Diffusing wave spectroscopy in elastomer fracture

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Supervisors: Costantino Creton, Tetsuharu Narita

In collaboration with Luca Cipelletti (Montpellier, CNRS)

Fracture at different length scale

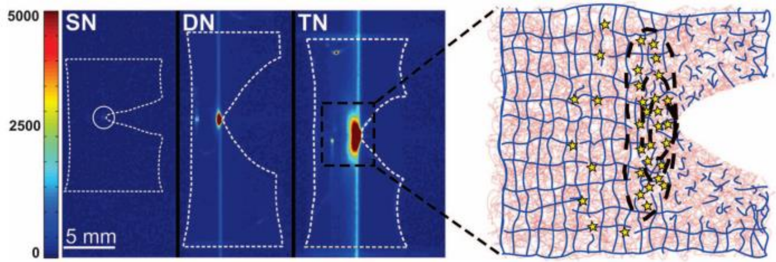
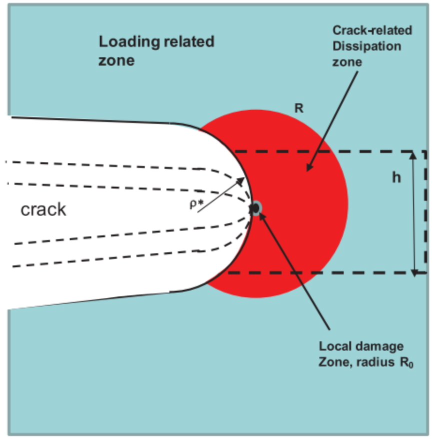
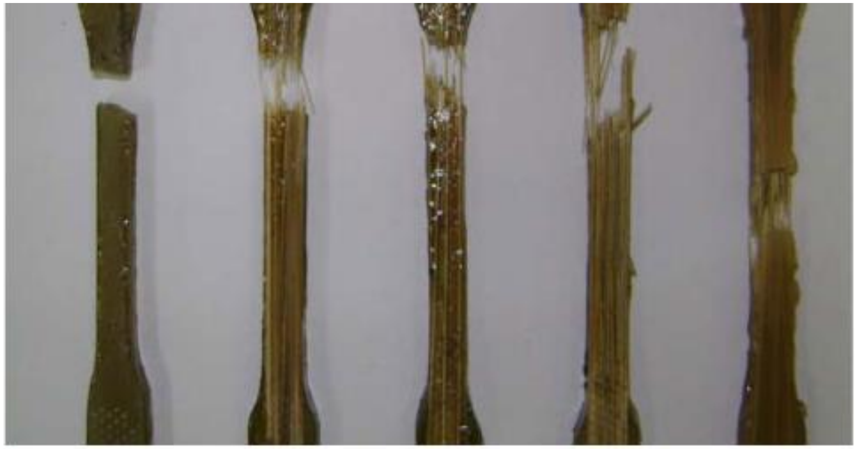


Fig. 5. Mapping of where bonds break during crack propagation. Intensity-colorized images of propagating cracks on notched samples containing dioxetane cross-linker in the first network, showing the light emission due to the breakage of bonds in SN, DN, and TN samples. The size and geometry of the sample are shown with a white dashed line. Vertical lines in the DN and TN image are due to detector artifacts; in DN and TN, spots far from the crack tip are due to reflection of the light on surface inhomogeneities and do not correspond to a local light emission. (Right) Schematic of the sacrificial bond-breaking mechanism in front of the crack tip for the DN and TN; the first network is represented in blue, and the second and third networks are in red.





Molecular scale (~10nm)

Ducrot, Etienne, et al.2014

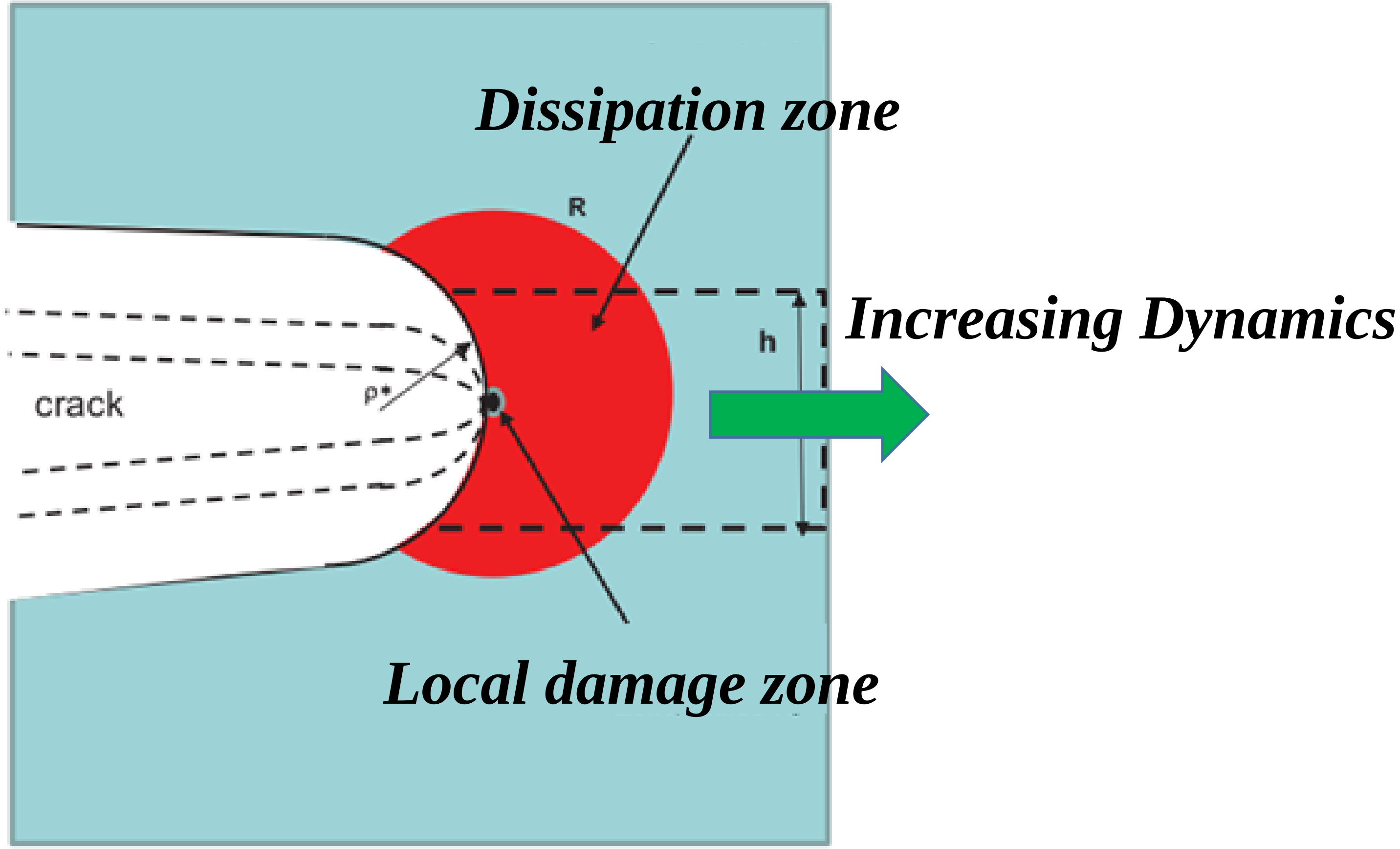
Mesoscopic scale (0.1 ~10 μm)

Creton, Costantino, and Matteo Ciccotti.2016

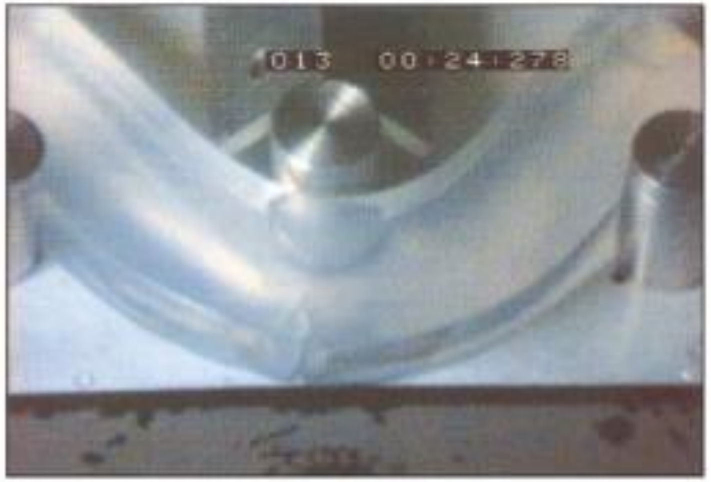
Macroscopic scale

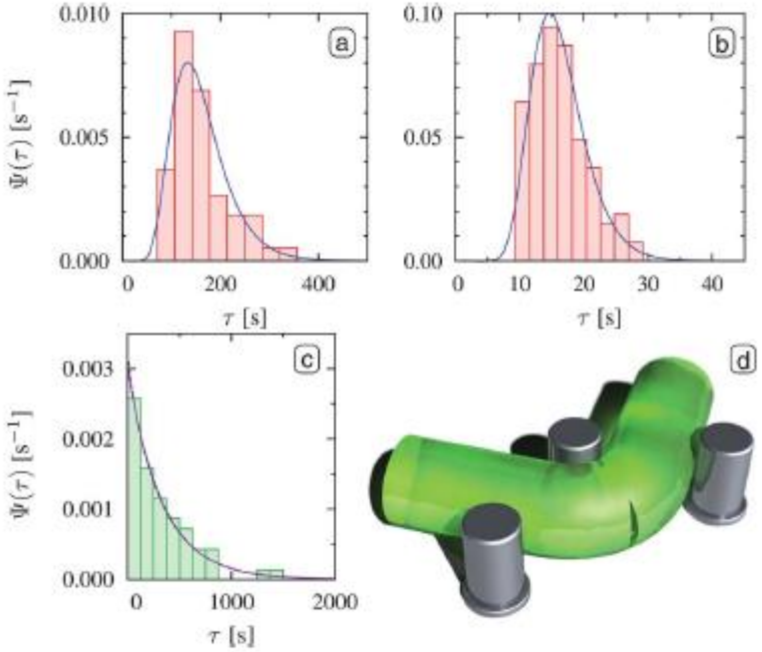
Martínez, A. B., et al. 2017

Fracture precursor



Delayed fracture

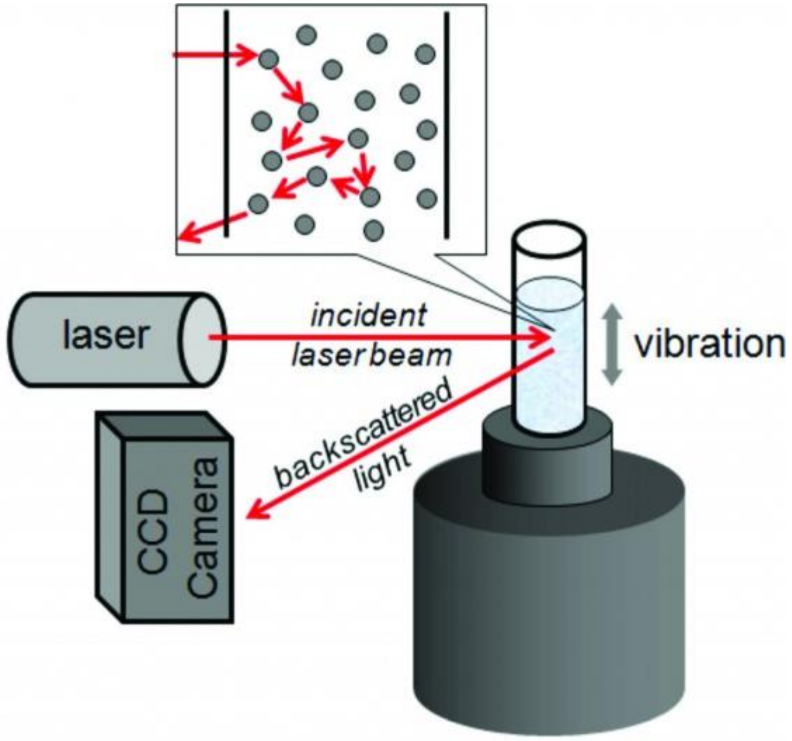


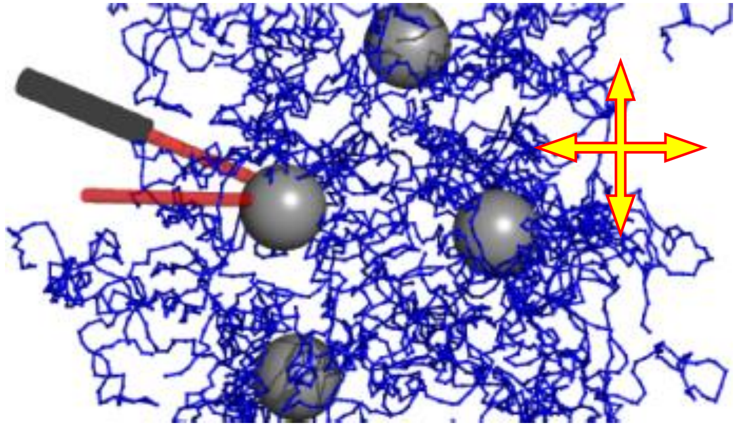


Bonn, Daniel, et al. "Delayed fracture of an inhomogeneous soft solid." *Science* 280.5361 (1998): 265-267.

Lindström, Stefan B., et al. "Structures, stresses, and fluctuations in the delayed failure of colloidal gels." *Soft Matter* 8.13 (2012): 3657-3664.

DWS technology in polymer network



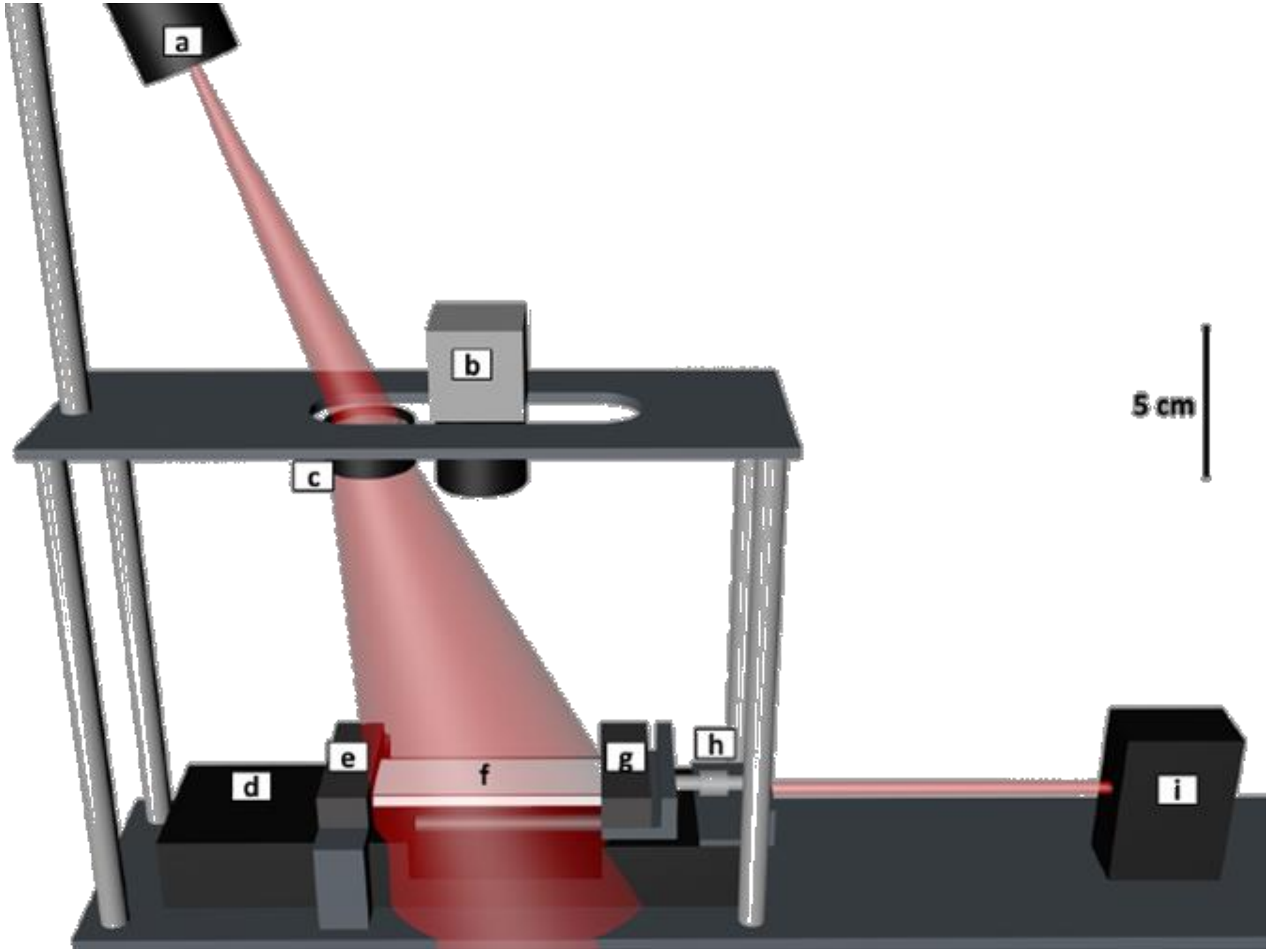


Polymer network + Probe particles

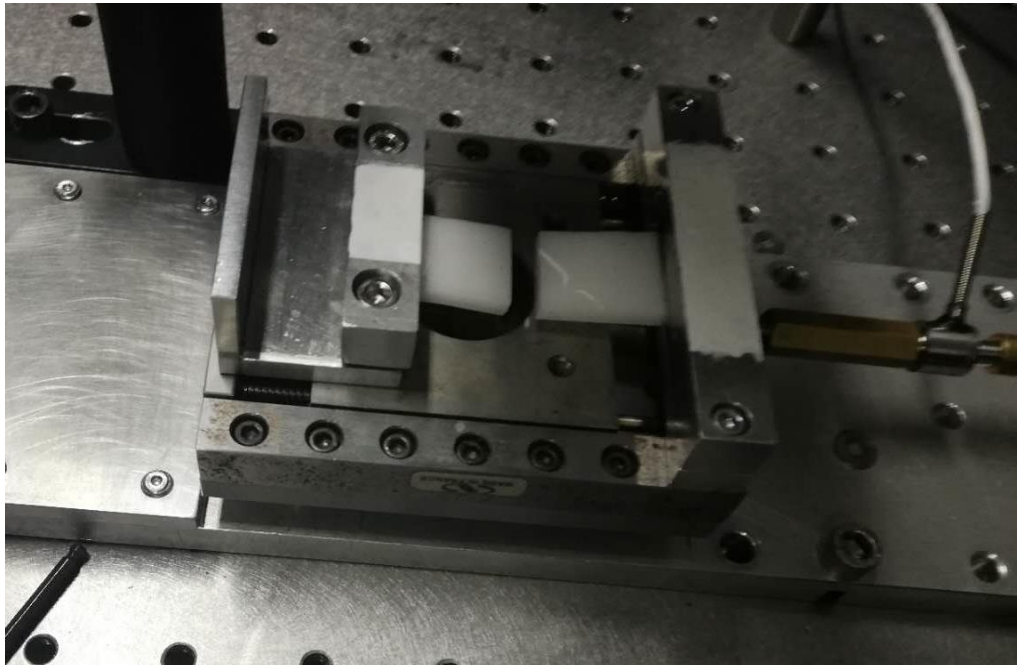
DWS: Multiple scattering

Krajina, Brad A., et al. 2017

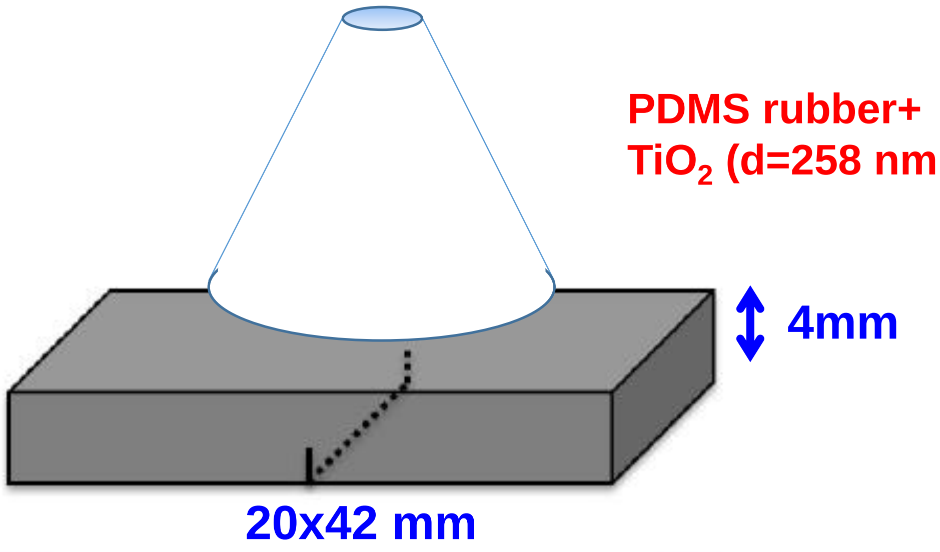
Experimental set-up



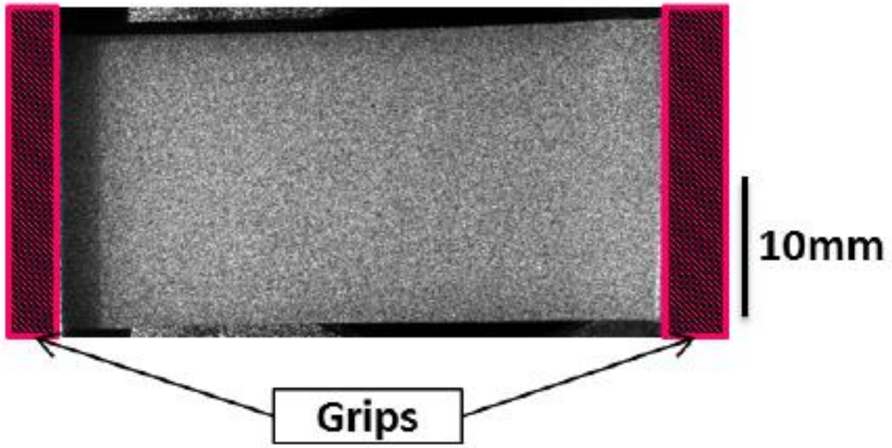
a: Laser
b: Camera
c: Lens
d: Motor
e: Fixed clamp
f: Sample
g: Moving clamp
h: Tensile sensor
i: Displacement sensor

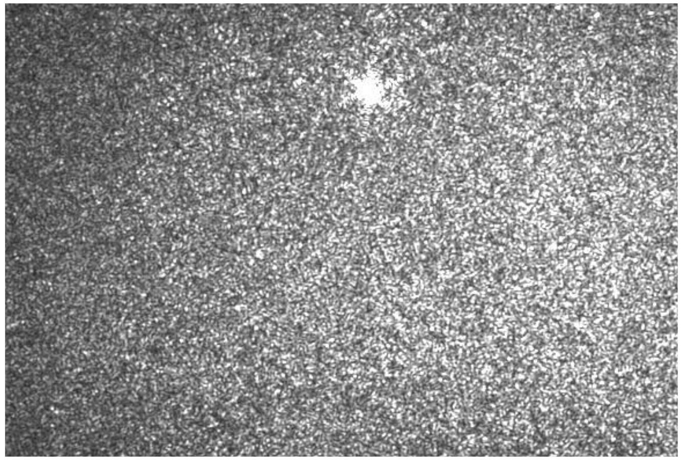


DWS measurement on PDMS



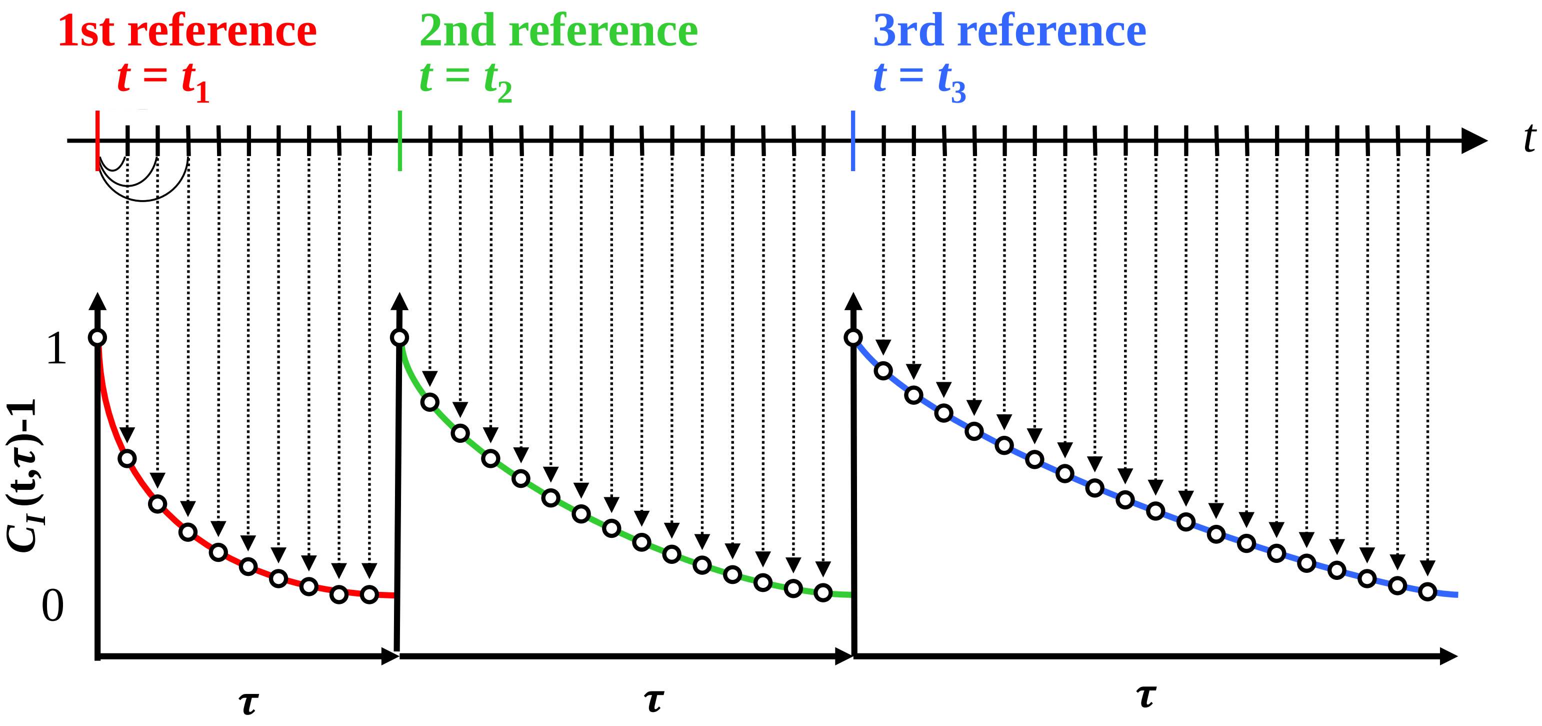
PDMS rubber+
TiO_2 (d=258 nm, ϕ=0.05%)





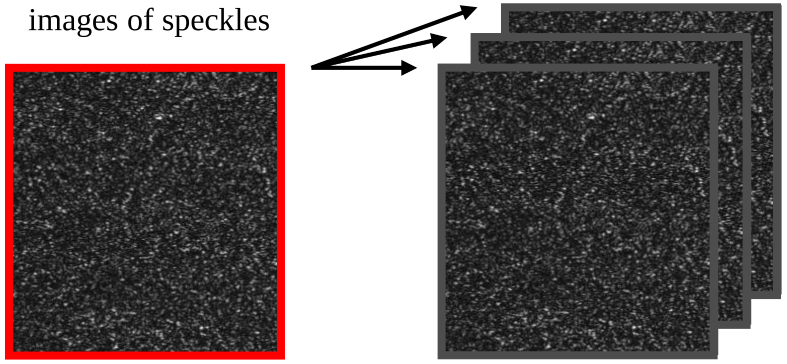
Macroscopic fracture —— Force data
Mesoscopic change —— DWS

Data analysais and fitting



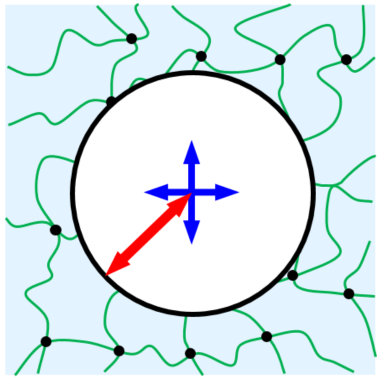
$$C_I(t,\tau) = \frac{\langle I_P(t) I_P(t+\tau)\rangle_P}{\langle I_P(t)\rangle_P \langle I_P(t+\tau)\rangle_P} - 1$$

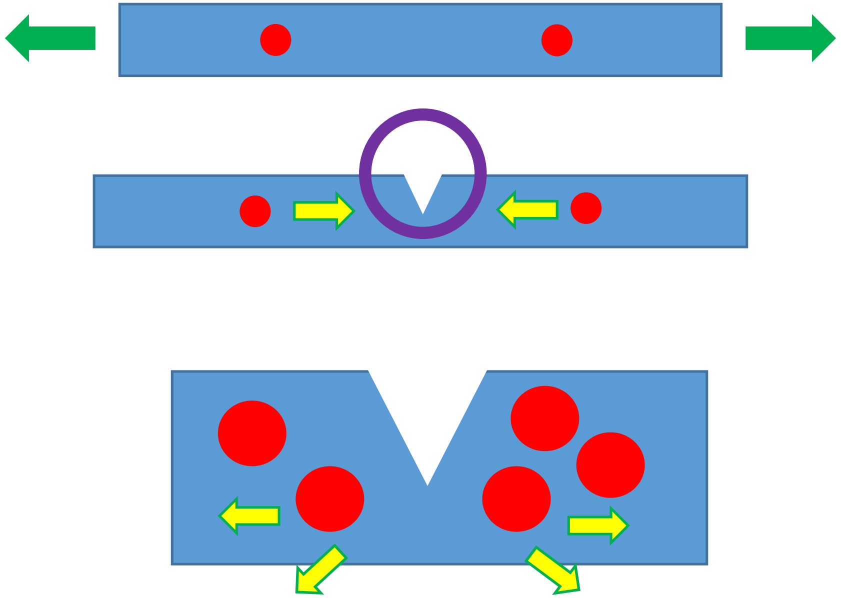
$$C_I(t_1,\tau) = A\exp\left(-\left(\frac{\tau}{\tau_0}\right)^P\right) + B$$

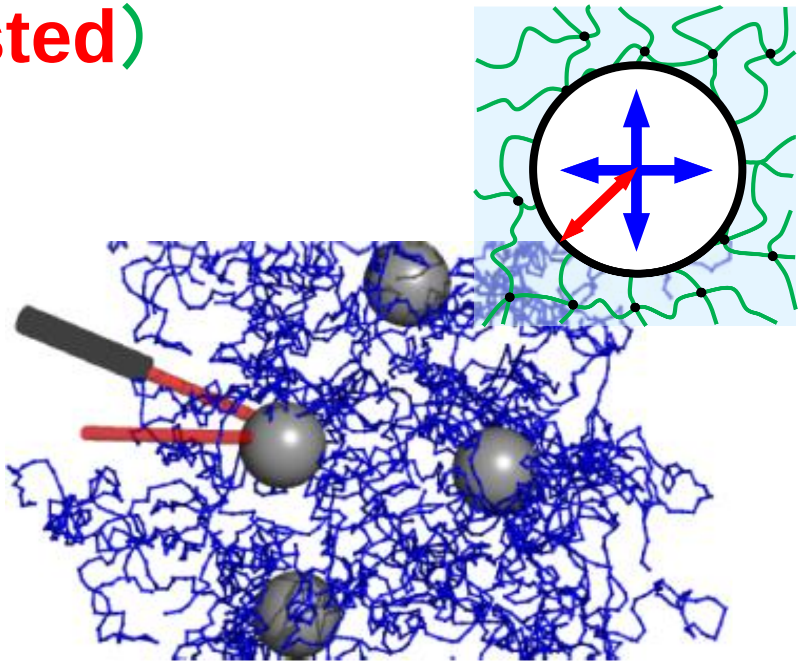


Particles dynamics in fracture

1. Intrinsic thermal fluctuation （baseline）
2. Translation due to fracture, relaxation and creep… （correction）
3. Displacement limited by chains （interested）

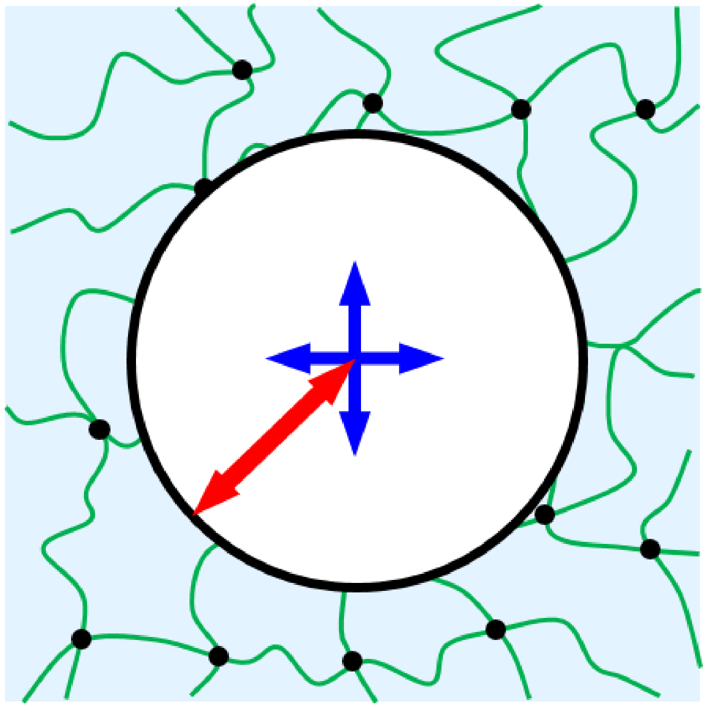






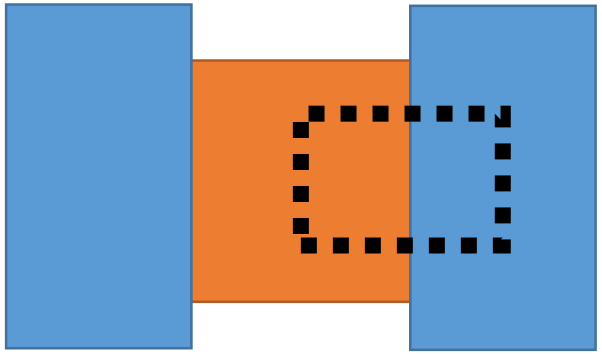
Particles dynamics in fracture

1. Intrinsic thermal fluctuation
2. Translation due to fracture, relaxation and creep…
3. Displacement limited by chains

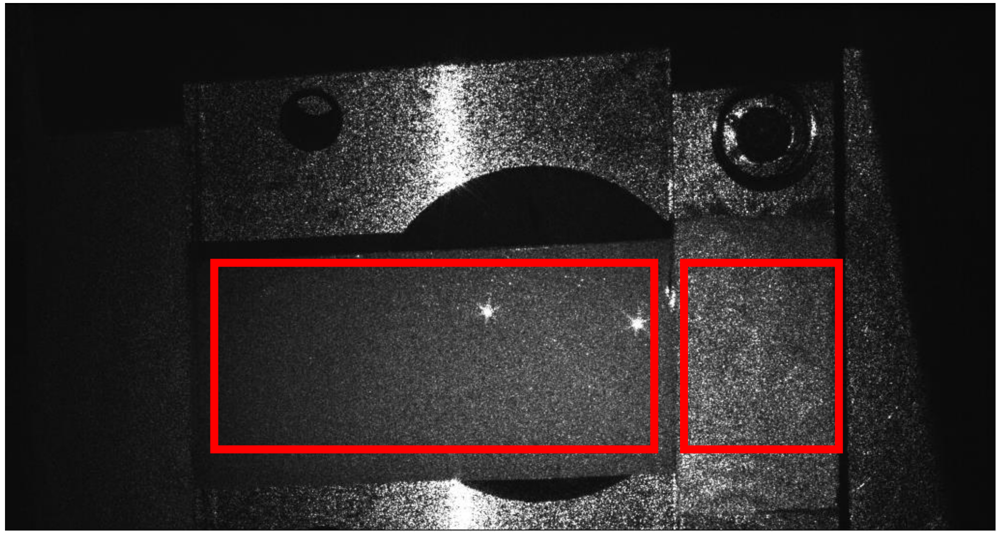


Yassine Nagazi PhD thesis, 2017

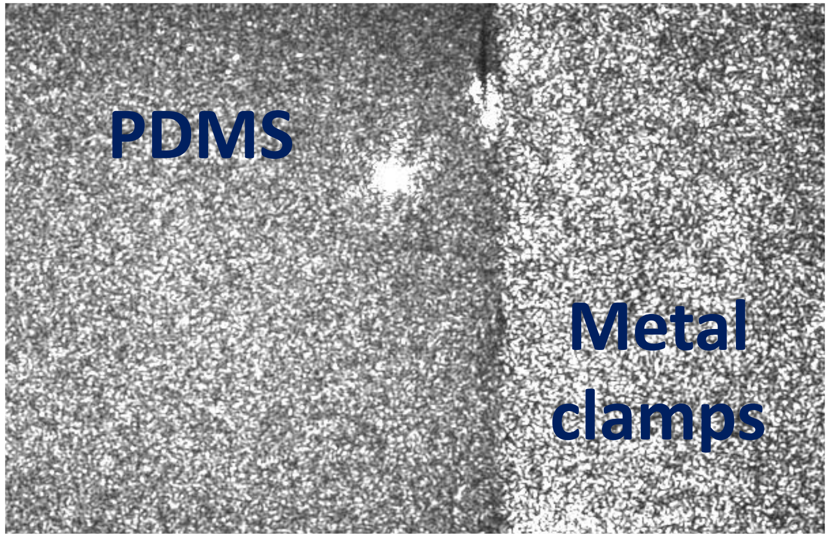
1. Static sample/clamp



Sample + system



No loading



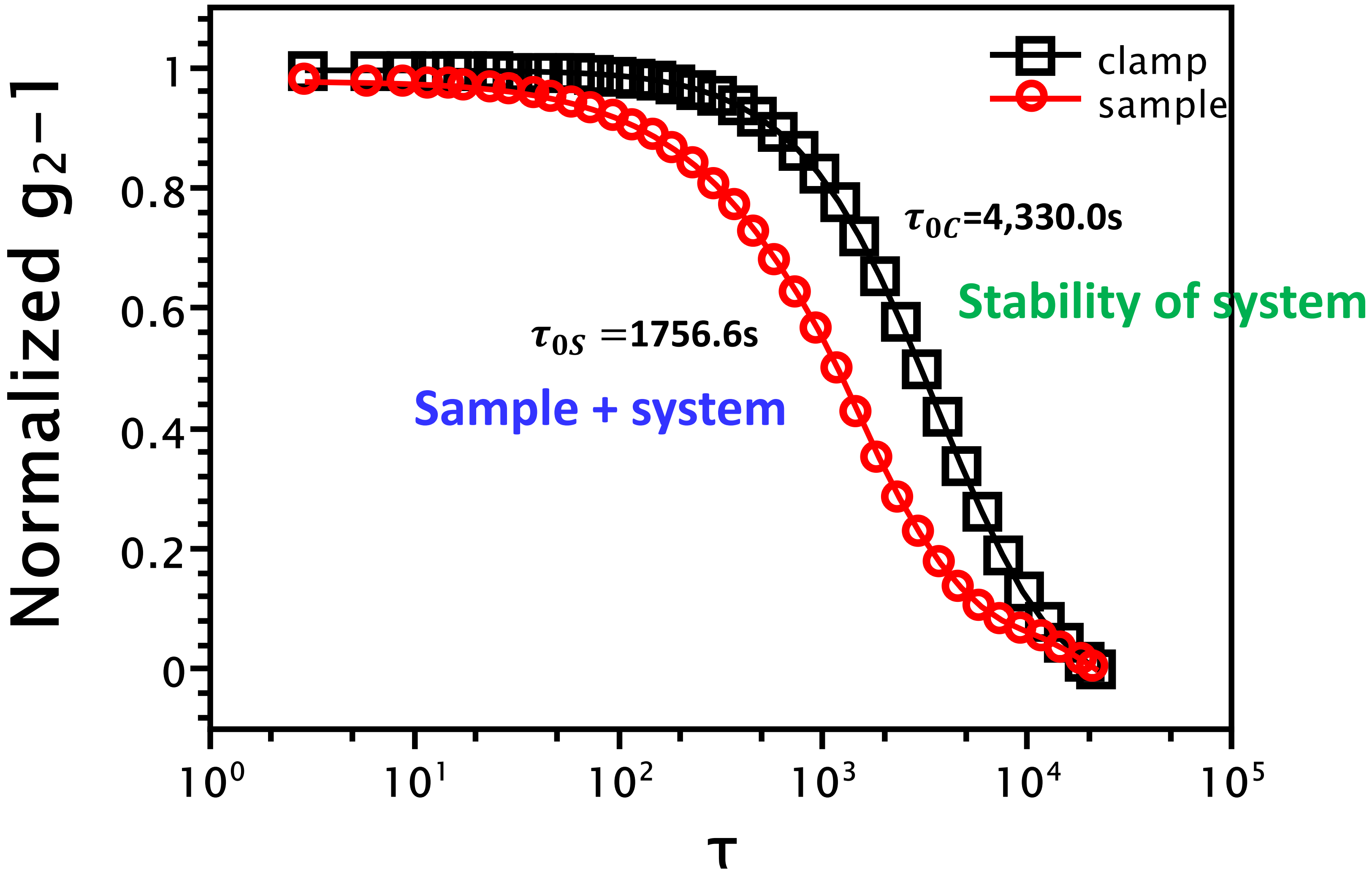
Stability of system:

Laser stability

Temperature fluctuation

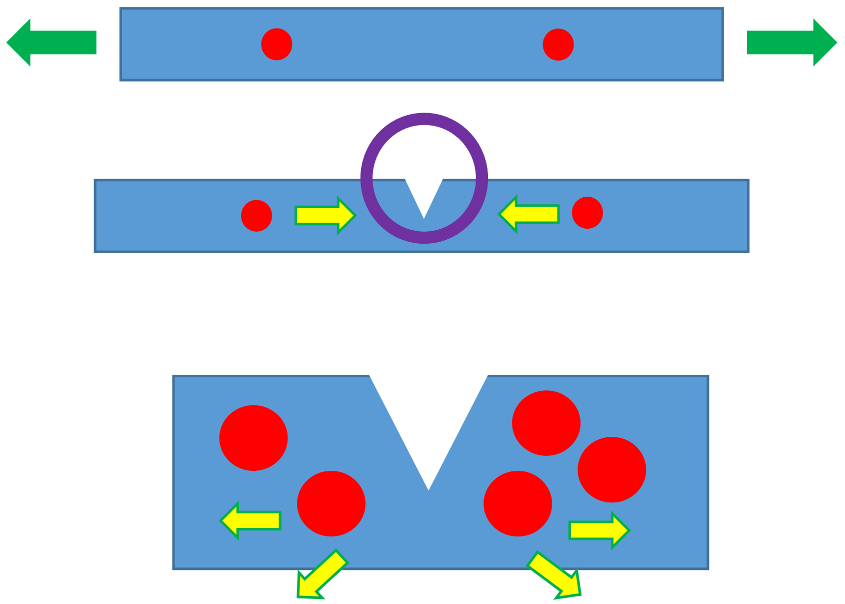
Environment Brightness

$$g_2(\tau)\text{-}1 = \langle C_I(t,\tau)\rangle_t = A\exp\left(-\left(\frac{\tau}{\tau_0}\right)^P\right) + B$$

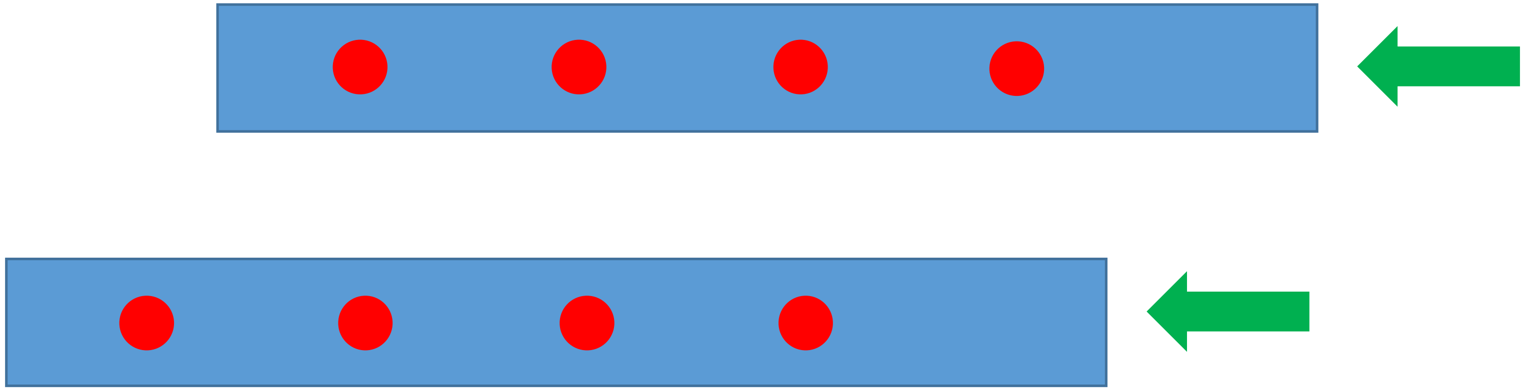


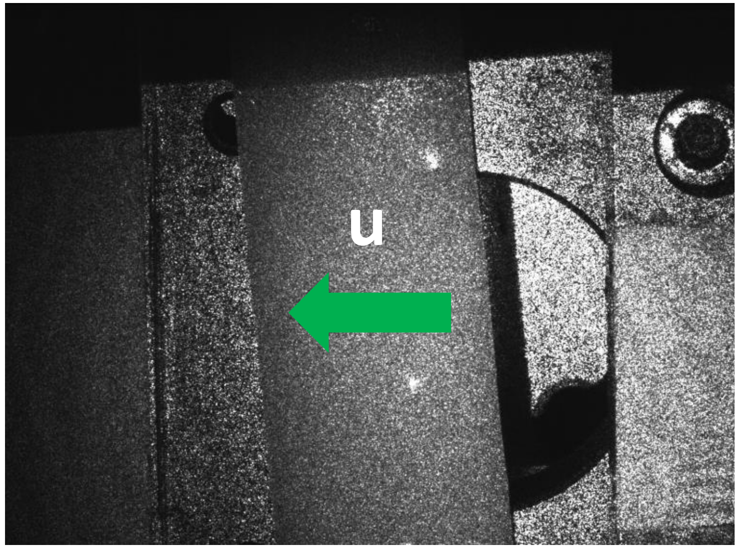
Particles dynamics in fracture

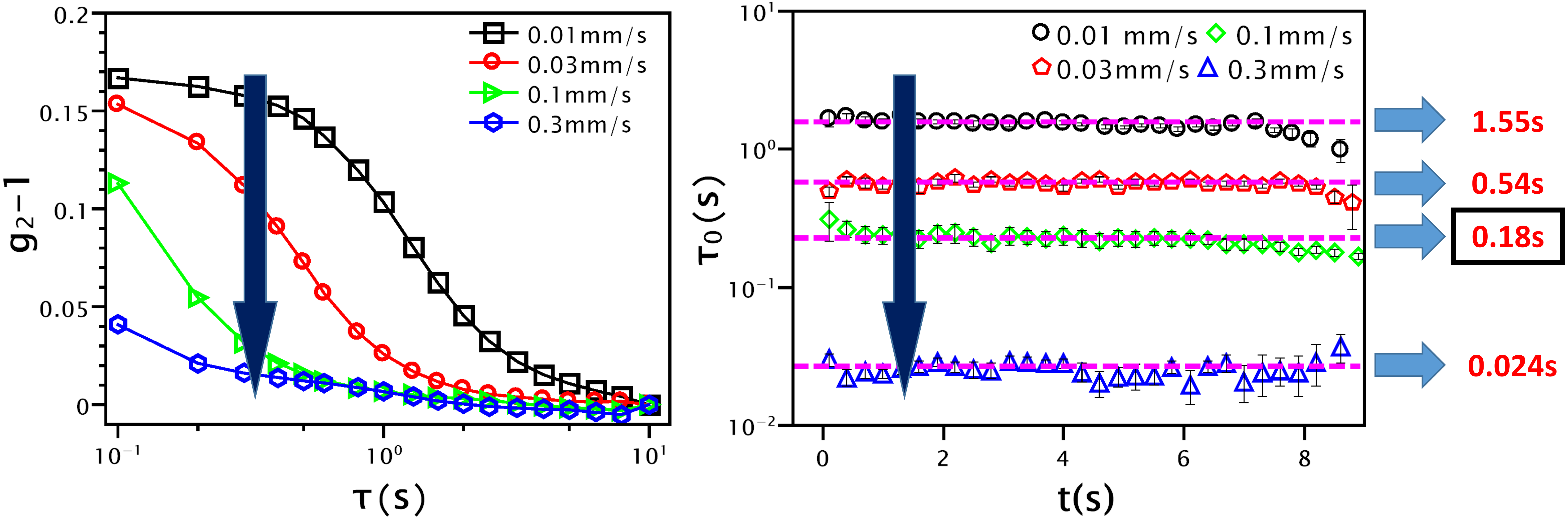
1. Intrinsic thermal fluctuation
2. Translation due to fracture, relaxation and creep…
3. Displacement limited by chains

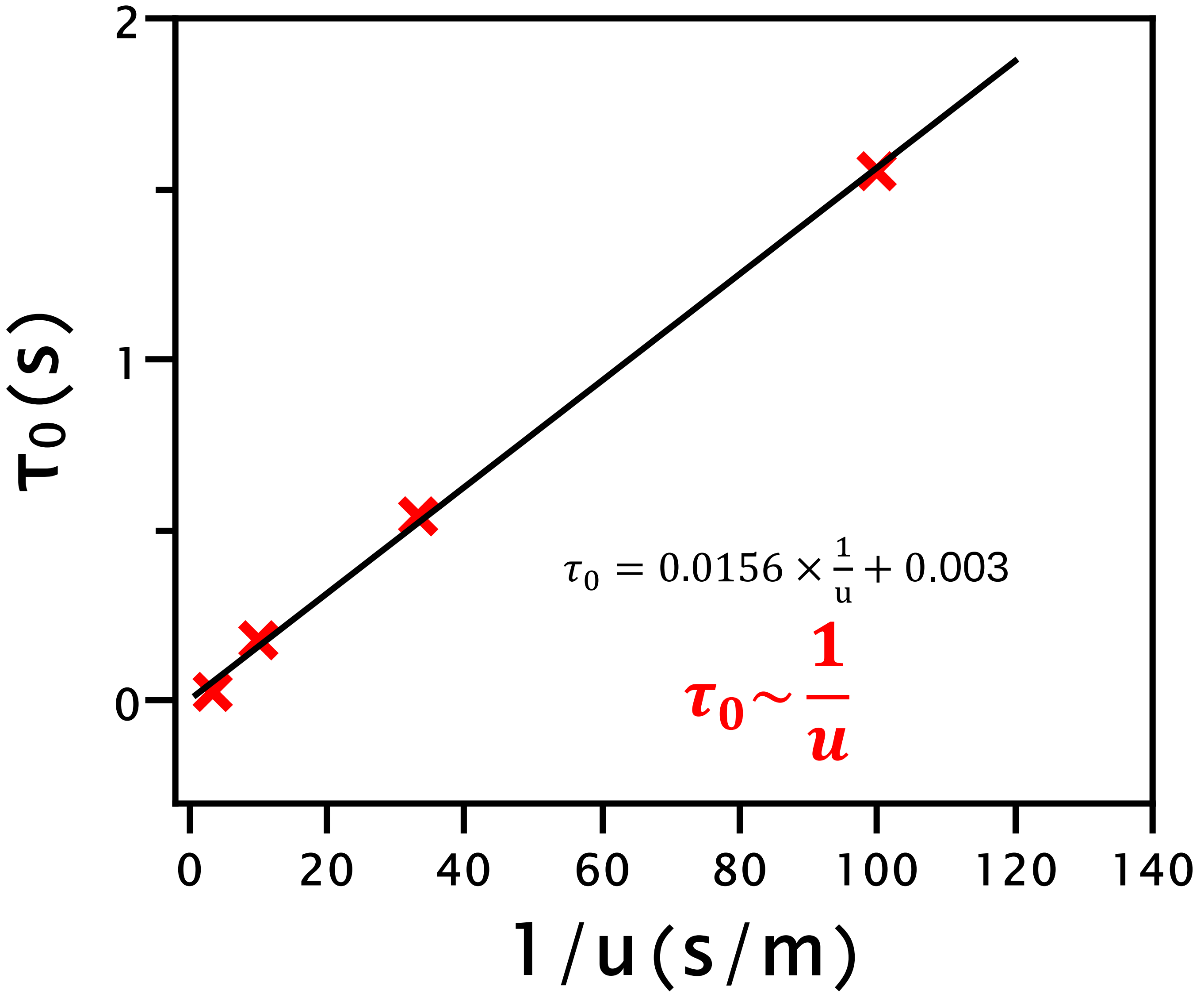


2. Translation





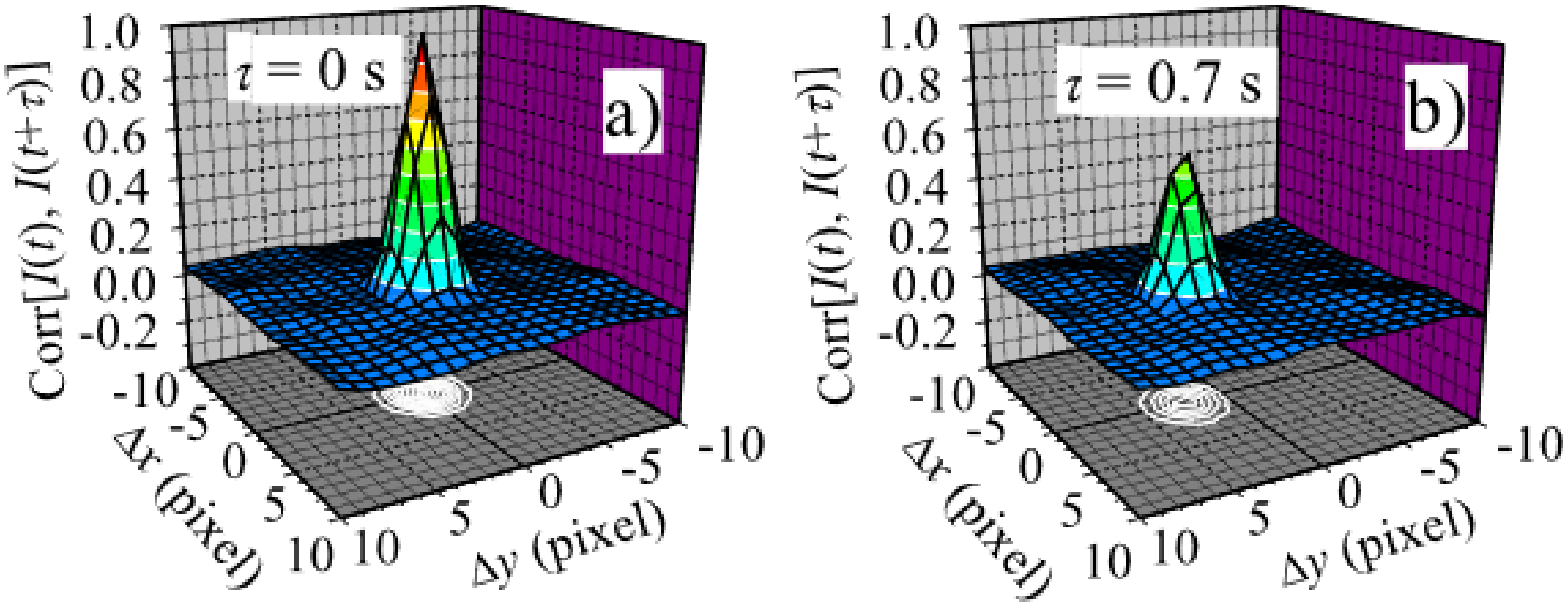

0.2
0.15
0.1
0.05
0
g2−1
10−1
10^0
10^1
τ(s)
0.01mm/s
0.03mm/s
0.1mm/s
0.3mm/s
10^1
10^0
10−1
10−2
τ0(s)
0
2
4
6
8
t(s)
0.01 mm/s
0.1mm/s
0.03mm/s
0.3mm/s
1.55s
0.54s
0.18s
0.024s



Translation speed∼Loss rate of correlation

Practical complex drifting field: Drifting correction

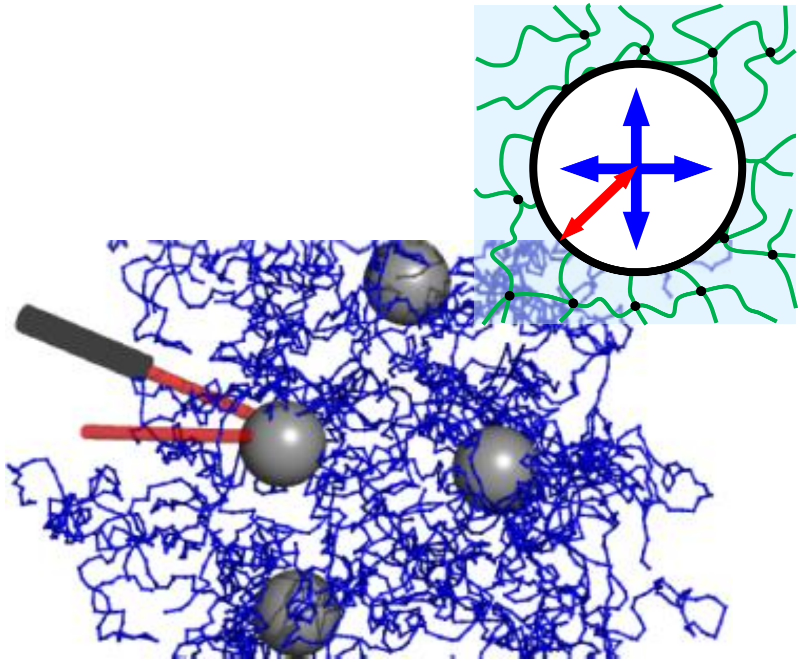
$$\langle C_I(t,\tau)\rangle_t \rightarrow \langle C_I(t,\tau,x,y)\rangle_t$$



Cipelletti, Luca, et al. *Optics express* 2013

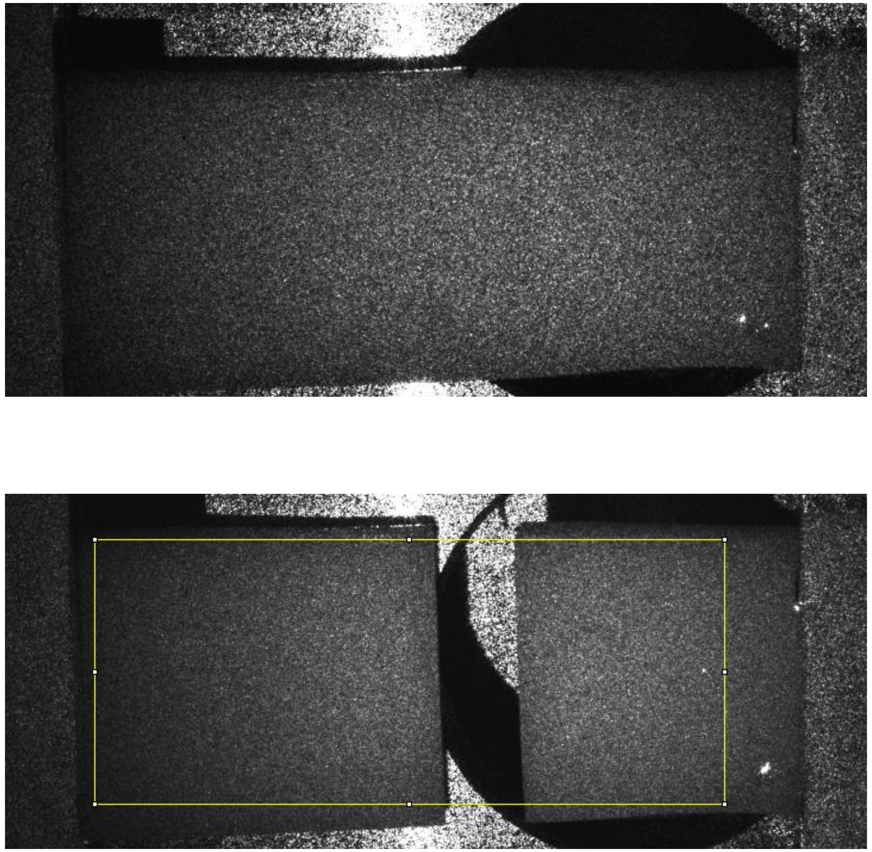
Particles dynamics in fracture

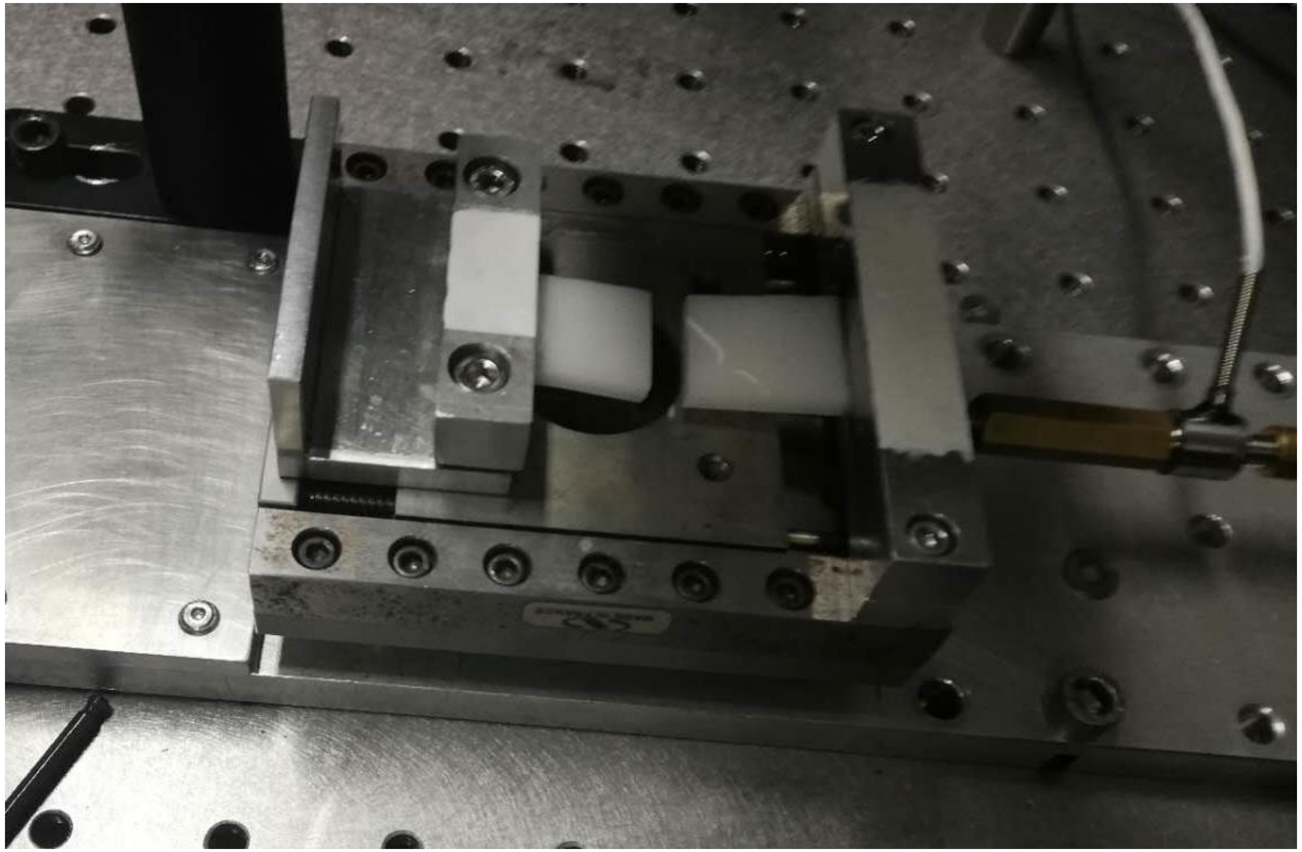
1. Intrinsic thermal fluctuation
2. Translation due to fracture, relaxation and creep…
3. Displacement limited by chains (dynamics change)



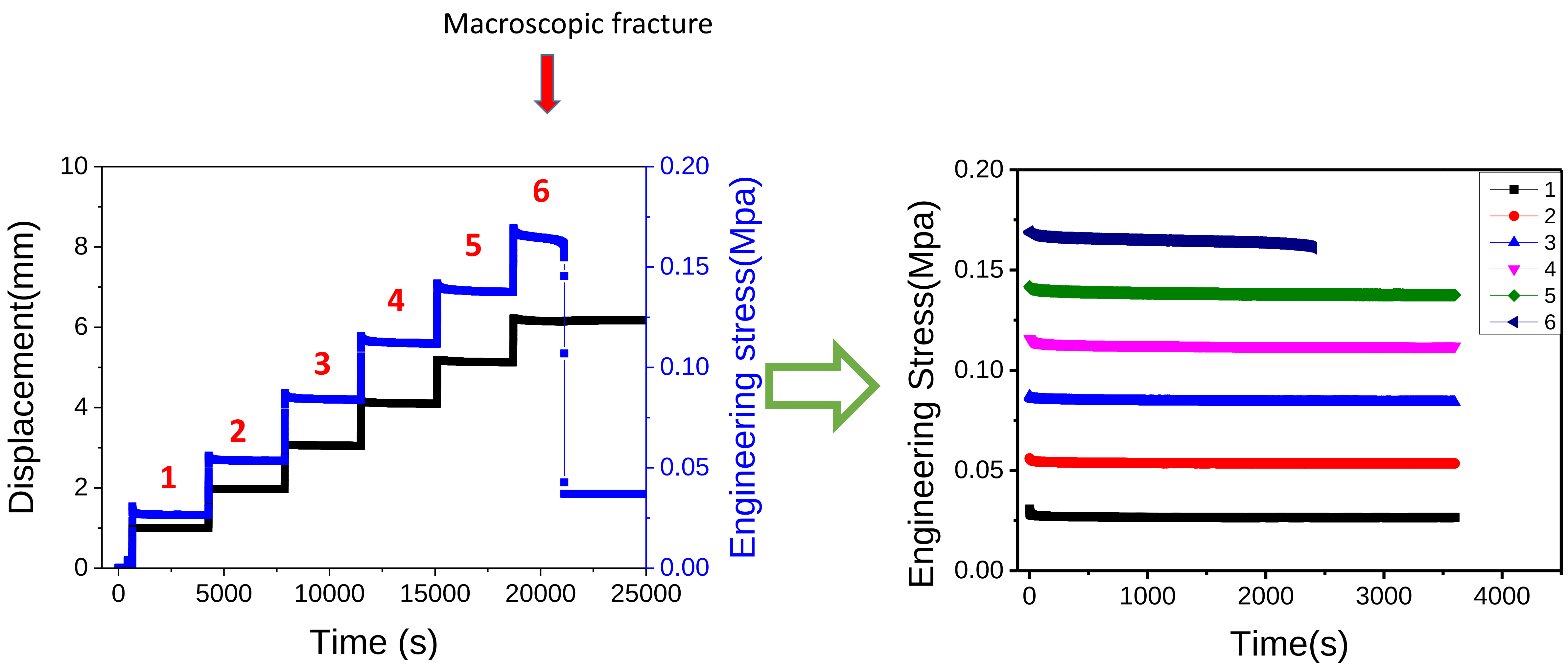
3. Multi-steps delayed fracture

Yassine Nagazi thesis, 2017

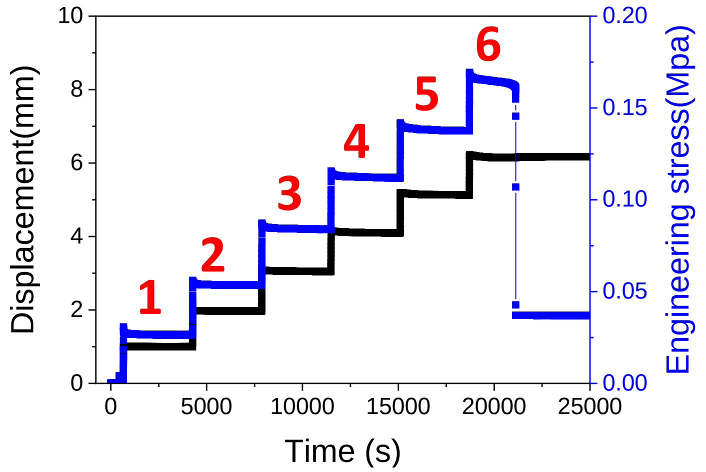




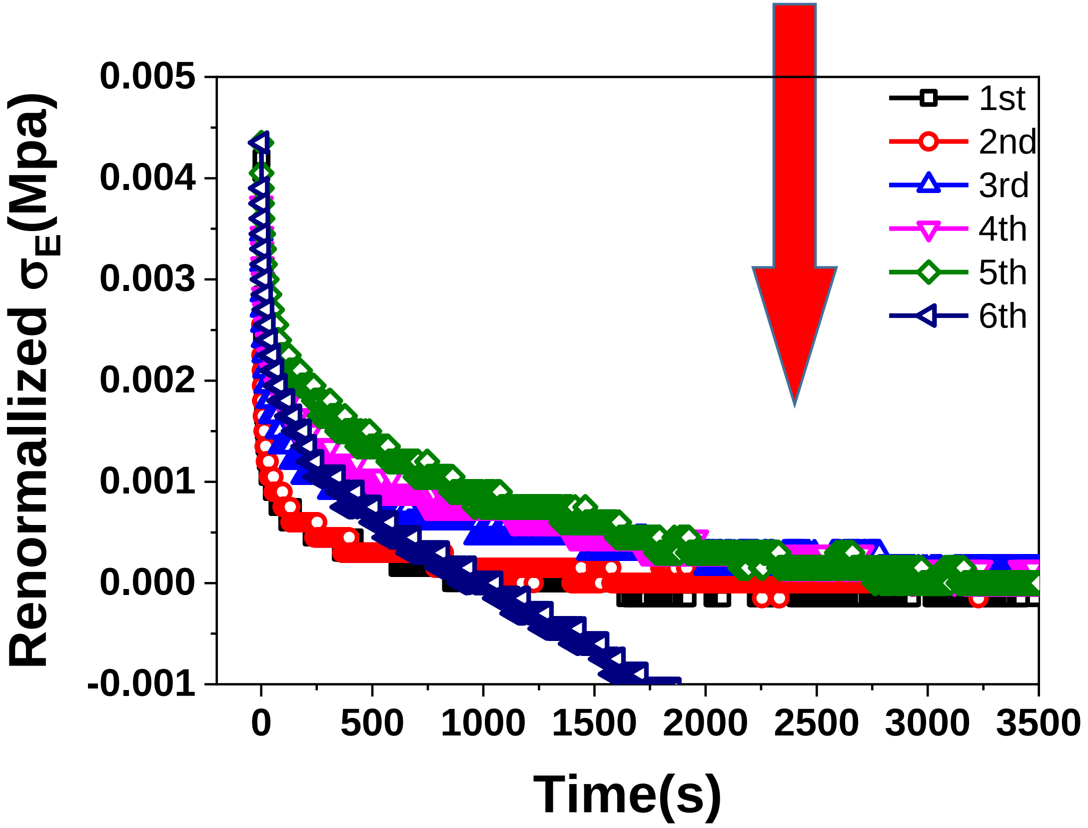
Extension length/Force

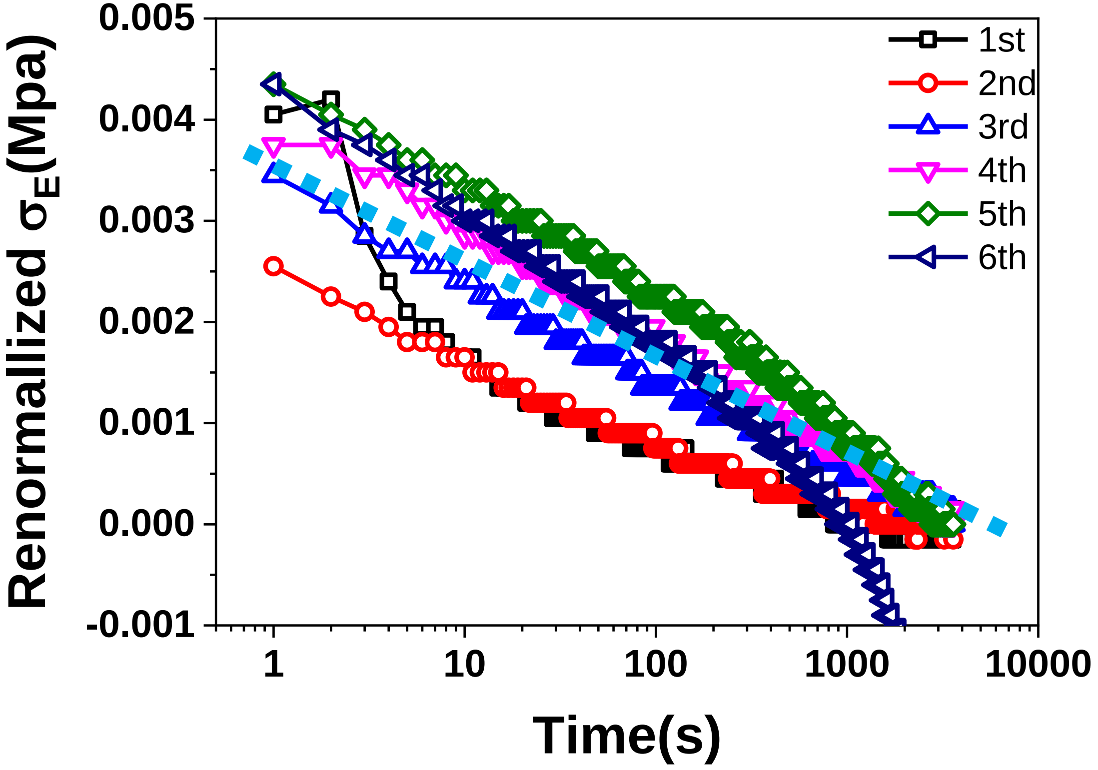


Macroscopic measurement

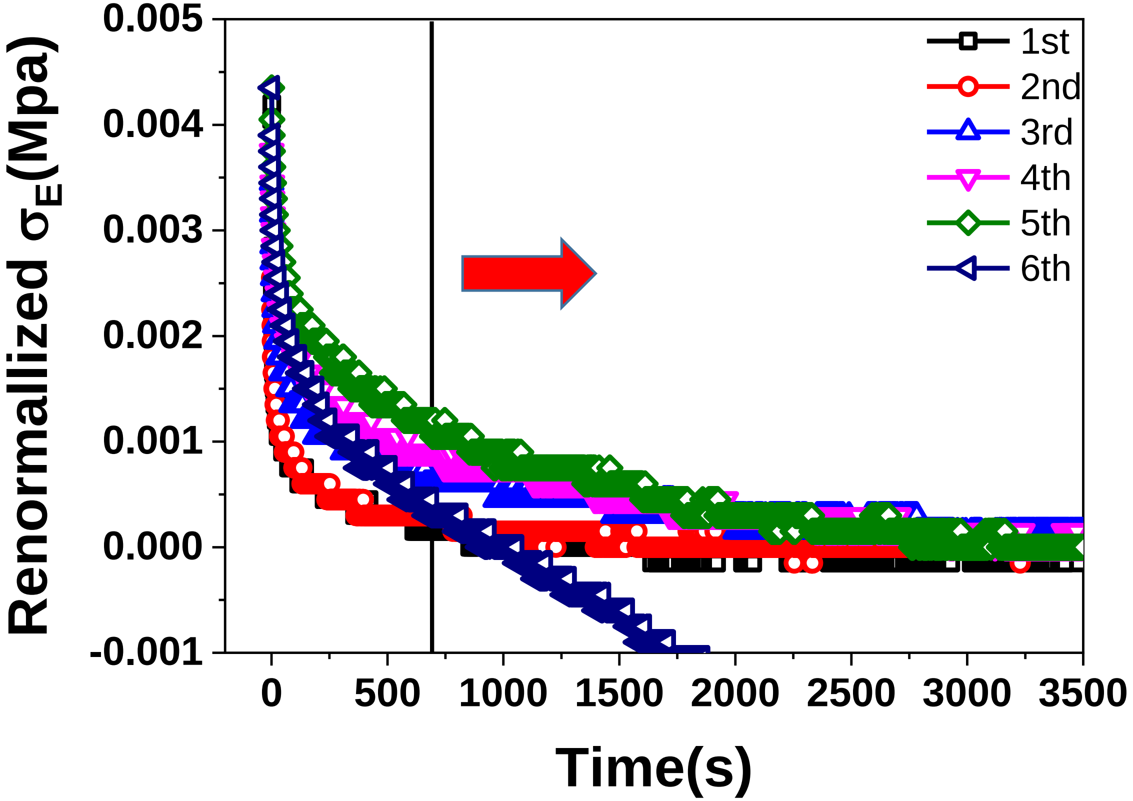


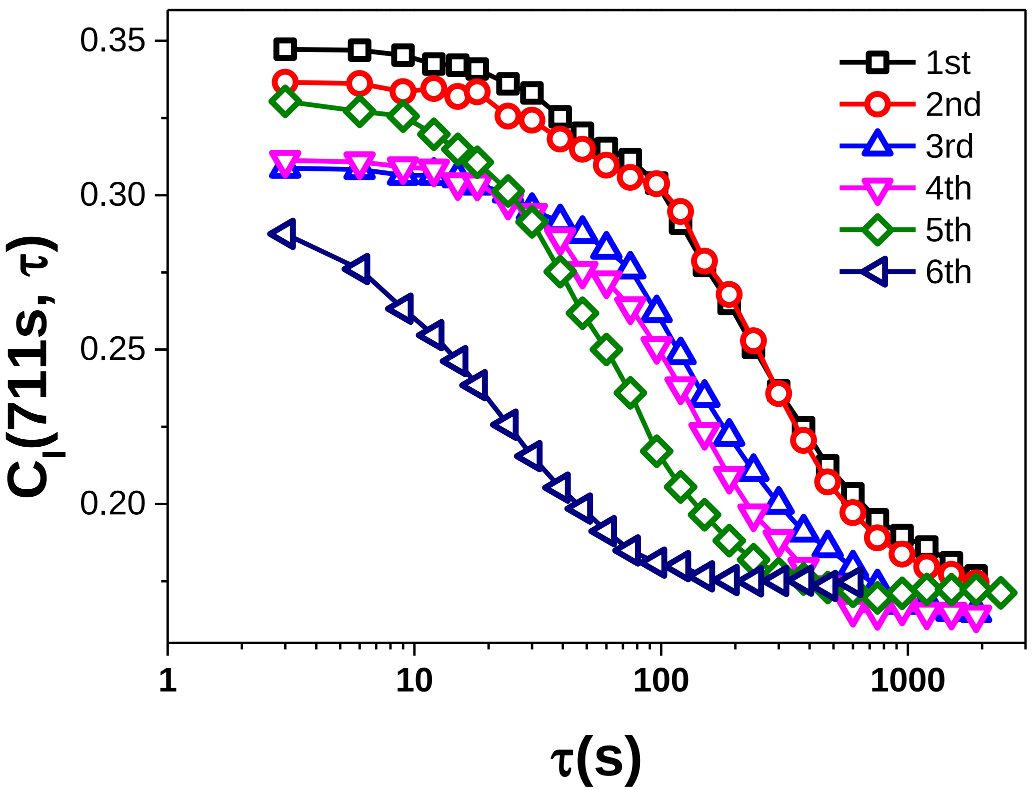
Macroscopic fracture:2400 s





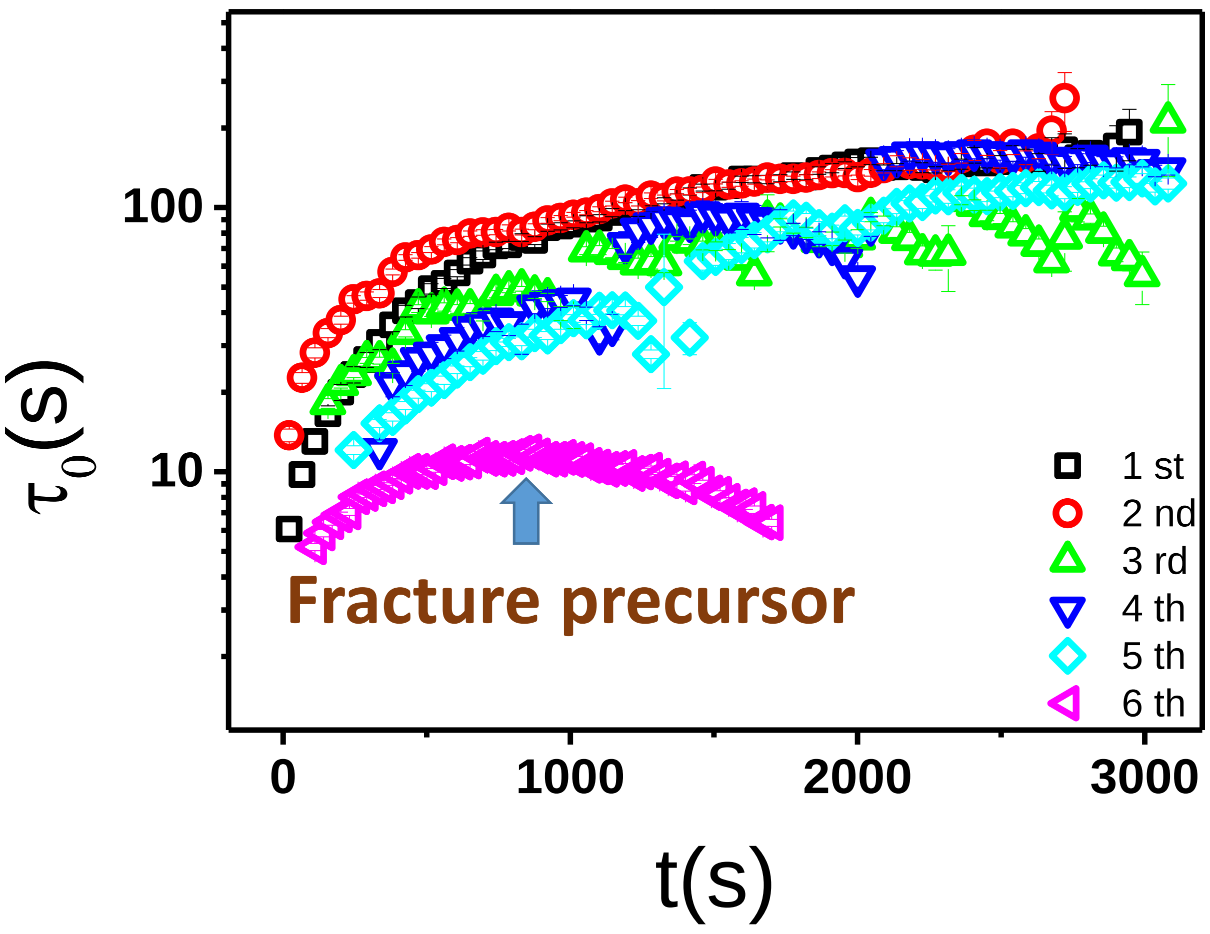
Mesoscopic measurement





$$C_I(t,\tau) = \frac{\langle I_P(t) I_P(t+\tau)\rangle_P}{\langle I_P(t)\rangle_P \langle I_P(t+\tau)\rangle_P} - 1$$

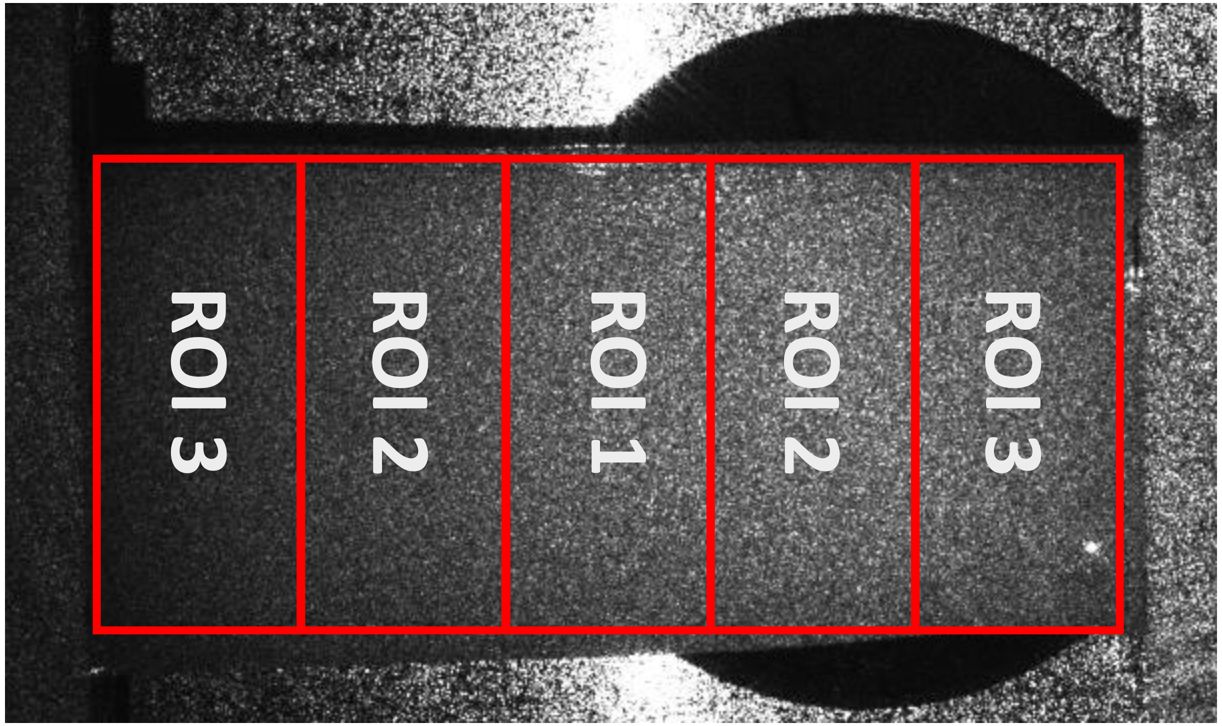
Fitting results

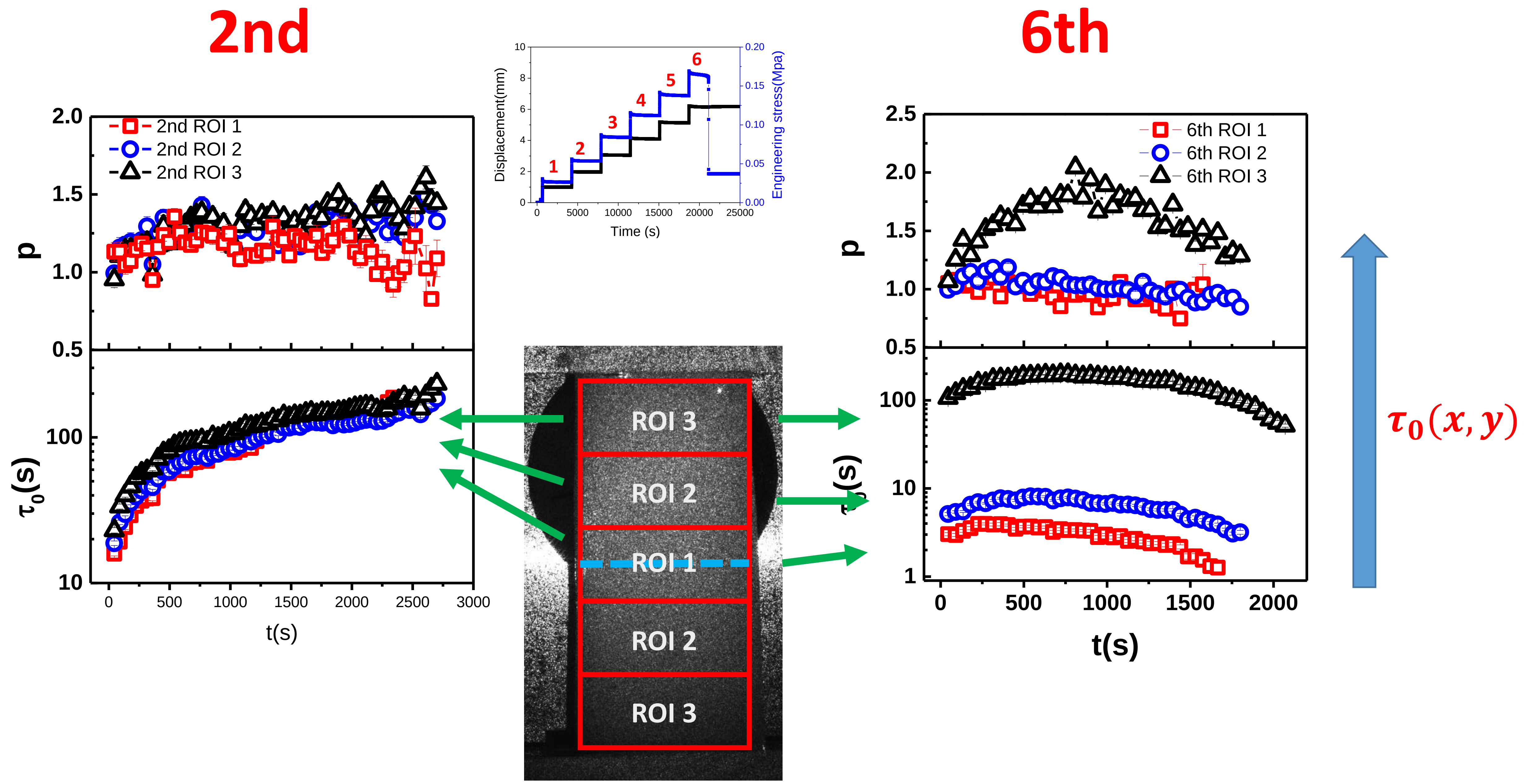


τ_{0S} =1756.6 s

$\tau_{0\rightarrow 0.1mm/s}$ = 0.18 s

Spatial distribution

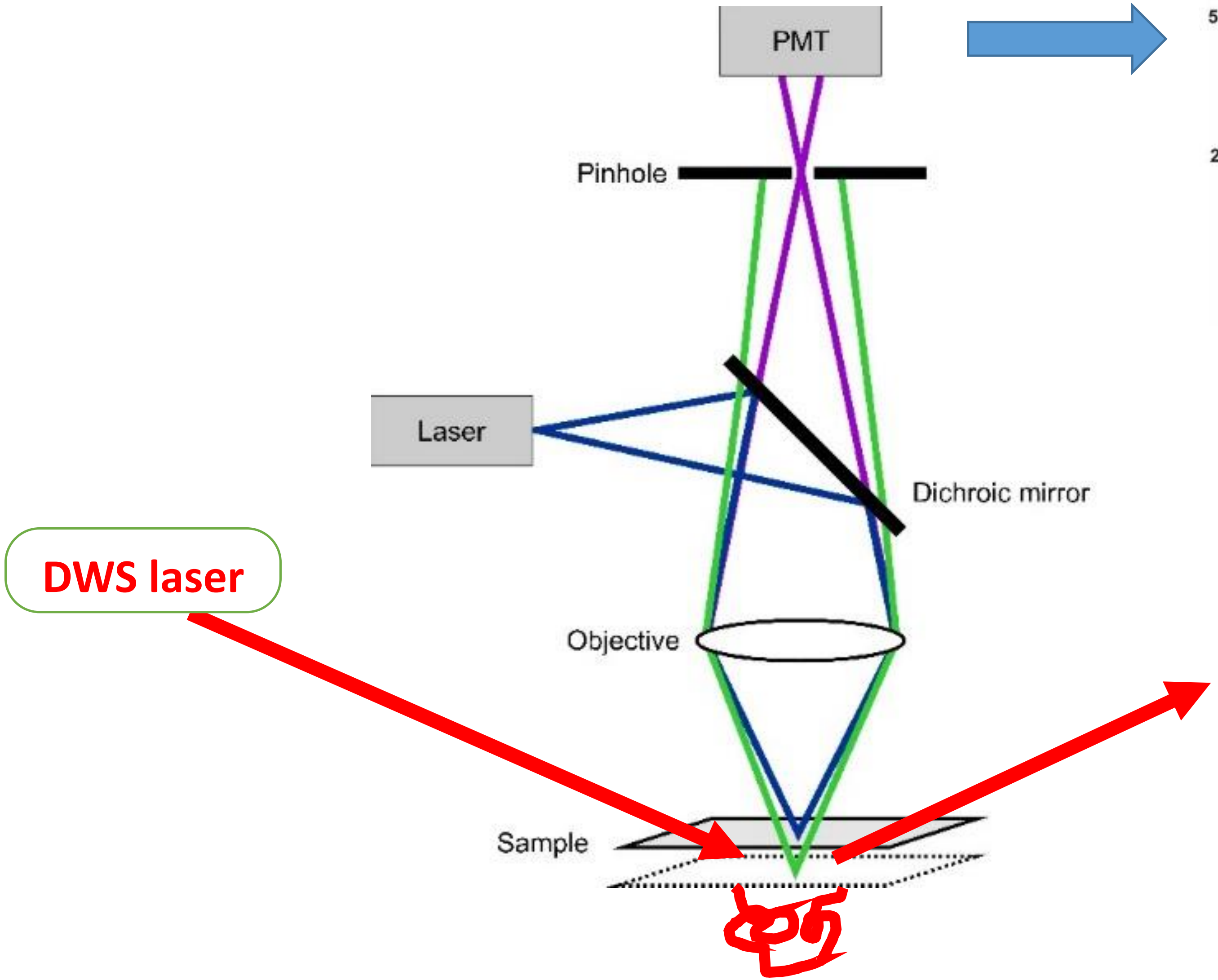


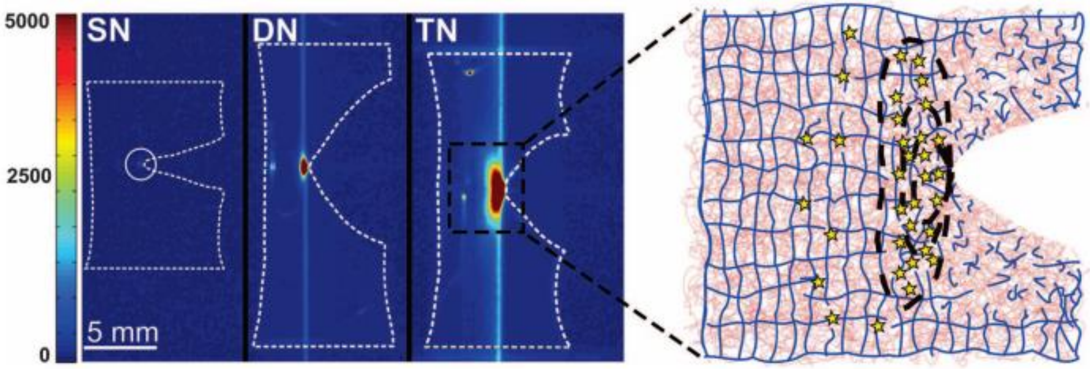

2nd
6th
2nd ROI 1
2nd ROI 2
2nd ROI 3
p
τ_0(s)
t(s)
Displacement(mm)
Engineering stress(Mpa)
Time (s)
1
2
3
4
5
6
ROI 3
ROI 2
ROI 1
ROI 2
ROI 3
6th ROI 1
6th ROI 2
6th ROI 3
$\tau_0(x, y)$

4. Conclusion

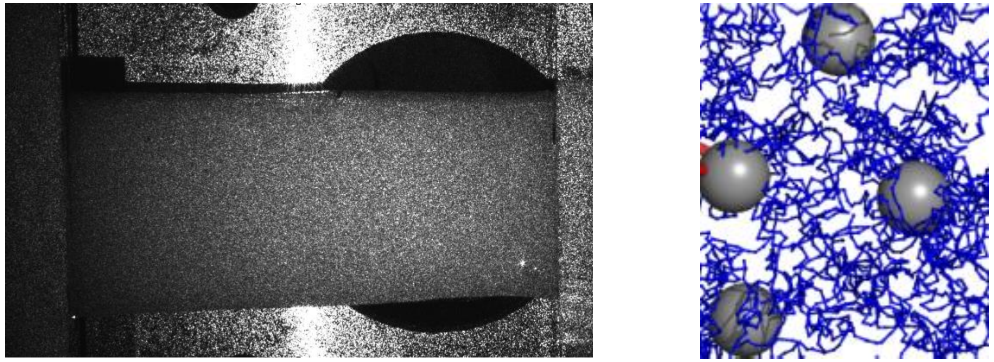
- With the combination of DWS and mechanical testing, different dynamics can be distinguished
- Fracture precursor is observed but the corresponding chain behavior is not clear
- Relaxation time of loading force and correlation function

Outlook: DWS & Mechanophore & more



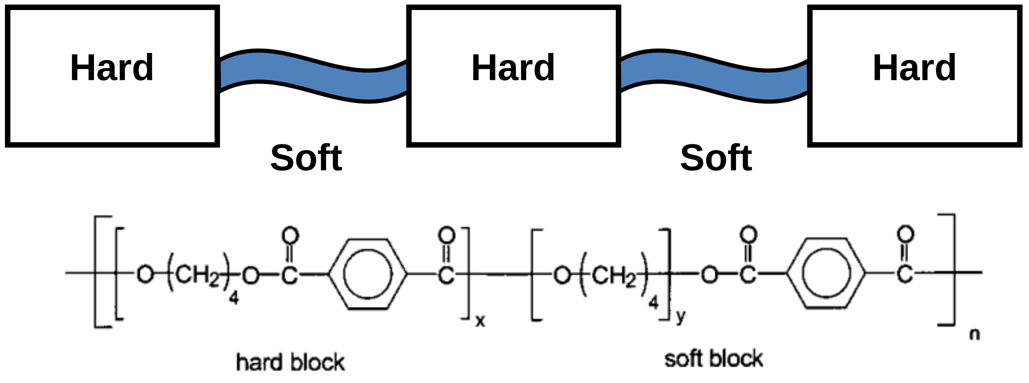


Molecular scale (~10nm)



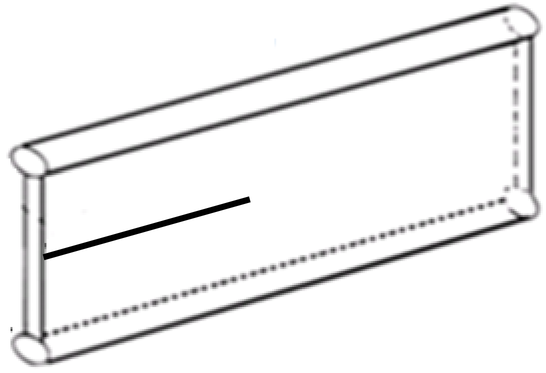
Mesoscopic scale (0,1 ~10 μm)

Fatigue testing on EL-250 (DSM)



PBT PTMO

injection: Pure Shear sample: 5x37x1mm



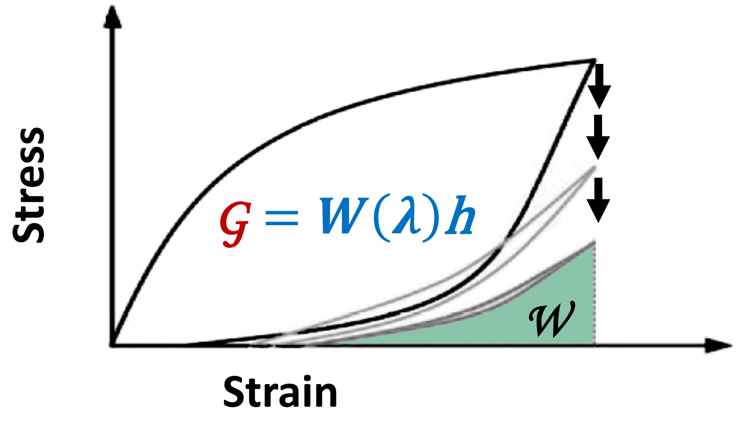
Accommodation(uncut)

Material strained to a highest strain than the maximum strain used in cyclic fatigue.

Getting rid of Mullins and other transient effects.

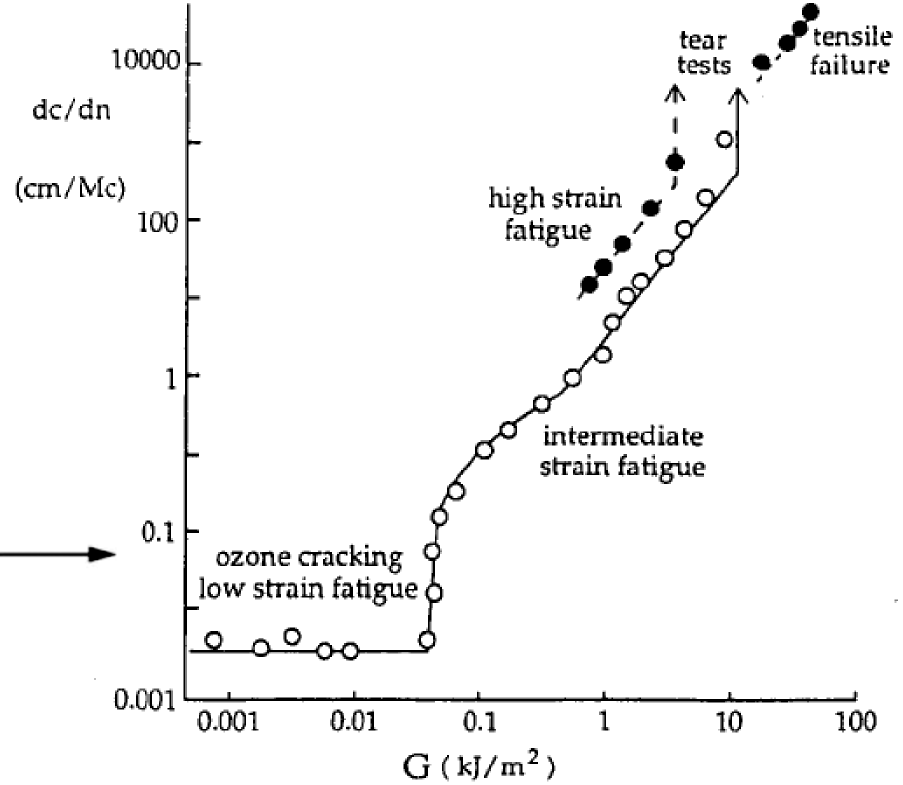
Characterization(uncut)

Evaluation of energy release rate $\mathcal{G}$ for different imposed deformations



Crack Propagation(cut)

Crack propagation rate
As a function of applied $\mathcal{G}$



Fracture property

Rate dependence
Temperature dependence
。。。

Investigation approach

Fixed G/ λ at different temperature/frequency

Thank you！