In-situ Cryogenic Cleaning Techniques for Tin-Contaminated EUV Mirrors

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Goal: Develop new tin-cleaning schemes for the EUV collector

Tests: Examine removal of tin droplet splats from EUV mirrors

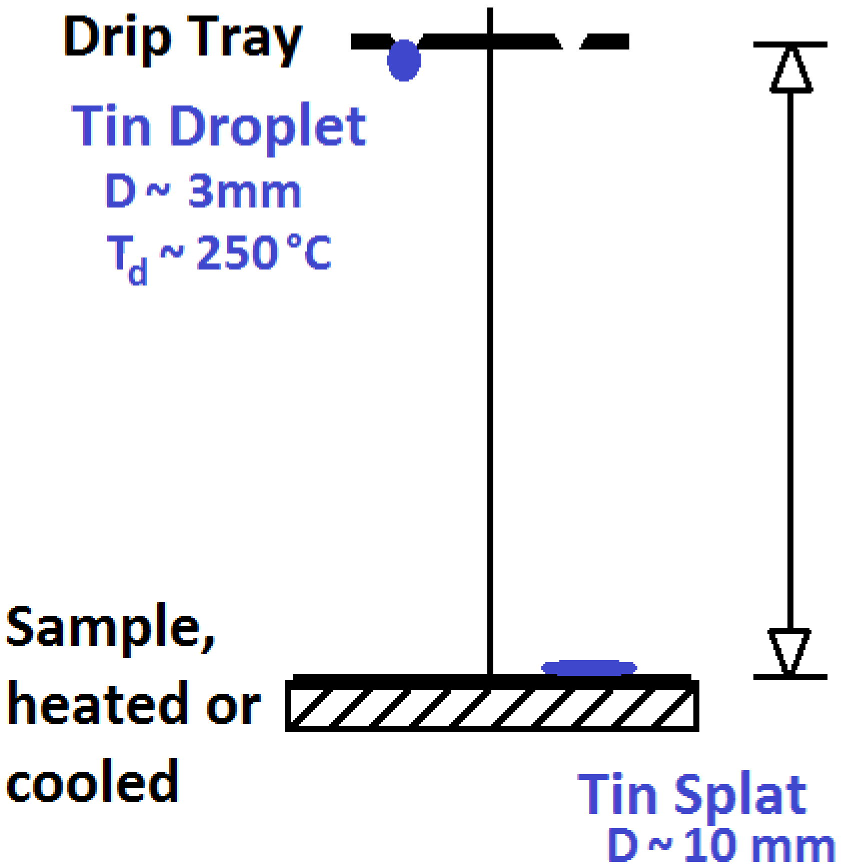
Motivation: Tin- Cleaning of EUV Collector

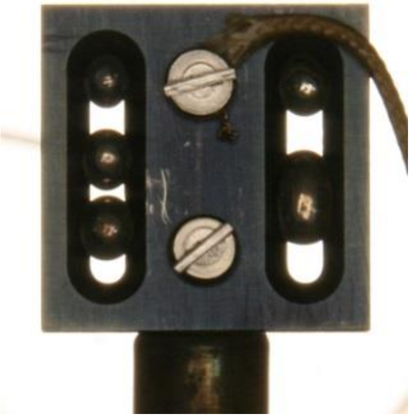
- Considerable contamination of the collector mirror occurs by tin debris
- Chip manufacturing needs increased source availability and productivity

- Increase of collector lifetime needed: more than several months, > 400 Gp
- Internal tin-cleaning by plasma etching is too slow for thick deposits
- Improved tin-cleaning efficiency can increase uptime and reduce costs

Dripping Molten Tin Drops onto EUV Mirrors

Tin dripping scheme:





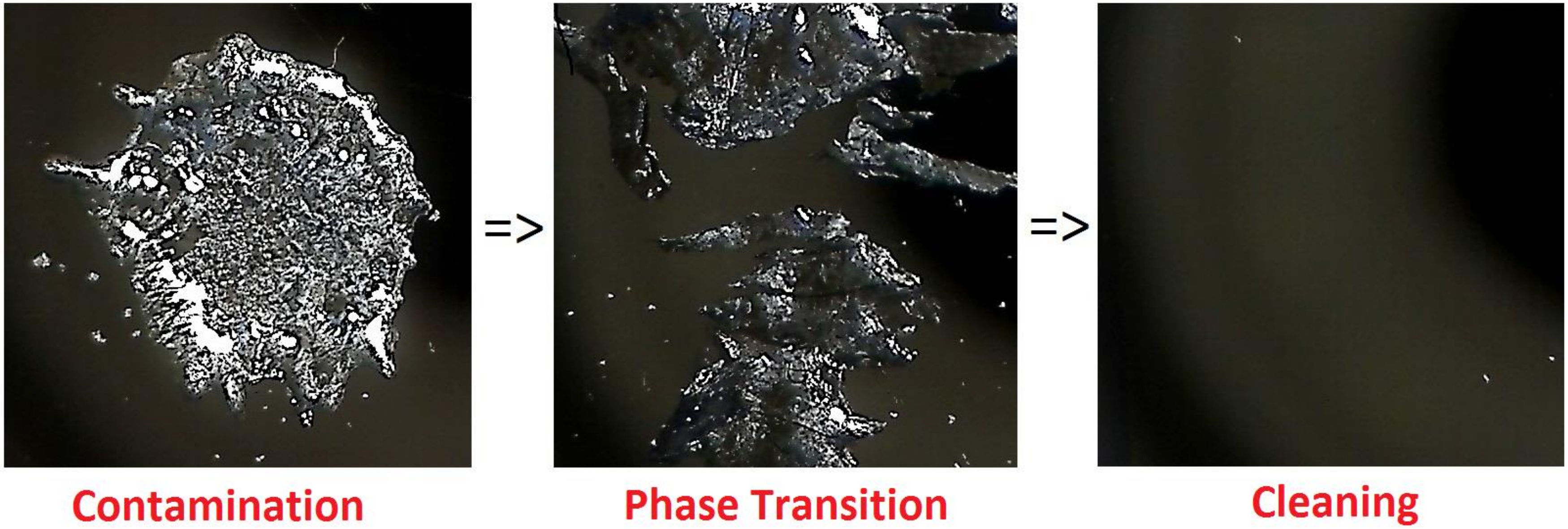
Mass of Tin Drop m ~ 0.1g
Drip Distance d ~ 45 cm
Dripping Time t ~ 0.3 s
Impact Velocity v ~ 3 m/s
Weber Number ~ 300

T_s~ 90 °C, 23 °C, -50 °C

Regime: tin dripping below droplet splashing threshold, radial „finger“ pattern developing at edge of splat

Phase Transformation of Tin Drop by Cooling

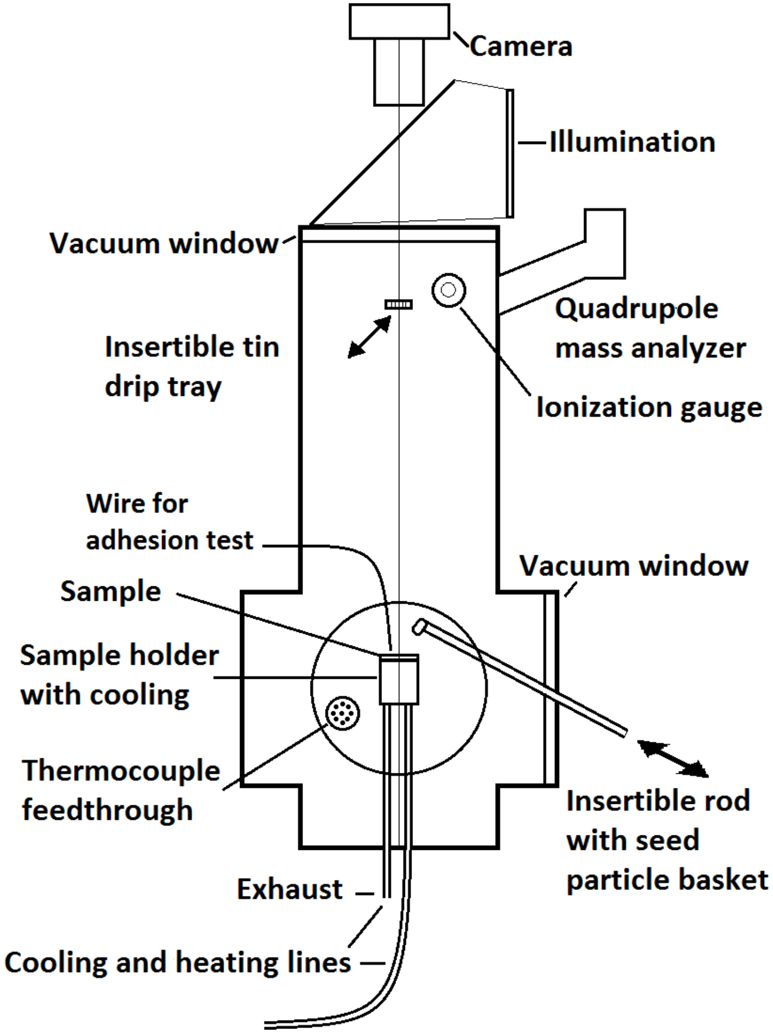
Induction of tin pest leads to embrittlement of tin-drop contamination



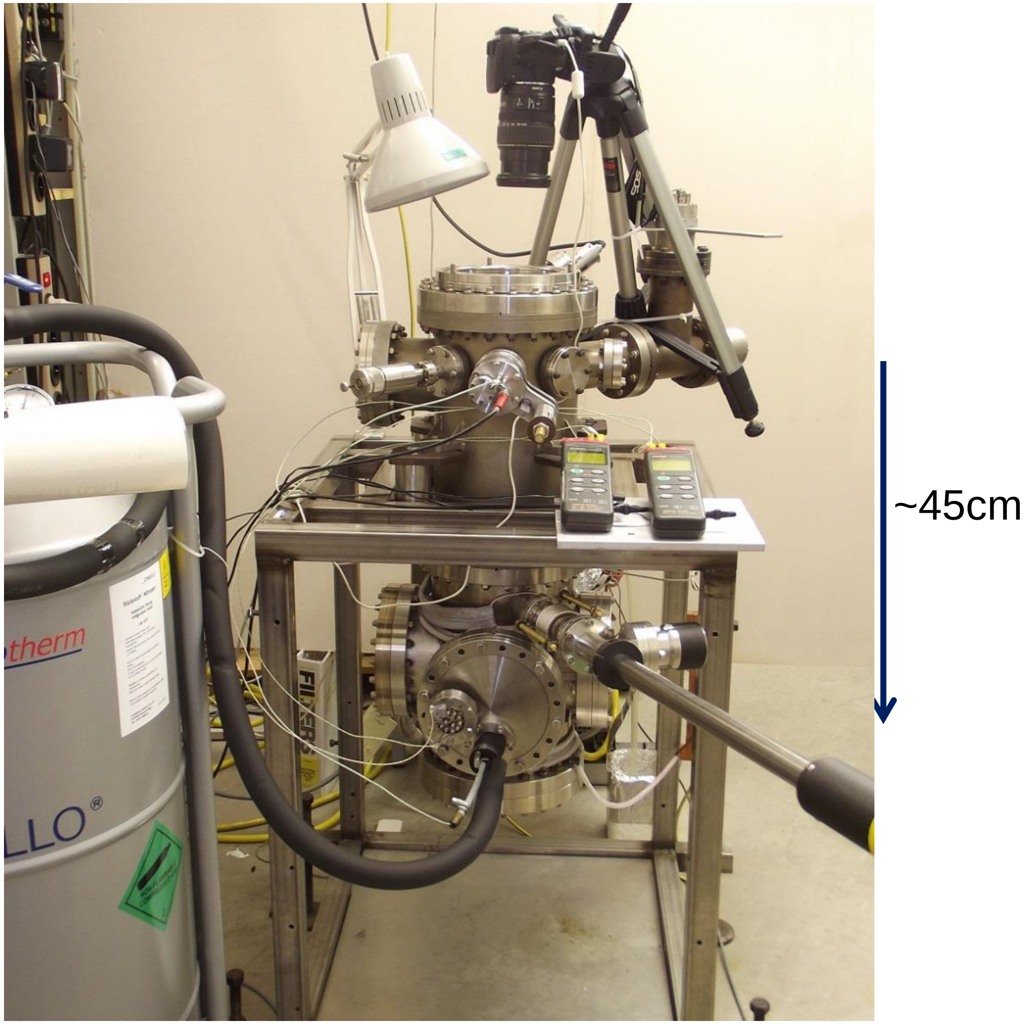
- Contamination of ML-coated mirror by large tin drops (99.999% purity)
- Seed initiation of phase transition at -25 °C to gray tin which breaks apart
- Subsequent surface cleaning by vibrations or flows of inert gas

First ex-situ tests: N. Böwering, Mater. Chem. Phys. **198**, 236 – 242 (2017)

Setup for In-Situ Tin Dripping in Vacuum



Can fit in MLM-samples and up to 6-inch wafers



Sample cooling and heating capability; chamber base pressure: 10^{-7} mbar

Sticking Behavior of Tin Drops on Surfaces

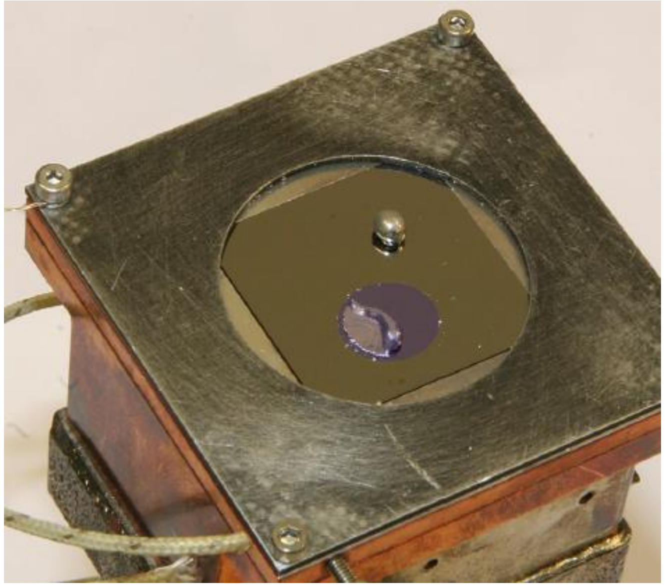
Sticking depends on properties of substrate and coating:

Influence of material, substrate temperature and effusivity (heat conductance)

- Droplet splat sticking observed on heated smooth substrates, T_s = ~90°C
- Non-sticking on Si substrates at room temperature or when cooled to -50 °C
  Delamination behavior seen: droplet self-peeling or contraction dominates
  Bending force by surface tension of splat exceeds adhesion to substrate

Sample type/T_s	~90 °C	23 °C	– 50 °C
Si wafer	Sticks	Peels	Peels
Si–ZrO_2	Sticks	Peels, contracts	Contracts
Si–Mo/Si	Sticks	Peels	Contracts
Si–Mo/Si–ZrN	Sticks	Sticks or peels	Peels
Si–Mo/Si–ZrO_2	Sticks	Sticks	Peels
Oxidized Si wafer	Sticks	Sticks	Peels
Glass slide	Sticks	Sticks	Sticks
Rough Si wafer	Peels	Peels, contracts	Contracts

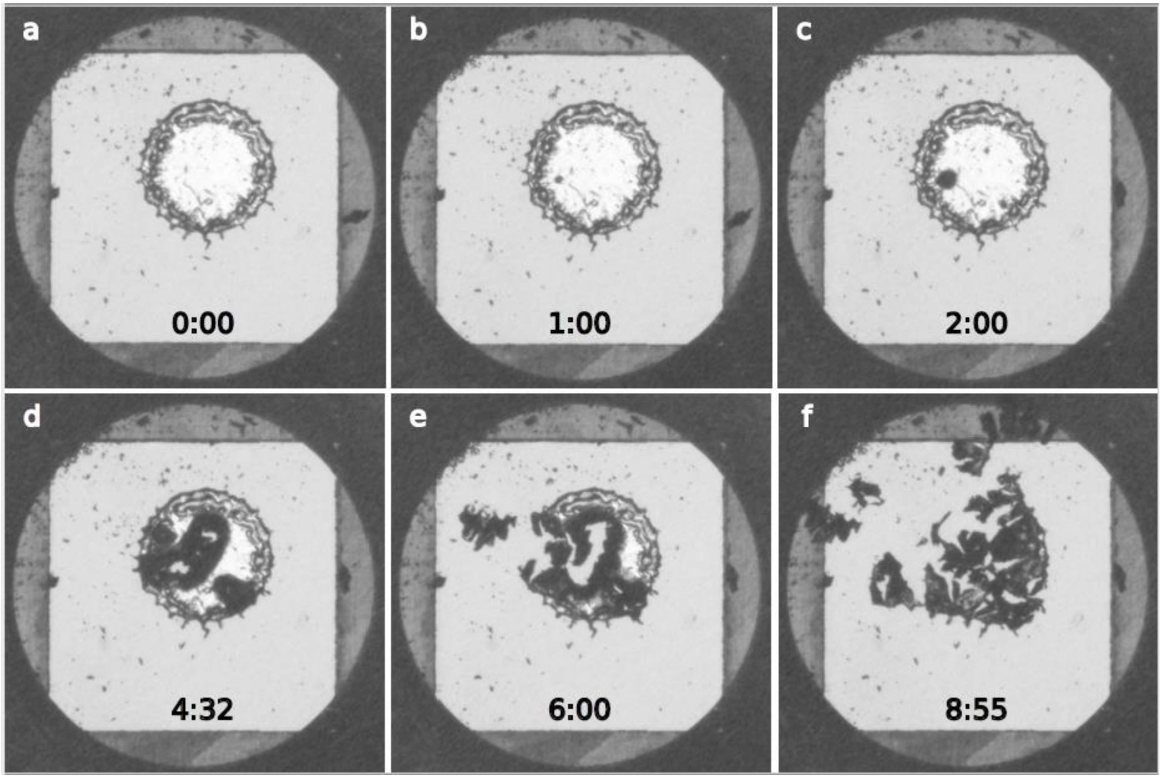
Sticking behavior: N. Böwering, C. Meier, Appl. Phys. A **125**, 633 (2019)



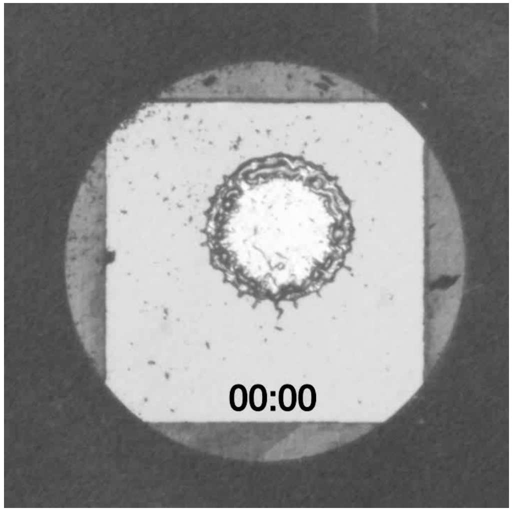
Fully and partially contracted splats

In-Situ Transformation of Tin Splat

Tin drop (154 mg) dripped on sample at T_s = 92 °C. Growth of brittle gray tin: v = 10-15 µm/min.
Substrate cooling at T_s of -30 °C to -40 °C; ~9 hrs. to complete the transformation



T_s~ -35°C



Time lapse video
(not included in pdf)

Coating of Si-sample: Mo/Si MLM with 3 nm thick ZrO_2 cap layer (R_{max} = ~56% at 13.5 nm)

Sticking behavior and transformation: N. Böwering, C. Meier, Appl. Phys. A **125**, 633 (2019)

Tin Drop Transformation on Multilayer-Coated Mirror Sample: EUV-Reflectance

<u>EUV Reflectance at 13.6 nm for uncapped Mo/Si-coated sample:</u>

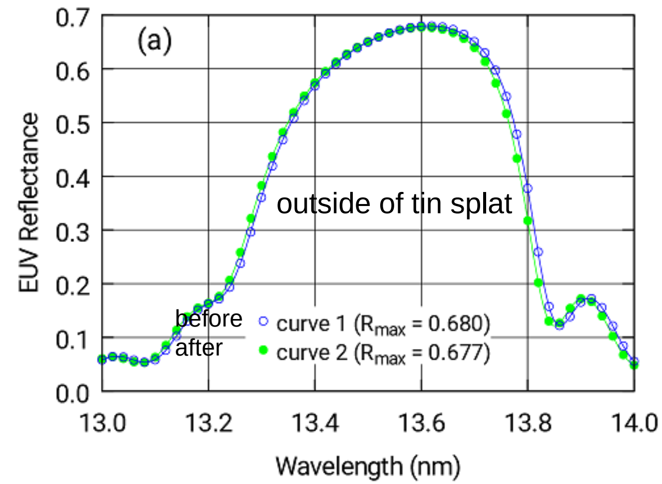
Tin drop conversion during cooling in ~7 hours

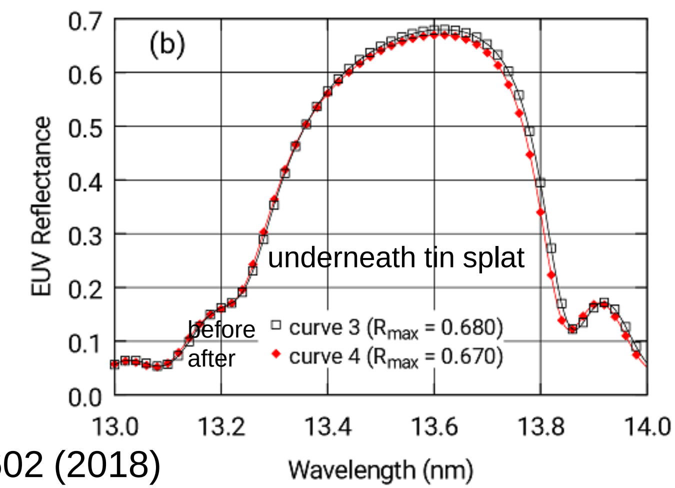
=>

T=-24°C

after tin dripping

after conversion





EUVR measurements by PTB:

Peak EUVR before tin dripping: R_{max} = 68.0%

Peak EUVR after tin cleaning: $R_{max} \geq$ 67.0%

(a) R_{max}: 67.2 % – 67.7 % outside of tin splat

(b) R_{max}: 67.0 % underneath tin splat

EUVR results: N. Böwering, C. Meier, J. Vac. Sci. Technol. B **36**, 021602 (2018)

Tin Removal by CO_2 Snowflake Aerosol

Ex-situ removal by impact of CO_2 snow-flakes using a gun-jet nozzle with expansion of liquid carbon dioxide at high gas pressures

During expansion phase change of liquid CO_2 to small snow crystals with temperature of ~-70 °C

pressure: ~50 bar, filter nozzle: 1 mm diam.

Fast ex-situ removal technique with minute duration; works for thin and thick Sn deposits and also for other adhering contaminations

Process can be automated for repeated sweeping strokes across curved substrates

High-purity CO_2 gas should be used!
Organic residue seen when using industrial grade liquid CO_2 (99.5%)

Setup for CO_2 snow-jet cleaning of 4“ Mo/Si-coated MLM wafer

EUV mask cleaning with CO_2 snow: Varghese et al., Proc. SPIE **8166**, 816615 (2011)

Alternative Cryogenic Sn-Removal Techniques

We have examined 5 different schemes at low temperatures

In-situ tin splat removal:

- Non-sticking at low substrate temperatures *(cooling to ~ -50 °C)*
- For sticking deposits:
  - Conversion to gray tin by tin pest *(phase transition, at -25…-40 °C)*
  - Delamination by strong cooling *(to ~ -120 °C, expansion difference)*

Ex-situ tin splat removal:

- Delamination after immersion in liquid nitrogen *(contraction difference, ~ -196 °C)*
- Removal by high-pressure CO_2 snow-flake aerosol *(cooling and impact, ~ -50 °C)*

Delamination due to thermal contraction mismatch:
CTE at 300 K of Si: $2.6*10^{-6}$/K
CTE at 300 K of β-Sn: $22*10^{-6}$/K
No damage to ML coating seen

Compared to tin-etching by hydrogen radicals, all cryogenic techniques are faster by orders of magnitude!

Top of tin drop

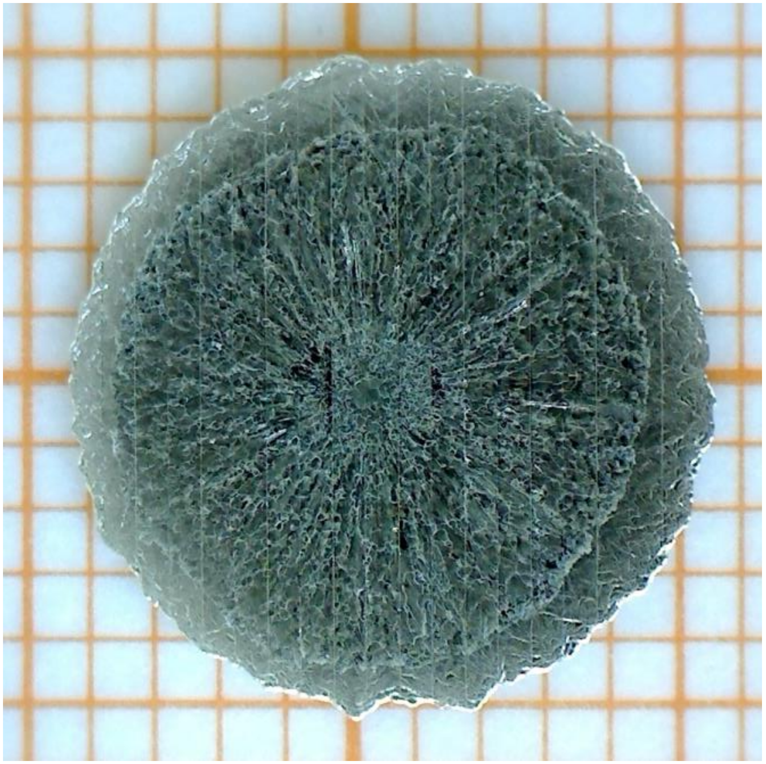
Bottom of tin drop

6“ Structured Si-Wafer Grating Sample

EUV collector mirrors use grating structures for suppression of IR radiation

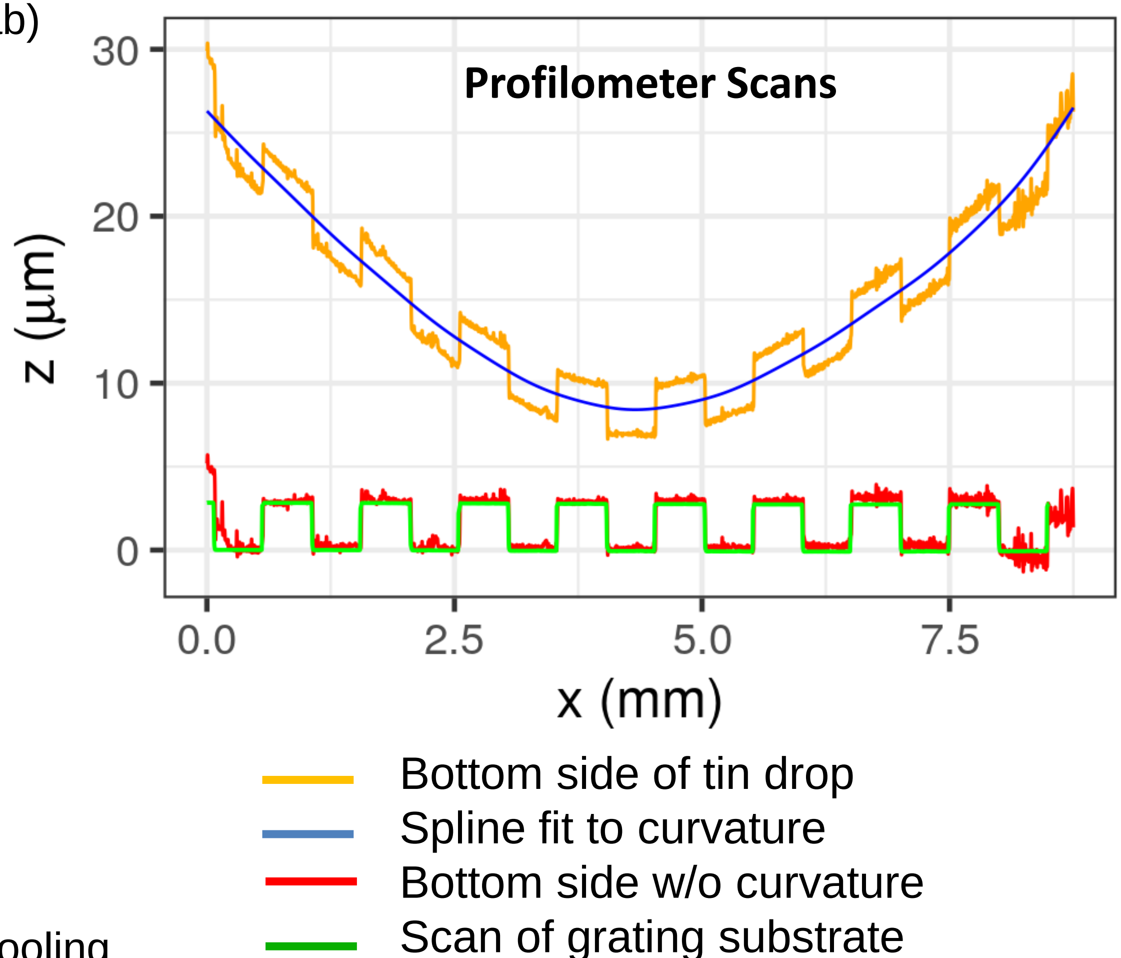
M. Kriese et al., Proc. SPIE **9048**, 90483C (2014); T. Feigl et al., Proc. SPIE **9422**, 94220E (2015)

6“ laminar grating on Si substrate
etched straight grooves (optiXfab)
0.5 mm wide, ~3 µm deep



Drop removed from grating sample by cooling

Grating structure imprinted on bottom of tin drop

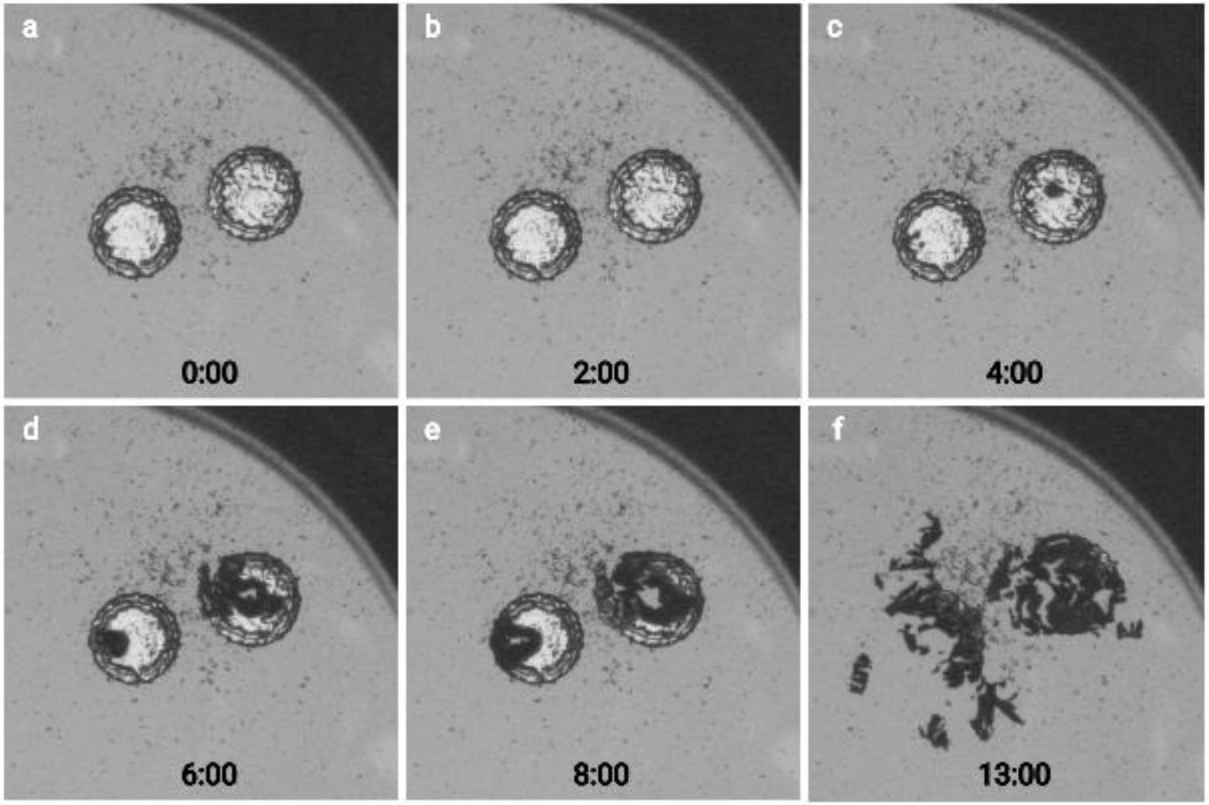


In-situ Transformation on 6” Grating Sample

Similar phase transformation behavior observed on uncoated Si grating wafer*

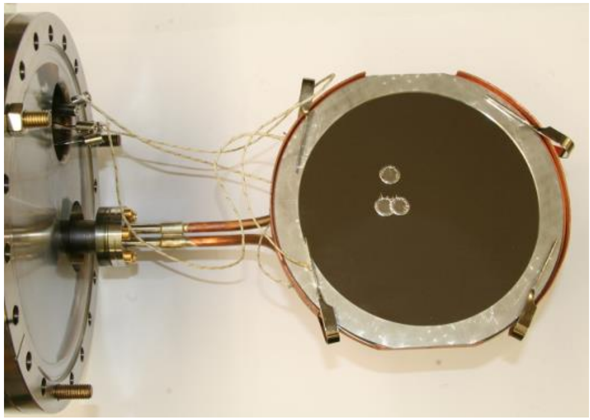
* supplied by optiX.fab

T_s~ -40°C

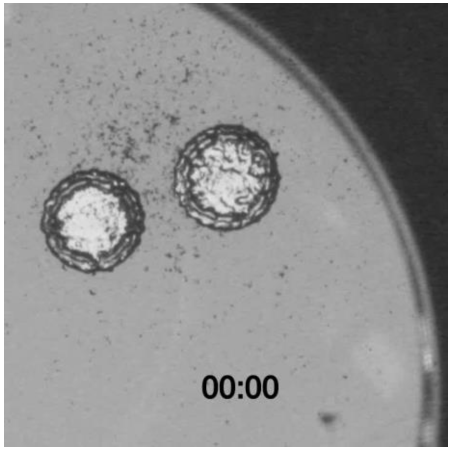


Droplet splat conversion to gray tin on uncoated Si grating wafer

“Gray tin on the grating”: N. Böwering, C. Meier, to be published



Cooled sample holder



Time lapse video
(not included in pdf)

Summary: Cryogenic EUV Optics Tin-Cleaning

Prerequisite: *Cooling to low temperatures of optics to be cleaned*

Several simple, low-cost methods were tested successfully on different substrates:

- Non-sticking tin drops at/below room temperature on Si substrates
- Induction of tin pest for sticking tin deposits on Si-MLM
- Phase transformation applies specifically to tin contaminations
- In-situ cleaning demonstrated inside of vacuum chamber
- Fast cleaning of thick deposits by conversion in less than 24 hours
- No damage of multilayer coating and grating
- Reduction in EUVR is less than 0.5%
- Alternatives: Delamination by cooling to very low temperatures (< -100°C)
- Ex-situ removal by impact of CO_2 snowflake jet
- 5 different schemes examined on different substrates

-> EUV collectors should be cooled more strongly to mitigate tin contaminations

Acknowledgements

- Thanks to optiXfab for providing ML-coated mirror and Si-wafer samples
- Thanks to PTB Berlin for a measurement of EUV reflectance

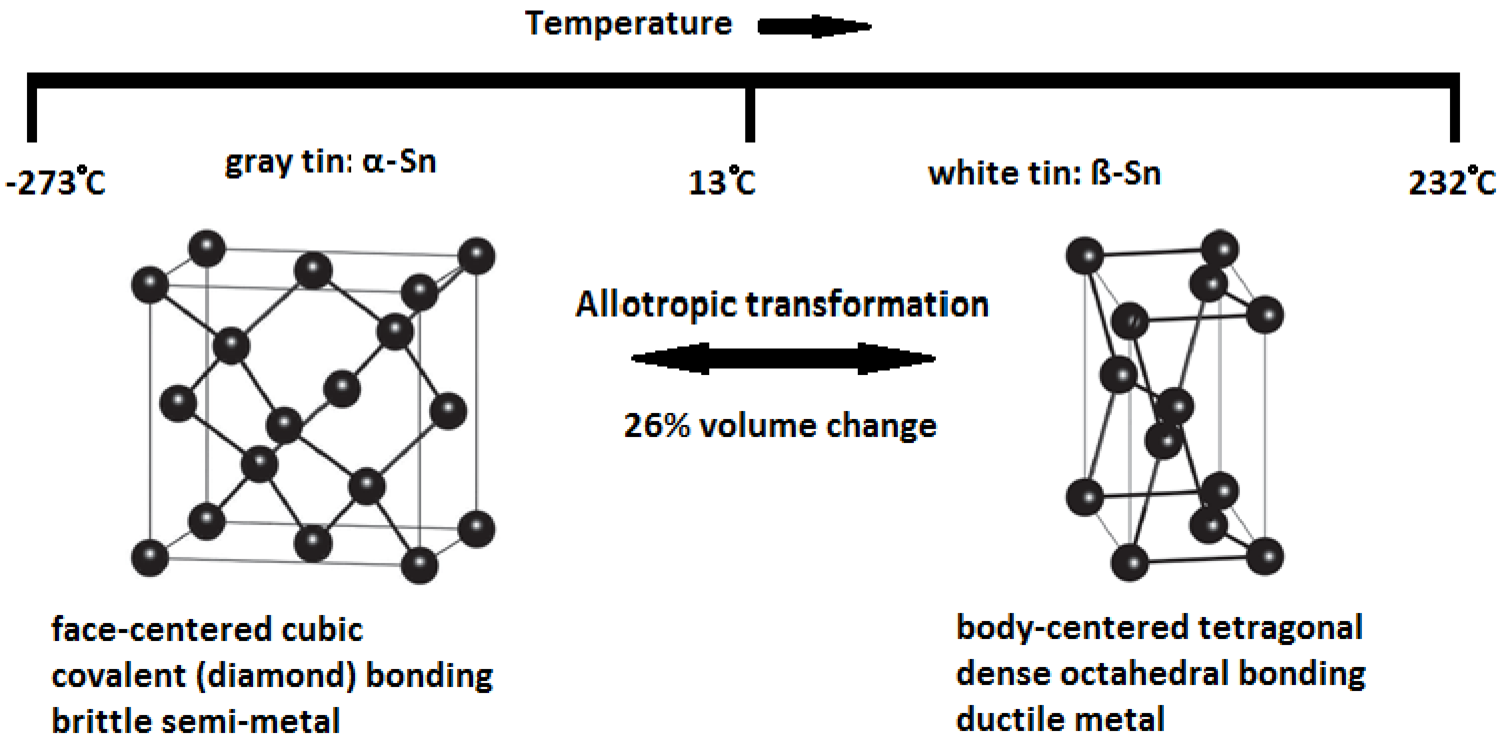
May the source be with you!

Thank you for your attention!

Research carried out without funding.
Any development funding welcome!

Add'l. Slide: Transformation of Tin β-Sn→α-Sn

- At -20°C to -50°C, white tin can undergo a phase transformation to brittle semi-metallic gray tin.
- The phase transition occurs only rarely, but it can be triggered by inoculation with seed particles.
- Tin breaks apart due to large volume increase and brittleness.



Add'l: Model for Splat Sticking or Detachment

Two semi-infinite bodies brought suddenly into contact, hot tin drop splat and colder silicon substrate:

$$T_c = T_s + (T_d - T_s)\,(1 + e_s/e_d)^{-1}\ ;\ \text{with } e = (k\rho c_p)^{1/2}$$

T_c: thermal contact temperature; T_s: substrate temperature; T_d: droplet temperature
e_s: substrate effusivity; e_d: droplet effusivity; k: thermal conductivity; ρ: density; c_p: heat capacity

For Si and tin drops with T_d = ~ 250 °C, T_s = 90 °C, 23 °C, -50 °C:
Initial temperature at contact surface: T_c = 138 °C, 90 °C, 39 °C

for T_c > ~100 °C: adhesion strength > bending force: splat sticks at T_s = ~90 °C
for T_c < ~100 °C: adhesion strength < bending force: splat peels at T_s = ~-50 °C

For splat detachment, the bending force during solidification has to overcome adhesion.