NANOINDENTATION AND THE DYNAMIC CHARACTERIZATION OF VISCOELASTIC SOLIDS

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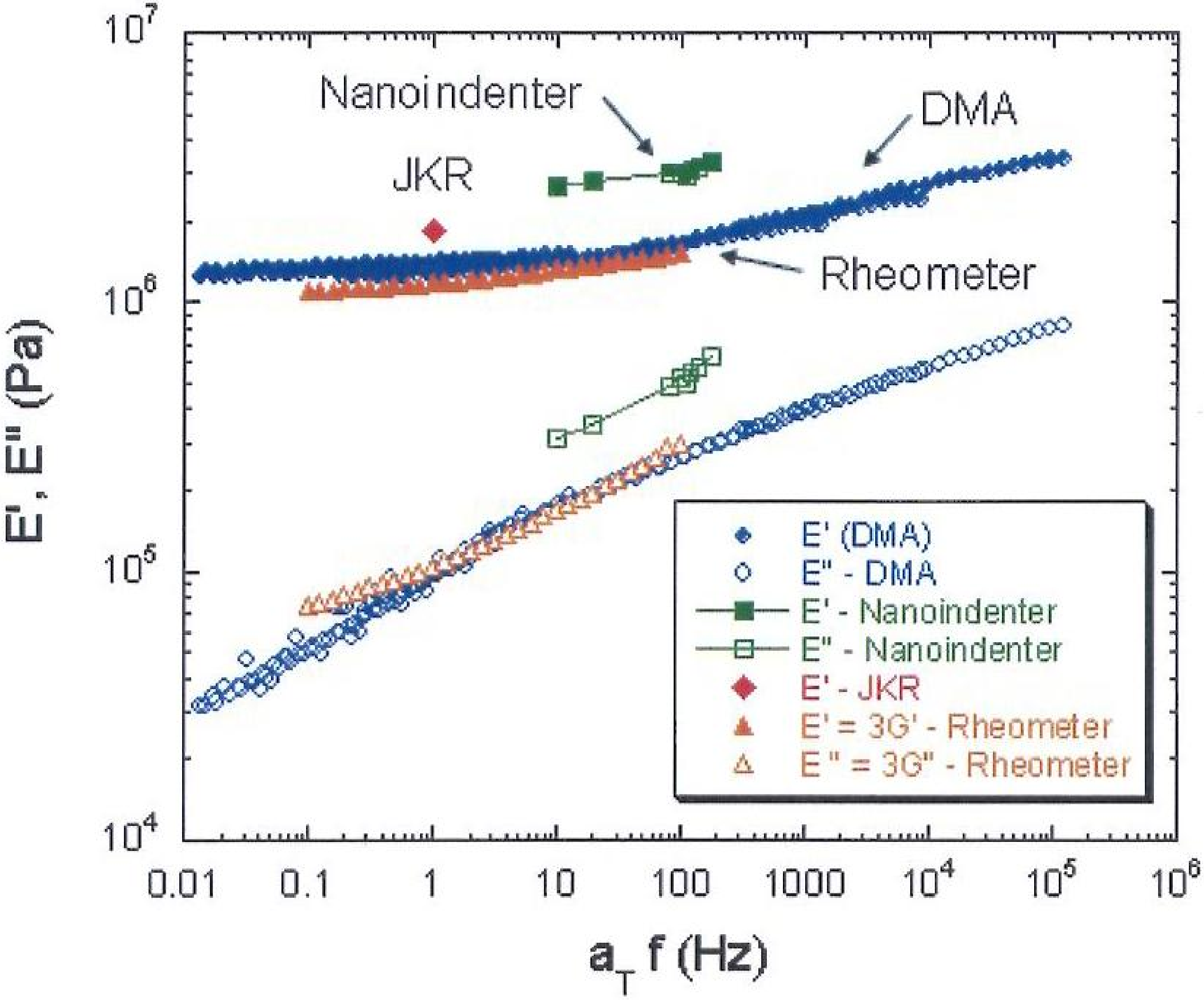
MOTIVATION

- ***Extend the applicability of nanoindentation to time-dependent behavior***
  - – Viscoelastic functions and ***dynamic behavior***
  - – ***Complex modulus***

  - – What we're after:
    - Constitutive behavior of small volumes of viscoelastic solids subjected to sinusoidal loading

$$\sigma = \varepsilon_o E' \sin \omega t + \varepsilon_o E'' \cos \omega t$$

NANOINDENTATION & DMA COMPARISON



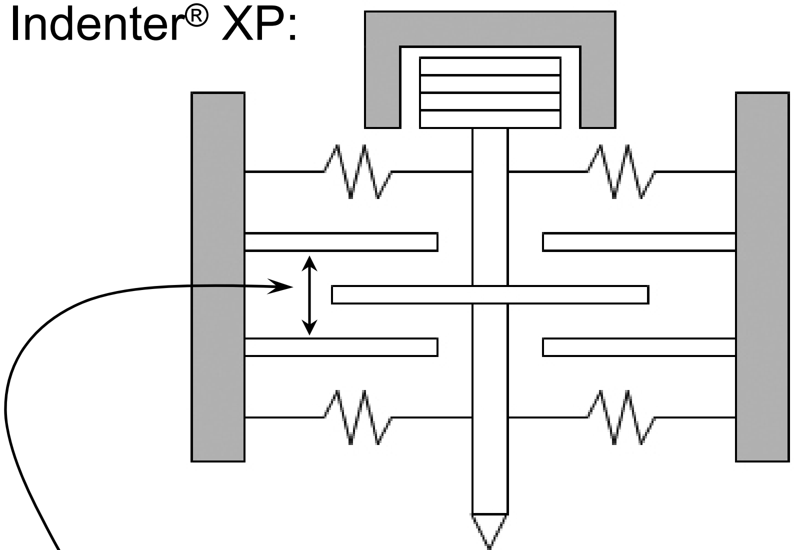
C. C. White et al., Mater. Res. Soc. Symp. Proc. **841** (2005)

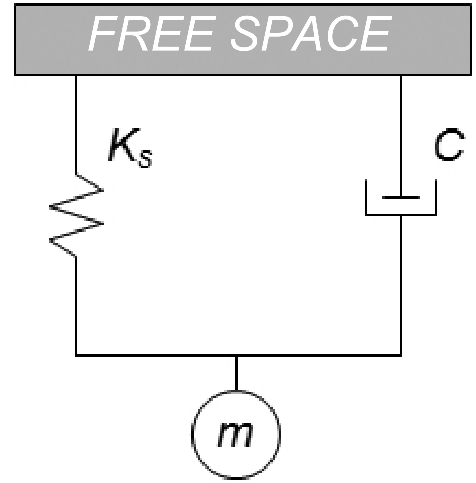
MODELING THE INSTRUMENTATION

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

MODELING THE INSTRUMENTATION

Nano Indenter® XP:





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

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

$$F_o e^{i\omega t} = m\ddot{h} + C\dot{h} + Kh$$

$$h(t) = h_o e^{i(\omega t - \phi)}$$

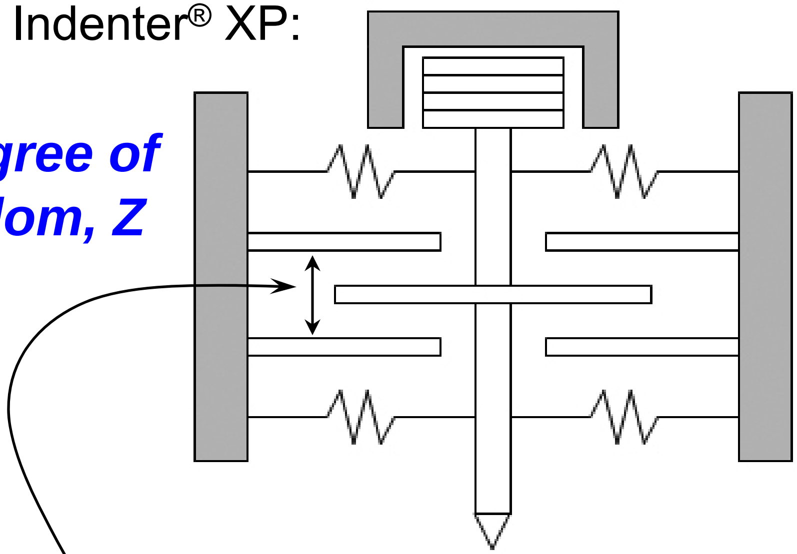
$$\frac{h_o}{F_o} = \left[\left(\left(K - m\omega^2\right)^2 + \omega^2 C^2\right)^{1/2}\right]^{-1}$$

$$\tan\phi = \frac{C\omega}{K - m\omega^2}$$

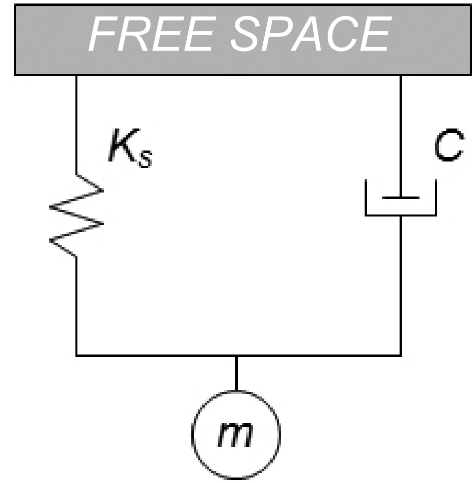
MODELING THE INSTRUMENTATION

Nano Indenter® XP:

1 degree of freedom, Z



Raw displacement, ± 1 mm



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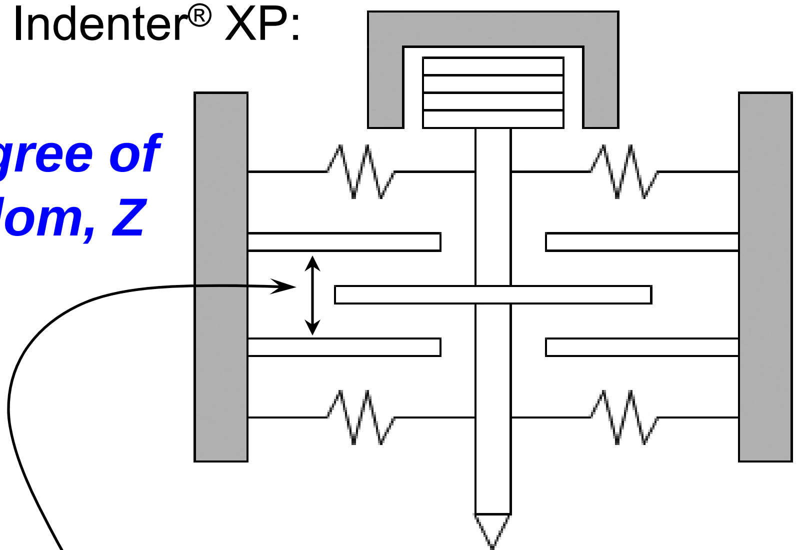

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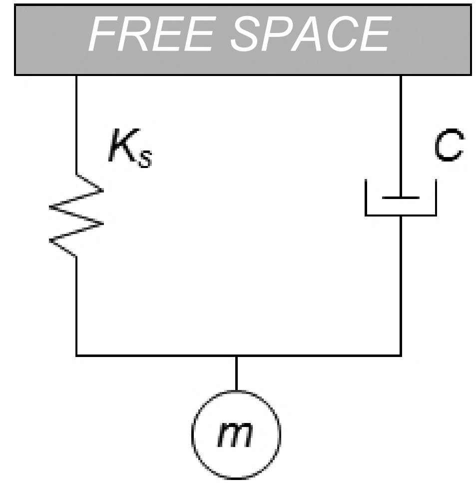

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FUNCTION OF POSITION

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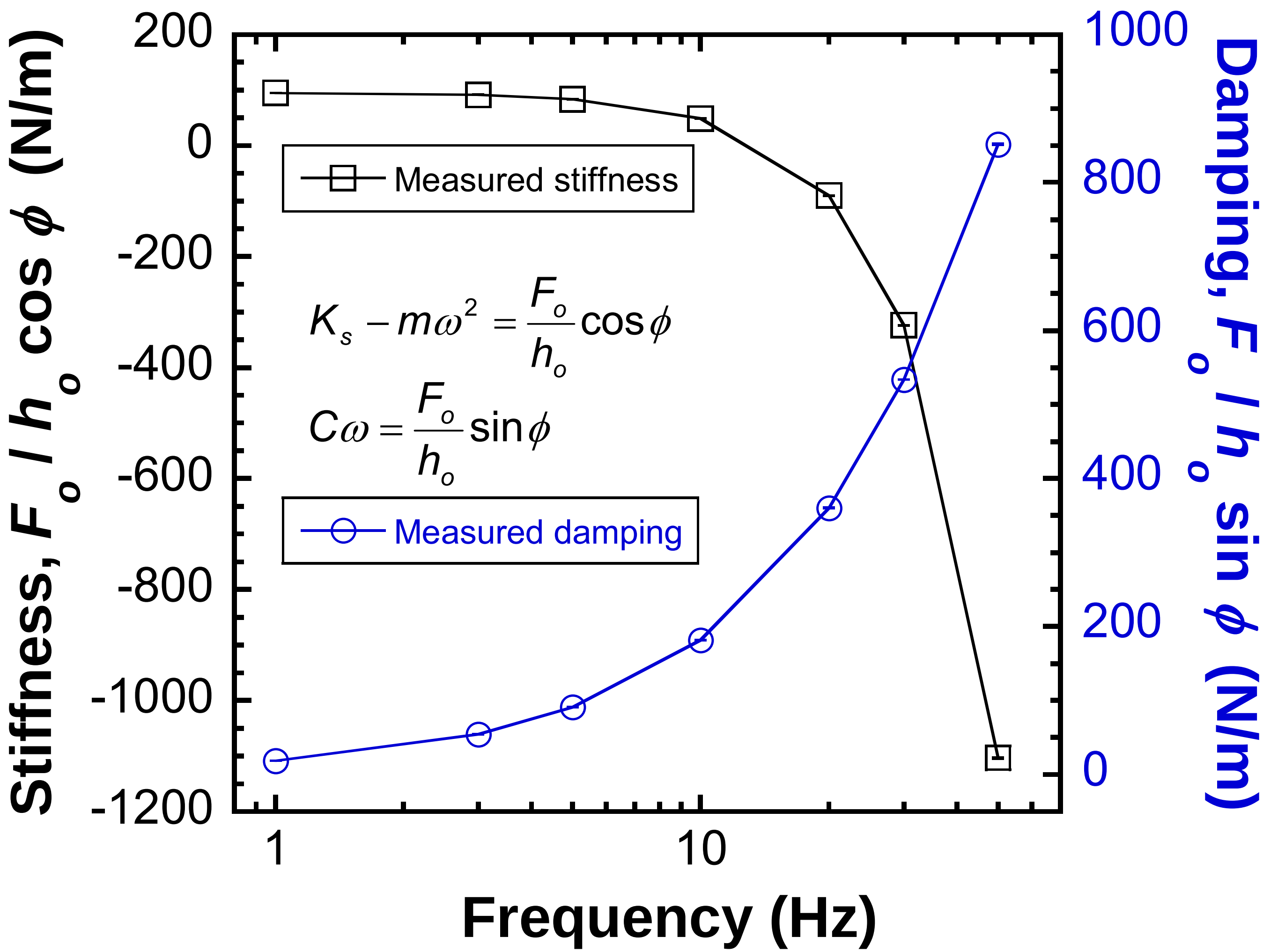
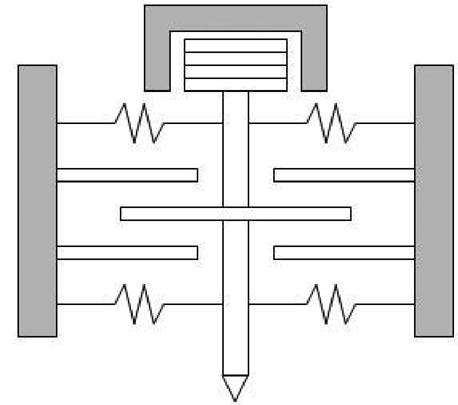
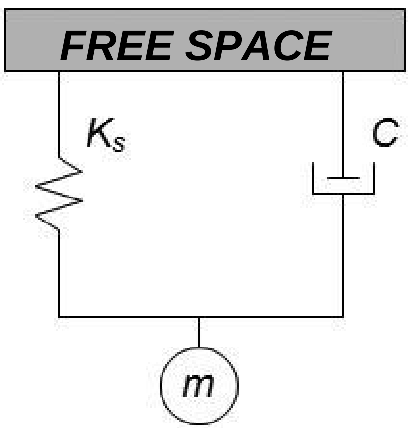
Measured stiffness and damping in free space, position = 18.8 μm







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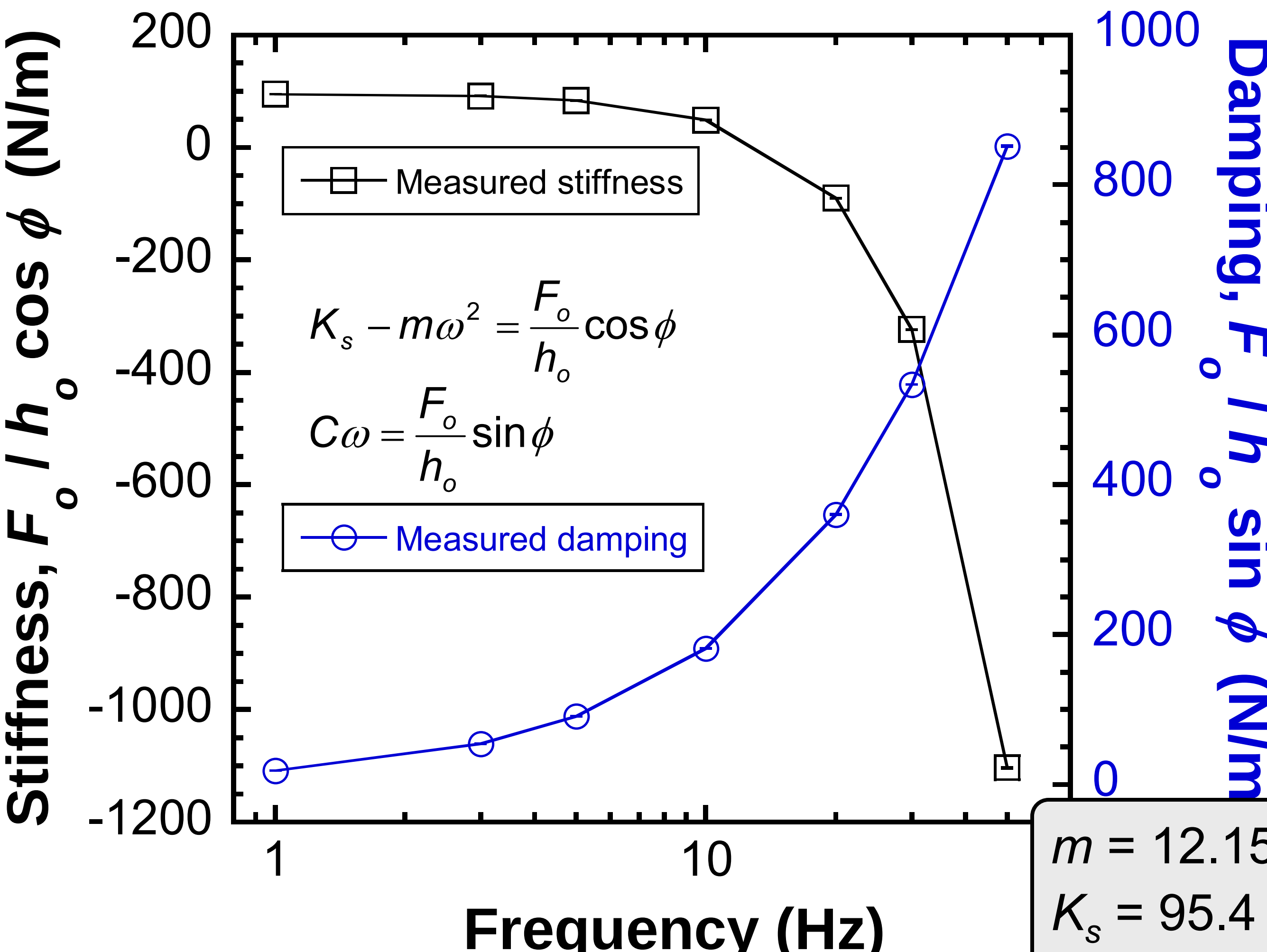

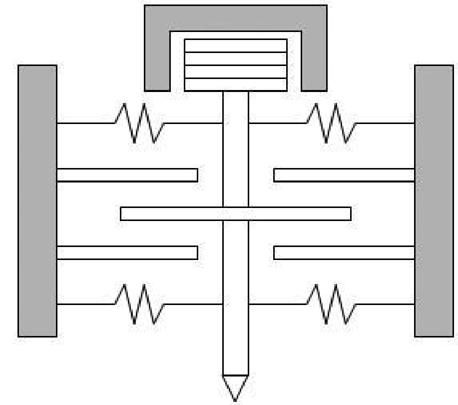

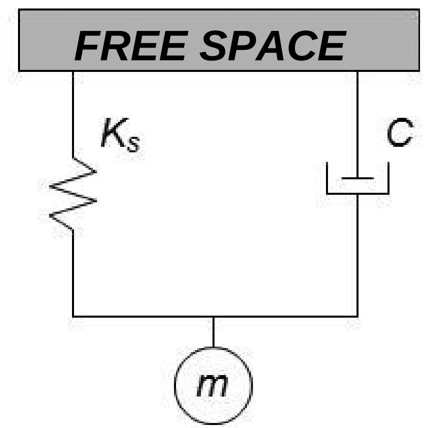

m = 12.15 g
K_s = 95.4 N/m
C = 2.81 Ns/m

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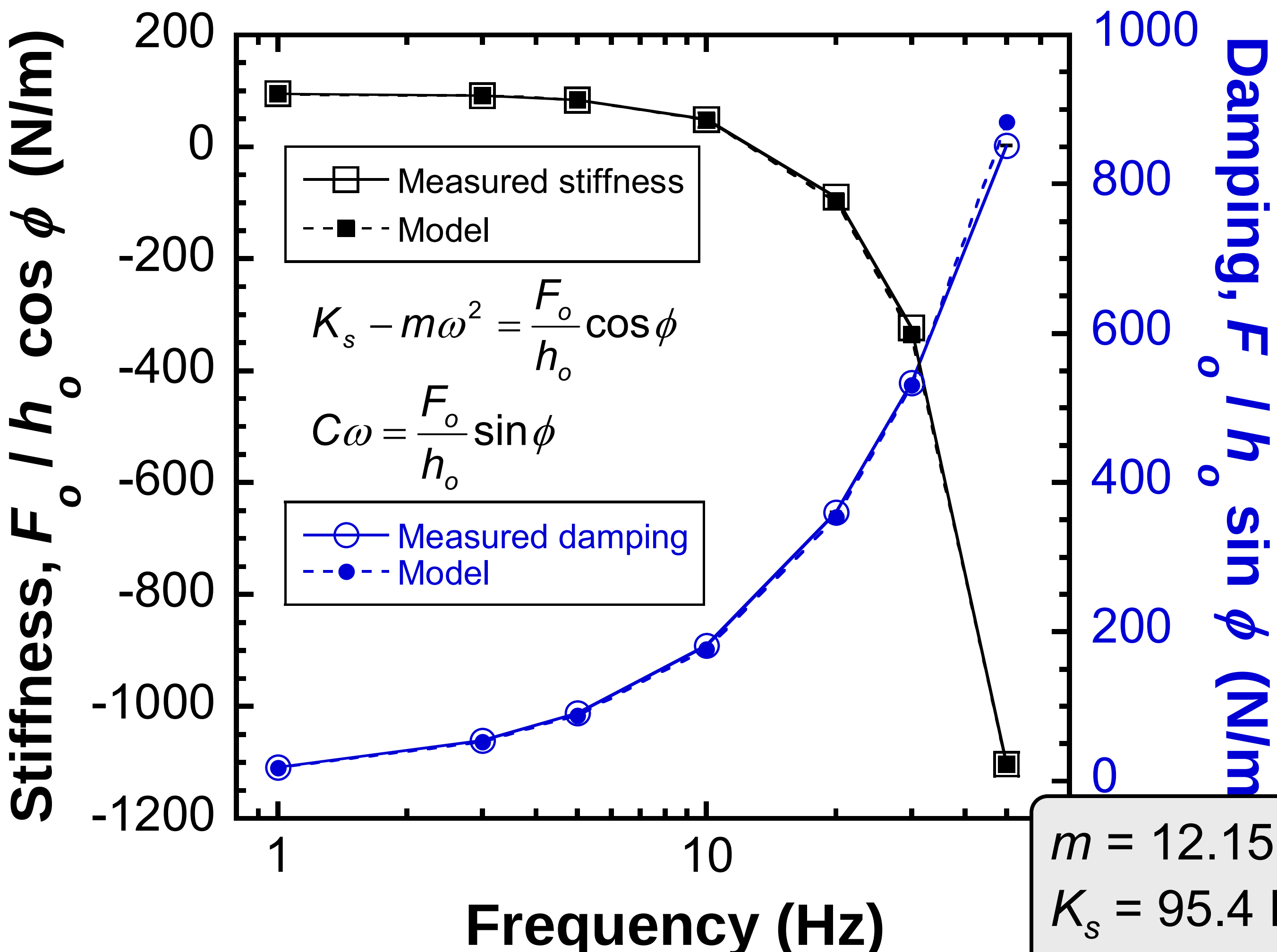

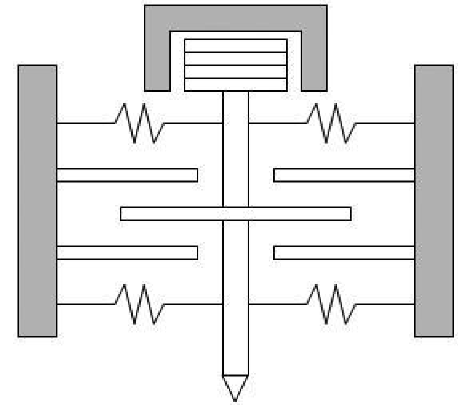

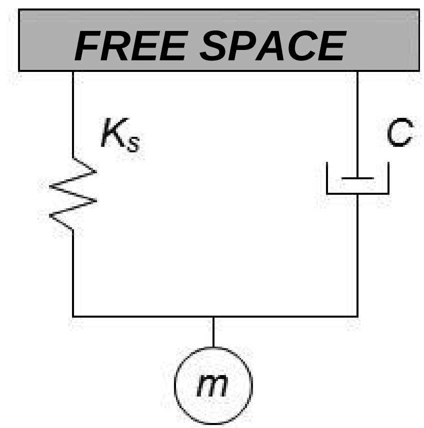

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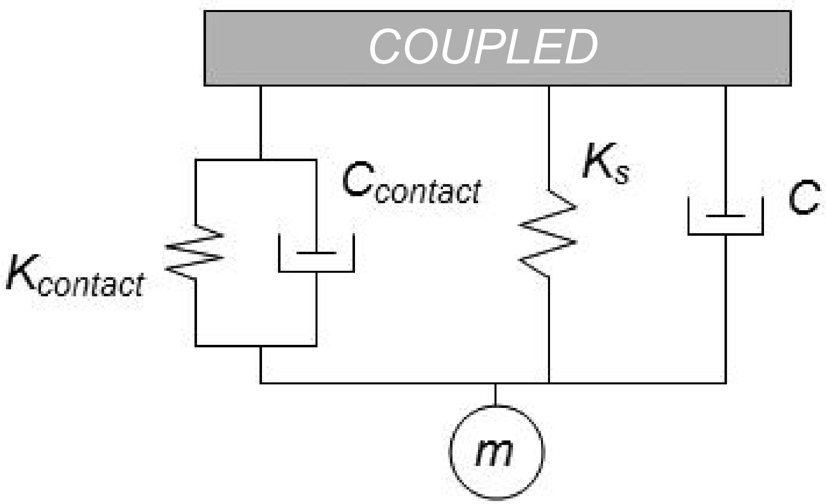
ADD THE CONTACT



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

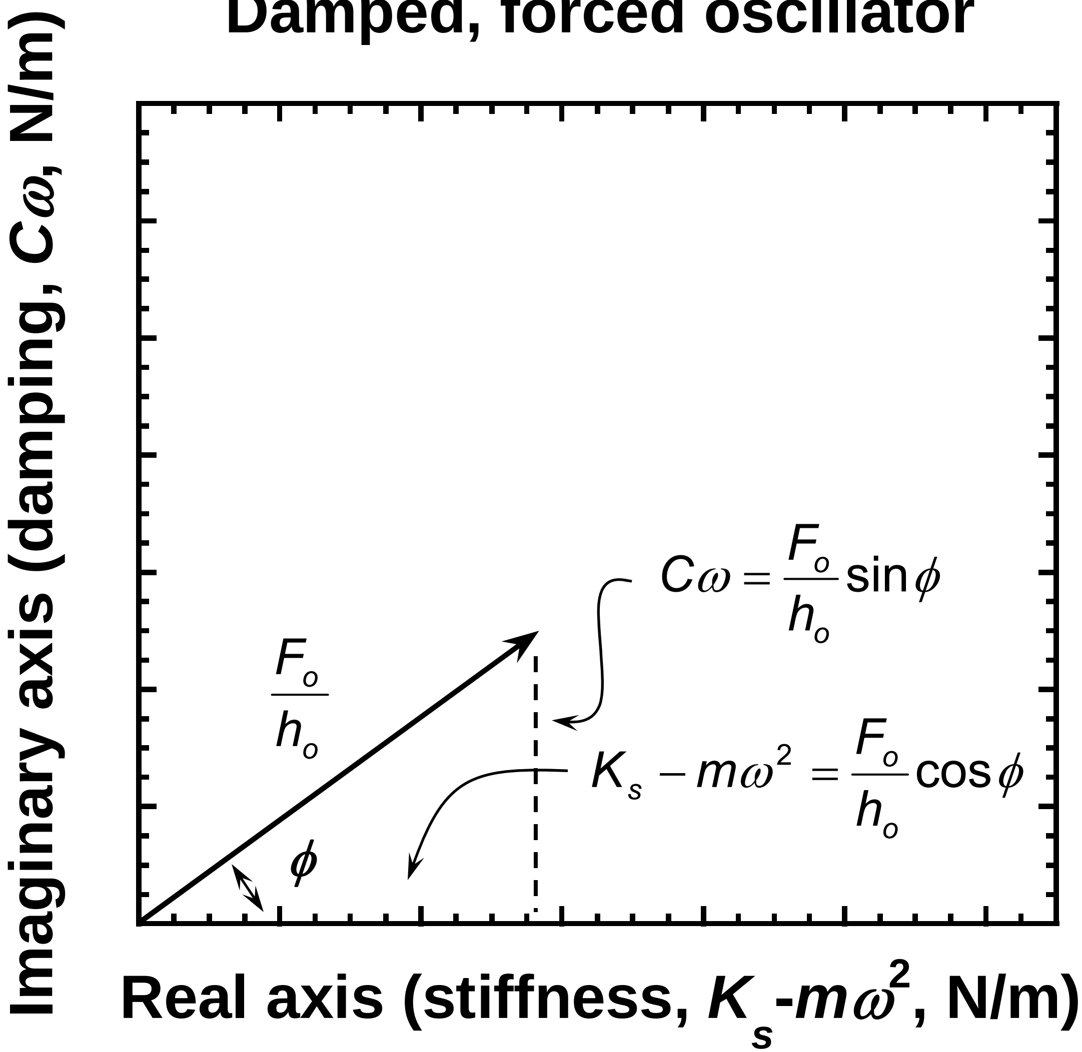
$$C = \frac{F_o}{h_o}\frac{\sin\phi}{\omega}$$

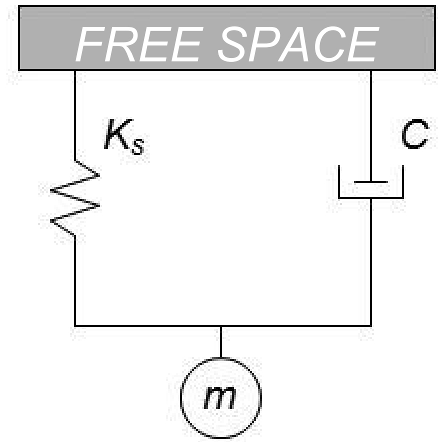
COUPLED RESPONSE = SAMPLE + INSTRUMENT

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

PHASOR DIAGRAM: PHYSICAL INSIGHT

Damped, forced oscillator





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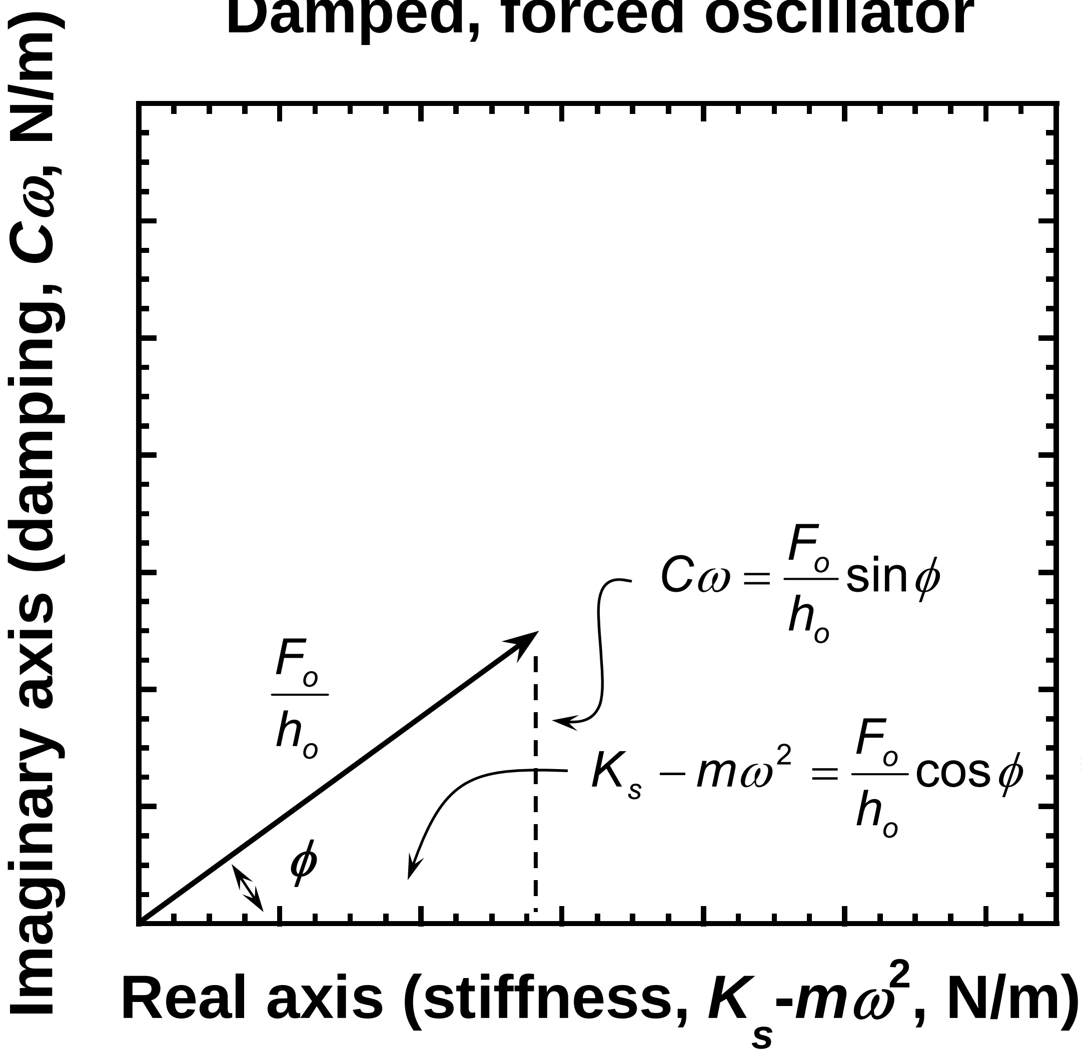

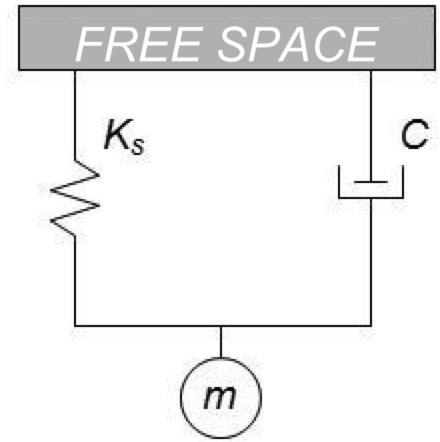

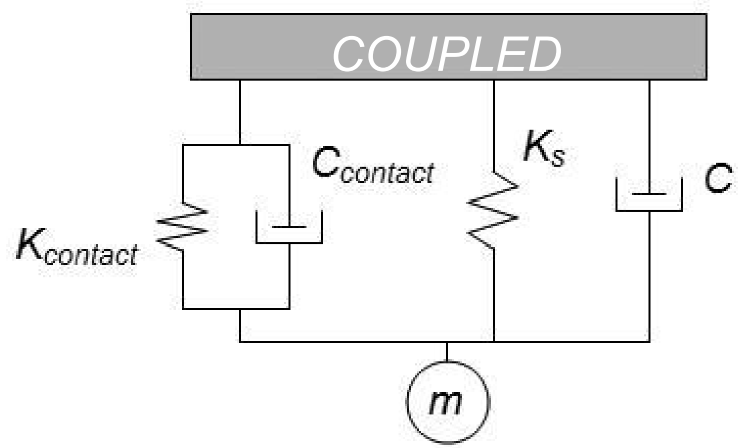

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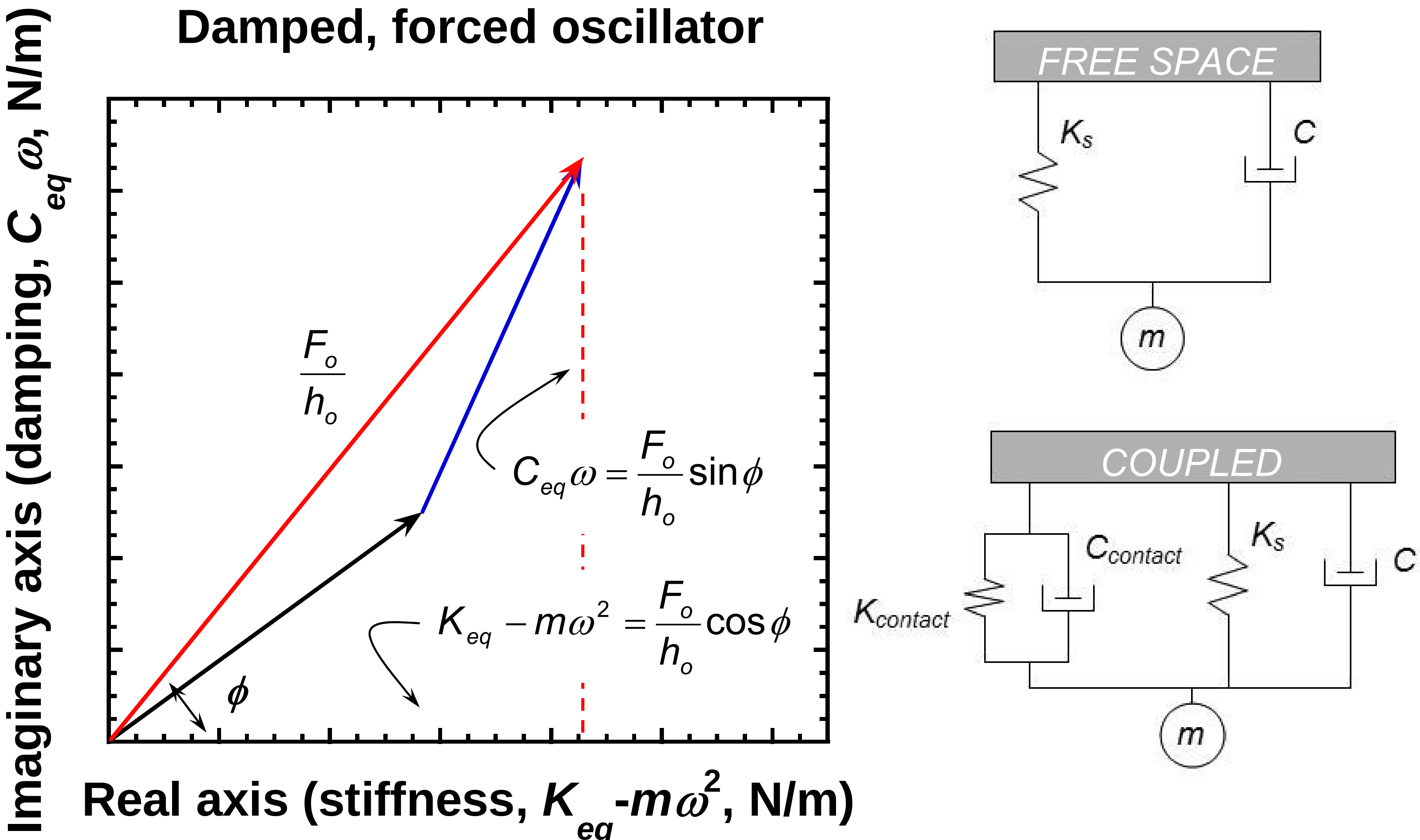

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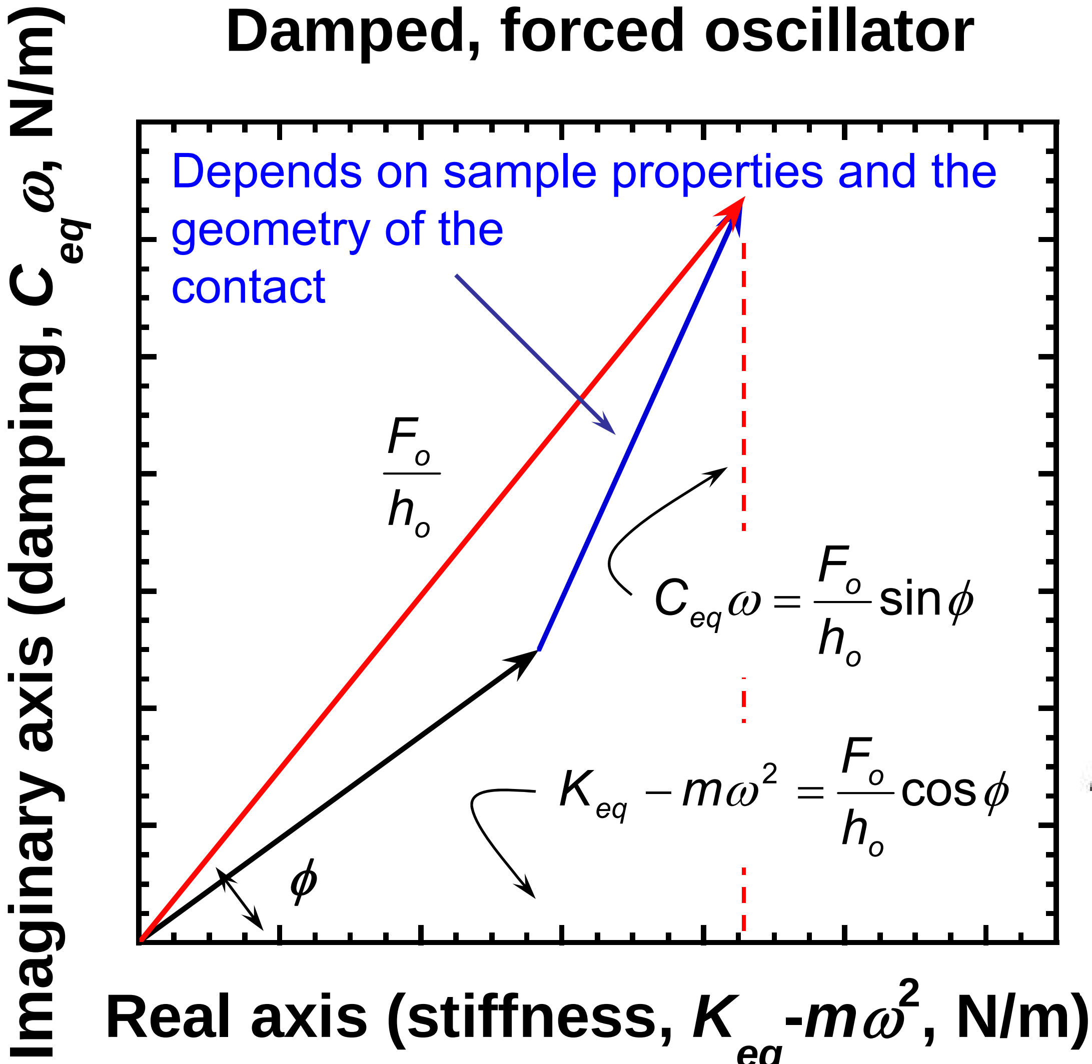
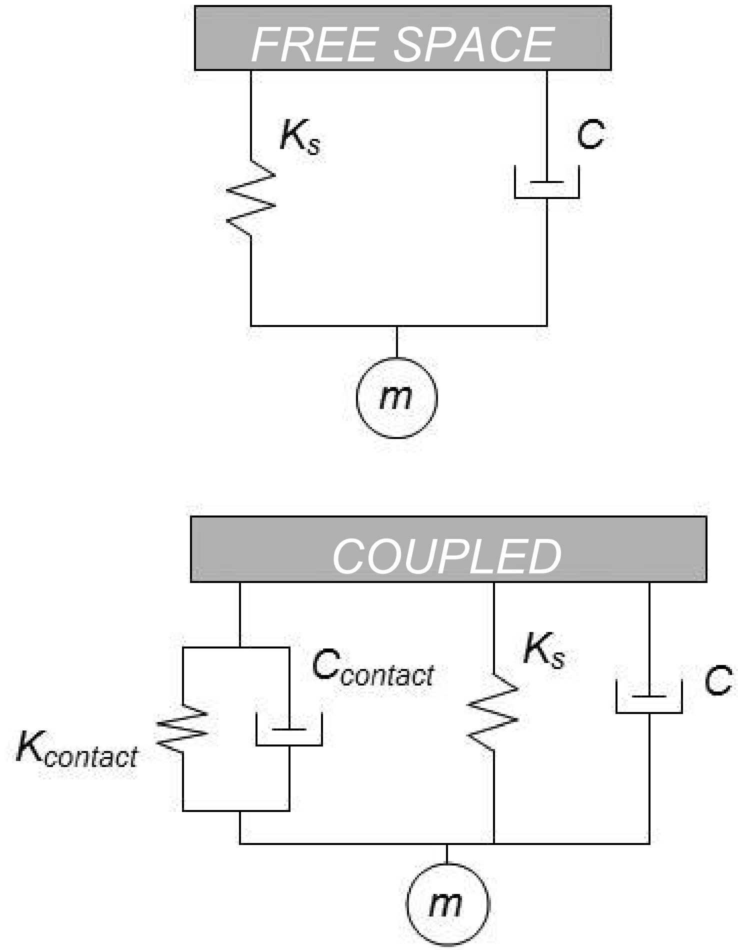
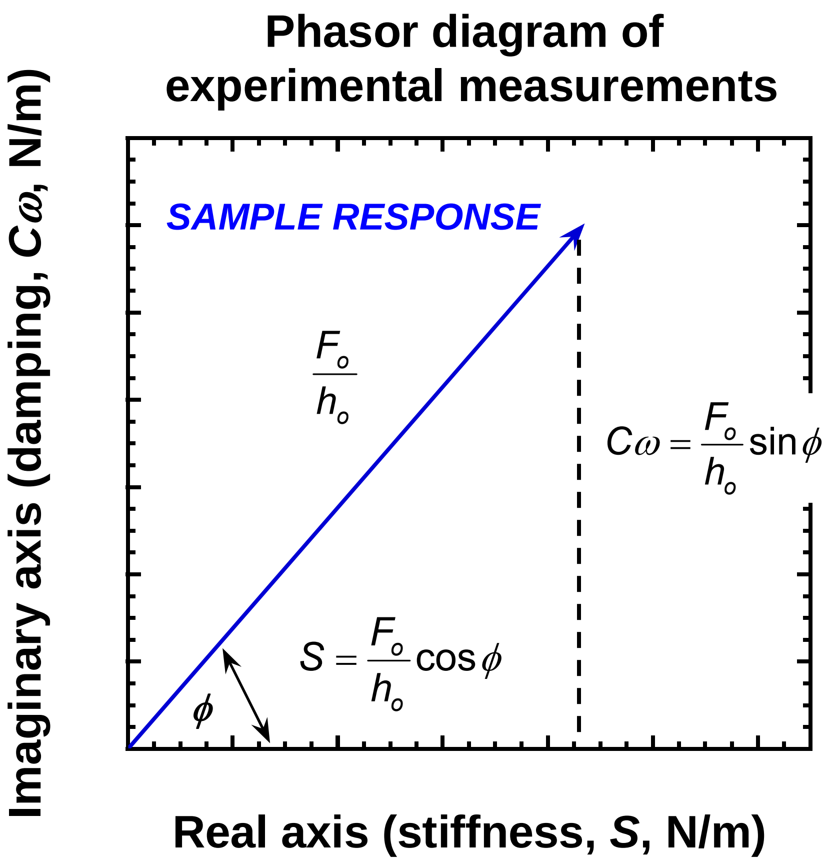
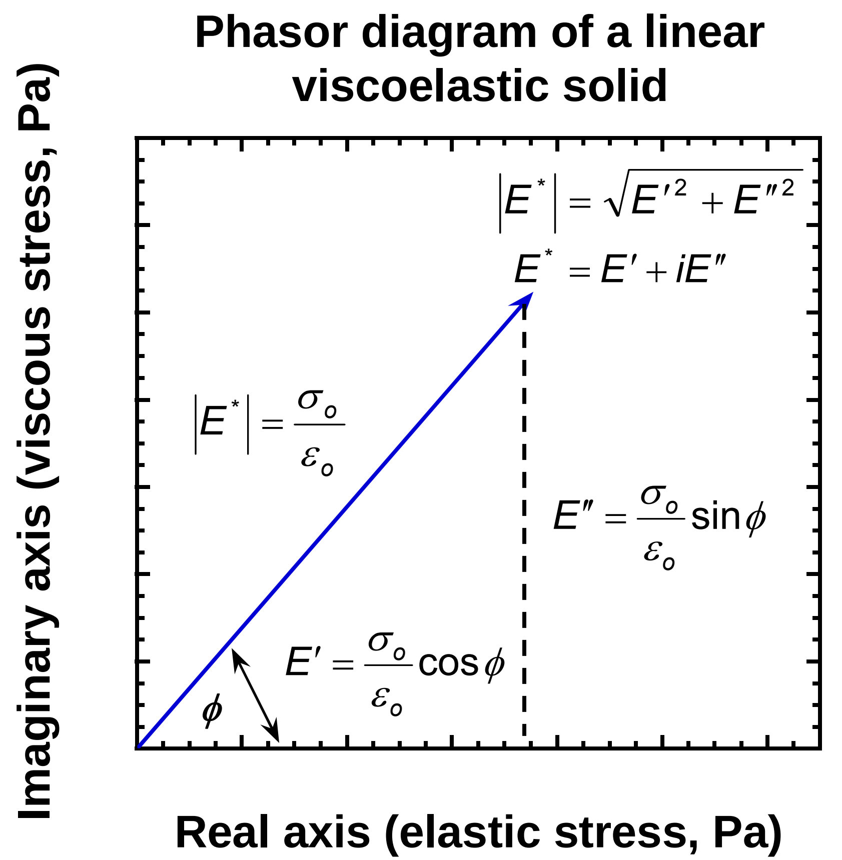




FROM *S* AND *Cω* → *E*' AND *E*"





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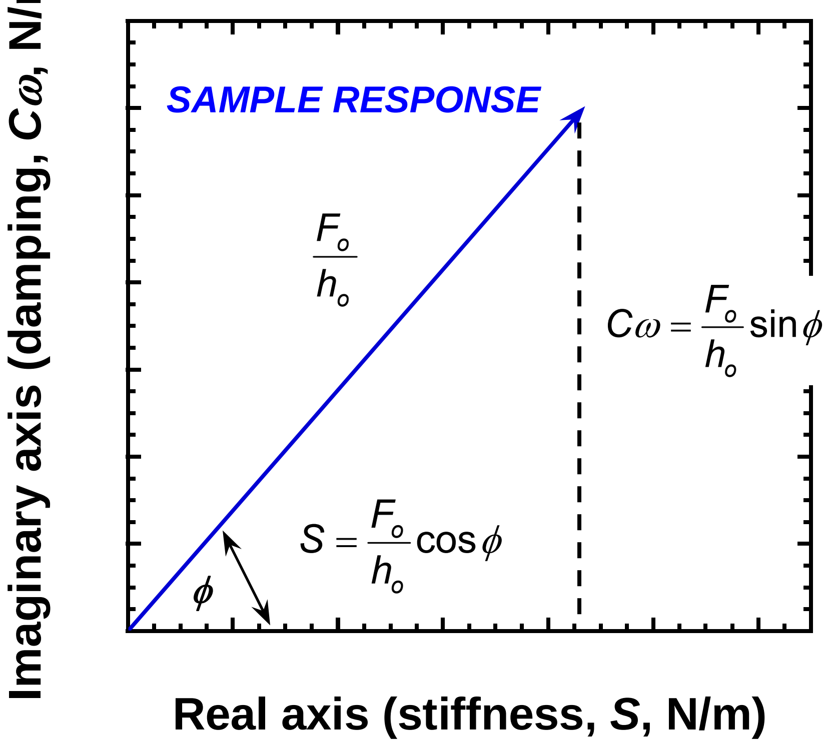
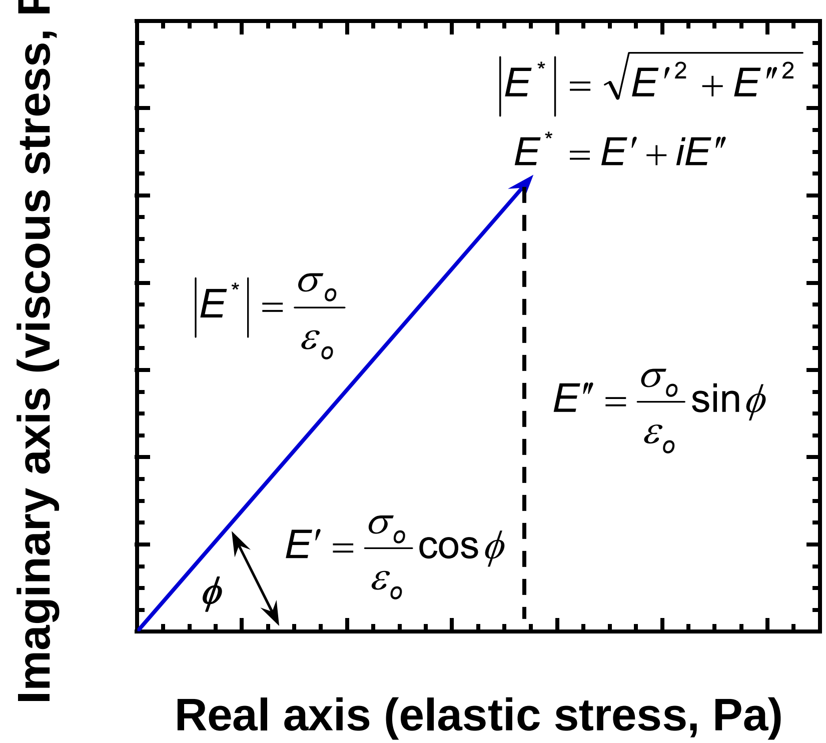
FROM S AND $C\omega \rightarrow E'$ AND E''

Phasor diagram of experimental measurements



Phasor diagram of a linear viscoelastic solid

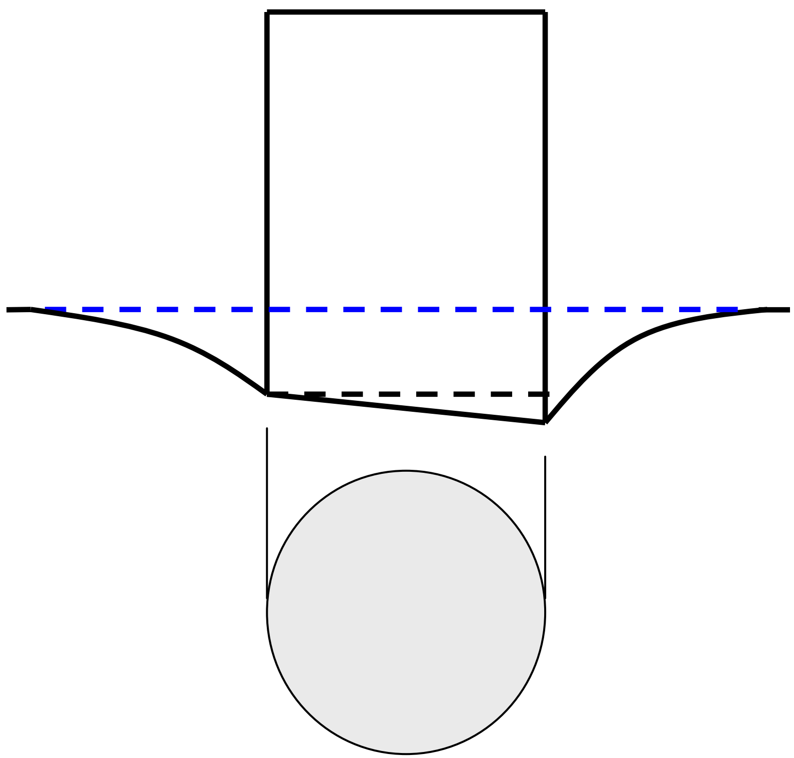


The fundamental equation of nanoindentation:

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

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

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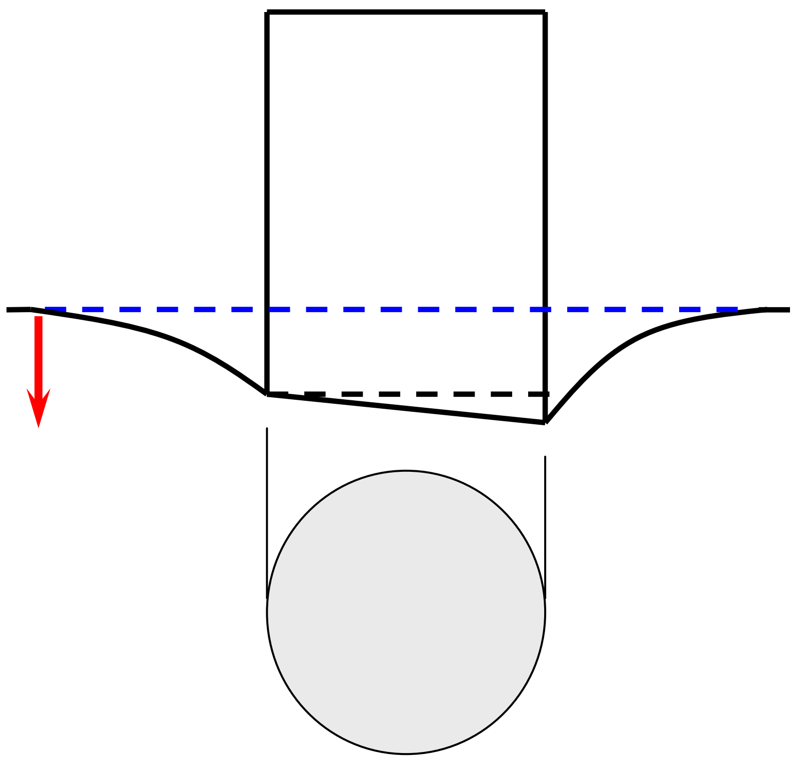

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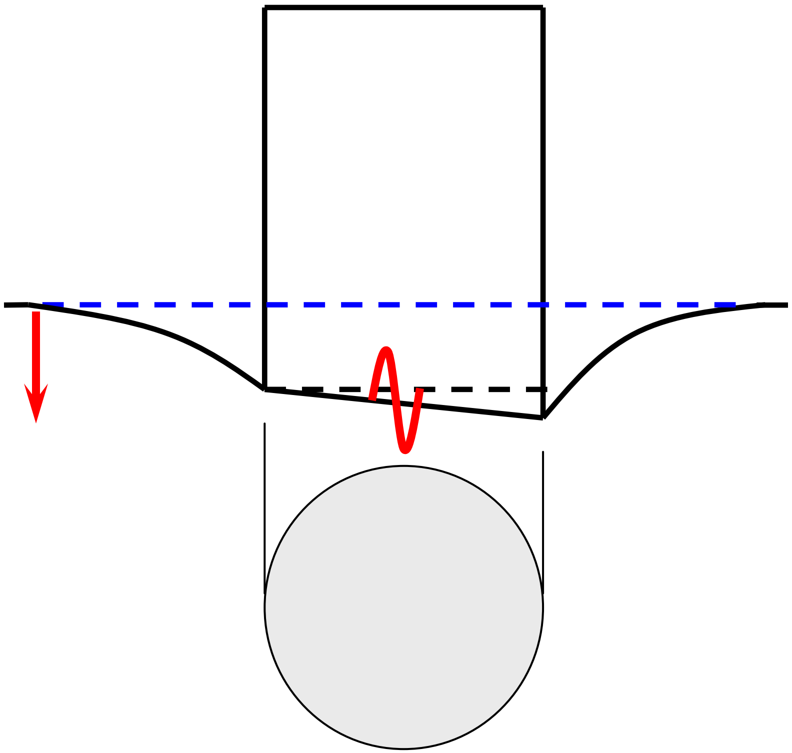

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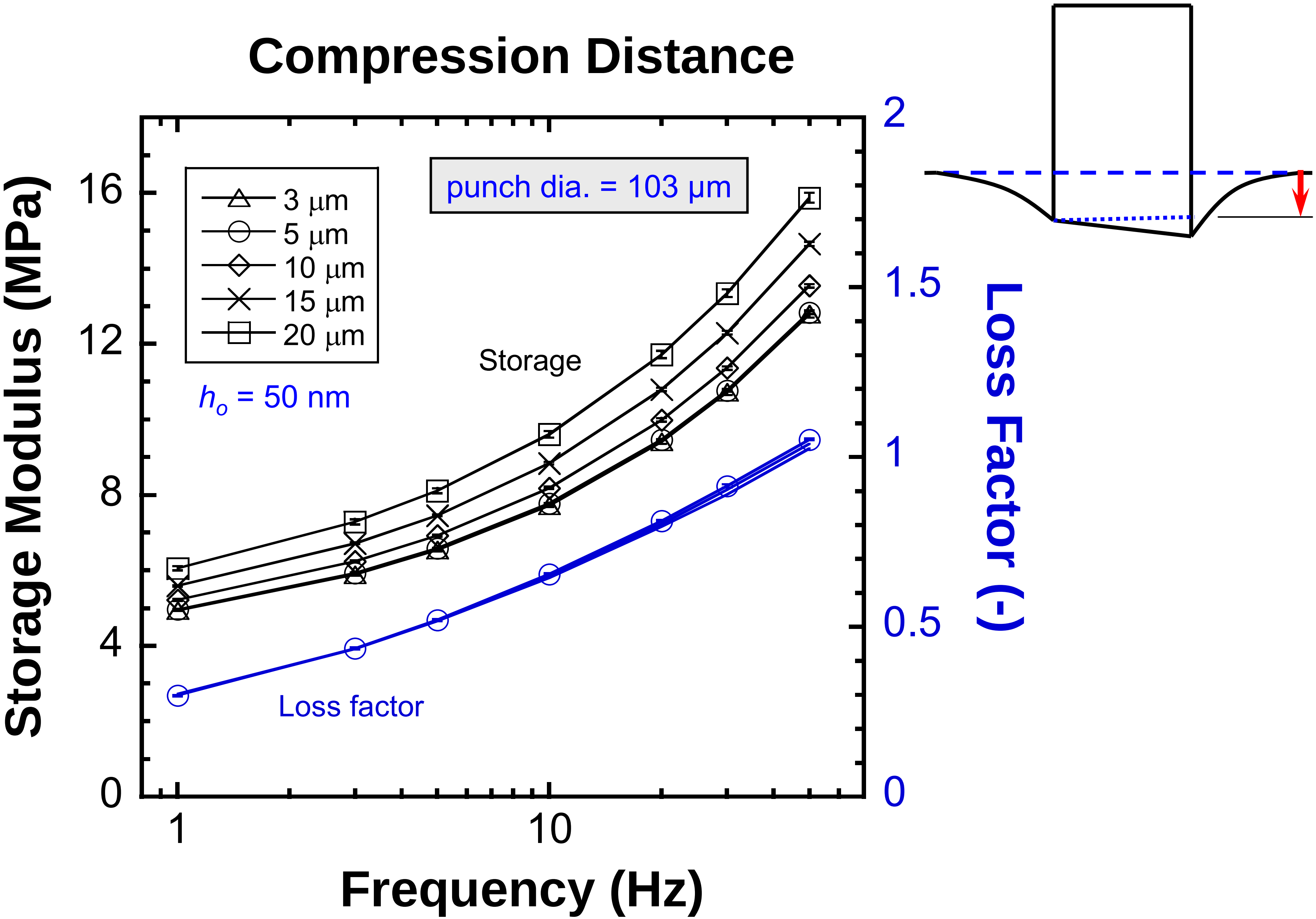
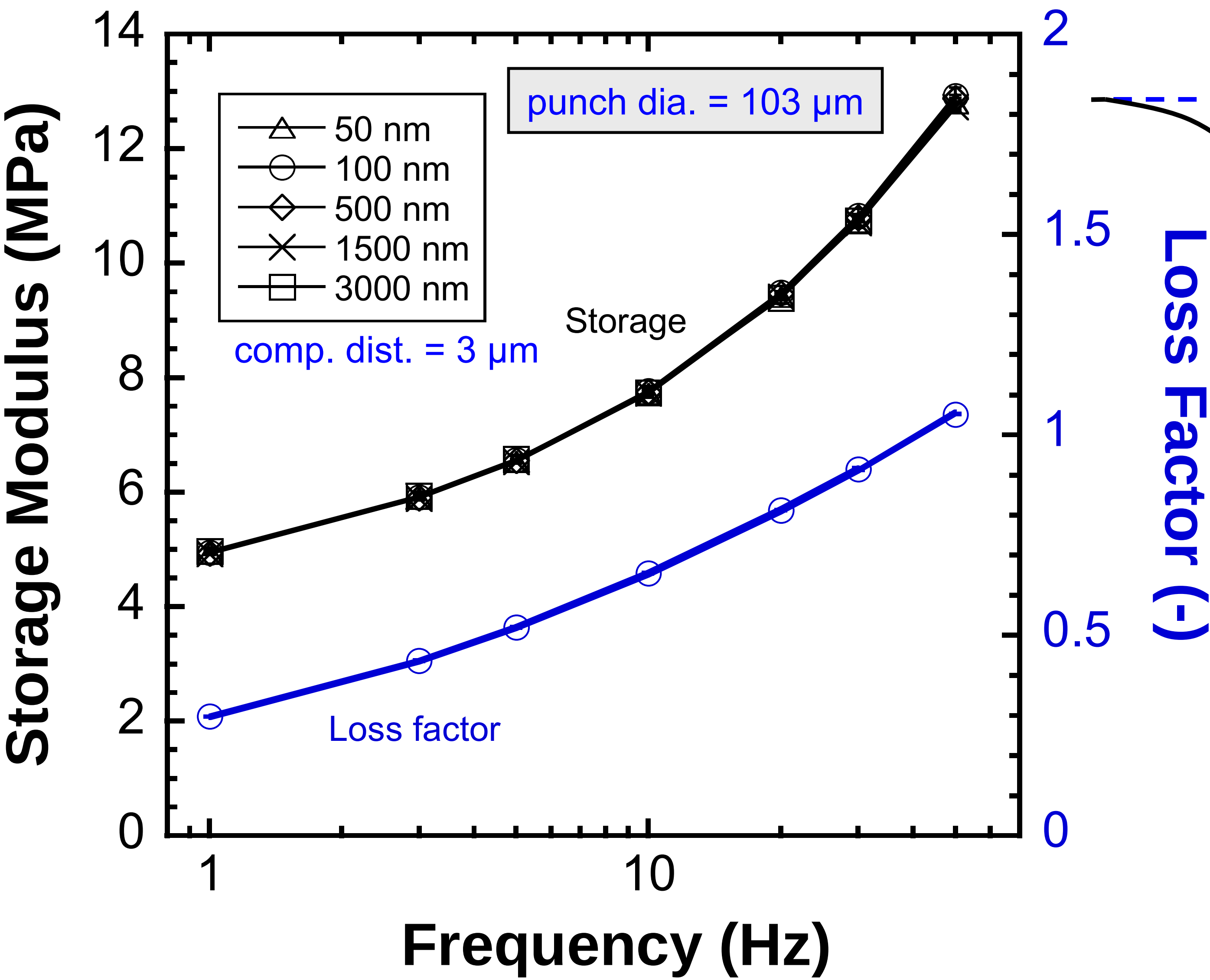
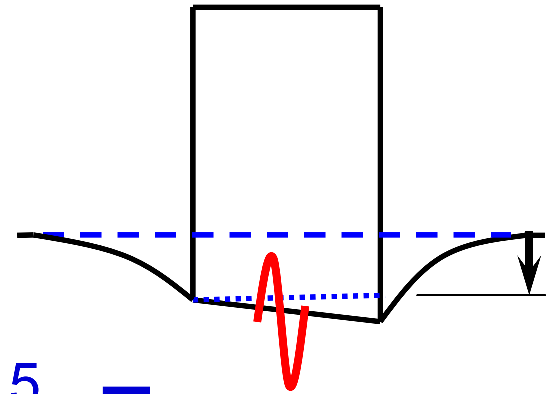
Compression Distance



Oscillation Amplitude





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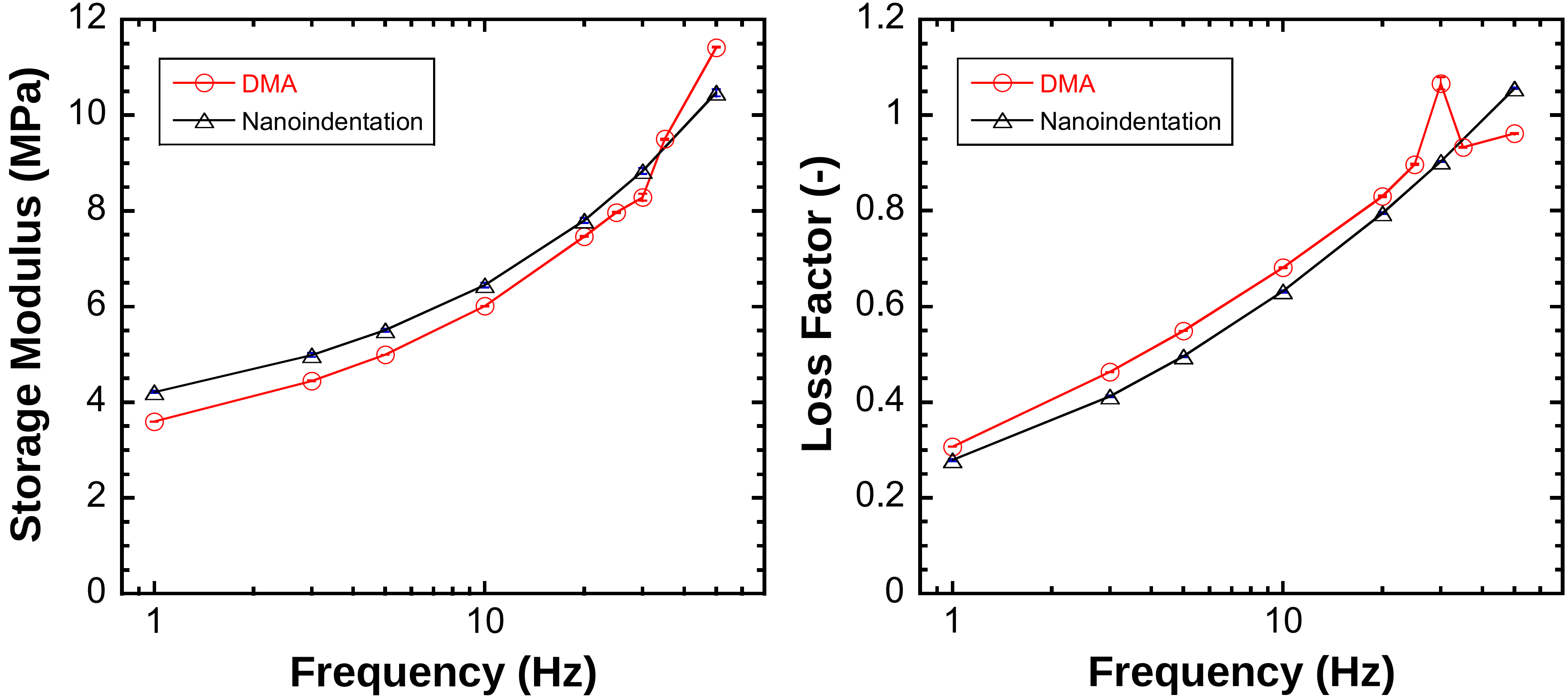
DMA VS. NANOINDENTATION

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



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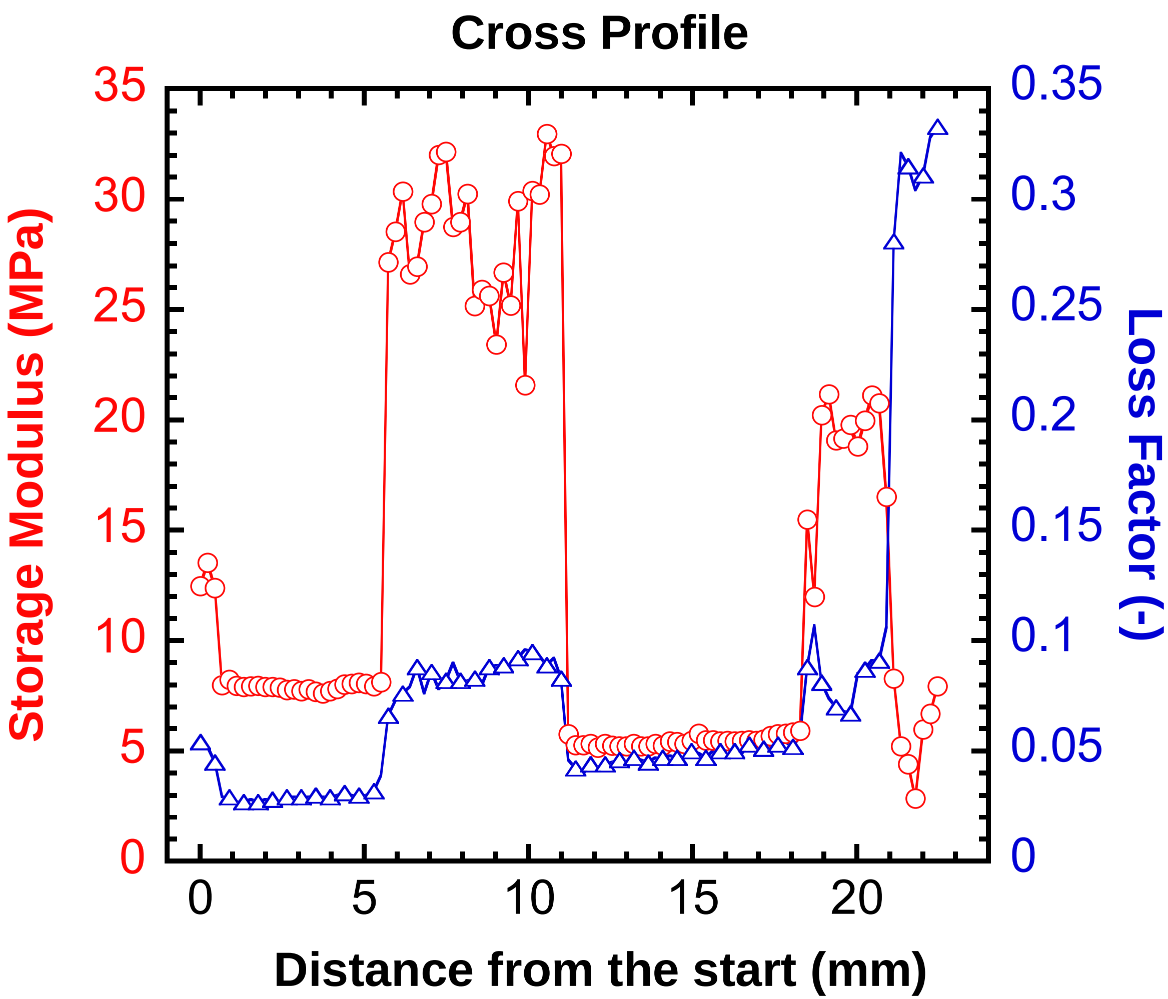
APPLICATION: GOODYEAR TIRE AND RUBBER CO.

Courtesy Tom Fleischman and Remi Granier



- Lab cure vs. actual
- 45 μm dia. flat punch
- 10 Hz
- 103 measurements
- 90 seconds per test
- No failed tests

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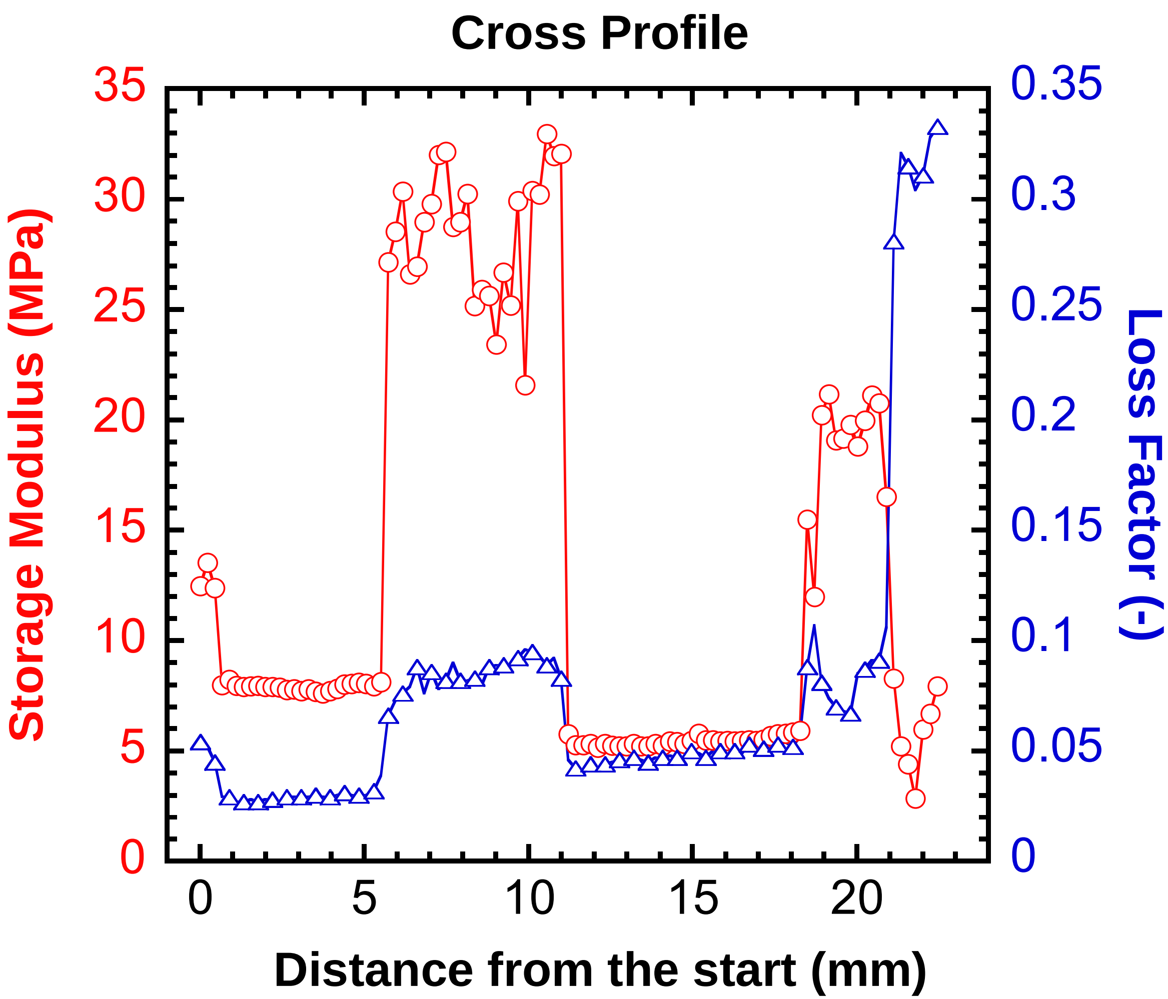

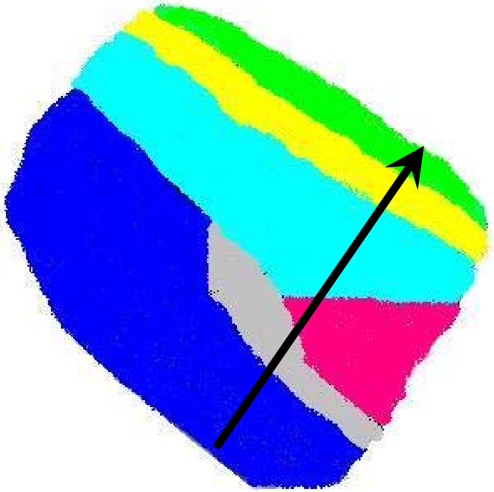

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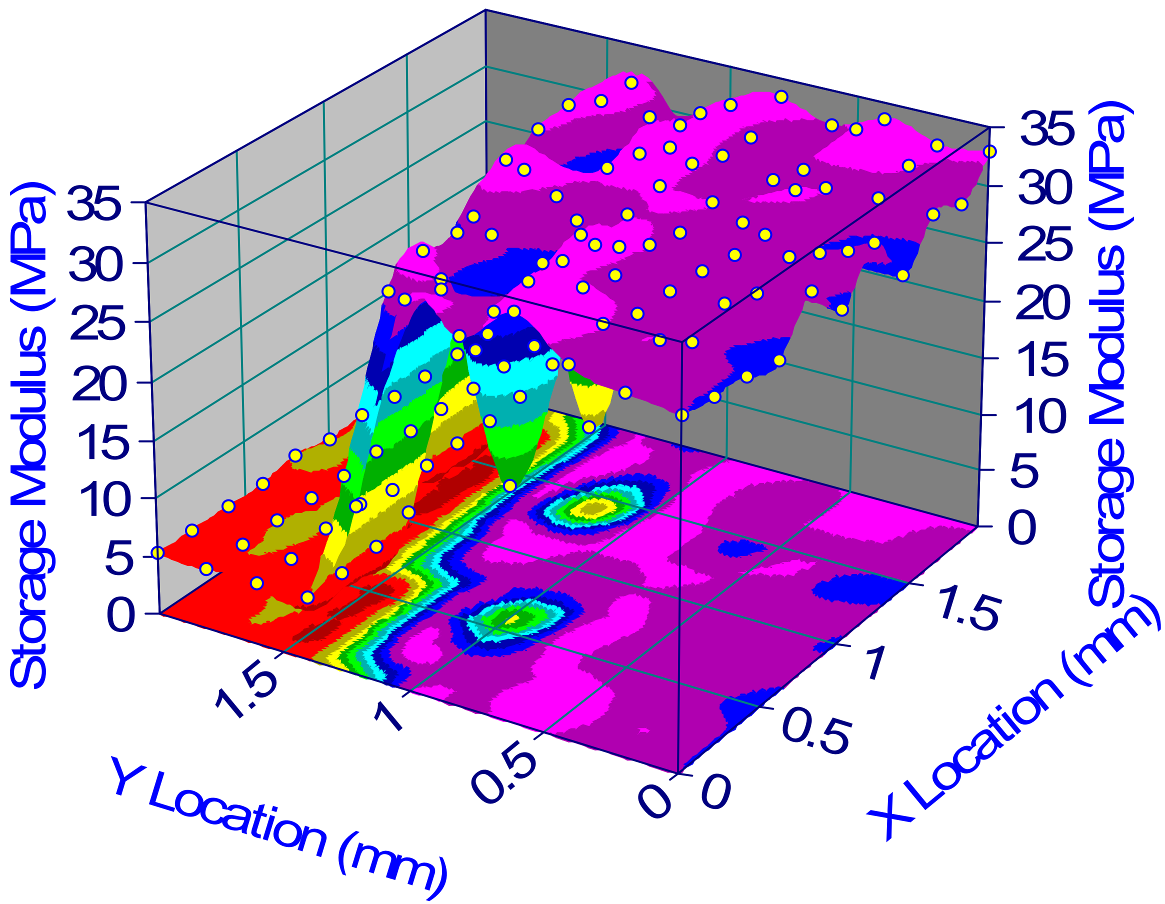

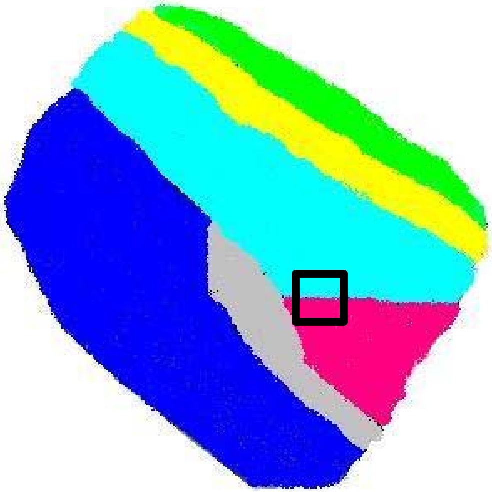

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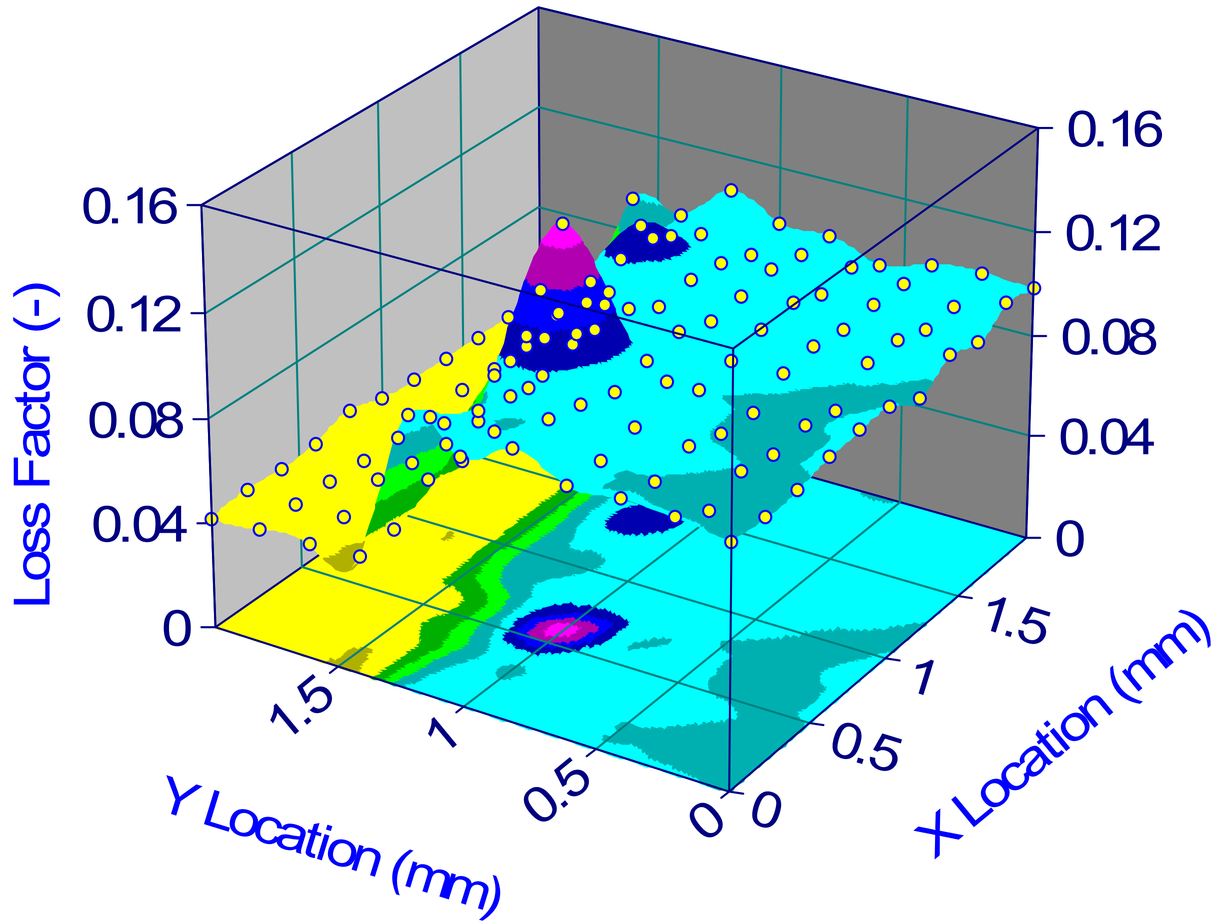

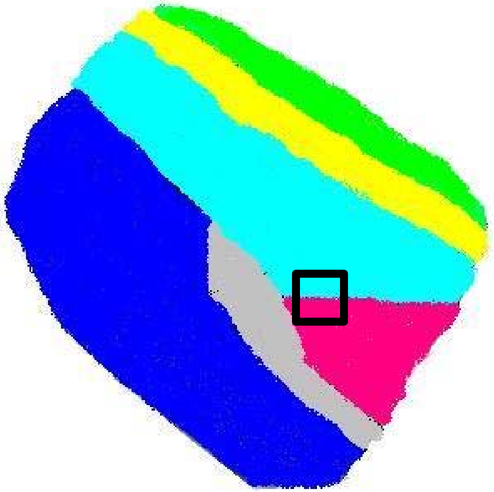

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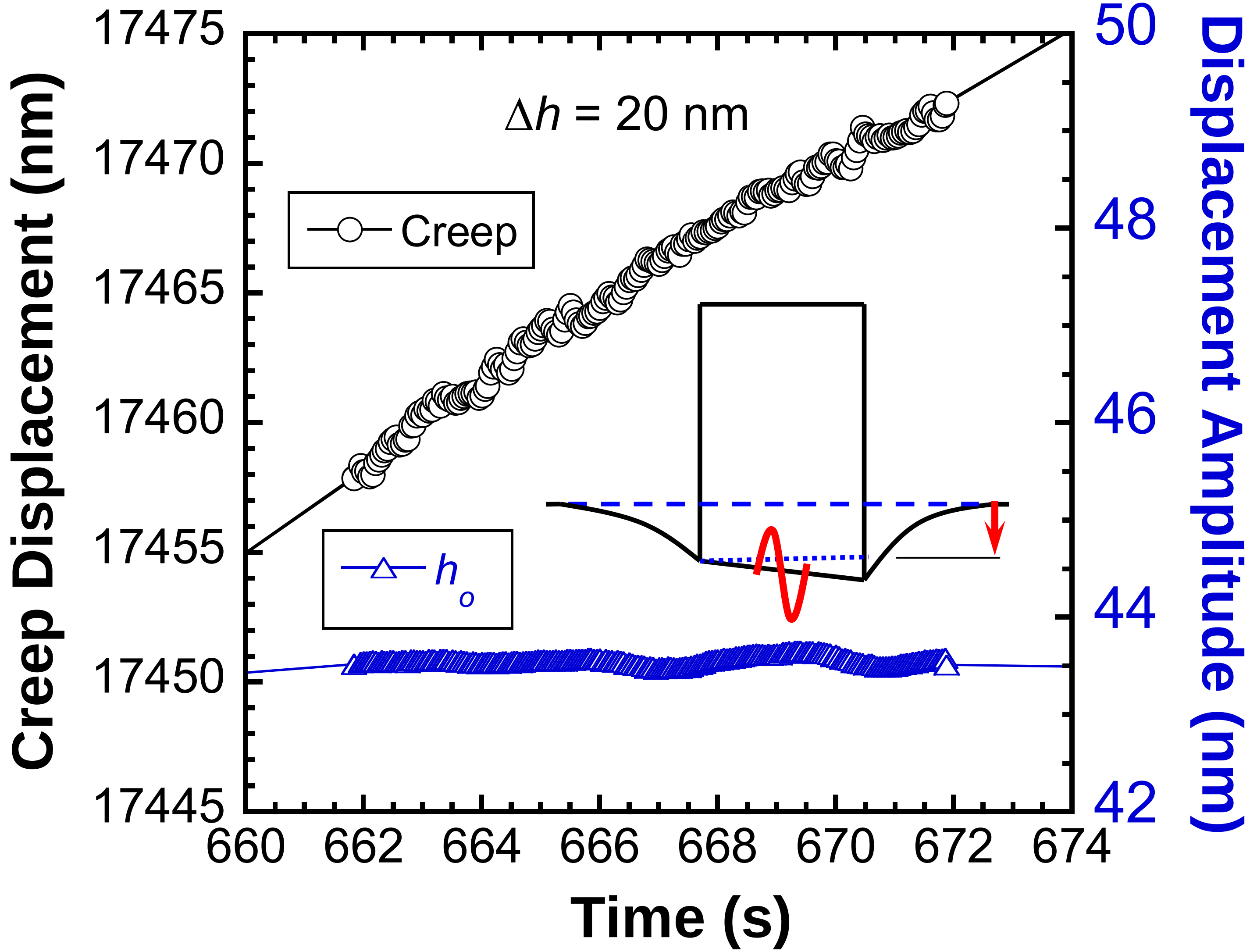
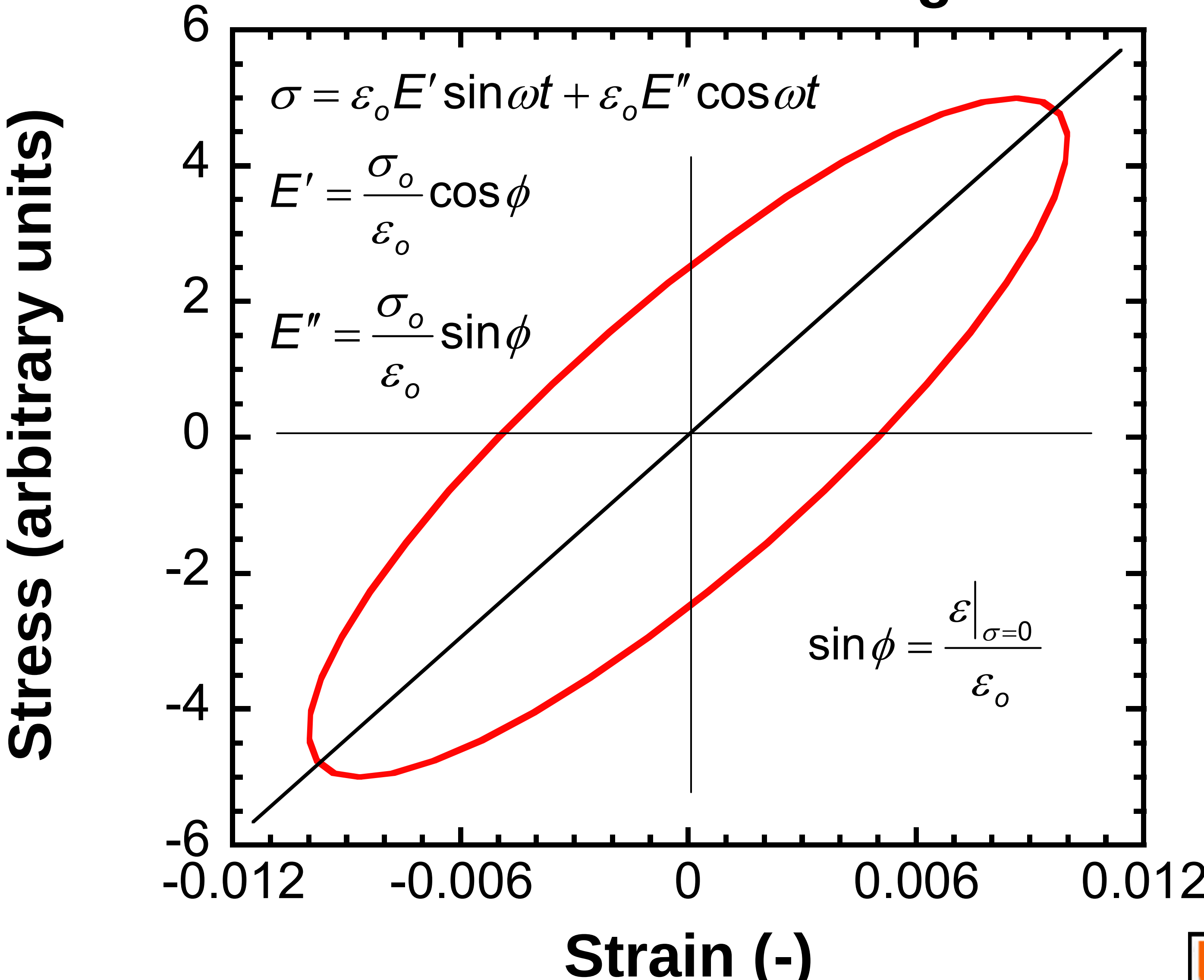
CLOSING REMARKS

- Goals of a dynamic nanoindentation experiment:
  - Robust dynamic characterization of the instrument
  - Known contact geometry
  - Steady-state harmonic motion
  - Linear viscoelasticity
- If these criterion are met, dynamic nanoindentation can be used to make accurate and precise measurements of the complex modulus.
- These experiments can be performed in a completely automated fashion, in 90 seconds or less, and with a 100% success rate – no failed measurements.

Steady-State Harmonic Motion



Linear viscoelasticity, sinusoidal loading



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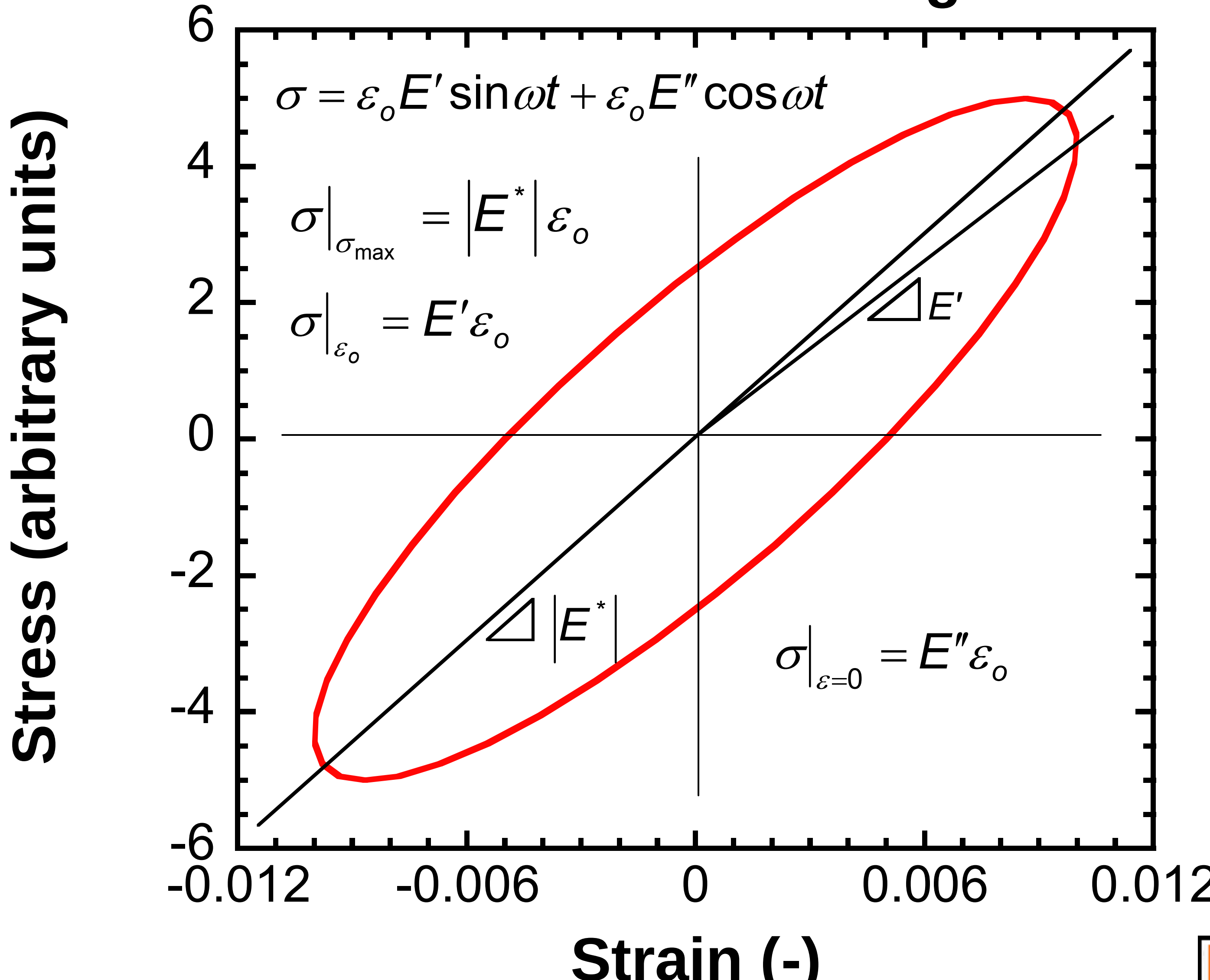

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Full Contact