



Effect of Heat of Treatment on the Optical Absorption of Dielectric Coatings: effect of temperature and Oxygen pressure

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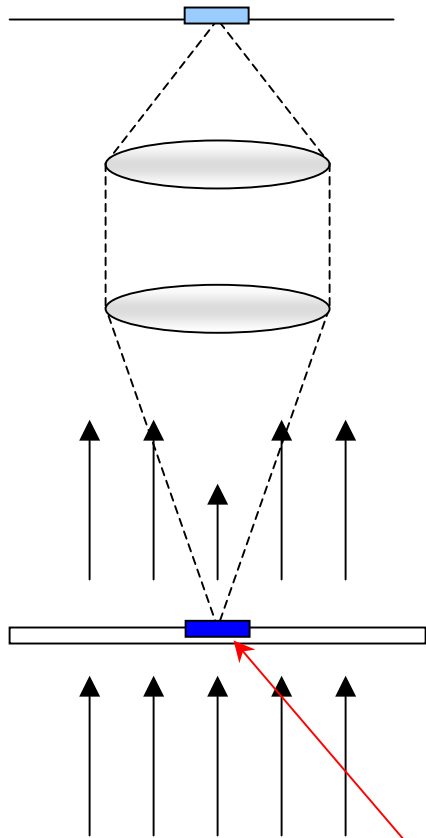
INTRODUCTION

The specifications for optical absorption losses of Advanced LIGO optics, of 0.5 ppm level, can now be reached using different techniques^{1,2}, among which ion-beam sputtering is anticipated as the most suitable for high-damage threshold and low loss dielectric optical coatings. The current challenge for AdLIGO optics is the optimization of the other parameters, like mechanical/elastic noises, spurious charge build-up, etc.

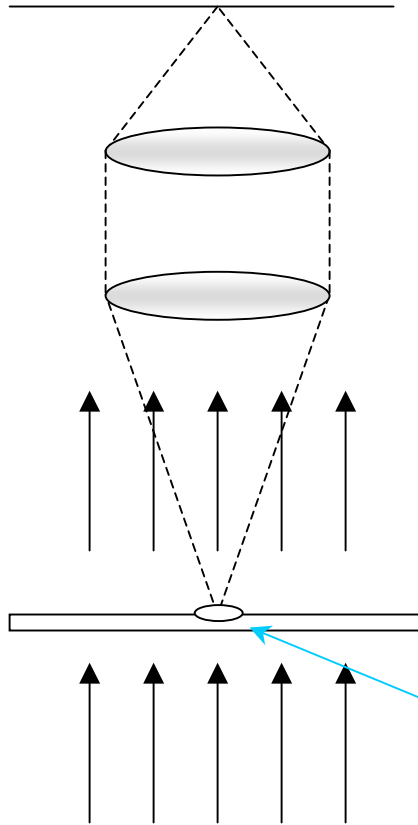
With this aim, the Stanford and Caltech teams in collaboration with CSIRO have been investigating the optical and mechanical properties of various coatings, both single- and multilayer, related to the LIGO AR and HR coating “recipes”.

This poster summarizes the results on the optical absorption study of coatings deposited at different conditions as well as the effect of post-annealing.

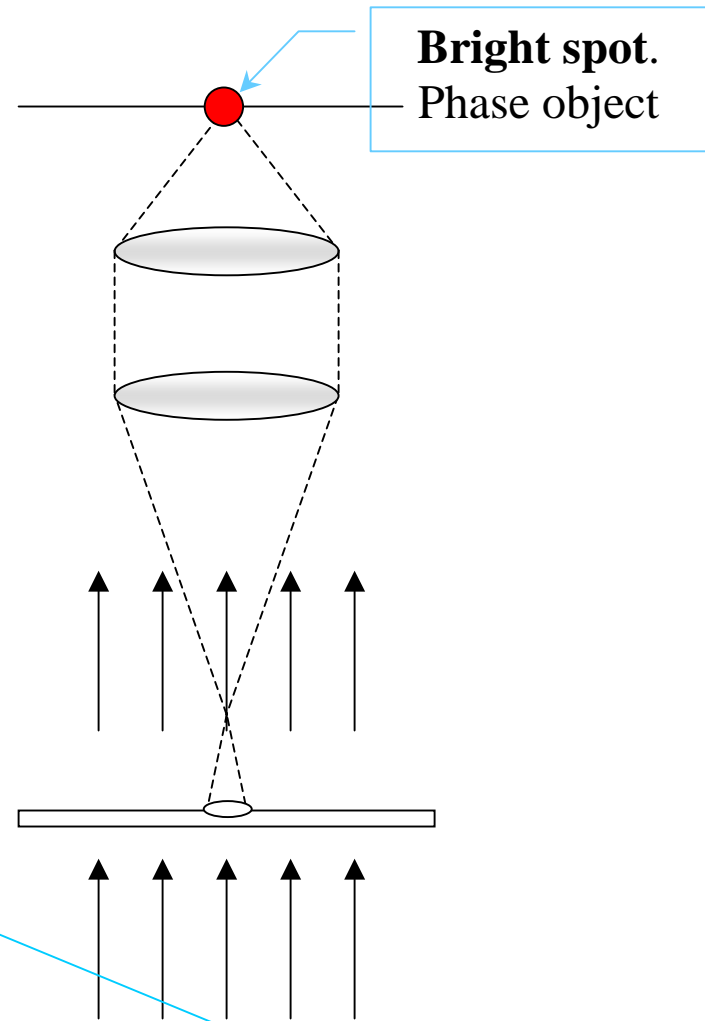
The PCI Method (idea of thermal lensing)



Absorptive material: amplitude object. Contrast image



Transparent lens: phase object.
No amplitude change, no contrast, no image



Bright spot.
Phase object

Physical Principles

The probe phase is distorted due to heating:
$$\Delta\varphi = \left(\frac{2\pi}{\lambda}\right) \times L \times \frac{\partial n}{\partial T} \times \Delta T$$

$\Delta\varphi$ transforms into an intensity distortion

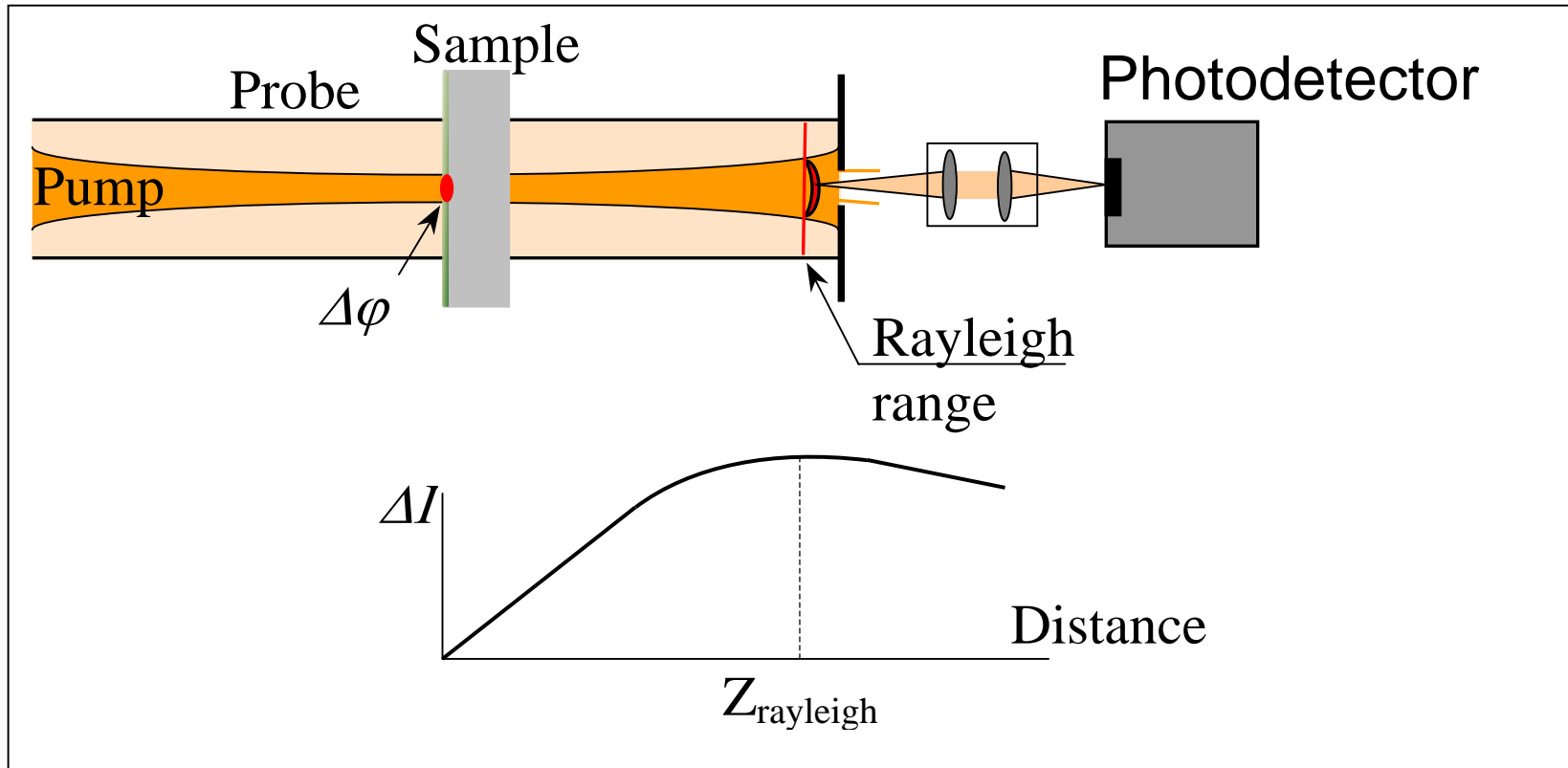
(maximum at the Rayleigh length):
$$\Delta I/I \propto \Delta\varphi \ll \pi$$

The PCI signal $\Delta I/I$, for a pump beam of power W_P and waist w_0 “chopped” at a frequency f , can be written as:

$$\boxed{\frac{\Delta I}{I} \propto B = \left(\frac{L}{2\lambda}\right) \times \left(\frac{\alpha}{\kappa}\right) \times \left(\frac{\partial n}{\partial T}\right) \times \left(\frac{W_P}{w_0^2}\right) \times f^{-1}}$$

where L is the interaction length, α is the thermalized absorption loss, and κ stands for the thermal conductivity.

PCI method (how it works)



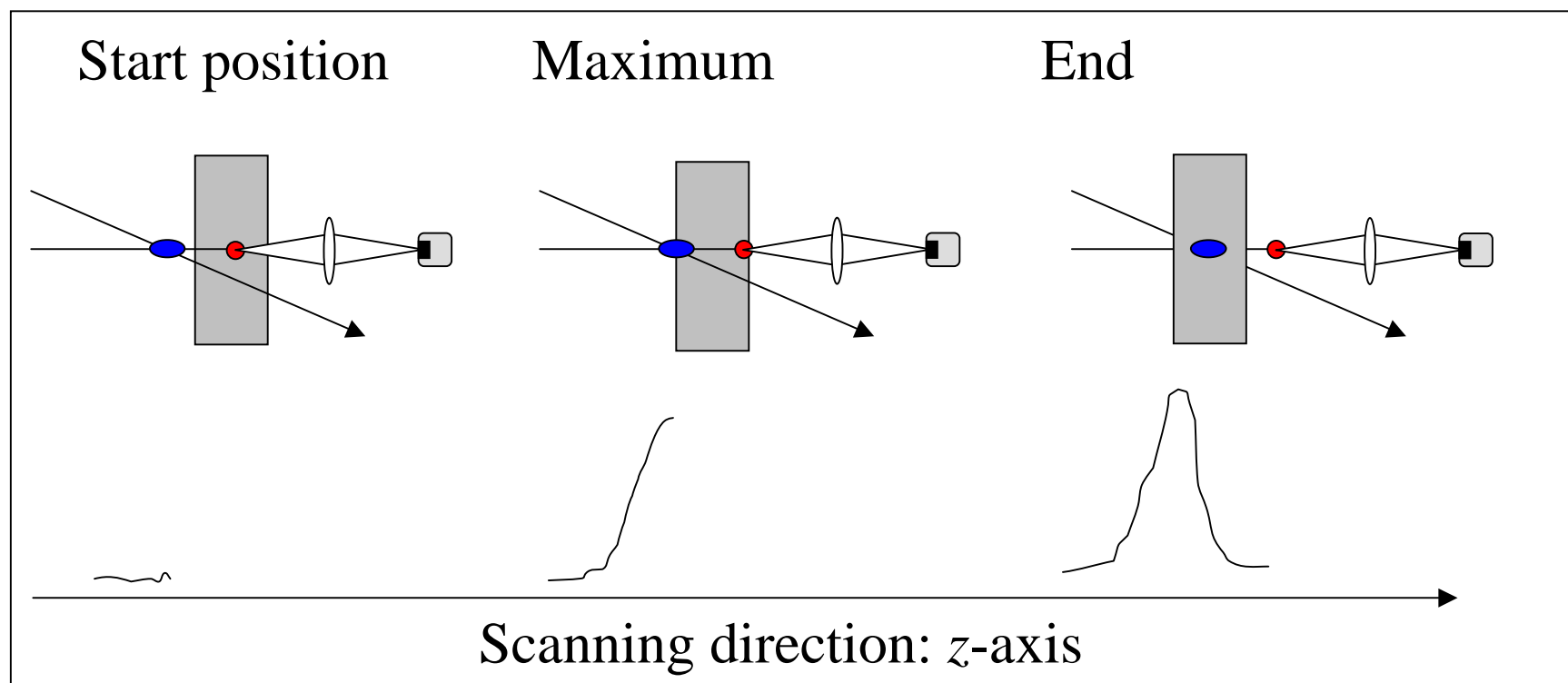
$w_{\text{pump}} < w_{\text{probe}}$. When the pump is on, the probe experiences phase distortion ($\Delta\phi$) in the heated area. At some distance, $\Delta\phi$ transform into ΔI and an effective interference occurs between the central spot (distorted) and outer ring area (undistorted) of the probe

A small angle, $\leq 7^\circ$, between the probe and pump beams allows localization of the heated area and isolation of the probe output in space.

Sensitivity: **better than 0.1 ppm** (surface absorption)

better than 0.2 ppm / cm (bulk absorption)

Optical loss measurement scheme for surface absorption

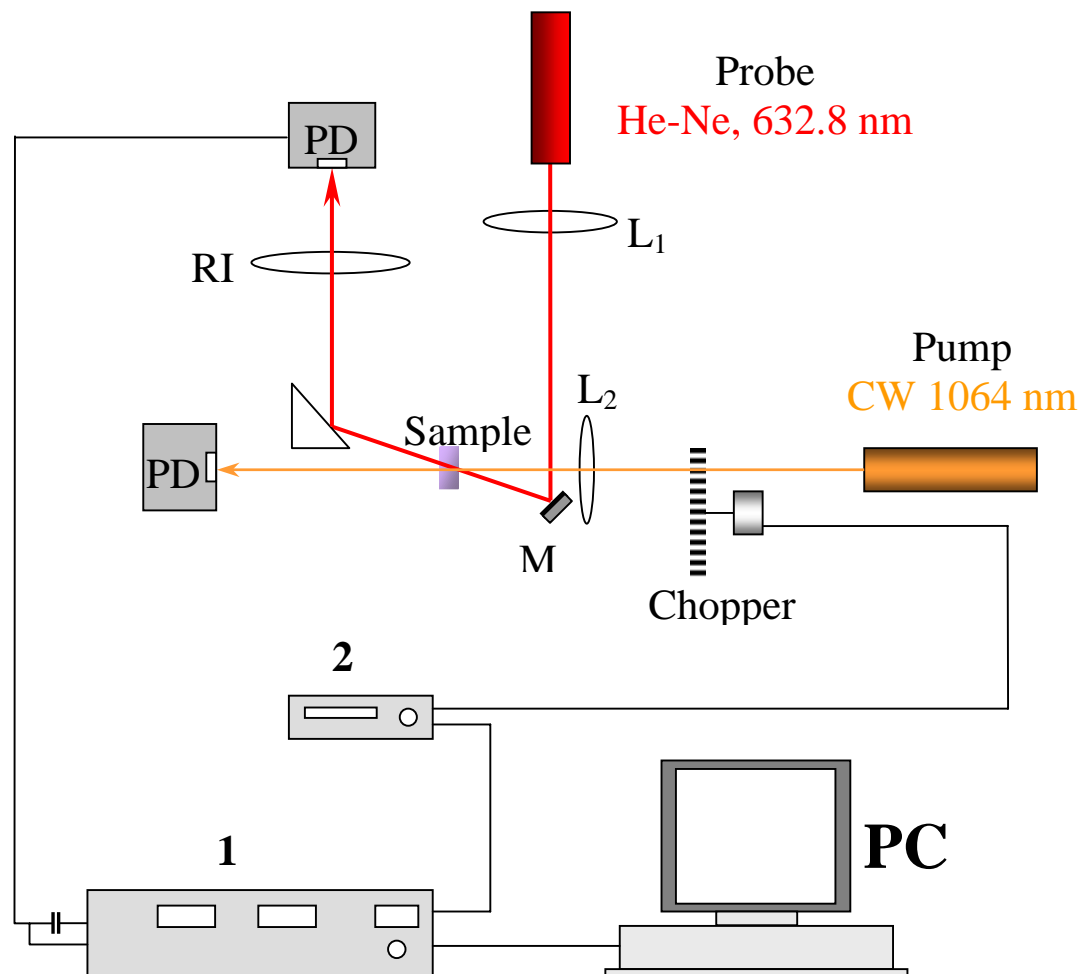


Blue: the position where the pump and probe beam cross:

Red: the position where the imaging system collects the signal from.

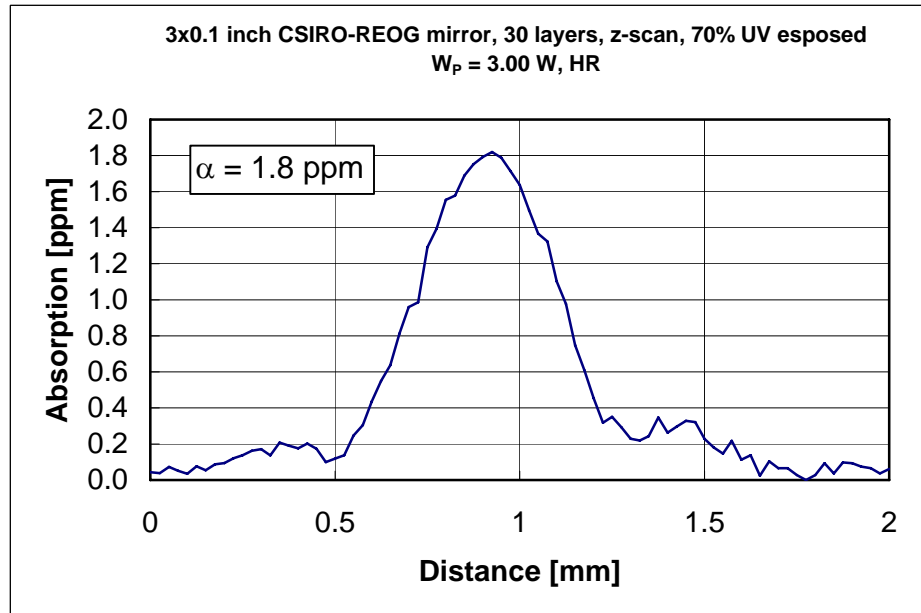
PCI setup

- RI** Re-imaging System
PD Photodetectors
L_i Lenses
M Mirror
1 Lock-in Amplifier
SR830 DSP
2 Chopper Controller
SR340
PC with GPIB-connection
(LabVIEW software)

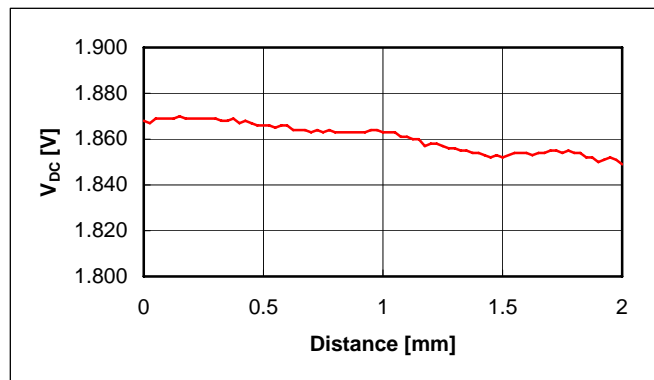


The probe wave (632 nm) with a larger waist (90 μm) senses the lensing effect of the pump (1064 nm) with a smaller waist (35 μm). Interference occurs between different parts of the probe wave due to phase mismatch.

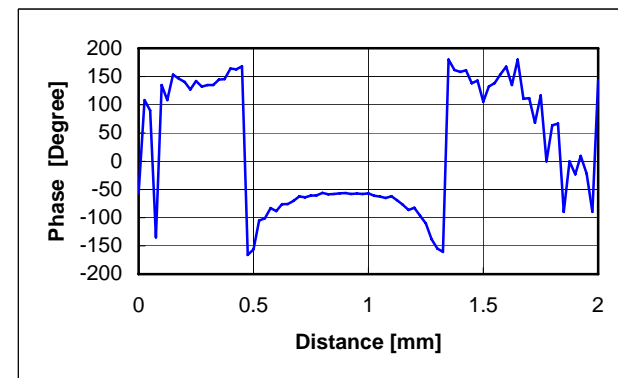
The PCI signal and additional monitored parameters



**Intensity of the AC PCI signal,
AC signal \propto to absorption**



DC signal from probe $\propto W_{probe}$



**Phase delay of the AC signal
(characterizes the substrate material)**

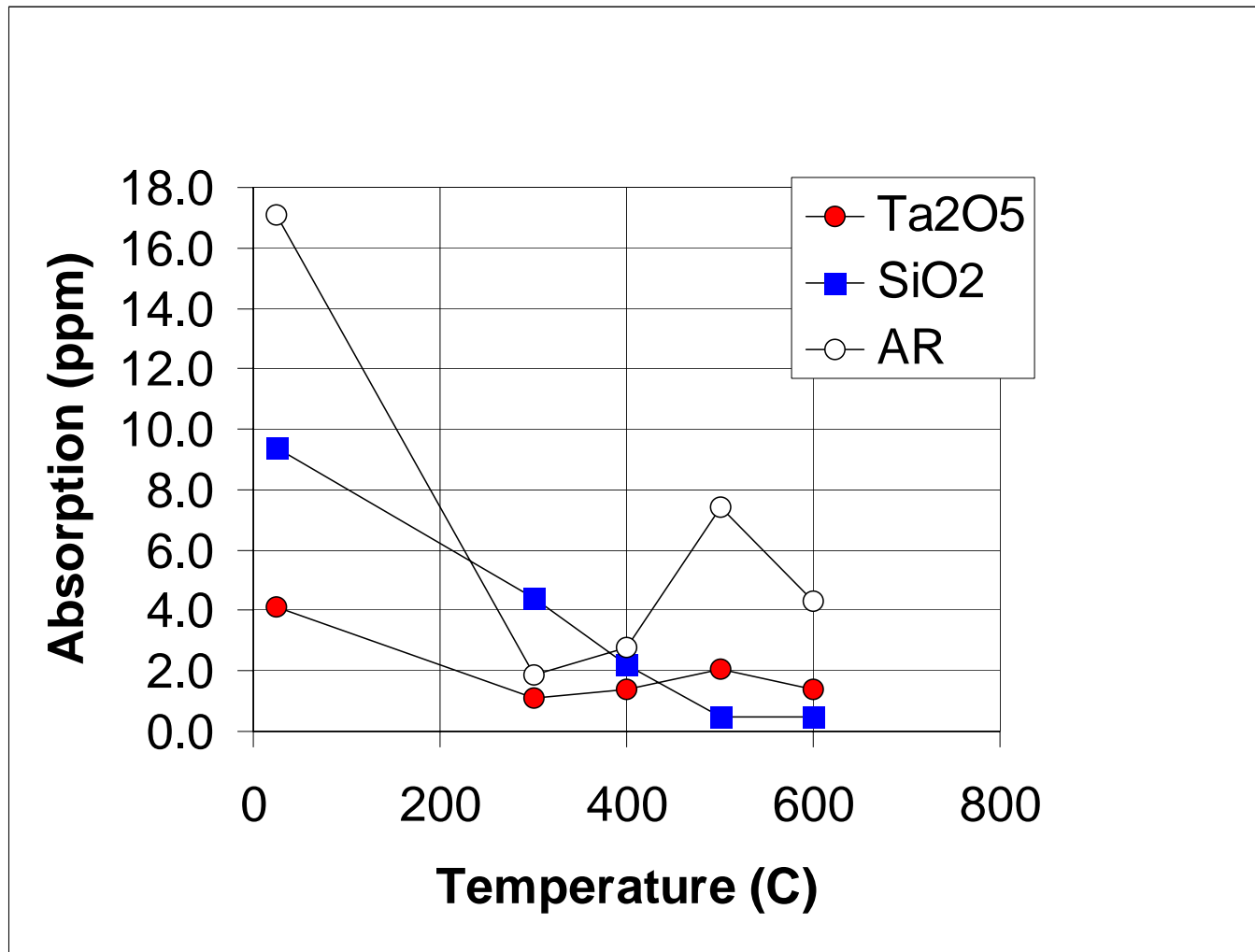
1) Monolayer coatings

| Sample | Thickness (nm) | Absorption (ppm) |
|--|-----------------------|-------------------------|
| Ta ₂ O ₅ | 500 | 1 to 4 ppm |
| SiO ₂ | 500 | <0.5 to 9 ppm |
| Nb ₂ O ₅ | 500 | 25 to 40 ppm |
| Ta ₂ O ₅ /SiO ₂ | 48/238 | 1.85 to 17 ppm |

Annealing effect

| Sample | Alpha before (ppm) | Alpha after (ppm) | Annealing regime |
|--|---------------------------|--------------------------|-------------------------|
| Ta ₂ O ₅ | 4.1 | 1.15 | 350, 24 hrs |
| SiO ₂ | 9.4 | 3.1 | 350, 24 hrs |
| Ta ₂ O ₅ /SiO ₂ | 17.1 | 2.4 | 350, 24 hrs |
| Ta ₂ O ₅ | 1.9 | 1.65 | 600, 19 hrs |
| Ta ₂ O ₅ /SiO ₂ (AR) | 4.3 | 4.05 | 600, 19 hrs |

The goal of this 1st phase annealing was the study of the stress removal effect.



As seen, low-temperature annealing, 300 to 350°C, is more efficient. The increase of the optical absorption at higher temperatures is possibly due to the growth of crystallites.

2) Double-layer AR coatings (oxygen effect)

Tantala/Silica double layers (~41 nm Ta₂O₅/241.4 nm SiO₂) were deposited at different oxygen excess in chamber, 25, 33, and 40 cm³, and were annealed subsequently at temperatures varying from 350 to 550°C in vacuum or air.

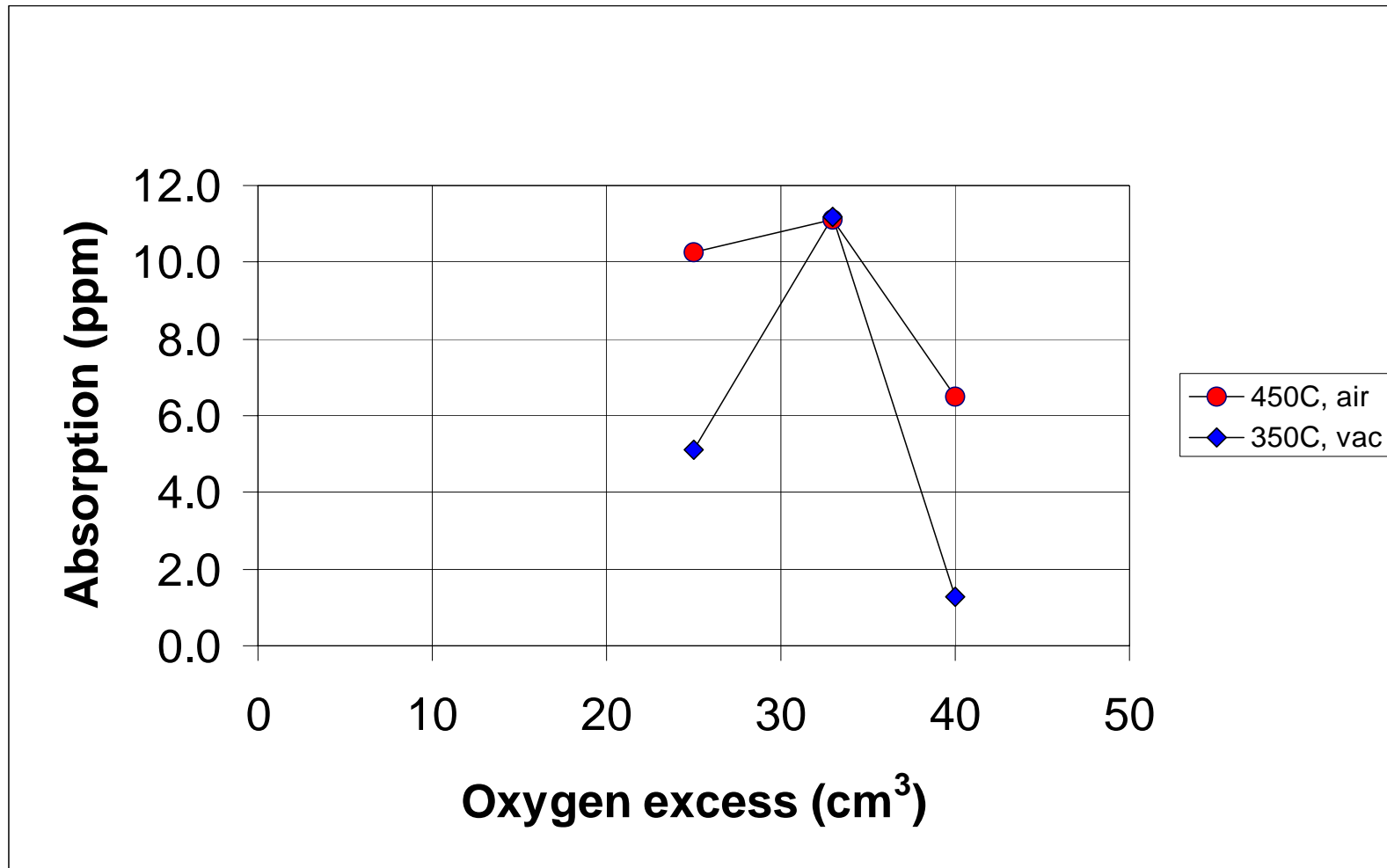
| Oxygen 40 cc in chamber | | |
|--------------------------------|------------------|--------------------|
| Sample # | Annealing | Alpha (ppm) |
| 21 | None | 4.5 |
| 22 | 450 air | 6.5 |
| 23 | 550 air | 8.05 |
| 24 | 350 vac | 1.28 |
| 25 | 450 vac | 8.55 |
| 26 | 550 vac | 15.65 |

| Oxygen 33 cc in chamber | | |
|--------------------------------|------------------|--------------------|
| Sample # | Annealing | Alpha (ppm) |
| 28 | None | 12.3 |
| 29 | None | 10.6 |
| 31 | 450 air | 11.1 |
| 32 | 550 air | 3.05 |
| 33 | 350 vac | 11.2 |
| 34 | 450 vac | 14.2 |

| Oxygen 25 cc in chamber | | |
|--------------------------------|------------------|--------------------|
| Sample # | Annealing | Alpha (ppm) |
| 35 | None | 16.75 |
| 36 | 450 air | 10.25 |
| 37 | 550 air | 9.95 |
| 38 | 350 vac | 5.1 |
| 39 | 450 vac | 12.7 |
| 40 | 550 vac | 14.05 |

The trends are

- 1) Annealing in air is better than in vacuum;**
- 2) Annealing temperature must be below 450C.**
- 3) Oxygen excess 40 cc in chamber is better in general**



Variation of the optical absorption of two AR Ta₂O₅/SiO₂ coatings versus oxygen excess content in the chamber for depositions in air and vacuum.

2) Double-layer AR coatings (effect of substrate temperature)

Tantala/Silica double layers (~41 nm Ta₂O₅/241.4 nm SiO₂) were deposited at different target temperature. Oxygen pressure was slightly different for SiO₂ (35 cm³) and Ta₂O₅ (49 and 45 cm³). Samples were annealed subsequently at 450°C in 0.2 atm O₂ .

| Sample # | Deposition Temp (C) | Absorption (ppm) | Notes |
|-----------------|----------------------------|-------------------------|-----------------------------------|
| 85 | 50 | 1.6 | |
| 93 | 100 | 11.8 | uv-ozone cleaned substrate |
| 67 | 100 | 9.55 | |
| 78 | 100 | 13.8 | uv-ozone cleaned substrate |
| 80 | 100 | 10.9 | |
| 68 | 125 | 9.75 | |
| 90 | 125 | 13.15 | |

Conclusion: lower the target temperature lower the optical absorption

Summary of the Results

The deposition and post deposition conditions are optimized for monolayer and AR (1064 nm) coating using the ultra-low optical absorption measurements PCI setup.

The trends favorable for lower optical absorption are:

- 1) Lower deposition target temperature (50°C)
- 2) Slightly oxygen enriched atmosphere with respect to air (25 cm³ in SCIRO IBS chamber)
- 3) Moderate stress releasing post-annealing temperature (300 to 350°C)

References

¹*Handbook of Deposition Technologies for Films and Coatings*, R.F. Bunshah (ed), William Andrew Publishing (1994).

²*Thin Solid Films* **260** (1995) 86, N. Kaiser, H. Uhlig, et al.

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