

# COATING RESEARCH & DEVELOPMENT @ LMA

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in collaboration with:

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# Outline

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1. Investigation of Multilayer-Coating Loss
2. Updates on Atomic Layer Deposition

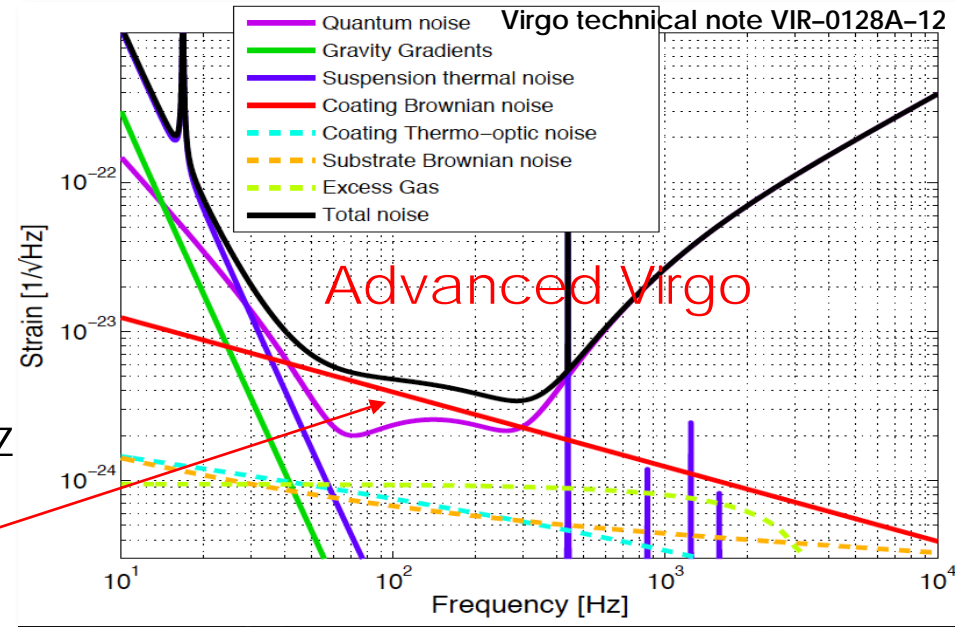
# Investigation of Multilayer-Coating Loss

in collaboration with T. Epicier & B. Van de Moortèle, *CLYM*

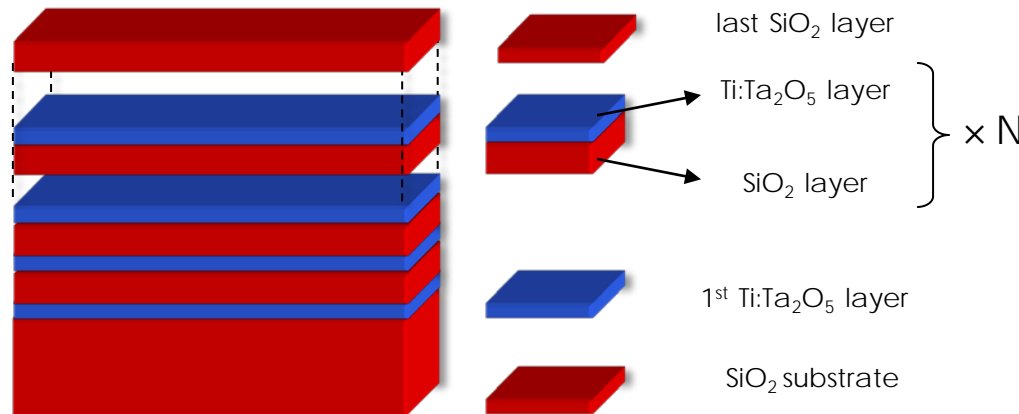


# Thermal Noise of Coatings

the sensitivity gravitational-wave interferometers of 2nd and 3rd generation will be **limited by coating thermal noise** around  $10^2$  Hz



coatings are multilayer stacks realized through Ion Beam Sputtering (IBS)



$\text{Ti:TaO}_2\text{O}_5$ : HIGH (H) refractive index

$\text{SiO}_2$ : LOW (L) refractive index

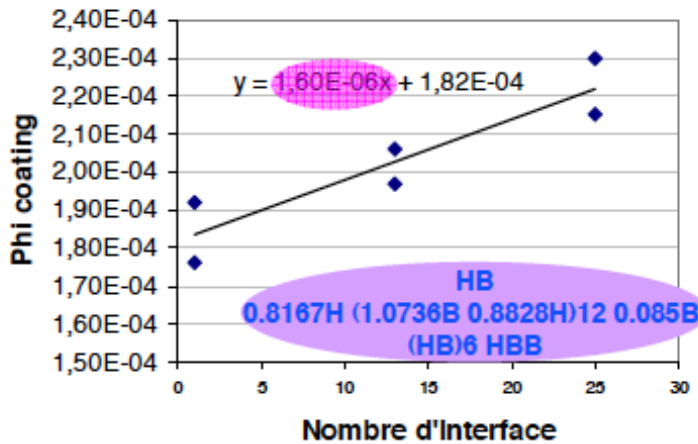
# The Story So Far



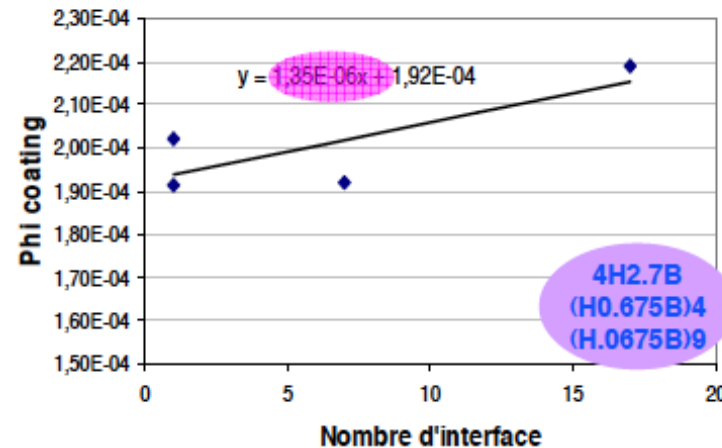
## Mechanical losses: multi-layers



### Dependence of coating multi-layers losses vs number of interfaces



Ratio Ti:Ta<sub>2</sub>O<sub>5</sub>/SiO<sub>2</sub> ~ 0.6-0.7



Ratio Ti:Ta<sub>2</sub>O<sub>5</sub>/SiO<sub>2</sub> ~ 1

- Estimate contribution to mechanical losses due to interfaces
- Deduce losses of Ti:Ta<sub>2</sub>O<sub>5</sub> and SiO<sub>2</sub> from multi-layers
  - Ti:Ta<sub>2</sub>O<sub>5</sub> ~ 2.4 ± 0.2 10<sup>-4</sup>; SiO<sub>2</sub> ~ 1.3 ± 0.4 10<sup>-4</sup>
- Even if error is large, losses of silica remain significantly larger than what is measured on monolayers. Why?
  - Error in the measurement?
  - Silica behaving differently when stacked?

credits: R. Flaminio

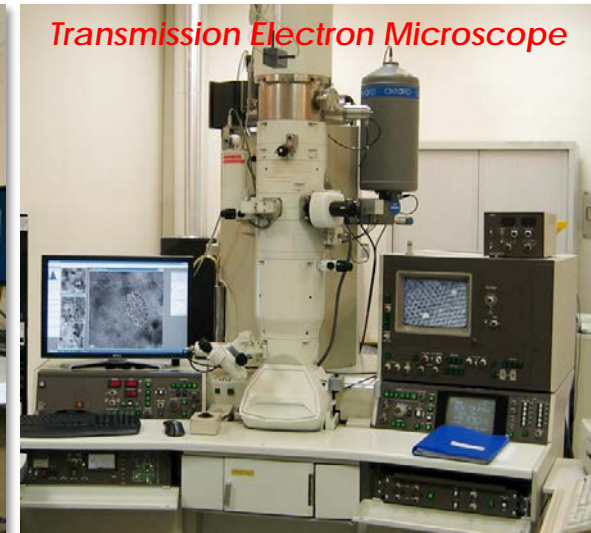
Amaldi 9, Cardiff, 2011

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# Later Developments

physico-chemical characterization of  $\text{SiO}_2/\text{Ti:Ta}_2\text{O}_5$  interfaces:

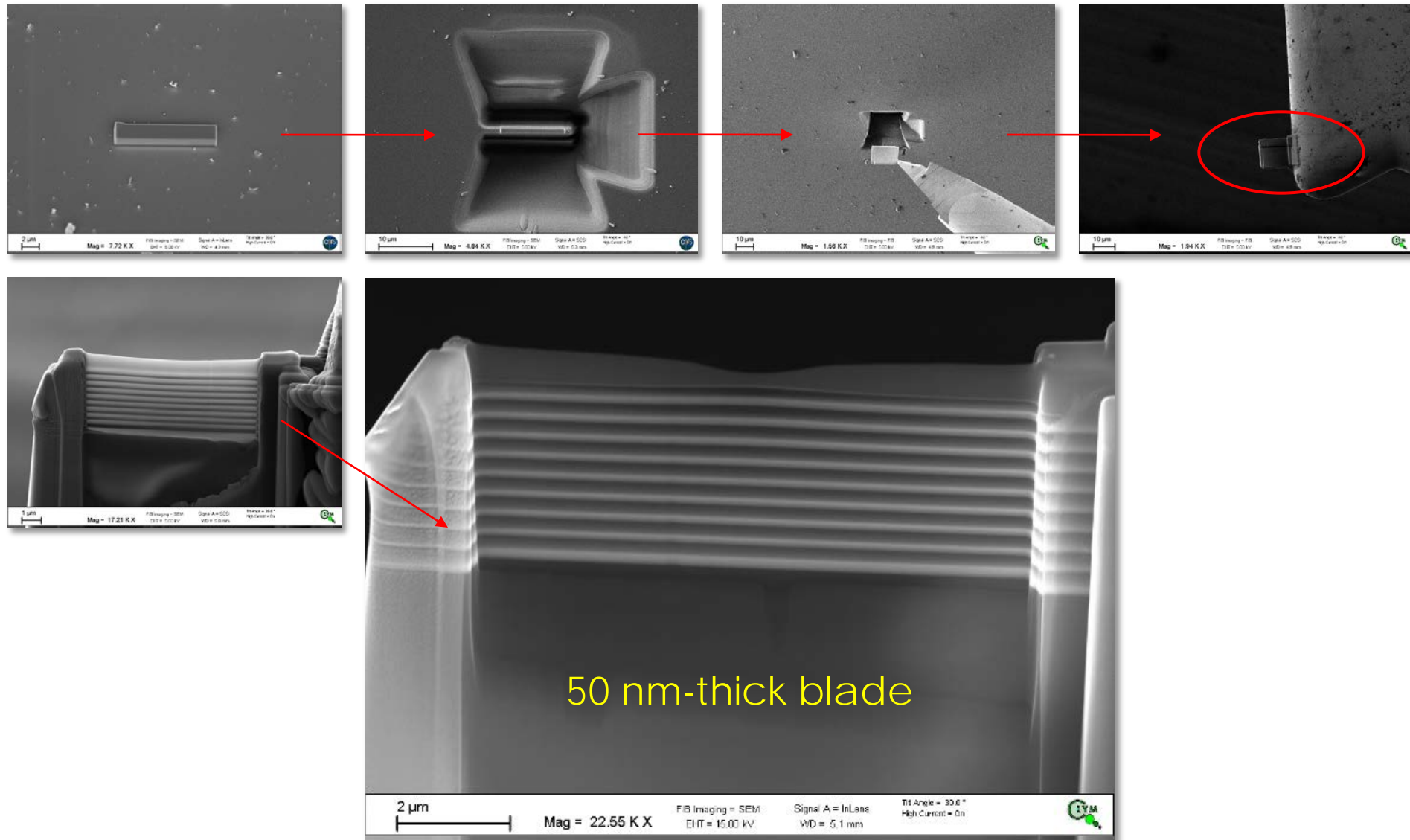
- sample: ITM-HR coating, 1.031H(1.3927L 0.5854H)8 1.199L [18 layers]
- Scanning/Transmission Electron Microscopy (SEM/TEM) @ CLYM:  
study at atomic scale



- imagery
- density profiles
- energy-dispersive X-ray analysis (EDX) → atomic concentration

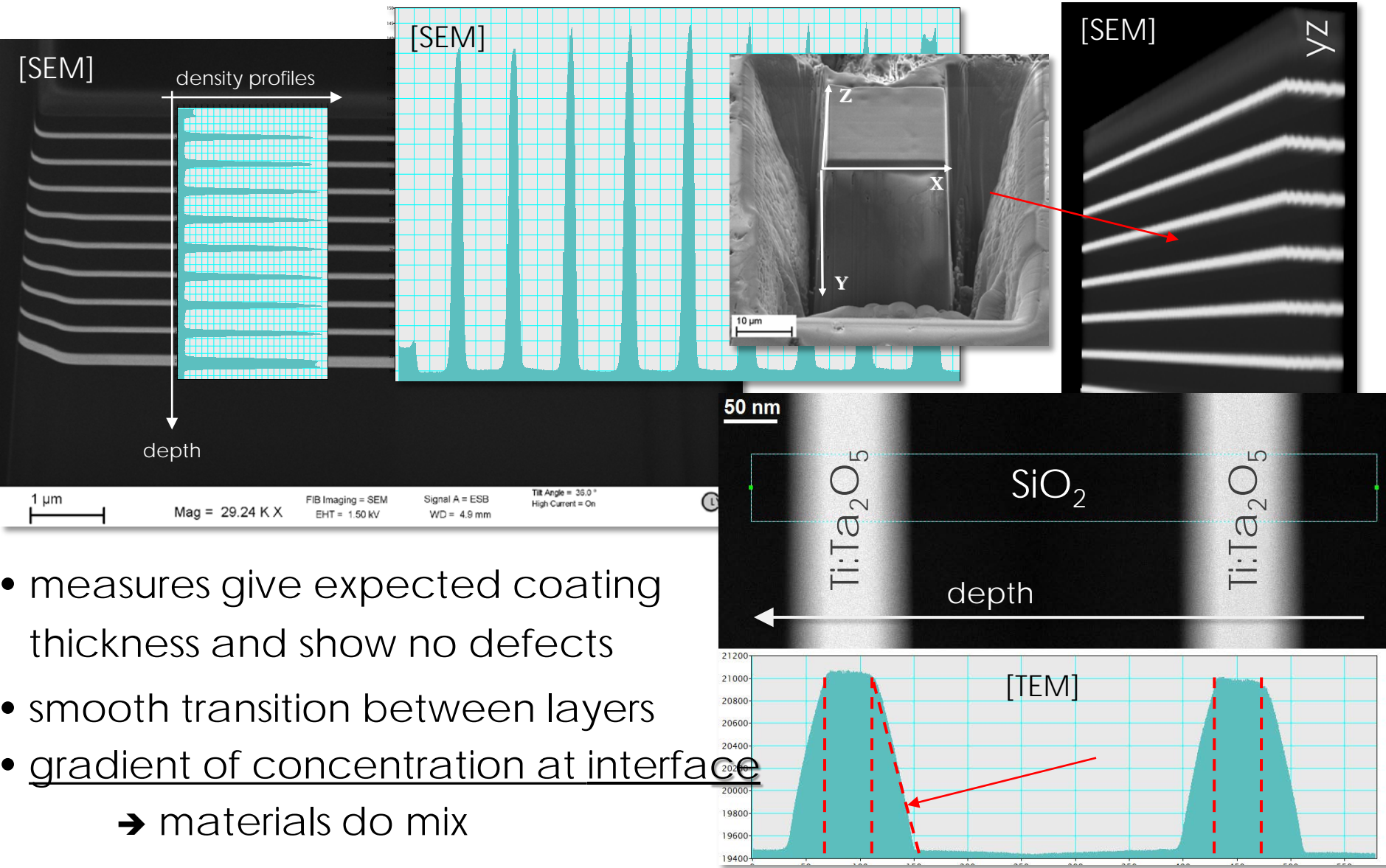
hypothesis: interdiffusion of materials?

# Preparation of the Sample





# Tomography & Density Profiles

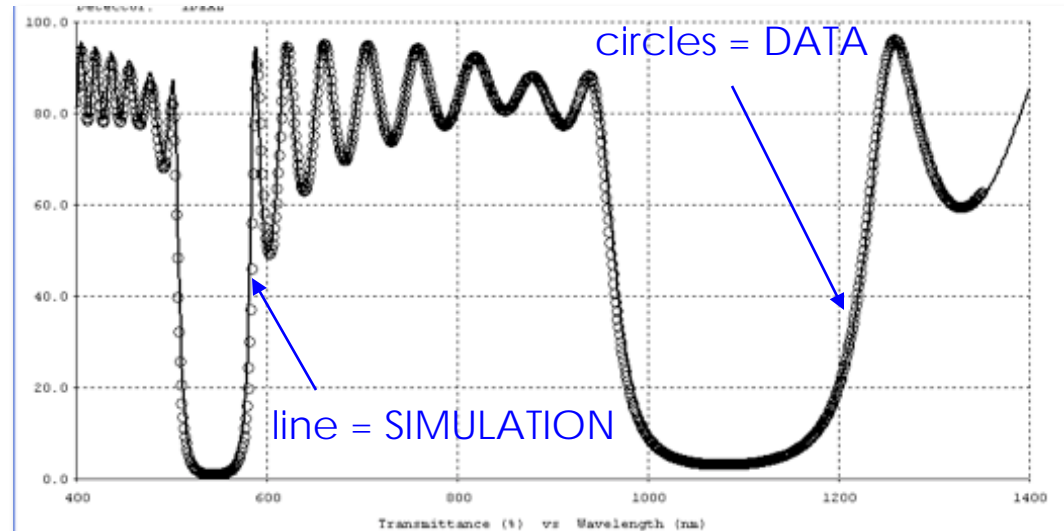


- measures give expected coating thickness and show no defects
- smooth transition between layers
- gradient of concentration at interface  
→ materials do mix



# The Picture

- no crystalline phase observed in layers
- analyses reveal a mixing interface of 20 to 35 nm
- simulated optical spectra of multilayer coatings with mixed materials at interfaces are compatible with measures on 1" substrate
- optical simulations also show that gradients should be symmetric:  $\text{Ti:Ta}_2\text{O}_5$  and  $\text{SiO}_2$  diffuse into each other
- analyses of atomic concentrations are not conclusive → new measures in progress



# Mixed Materials

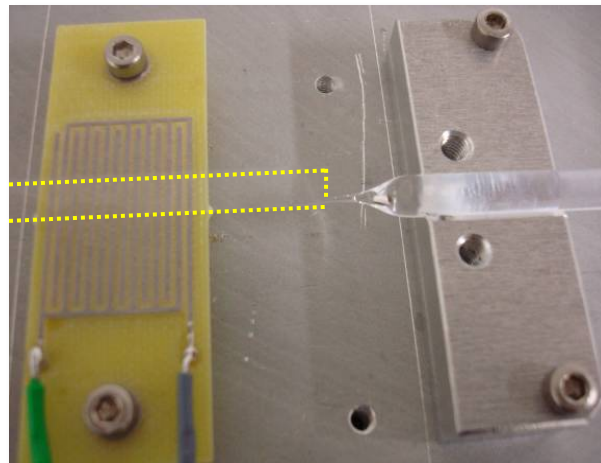
- could mixed interfaces explain exceeding loss?
- simple model of the interface: discrete gradient  
mixed material 1 = 65% SiO<sub>2</sub> + 35% Ti:Ta<sub>2</sub>O<sub>5</sub>  
mixed material 2 = 65% Ti:Ta<sub>2</sub>O<sub>5</sub> + 35% SiO<sub>2</sub>
- coating of a SiO<sub>2</sub> blade with mixed material 1

- measure of loss of mix1:

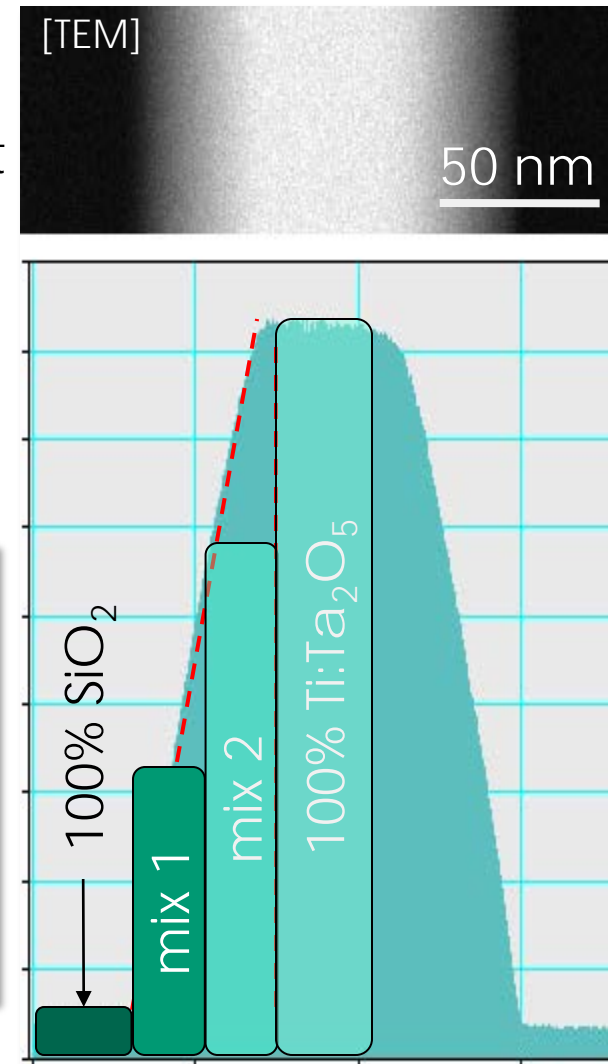
$$\Phi_{\text{mix1}} = (2.7 \pm 0.2) \times 10^{-4}$$

loss similar to Ti:Ta<sub>2</sub>O<sub>5</sub>

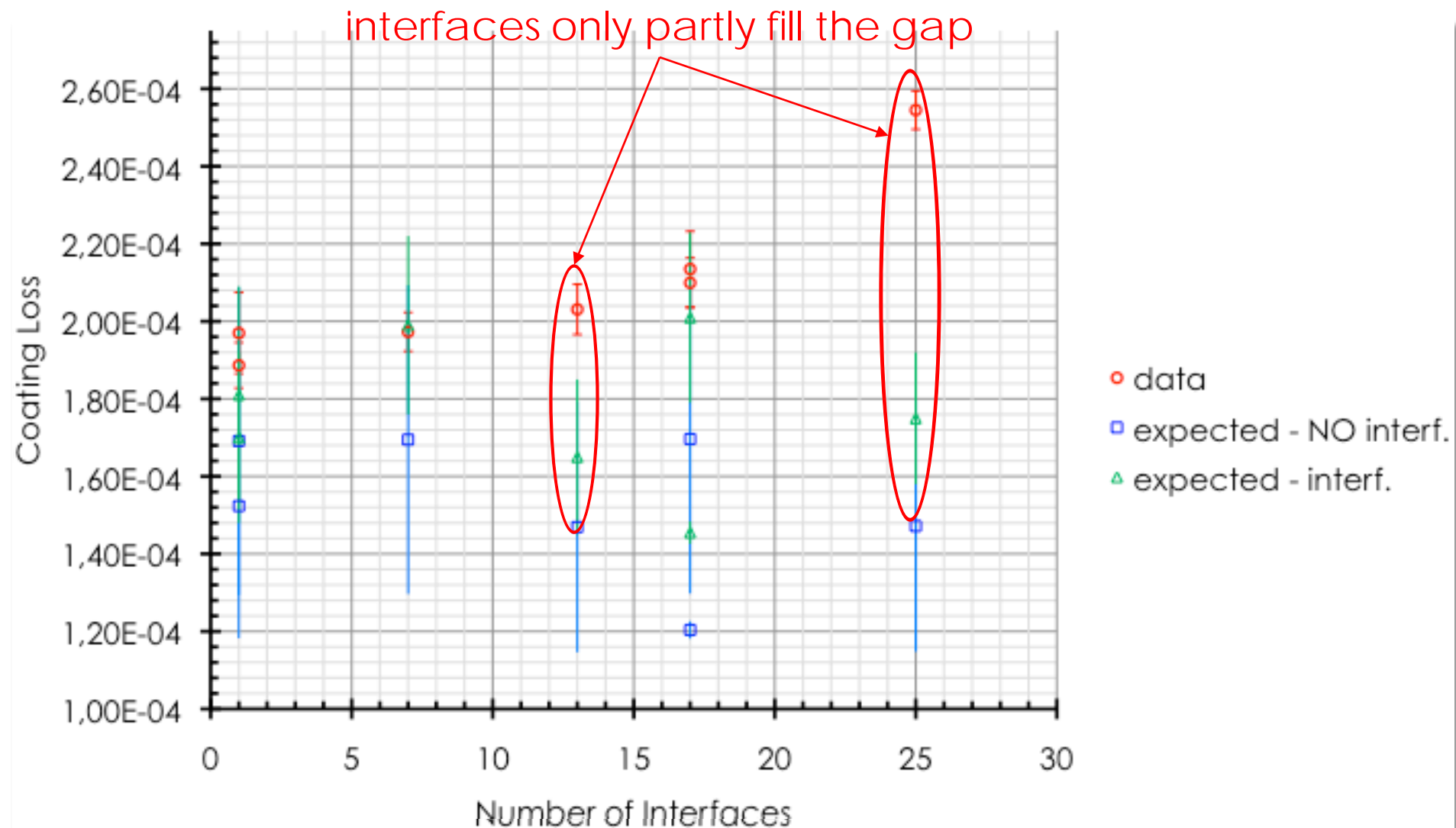
- to be done: deposit and measure mix 2



setup for ringdown technique



# Interfaces – Results



# Interfaces – Conclusions

- excess of mechanical loss is observed in multilayer coatings, depending on the number of interfaces between layers
- interfaces have been characterized
  - analyses revealed a mixing zone with gradients of atomic concentration
- loss of a mixed material (65% SiO<sub>2</sub> + 35% Ti:Ta<sub>2</sub>O<sub>5</sub>) has been measured
  - loss is similar to that of Ti:Ta<sub>2</sub>O<sub>5</sub> ...
- ... but the presence of mixing interfaces only *partially* explains the exceeding loss [mixed material 2 has to be measured]

do we need a more complex model of the gradient?

# Updates on Atomic Layer Deposition

in collaboration with E. Härkönen, M. Ritala & M. Leskelä *University of Helsinki*



# Atomic Layer Deposition (ALD)

the idea:

investigation of new techniques to improve quality of coatings

→ we want lower mechanical losses



test of ALD realized @ University of Helsinki

advantages: ✓ self-limiting mechanism of film growth  
✓ excellent coating uniformity - even on large areas  
✓ homogeneous stoichiometry  
✓ high reproducibility

→ coatings of good quality factors<sup>[\*]</sup> and optical properties

major concern: x low deposition rate

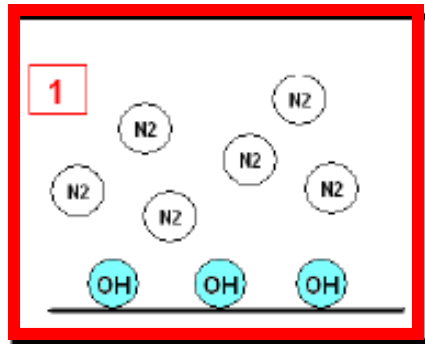
[\*] O Hahtela & al., *J. Micromech. Microeng.* 17, 2007



# Reminder

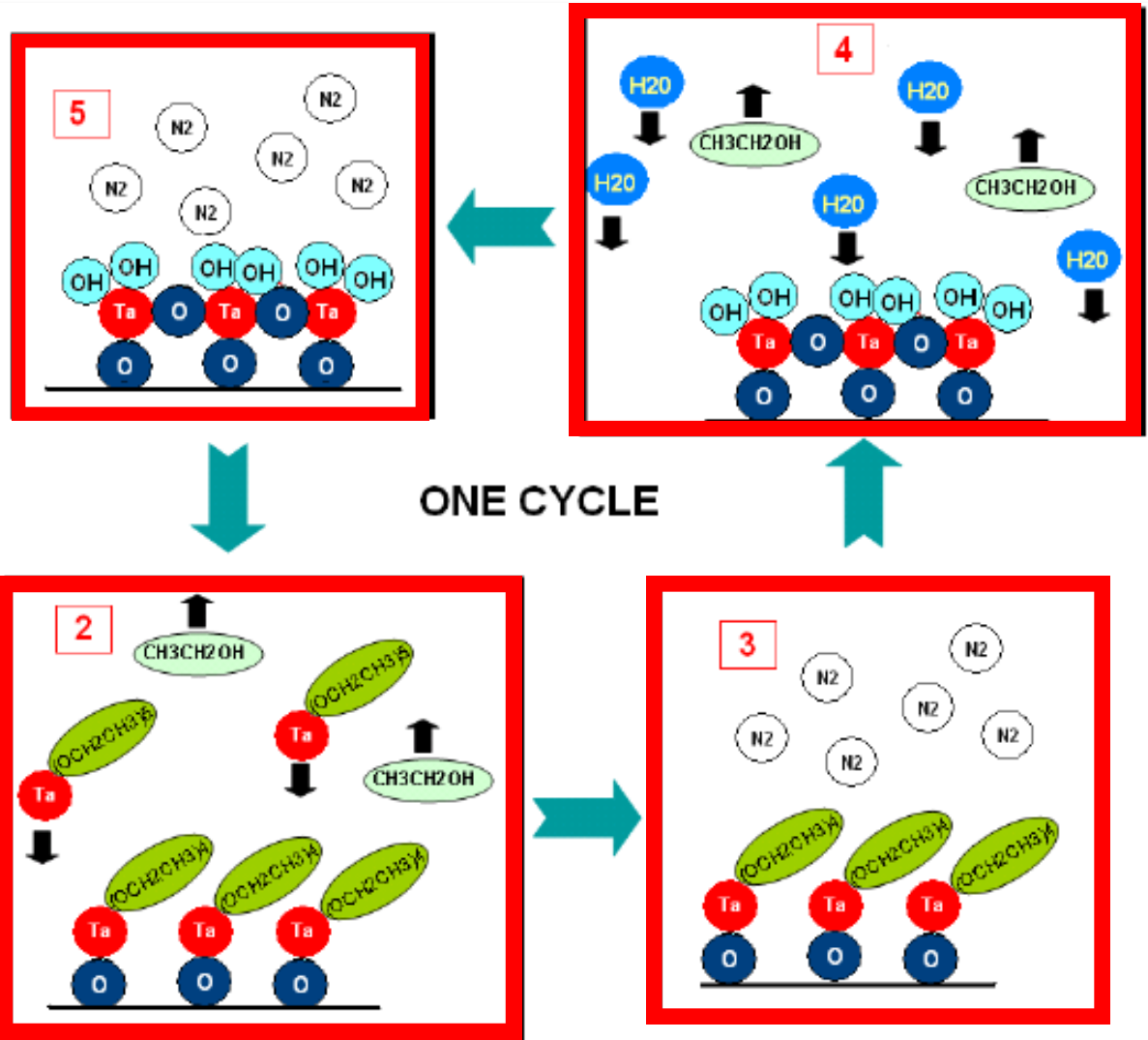
method based on alternate saturative surface reactions

1. purge [ $N_2$  – 5 s pulse]
2. precursor 1 [ $Ta(OCH_2CH_3)_5$  – 0.3 s pulse]
3. purge [ $N_2$  – 5 s pulse]
4. precursor 2 [ $H_2O$  – 0.3 s pulse]
5. purge [ $N_2$  – 5 s pulse]  
→ back to step 2



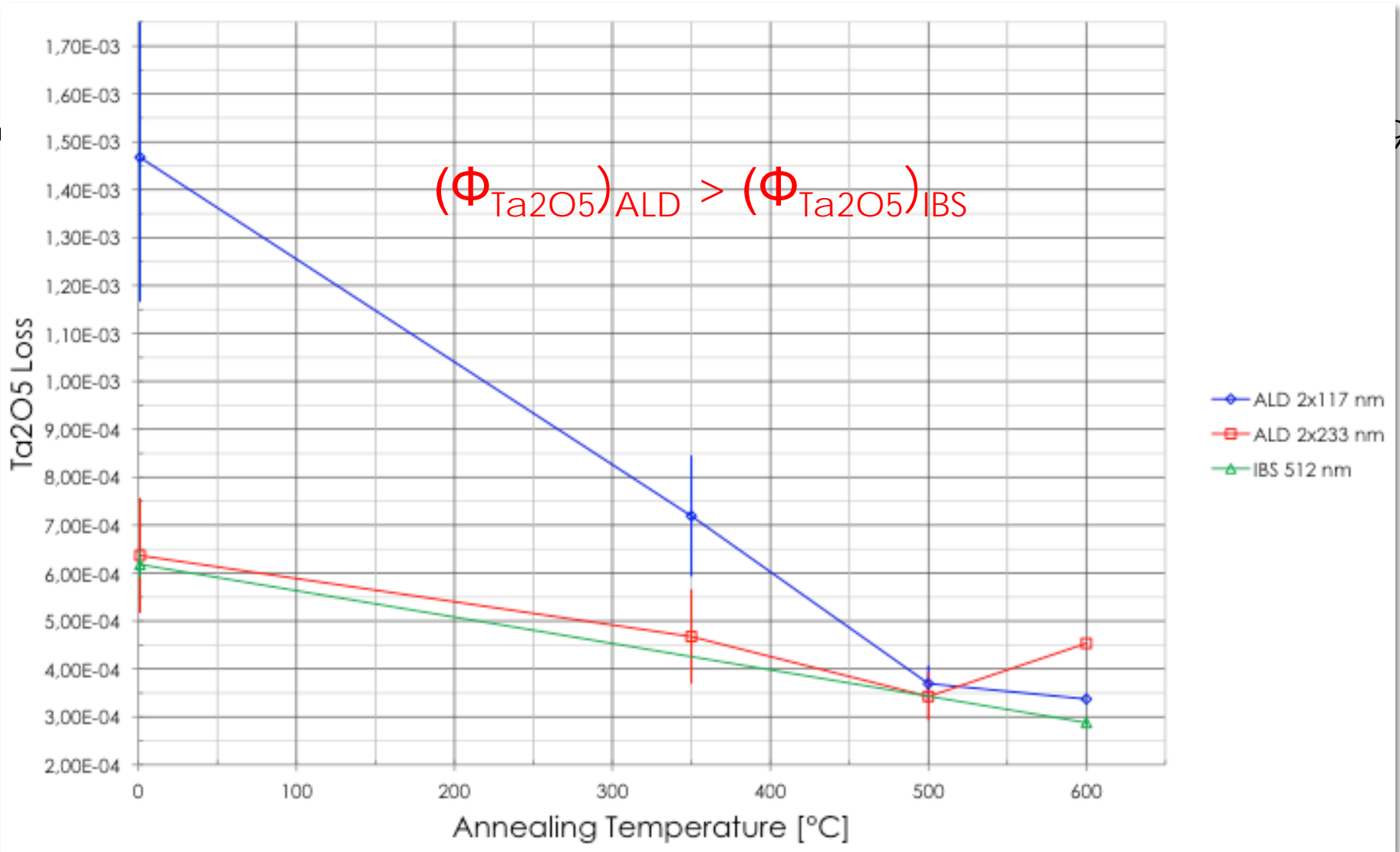
substrate @  $T = 250\text{ }^\circ\text{C}$

credits: J. Franc



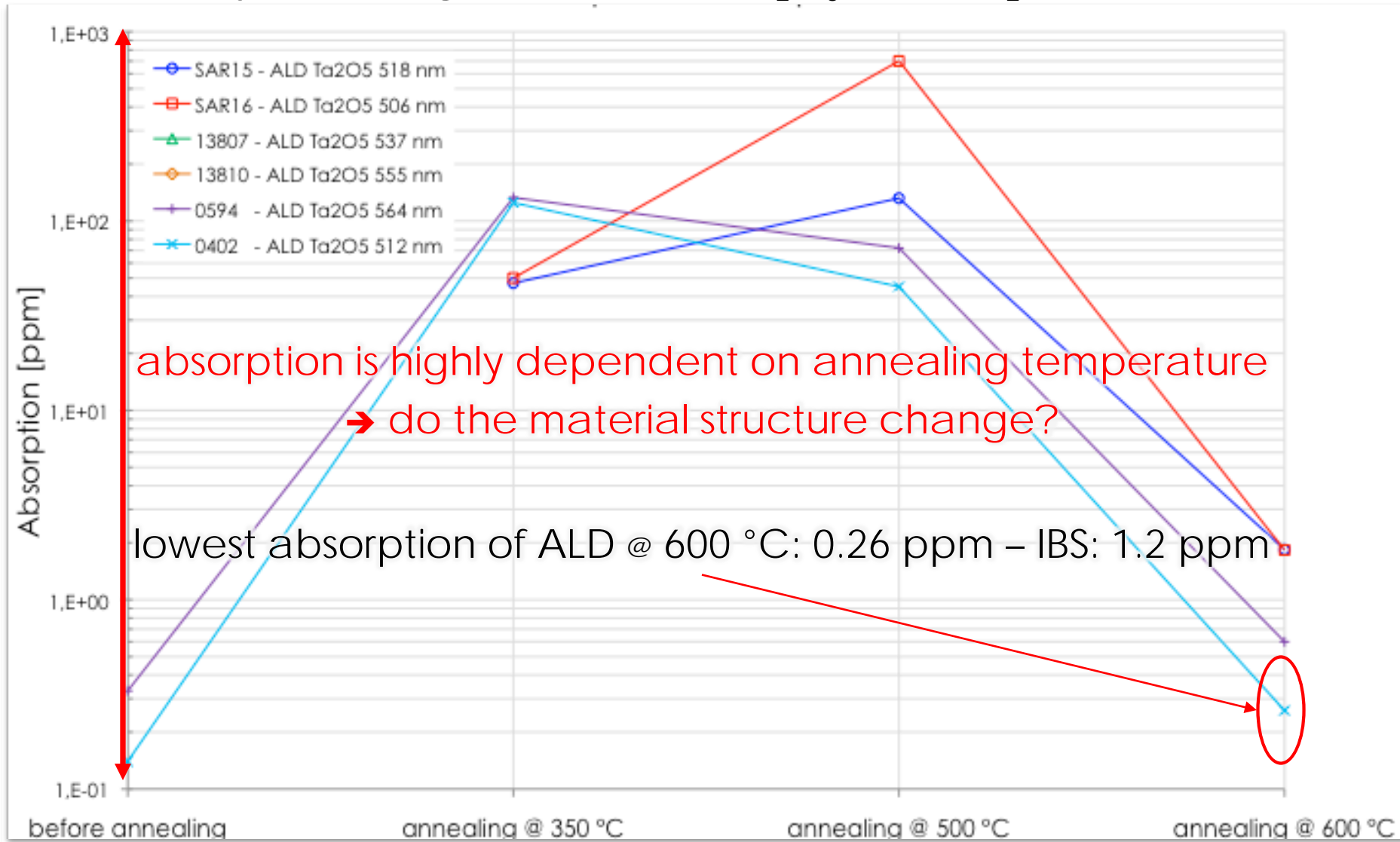
# ALD – Results

monolayer coatings of undoped Ta<sub>2</sub>O<sub>5</sub> on SiO<sub>2</sub> cantilever blades



# ALD – Results

monolayer coatings of undoped Ta<sub>2</sub>O<sub>5</sub> on 6 SiO<sub>2</sub> substrates of 1"



# ALD – Conclusions

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- ✓ ALD is a well-established technique to realize high-quality coatings
- x deposition rate is low
  
- x mechanical losses of ALD coatings are larger
  - no specific advantage in using ALD instead of IBS
  
- ✓ ALD coatings show good optical absorption
  - unusual behavior wrt annealing might be worth further investigation

# Summary

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- the structure of multilayer coatings has been characterized
- analyses revealed interfaces of mixed materials
- mixed interfaces can only partly explain the gap between predicted and measured losses
  
- ALD coating technique has been investigated
  - x losses are larger compared to IBS coatings
  - ✓ optical absorption seems lower for  $T_{\text{annealing}} > 600 \text{ }^{\circ}\text{C}$
  
- next step: further characterization of optical absorption wrt  $T_{\text{annealing}}$

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*Thank you for your attention.*