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# Calibration of Nanoindentation Data

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# Project Relation to LIGO

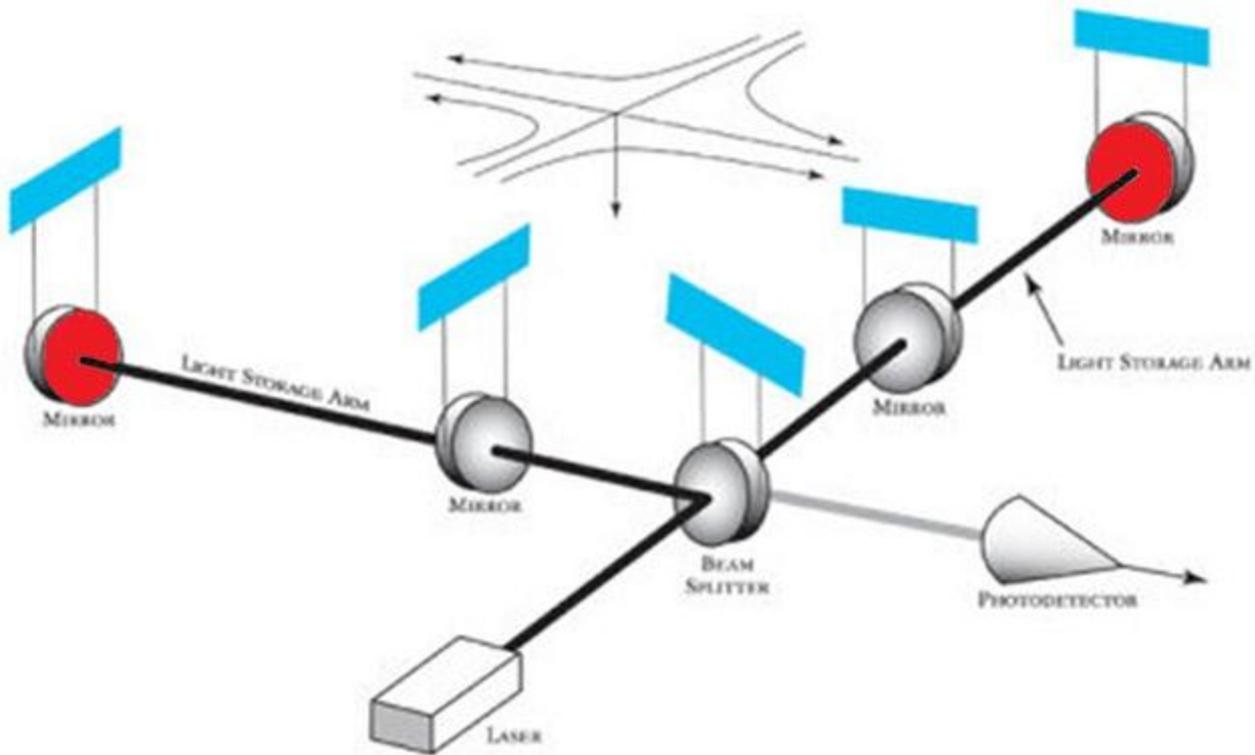


Photo credit: LIGO

# Noise Sources in Coatings

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- Three main sources of noise from the coatings:
  - » Brownian
  - » Thermo-elastic
  - » Thermo-refractive

# Brownian Noise

- Brownian thermal noise from a multilayer mirror:

$$S_x(f) = \frac{2k_B T}{\pi^{3/2} f} \frac{1}{\omega Y} \left\{ \phi_{\text{substrate}} + \frac{1}{\sqrt{\pi}} \frac{d}{\omega} \left( \frac{Y'}{Y} \phi_{\parallel} + \frac{Y}{Y'} \phi_{\perp} \right) \right\}$$

where  $k_b$  is the Boltzmann constant,  $T$  is temperature,  $f$  is frequency,  $Y$  is the Young's modulus of the substrate,  $Y'$  is the Young's modulus of coating,  $d$  is the thickness of the coating,  $\omega$  is the radius of the laser beam at  $1/e^2$  of maximum light intensity,  $\phi$  are the mechanical losses of the substrate, and the parallel and perpendicular directions of the coating

- Assuming  $\phi_{\parallel}$  and  $\phi_{\perp}$  are equal, and setting  $Y'=72$  GPa and  $Y=140$  GPa, changing  $Y'$  by 20% causes the noise to change by approximately 12%.

# Ways to Measure Young's Modulus

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- Acoustics
  - » Good for finding mechanical properties of thin films (on the order of microns)
- Nanoindentation
  - » Good for finding mechanical properties on the atomic scale
- Purpose
  - » Determine if thin film and atomic mechanical properties of a material are the same as the bulk properties

# Nanoindentation

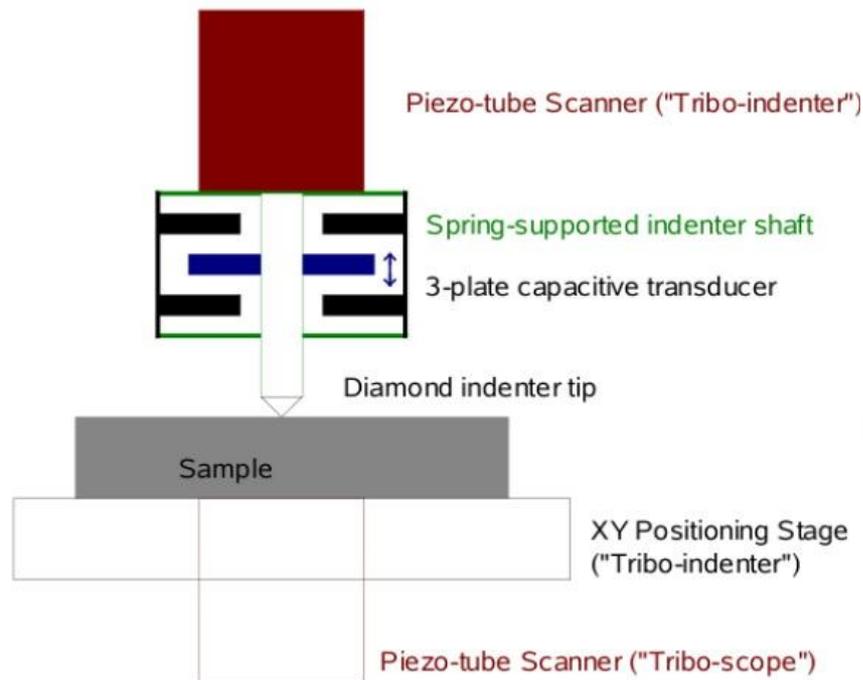
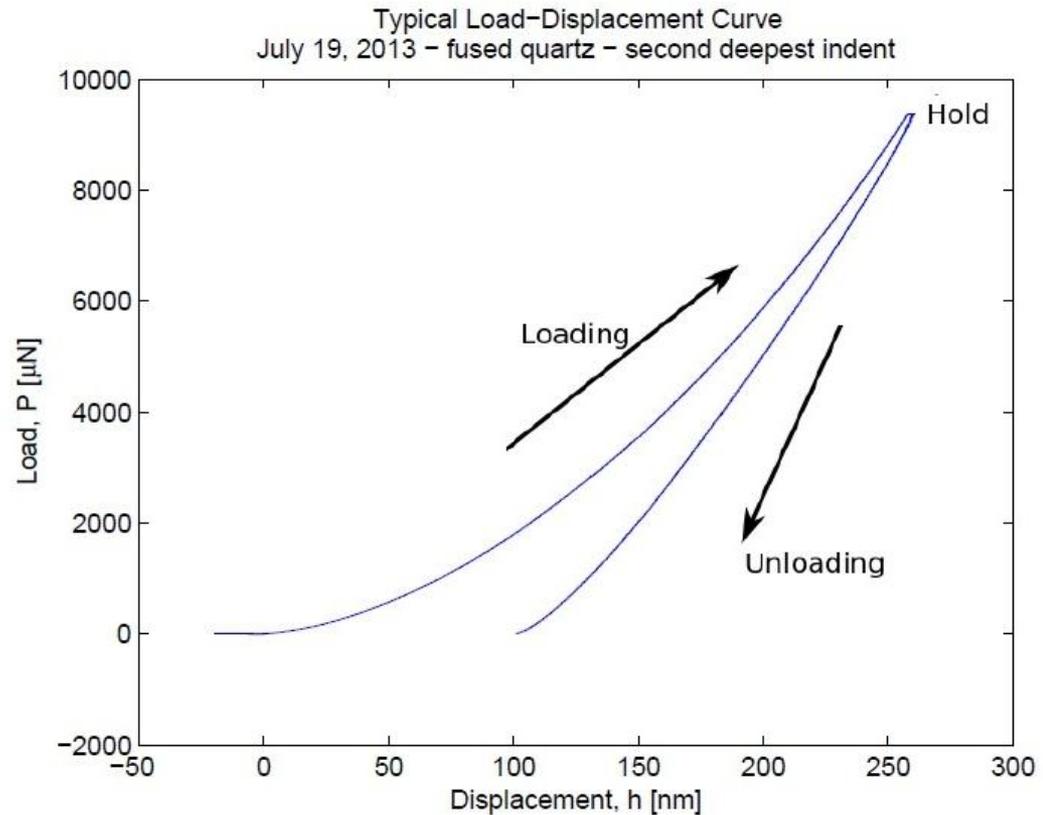


Photo credit: M. Oyen



# Nanoindentation

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- Two parameters of the nanoindentation system that affect sensitivity are variable: machine compliance and area function of the indenter tip
- Variation of 10% of the machine compliance causes a 9.2% variation of the Young's modulus
- Variation of 10% of the area function causes a 4.7% variation of the Young's modulus

# Calibration

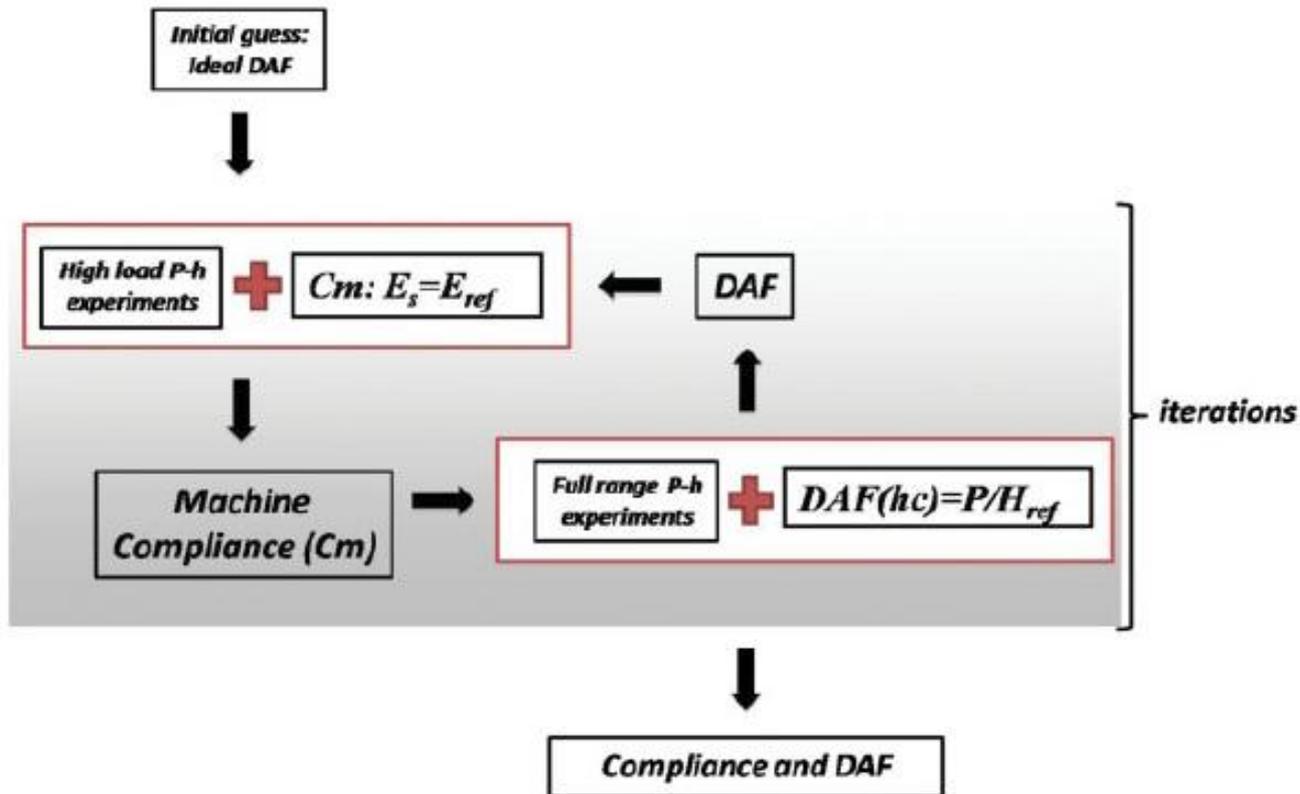


Photo credit: Barone et al., *Microsc. Res. Techniq.* 73:1001, 2010

# Calibration

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- Calibration is done on a material of known properties.
  - » Fused quartz is the standard calibration material.
- Calibration check: reverse analysis
  - » Run calibration analysis on a fused quartz data set, assuming the known value of the Young's modulus,  $E = 72 \text{ GPa}$
  - » Apply the calibration and run a Young's modulus analysis
  - » Should get the same number as the input value, with tolerance for rounding errors
- For each reverse analysis completed, results were consistent with the input value

# Future Work

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- Complete Young's modulus analysis for layered samples
  - » Analysis following the method of Song and Pharr
  - » Include Hay model to remove substrate effects
- Compare results from Young's modulus analysis of nanoindentation with fully analyzed results from Embry-Riddle acoustics group to uniquely determine Young's modulus and Poisson's ratio

# Acknowledgements

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- California Institute of Technology
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- Tamaryn Shean and the Oyen group at Cambridge University
- Lucas Meza and the Greer group at Caltech

# References

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- M.R. Abernathy. *Mechanical Properties of Coating Materials for Use in the Mirrors of Interferometric Gravitational Wave Detectors*. PhD thesis, University of Glasgow, 2012.
- A.C. Barone, M. Salerno, N. Patra, D. Gastaldi, E. Bertarelli, D. Carnelli, and P. Vena. Calibration Issues for Nanoindentation Experiments: Direct Atomic Force Microscopy Measurements and Indirect Methods. *Microsc. Res. Techniq.*, 73:996-1004, 2010.
- W.C. Oliver and G.M. Pharr. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *J. Mater. Res.* 7(6):1564-1583, 1992.
- W.C. Oliver and G.M. Pharr. Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology. *J. Mater. Res.* 19(1):3-20, 2004.



# Nanoindentation Equations

- Unloading data relation:

$$P = \alpha(h - h_f)^m$$

- Contact stiffness:

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A}$$

- Reduced Young's modulus:

$$\frac{1}{E_r} = \frac{(1 - \nu_s^2)}{E_s} + \frac{(1 - \nu_i^2)}{E_i}$$

- 6 term area function

$$A(h) = C_0 h^2 + C_1 h + C_2 h^{1/2} + C_3 h^{1/4} + C_4 h^{1/8} + C_5 h^{1/16}$$

# Nanoindentation Equations

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- Modeling load frame and specimen as two springs in series, total compliance of the system is

$$C = C_{\text{machine}} + C_{\text{specimen}}$$

- Since specimen compliance is the inverse of contact stiffness, the final equation is

$$C = C_{\text{machine}} + \frac{\sqrt{\pi}}{2E_r} \frac{1}{\sqrt{A}}$$

# Calibration Methods

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- Calculating machine compliance can be done:
  - » Graphically, by fitting the second equation on slide 15; the intercept is the machine compliance
  - » Analytically, by calculating both sides of the second equation on slide 14, and taking the average difference
- Applying the machine compliance can be done:
  - » To the load
  - » To the displacement

# Calibration Results

Date	Calibration method	Young's modulus
July 19	Graphical, load	$71.72 \pm 1.89$
July 19	Analytical, load	$73.69 \pm 1.79$
July 19	Graphical, displacement	$71.67 \pm 1.88$
July 19	Analytical, displacement	$73.47 \pm 1.72$
August 5	Graphical, load	$74.90 \pm 2.64$
August 5	Analytical, load	$75.54 \pm 2.53$
August 5	Graphical, displacement	$74.95 \pm 2.49$
August 5	Analytical, displacement	$75.35 \pm 2.47$

# Future Work - Comparison

