AN EXPERIMENTAL INVESTIGATION OF THE VISCOELASTIC PROPERTIES OF SMALL VOLUMES OF MATERIAL

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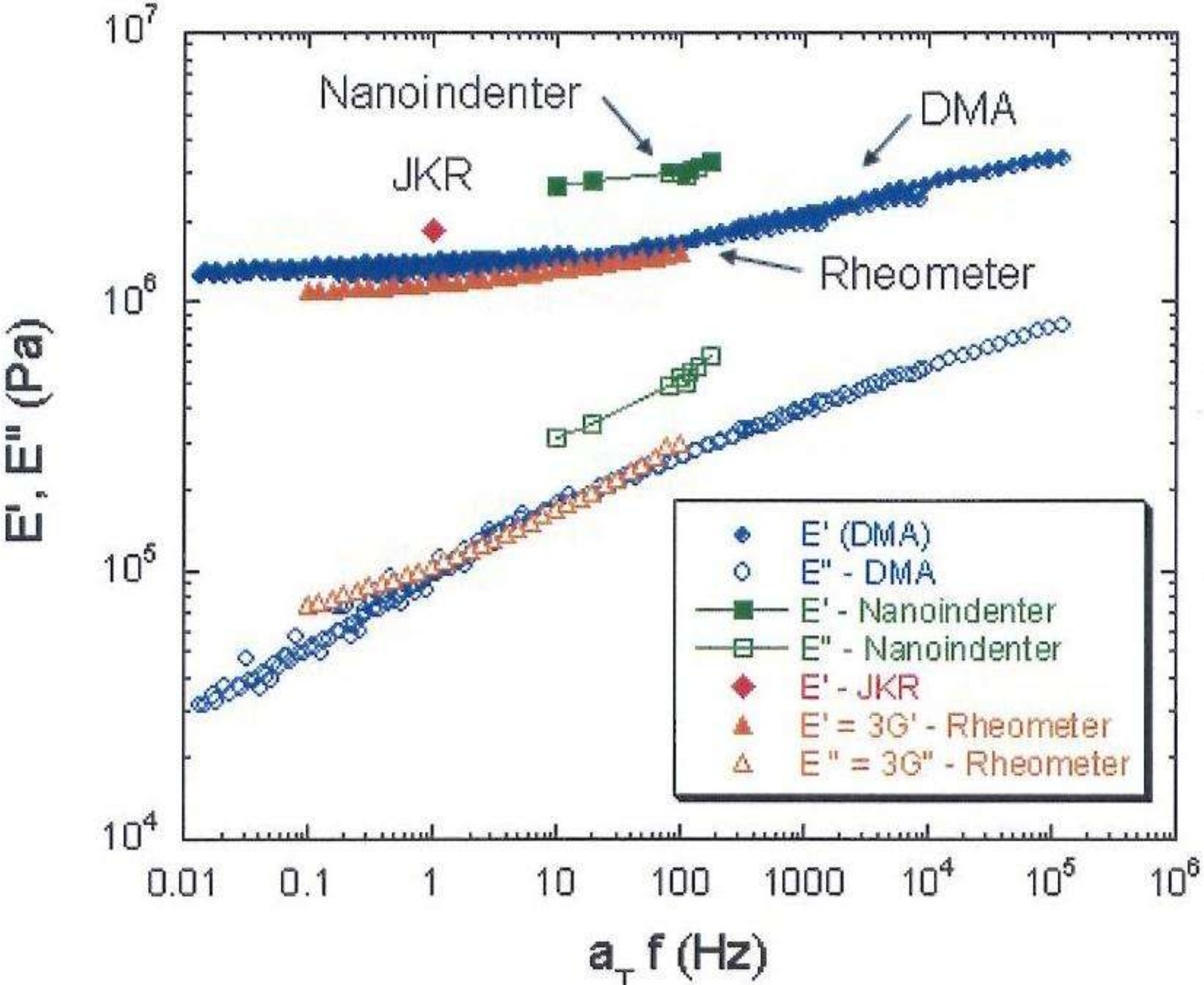
MOTIVATION

- ***What we're after:***
  - Constitutive behavior of small volumes of viscoelastic solids subjected time varying excitation over as wide a range of time or frequency as possible.
- ***Extend the applicability of nanoindentation to time-dependent behavior***
  - *Flat punch indentation, complex test geometry*
  - ***DMA, triple clamp fixture, complex geometry***
  - ***Simple test geometry, uniaxial compression***

- Material's response in the frequency domain - short time
- Material's response in the time domain - long time
- Bringing them together
- Do it all with flat punch indentation
- Material selection: Highly plasticized PVC

NANOINDENTATION & DMA COMPARISON

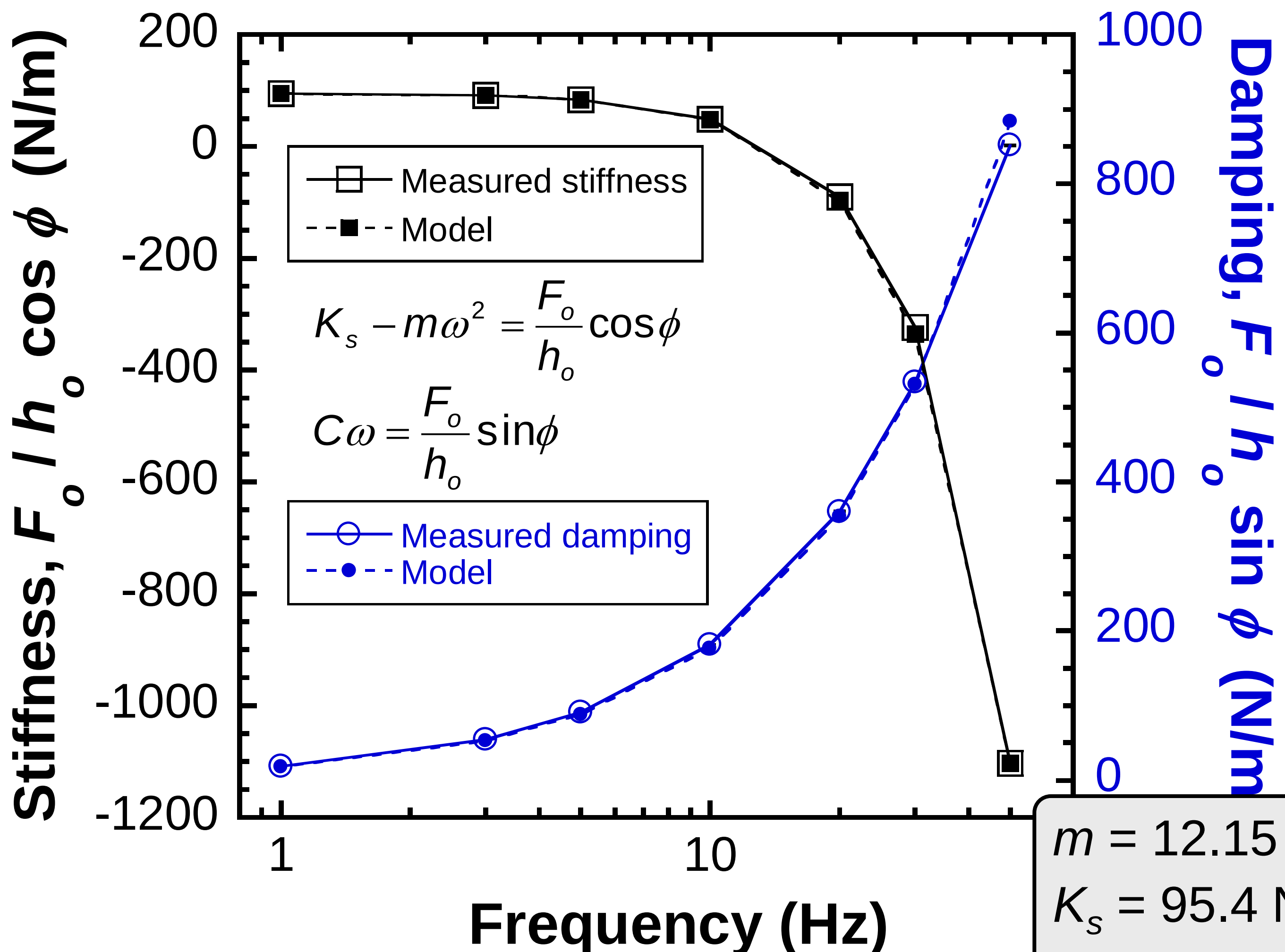


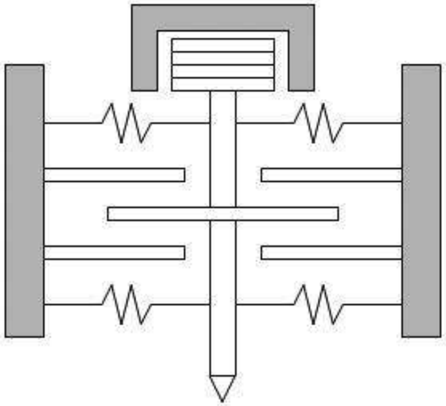
C. C. White et al., Mater. Res. Soc. Symp. Proc. **841** (2005)

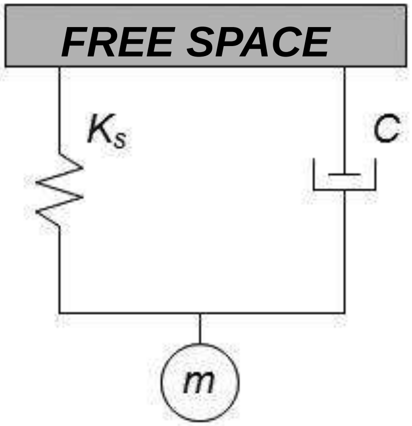
MODELING THE INSTRUMENTATION

- Measure time-dependent material properties
- Then we need to understand the time-dependent properties of the measurement tool

Measured stiffness and damping in free space, position = 18.8 μm







m = 12.15 g
K_s = 95.4 N/m
C = 2.81 Ns/m

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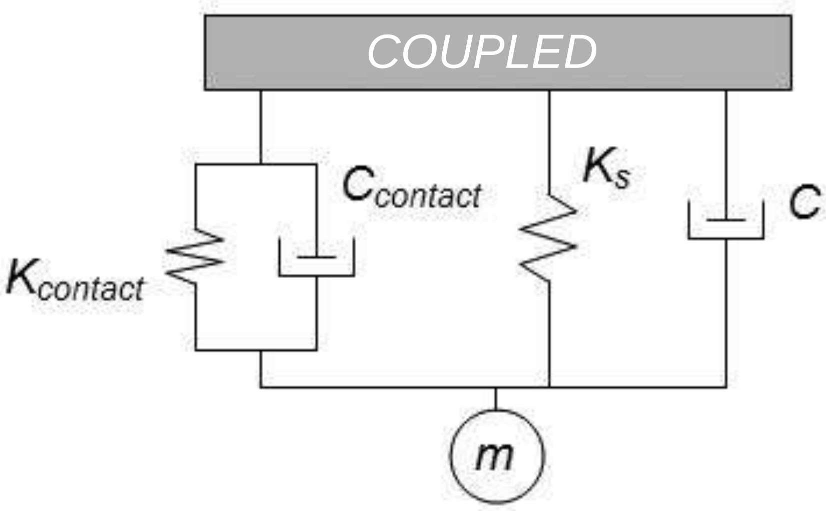
ADD THE CONTACT



$$S = \frac{F_o}{h_o}\cos\phi + m\omega^2$$

$$C = \frac{F_o}{h_o}\frac{\sin\phi}{\omega}$$

COUPLED RESPONSE = SAMPLE + INSTRUMENT

$$K_{\text{contact}} = \left[\left.\frac{F_o}{h_o}\cos\phi + m\omega^2\right|_{\text{coupled}}\right] - \left[\left.\frac{F_o}{h_o}\cos\phi + m\omega^2\right|_{\text{inst.(free space)}}\right]$$

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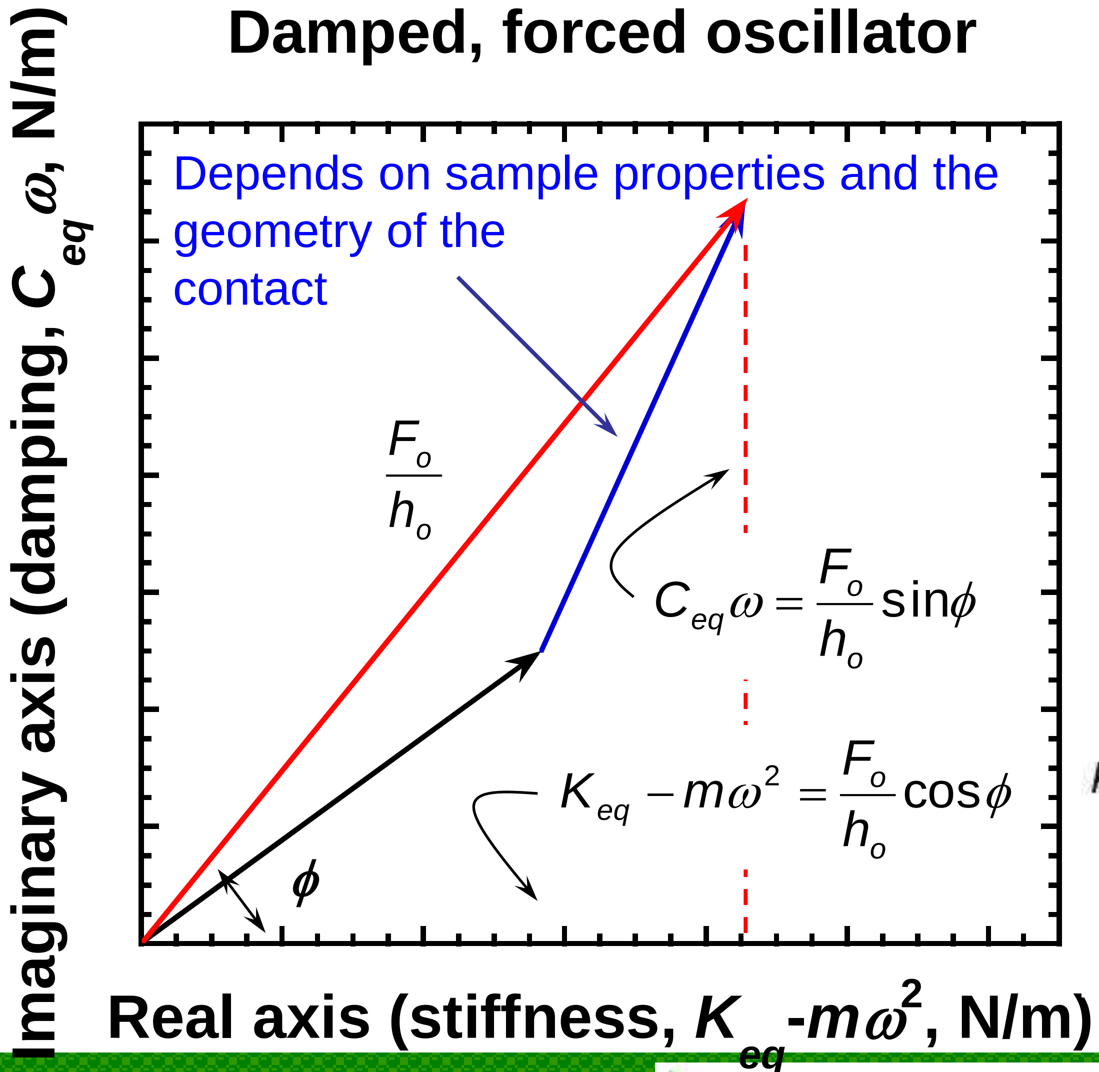
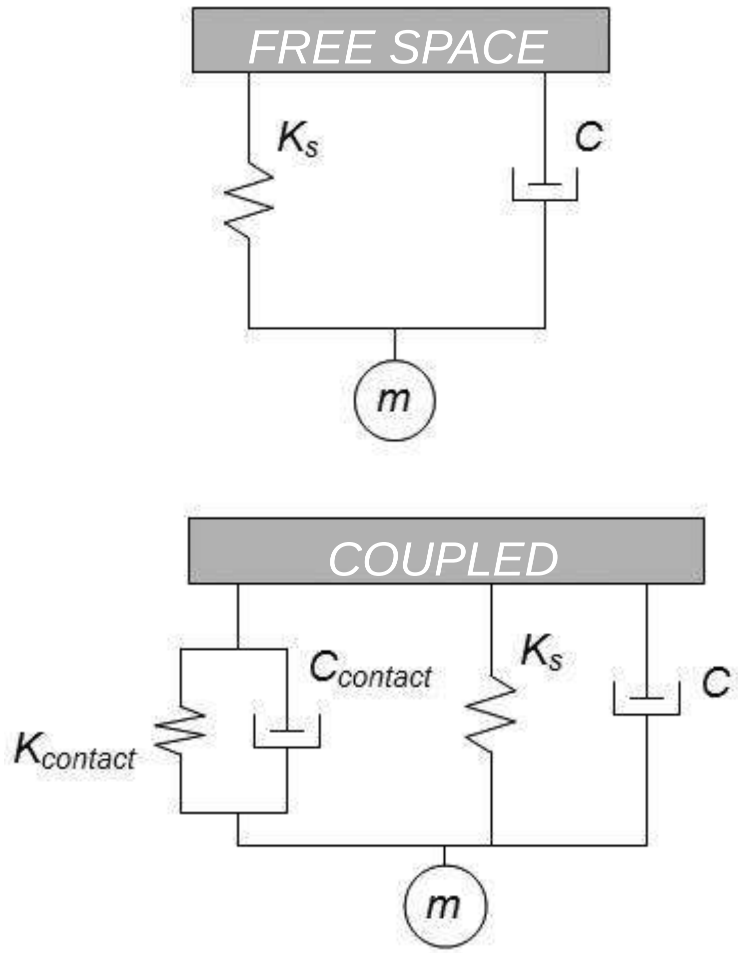
PHASOR DIAGRAM: PHYSICAL INSIGHT

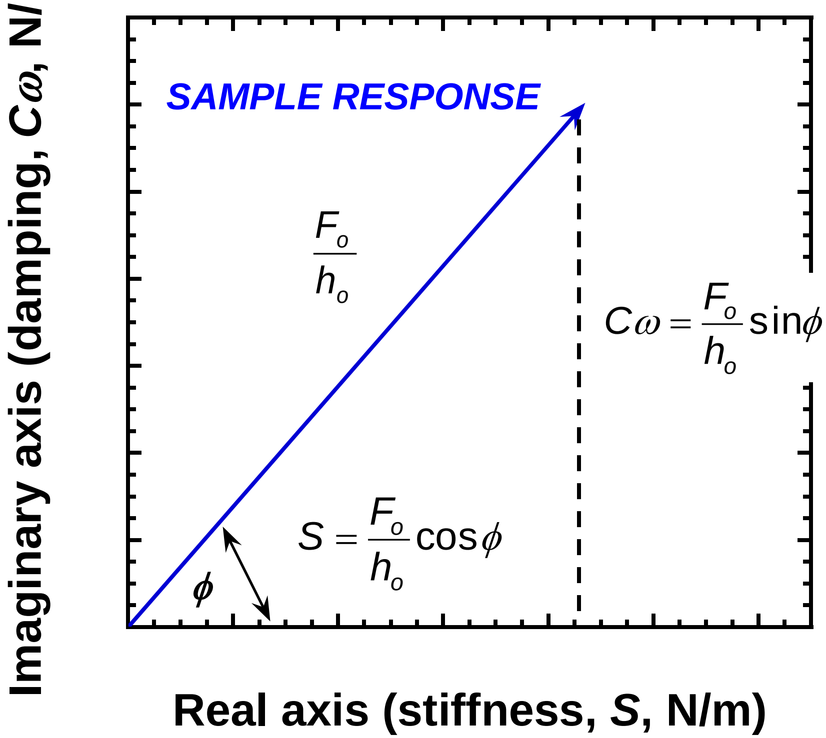
Damped, forced oscillator



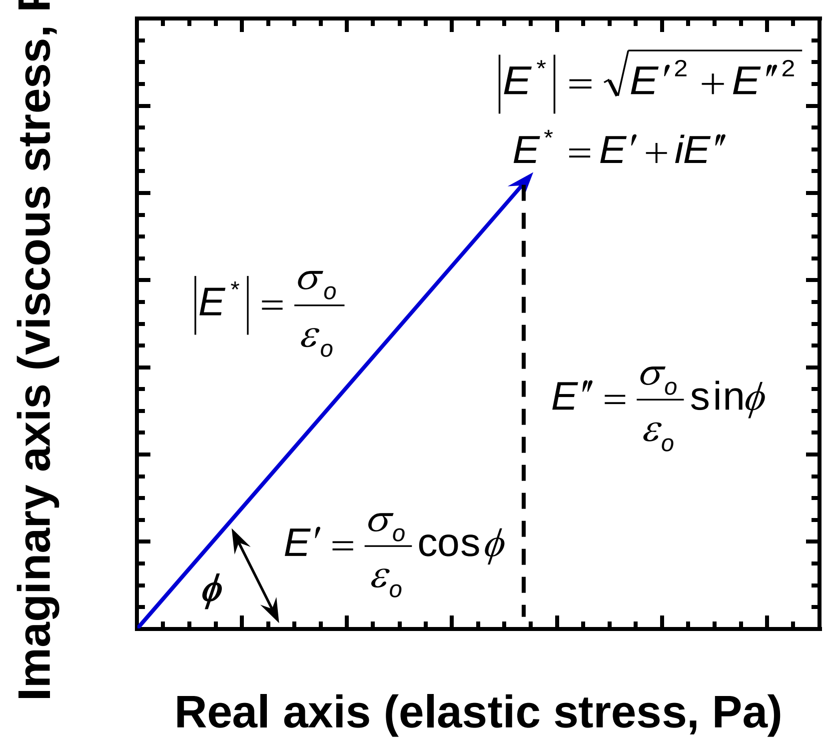


FROM S AND $C\omega \rightarrow E'$ AND E''

Phasor diagram of experimental measurements



Phasor diagram of a linear viscoelastic solid

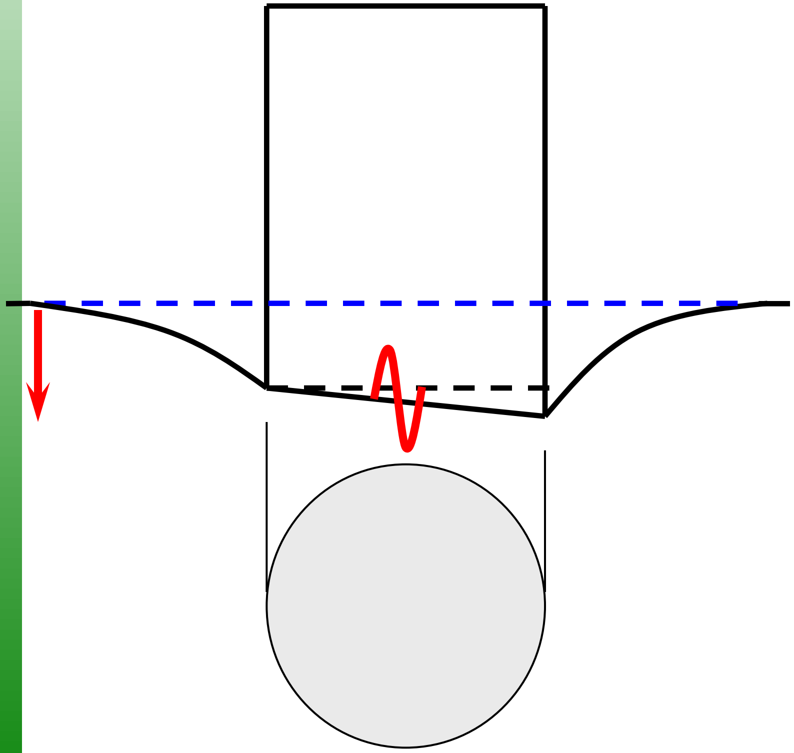


The fundamental equation of nanoindentation:

$$E' = S\frac{\sqrt{\pi}}{2}\frac{1}{\beta}\frac{1}{\sqrt{A}} \qquad E'' = C\omega\frac{\sqrt{\pi}}{2}\frac{1}{\beta}\frac{1}{\sqrt{A}}$$

GEOMETRY OF THE CONTACT

Circular flat punch:



Advantages:

– Known contact area
– Area not affected by creep or thermal drift

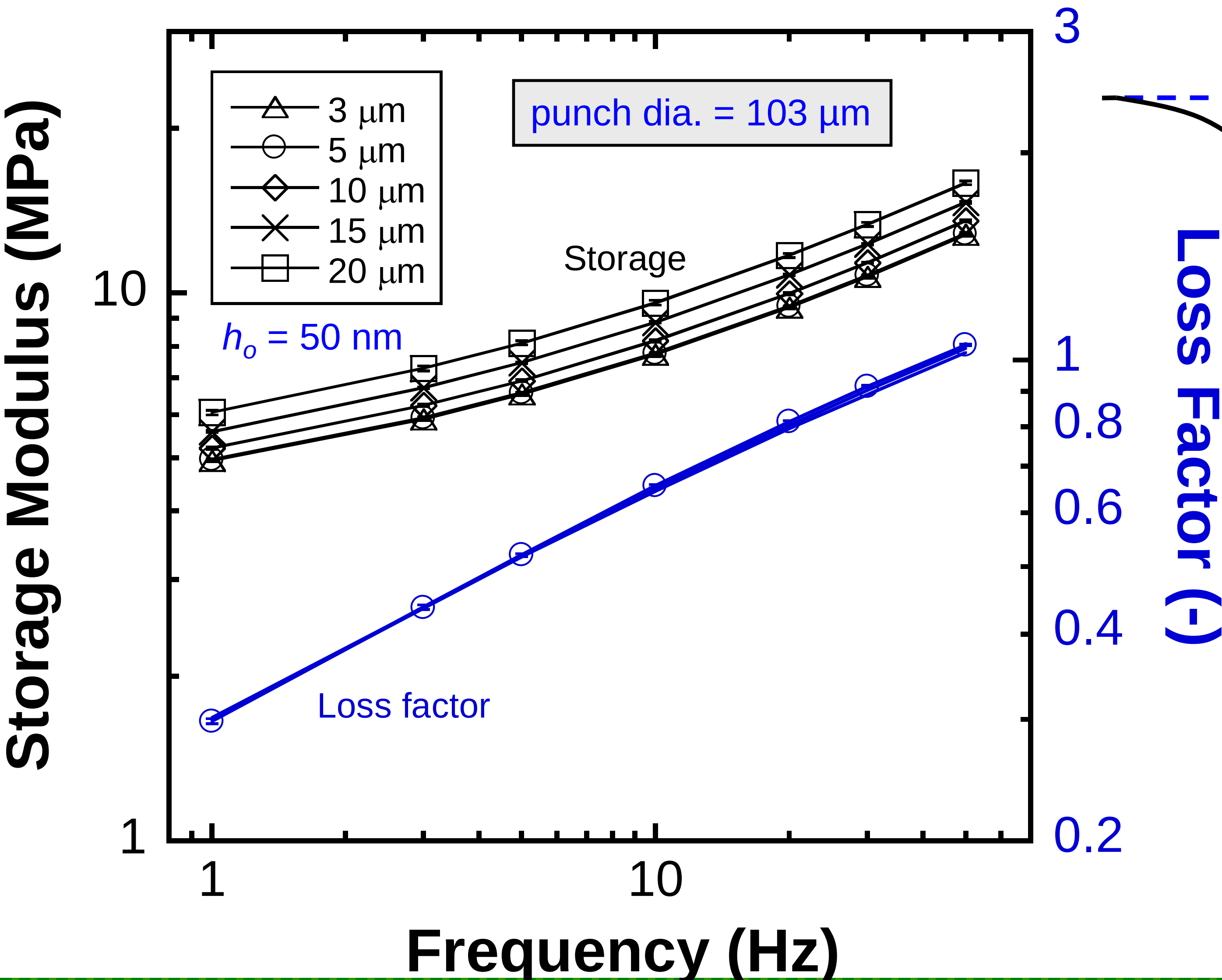
Disadvantages:

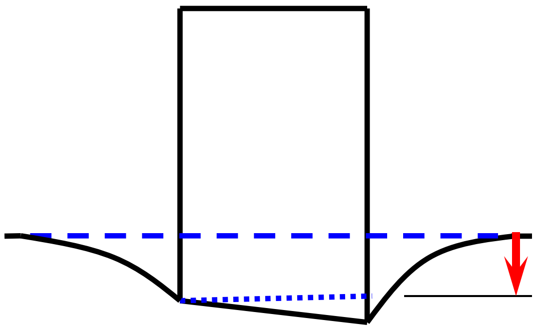
– Full contact
– Stress concentration

Any tip geometry, consider:

– Steady-state harmonic motion
– Linear viscoelasticity
  • Compression distance
  • Oscillation amplitude
– Loading rate

Pre-Compression Dependence





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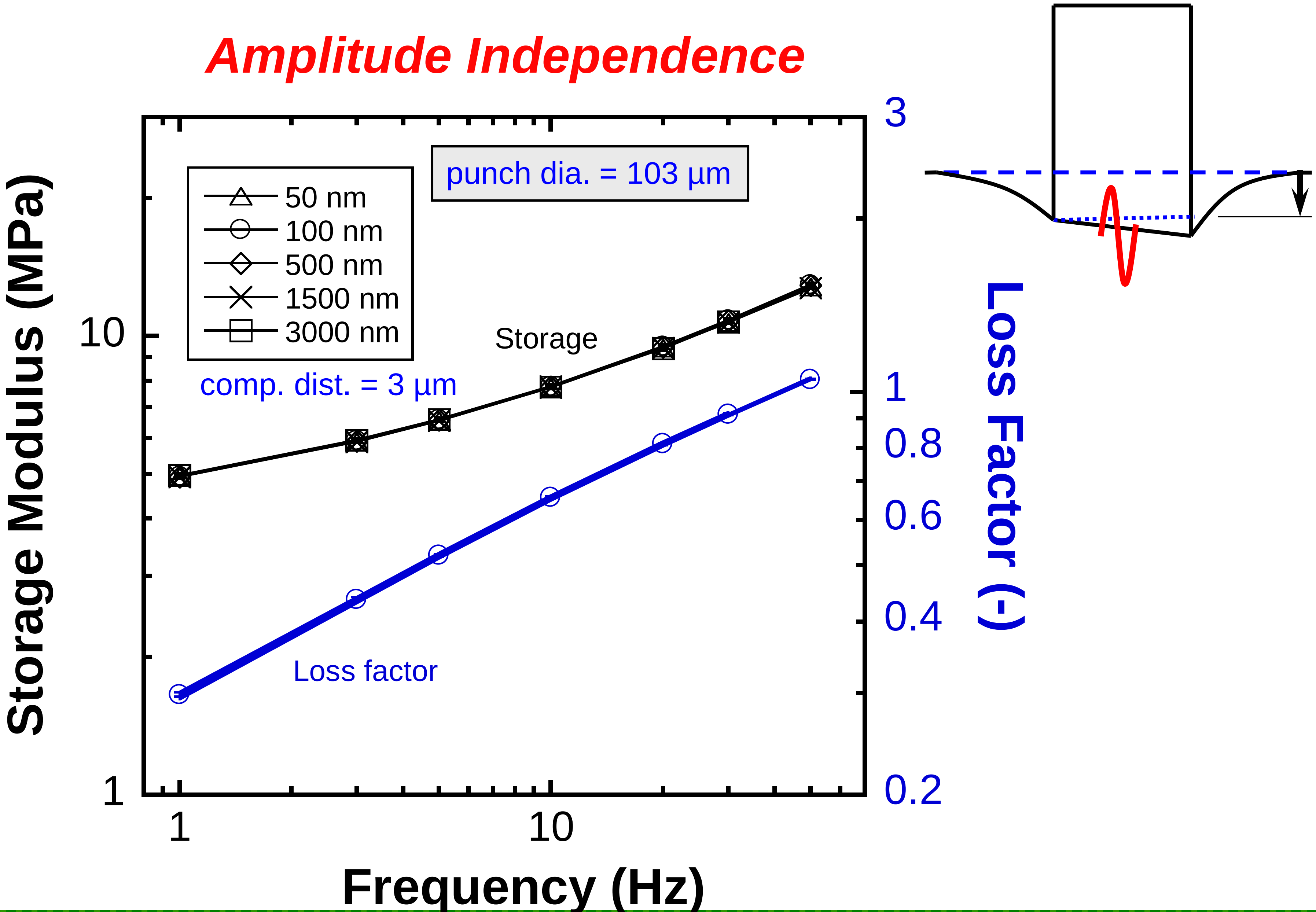
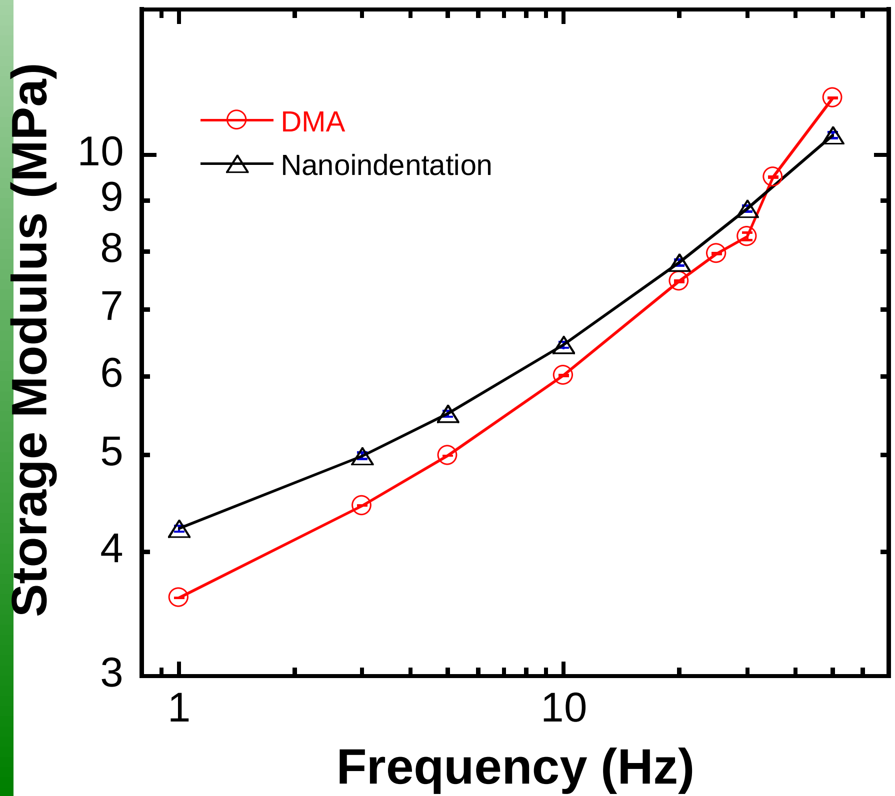
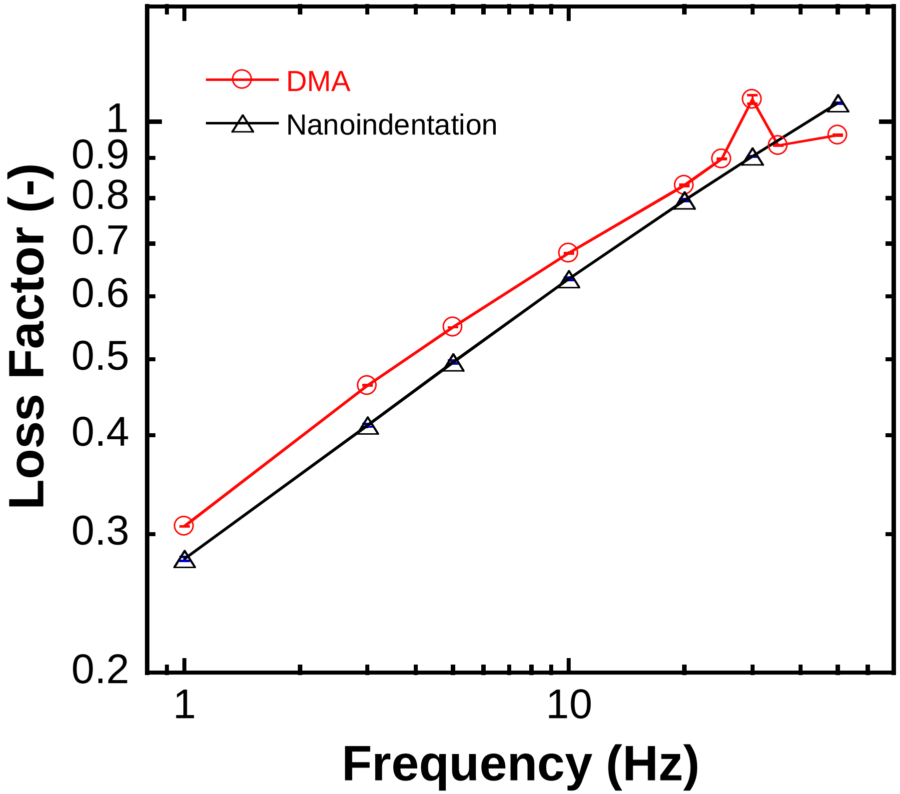
Amplitude Independence



DMA VS. NANOINDENTATION

Highly plasticized polyvinylchloride, the complex modulus at 22 ^{o}C





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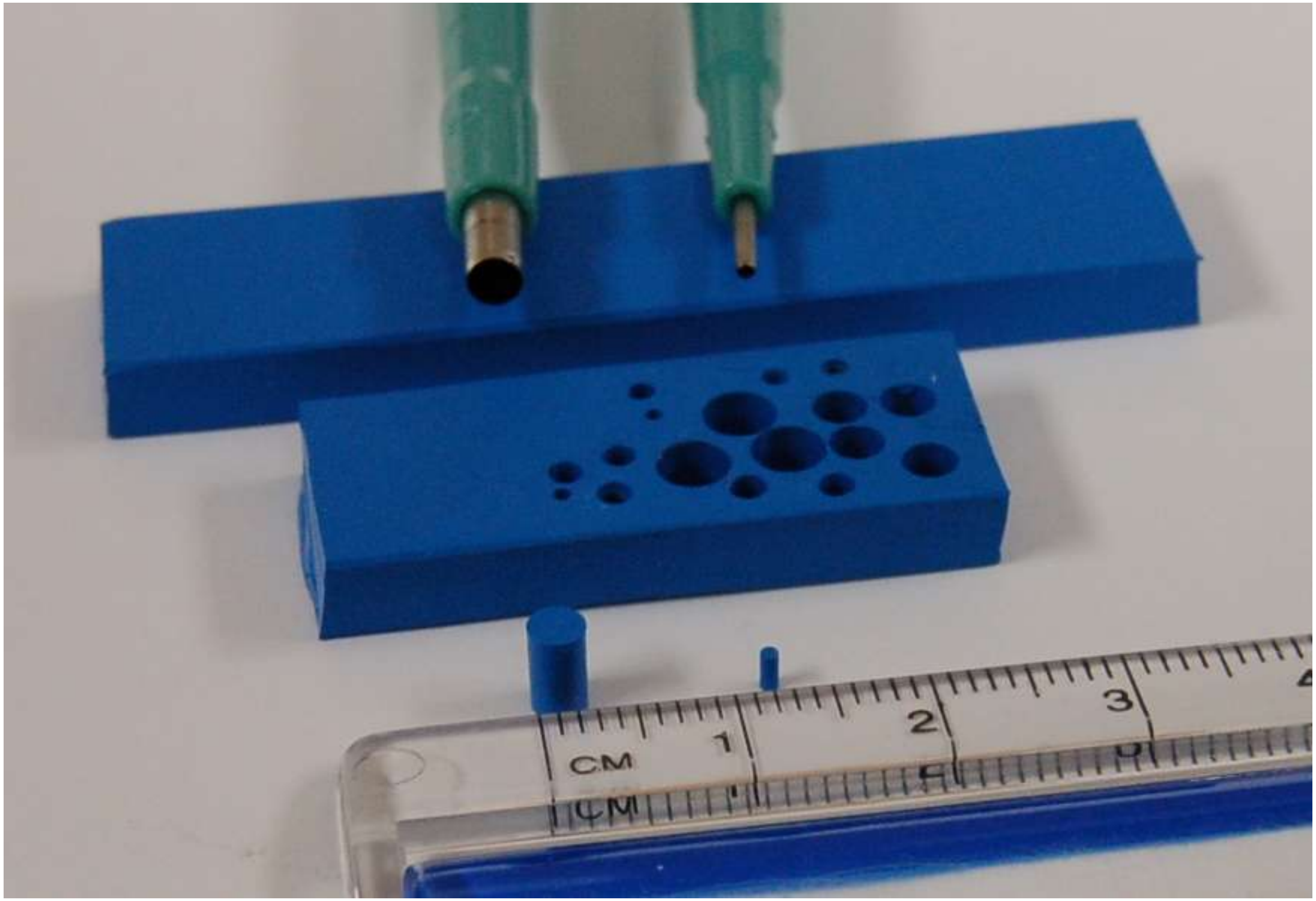
NEXT STEPS

- Do a better job comparing flat punch experiments to tests with simpler geometry.
- Study creep compliance with a flat punch.
- Combine frequency specific results with creep compliance.

COMPRESSION SAMPLES



COMPRESSION & INDENTATION

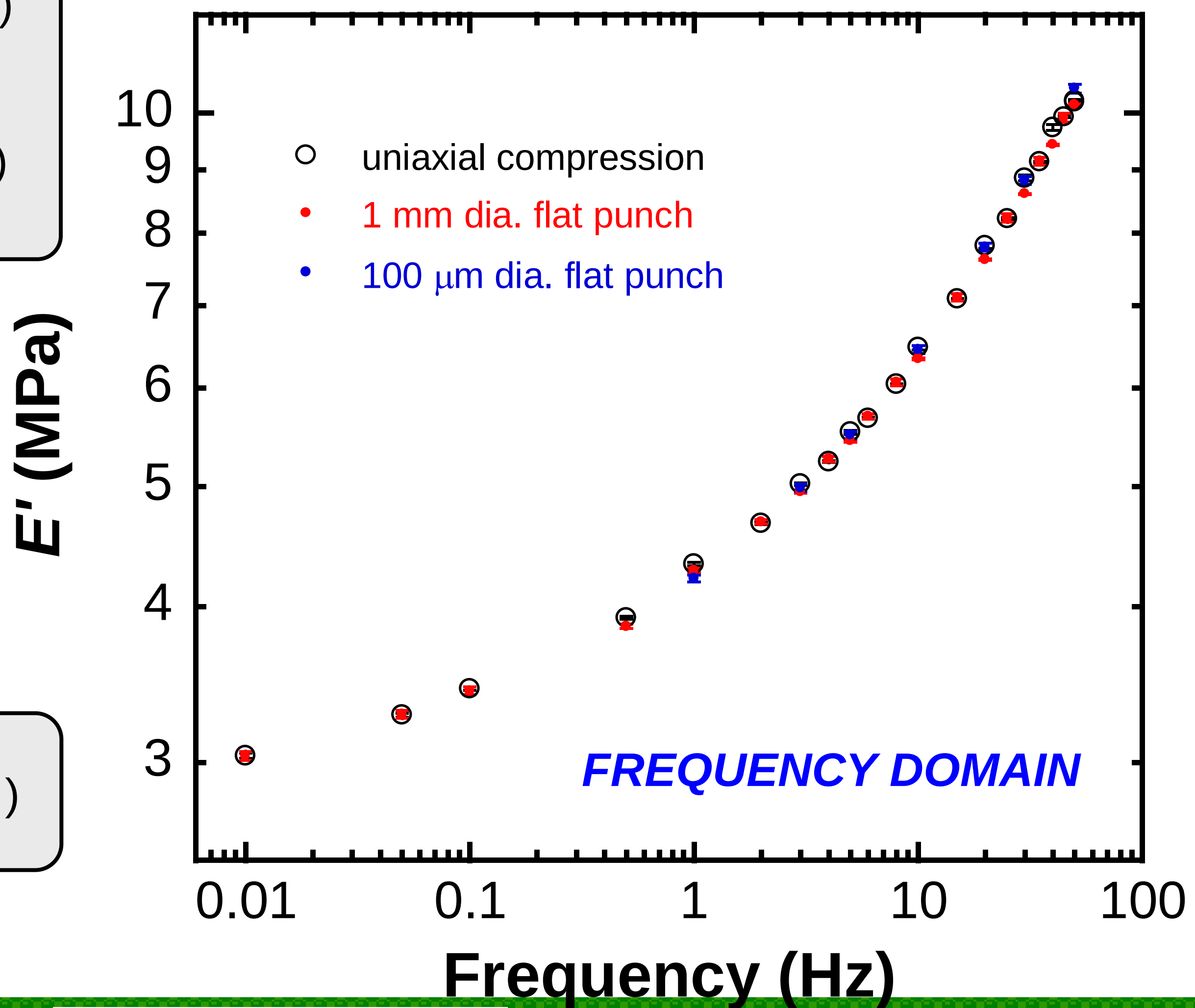
$$E' = \frac{F_o}{h_o}\cos\phi(geometry\,factor)$$

$$E'' = \frac{F_o}{h_o}\sin\phi(geometry\,factor)$$

Geometry factors:

Compression: $\frac{L}{A}$

Indentation: $\frac{\sqrt{\pi}}{2}\frac{1}{\beta}\frac{1}{\sqrt{A}}(1-\upsilon^2)$



COMPRESSION & INDENTATION

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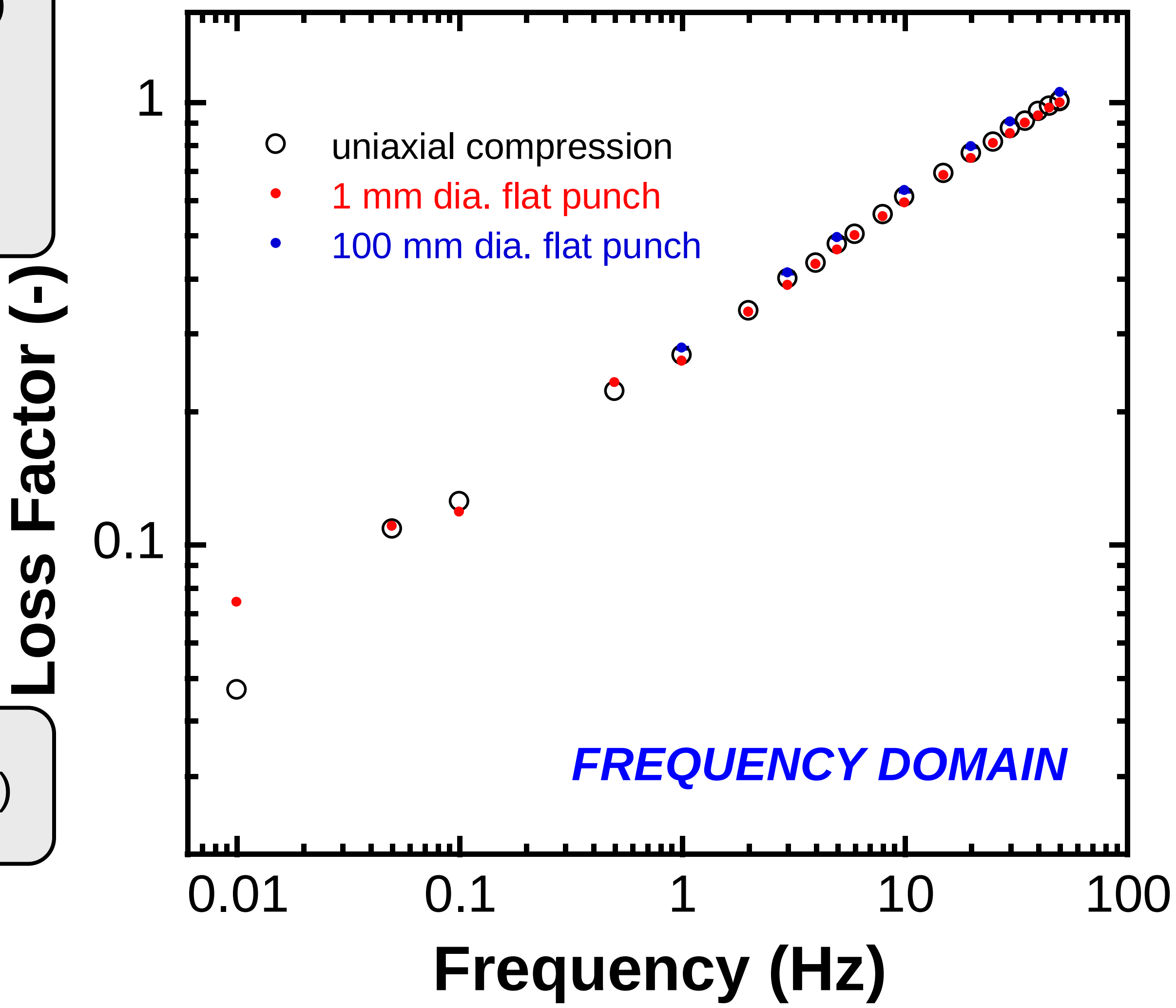

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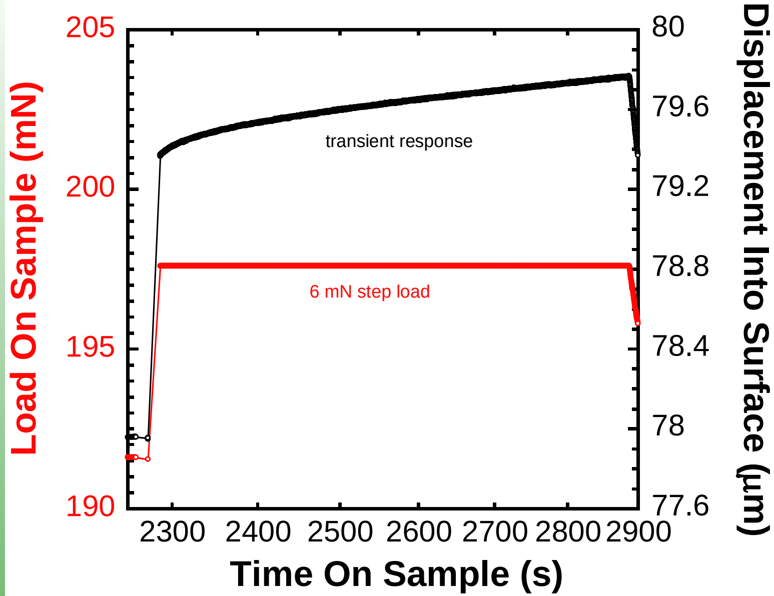


Compression:

$$\sigma = \frac{P}{A} \qquad \varepsilon = \frac{\Delta L}{L}$$

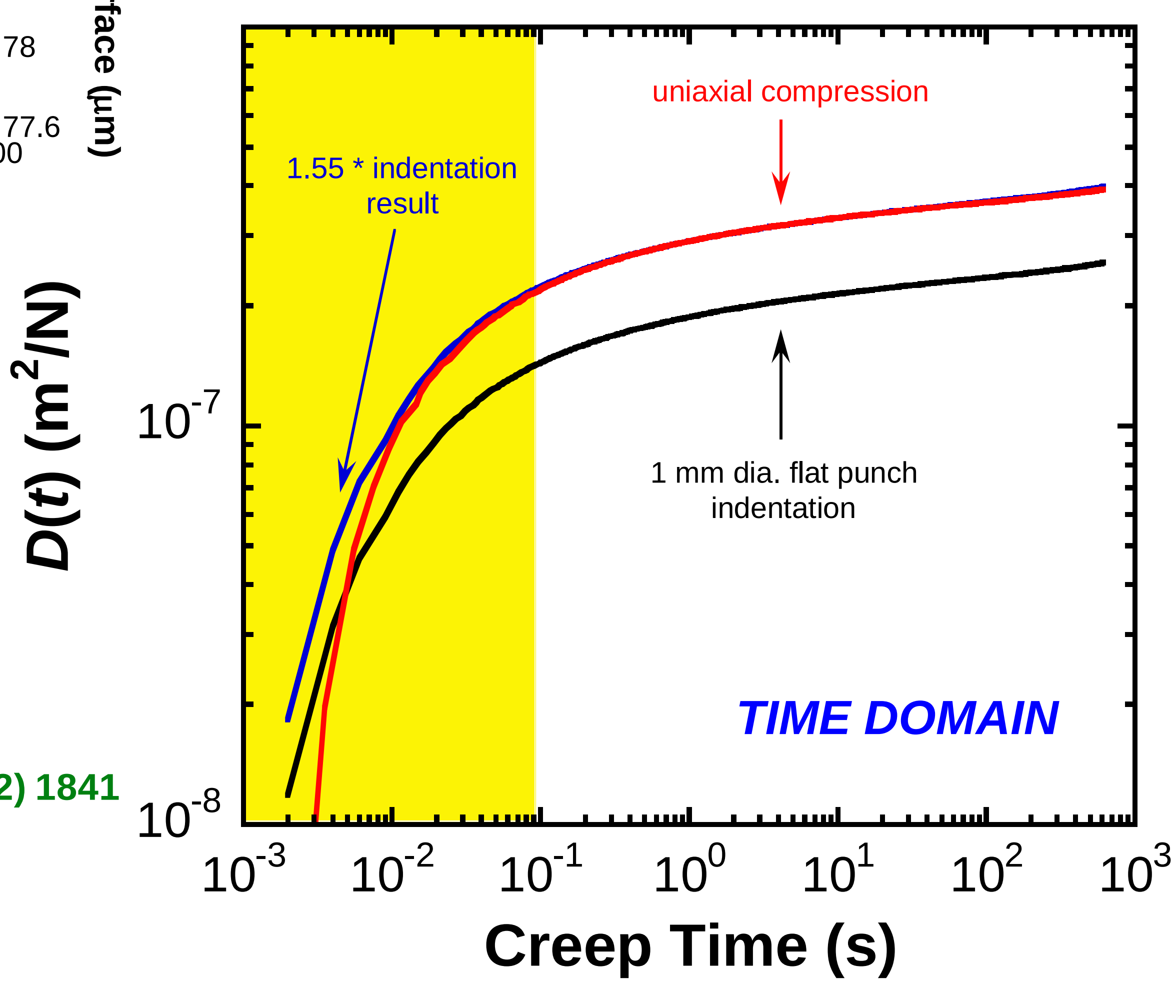
$$D(t) = \frac{A \Delta L}{LP}$$

Indentation:

$$\sigma \,\alpha\, H = \frac{P}{A} \qquad \varepsilon \,\alpha\, \frac{h}{D}$$

$$D(t) = C \frac{h}{HD}$$

$$D(t) = \frac{\varepsilon(t)}{\sigma_o}$$



Sakia, M., Phil. Mag. A 82 10 (2002) 1841

TRANSFORMING FROM FREQUENCY TO TIME

4 term Prony series:

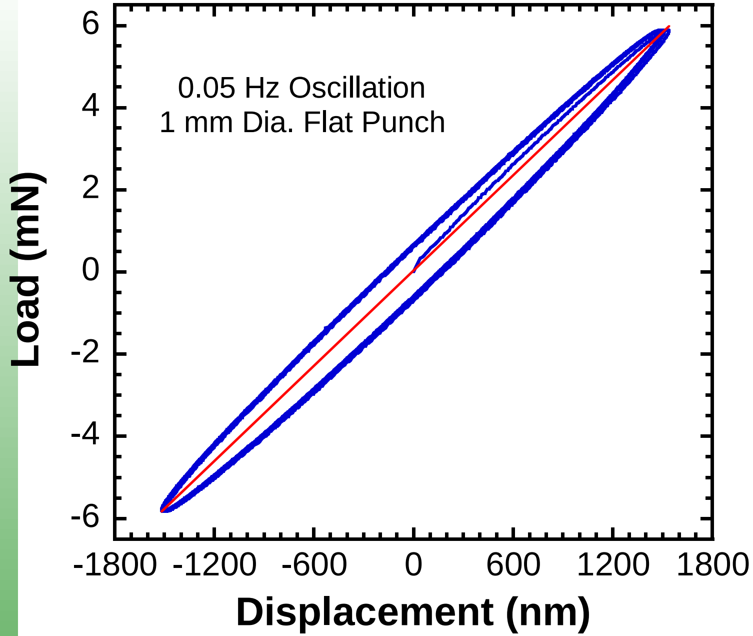
FREQUENCY DOMAIN:

$$J' = J_0 + \sum_{i=1}^{4} \frac{J_i}{1 + \tau_i^2 \omega^2}$$

$$J' = \frac{E'}{E'^2 + E''^2}$$

TIME DOMAIN:

$$D(t) = j_0 + \sum_{i=1}^{4} J_i (1 - e^{\frac{-t}{\tau_i}})$$



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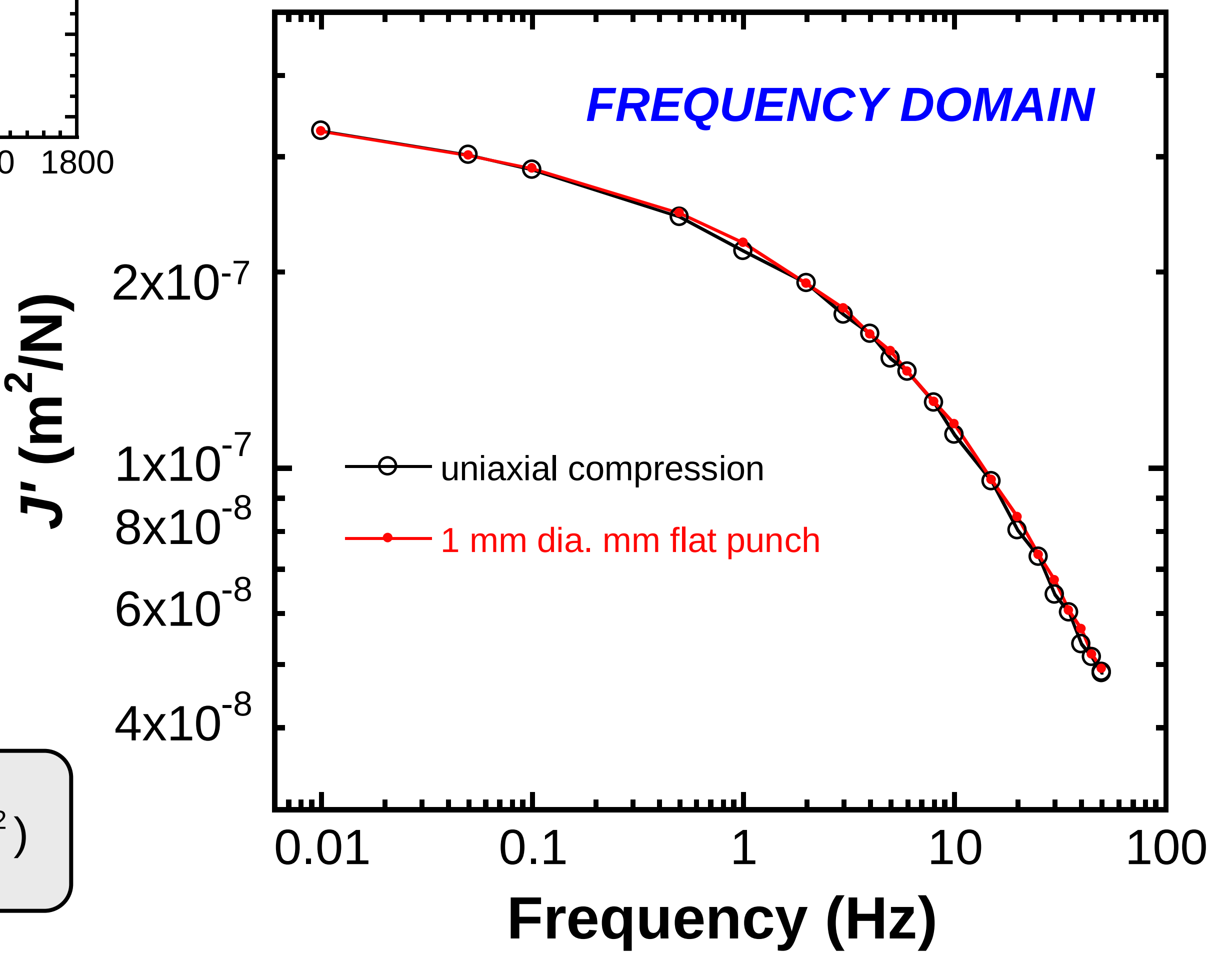


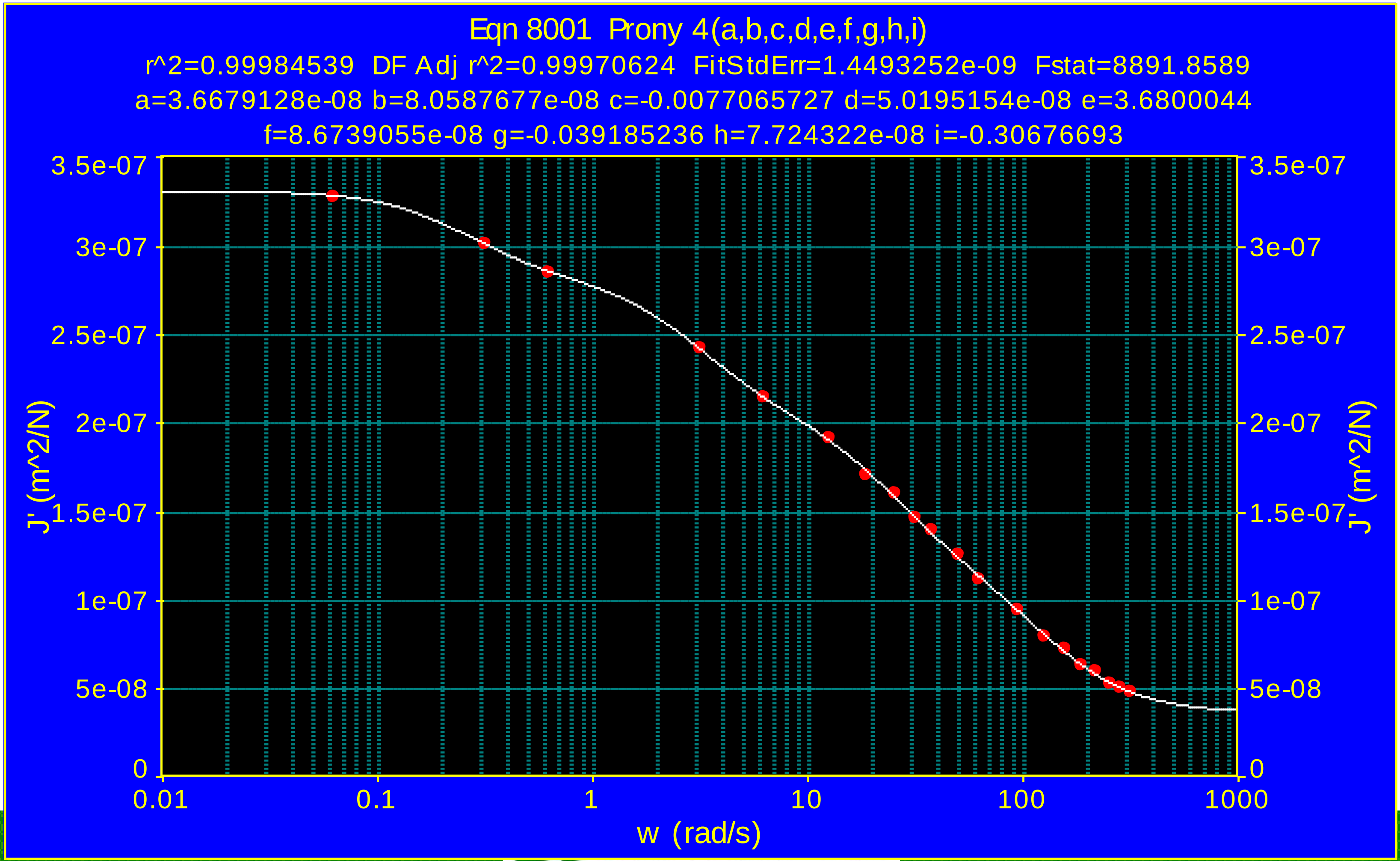
Geometry factors:

Compression: $\frac{L}{A}$

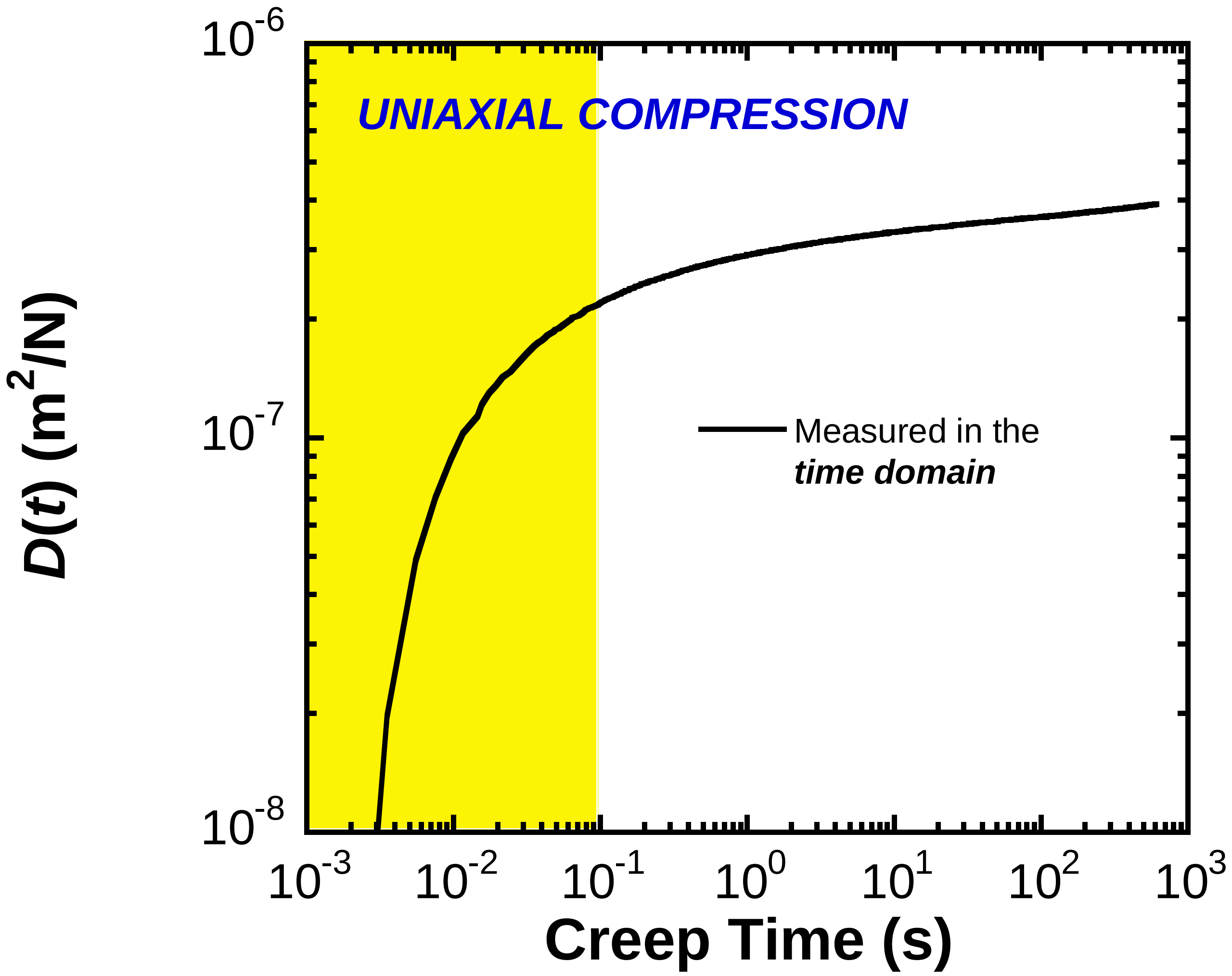
Indentation: $\frac{\sqrt{\pi}}{2}\frac{1}{\beta}\frac{1}{\sqrt{A}}(1-\upsilon^2)$

J' (FREQ. DOMAIN) FIT TO PRONY SERIES

UNIAXIAL COMPRESSION



VERIFICATION OF THE PATH FROM *J'* TO *D*(*t*)



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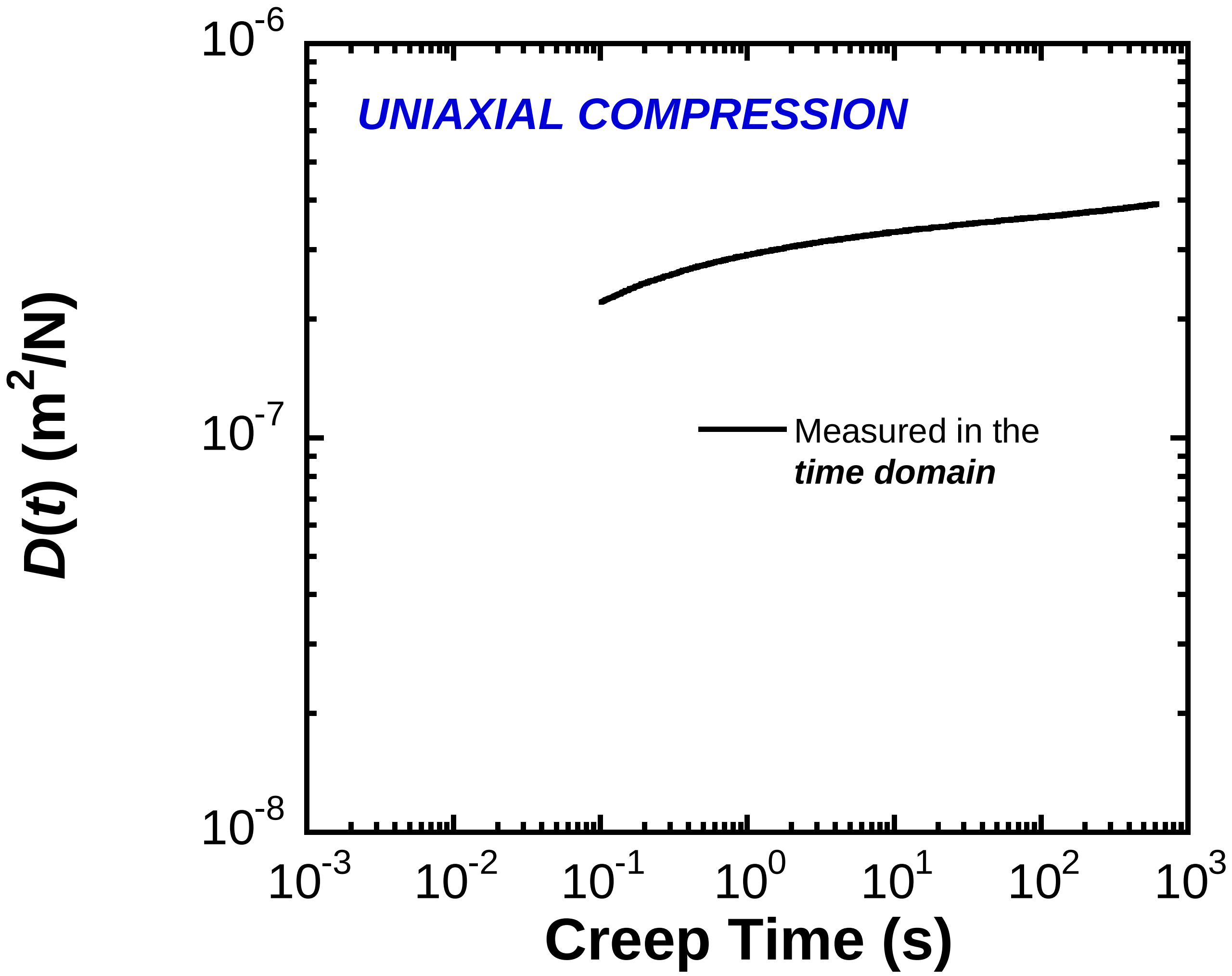

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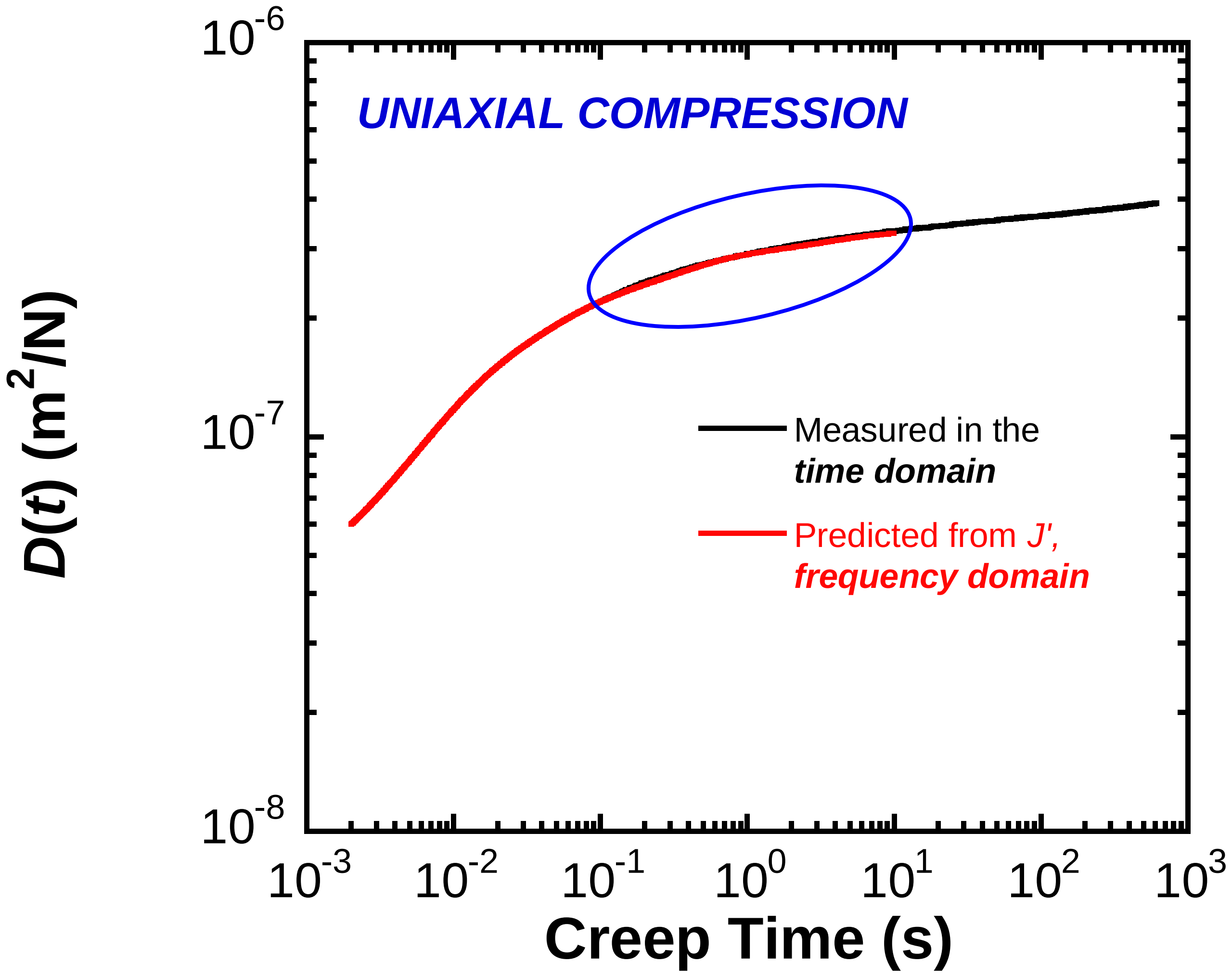

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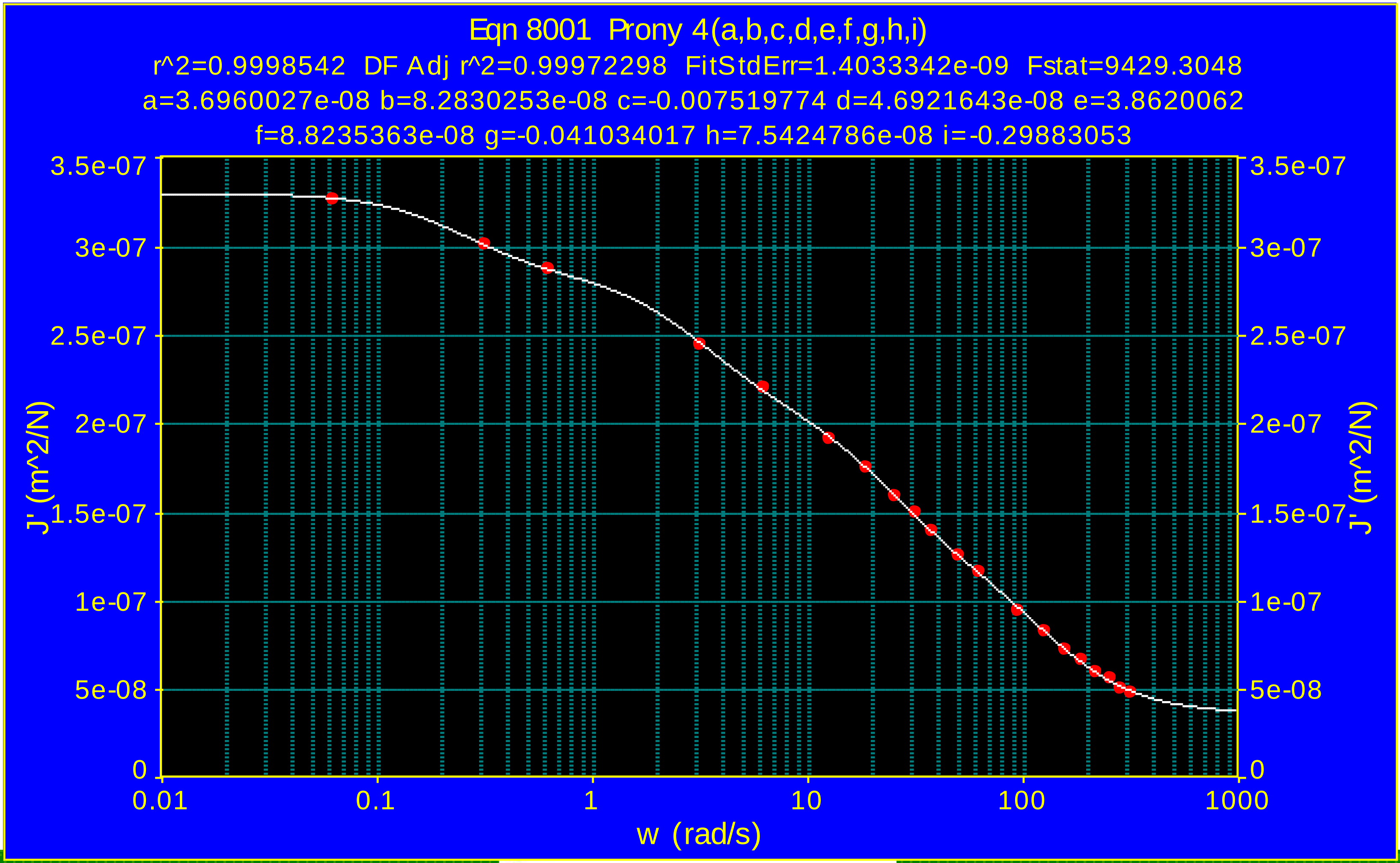
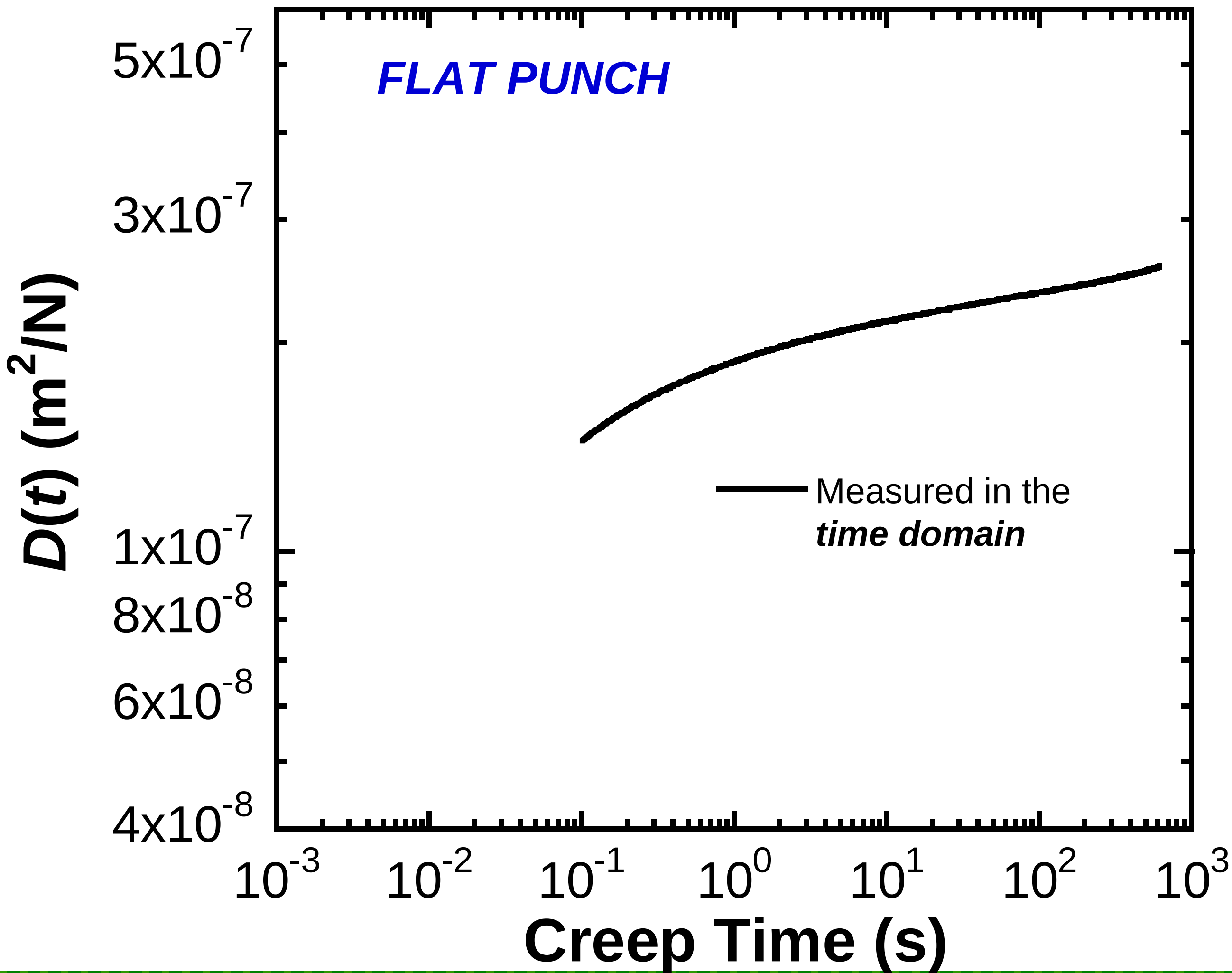
J' (FREQ. DOMAIN) FIT TO PRONY SERIES

FLAT PUNCH INDENTATION



MAXIMIZING THE TIME AND FREQUENCY RANGE



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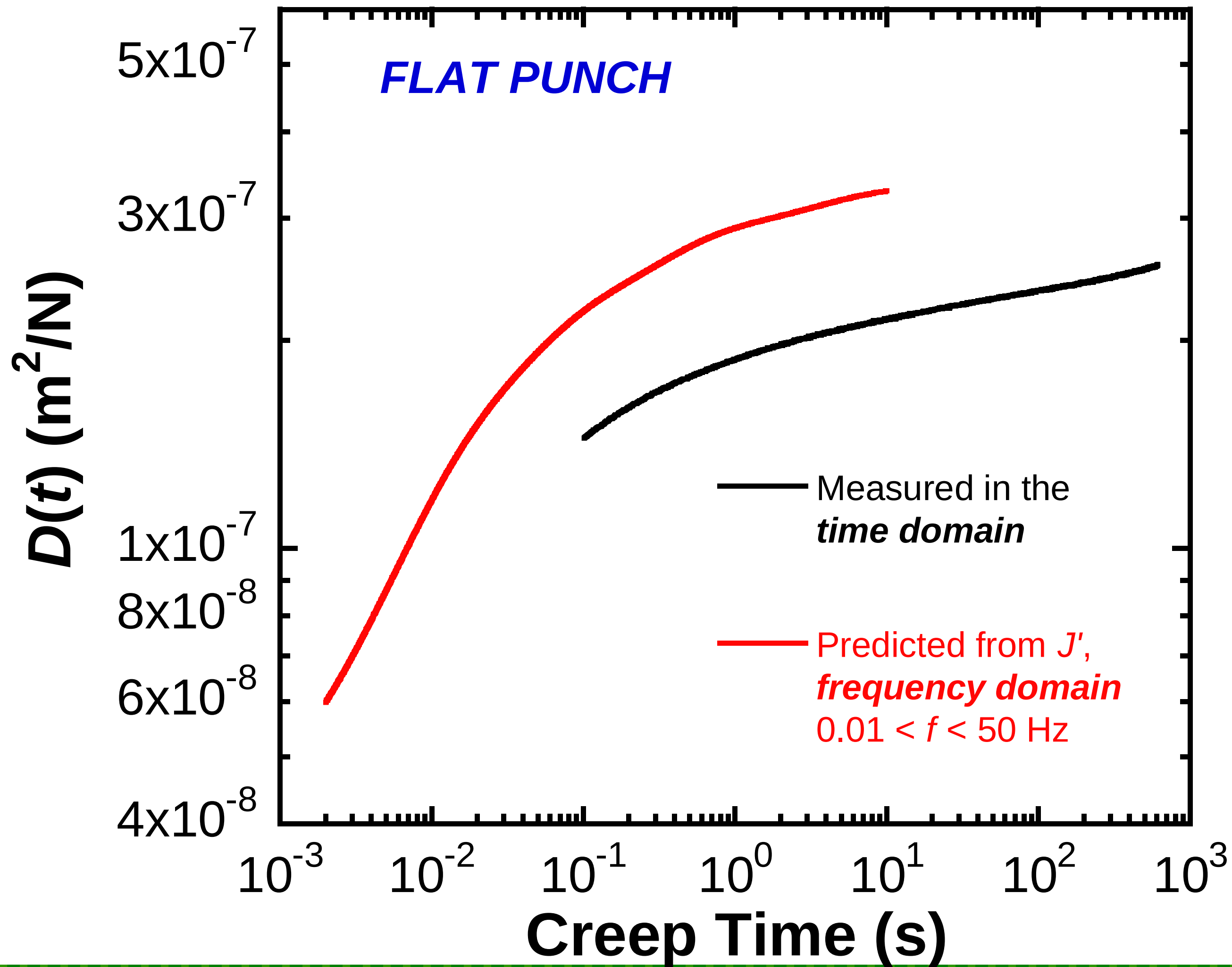

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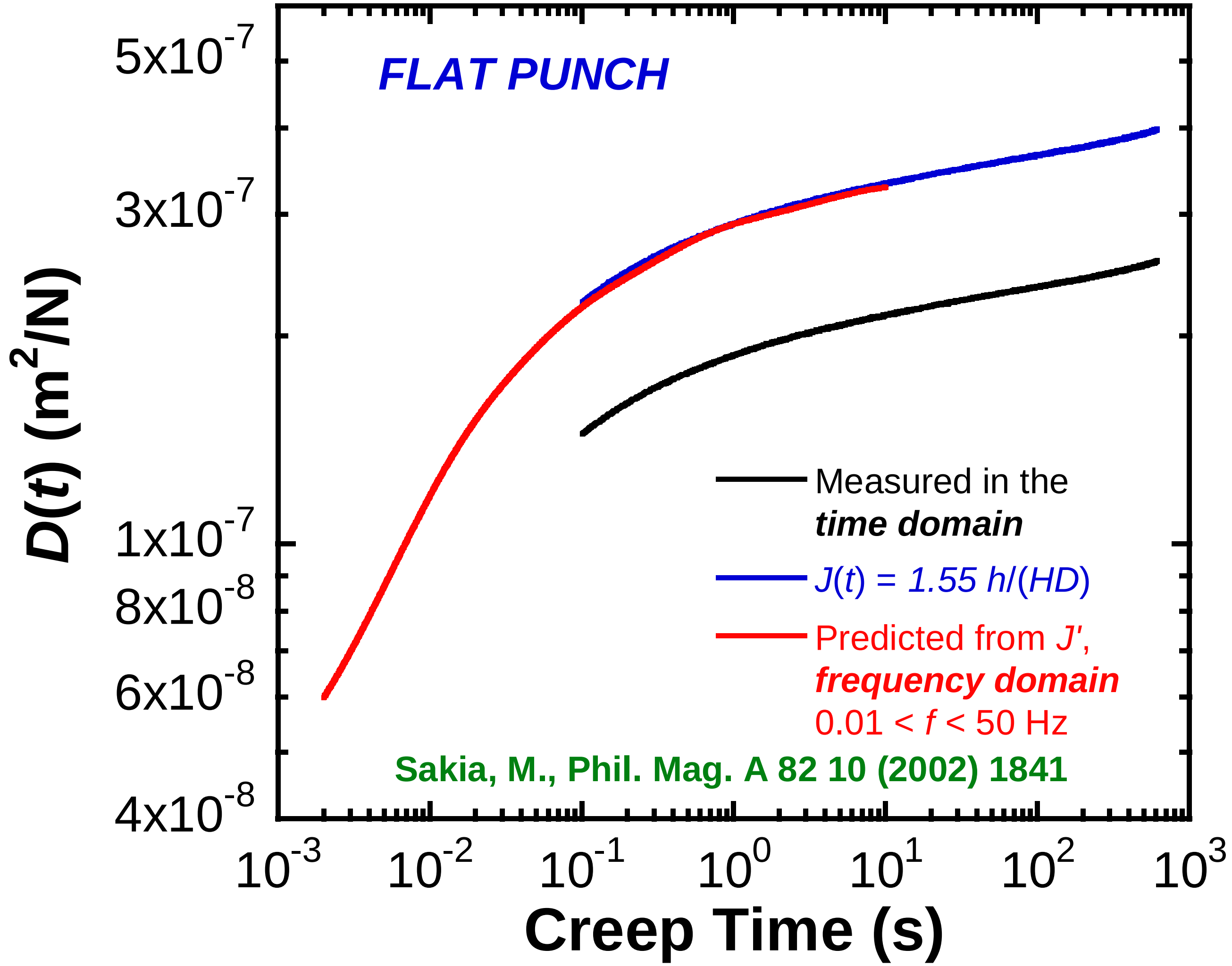

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CONCLUSIONS

- Dynamic nanoindentation of viscoelastic solids requires *robust dynamic characterization of the measurement tool itself*, a *known contact geometry*, *steady-state harmonic motion*, and *linear viscoelasticity*.
- In the frequency domain, Sneddon's stiffness equation works remarkably well.
- The Prony series model provides a valid path to transition between the frequency and time domains.
- It is possible to combine frequency and time domain data from a flat punch indentation experiment and therefore characterize the sample's behavior over the widest possible range of time and frequency.
- Sakia's calculation of the geometric factor relating flat punch time domain results to uniaxial results is confirmed.