In-situ stress measurement of single and multilayer films for x-ray astronomy optical applications

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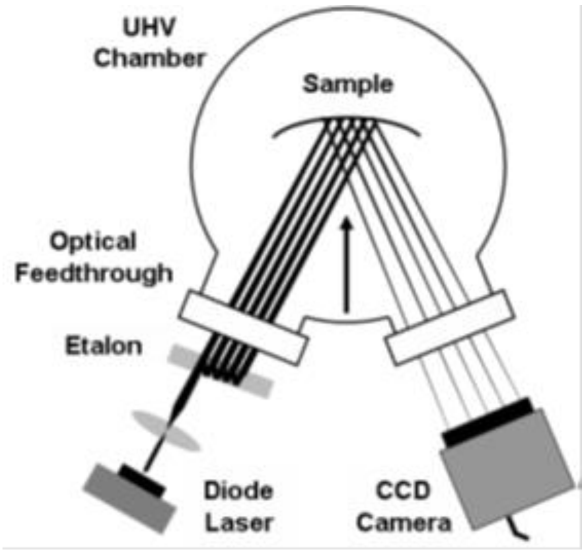
Talk Outline

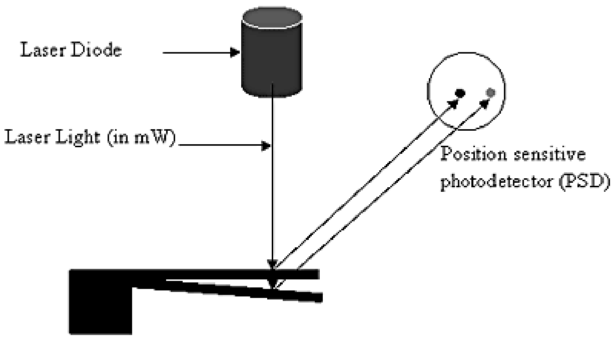
- Thin film stress measurement:
- Ex-situ example
- In-situ
  - Example: Stress behavior in polycrystalline materials during film growth
  - Current optical methods of in-situ measurement
    - Limitations, sensitivity
  - New method of in-situ stress measurement using fiber optic displacement sensor
    - Two embodiments: circular, cantilever-substrate
    - Sensitivity
    - Repeatability performance
    - Device validation
  - Effect of material interfaces on film stress: Ir/B4C, Ir/Si, Mo/Si, Mo/B4C,…
    - Multilayers to compensate stress in x-ray optical coatings?
      - W/Si example

Current methods of optical in-situ thin film stress measurement:

Multi-beam stress sensor (MOSS):



Micro cantilever:



These methods determine the substrate curvature by various optical means from which the integrated stress is calculated from the Stoney Eqn.:

$$\sigma h_f = \frac{E_s h_s^2}{6(1-\nu_s)}\kappa$$

Minimum detectable stress Δσhf:

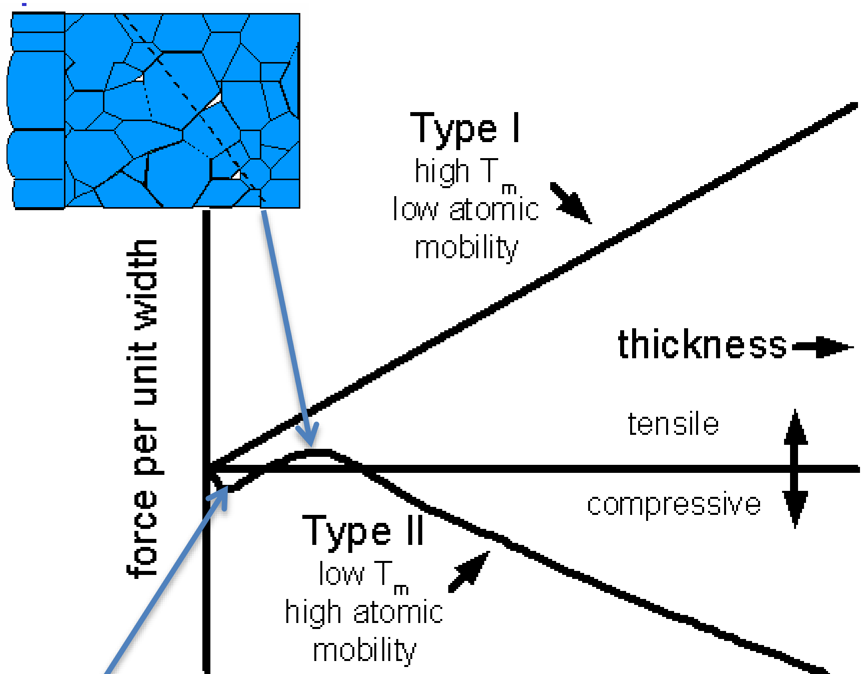
- Ranges from 0.005-0.050 N/m depending on method and substrate (i.e. geometry and mechanical properties)
- MOSS is 50 MPa*nm for 100 µm thick silicon substrate

Draw backs with current optical methods:

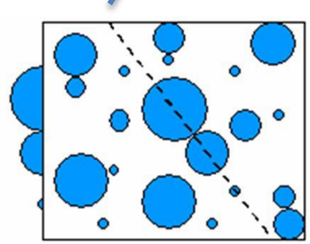
- Requires external optical access to the substrate through angled viewports
- Limited to specific deposition geometries
- Complex
- Requires the use of opaque substrates such as crystalline silicon.
- Film side is measured which can result in destructive interference effects when measuring transparent films.

Stress evolution in polycrystalline films

Island coalescence

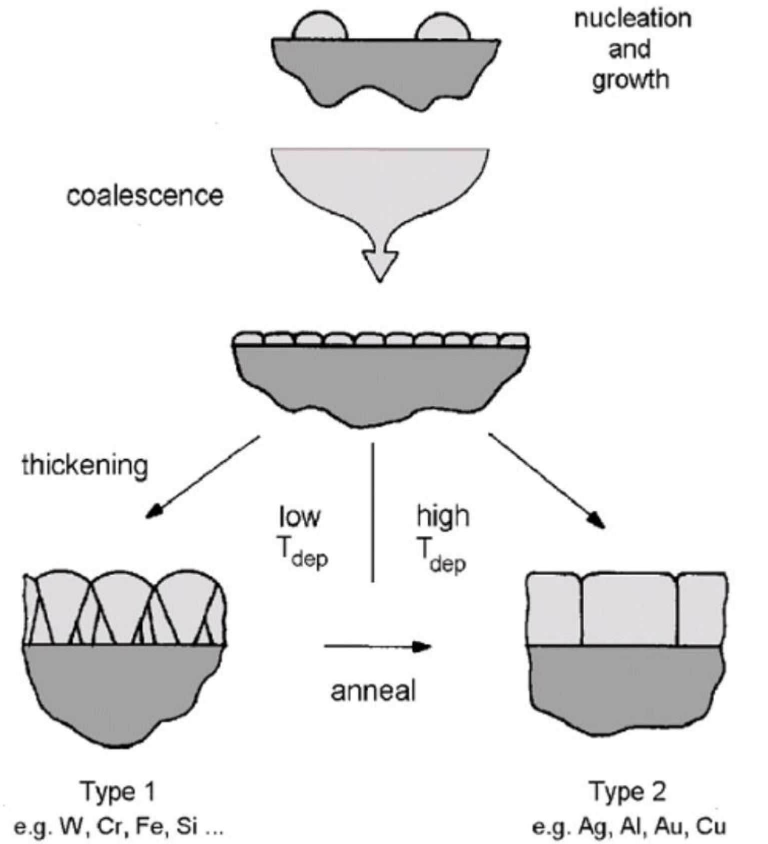


Volmer-Weber Growth Mode



Nucleation & island growth

<u>Depends on:</u>
Substrate temperature
Argon pressure
Mass of sputtered atoms
Substrate bias
Surface energy



Surface roughness increases with film thickness

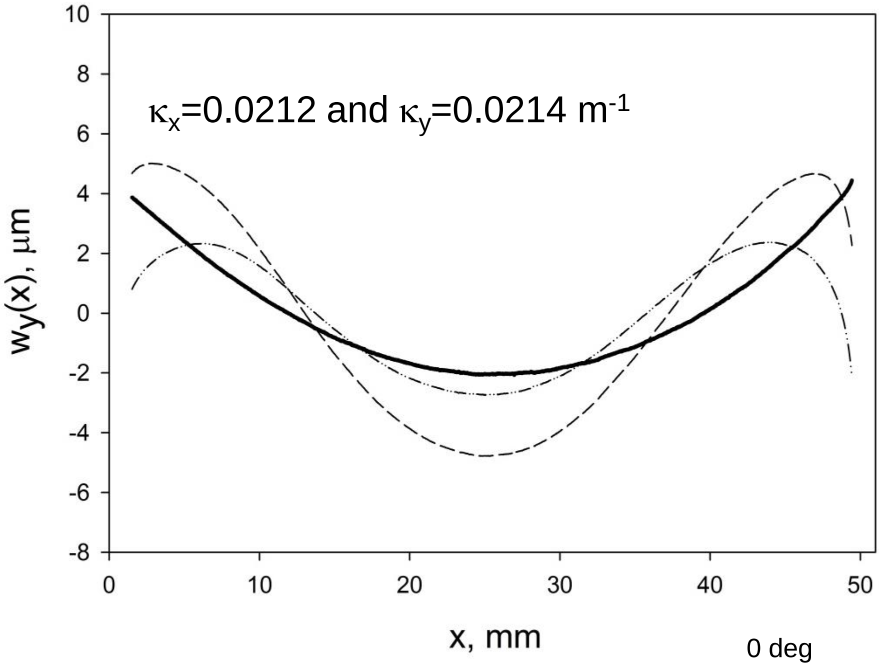
Low surface roughness

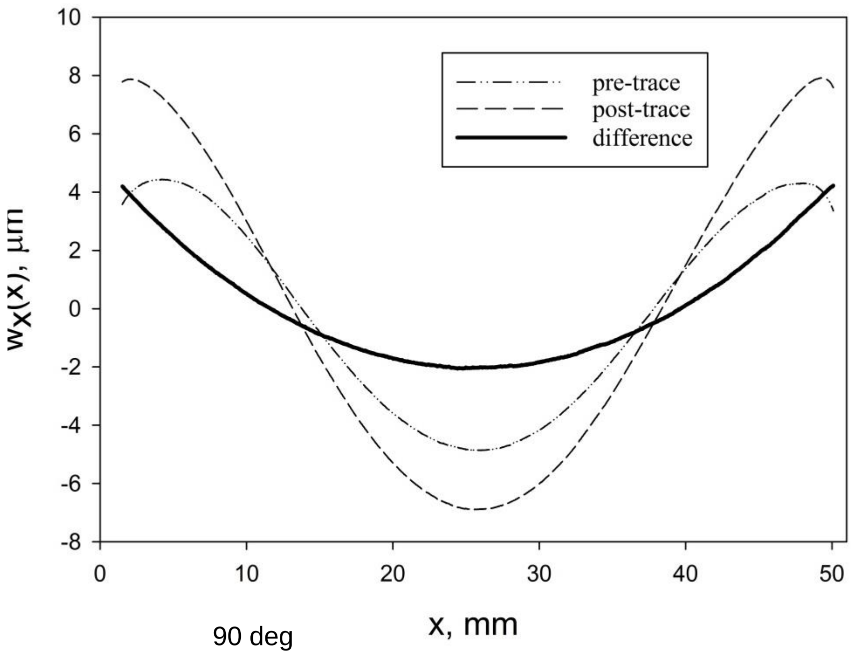
Ex-situ measurement of thin film stress

$$Stoney's\ Eqn:\ \sigma h_f = \frac{E_s h_s^2}{6(1-\vartheta s)}\kappa$$

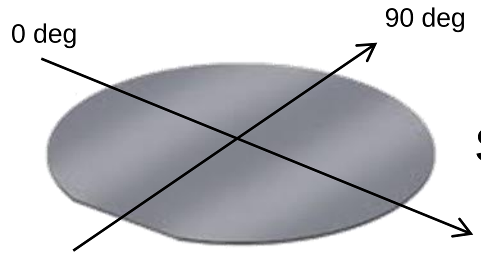
$$Spherical\ Deformation\ Mode:$$
$$A = \sigma h_f \frac{D_s^2}{h_s^3}$$

Tallysurf stylus profilometer





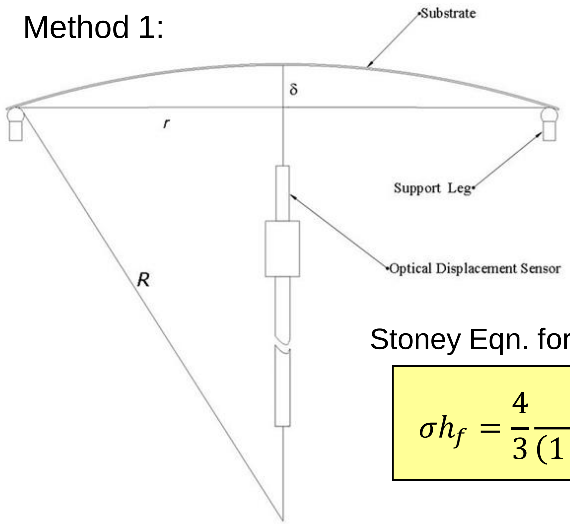
$$\kappa \approx \frac{d^2 w}{d^2 x} = const$$



Si <111>

New approach to in-situ stress measurement

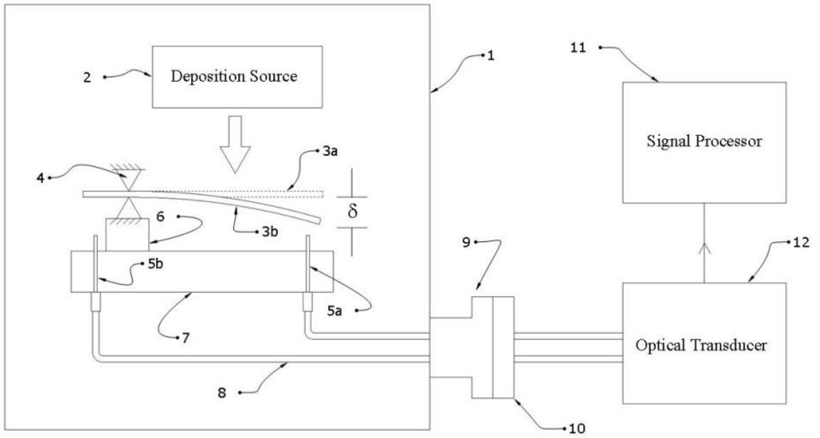
Method 1:



Stoney Eqn. for circular substrate:

$$\sigma h_f = \frac{4}{3}\frac{E_s}{(1-\nu_s)}\left(\frac{h_s}{D_s}\right)^2 \delta$$

Method 2:



Stoney Eqn. for cantilever:

$$\sigma h_f = \frac{E_s h_s^2 \delta}{3(1-\nu_s)L^2}$$

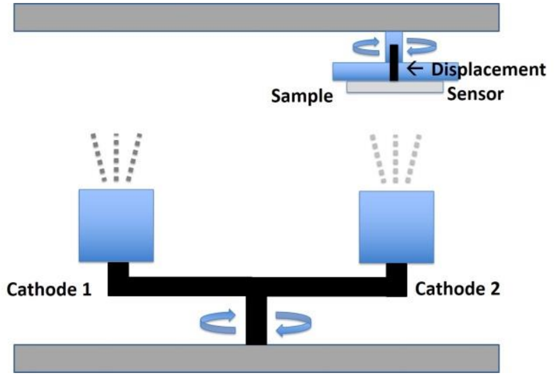
<u>New approach to in-situ stress measurement:</u>

- Utilizes a high resolution (i.e. 5nm) vacuum compatible fiber optic displacement sensor.
- Curvature determined from out-of-plane displacement measurement of the substrate.
- Uses double-side polished substrate.
- Same arrangement can be used for thermal annealing.
- Glass substrates can be utilized.
- Easily implemented into existing deposition systems.
- Very sensitive method.

<u>Ongoing work:</u>

- Adapting to rotating substrates
- Adapting to curved (i.e. segmented) substrates

D.M. Broadway, U.S. Patent 9,601,391 (Granted March 2017).
D.M. Broadway, U.S. Patent Application 15/425,740 (Filed February 2017).
Pending publication in Review of Scientific Instruments



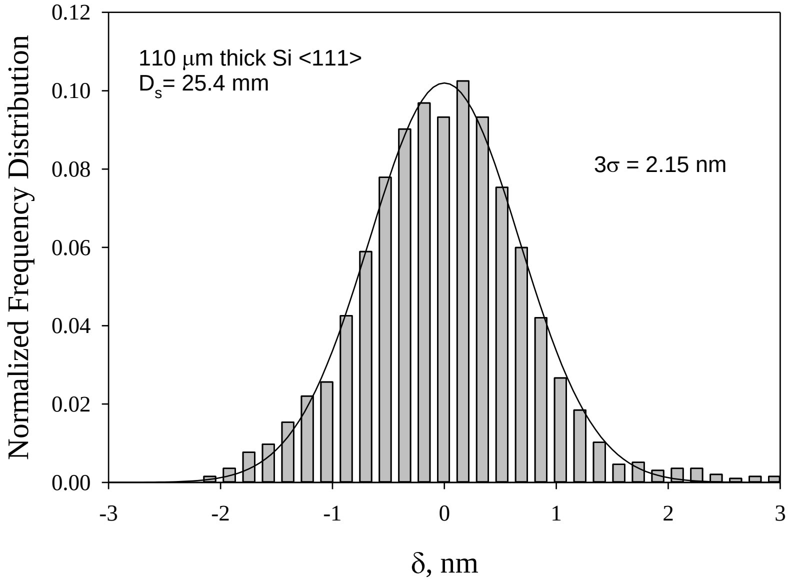
Minimum detectable integrated stress, $\Delta(\sigma h_f)$

- The minimum detectable stress is limited by the combined ambient vibrational background of the substrate and electronic noise of the displacement sensor.
- The sensitivity further depends on the mechanical and geometric properties of the substrate.
- The cantilever approach is more sensitive to a given integrated stress but is also more sensitive to vibrational noise—compensating effect.
- The cantilever approach is advantageous because it is flexible in its orientation and easily adapted to various deposition geometries.

Sensitive enough to measure stress in x-ray multilayers

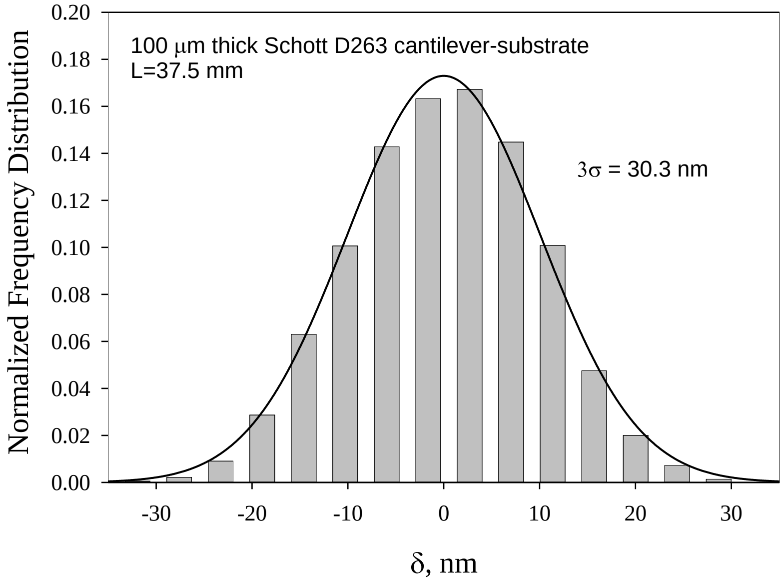
$$\Delta(\sigma h_f) = \frac{4}{3}\frac{E_s}{(1-\nu_s)}\left(\frac{h_s}{D_s}\right)^2 \Delta\delta$$

15 MPa*nm

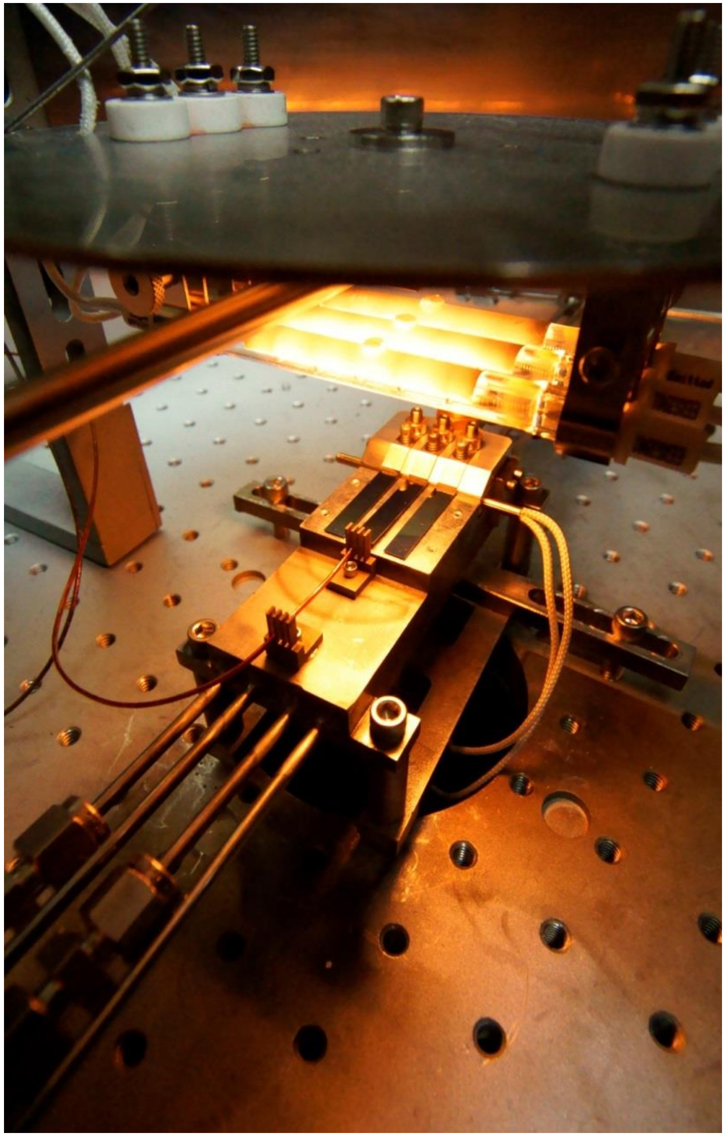


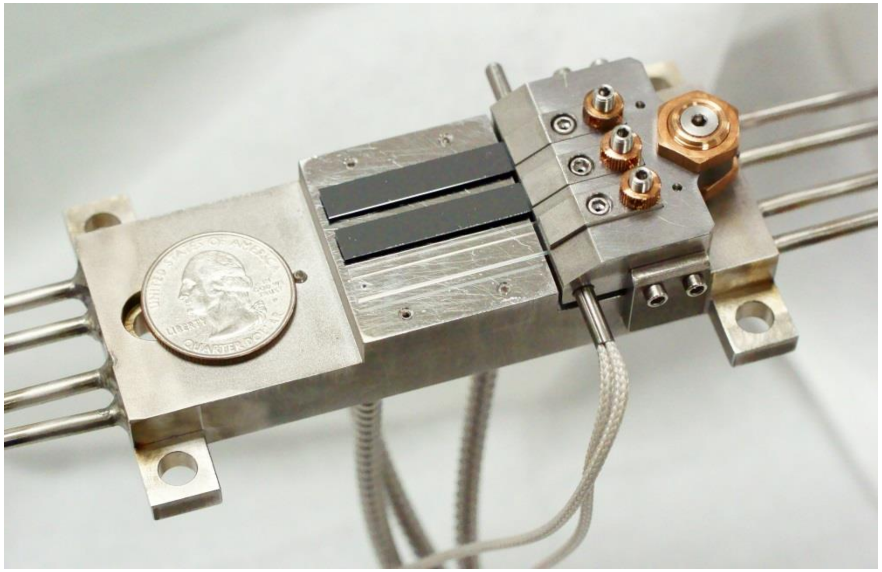
$$\Delta(\sigma h_f) = \frac{E_s h_s^2 \Delta\delta}{3(1-\nu_s)L^2}$$

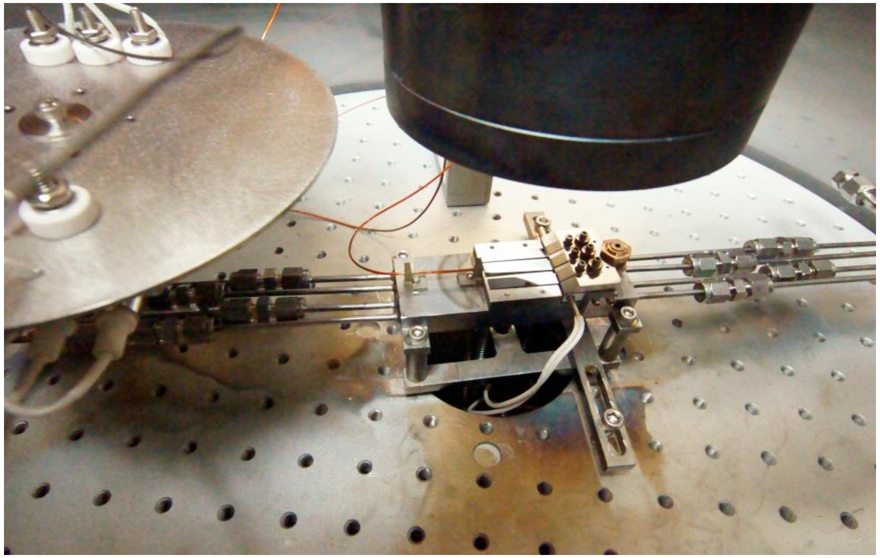
9 MPa*nm



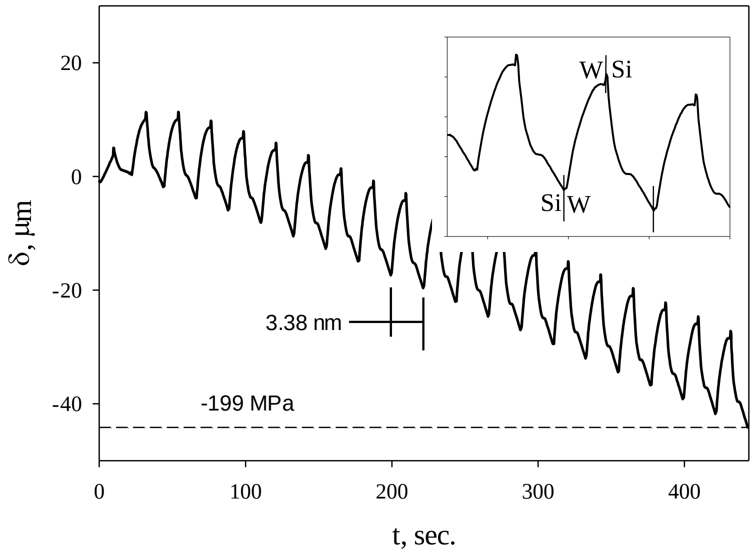
Refined in-situ stress sensor (Testing Underway)

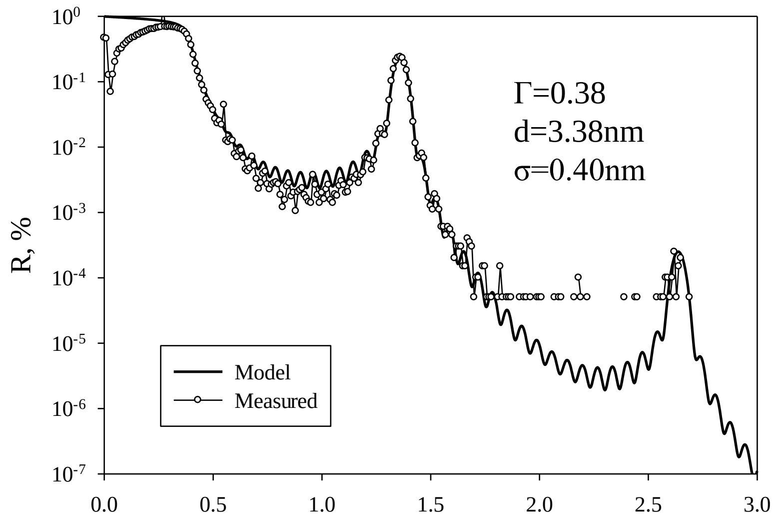






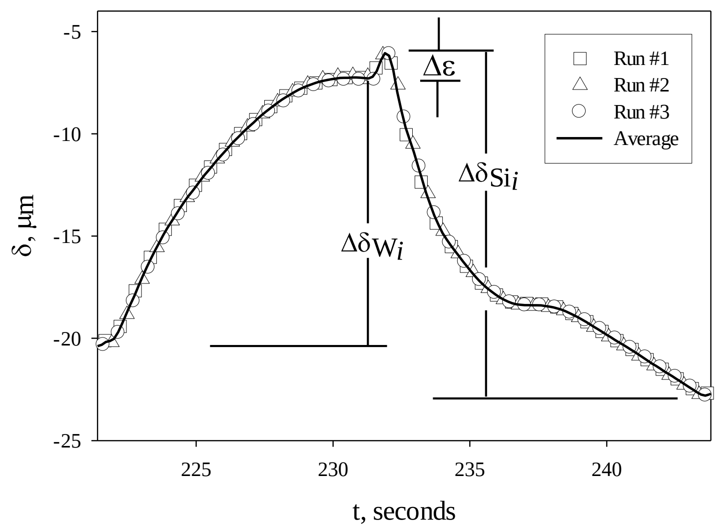
Device performance

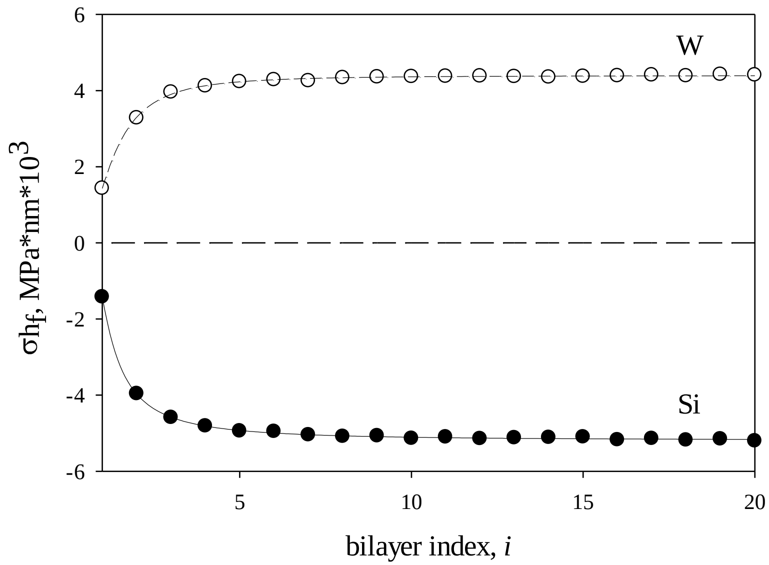




±0.5% within run repeatability

±2.5 % run-to-run repeatability



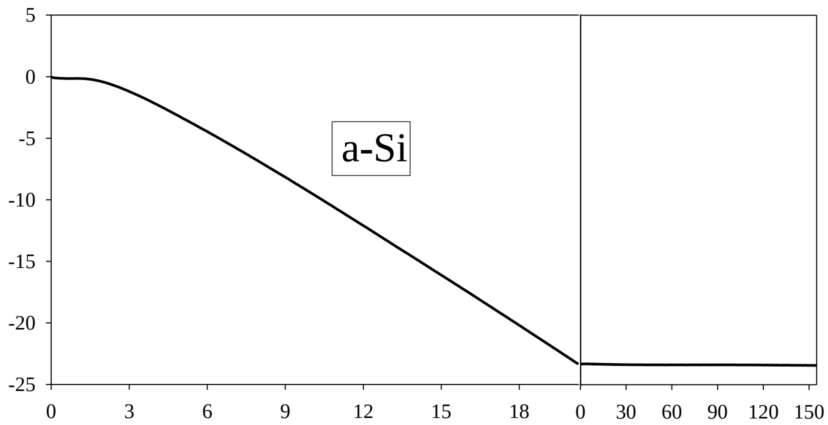


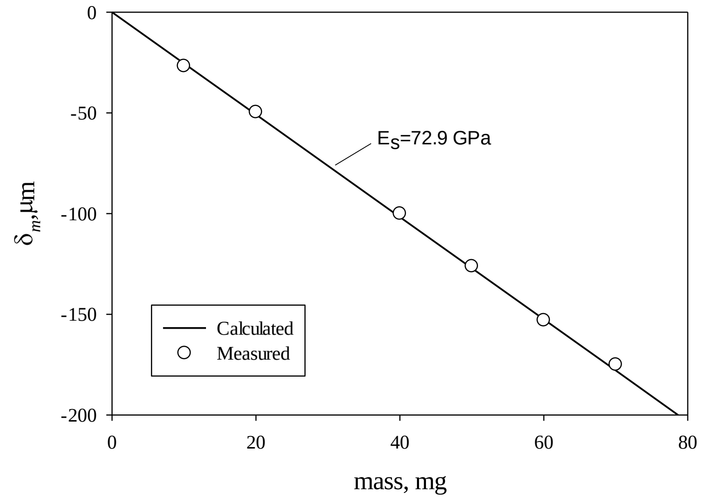
In-situ stress of single layer thin films

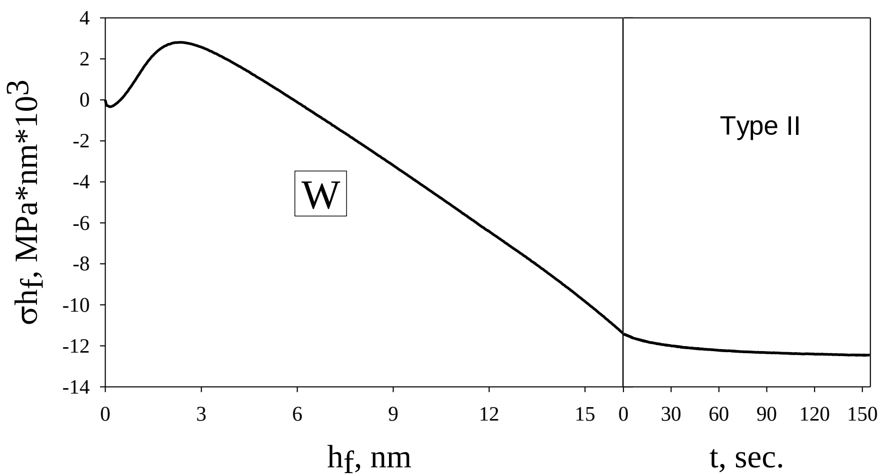
T_s~27-30^{o}C, 2.5 mTorr Ar
100µm thick Schott D263

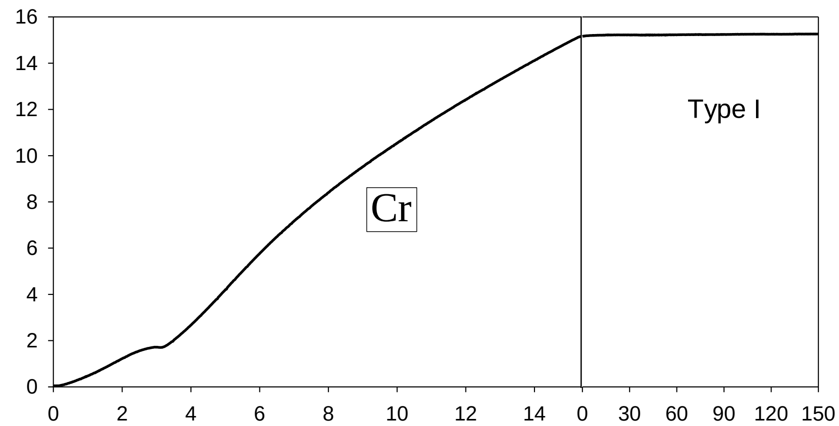
Calibration masses placed on cantilever tip used to validate substrate modulus and linear range of the sensor:

$$\delta_m = \frac{2mgx^2}{E_s b h_s^3}(3L - x)$$



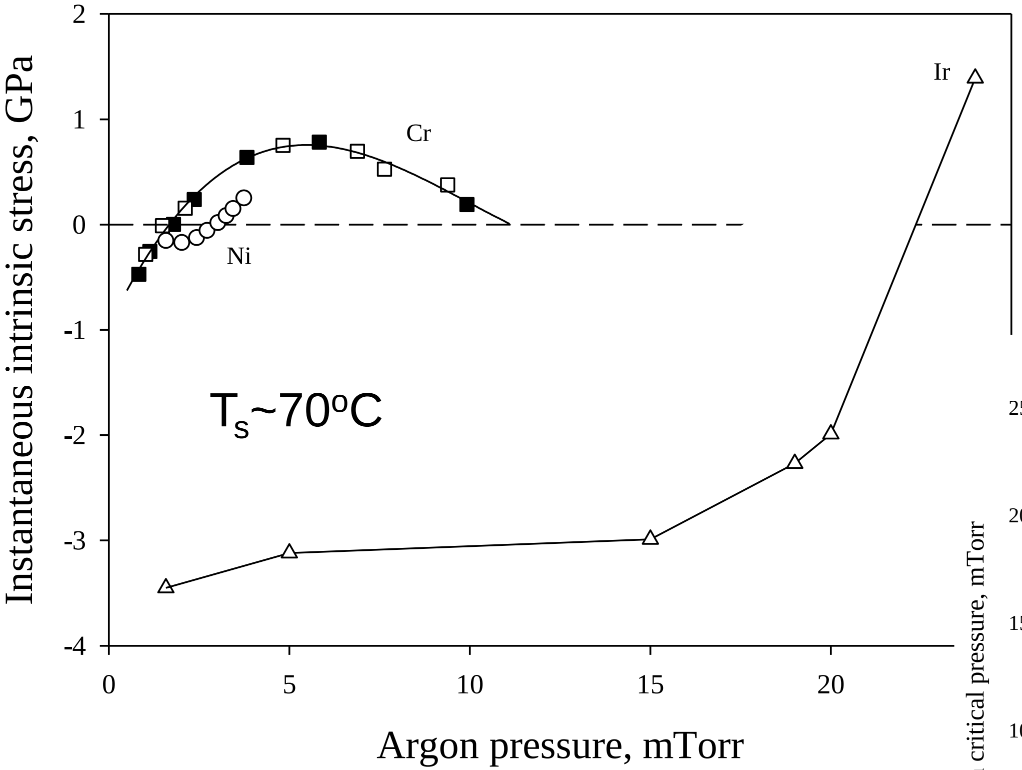






Stress reversal in polycrystalline films (circular substrate)

Results are consistent with D.W. Hoffman, Internal stress of sputtered Chromium, Thin Solid Films, 40 (1977) 355-363

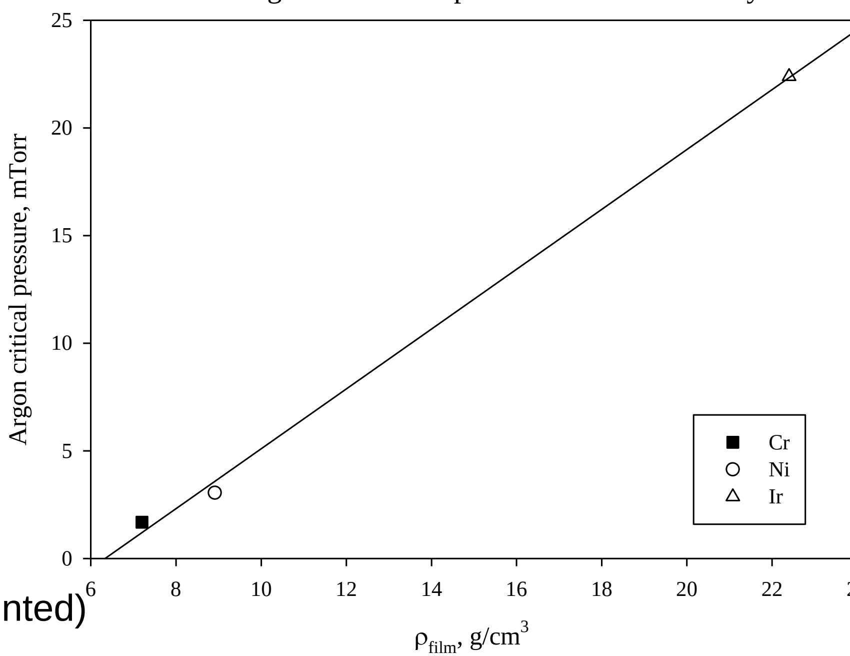


Instantaneous stress:

$$\frac{d(\sigma h_f)}{dh_f} = -\frac{E_s}{3\xi(1-\vartheta s)}\left(\frac{h_s}{r}\right)^2 \frac{d\delta}{dt}$$

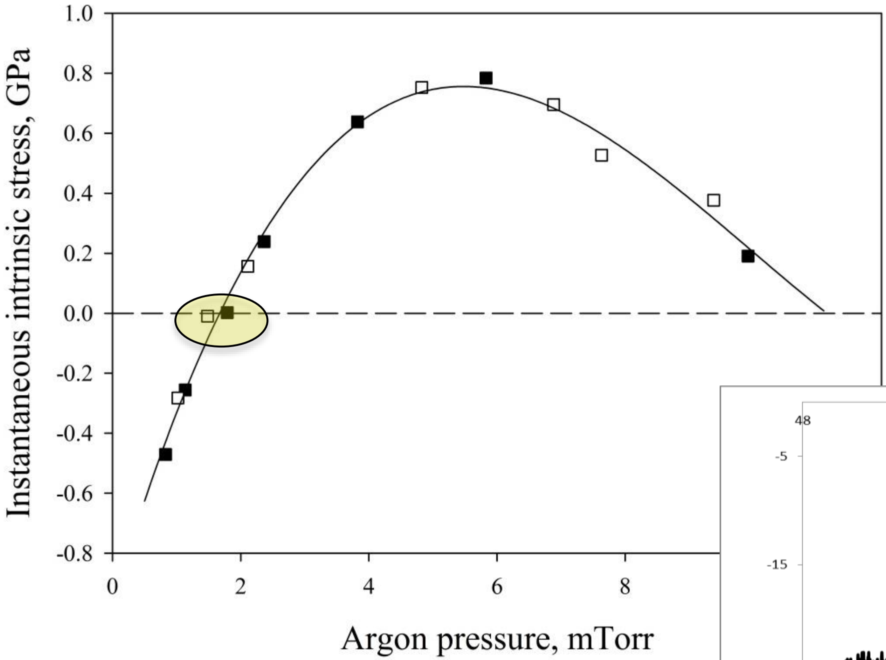
- Efficient for parametrizing the stress
- Independent of film thickness in the steady-state regime of film growth
- Substrate in thermal equilibrium

Scaling of the critical pressure with film density



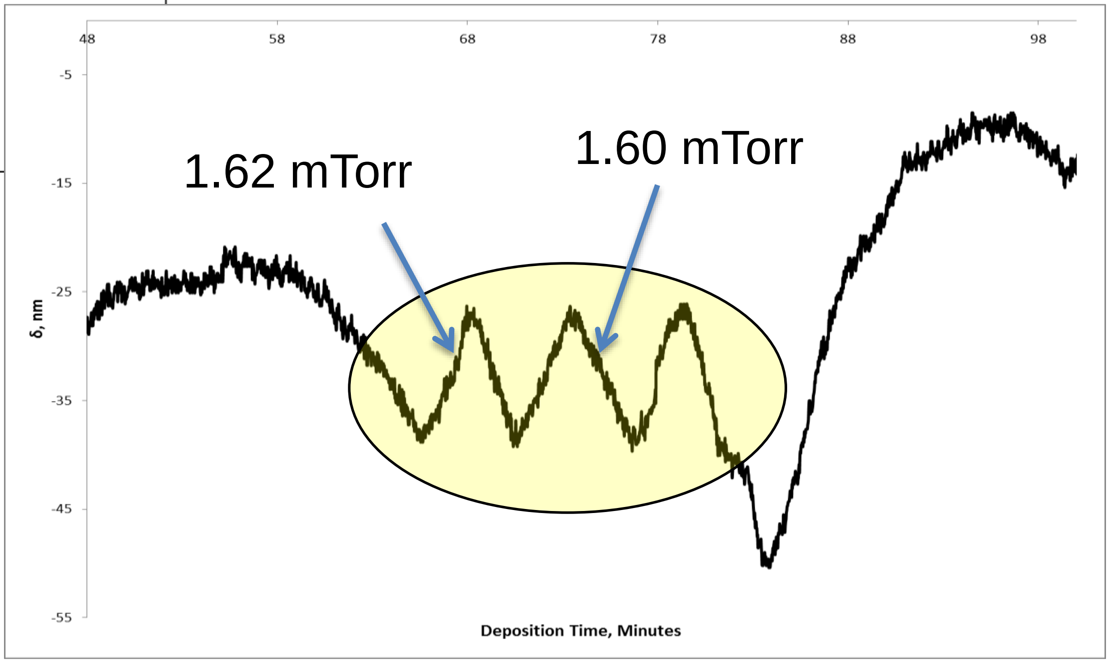
- State of stress (i.e. tensile or compressive) at low pressure is strongly influenced by substrate temperature for low density metals like chromium.
- Therefore, the state of stress will depend on the heat transfer mechanisms of the substrate (i.e. how it is mounted) for a given deposition system.

Sensitivity at the transition pressure (circular substrate)

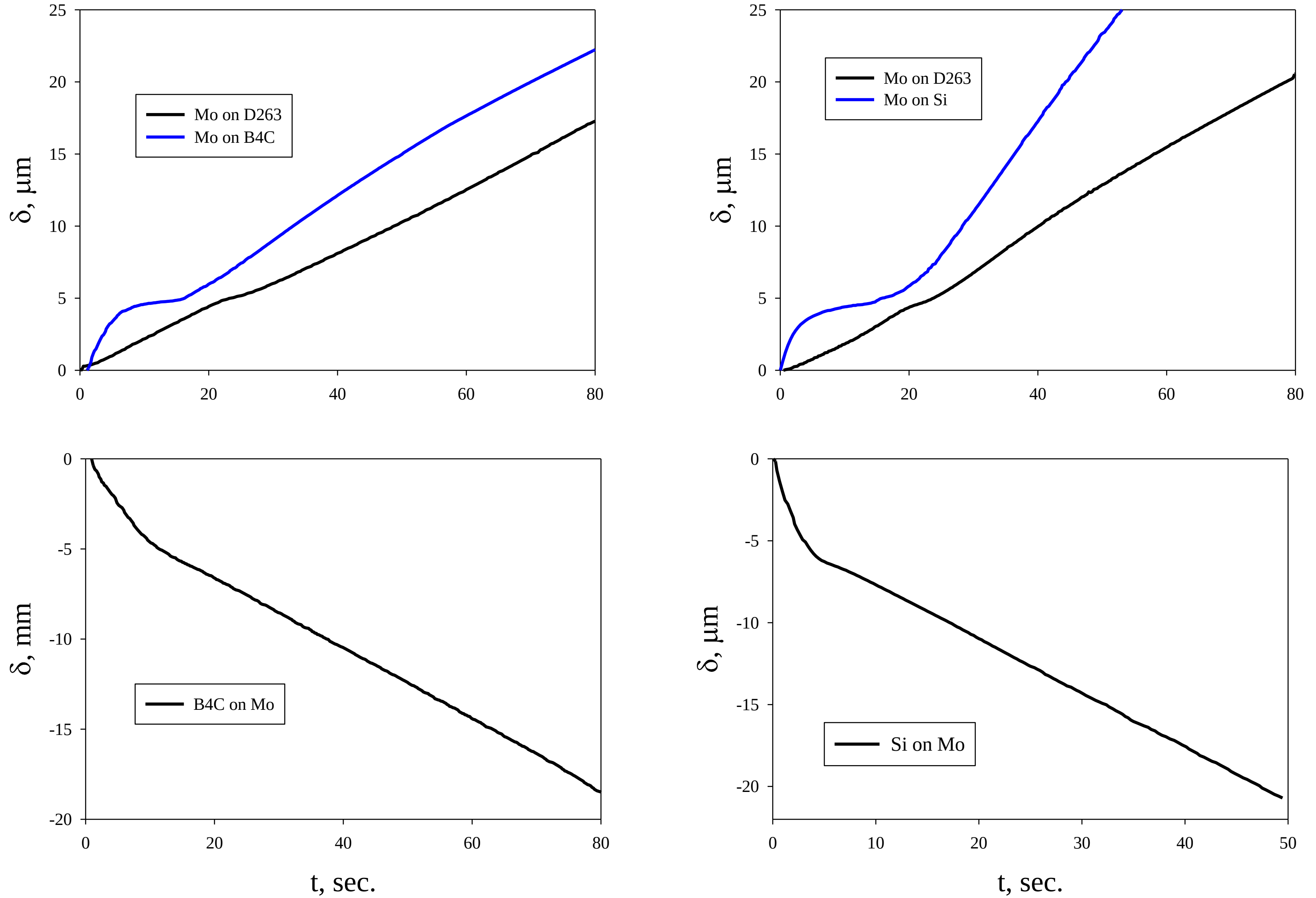


Stress reversal in Cr with argon pressure has been measured with the instrument. Consistent with the previous work of Hoffman (i.e. stress reversal).

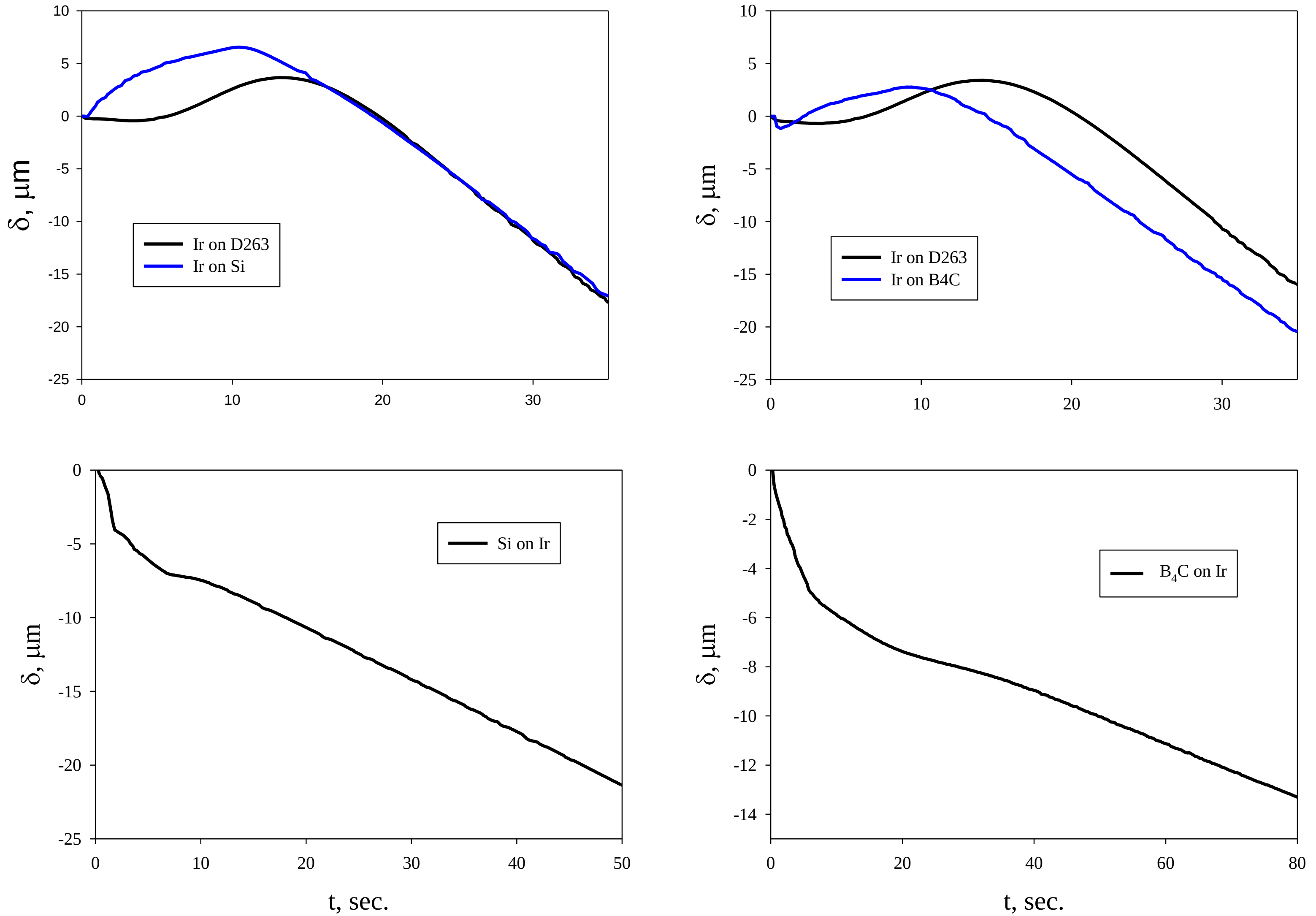
Measurement sensitivity is better than resolution in the control of Argon pressure.



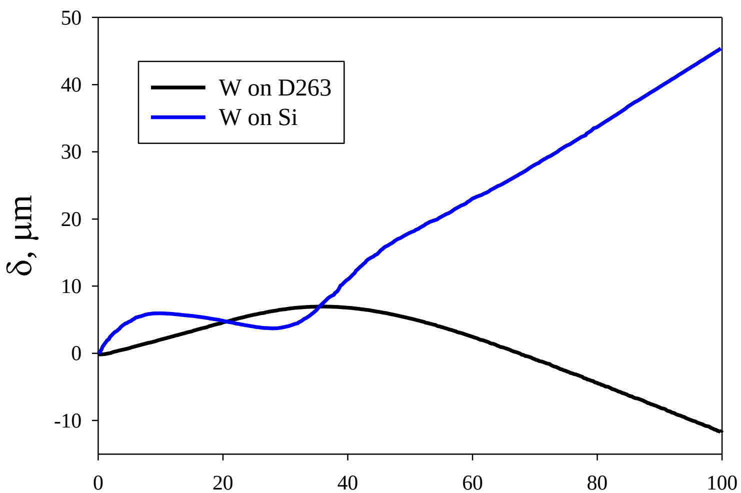
Effect of material interfaces on the film stress (Mo-based)

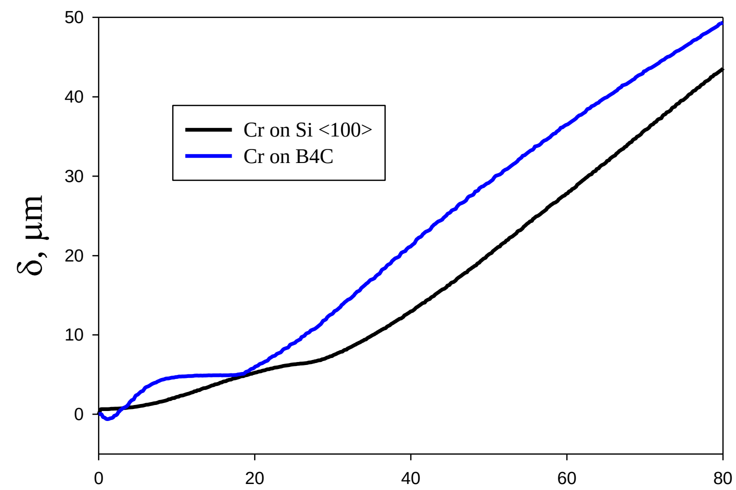


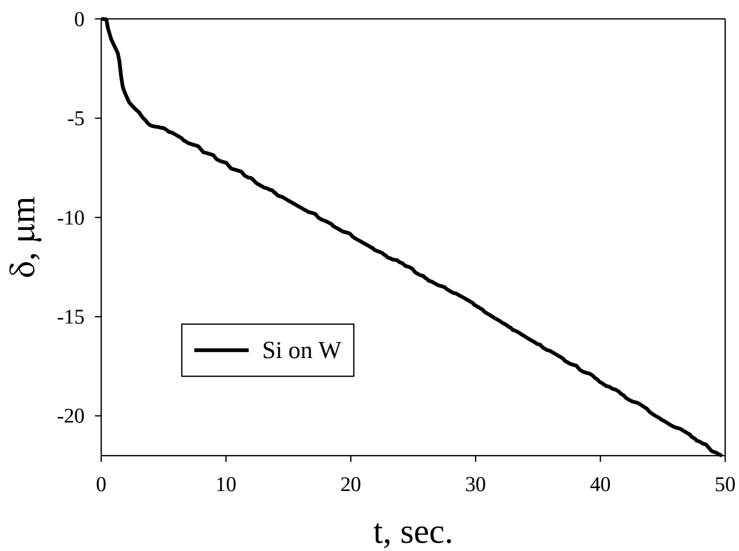
Effect of material interfaces on the film stress (Ir-based)

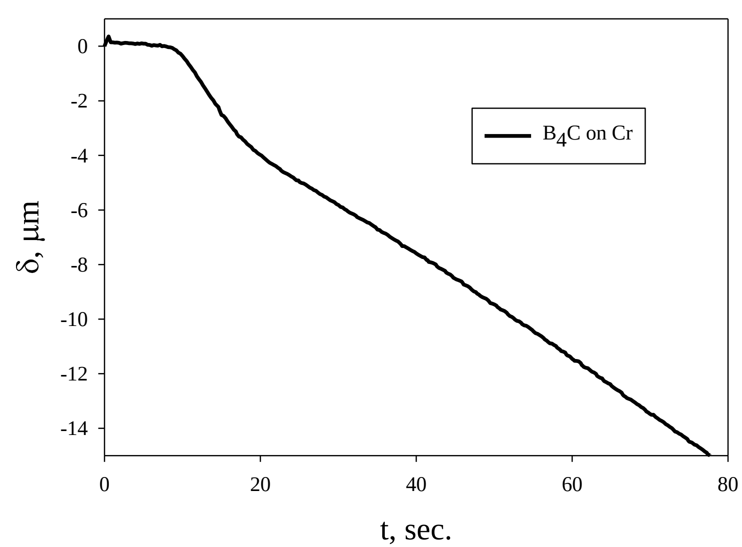


Effect of material interfaces on the film stress (W, Cr-based)



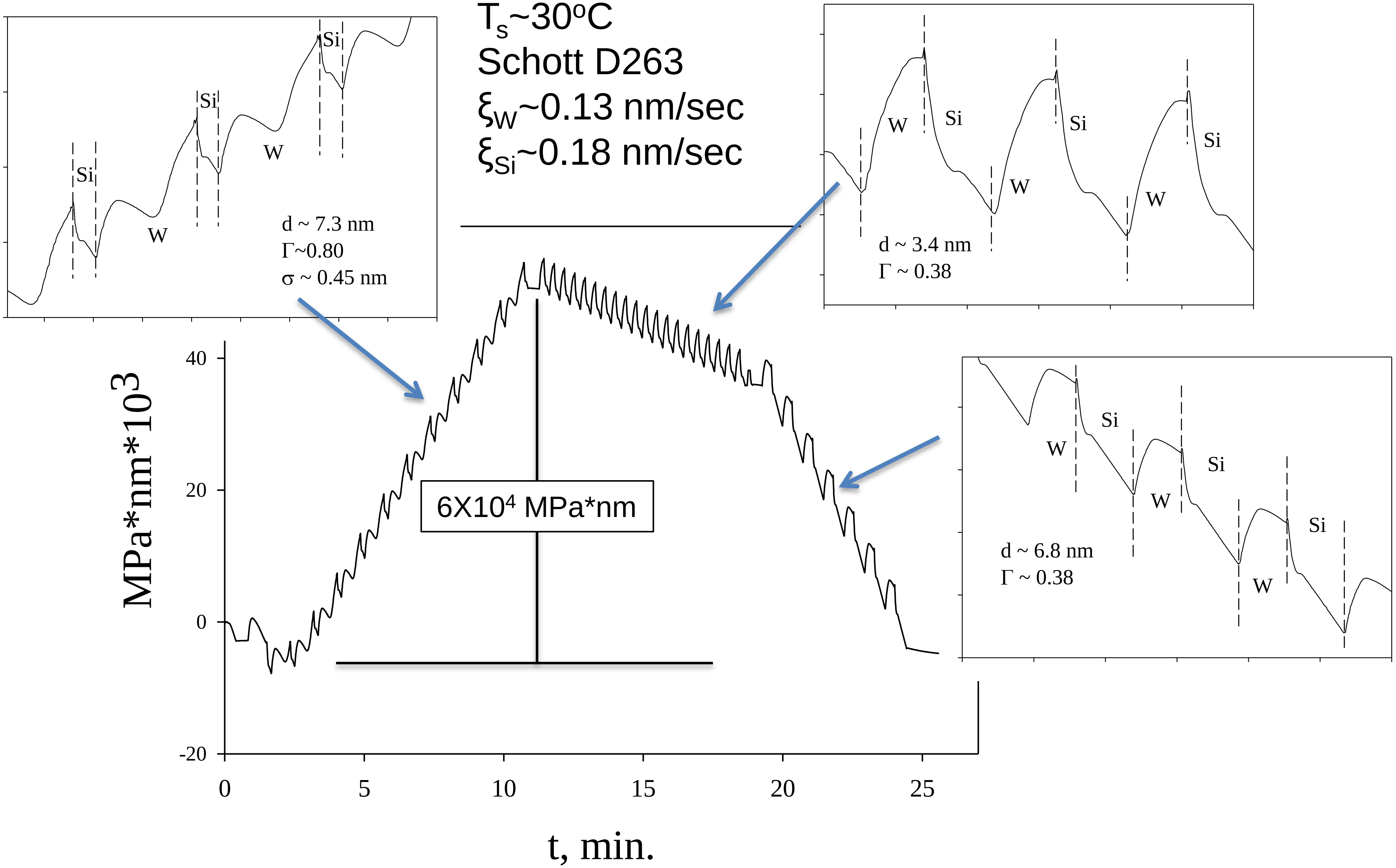






In-situ stress in W/Si multilayers

T_s~30°C
Schott D263
ξ_W~0.13 nm/sec
ξ_{Si}~0.18 nm/sec



Multilayers to compensate integrated stress

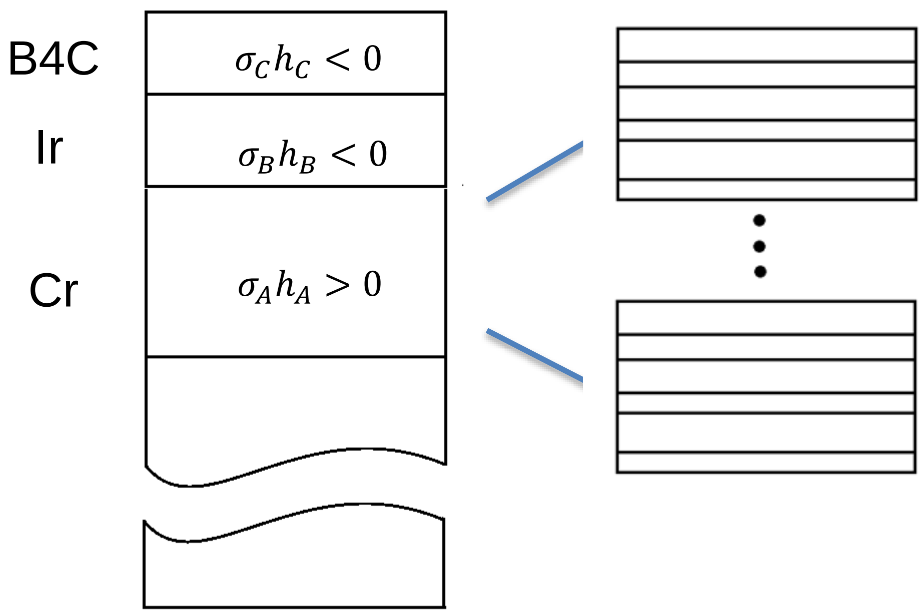
- Currently single layer films (i.e. Cr) with tensile stress are used as one technique to compensate the integrated stress in x-ray optical coatings to near-zero:

$$\left(\sigma h_f\right)_{Net} = \sigma_A h_A + \sigma_B h_B + (\sigma h)_{CTE} \approx 0$$

- The columnar microstructure of metal films in tension results in increasing surface roughness as the film thickness increases—thereby limiting the method's applicability.
- The increased surface roughness can severely degrade the optical coating's performance; particularly for high energy broadband multilayers.
- Multilayers interrupt the columnar growth so roughness doesn't increase with film thickness (for Glass & Si)

Example:

Stress compensating ML: Cr/B4C, Mo/B4C, …



Future work:

- Influence of substrate temperature and deposition rate.
- Impact to total deposition time.
- Optimization of layer thicknesses.
- Surface roughness characterization.
- Addition of N to B4C based ML's to increase dep. rate and smooth interfaces.

Conclusions

- We have introduced a novel method for the in-situ measurement of film stress using a fiber optic displacement sensor.
- The device is less complex than other current optical methods and easily implemented into an existing deposition system.
- The device's sensitivity is 0.009 N/m (9 MPa*nm) for a 100 μm thick glass substrate.
- This sensitivity is capable of detecting changes in stress due to small changes in deposition parameters such as argon process pressure (i.e. ±0.02 mTorr).
- The sensitivity can easily detect changes in the integrated stress in the individual layers of multilayer films of sub-nanometer thickness.
- The in-situ stress measured with the device is in good qualitative agreement with the known behavior of metals films (i.e. stress reversal, Volmer-Weber growth).
- We presented the influence of the material interfaces on the evolution of the film stress for several material pairs including: Mo/Si, Mo/B4C, W/Si, Ir/B4C,…
- We have proposed a new stress compensating method that utilizes multilayers
  - This method might be applicable to balance the stress in broadband multilayer that are more than a micron in total thickness.
  - More investigation is needed to study the impact to the total deposition time through optimization of the layer thicknesses