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X-ray optical thin film development at MSFC

NASA MSFC
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A device for the in-situ measurement of thin film stress

TECHNOLOGY DRIVES EXPLORATION

Non-Proprietary

Presentation Outline

- X-ray astronomy group at MSFC
- Overview of technology
- Current development status
- Thin film stress
  - Technological challenges associated with
  - Motivation for device development at MSFC
  - Thin film stress measurement techniques
    - Deflectometry
    - Our method
      - Sensitivity
      - Some results
      - Advantages
- Target technology markets
- Summary of key points
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NASA MSFC X-ray Astronomy Group

- NASA MSFC
- X-ray astronomy group (ZP12)
- 9-civil servants, 3-post docs, 1- graduate student
- A subset of the group specializes in x-ray optic instrumentation including: full-shell replication, thin film x-ray optical coatings, detectors, and telescope module assembly and alignment.
- Funding provided by NASA Astronomy and Physics Research and Analysis Program

Overview of Technology

- We introduce a simple optical method for the in-situ measurement of the internal stress during film growth and thermal annealing processes.
- In-situ measurement more efficiently facilitates the control of film stress which can be influenced by the many process parameters including: substrate temperature, substrate bias, process gas pressure, and deposition rate.
- Current TRL level: 4-5
- The key attributes of this instrument lies in its high sensitivity, simplicity, and ease of implementation—hence, fostering general use.
- Our method overcomes many of the difficulties associated with other, more complex, optical in-situ techniques.
- We have demonstrated the use of the instrument through good qualitative agreement to published results, including the stress reversal in chromium and nickel.

Current Development Status of Technology

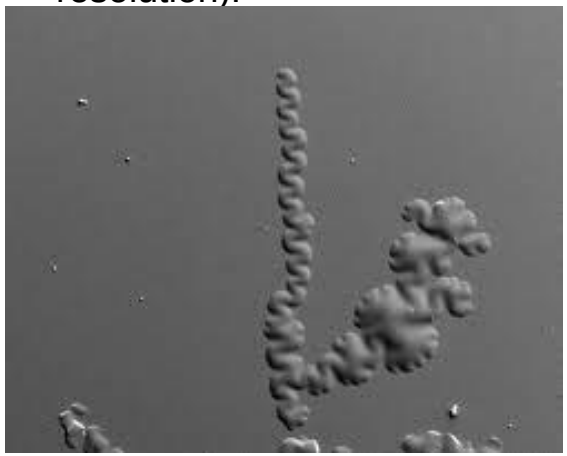
- The instrument is currently being used to collect data and conduct experiments for the investigation of the stress in thin films.
- A patent application has been filed.
- A manuscript which describe the methodology has been submitted to a peer reviewed journal (awaiting review).
- The next step of development include the extension of the methodology to heated and rotating substrates.
- The commercialization of the instrument could be appealing to a broad range of industries whose core competencies are in thin film devices, sensors, and optics (i.e. semi conductor, lithography, etc.).
- This device offers a cheaper alternative to current commercially available systems without sacrificing sensitivity.

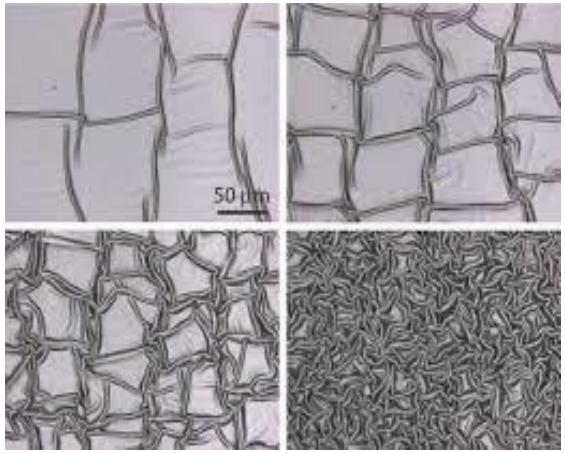
Thin Film Stress (A leading technological challenge)

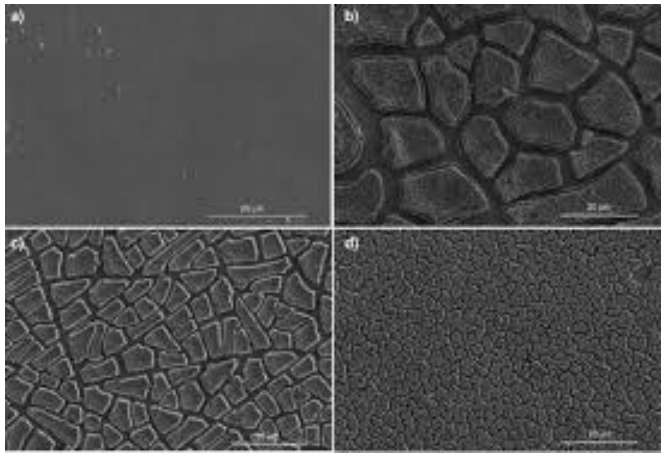
- Despite the small thickness of the films (i.e. sub-nanometer) the stress can be enormous for some materials (i.e. GPa's).
- An intrinsic property associated with the microstructure of the film.
- Can disqualify the use of a material which might otherwise be sought for its attractive physical characteristic(s).
- Can often be associated with a relevant device-specific performance characteristic.
- For magnetron sputter deposition the stress is highly process dependent and influenced by deposition conditions:
  - Gas Pressure (stress reversal)
  - Deposition Rate (cathode power)
  - Substrate Temperature
  - Substrate bias

Internal stress is a leading technological challenge in a broad range of thin film devices, sensors, and optic applications.

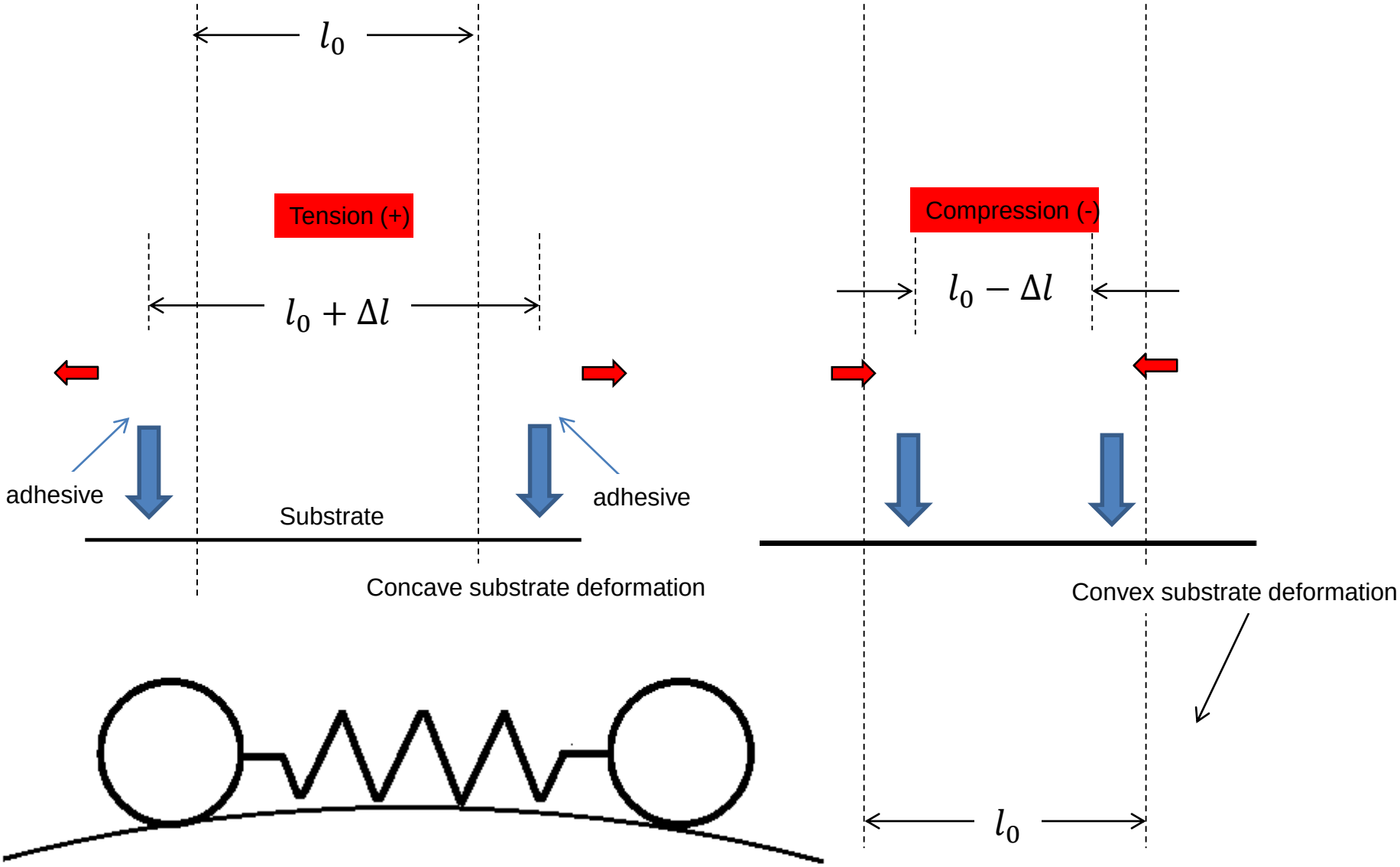
- The stress can cause the film to buckle, crack or delaminate from the substrate.
- Substrate distortion and deformation necessarily arise as a consequence of film stress and is a significant concern for films deposited on precisely figured substrates (i.e. degrades focal spot size and imaging resolution).







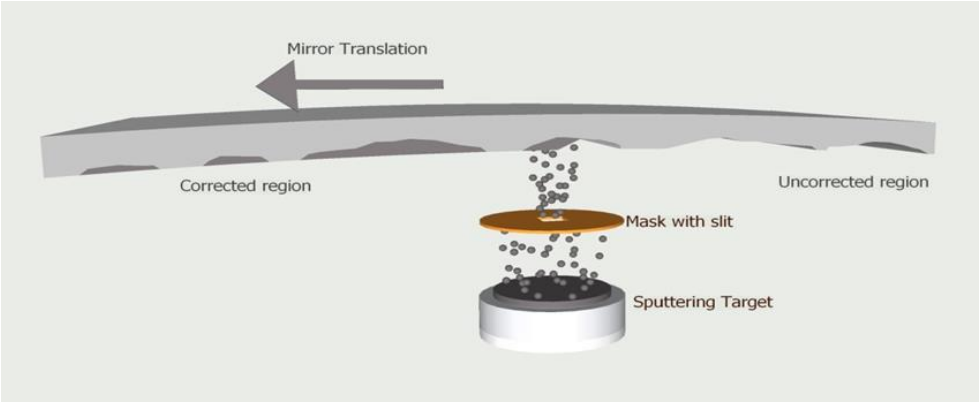
Stress/Spring Model



The film will delaminate if the stress is greater than the adhesion between the film as substrate

MSFC motivation for in-situ stress measurement capability: X-ray optics

- MSFC specializes in thin (10's of μm's) full shell replicated optics for x-ray astronomy.
- A technique for the preferential deposition of material is being used in order to correct mid-spatial-frequency figure errors resulting from internal stresses in the electroforming process or imperfections in the formed mandrel. This technology holds promise for improving the angular resolution of the next generation of space telescopes into the sub-arc second regime.
- In-situ monitoring of the film stress is highly beneficial since the stress in the filler layer can be "tuned" to zero during the deposition process. Otherwise, the stress in the correcting layer can itself distort the figure.
- The internal surfaces are coated with a highly stressed iridium x-ray reflective coatings (i.e. GPa's). In-situ monitoring of the stress is helping us to quickly identify methods for reducing or eliminating the stress in this sought after x-ray reflective material.



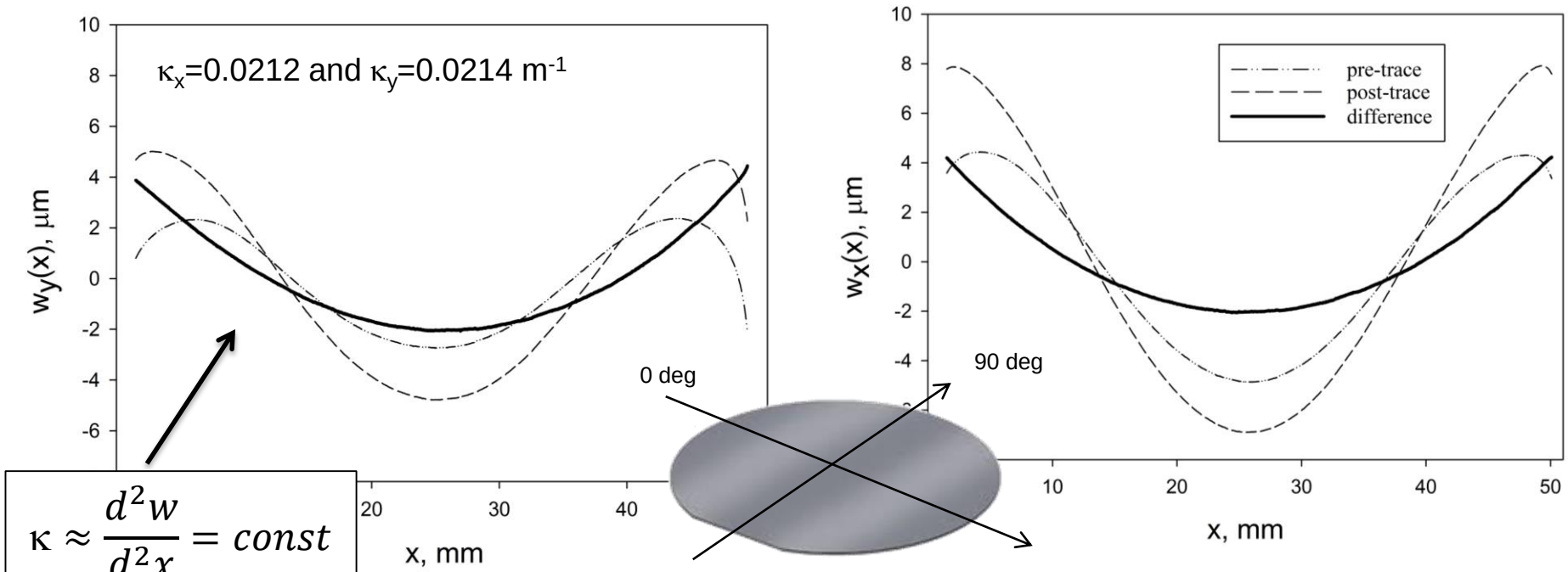
Measurement of thin film stress (Ex-situ method)

- The change in substrate curvature, κ, is measured before and after the thin film process--usually by profilometry or laser deflectomerty.
- The curvature, κ, of the deformed substrate is proportional to the product of film stress and film
- thickness, σh_f, through a constant that describes the geometric and mechanical properties of the substrate (Stoney's Equation)--namely, the substrate's thickness, h_s, and biaxial modulus, $\frac{E_s}{(1-\vartheta_s)}$.

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

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

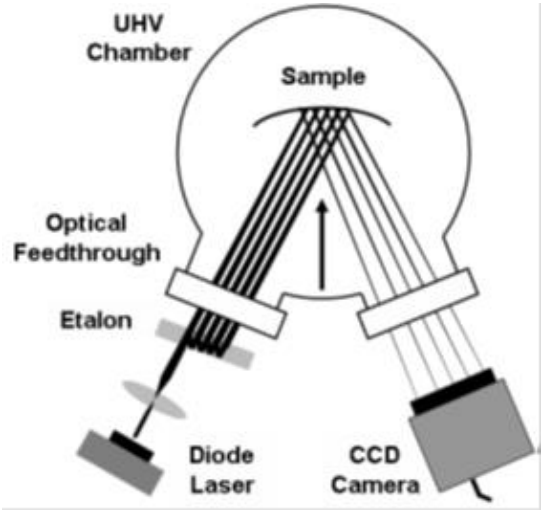
- The substrate will deform spherically provided the substrate is thick enough.
- Spherical deformation implies a constant curvature.



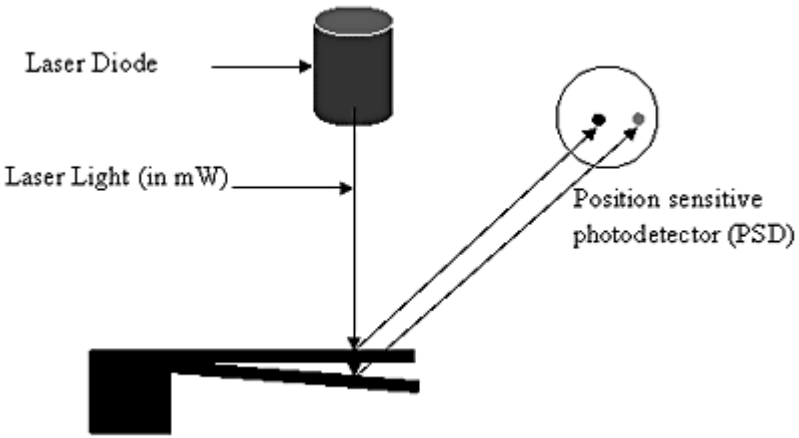
$$\kappa \approx \frac{d^2w}{d^2x} = const$$

Other optical in-situ film stress measurement methods (deflectometry):

Multi-beam optical stress sensor (MOSS):



Micro cantilever:



Disadvantages of optical deflectometry techniques:

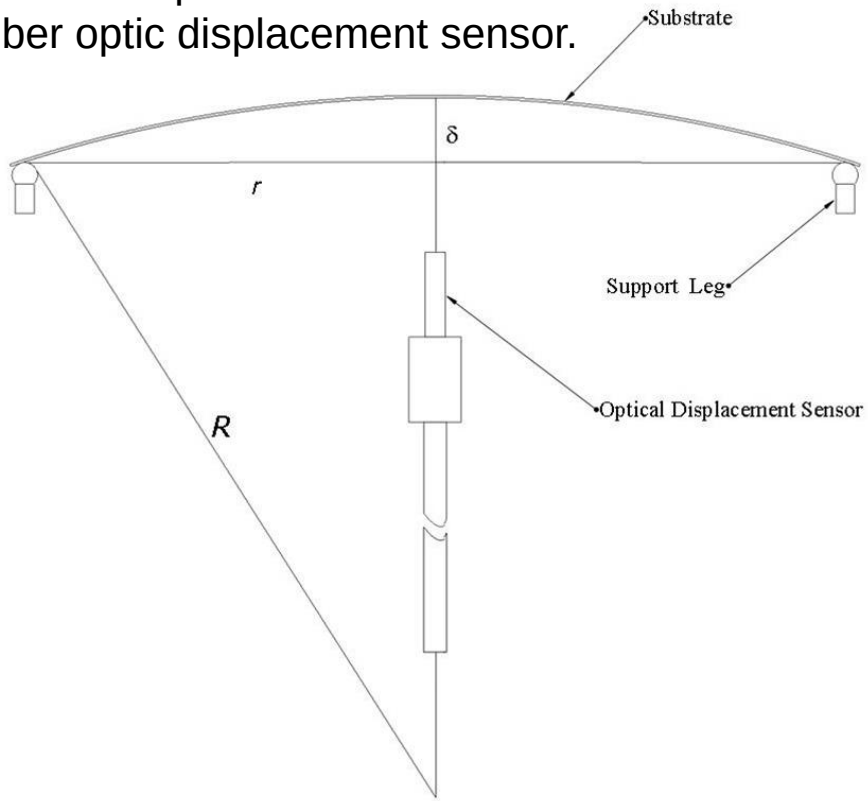
1. Requires optical access to the substrate (i.e. substrate is in vacuum).
2. High quality glass viewports are often needed.
3. Interference between film and substrate can occur for transparent films causing low reflectivity and loss of signal.
4. The vacuum system must be designed to geometrically accommodate the optical components.
5. High cost of implementation can discourage their general use.

In-Situ methodology (sphereometry):

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

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

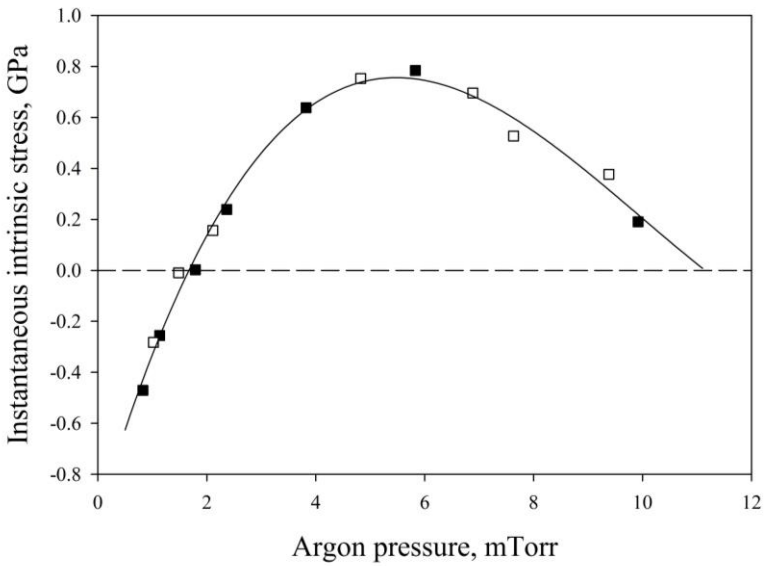
The curvature measurement is performed during deposition by measuring the backside of a double side polished substrate with a non-contact variation of the classic spherometer using a fiber optic displacement sensor.



Patent application filed

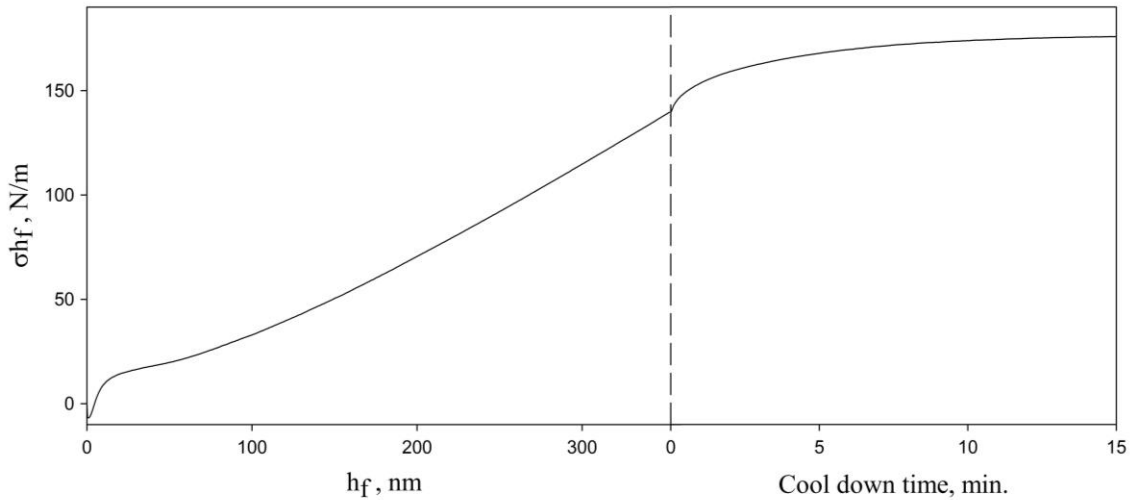
Manuscript on methodology recently submitted to The Journal of Thin Solid Films

In-Situ stress in Cr films



Results obtained with the instrument are consistent with other published results:

[1] D.W. Hoffman, Internal stress of sputtered Chromium, Thin Solid Films, 40 (1977) 355-363

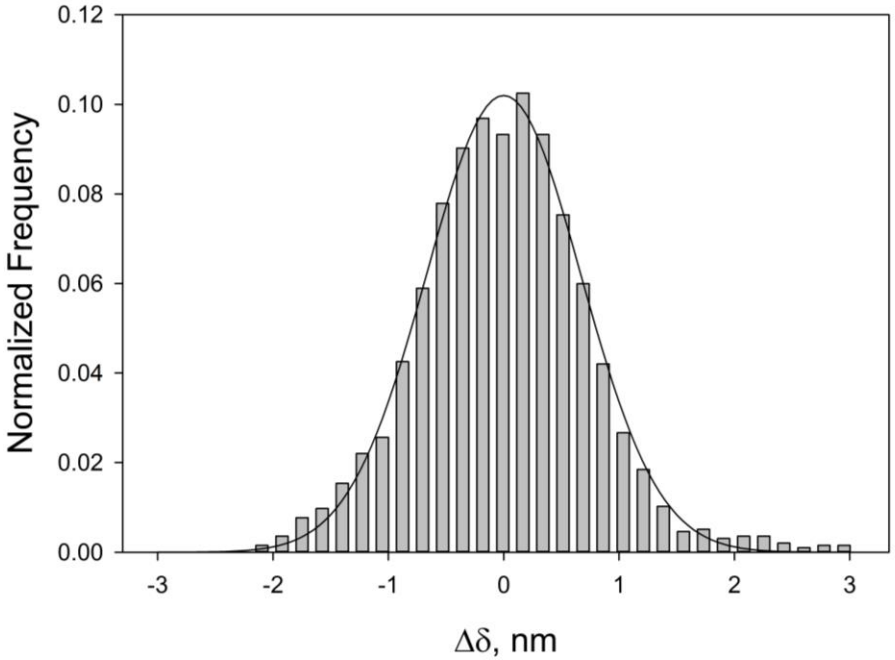


Stress Measurement Sensitivity:

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.

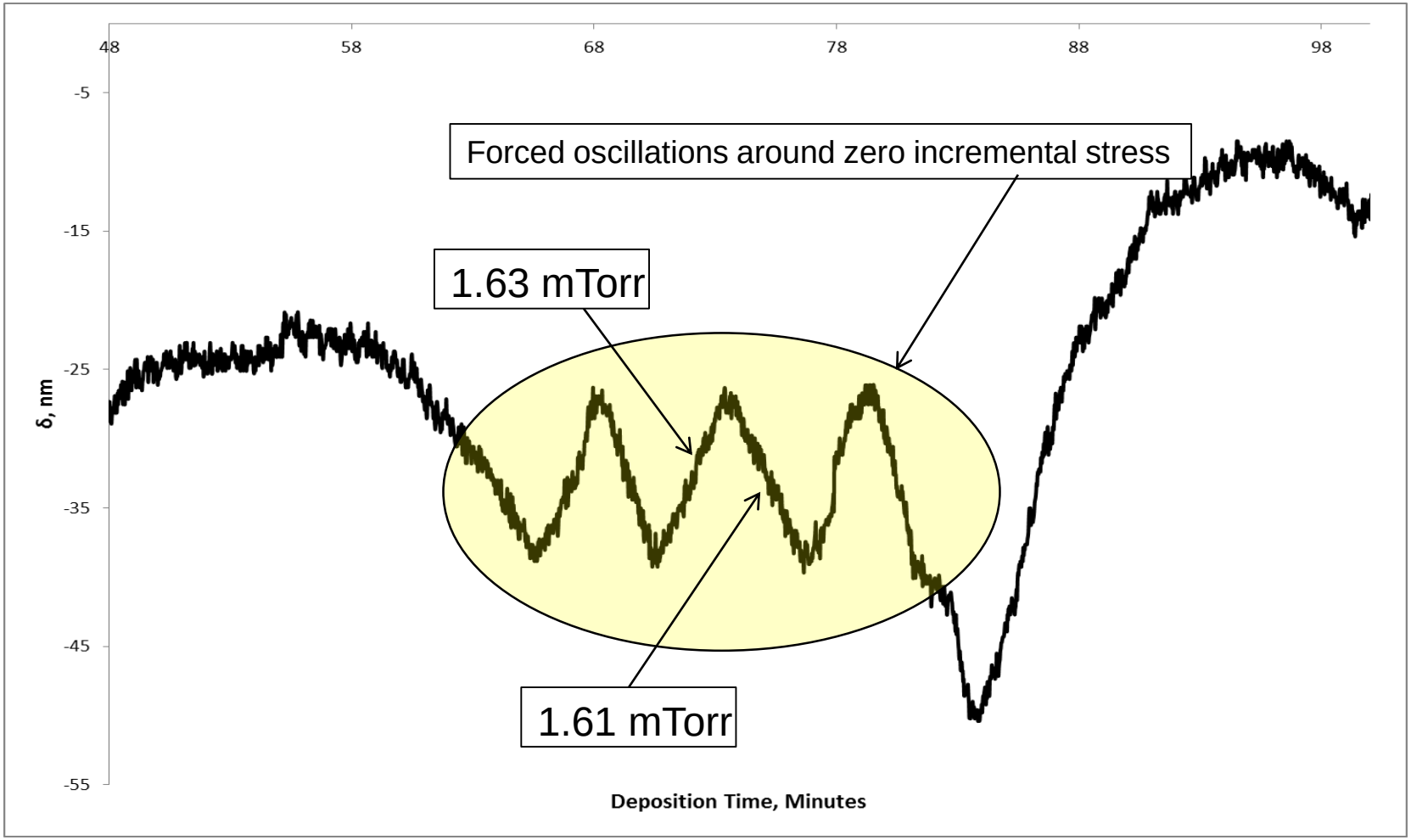


Sensitivity Comparison (110 µm):
MOSS: 0.050 N/m
Cantilever: 0.020 N/m
Spherometry: 0.015 N/m

The highest sensitivity of 0.015 N/m can be achieved with the use of a second sensor to subtract the systematic thermal expansion error of the device.

Sensitivity (Cont'd)

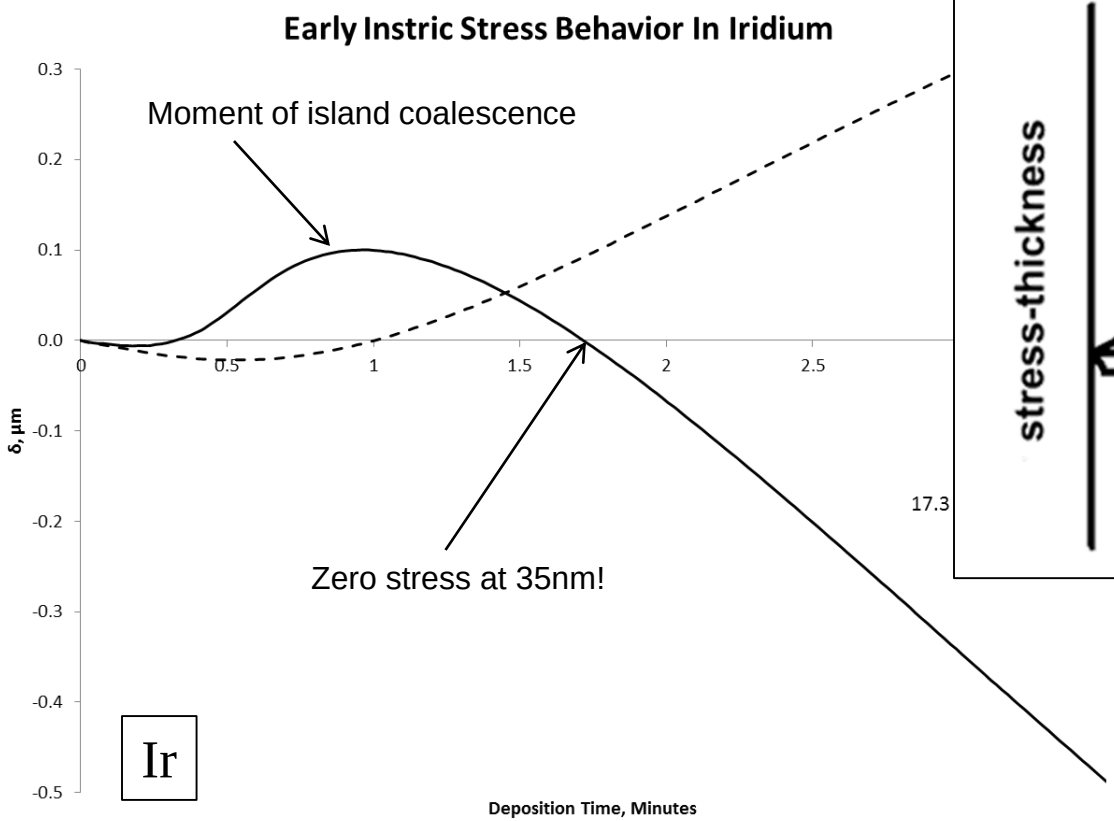
Measurement sensitivity is better than resolution in the control of process parameters.

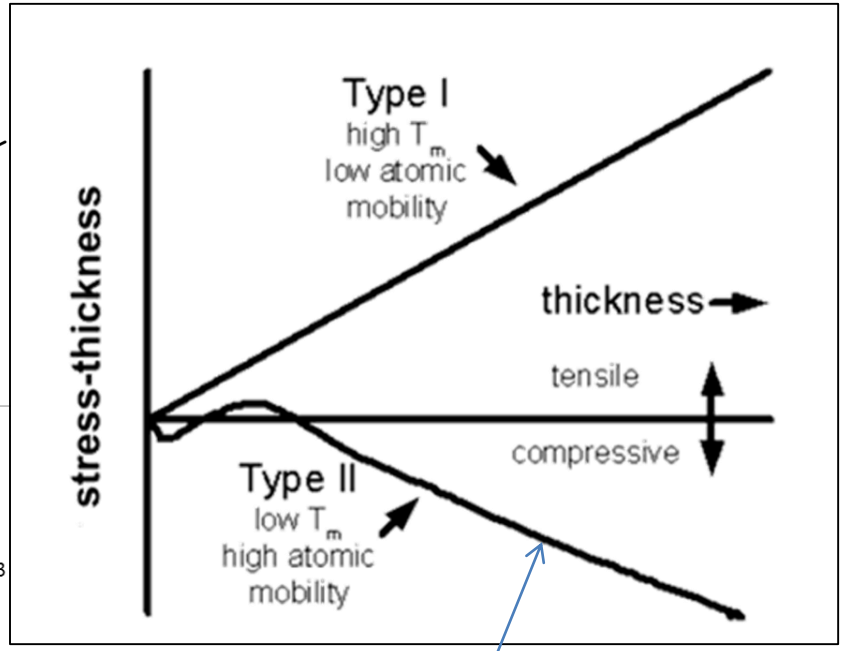


Stress during early growth of iridium films

Iridium is a material of interest for grazing incidence X-ray reflectivity due to its high optical density which results in a large critical angle (i.e up to 10 keV)

Most X-ray optical coatings range in thickness from a few tenths to tens of nanometers (~ 30 nm for total reflection optics).





Our results are qualitatively consistent with other published results

After R. Abermann, Vacuum 41, 1279 (1990) (top); Measured In-Situ (bottom)

Target Technology Market Applications

- The commercialization of the instrument could be appealing to any of a broad range of industries whose core competencies are in thin film devices, sensors, and optics.
- The application of the device is as broad as thin film technology itself.

Summary of Key Points

- In-situ—Conducts measurements during thin film deposition, providing immediate feedback on the effects of process changes.
- Real-time measurement—Allows for the adjustment of process parameters (e.g., process gas pressure) to optimize or eliminate stress during thin film fabrication process
- Low-cost, simple design—Uses off-the-shelf fiber optic probes for a less complex and less expensive system than traditional ex-situ approaches, reducing costs by an order of magnitude or more.
- Sensitive—Offers sensitivity of 0.015 N/m, comparable to a multi-beam optical stress sensor (MOSS) and potentially as sensitive as the micro-cantilever method
- Flexible—Measures stress during film growth, or the evolution of stress with thermal annealing.
- Versatile—Applies to a wide range of thin film and bulk material applications subject to stress, with no limitation on substrate size or reflective characteristics of deposited films.
- Easily implemented—Requires little or no modification to the vacuum chamber; the fiber optic probe can easily be inserted through flanges commonly found in vacuum systems.
- Other advantages-No interference effects between a transparent film and substrate that can sometimes cause a potential loss of signal as with other methods.

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Thank you!