The formation and aging of diffractive optical elements in photo-thermo-refractive glass: In-situ investigations

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Photo-thermo-refractive (PTR) glass: process

is a **Na**–K-Zn-Al-**F**-**Br** silicate glass doped with As, Sn, Ce, and **Ag**.

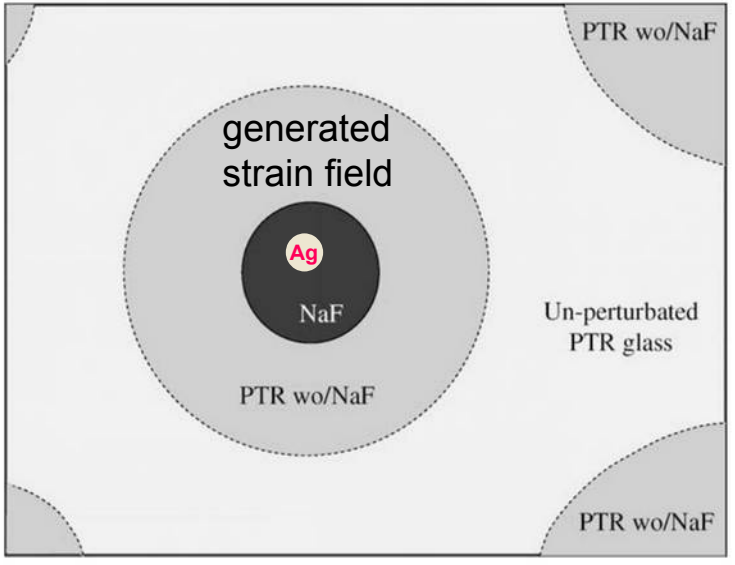
PTR glass exhibits a **localized refractive index change** ($\Delta n < 0$!)

1) after UV-exposure and

2) successive thermal treatment above the glass transition temperature, Tg,

which results from the **crystallization of about 0.1 wt% NaF** nano-crystals.

We have a technology but do not really know how/why it works



L. Glebov et al, Optical Materials 32 (2009) 139–146

$n_{NaF} \sim 1.3$

$N_{SiO2} \sim 1.5$

$\Delta n \sim 10^{-4}$

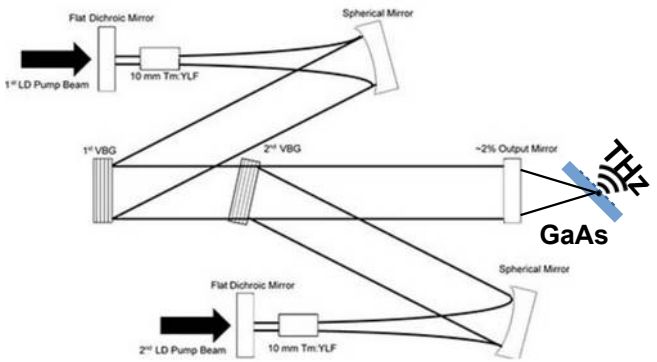
Its useful for: Applications

- Monochromatic THz emission by means of parametric oscillation

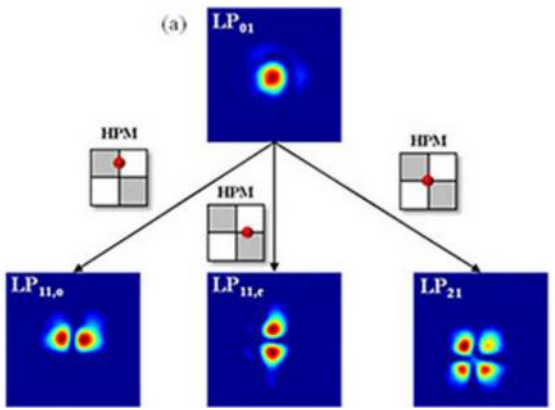
- Holographic phase elements for mode conversion, holograms

- Multiplexed volume Bragg gratings

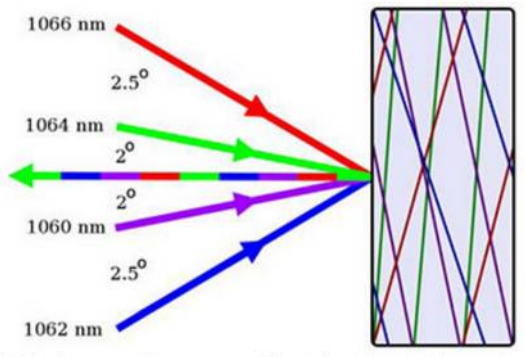
- …



Monochromatic 0.4 - 1.7 THz, Thz ~ μW??
optical continuous wave power levels from 80 to 100W



Fiber mode conversion from gound mode (LP01 to higher modes using a holgraphic phase plates

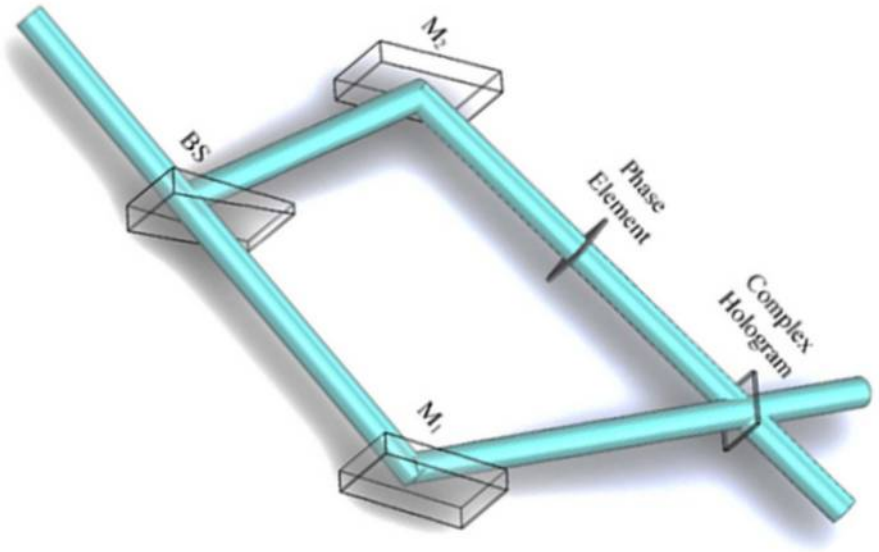


Generation of high power laser radiation combining multiple lasers into a single beam

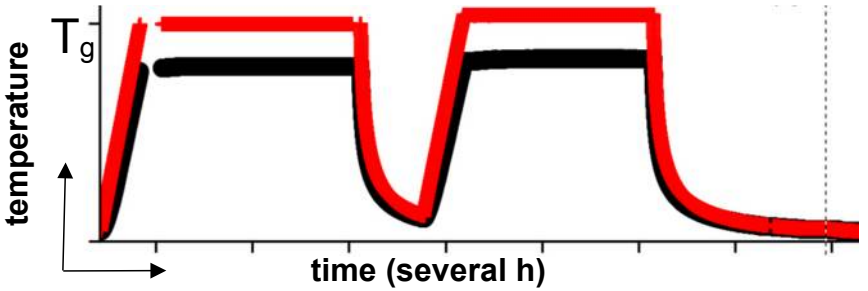
ppl.creol.ucf.edu/Research.aspx

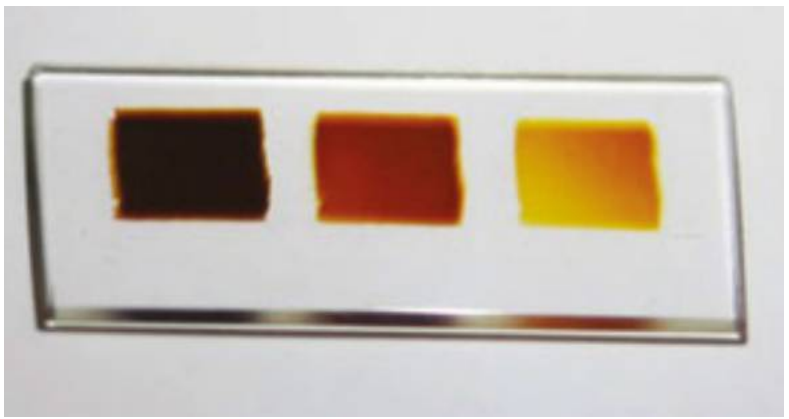
Photo-thermo-refractive (PTR) glass: manufacturing like a photographic film

1) exposure (UV light)



2) development (2 step thermal annealing)

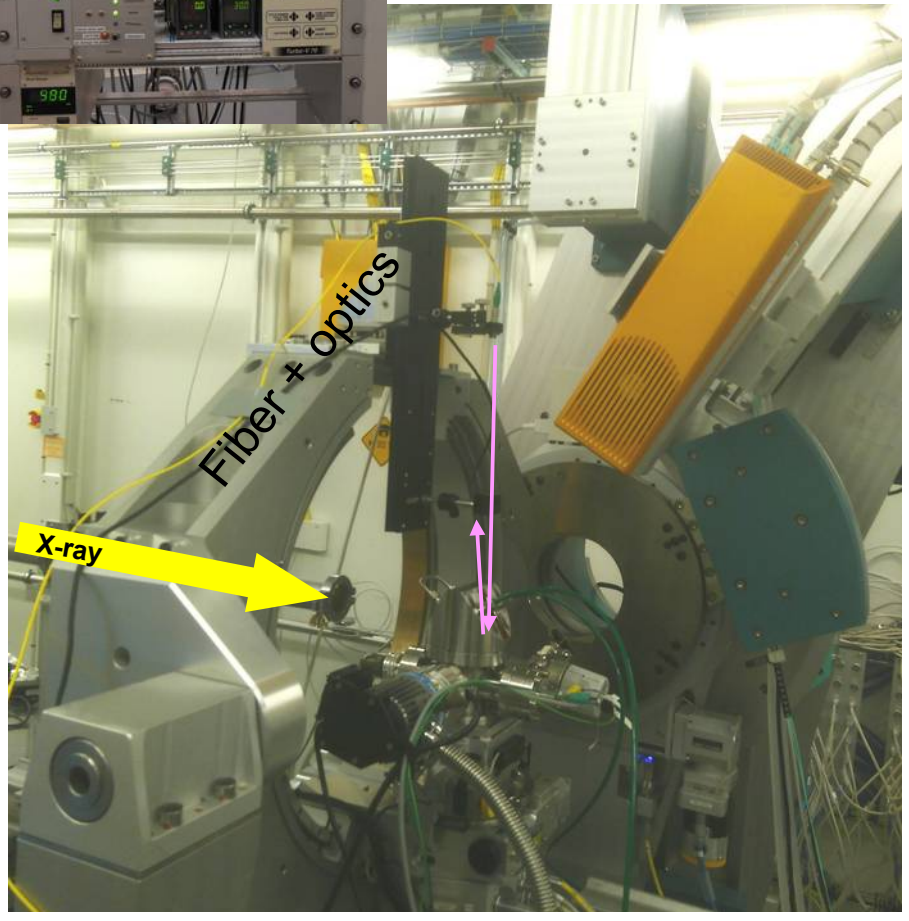




Study the whole process in-situ using X-rays

1st start with the development process

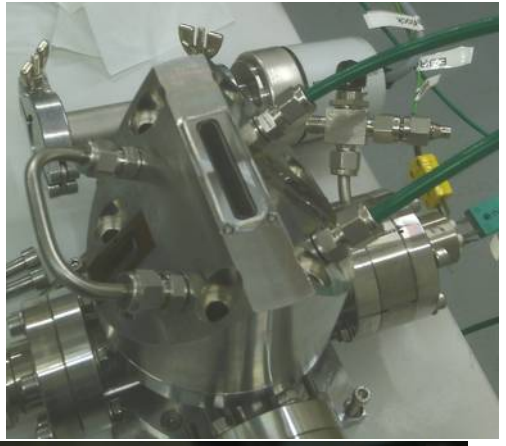
Combined diffraction + optical setup

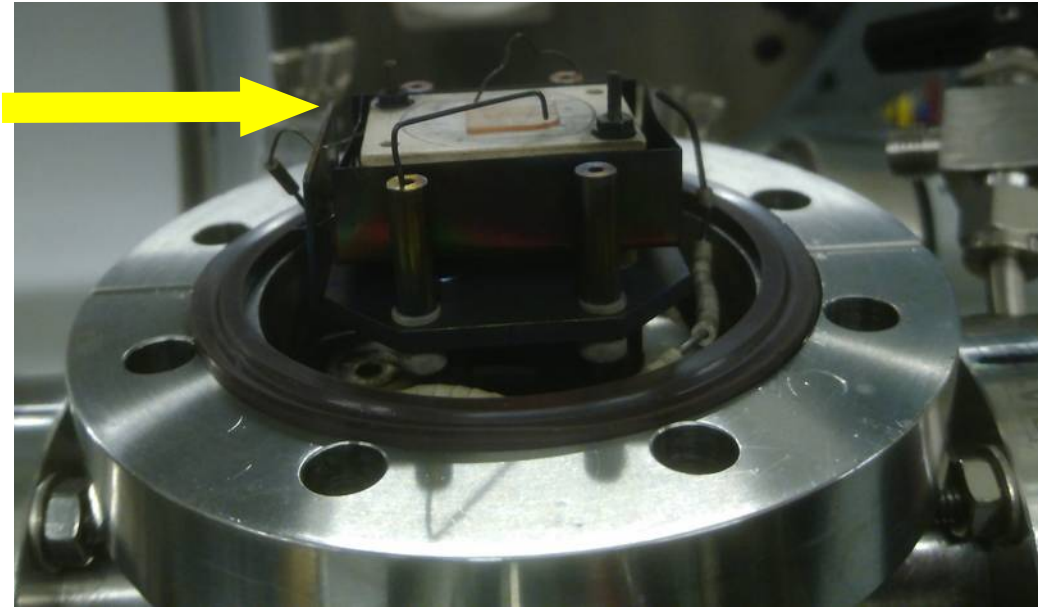


NIR signal almost ⊥ incidence

PETRAIII P23:

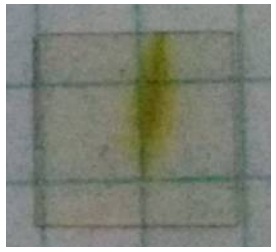
E_{phot} ~ 19keV
T_{int} ~ 10 sec/image
T_{max} ~ 600°C
α_i ~ 1.5°

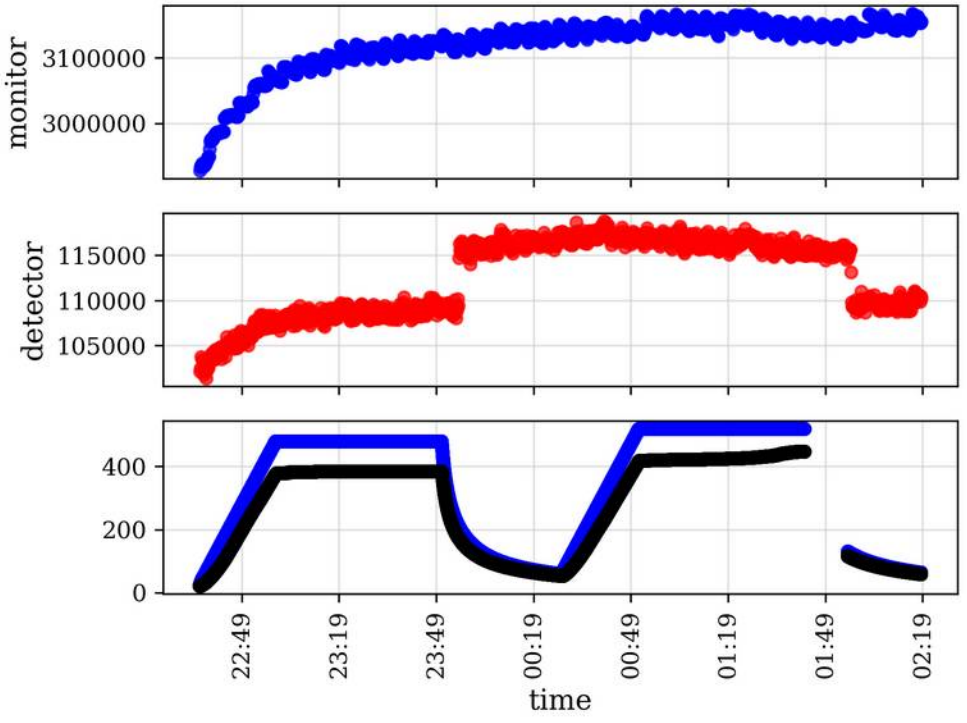


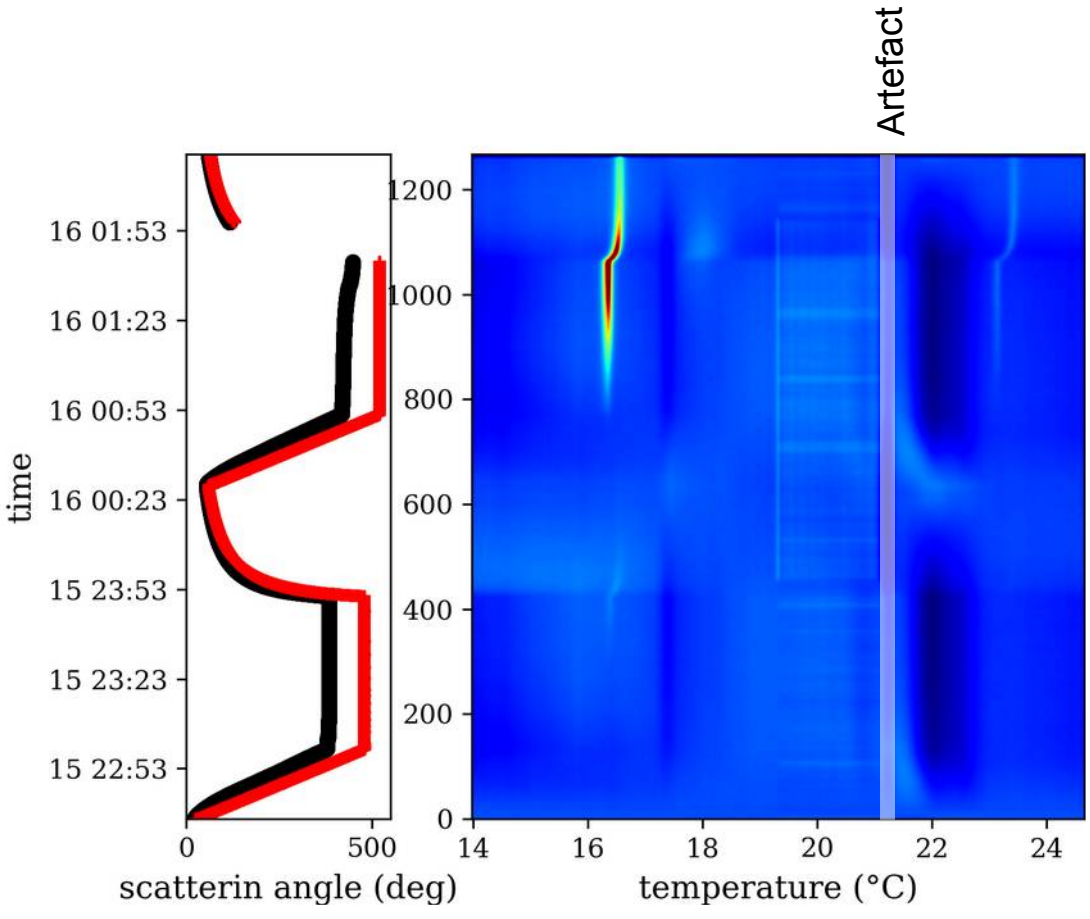


Sample 1 : UV exposed (XRD)

$E_{x\text{-}ray}$ = 20.1keV





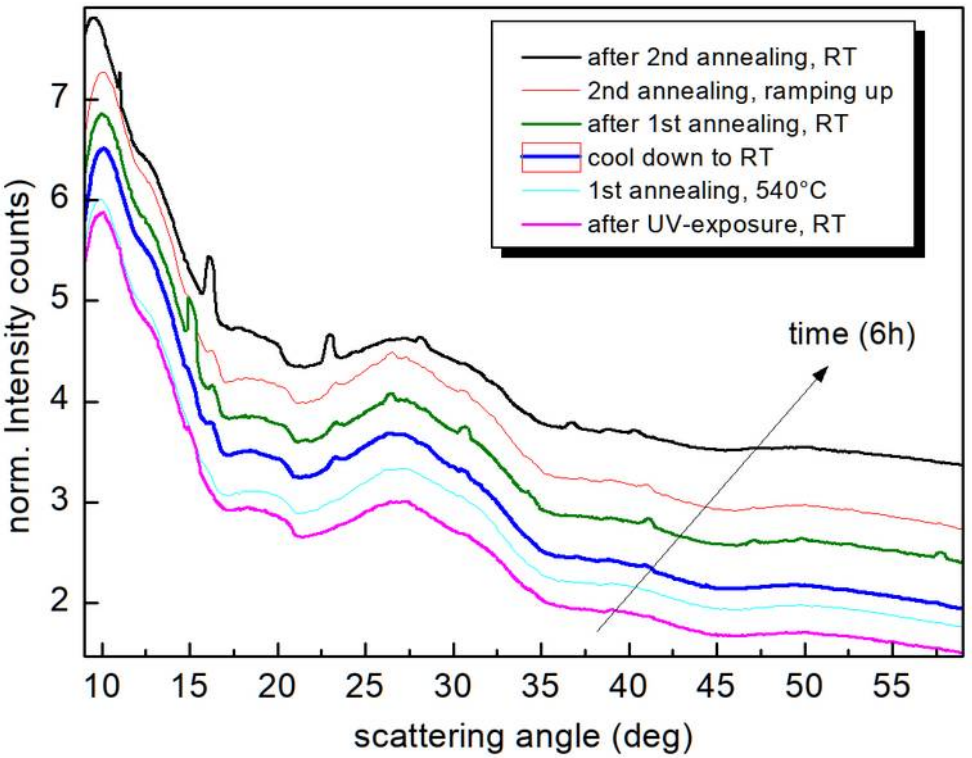


2 Step annealing 1st: 50min @480°C
2Nd: 50min @520°C

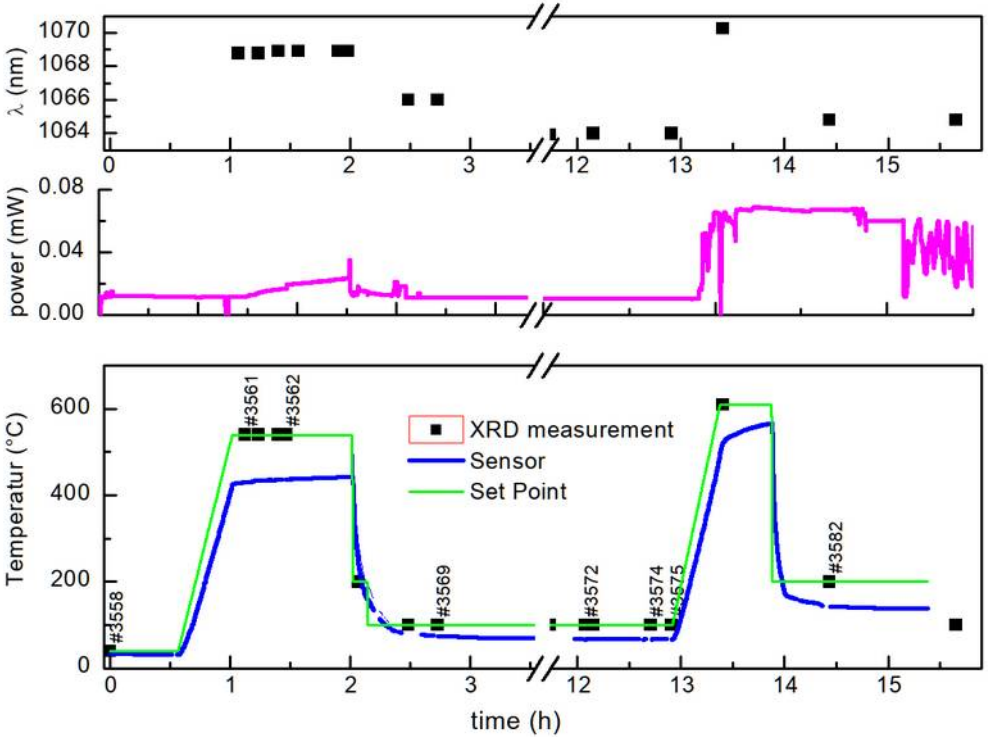
Image raw data:
P23_N94_irrad_3_00138-00138_org.tif.zip

Sample 2 : UV exposed (NIR+XRD)

$E_{x\text{-}ray}$ = 18.74keV

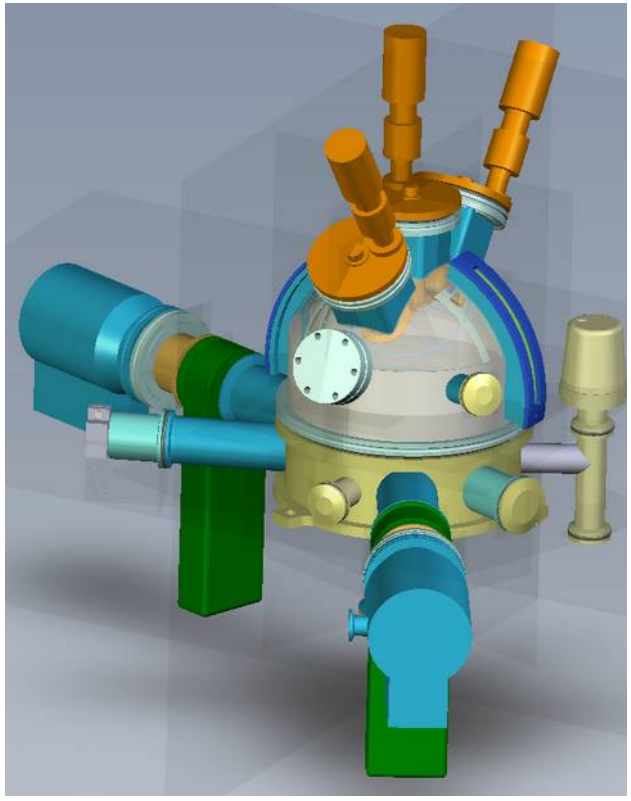


- NIR signal almost normal to sample surface
- reflected wavelength put always to max. reflected power
- sample thickness ~ 1mm
- 1st distinct peaks appear during the 1st cool down
  (Ag cluster precipitation?)
- after 2nd anneal reflected power increase > 10x

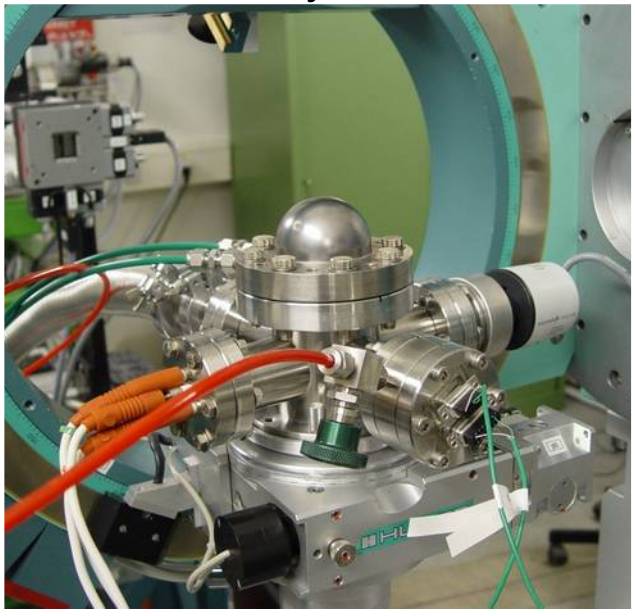


In-situ process chambers from material research @ P23

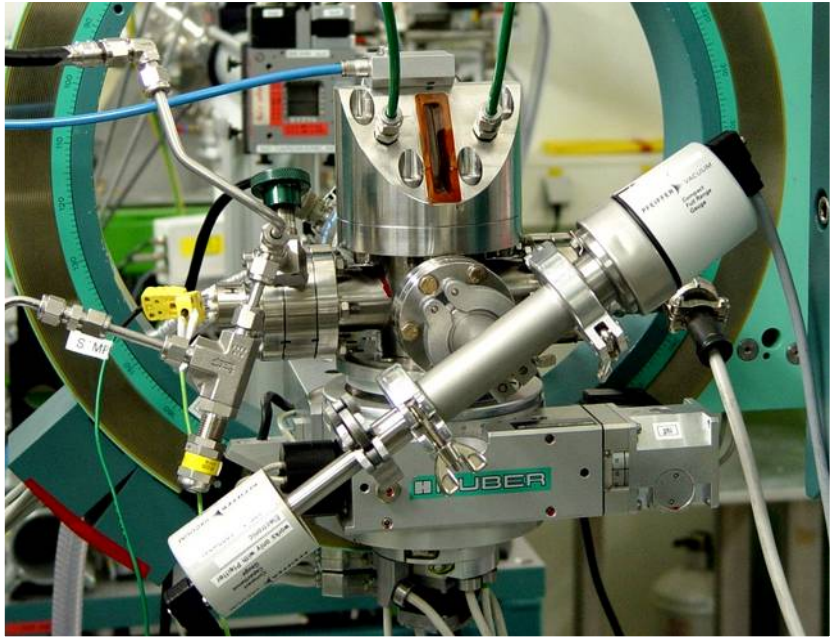
Magnetron deposition chamber



HT annealing chamber +
sheet resistivity measurement

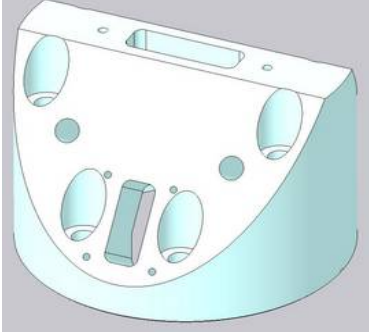


HT gas chamber suitable for
reactive gases, P ~ 1bar



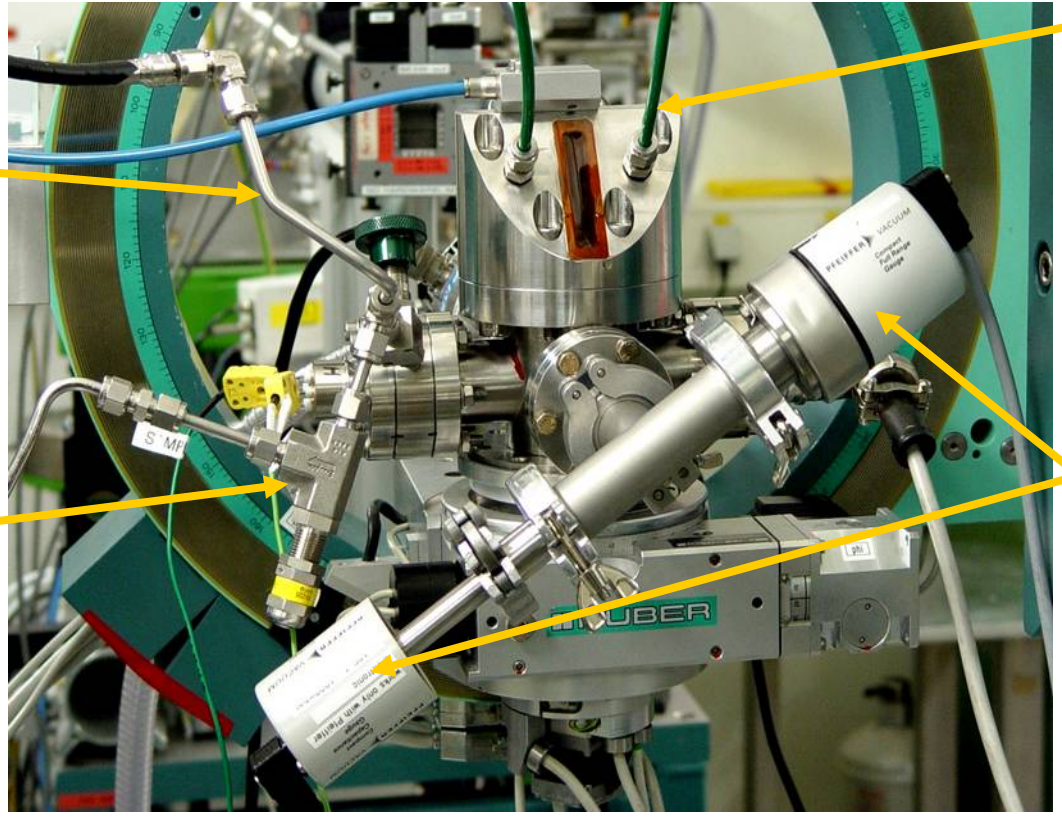
process chambers from HZDR (ROBL) transferred to Petra/P23

High temperature gas chamber



Be-dome: >10^{-7} mbar; 1400°C or 200 mbar @ 900°C

- Kapton dome: up to 800 mbar over pressure @ 900°C
- gas flow unit and pressure regulation, Ar, CH_4, H_2, C_2H_2 ….
- Optical port



Vacuum system ready for usage at P23

The in-situ x-ray deposition system

Deposition chamber

- p_{base} ~ $8*10^{-7}$ mbar
- Up to 3 1“ magnetrons
- 2 optical ports, port for XFLASH
- Target substrate distance: 10 cm
- **Separate test system available**

Deposition parameters

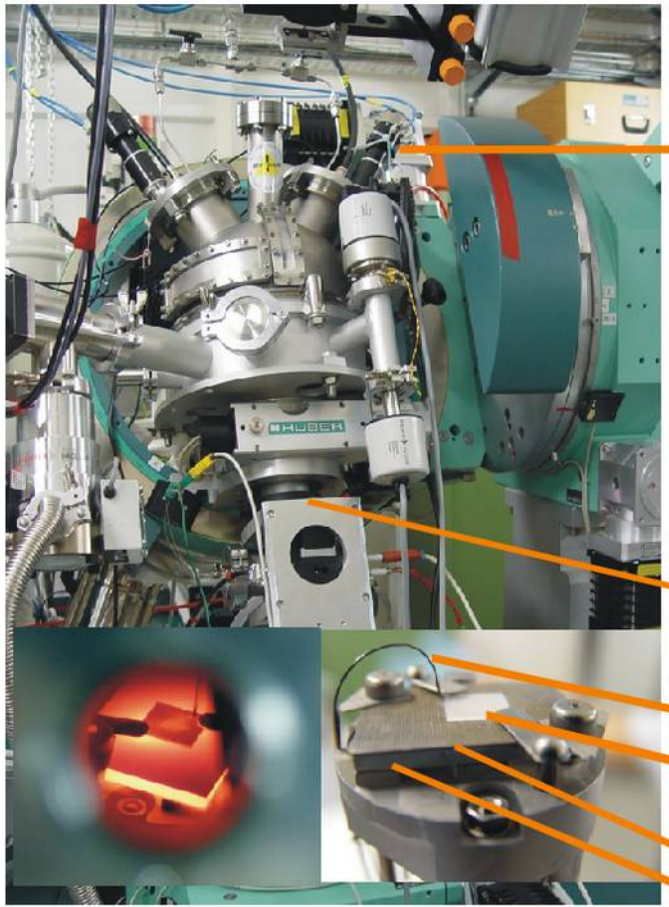
- $p_{deposition}$ ~ $3\text{-}8*10^{-3}$ mbar
- Sputter gas: Ar + N2
- AC or DC supply possible
- **Substrate temperature up to 800°C**

Methods

XRD, XRR, GID, GIXS, GISAXS,

Integration into the P23 diffractometer not finished

The in-situ x-ray deposition system



Integration into the P23 diffractometer not finished

Thank you

very much!