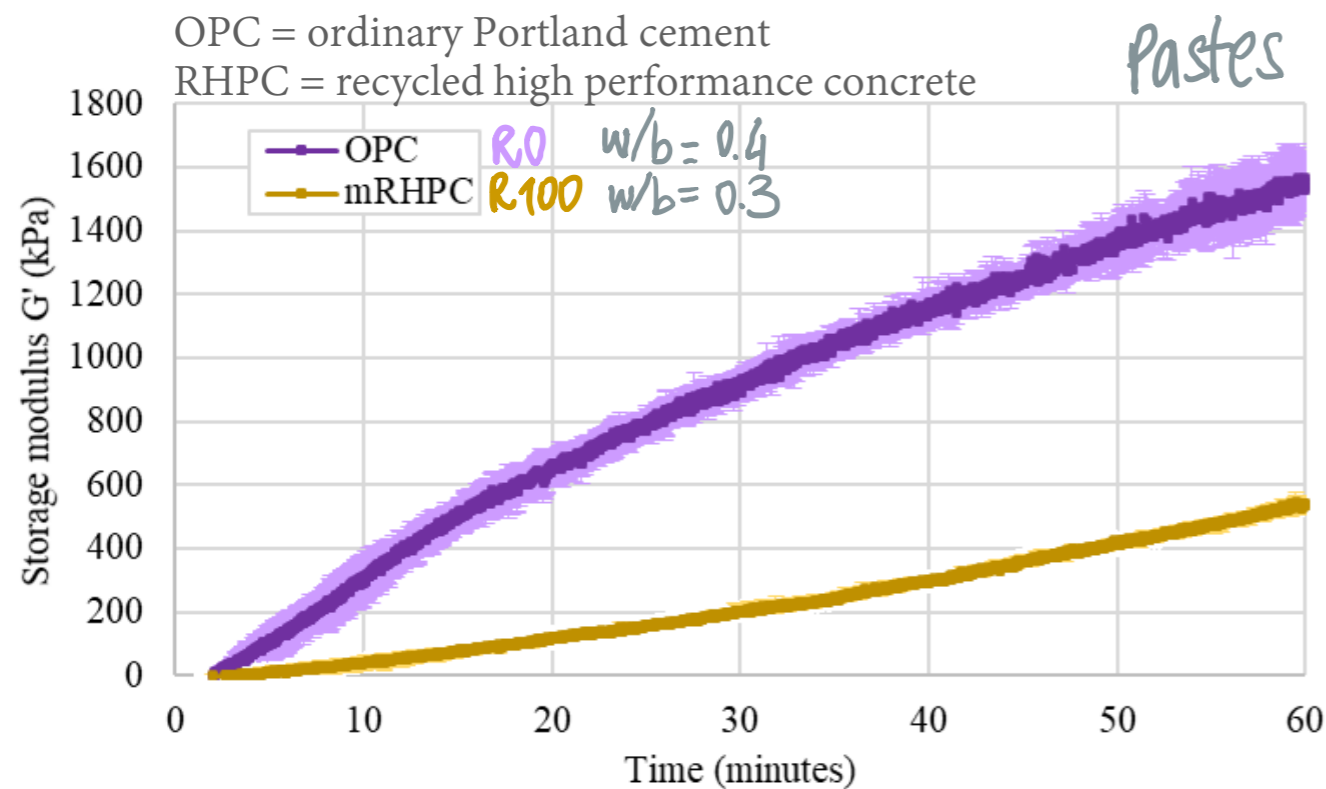
Concrete



Daneshvar et al. *Case Studies in Construction Materials* (2022)

Recycled Concrete

The residual reactivity detected by SAOS is confirmed by mechanical testing



....after 28 days!

Daneshvar et al. *Case Studies in Construction Materials* (2022)

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<https://www.bs.tuwien.ac.at>



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Bau &
Umwelt
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