Dynamic Mechanical Analysis & Viscoelasticity

Corso di Biomeccanica dei Tessuti
25/11/2019 – 16/12/2019

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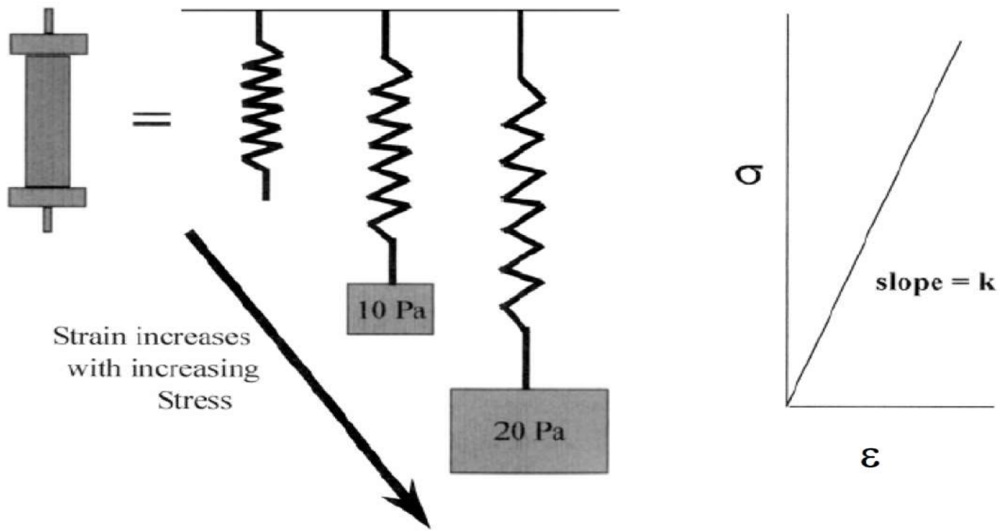
Elastic Solids



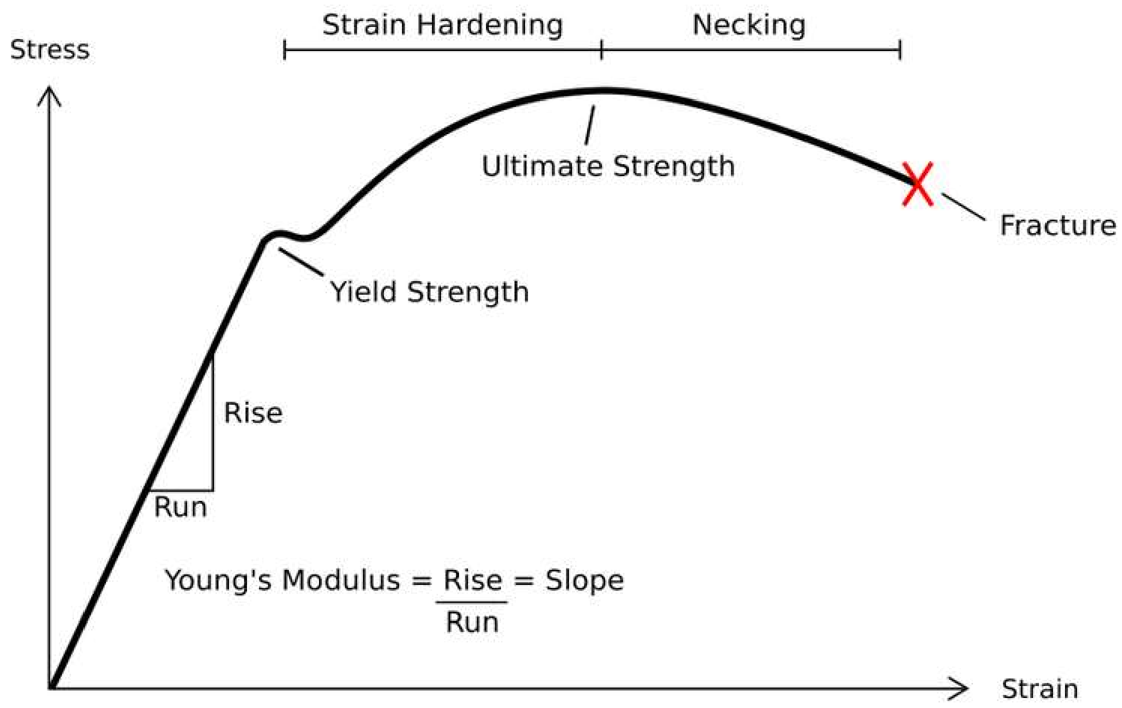
Stress is directly proportional to deformation:

$$\sigma = E \cdot \varepsilon$$

The **elastic modulus** (E) represents the resistance of a material to deformation (**stiffness**). The reciprocal of E (J) is known as **compliance**.

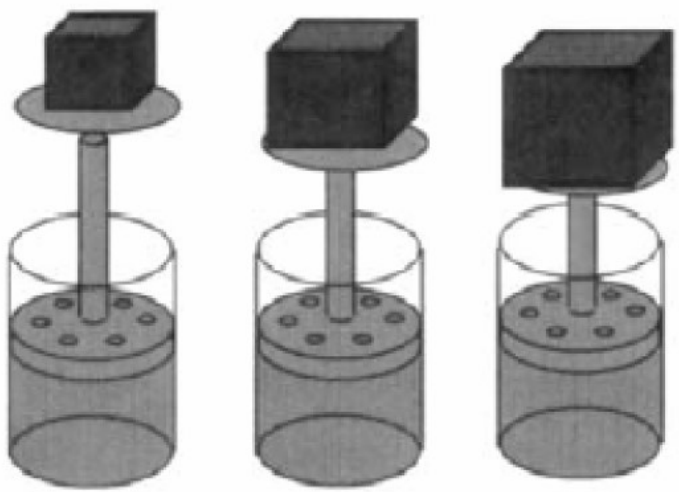
$$J = \frac{1}{E}$$

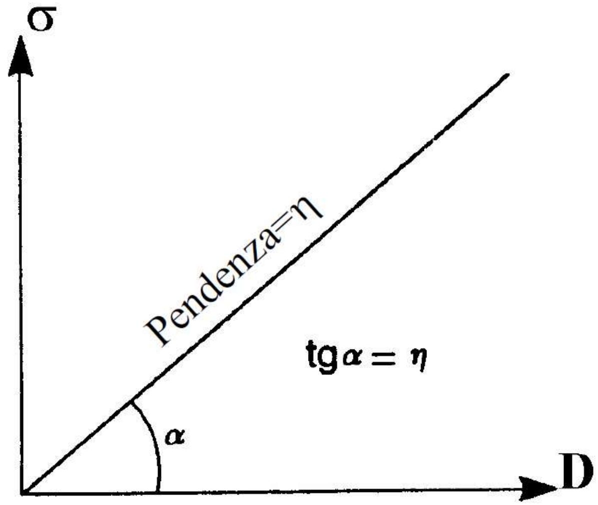
Elastic response



An elastic material has a linear response until a critical stress value (yield stress),
then it becomes not linear until the failure of the sample.

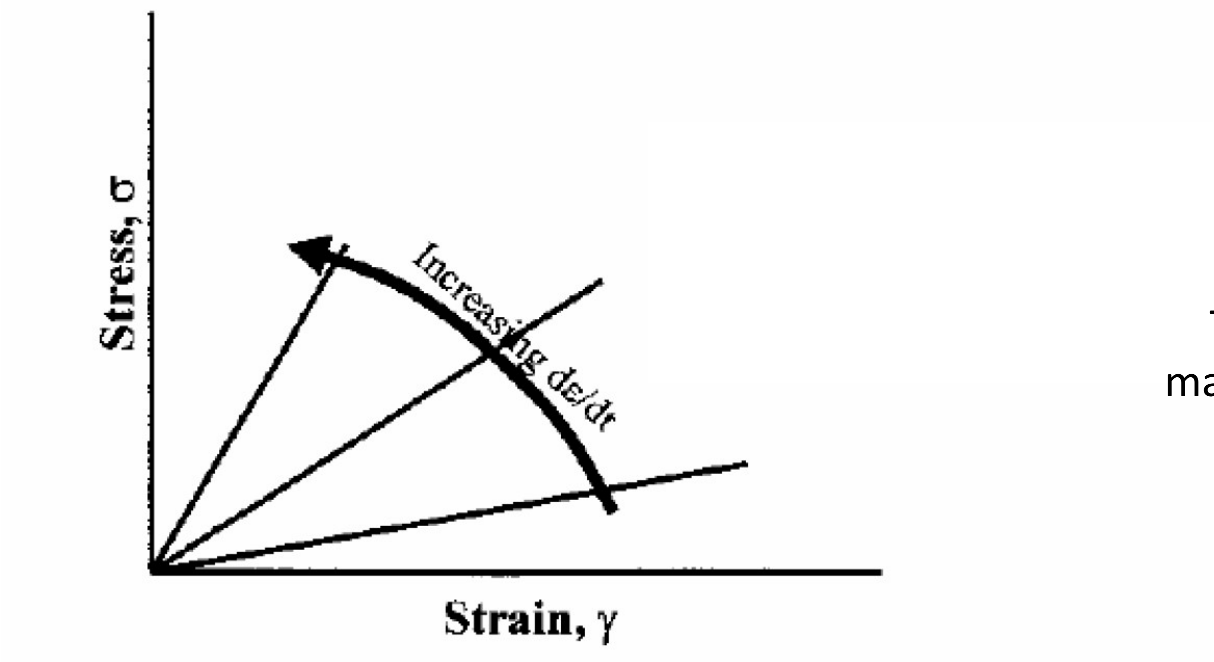
Viscous liquids





$$\sigma = \eta \cdot \frac{dV}{dy}$$

Viscoelastic materials

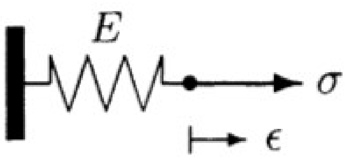


Time dependency:
The **apparent stiffness** of the material increases with increasing testing velocity

Lumped parameter models

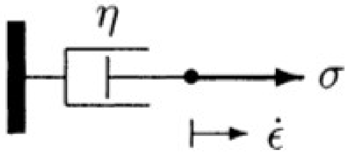
SPRING: ELASTIC SOLID

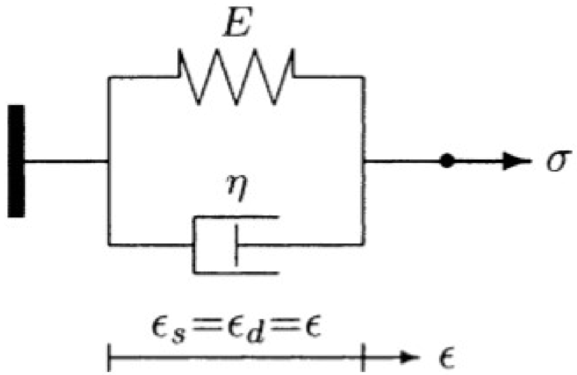
$$\sigma = E\,\epsilon$$



DASHPOT: VISCOUS FLUID

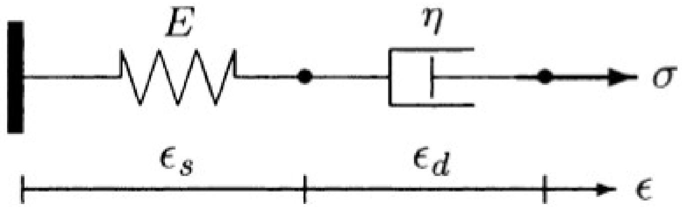
$$\sigma = \eta\,\dot{\epsilon}$$





Kelvin–Voight model

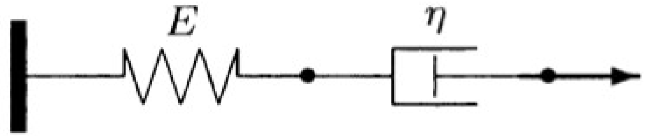
$$\sigma = E\varepsilon + \eta\frac{\mathrm{d}\varepsilon}{\mathrm{d}t}.$$

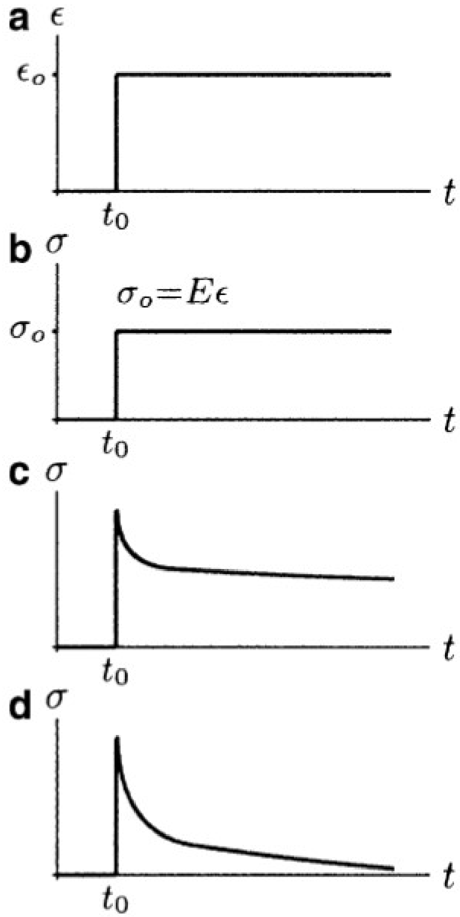


Maxwell model

$$\frac{d\varepsilon}{dt} = \frac{1}{E}\frac{d\sigma}{dt} + \frac{\sigma}{\eta}$$

Stress Relaxation





Stress relaxation

Stimulus = **strain step ε_0** (a)

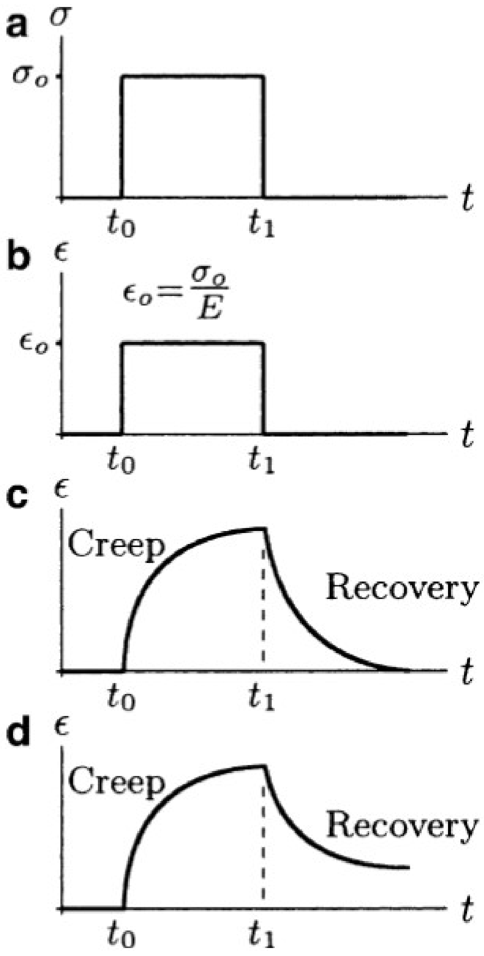
Response:
- (b) elastic material -> constant stress
- (c) viscoelastic solid -> **initial high stress that will decrease over time**, but stress level will **never reduce to zero**
- (d) viscoelastic liquid -> initial high stress that will decrease over time, and the stress will **eventually reduce to zero**

$$\sigma(t) = \varepsilon_0 E e^{-tE/\eta}$$

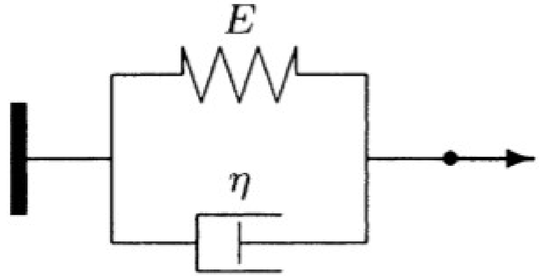
Relaxation time (τ_{SR}): The force drops to 1/e of its initial value

i.e. when $t=\tau$, $\sigma(\tau) = \sigma_0 e^{-1} = \sigma_0 * 0.33$

Creep



Creep and recovery



Stimulus = **stress step σ_0** (a)

Response:

- (b) elastic material -> **constant strain** at time t0. At time t1, the material will instantly and **completely recover the deformation**.

- (c) viscoelastic solid -> a **strain gradually increasing** between times t0 and t1. At time t1, gradual recovery will start. The *recovery will eventually be complete.*

- (d) viscoelastic liquid -> complete recovery will never be achieved and there will be a **residue of deformation** left in the material

$$\varepsilon(t) = \frac{\sigma_0}{E}(1 - e^{-tE/\eta})$$

Retardation time (τ_C): The strain achives to (1-1/e) of its final value

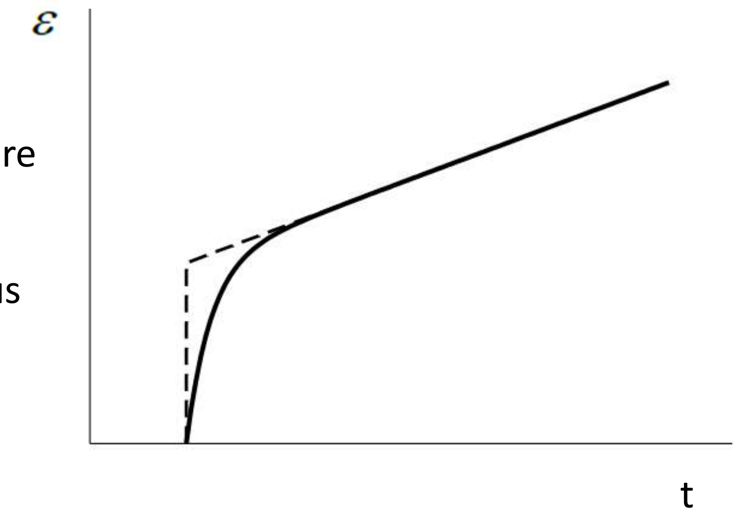
i.e. when t=τ, $\varepsilon(\tau) = \varepsilon_{equilibrium}(1-e^{-1}) = \varepsilon_{equilibrium}*0.67$

Creep and SR equations

- Creep -> Voigt

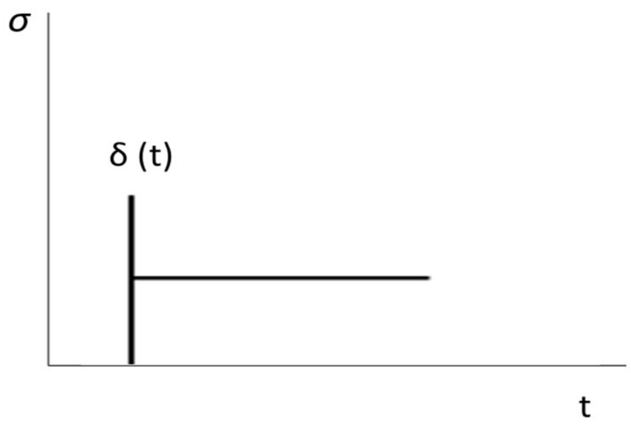
  because Maxwell does not describe correctly creep answer: the answer is more edgy and **does not describe the transition between short time** (elastic) **and long time behavior** (viscous)**.**
  Maxwell does not describe well all the situations in which the applied stimulus is a stress.

$$\varepsilon(t) = \sigma_0\left(\frac{t}{\eta} + \frac{1}{E}\right)$$



- SR -> Maxwell

$$\sigma(t) = \varepsilon_0\eta.\delta(t) + \varepsilon_0 E$$



Universal Testing Machine

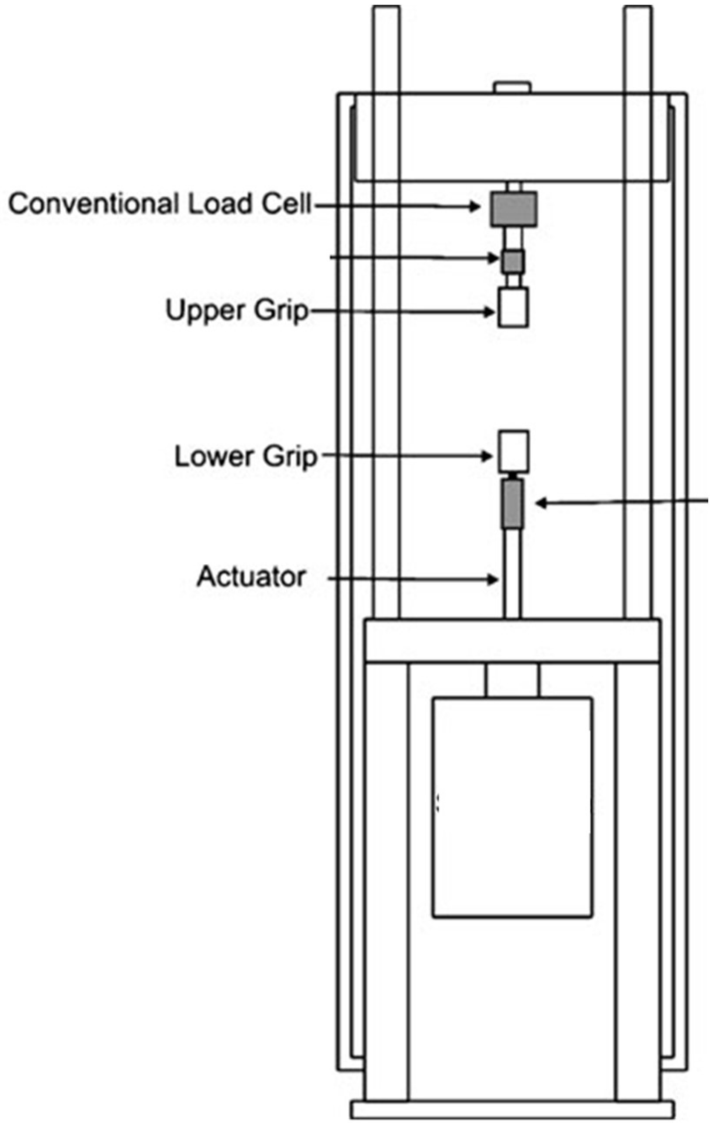
Bulk Mechanical Properties

Universal testing machines (UTM):

Compression and tensile tests

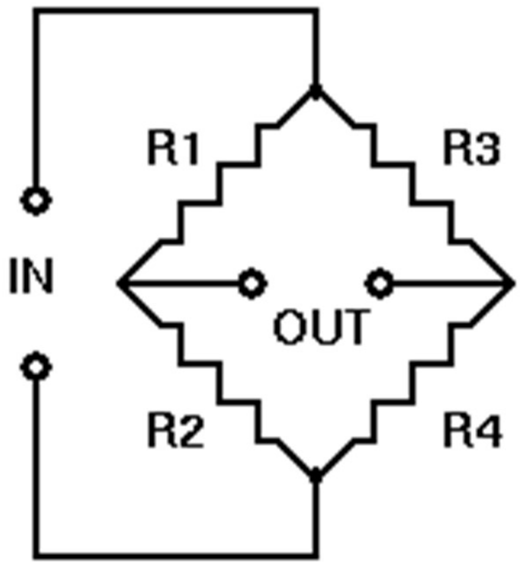
Main components:

- Load cell (different maximum loads)
- Actuator
- Sample holding system



Load cell

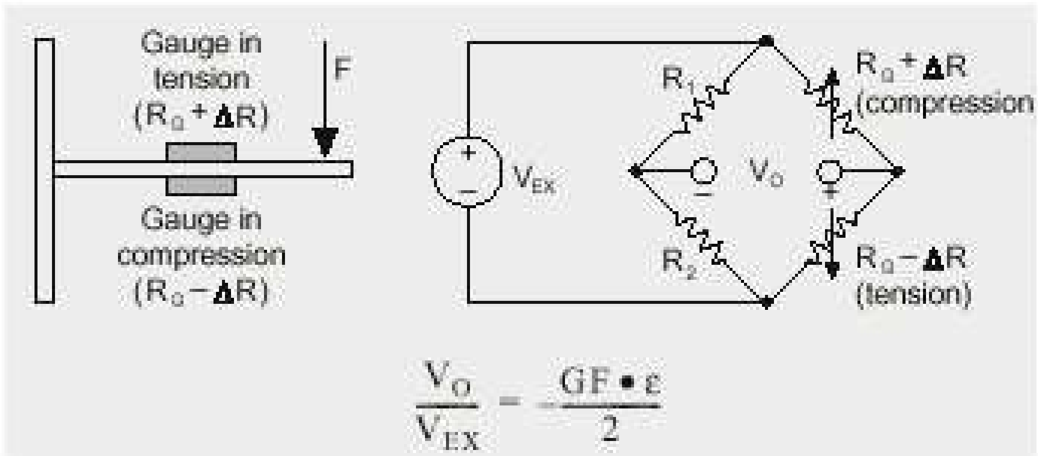
wheatstone bridge:



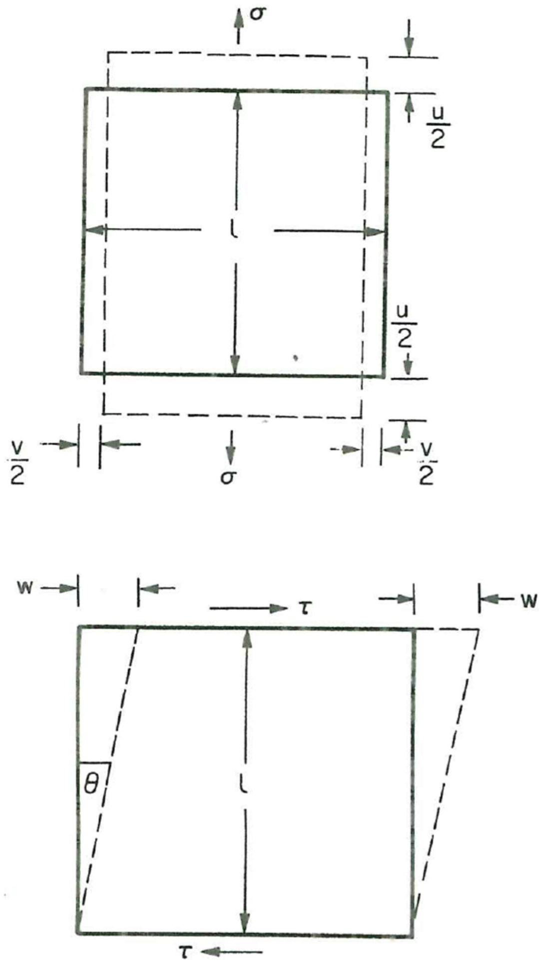
when $R1/R2 = R3/R4$ ➡ Vout=0

but if there is a change to the value of one of the resistors:

$$Vout = [(R3/(R3 + R4) - R2/(R1 + R2))] * Vin$$



Note: shear vs. elastic modulus



Nominal tensile strain,

$\epsilon_n = \frac{u}{l}$

Nominal lateral strain,

$\epsilon_n = -\frac{v}{l}$

Poisson's ratio,

$\nu = -\frac{\text{lateral strain}}{\text{tensile strain}}$

Engineering shear strain,

$\gamma = \frac{w}{l} = \tan\theta$

$$E = 2G(1-\nu)$$

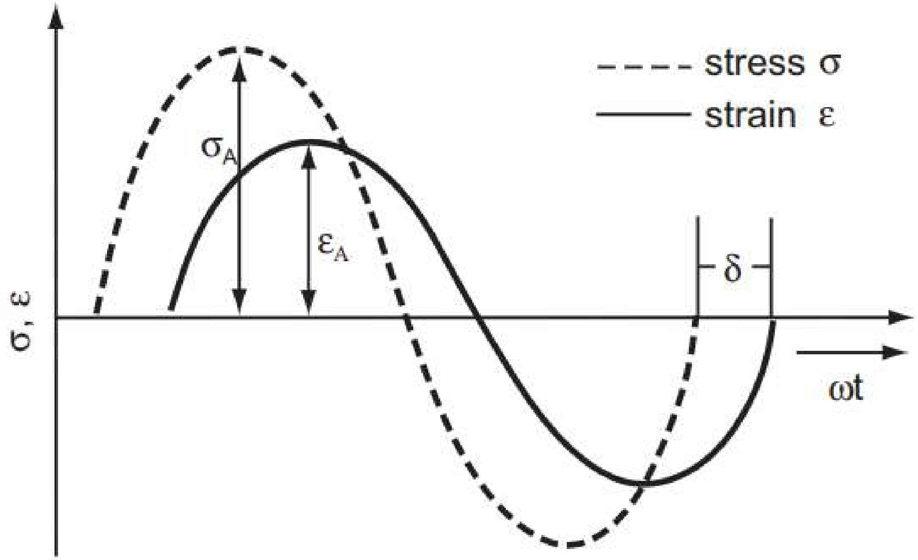
Elastic modulus

Shear modulus

$G = \tau / \gamma$

DMA

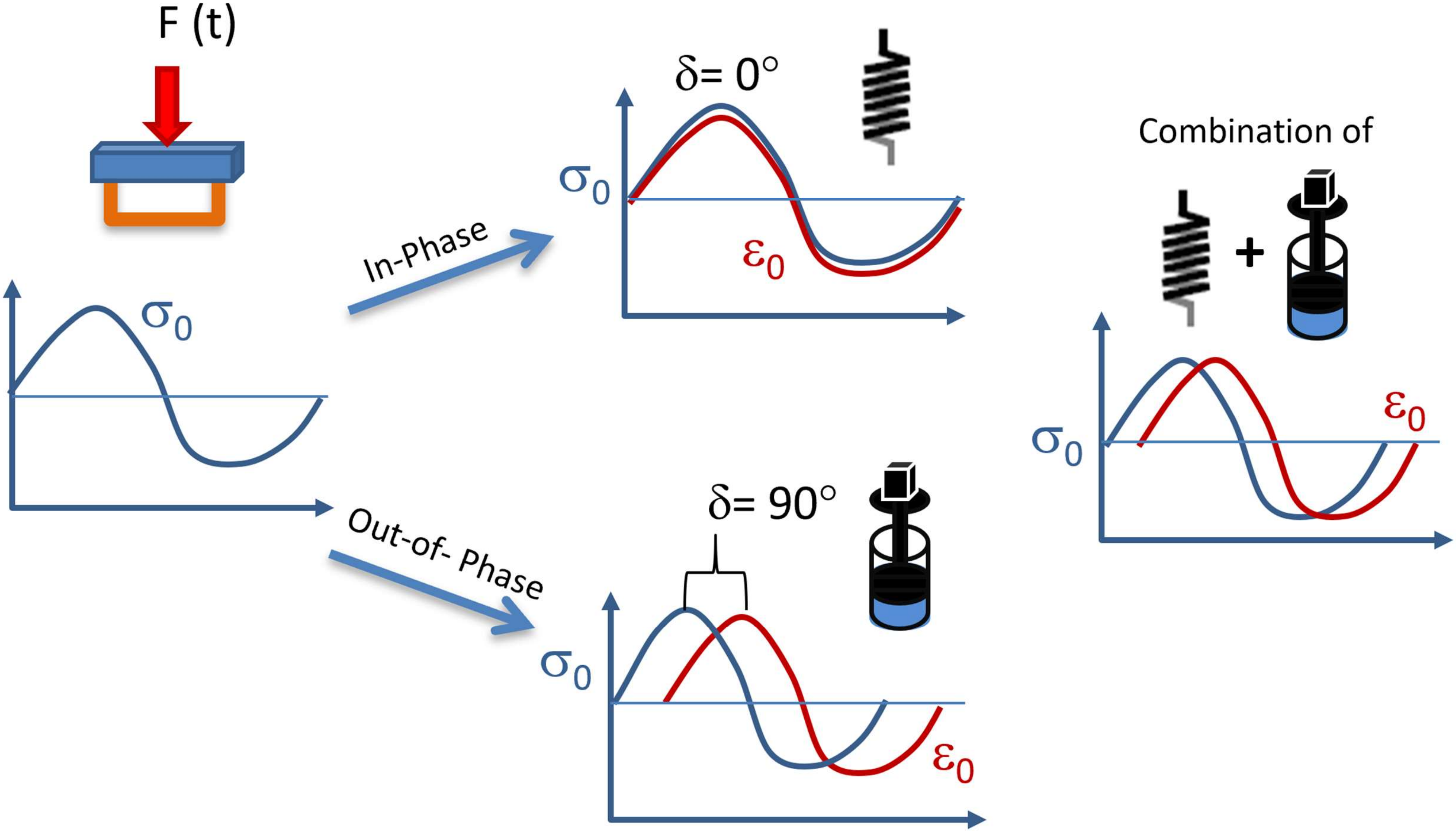
Dynamic Mechanical Analysis (DMA) is a technique where a *small deformation is applied to a sample in a cyclic manner*. This allows the **materials response to strain, temperature, frequency** and other values to be studied. (The sample can be subjected by a controlled stress or a controlled strain)



Stimulus = strain/stress sinusoid

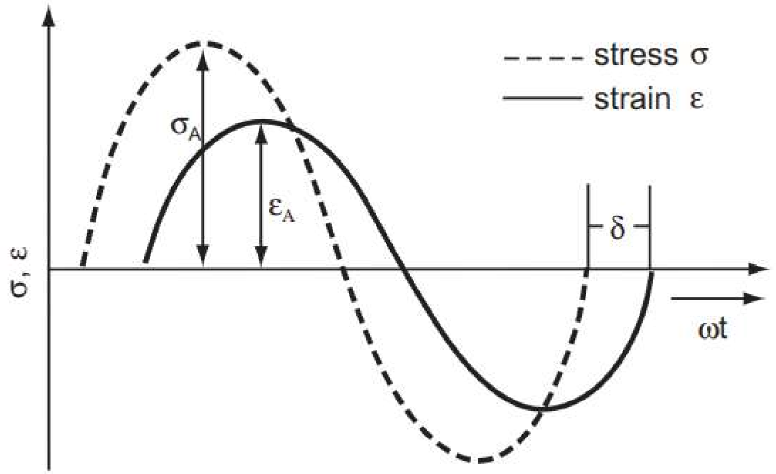
Response = stress/strain sinusoid:

- Amplitude <-> stiffness (i.e. E)
- **Delta = 0** for ideally **elastic material** (all energy stored in the material)
- **Delta = 90°** for an ideally **viscous liquid** (all energy dissipated)



F (t)
σ0
In-Phase
δ= 0°
σ0
ε0
Out-of- Phase
δ= 90°
σ0
ε0
Combination of
+
σ0
ε0

Complex Modulus



$$\sigma = \sigma o \exp(\omega t + \delta) i$$

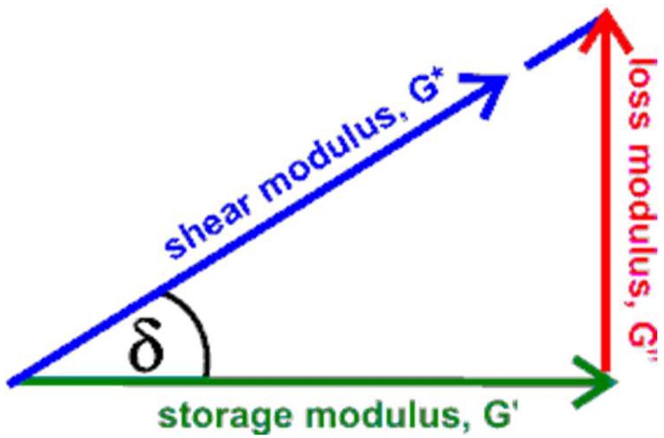
$$\varepsilon = \varepsilon_o \exp(i \omega t)$$

$$E^* = \frac{\sigma}{\varepsilon} = \frac{\sigma_o}{\varepsilon_o} e^{i\delta} = \frac{\sigma_o}{\varepsilon_o}(\cos\delta + i \sin\delta) = E' + iE''$$

$$E' = \frac{\sigma_o}{\varepsilon_o}\cos\delta$$

$$E'' = \frac{\sigma_o}{\varepsilon_o}\sin\delta$$

Complex (shear) modulus



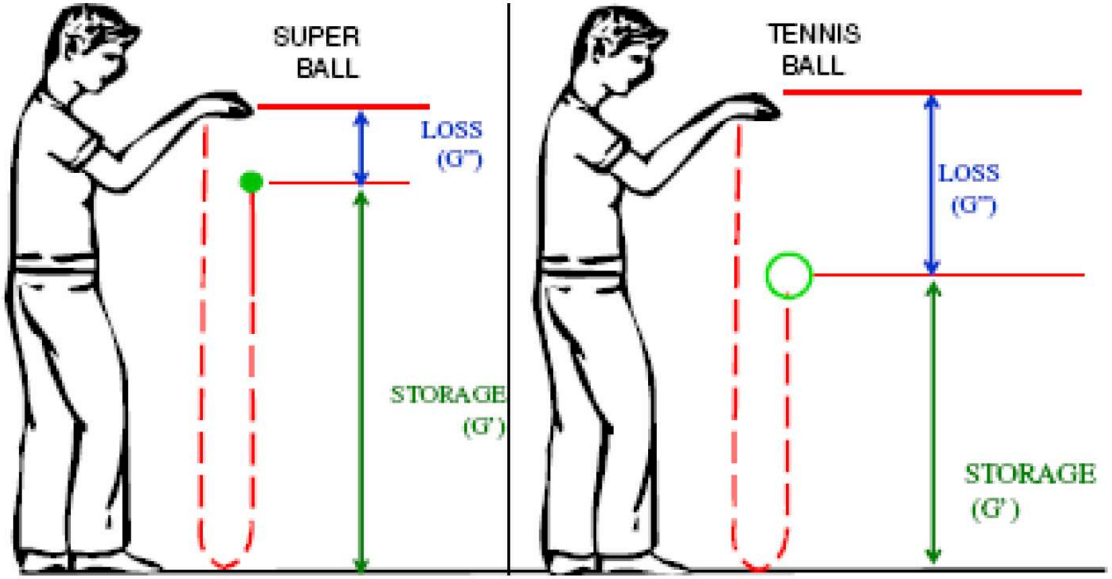
$$G^* = G' + iG''$$

$$\tan\delta = \frac{G''}{G'}$$

The **loss or damping factor** is a measure of the energy lost and represents mechanical damping or internal friction in a viscoelastic system. The loss factor tan G is expressed as a dimensionless number.

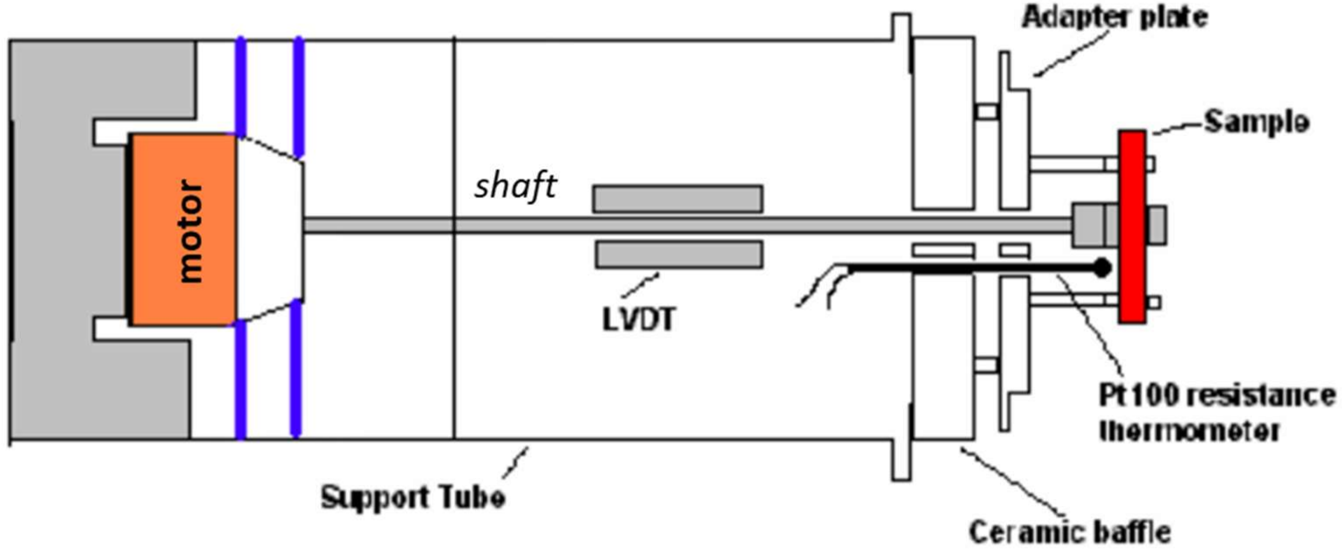
*A **high tan** value is indicative of a material that has a **high-nonelastic component**, while a low value indicates one that is more elastic.*

Storage and Loss moduli



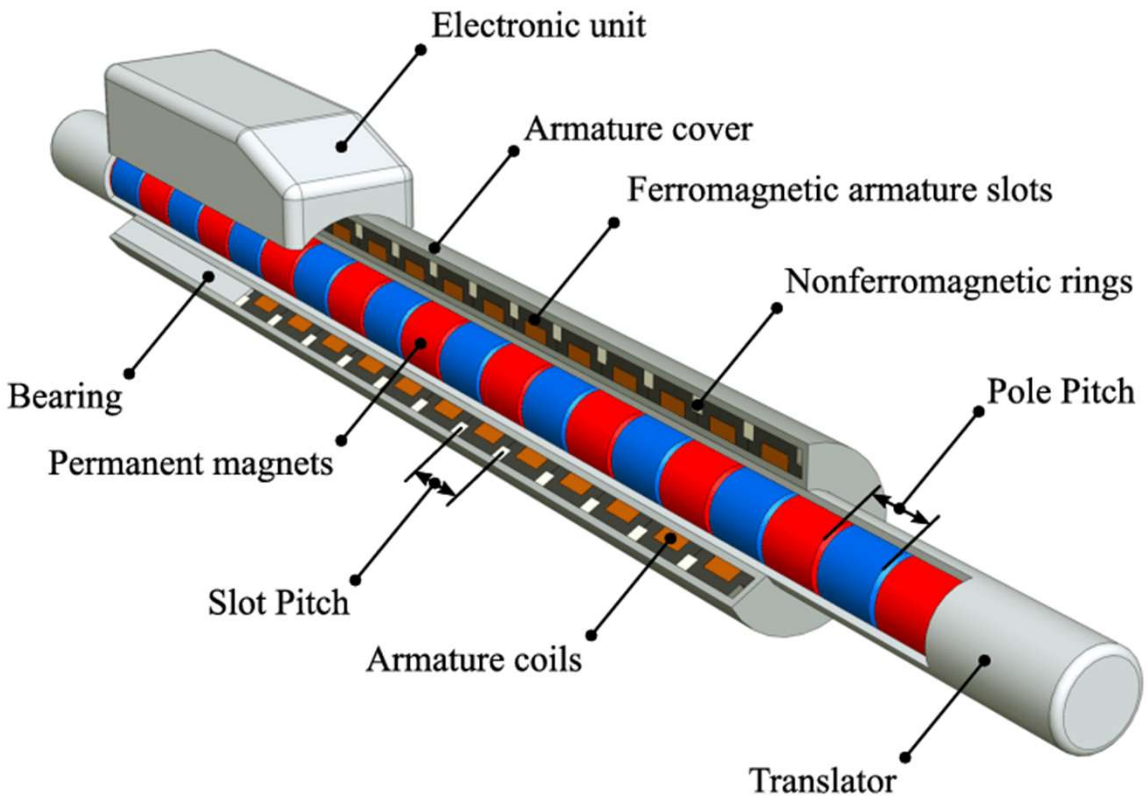
The **storage modulus** is often associated with "**stiffness**" of a material and is related to the Young's modulus, E. The dynamic **loss modulus** is often associated with "**internal friction**" and is sensitive to different kinds of *molecular motions, relaxation processes, transitions, morphology* and other structural heterogeneities.

Dynamic Mechanical Analyser

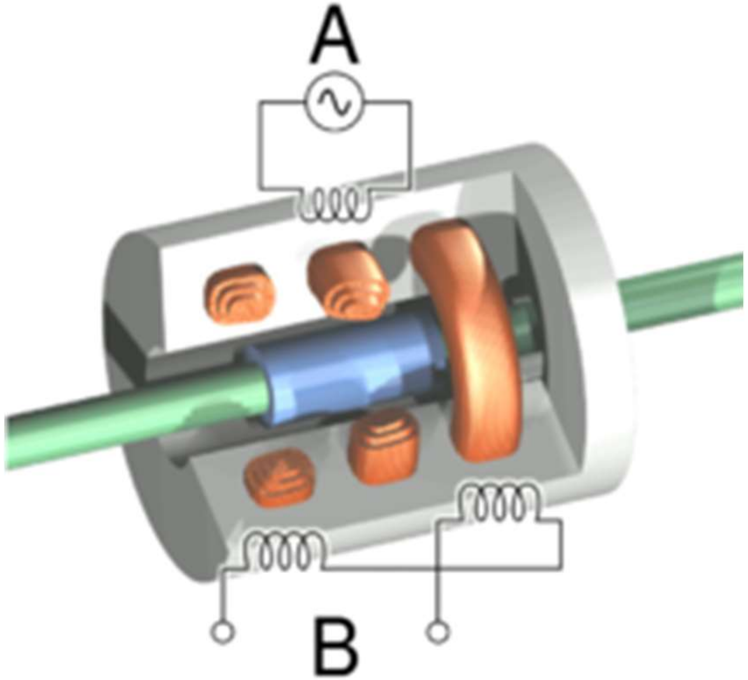


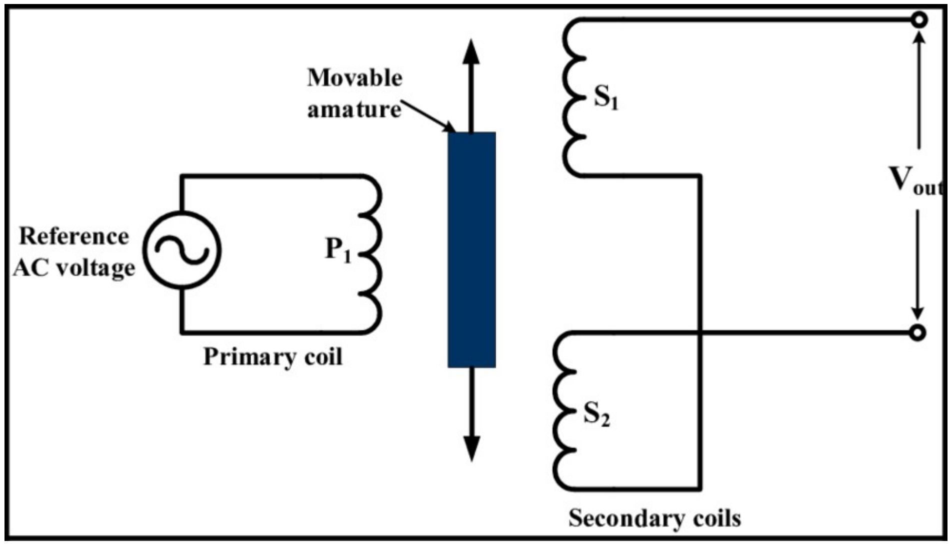
The stress is transmitted through the drive shaft onto the sample which is mounted in a clamping mechanism. As the sample deforms, the amount of displacement is measured by the LVDT positional sensor.

Non contact linear actuator

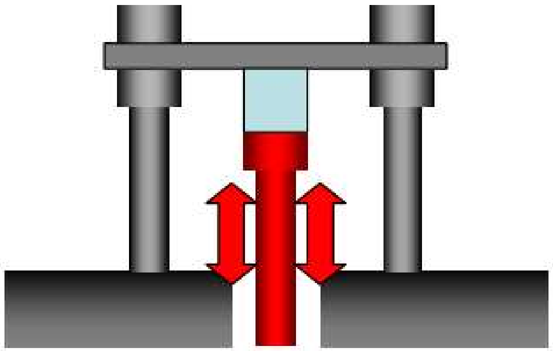


LVDT (linear variable displacement transducer)



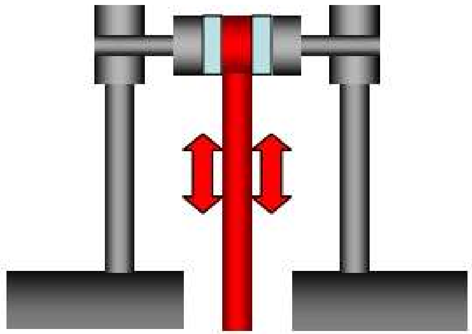


Dynamic Mechanical Analyser (2)



Compression

In compression mode an **axial load** is applied to the specimen held between two parallel plates.



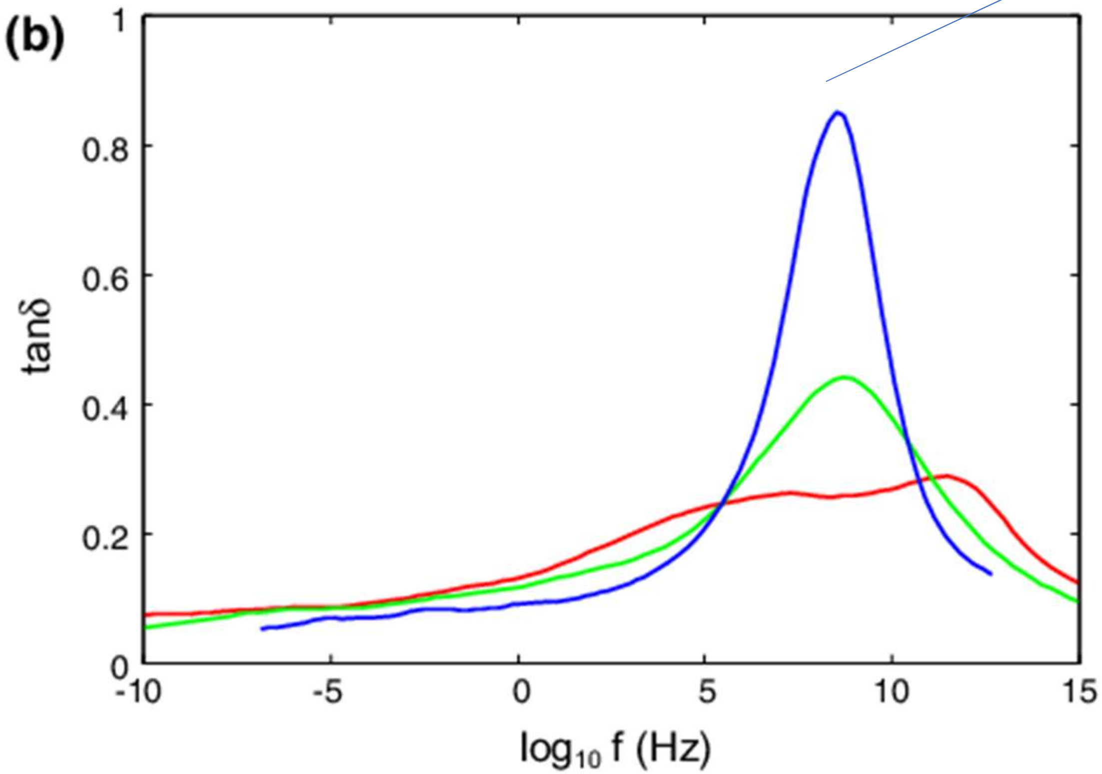
Shear

In shear mode the sample is placed in a **sandwich arrangement** between two plates and subjected to cyclical shear by the **displacement of a central push-rod**.

Frequency Sweep & Relaxation time

(constant Temperature)

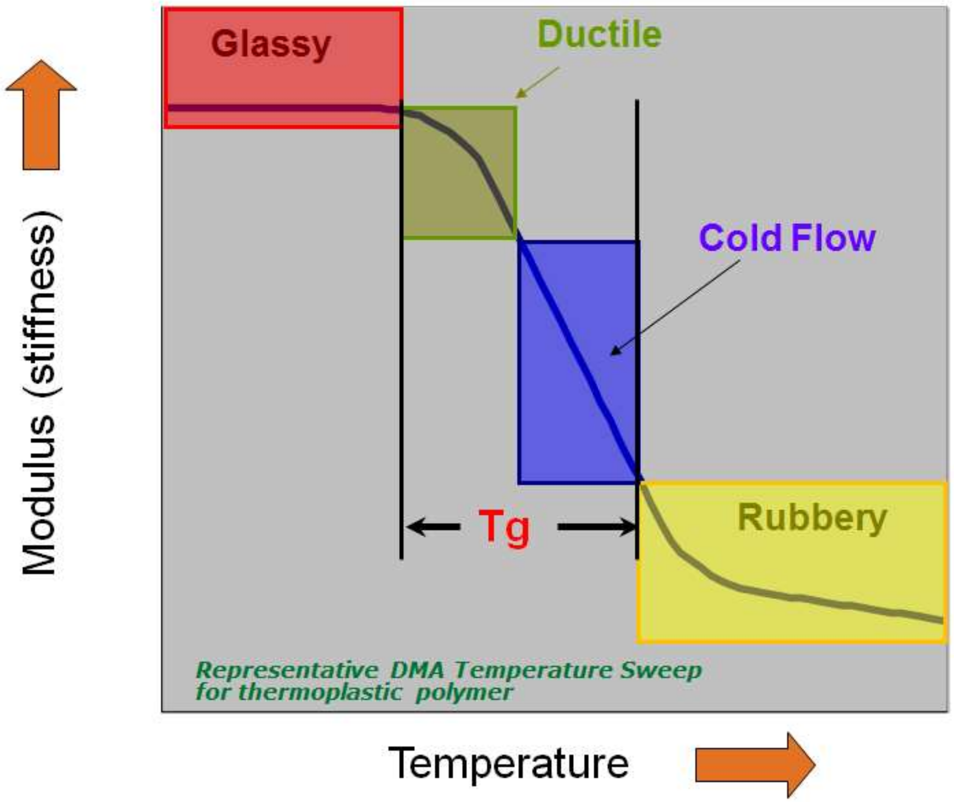
Relaxation frequency: from tanδ or E'' peak

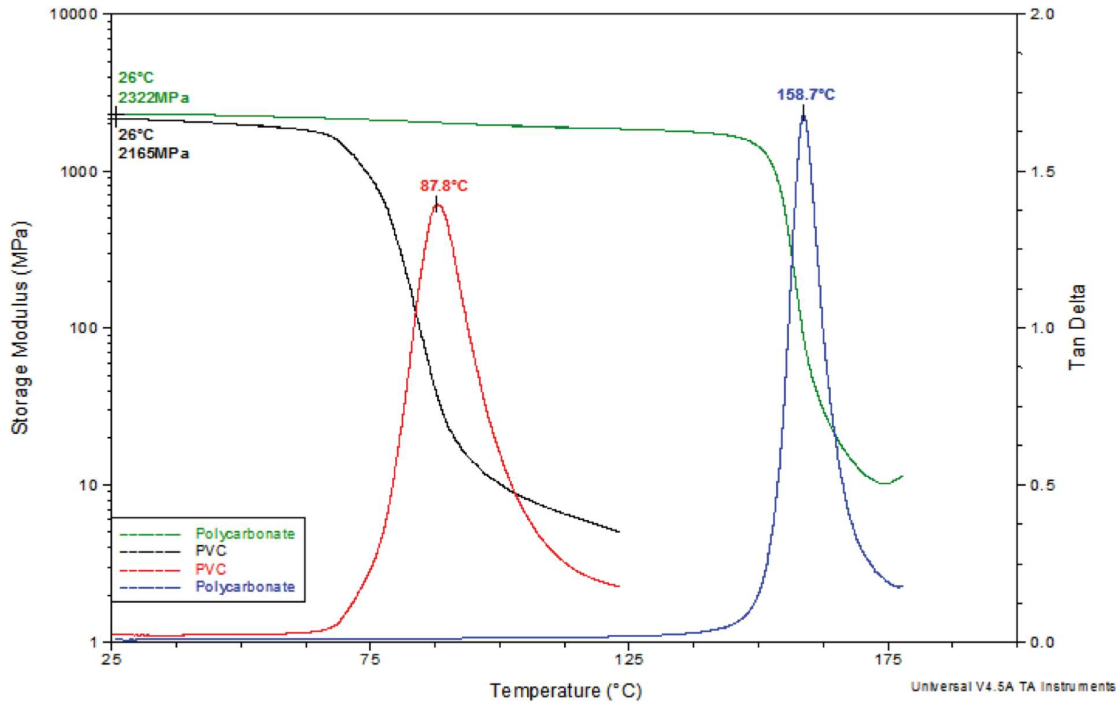


$$f = \frac{1}{\tau}$$

Relaxation time

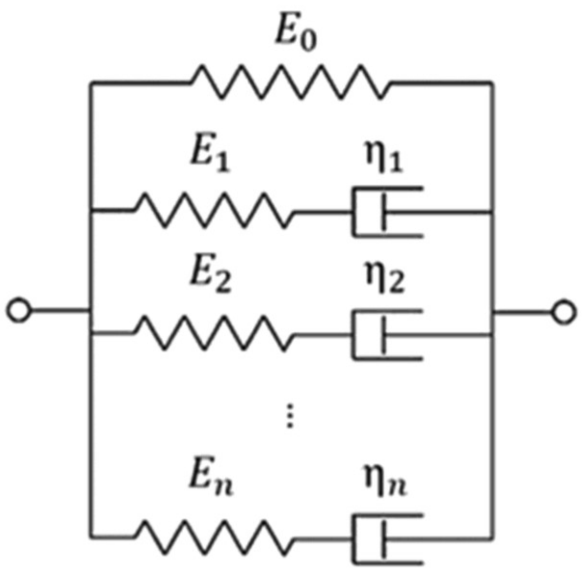
Temperature Sweep



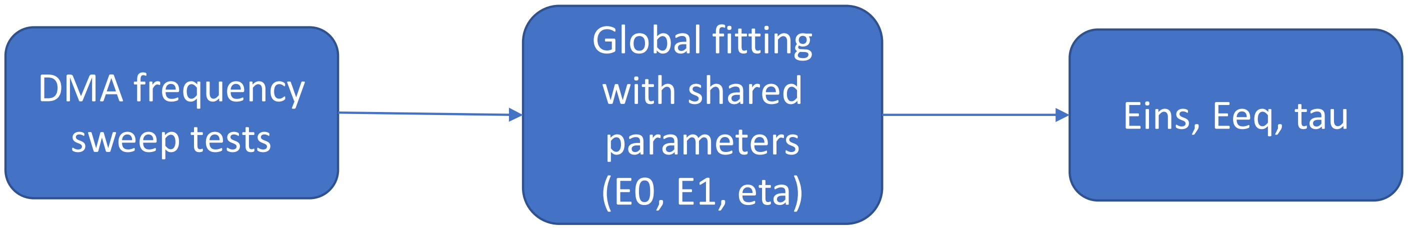


A **peak in tan_delta** appears at the **glass transition temperature (Tg)** of the polymer.

DMA & Lumped parameter models



$$E^*(f) = \left(E_0 + \sum_{i=i}^{n} \frac{4\, E_i\, \eta_i^2\, f^2\, \pi^2}{E_i^2 \,+\, 4\, \eta_i^2 f^2 \pi^2} \right) + i \left(\sum_{i=i}^{n} \frac{2\, E_i^2\, \eta_i\, f\, \pi}{E_i^2 \,+\, 4\, \eta_i^2 f^2 \pi^2} \right)$$



Articolo StepReconstructed DMA

- Qual è il pricipale vantaggio del metodo rispetto al DMA classico?
- Riflettere sulla struttura del paper

In generale per una tesi sperimentale inspiratevi a Research Articles, per una compilativa alle Review

ELSEVIER

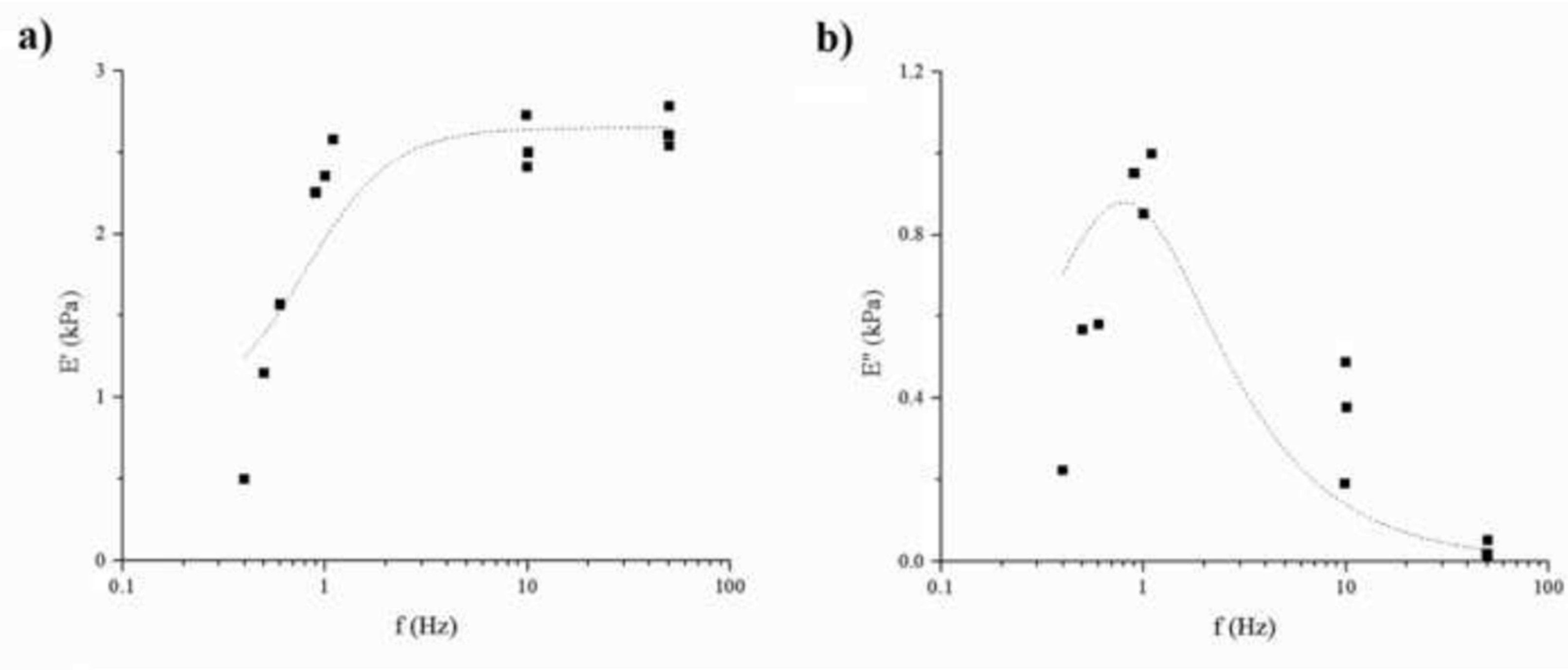
Journal of Biomechanics

Volume 47, Issue 11, 22 August 2014, Pages 2641-2646

Viscoelastic characterisation of pig liver in unconfined compression

G. Mattei [a, b], A. Tirella [a, c], G. Gallone [a, b], A. Ahluwalia [a, c]

SRDMA

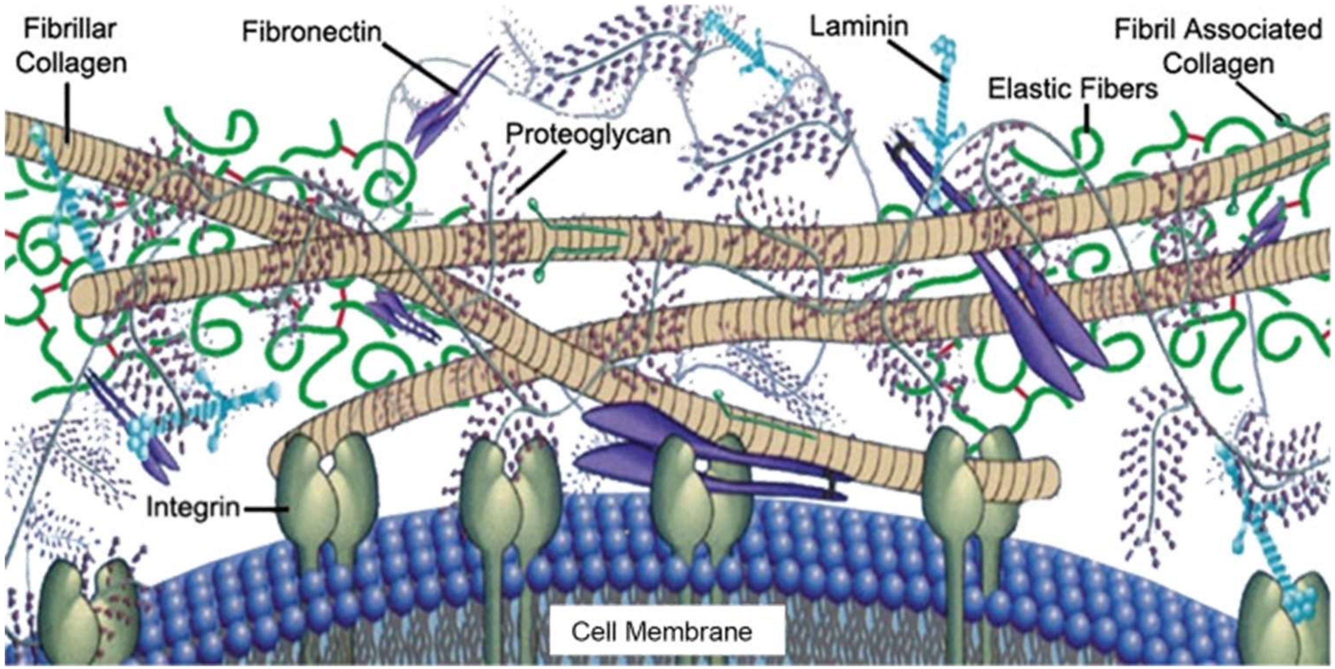


step-reconstructed DMA (SRDMA) is based on dynamic measurements around specific frequencies and then reconstruction of the mechanical behaviour in the entire frequency range of interest

Tissues from an Engineering Point of View

ECM – *biochemical composition and functions*

The ECM is generally composed of **water, proteins and polysaccharides**, even if the composition and topology is tissue-specific.



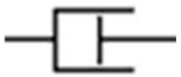
Frantz et al, J. Cell. Sci.., 2010

- **RGD** (Arg-Gly-Asp) sequence -> fundamental for cell adhesion
- **Nutrient and oxygen** diffusion

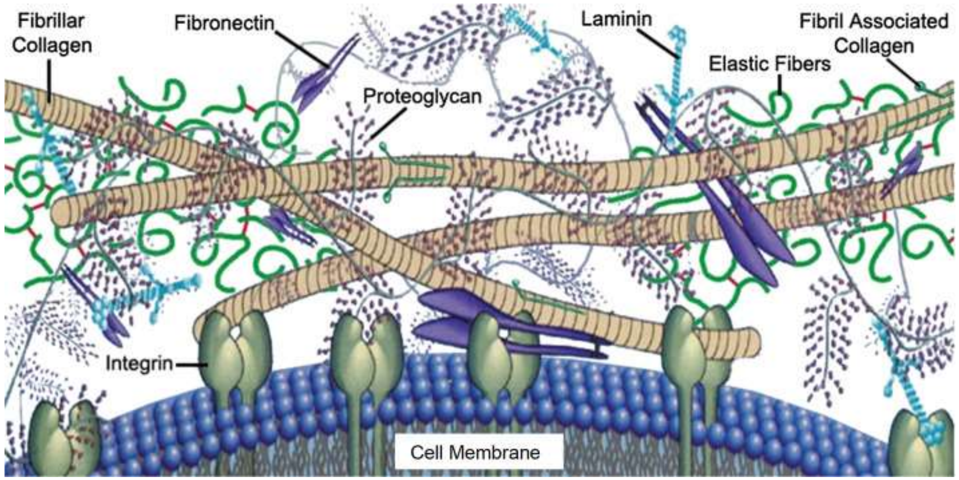
Tissues from an Engineering Point of View

ECM – *mechanical functions*

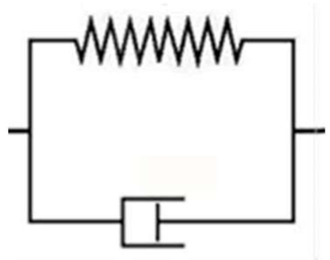
Elastic network

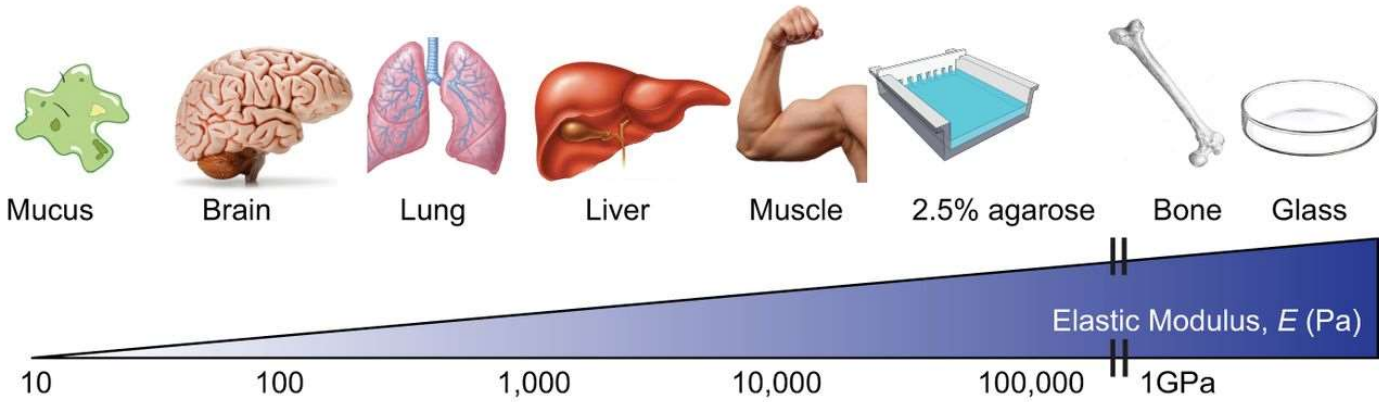


Liquid phase



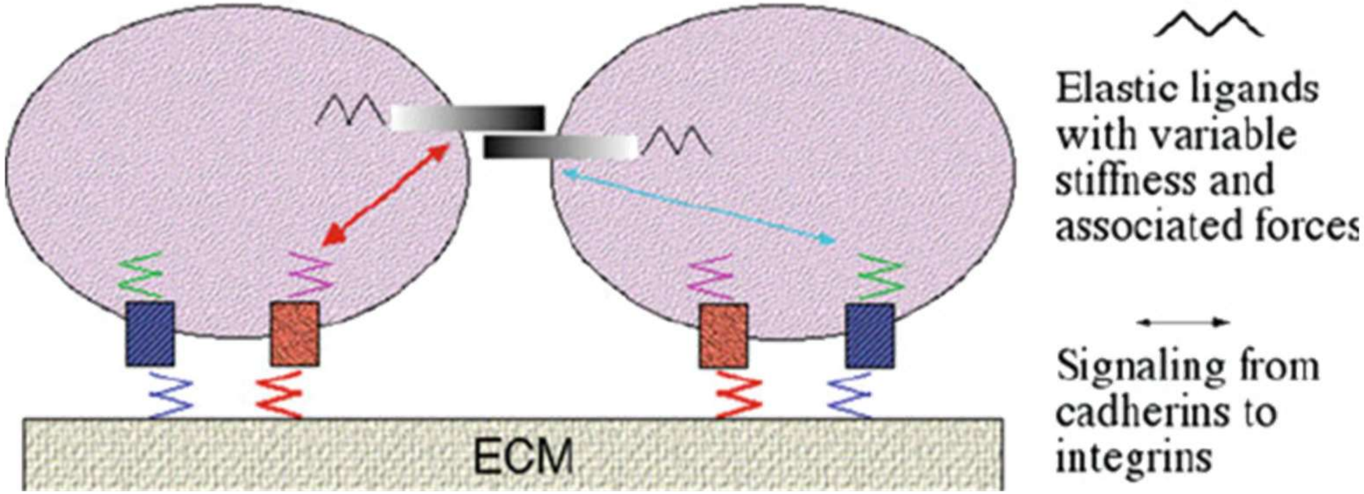
Viscoelastic behaviour





Tissues from an Engineering Point of View

Cells and Extracellular Matrix (ECM)



Jones et al, Mechanobiology, 2011

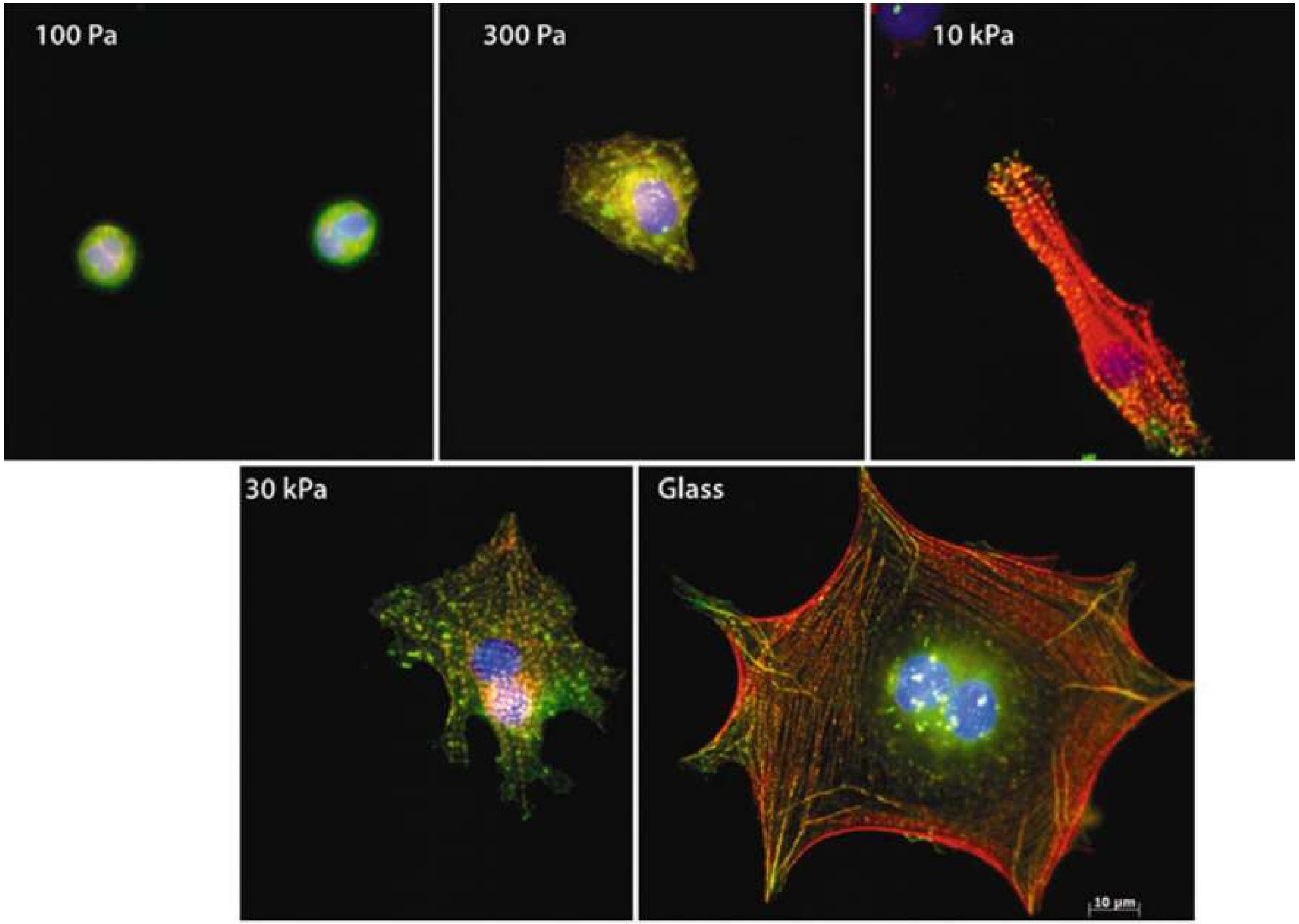
The ECM provides:

- essential **physical scaffolding** for the cells
- **biochemical and biomechanical cues** that are required for tissue morphogenesis, differentiation and homeostasis.

Tissues from an Engineering Point of View

ECM – *mechanical functions*

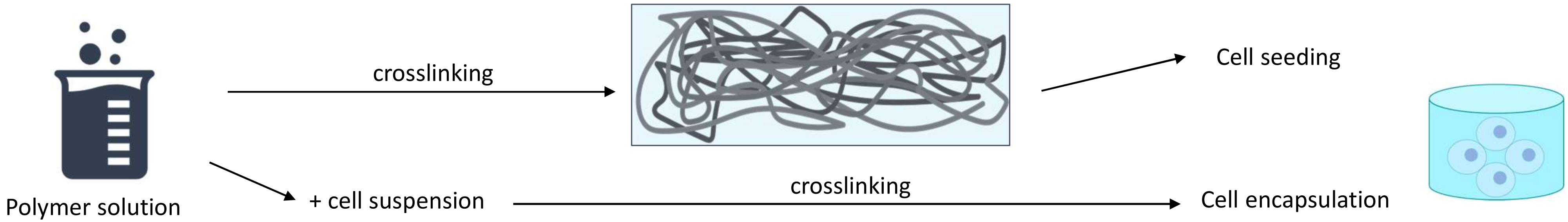
Jones et al, Mechanobiology, 2011



Engineered Tissues

Scaffolds

Hydrogels are composed of highly **hydrophilic polymers**, capable of holding large amounts of water in their three-dimensional networks (*Natural polymers: alginate, collagen, gelatin, agarose, etc. Synthetic polymers: PEG, PVA, PCL, PAAM, PU, etc)*



Hoffman, Adv. Drug Del. Rev. J.., 2001

Engineered Tissues

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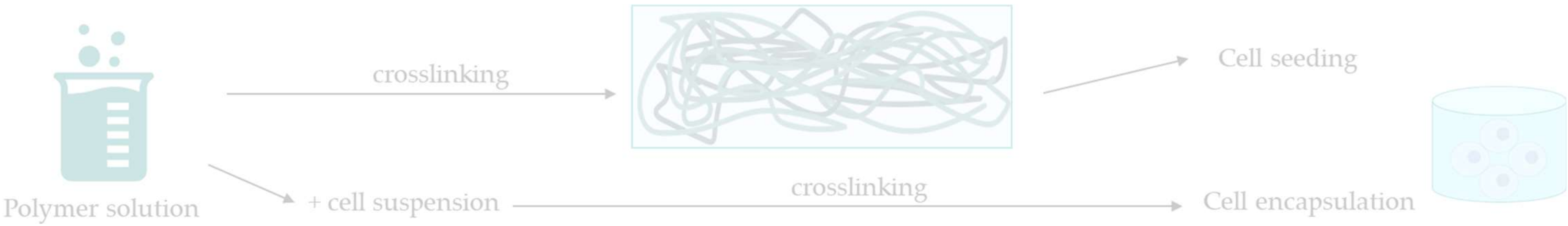
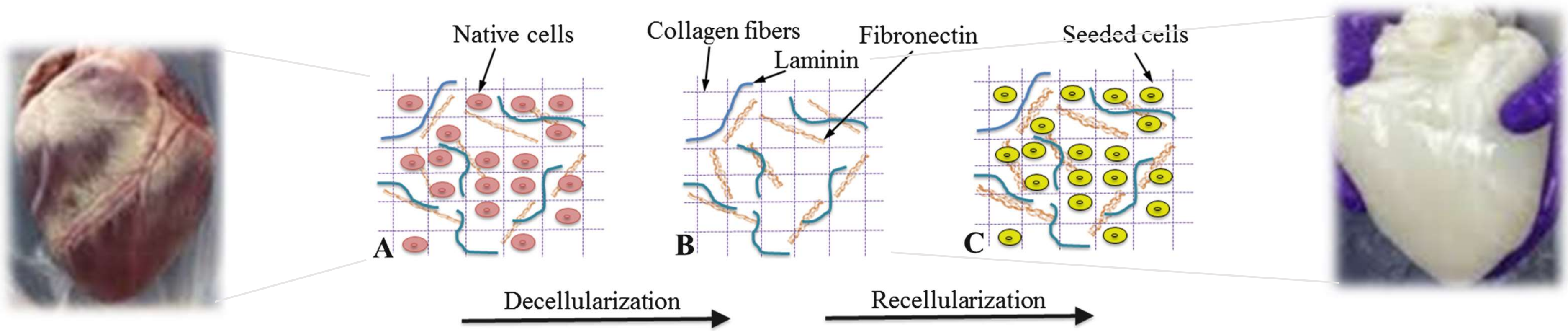


Tissue-derived Scaffolds



Hoffman, Adv. Drug Del. Rev. J.., 2001

Decellularization

& digestion

Decellularization maintains *microstructures of native extracellular matrices and its biochemical compositions*, providing tissue-specific microenvironments for efficient tissue regeneration.

Digestion is necessary to *solubilize decellularized ECM* (i.e. breaks down proteins into smaller peptides).

Lee et al, Biomacr., 2014

Decellularization

Example: Liver Decellularization & Digestion

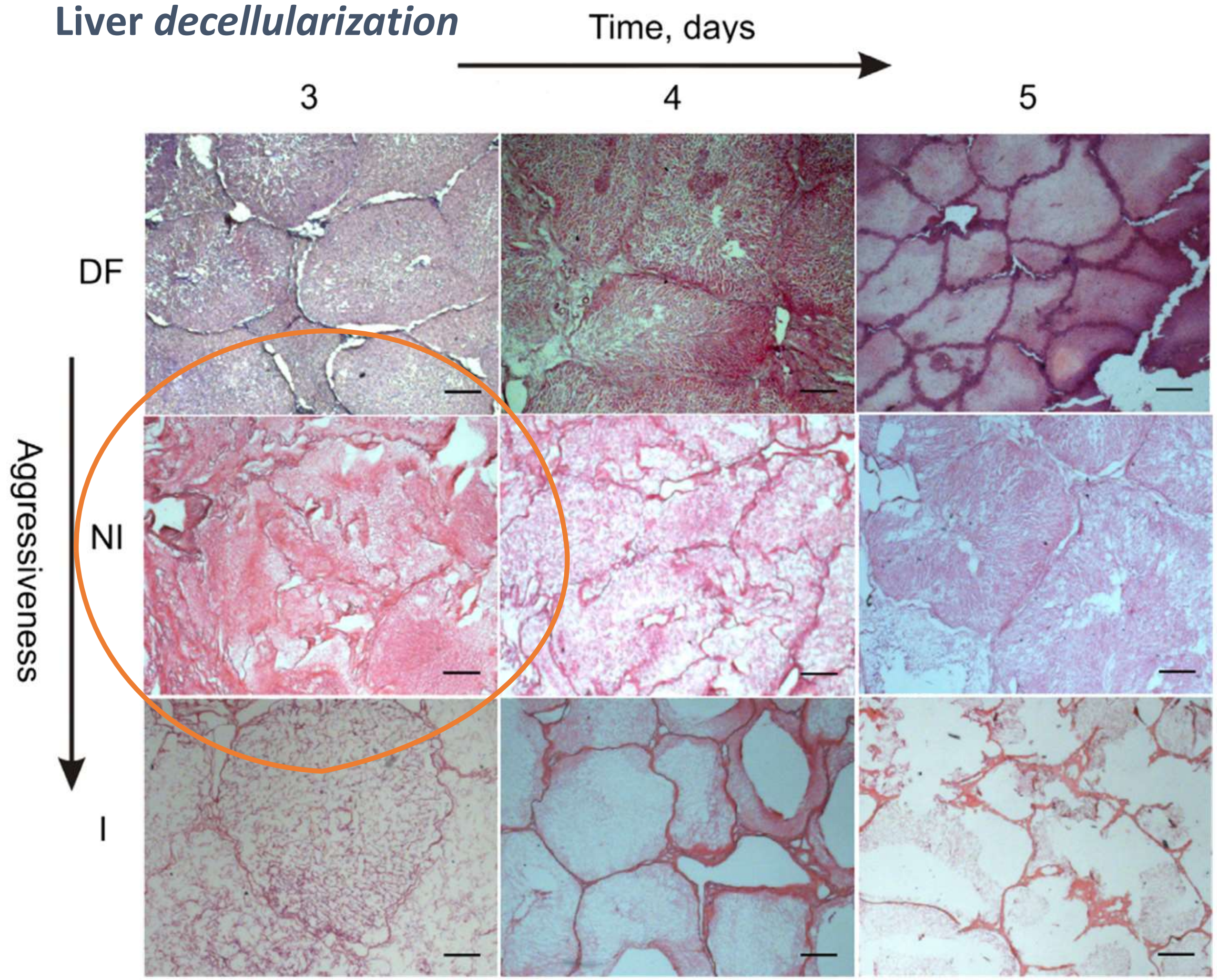
Tissue source: **pig liver**

Tunable ECM Gels

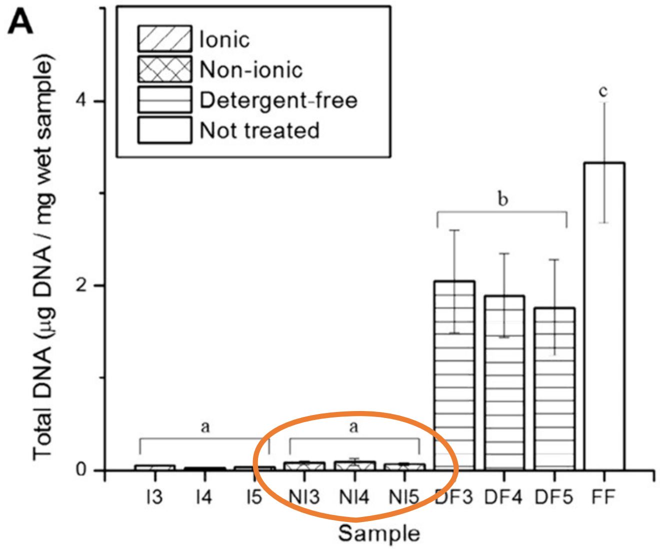
Mattei et al, Ac. Biom., 2014

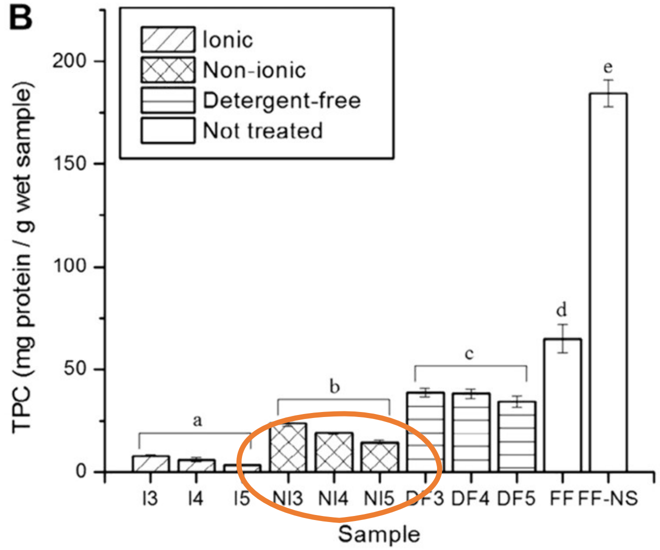
Decellularization

Liver *decellularization*



Mattei et al, Ac. Biom., 2014





Decellularization

Liver: *mechanical properties*

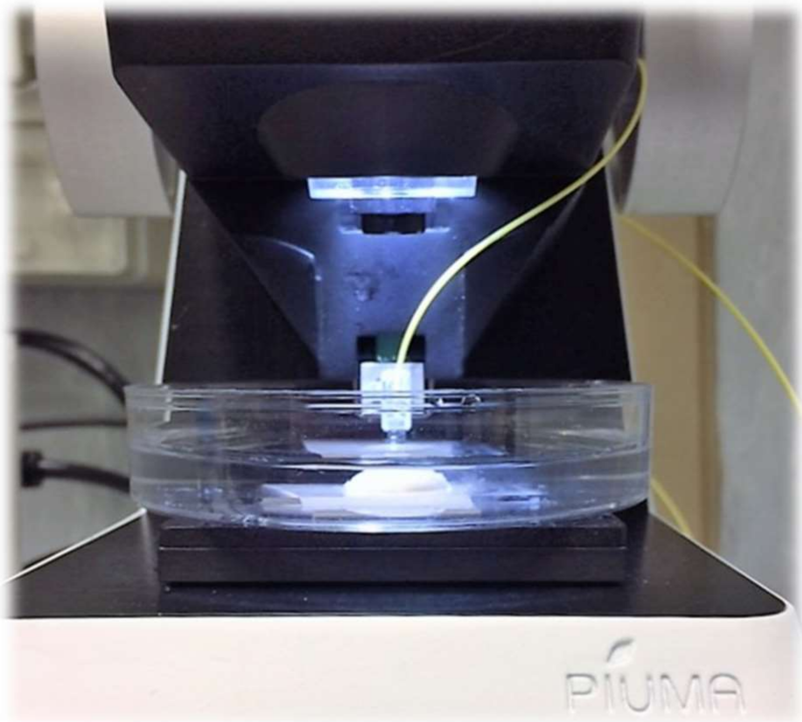
Fresh liver: E = 1.62 ± 0.13 kPa

No significant differences among liver lobes and different pigs.

→ Homogenous tissue source (healthy pigs from the same piggery)

Decellularized liver: I3 protocol -> 1.25 ± 0.07 kPa
NI3 protocol -> 1.31 ± 0.09 kPa

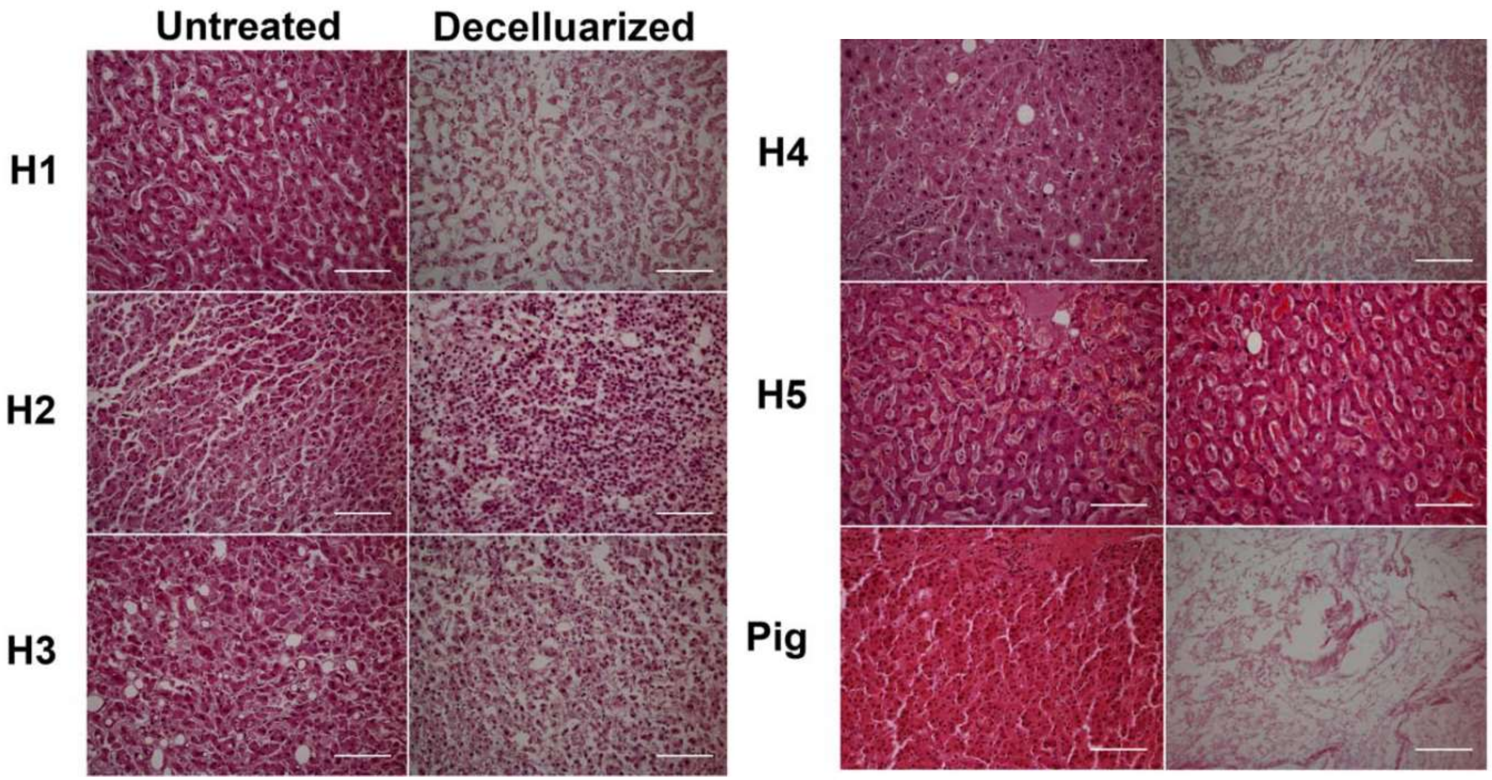
These values are slightly different from that of untreated liver samples mainly because of the cell removal



Mattei et al, Ac. Biom., 2014

Decellularization

Variability of Human decellularized-tissues: *the importance of Healthy Tissue Sources*



Applying the same decellularization protocol to human liver samples obtained from **five different patients** yielded five different outcomes:

- different levels of remaining cells and matrix
- different protein and GAG content per unit area after decellularization

Mattei et al, Art. Org., 2016