Status of x-ray multilayer development at MSFC

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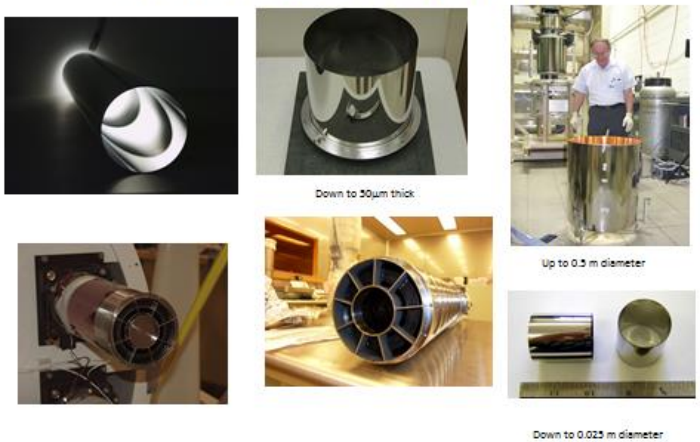
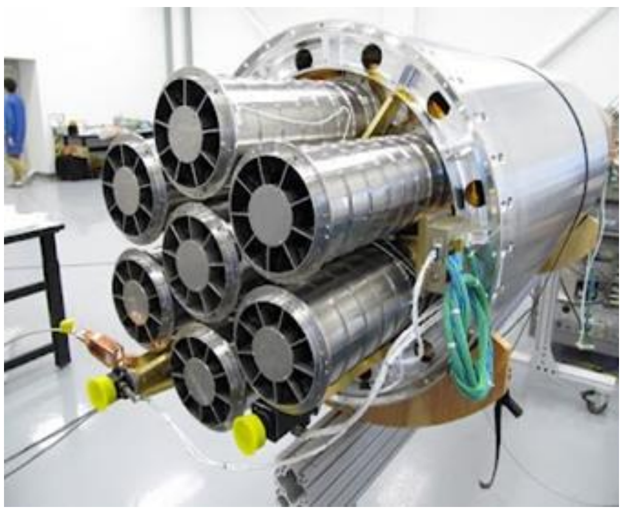
Presentation outline

- Technological challenges associated with stress in X-ray optical coatings (single and multilayer thin films)
- MSFC method of in-situ stress measurement (prototype)
- Instrument sensitivity
- A method for reducing the stress in iridium thin-films
- Refinements to the prototype and expanded capability

Motivation for in-situ stress measurement capability at MSFC

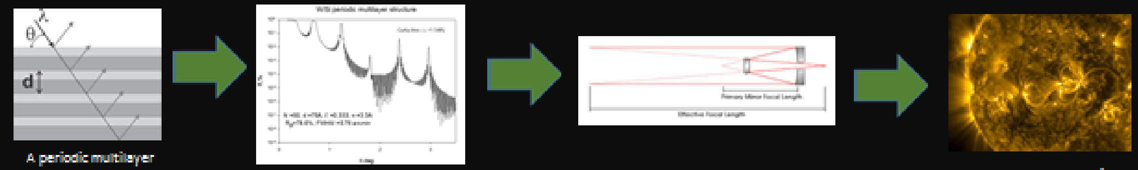
Electroformed X-ray Optics





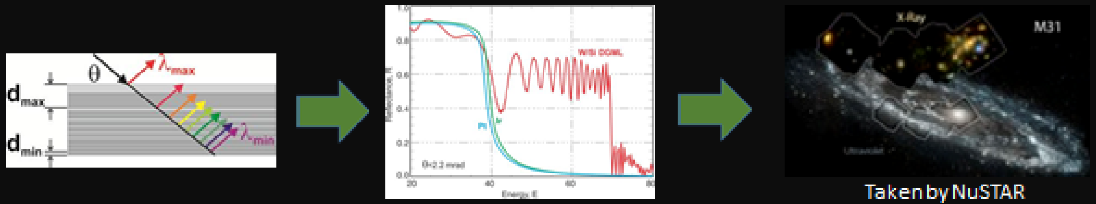
Multilayer thin-film reflective coatings

- Needed to efficiently reflect light at the high-energy region of the spectrum, from EUV to hard x-rays.
- Periodic multilayers are used as selective optical elements due their inherently narrow spectral response.
- At EUV energies the can be designed to reflect at normal incidence.
- Enabled the fabrication of Cassegrain-type EUV reflecting telescopes.



Taken by SDO/AIA

- A depth-graded multilayers is a film stack containing a range of layer thicknesses
- They are designed to give a spectral response at grazing incidence this is several times broader than the total external reflection regime of a single layer films.



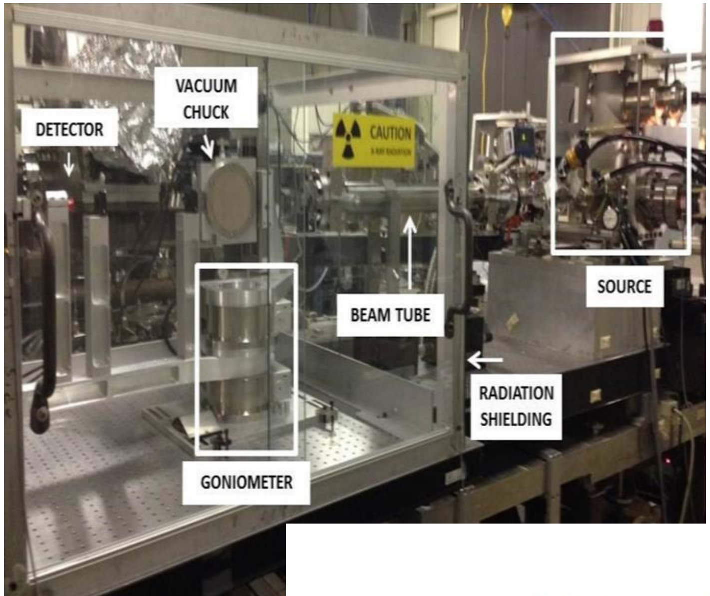
Taken by NuSTAR

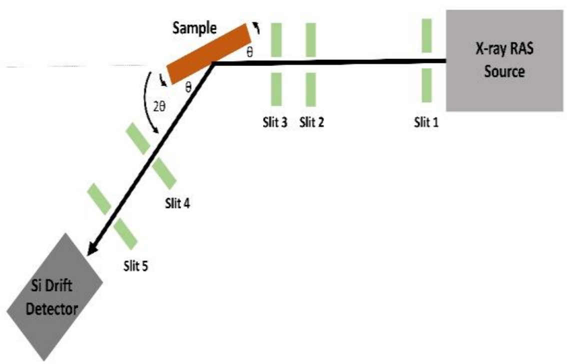
Tightly nested thin shells needed to achieve large light collecting areas.

Even modest stress in the x-ray optical coatings easily deforms the precise figure of the thin shells and severely degrades imaging resolution.

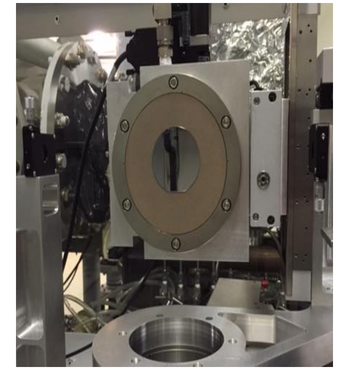
Stress must be controlled to “near-zero” values while also achieving low surface roughness.

Multilayer Coatings for X-ray Optics - MSFC

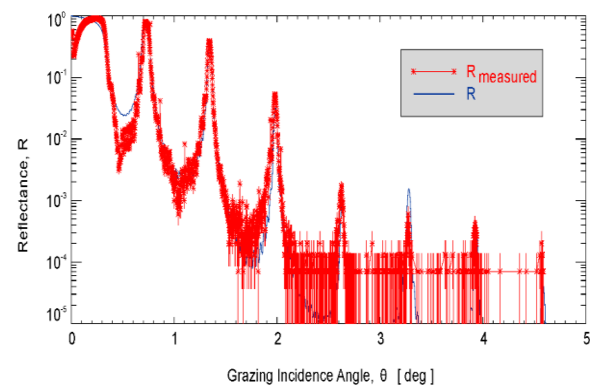




X-ray Reflectometer

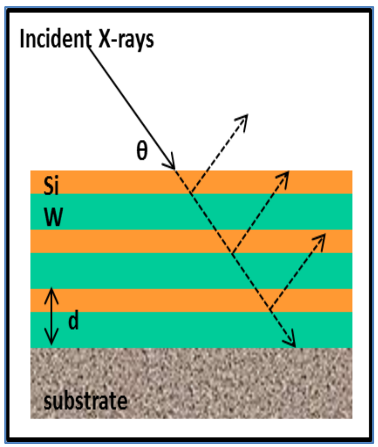


W/Si multilayer undergoing test



Reflectivity Curve

Multilayer Coatings for X-ray Optics - MSFC



How multilayers work

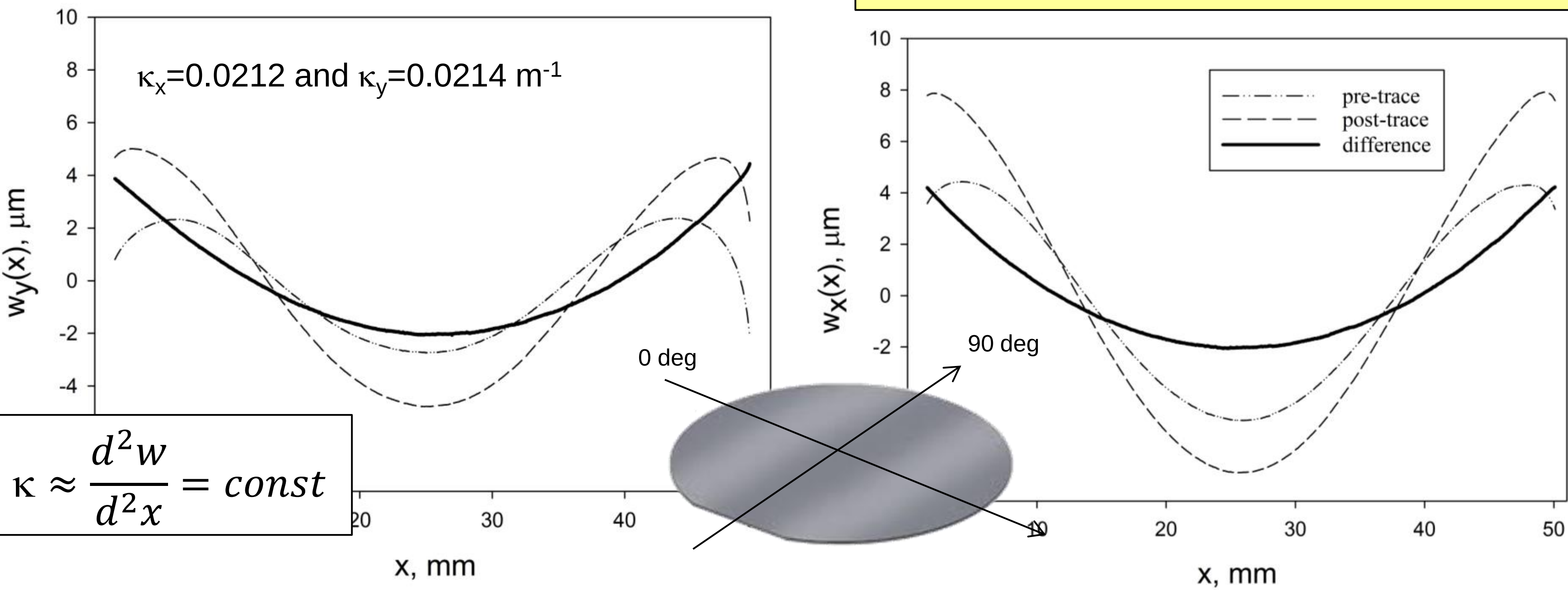
Multilayer DC Magnetron Desposition System

Measurement of thin film stress (ex-situ method)

- The change in substrate curvature, κ, is measured before and after the thin film process--usually by profilometry or laser deflectometry.
- The curvature,κ, of the deformed substrate is proportional to the product of film stress and film thickness according to the Stoney equation.
- Since substrates are seldom flat, the pre and post traces must be subtracted in order to reveal the parabolic displacement corresponding to a constant value of curvature (i.e. spherical deformation).
- For small out-of-plane displacements, the curvature is approx. the second derivative of this displacement.
- The substrate will deform spherically provided the deformation mode criterion is satisfied.

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

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



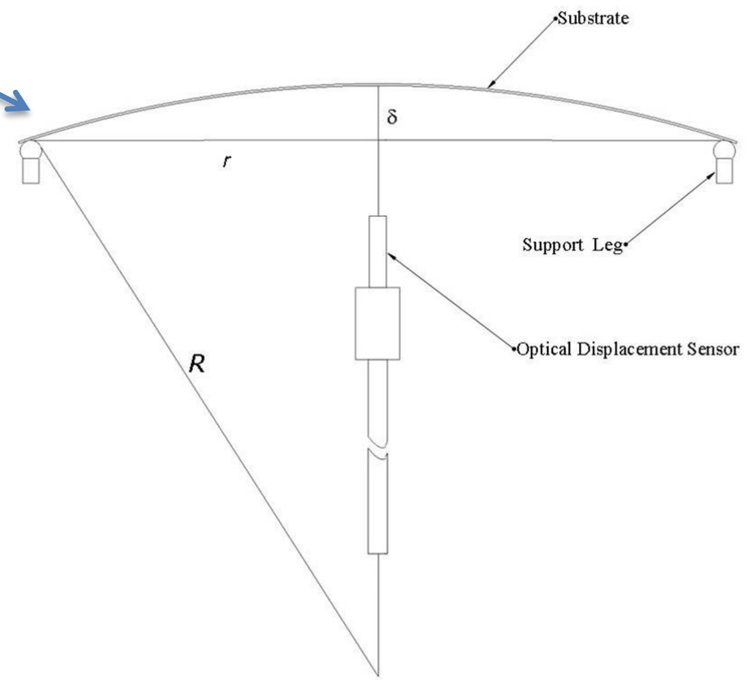
$$\kappa \approx \frac{d^2 w}{d^2 x} = const$$

In-Situ methodology:

Since substrate deformation is spherical we need only measure the sagitta, δ, to infer its curvature from which the Stoney equation can be employed:

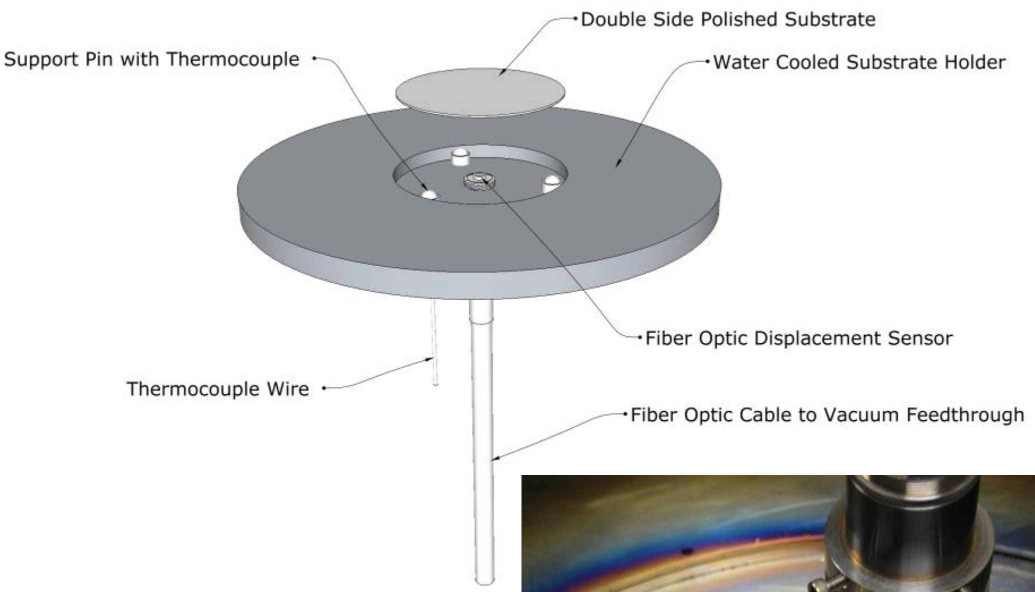
$$\sigma h_f = \frac{E_s h_s^2}{6(1-\vartheta s)}\kappa \quad \text{,where} \quad \kappa = \frac{2\delta}{r^2+\delta^2} \quad \xRightarrow{r \approx \frac{D_s}{2} \gg \delta} \quad \boxed{\sigma h_f = \frac{4}{3}\frac{E_s}{(1-\vartheta s)}\left(\frac{h_s}{D_s}\right)^2 \delta}$$

Effective spherical deformation

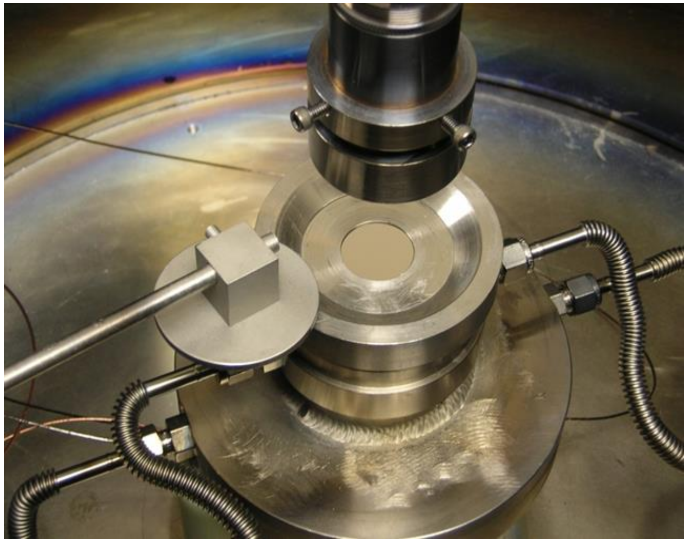


The curvature measurement is performed during deposition by measuring the displacement of the substrate center. The measured is performed on the opposing coated surface of a double side polished substrate using a high resolution fiber optic displacement sensor (i.e. ± 5nm). The displacement is "zeroed" prior to the process in order to measure the effective spherical deformation.

Prototype Instrument

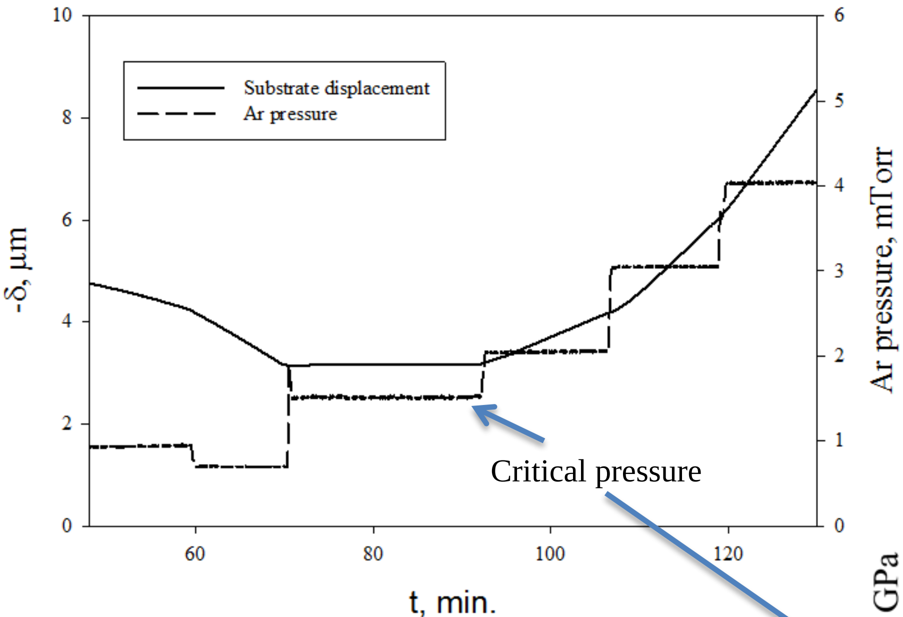


Spherometer



“Mechanical stress measurement during thin film fabrication”, NASA, United States Patent Application #14,645,994, (2015)

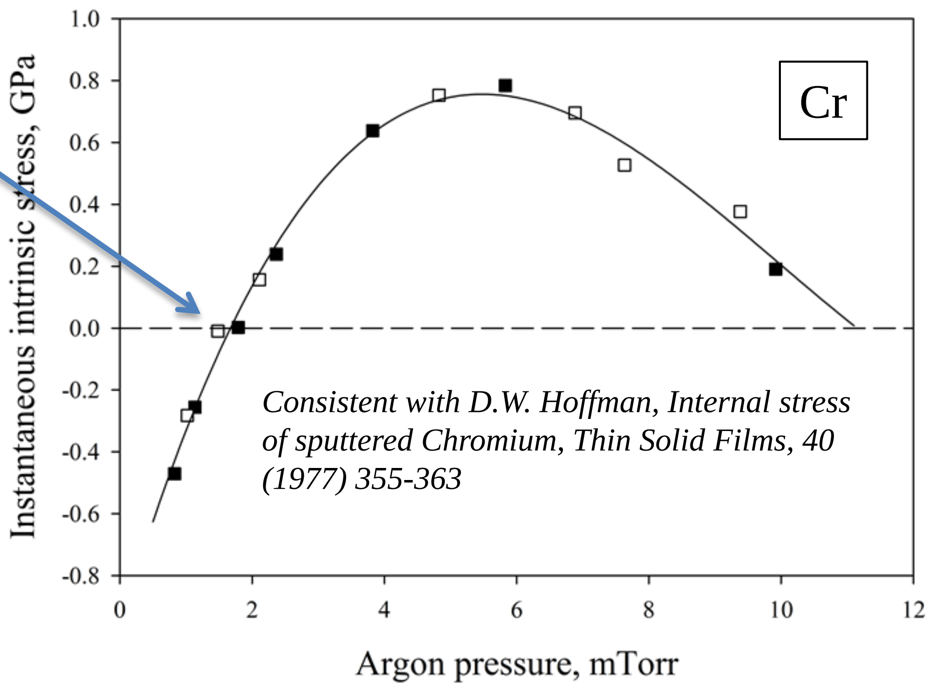
Incremental or instantaneous intrinsic stress



The incremental or instantaneous stress represents the change in the cumulative stress due to the stress in a film of differential thickness, dh_f, being deposited on the surface of the growing film at a rate of ξ (nm/s).

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

- No systematic error in the measurement since the substrate is in thermal equilibrium.
- The incremental stress is a convenient quantity since it is independent of film thickness (i.e. constant) for most metals shortly after the start of deposition (i.e. >10nm).
- Many data points can be collected in a single deposition run.
- Approach might be used to "tune" process pressure to zero stress.

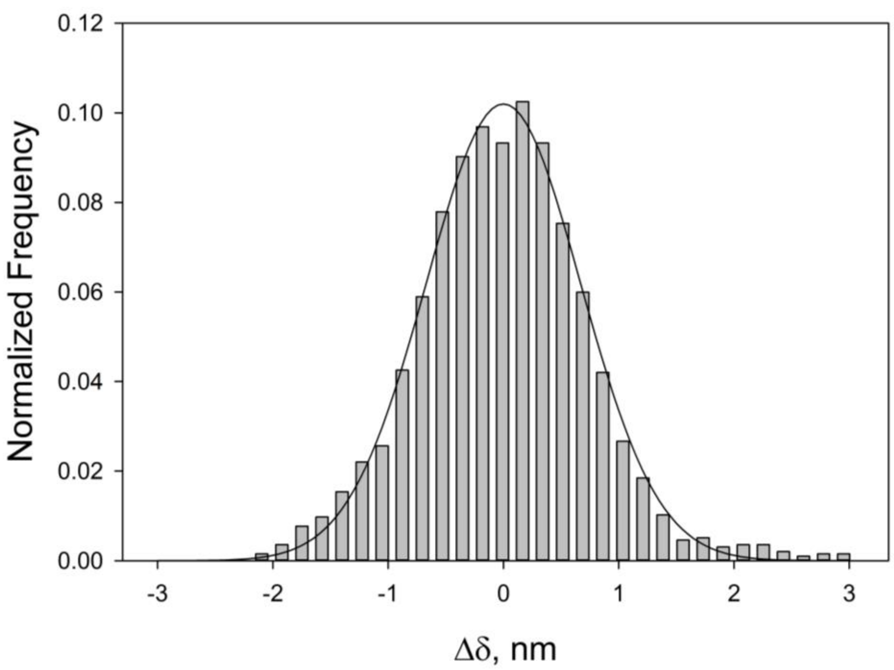


Stress Measurement Sensitivity:

To establish an estimate of the minimum detectable force per unit width of our apparatus, we sampled displacement data at 2.5 Hz during a thirty-eight minute deposition process of chromium for a thermally equilibrated substrate.

The normal distribution of random noise resulted in a FWHM of 1.69 nm.

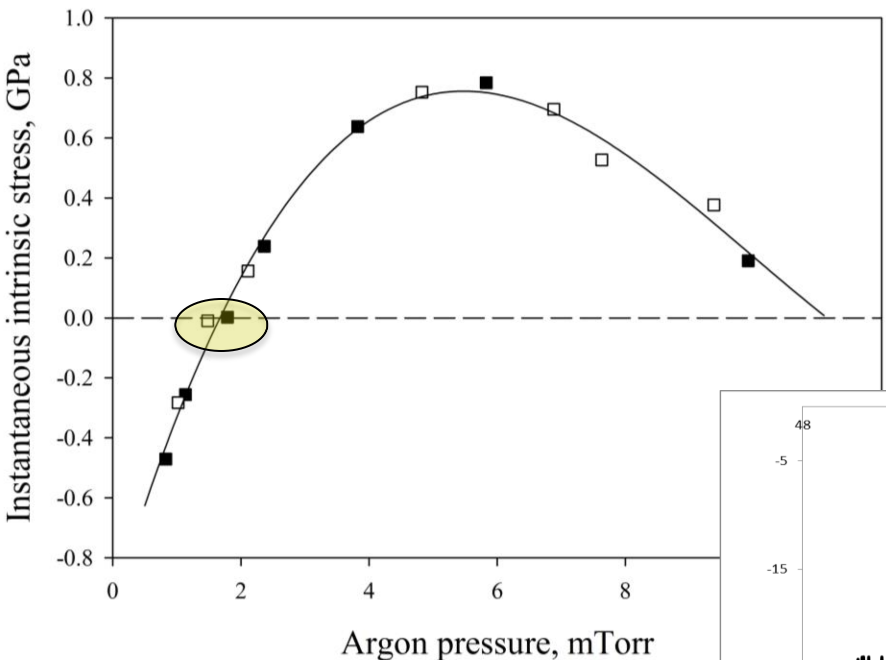
From this value, the sensitivity of the instrument for a 110 µm thick silicon substrate was calculated to be 0.015 N/m.



Sensitivity Comparison (110 µm):
MOSS: 0.050 N/m
Cantilever: 0.020 N/m
Spherometry: 0.015 N/m

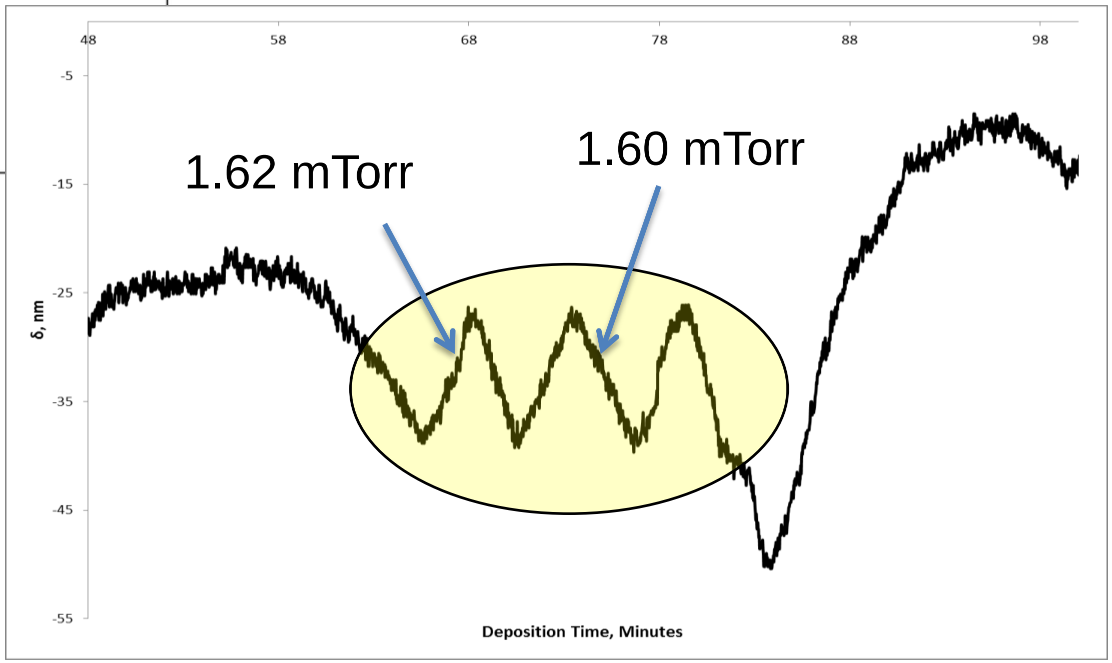
Sensitive enough to measure the stress in multilayers thin-films for x-ray optics.

Sensitivity at the transition pressure



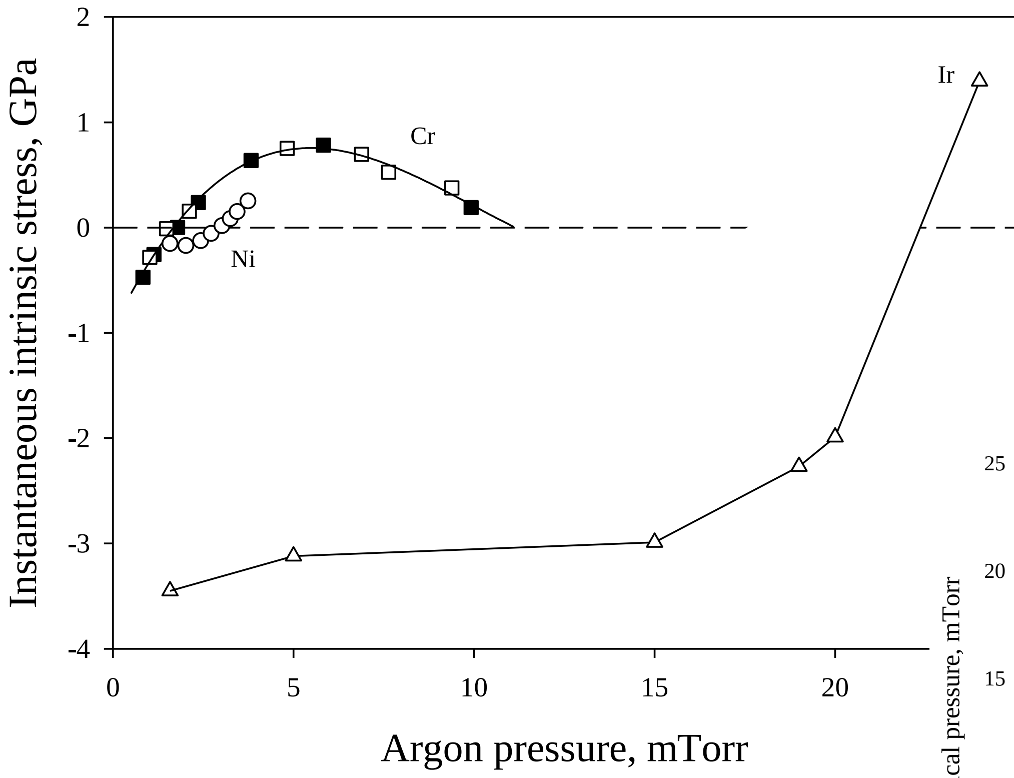
Stress reversal in Cr with argon pressure has been measured with the instrument. Consistent with the previous work of Hoffman (i.e. stress reversal).

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



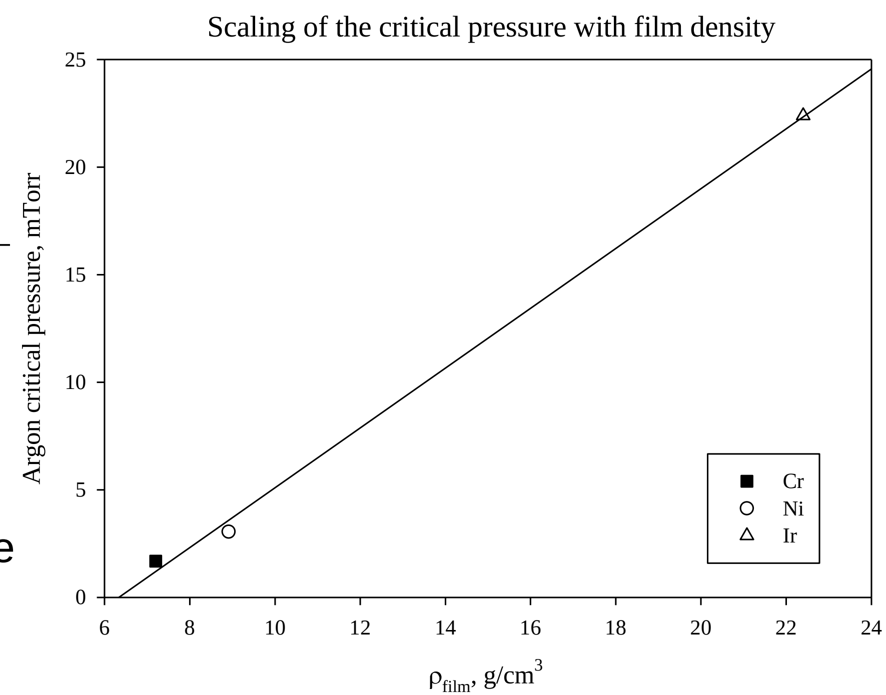
Correcting the raw data

Instantaneous stress in Ni, Cr, and Ir



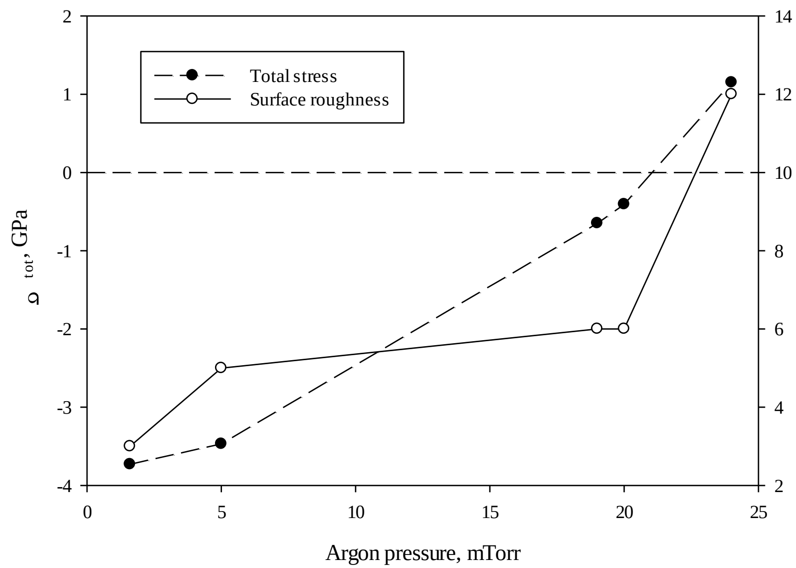
The critical pressure scales with density of the sputtered material

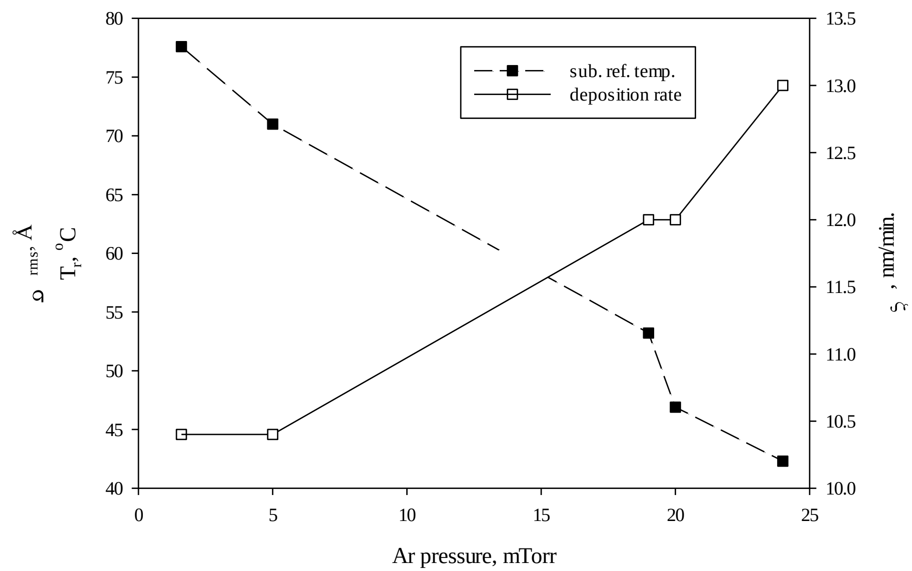
The stress in Cr which is commonly thought to have tensile stress can be made compressive by reducing argon pressure, for example.

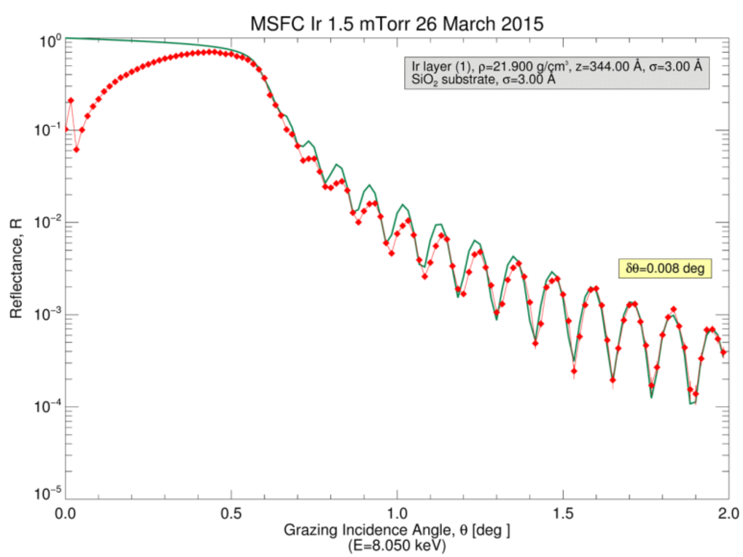


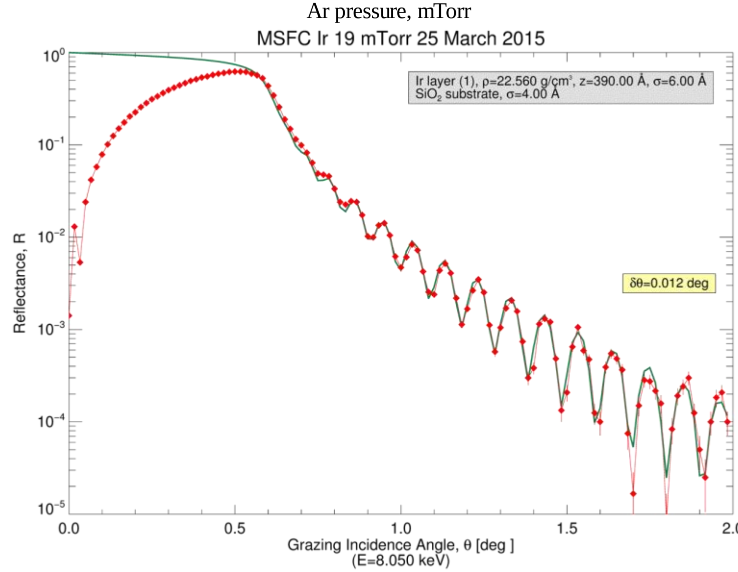
Single layer iridium films are used as a reflective coating for low energy x-rays.

Stress and roughness in the steady-state regime of film growth, Ir



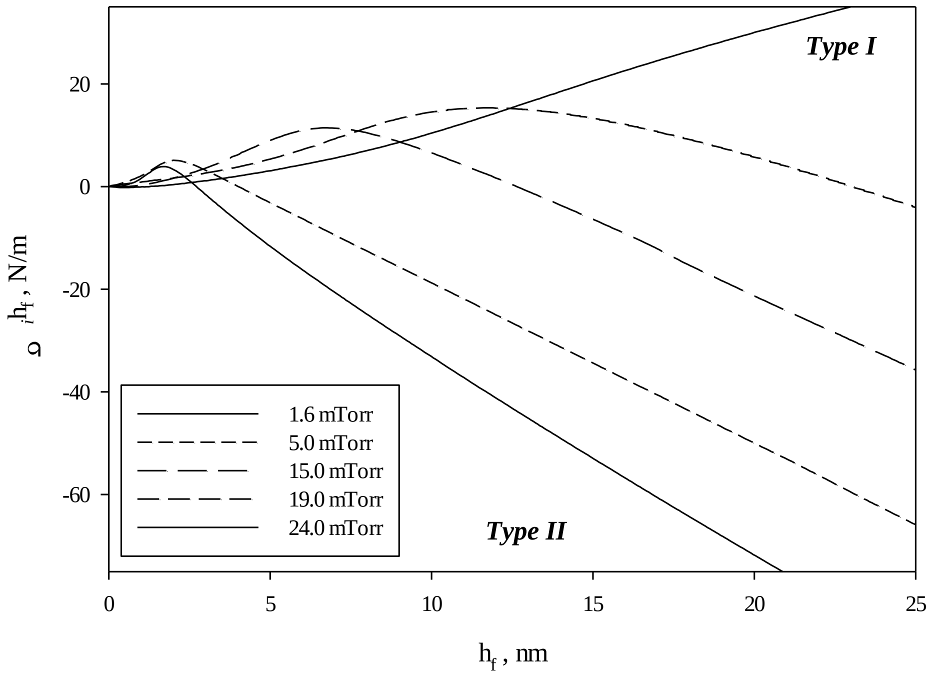


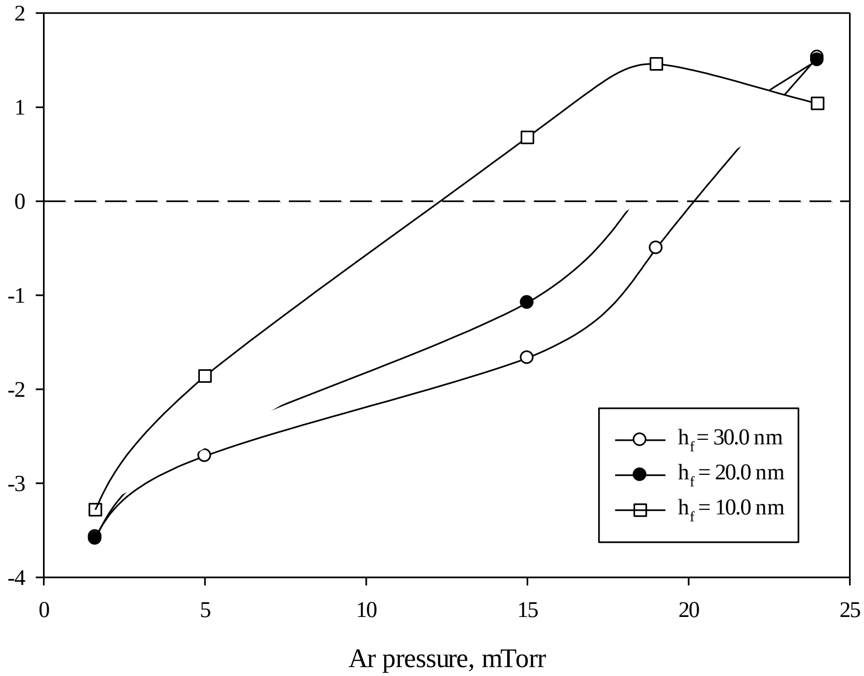




Intrinsic stress behavior in iridium

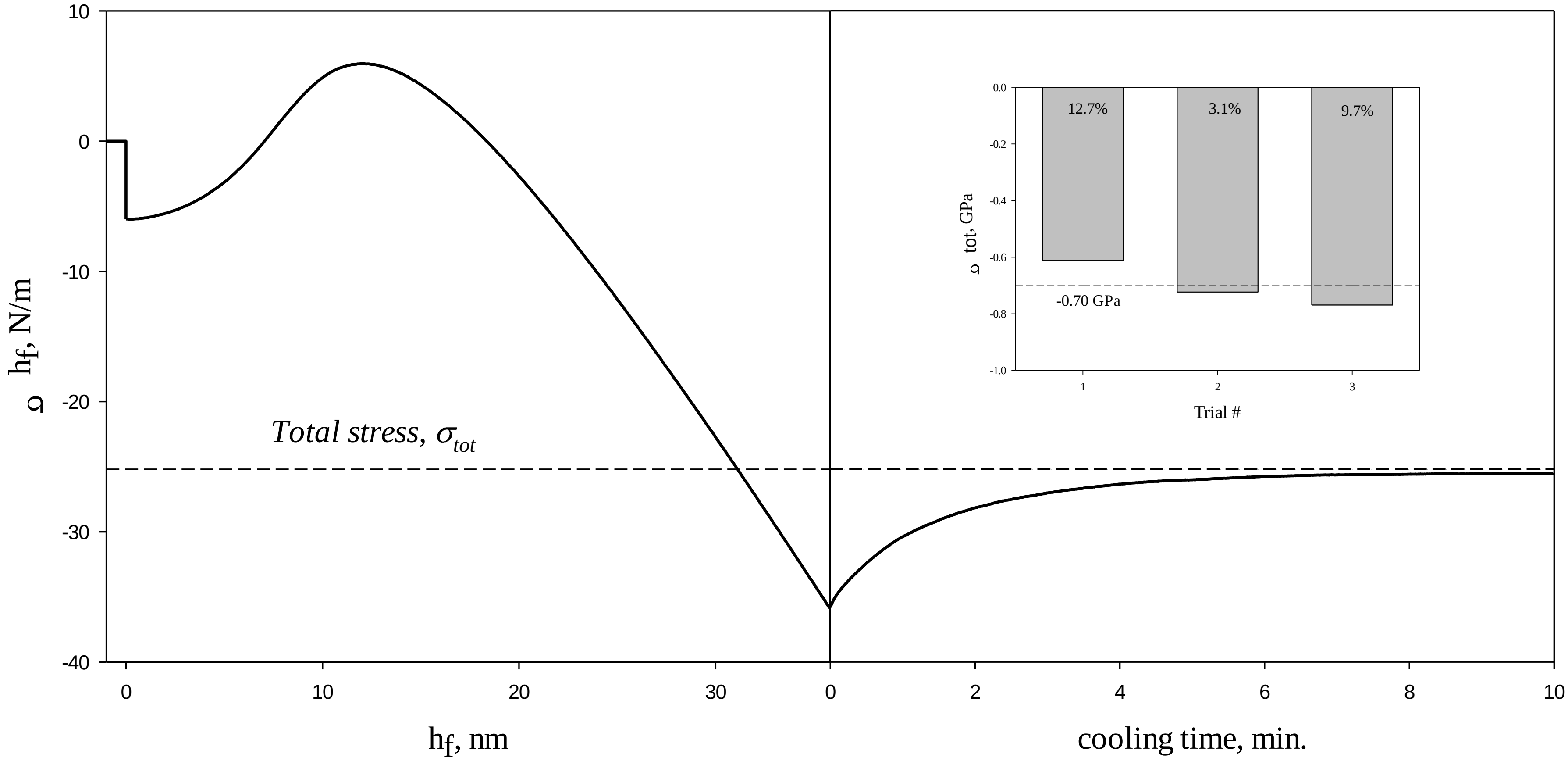
Zero stress can be achieved by increasing argon pressure



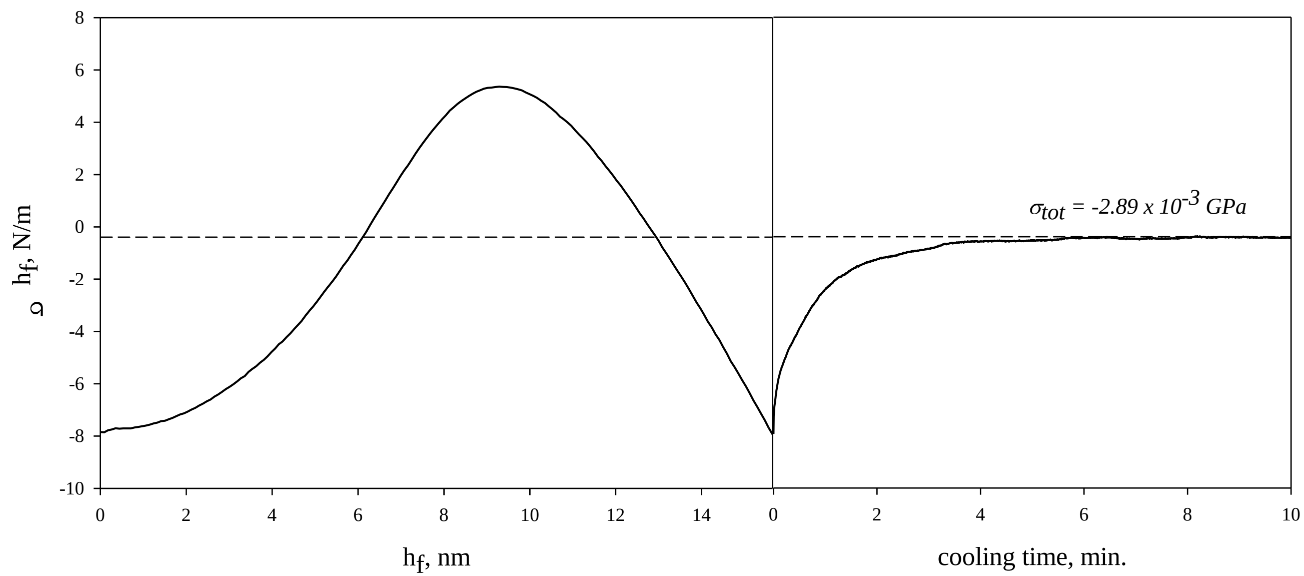


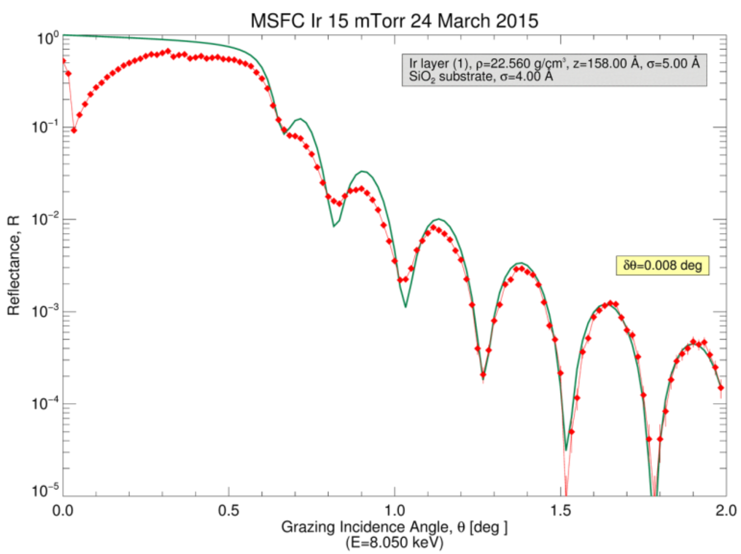
Total internal in-situ stress & measurement repeatability, Ir example

19.0 mTorr



Near-zero stress in Iridium (15.0 mTorr)





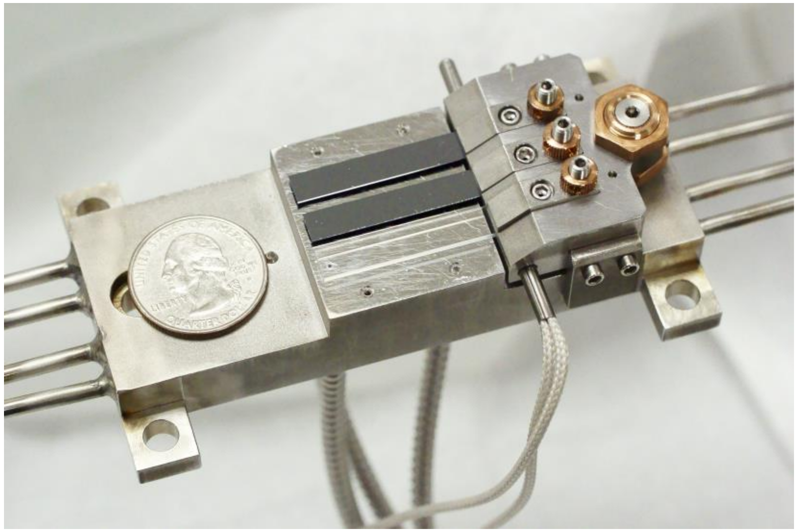
Reduction in the total stress by 3 orders of magnitude (i.e. to -2.89 MPa)

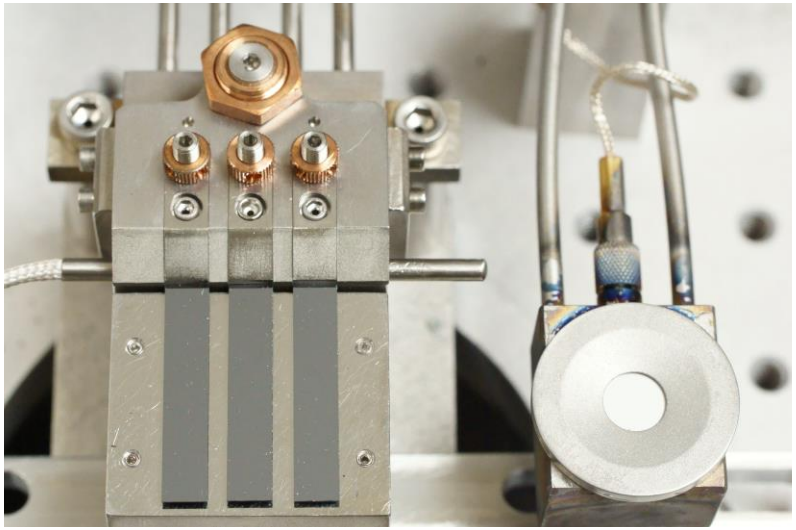
Good adhesion

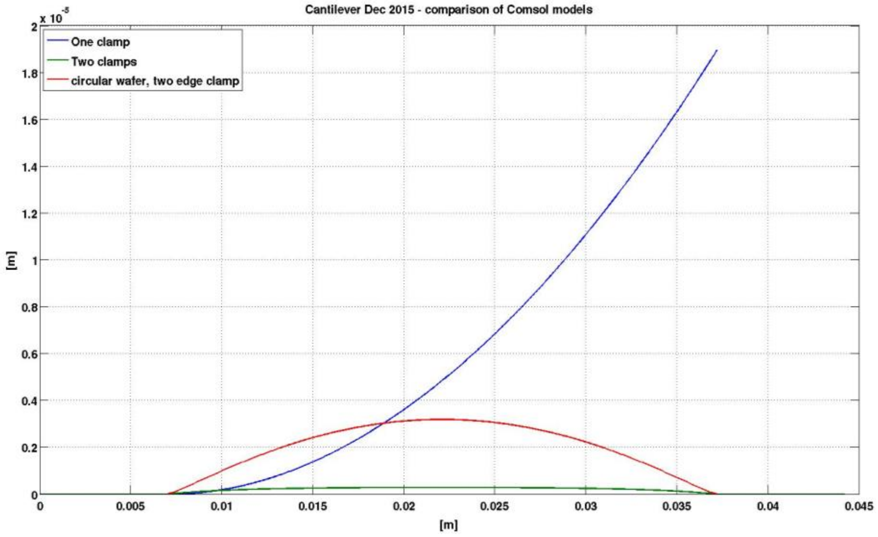
Promising result: 5Å rms roughness

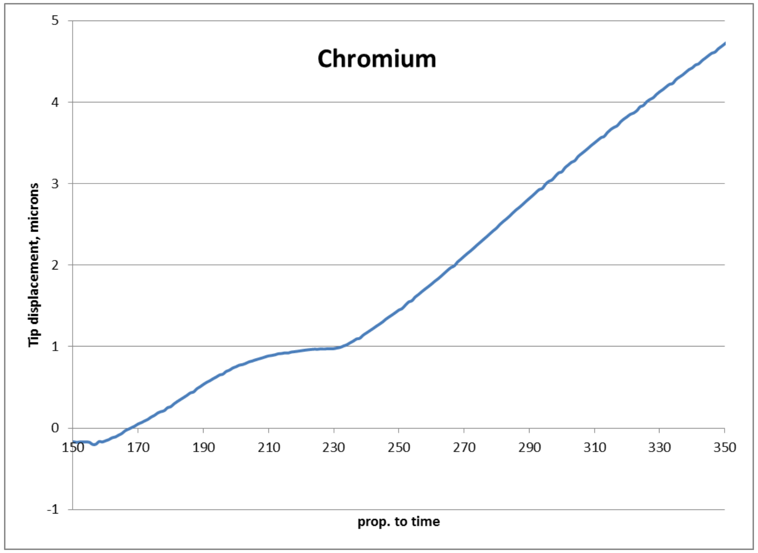
Further reduction in the roughness is possible through optimization of Ar pressure

Refined Instrument (Testing Underway)









Refined instrument capability

- Addition of a sensor to measure thermal and viabrational systematic error.
  - Heat generated during the deposition process causes systematic error in the measurement of the substrate displacement due to the thermal expansion of the substrate support mechanism.
  - The systematic error can be correlated to the substrate temperature and subtracted from the measured data. However, the sensitivity becomes limited by the resolution of the temperature measurement (i.e. from 0.015 N/m to 0.035 N/m).
- Capability to actively heat the substrate during deposition and for annealing.
- Capability to measure stress for films deposited on transparent substrates.
  - Use is currently limited to opaque substrates such as silicon.
  - Glass has consistent , good surface quality and is a cheaper alternative to silicon substrates.
  - Glass is amorphous and isotropic in its mechanical properties.
  - To be consistent with the substrate composition of the final optical instrument (i.e. such as slumped glass segmented substrates to be used by SAO for adaptive optics).
- Development of a method for extracting the intrinsic stress from the total measured stress.
  - Important since the intrinsic stress contains information about the microstructure of the film.
  - Requires accurate substrate temperature determination.

Thankyou!