



Status of low temperature coating loss measurements

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Overview

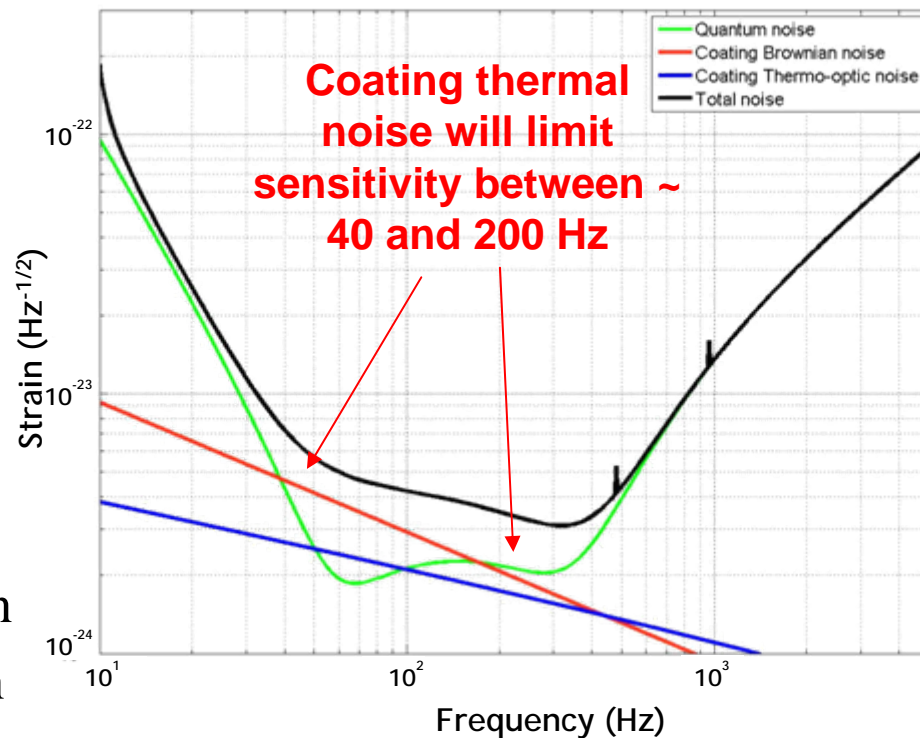
- Temperature dependence of coating dissipation
 - Effect of doping on low T dissipation in Ta_2O_5
 - Heat treatment of Ta_2O_5 coatings
 - Ion beam sputtered silica
 - Comparison to multilayer coating loss results
 - Comparison of silica deposition methods
 - Other possible coating materials – hafnia
- Studies of coating microscopic structure and short range order
- Coating material properties
 - Density
 - Young's modulus

Coating thermal noise

- **Thermal noise** associated with the dielectric mirror coatings expected to be a significant limit to the sensitivity of second generation detectors

- Level of thermal noise related to mechanical dissipation of coating material

- Current coatings use alternating layers of SiO₂ (low index) and Ta₂O₅ (high index)
 - Ta₂O₅ layers are the dominant source of mechanical dissipation
 - **Doping** the Ta₂O₅ with TiO₂ can reduce the dissipation

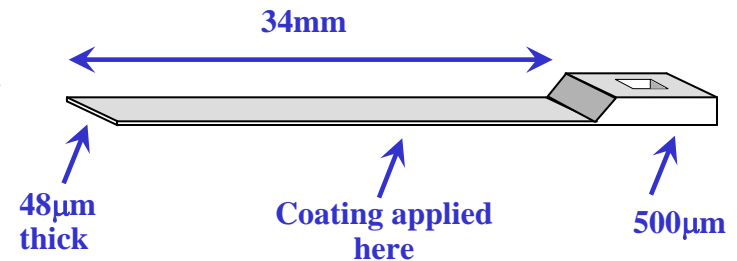


Projected Advanced LIGO sensitivity curve

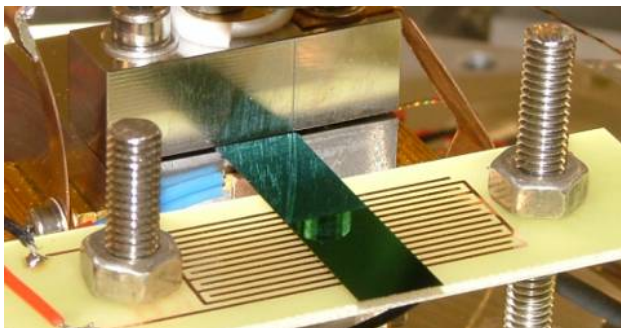
Cryogenic loss measurements of single layer coatings

- Studying the temperature dependence of the dissipation is of interest to:
 - Understand **dissipation mechanisms**
 - Evaluate coatings for possible use at **cryogenic temperatures**

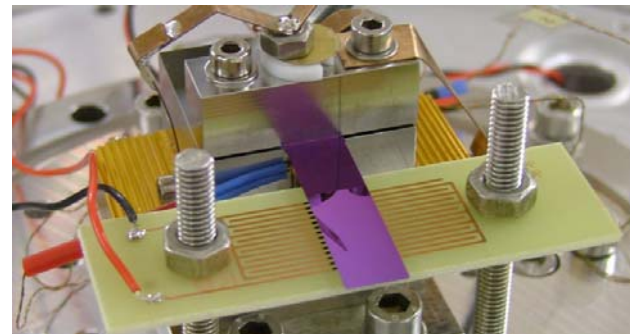
- Single layers of coating materials ($0.5 \mu\text{m}$ thick) applied to silicon cantilever substrates, fabricated at Stanford



- Coatings supplied by LMA and CSIRO



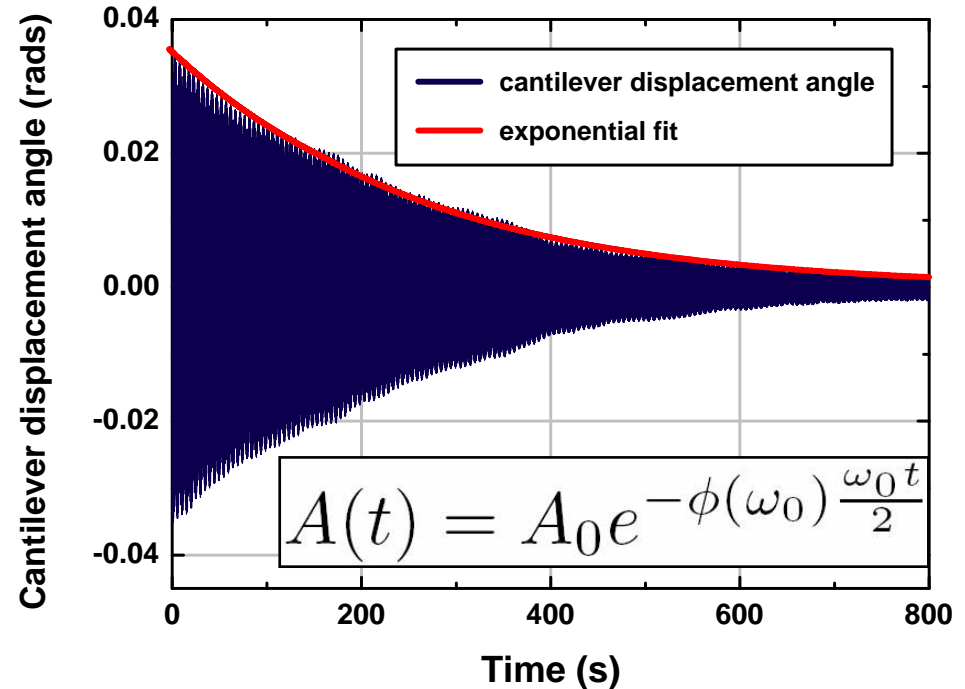
G0900645-nm
0645-nm doped tantala (LMA) coated silicon cantilever in clamp



Silica (LMA) coated silicon cantilever in clamp

Measuring coating loss

- Bending mode of cantilever excited electrostatically
- Loss $\phi(\omega)$ obtained from exponential fit of amplitude decay
- Coating loss calculated from the difference in loss of a coated sample and an identical uncoated sample:



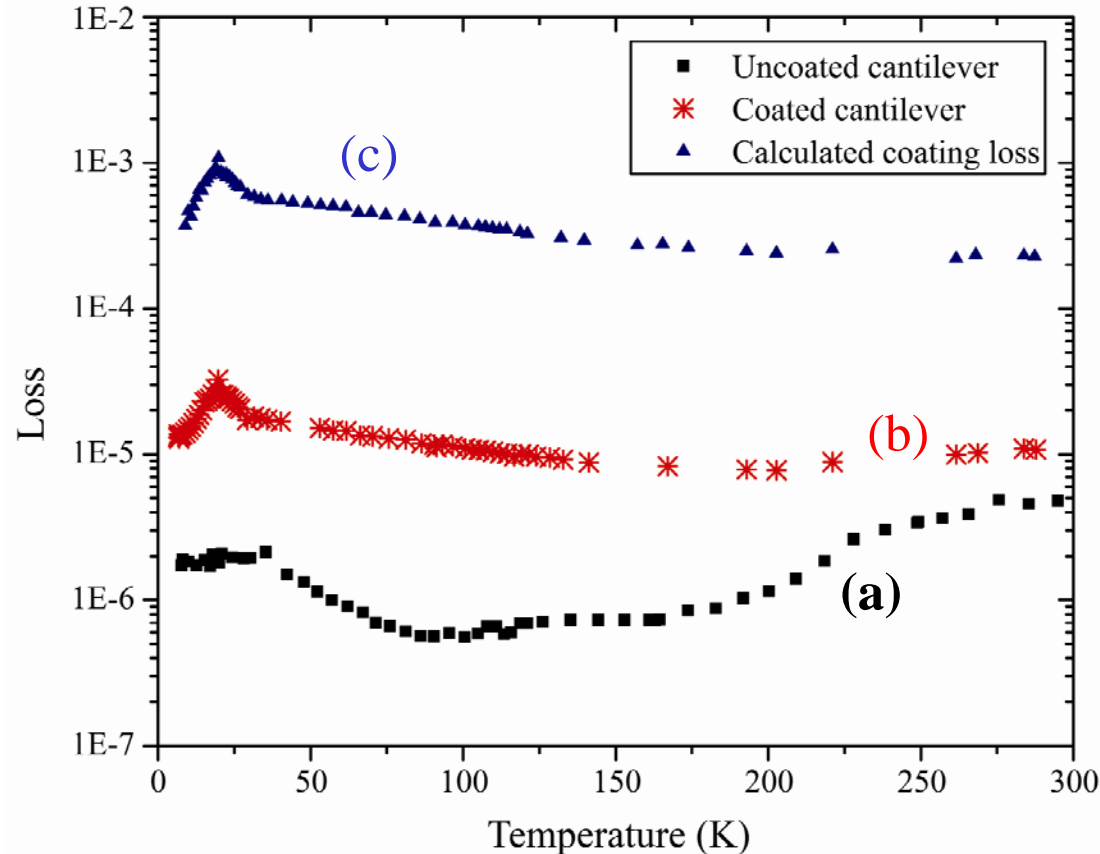
$$\phi_{\text{coating}} = \frac{Y_{\text{cantilever}}}{3Y_{\text{coating}}} \frac{t_{\text{cantilever}}}{t_{\text{coating}}} (\phi_{\text{coated-sample}} - \phi_{\text{un-coated-sample}})$$

Ratio of energy stored in cantilever to energy stored in coating

Difference in loss between coated and un-coated cantilevers

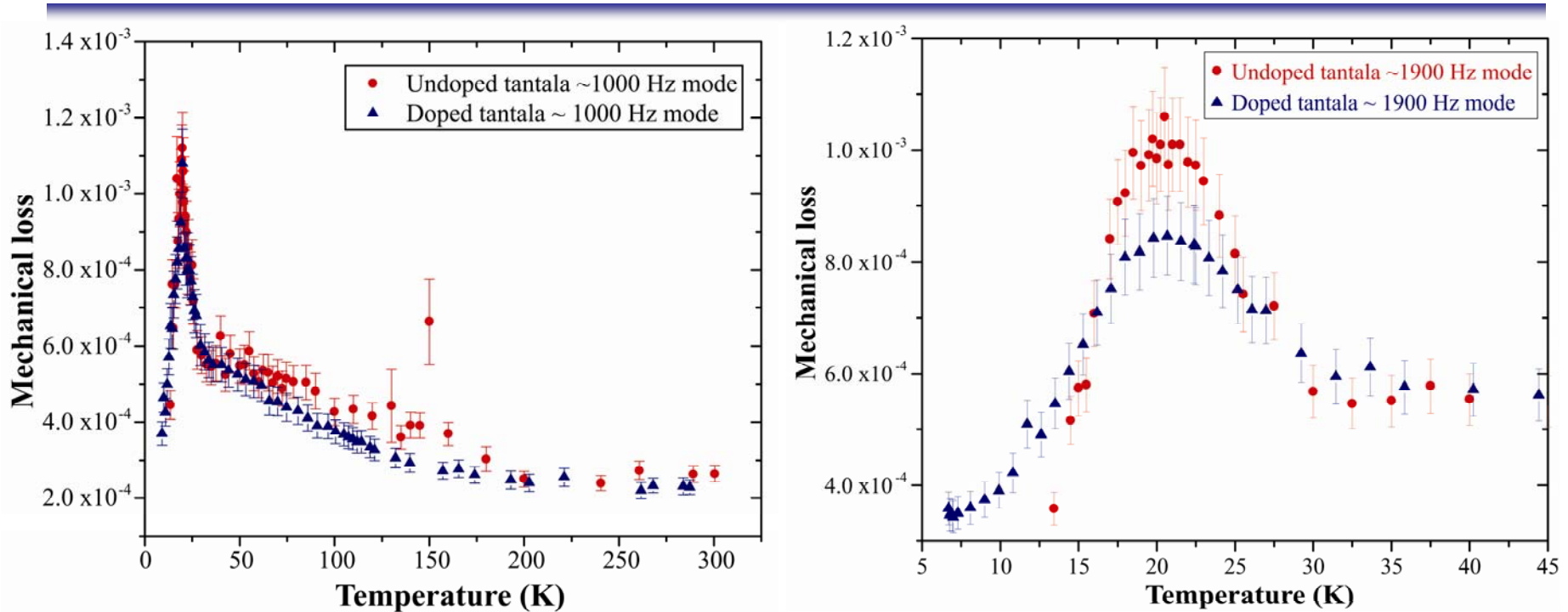
Dissipation peak in tantalum coatings

- Initial measurements below 77 K carried out in collaboration with Jena University
- Dissipation peak at ~ 18 – 20 K found in Ta₂O₅ coatings
- Loss of silicon cantilever substrate increases at low temperature due to **thermal oxide layer** - necessary for coating adhesion.



Loss of (a) uncoated silicon cantilever with thermal oxide layer, (b) cantilever coated with 500 nm of TiO₂-doped Ta₂O₅ (14.5 % Ti) and (c) the calculated loss of the coating layer

Effect of doping on loss of Ta₂O₅ coatings



Comparison of dissipation in **doped** (14.5 % TiO₂) and **un-doped** Ta₂O₅ for 3rd (left) and 4th (right) bending modes.

- Doping **reduces the height** of the peak and **slightly increases the width** of the peak
- Doping reduces loss of Ta₂O₅ throughout temperature range, with the exception of the wings of the peak

Analysis of coating loss peak (TiO₂ doped coating)

- Most dissipation mechanisms described as **Debye relaxations** between equilibrium states, with **relaxation time** τ :

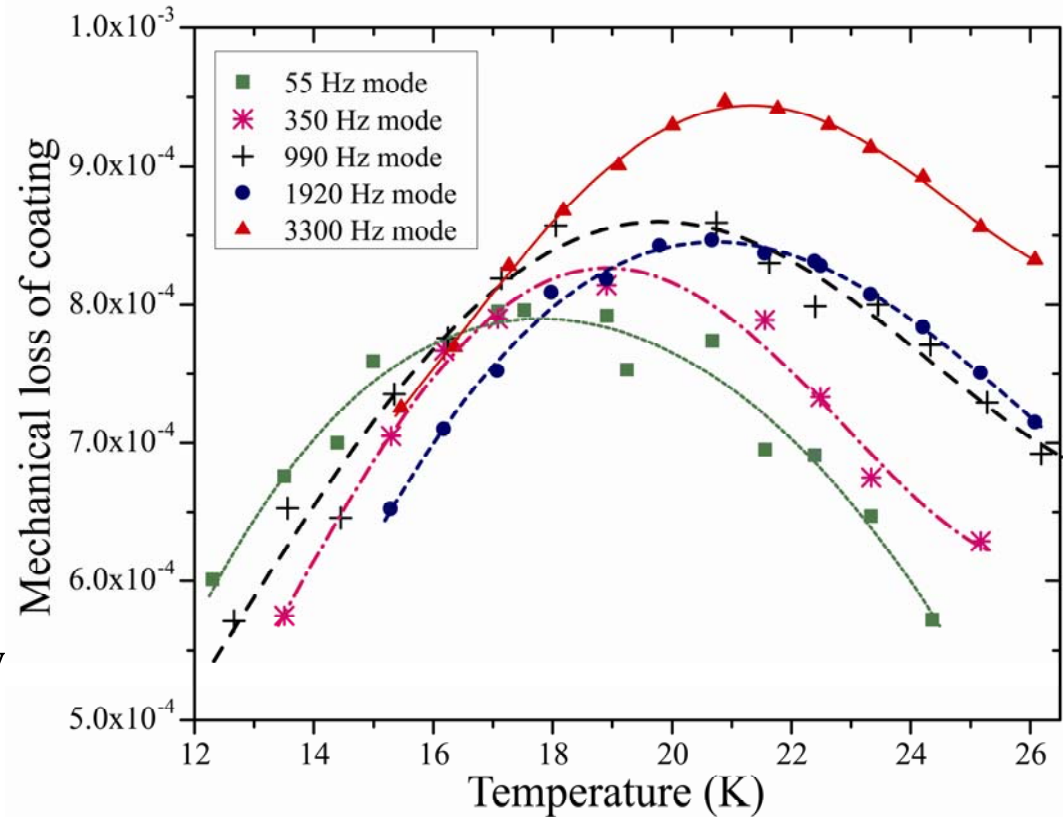
$$\phi(\omega) = \Delta \frac{\omega\tau}{1 + (\omega\tau)^2}$$

- T_{peak} increases with mode frequency – this indicates a **thermally activated** dissipation mechanism, where τ is given by the Arrhenius equation:

$$\tau = \tau_0 e^{\frac{E_a}{K_B T}}$$

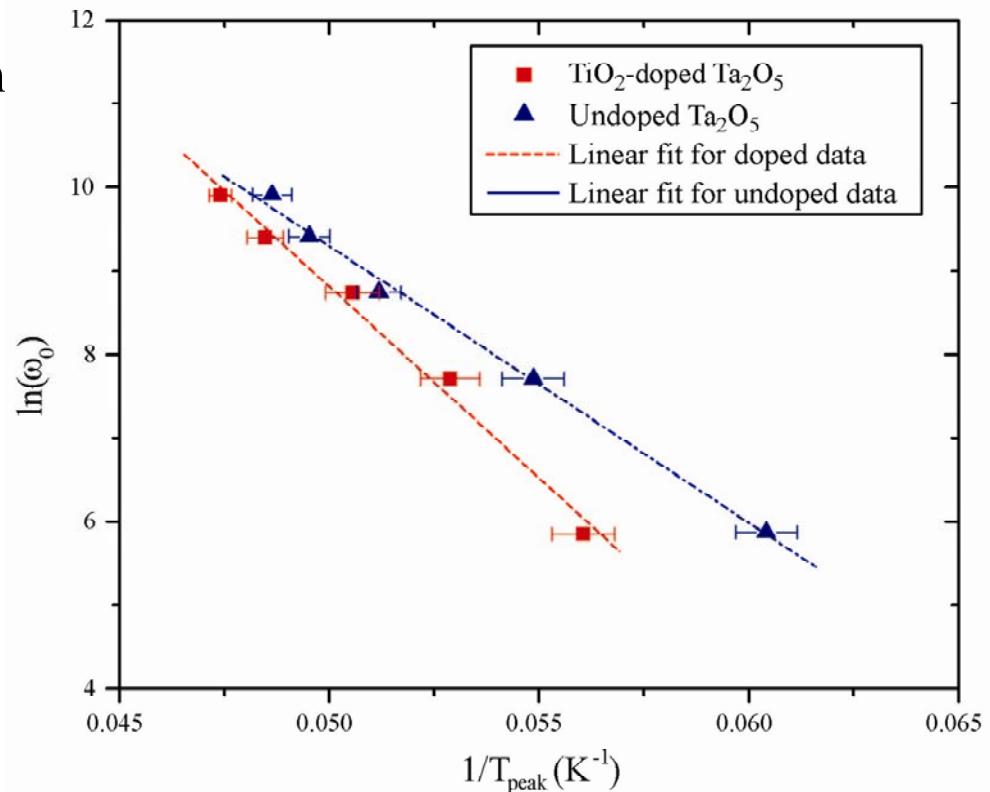
– τ_0 is the **relaxation constant**

– E_a is the **activation energy** for the dissipation process

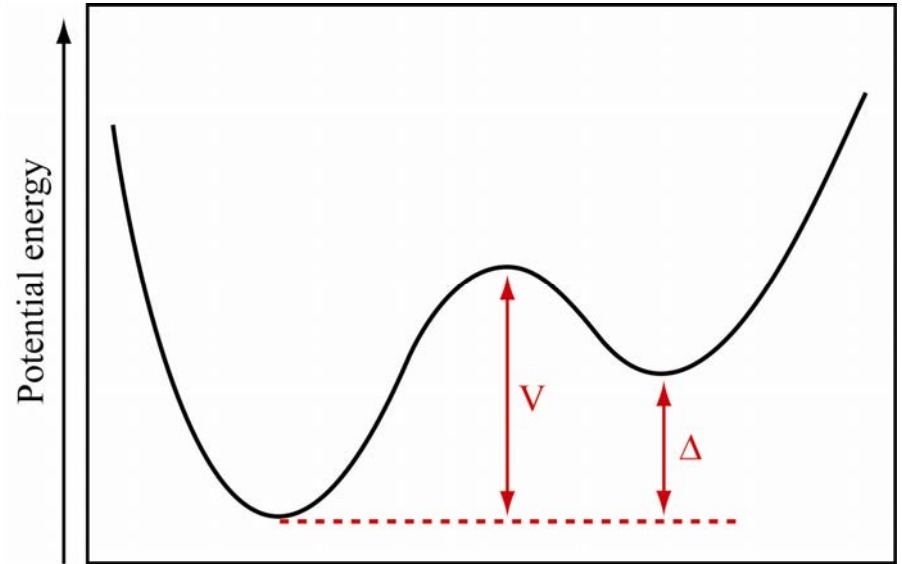


Analysis of coating loss peak

- **Activation energy** associated with the dissipation peak calculated from Arrhenius law
 - (40 ± 3) meV for doped Ta_2O_5
 - (29 ± 2) meV for undoped Ta_2O_5
- The low temperature dissipation peak in **fused silica** has a similar activation energy (44 meV)
- Oxygen atoms undergo thermally activated transitions between two stable bond orientations
- Width of peak thought to be related to the distribution of Si-O bond angles
- Similar dissipation mechanisms may be responsible for loss peak in Ta_2O_5



- Stable Si-O bond orientations represented by an **asymmetric double-well potential** (right)
- **Amorphous structure** gives a distribution $g(V)$ of the potential barrier height
- Calculated activation energy corresponds to the **average barrier height** in this distribution
- Following a similar analysis for silica^{1, 2}, the loss around the peak can be related to the distribution of barrier heights, $g(V)$

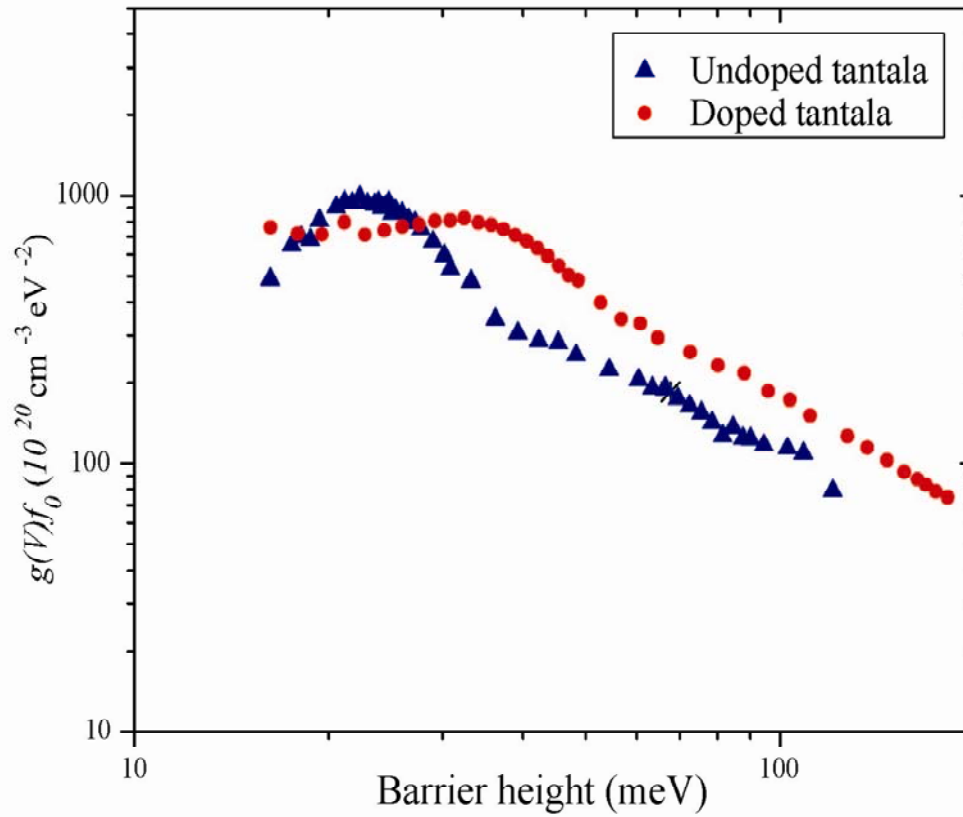


$$\phi = \frac{\pi\gamma^2 f_0}{C_{ii}} k_B T g(V)$$

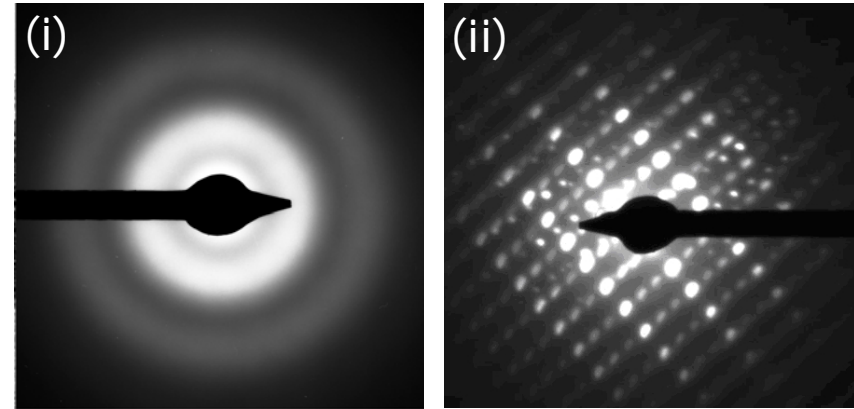
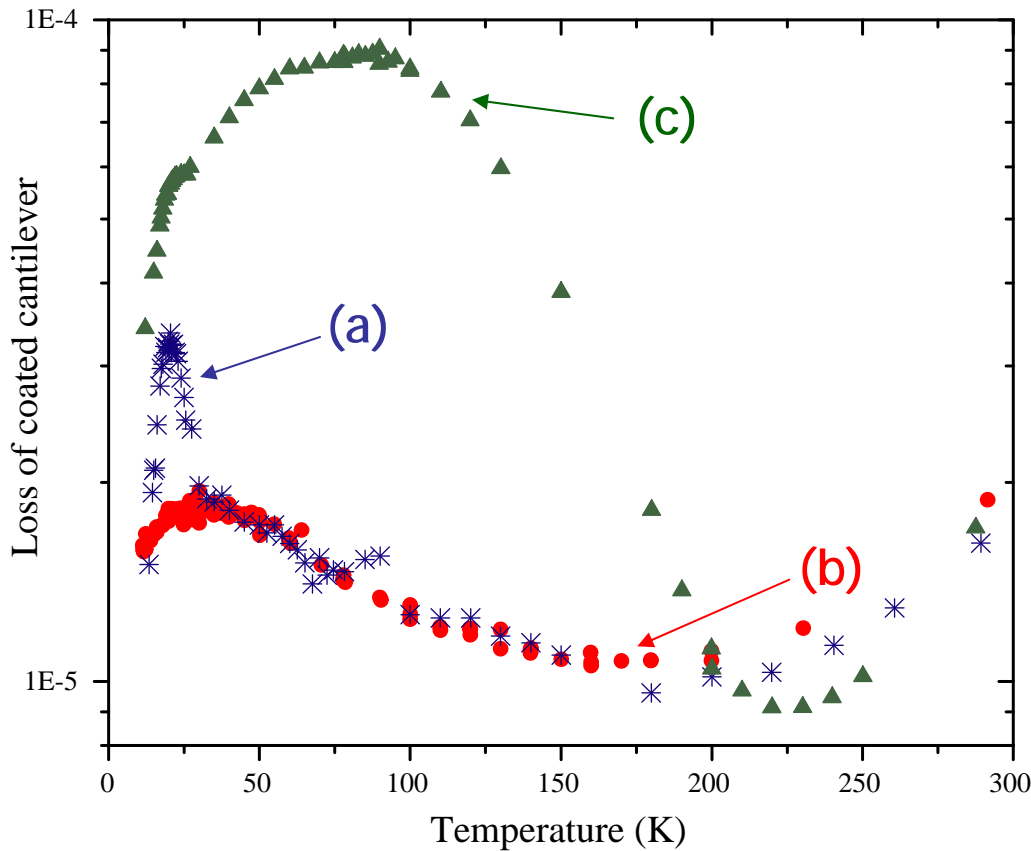
- γ represents the coupling between strain and the dissipation mechanism
- C_{ii} is the elastic constant of the material

ADWP model – barrier height distribution

- Thus the **barrier height distribution function** can be calculated, as shown.
- TiO₂ doping appears to shift distribution to a higher barrier height, and increase width of distribution.
- Other methods of altering the barrier height distribution – perhaps heat treatment?



Effect of annealing temperature

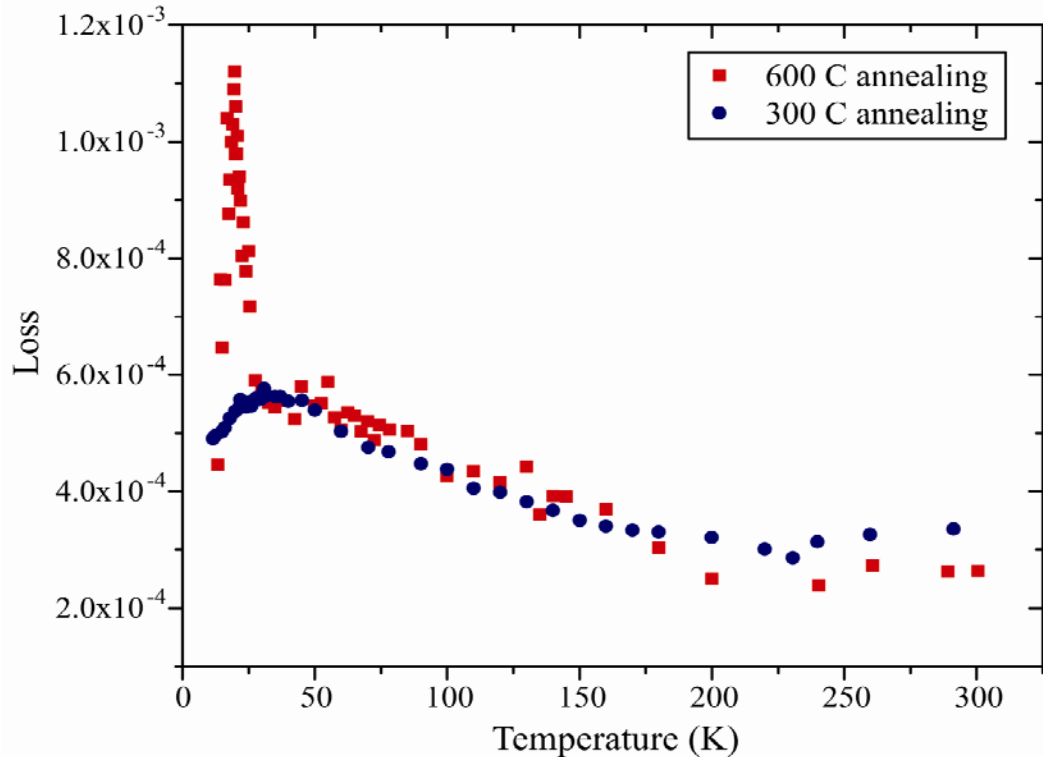


Above: Electron diffraction measurement of Ta_2O_5 annealed at (i) 600°C , showing amorphous structure and (ii) 800°C , showing crystalline structure.

Left: Loss at 1.9 kHz of $0.5\ \mu\text{m}$ Ta_2O_5 coatings annealed at (a) 600°C , (b) 300°C and (c) 800°C

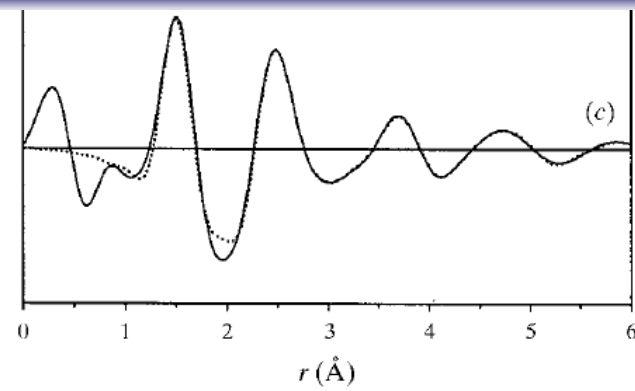
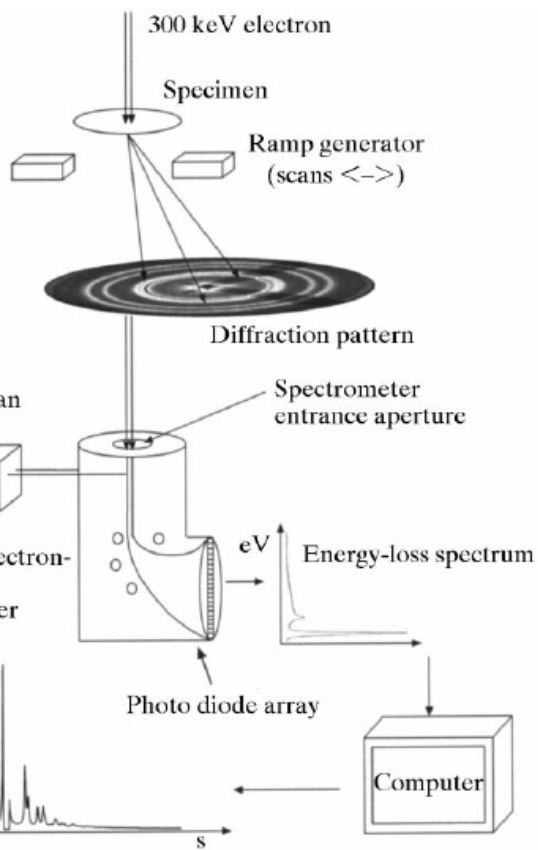
- Large loss peak at ~ 80 to 90 K in un-doped Ta_2O_5 coating (CSIRO) annealed at 800°C , perhaps due to (expected) **onset of polycrystalline structure**
- **Smaller, broader peak** at ~ 35 K in un-doped Ta_2O_5 annealed at 300°C – activation energy $138\ \text{meV}$.

Effect of annealing – coating loss



- 600 C heat treatment reduces loss above 200 K
- Peak at 35 K may also be present in coating heat treated at 600 C, in addition to sharp peak at 20 K
 - Peaks likely to be associated with different loss mechanisms e.g. different molecular transitions in a double-well potential?
 - RDF analysis of TEM electron diffraction patterns underway, result in models of short range structure, aid interpretation of loss data

Electron diffraction radial distribution function studies



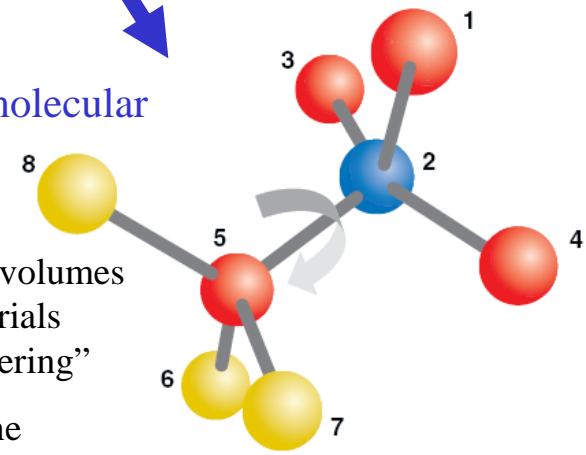
radial distribution function



structural information such as nearest neighbour distances and bond angles



Model of local molecular structure



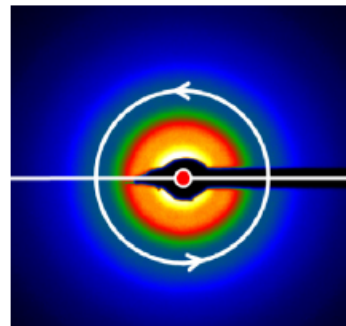
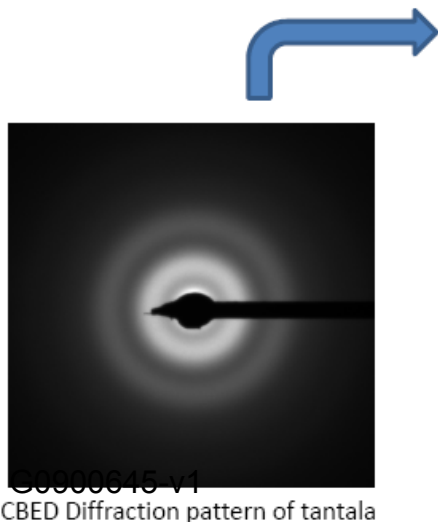
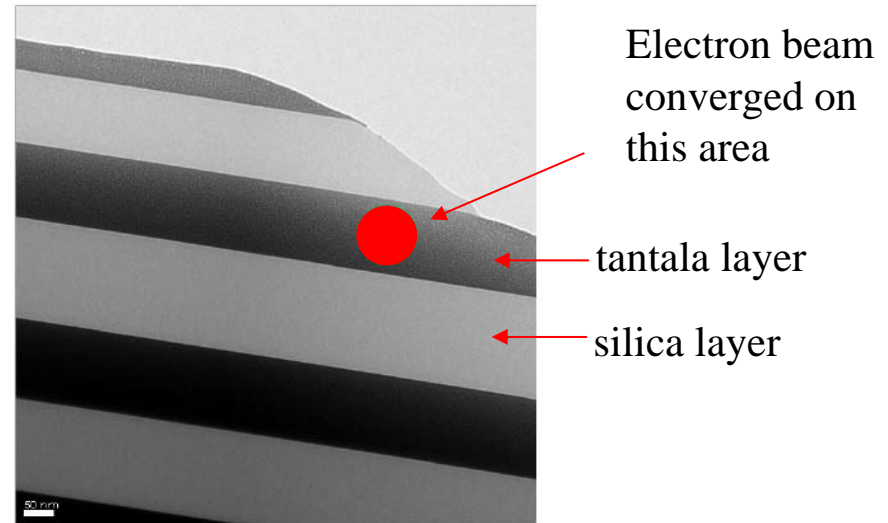
“The study of nanovolumes of amorphous materials using electron scattering”

David J H Cockayne

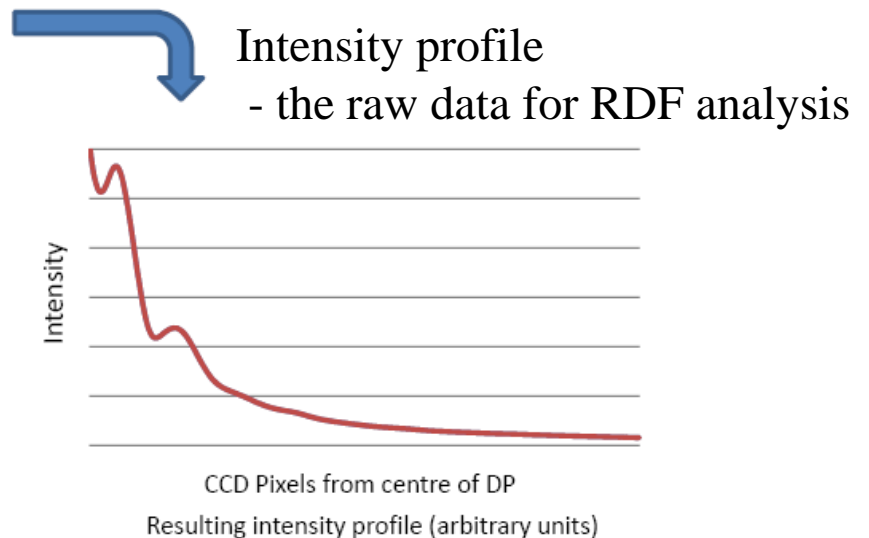
Fig. 1. A schematic diagram of the experimental set-up for the collection of the energy-loss data as a function of momentum transfer in a transmission electron microscope.

Radial distribution function studies

- TEM image of SiO₂/Ta₂O₅ coating (right)
 - Electron beam converged on to a spot of diameter ~ 100 nm (on one layer)
- Diffraction pattern recorded and processed
 - Centre detection
 - Azimuthal averaging

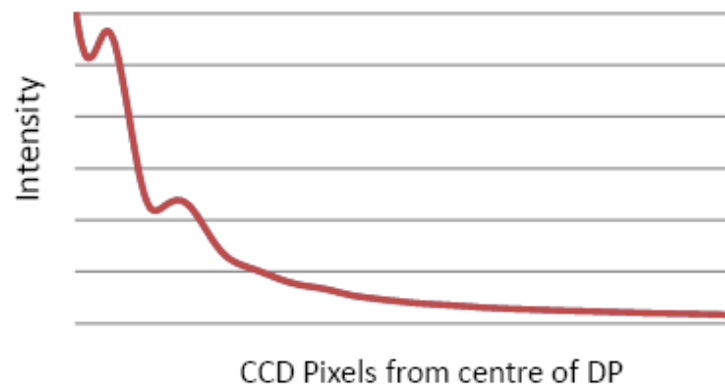


Same CBED image with a colour profile to easily detect diffraction rings and find centre which is then azimuthally averaged



Radial distribution function studies

Intensity profile for tantalum (Riccardo Bassiri)



Tantala intensity profile (arbitrary units)

The intensity profile, $I(q)$ of a diffraction pattern can be described as,

$$I(q) = N f^2(q) + 4\pi N f^2(q) \int_0^{\infty} [g(r) - \rho_0] \frac{r}{q} \sin(qr) dr, \quad (8)$$

where N is the number of atoms, $q = 4\pi \sin\theta/\lambda$, f is the scattering factor (which is obtained from Kirkland fitting parameters [13]) and r is the distance from the central atom. $g(r)$ is defined as pair distribution function and describes the position of two atoms in a homogenous isotropic system. The term, $N f^2(q)$ defines the mean intensity from the atoms scattering independently, and a second term that causes a deviation from the mean intensity as the density ρ_0 varies with r .

Radial distribution function studies

Example RDF (taken from 1st year report)

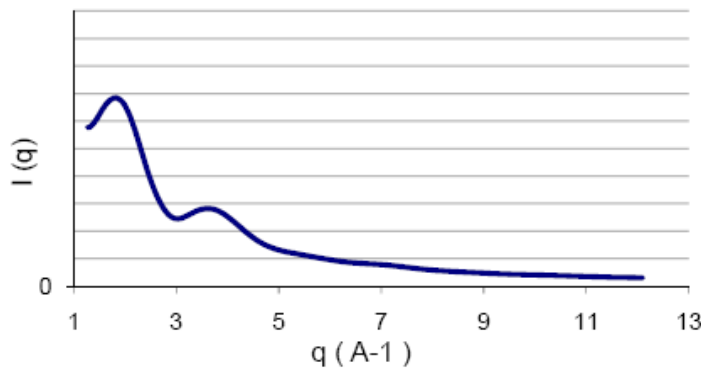
The reduced intensity function is defined as,

$$\varphi(q) = \left[\frac{I(q) - Nf^2(q)}{Nf^2(q)} \right] q,$$

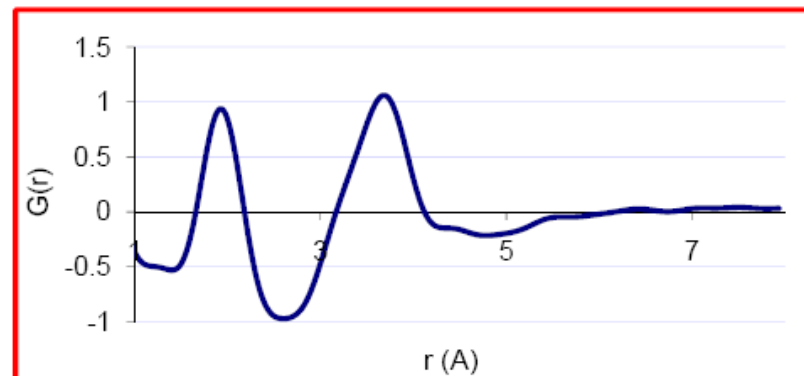
from which we arrive at the reduced density function, $G(r) \equiv 4\pi r[g(r) - \rho_0]$,

$$G(r) = 4 \int_0^{\infty} \varphi(q) \sin(qr) dq. \quad (10)$$

With the above equations [14, 15], it is now possible to obtain $G(r)$ from a diffraction pattern by firstly obtaining $\varphi(q)$ from $I(q)$ and performing the fourier transform on $\varphi(q)$.

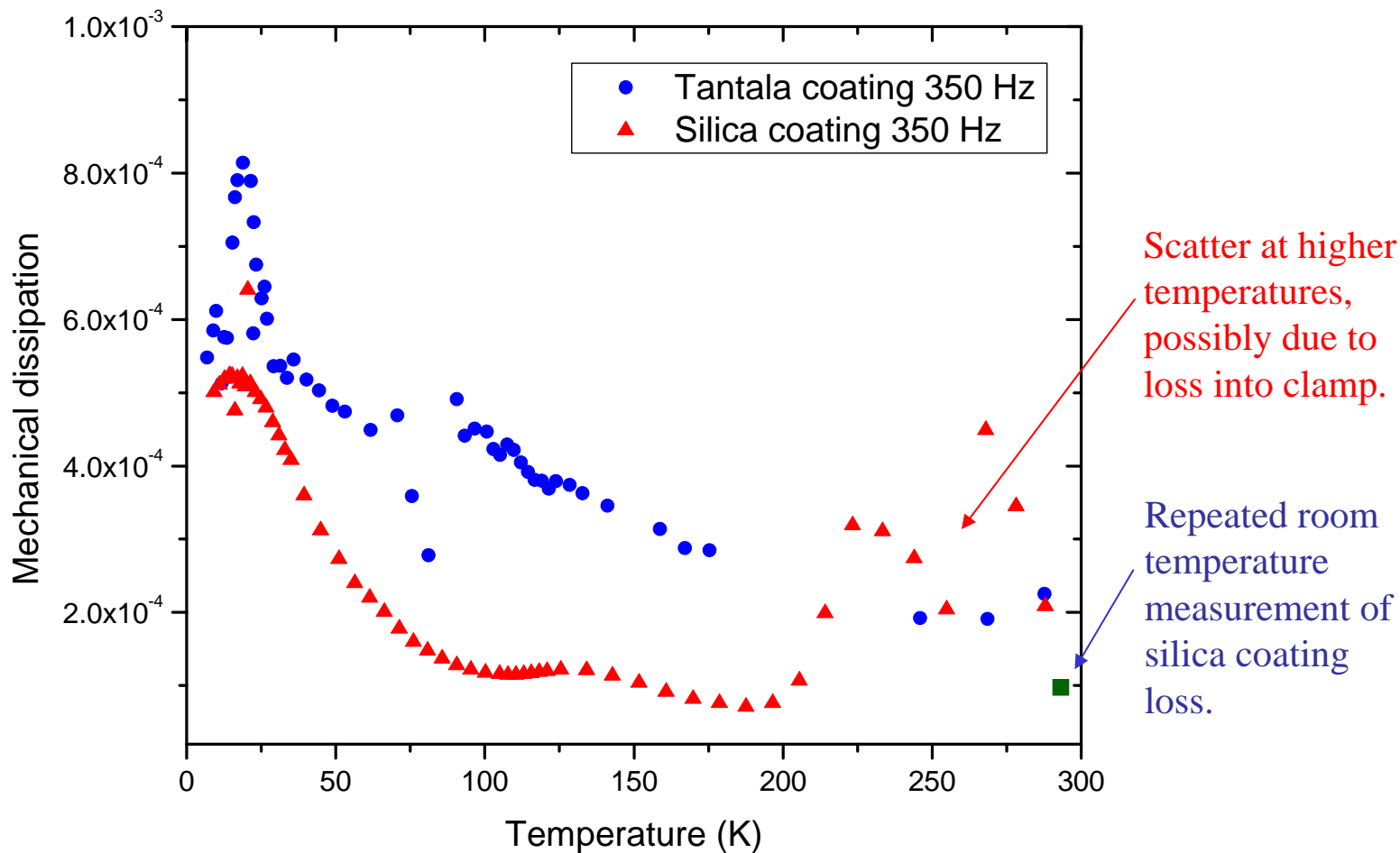


G0900645 Tantalum reduced intensity function



Tantala reduced density function (RDF)

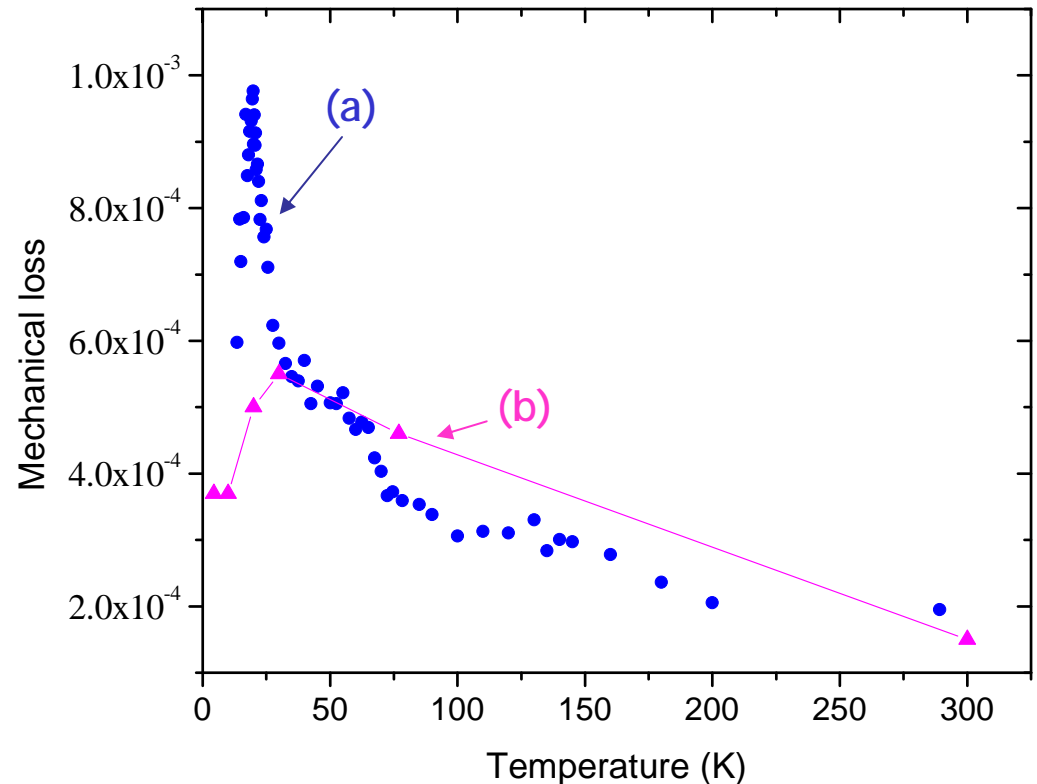
Comparison of SiO₂ and Ta₂O₅



- Loss of single layers of silica and tantala can be used to calculate the expected loss in a multilayer coating.

Comparison to multilayer loss results

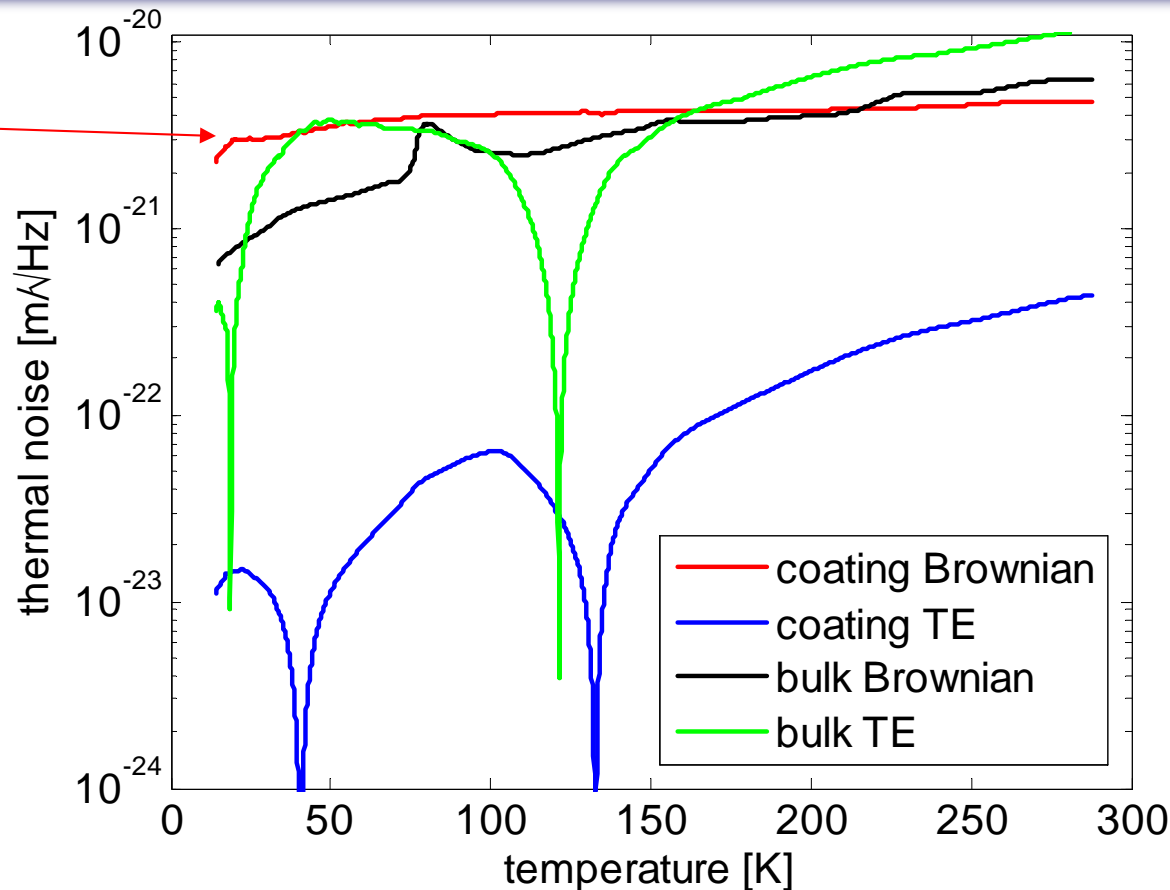
- Loss of single SiO_2 and Ta_2O_5 layers used to calculate loss in a 31 layer multilayer coating, as measured by Yamamoto et al*
- Yamamoto's results:
 - No large peak at 20 K – evidence of a peak at higher temperature
- Difference in results could possibly be explained by:
 - Differences in annealing temperature and / or coating layer thickness?
 - Further study of multilayer coatings of interest



(a) Calculated multilayer coating loss at 1 kHz compared to (b) Yamamoto's measured multilayer loss at 1.1 kHz

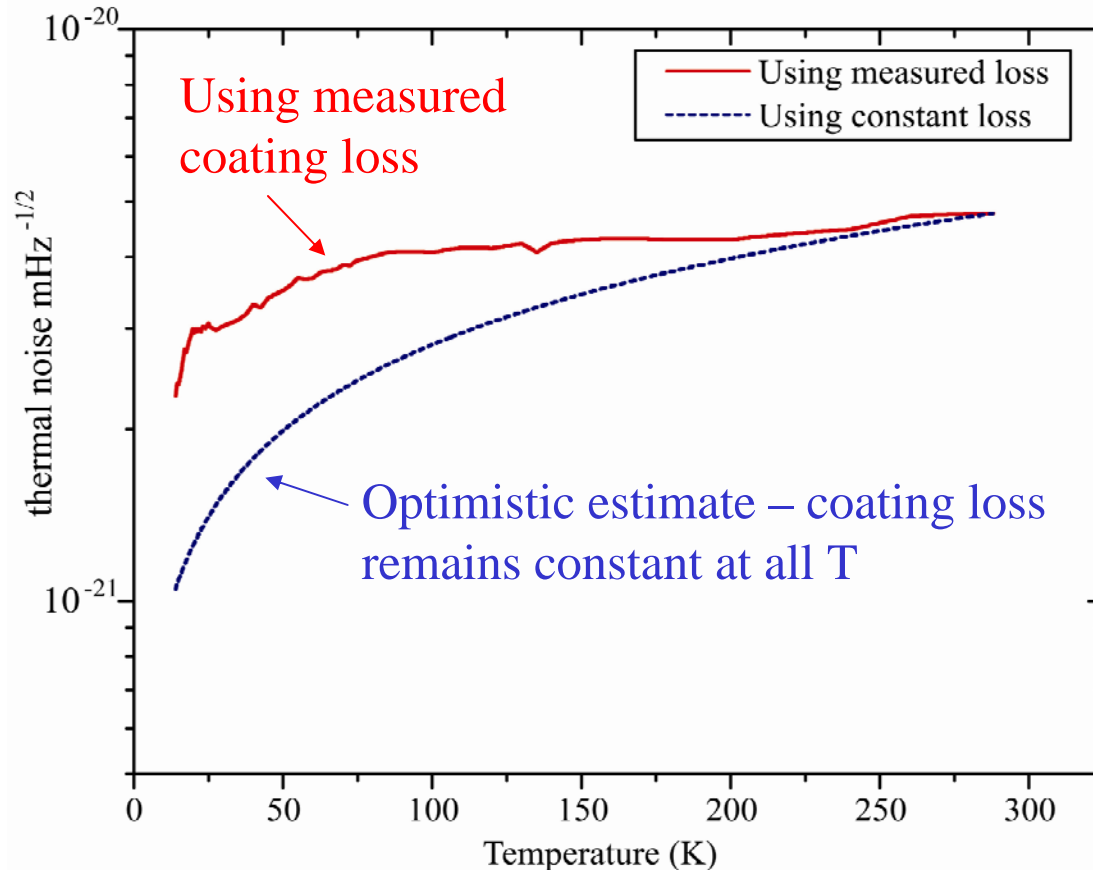
Coating thermal noise at 100 Hz

Small peak due to peak in coating loss



- Coating and bulk thermal noise (thermoelastic + Brownian) for a 30 layer $\text{SiO}_2/\text{Ta}_2\text{O}_5$ coating on a Si (111) substrate of Advanced LIGO geometry
- Coating likely to dominate thermal noise at low temperature (substrate TE also significant 40 – 100 K)

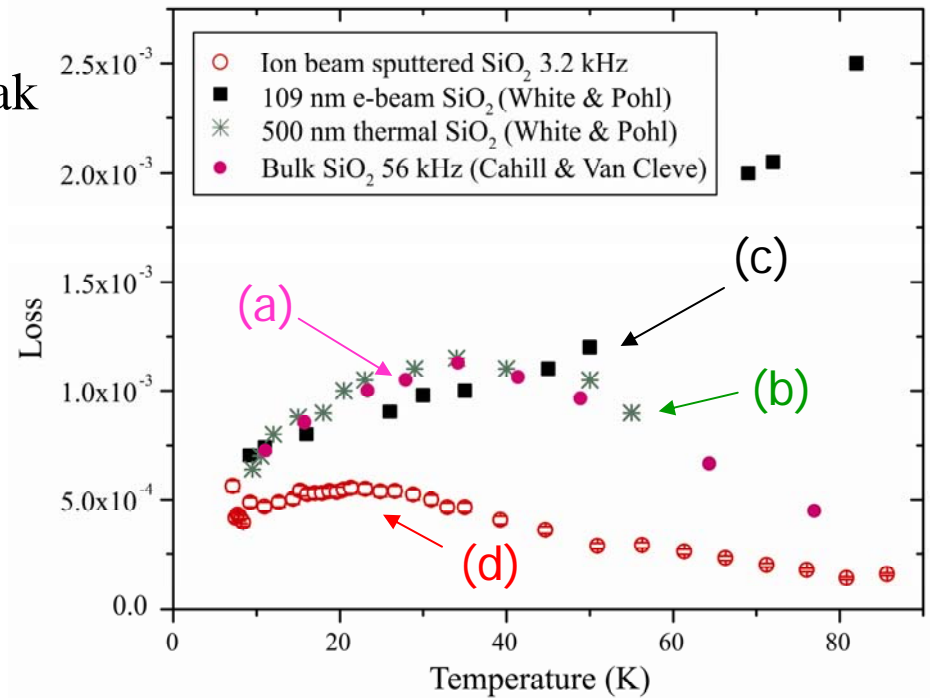
Coating thermal noise at 100 Hz



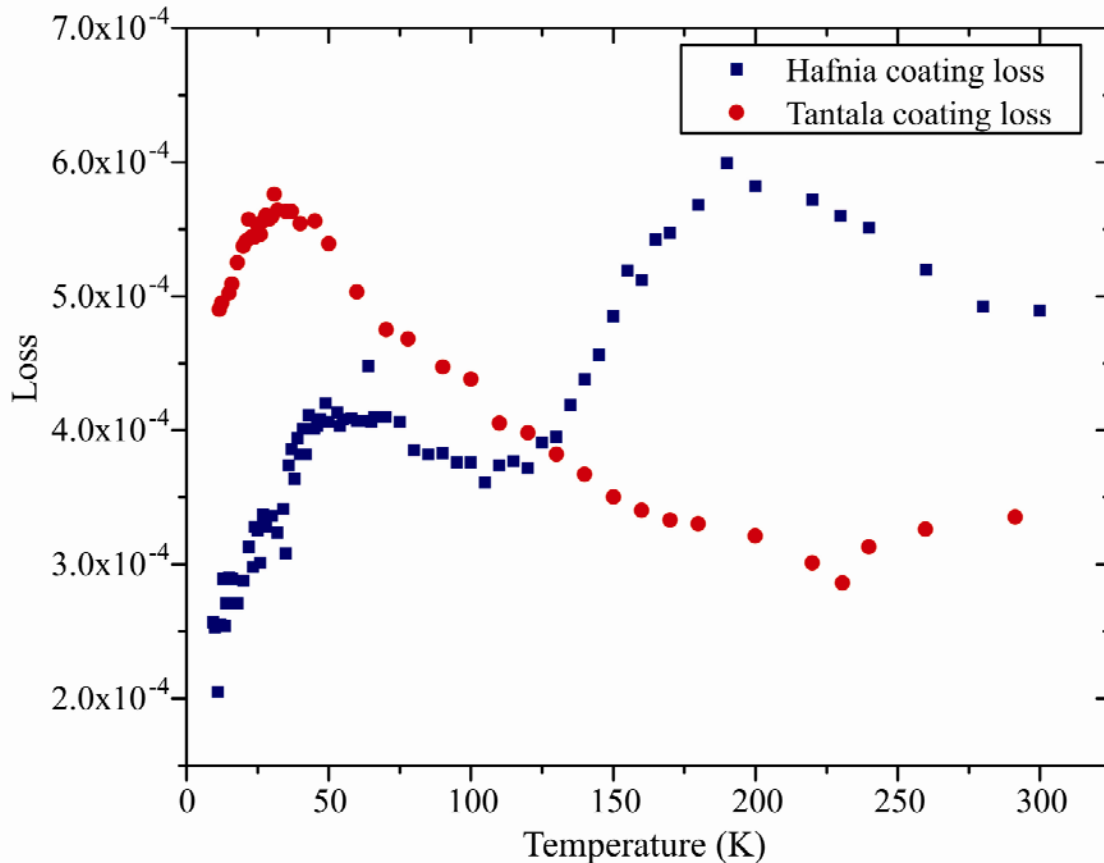
- If coating loss was constant with temperature, could gain factor of ~ 4 in TN noise at 18 K
- Measured coating losses imply we can only gain a factor of ~ 1.7 in coating TN by cooling to 18 K

Silica - comparison of deposition methods

- Bulk silica (a) & thermal oxide (b) grown on silicon have a dissipation peak at ~ 35 K^{1,2}
- e-beam SiO₂ (5.5 kHz)¹ (c)
 - 109 nm thick e-beam film shows no peak at 35 K
 - Higher loss than bulk and thermal oxide above 40 K
- Ion beam sputtered silica (d)
 - Broad loss peak at ~ 23 K
 - Significantly lower loss and different temperature dependence to similar thickness of e-beam SiO₂
- Deposition method has a significant effect on mechanical loss – studies of different method (loss + RDF analysis) planned



Alternative high index materials – hafnia?



Coating loss at ~ 1kHz for HfO_2 and Ta_2O_5 heat treated at 300°C. (E. Chalkley)

- Hafnia loss significantly lower below 125 K
- Two loss peaks at 50 K and 190 K
- Study of the effect of heat treatment on loss + microscopic structure planned
- **Material properties** for thin-film hafnia are **not well studied** – results depend on Young's modulus

- Initial **absorption measurement** of a $\text{SiO}_2/\text{HfO}_2$ coating by Markosyan et al. (Stanford) gave 60-80ppm, considerably higher than required. Further investigation required.

Other coatings research

Density

- Ellipsometric layer thickness measurements
- Coating removed by HF etch
- Initial density measurement of $7675 \pm 461 \text{ kg m}^{-3}$ for LMA undoped ion beam sputtered tantala

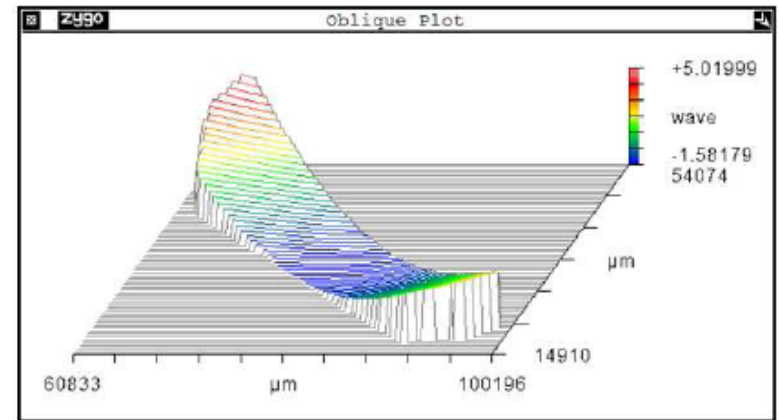


microbalance
 $\Delta m = \pm 10 \mu\text{g}$

Young's modulus measurements

- Measurements by nano-indentation with AFM
- Measure change in radius of curvature when a coated cantilever is heated, using Zygo interferometer

G0900645-v1



Zygo interferometer image of a coated cantilever

Conclusions

- Loss peaks found in amorphous tantala, at 35 K (coating annealed at 300 C) and at 20 K (coating annealed at 600 C)
- TiO₂ doping (14.5 %) **increases the activation energy** of the 20 K dissipation peak in Ta₂O₅ by ~ 30 %
 - Possibly due to a shift in the distribution of ADWP barrier heights to higher energy
- 35 K loss peak may be present in both coatings, peaks possibly associated with different dissipation mechanisms – further analysis required
- Preliminary results of the mechanical loss of hafnia show lower levels of loss than tantala below ~100 K
 - Optical properties and Young's modulus require further investigation
- Studies of short range structure with RDF analysis of electron diffraction measurements are in progress
- **Very low operating temperatures (~ 10 K), new or improved coating materials or a reduction of the total coating thickness** may be attractive to gain a significant reduction in coating thermal noise for 3rd generation detectors