Physikalisch-Technische Bundesanstalt
Braunschweig und Berlin
Nationales Metrologieinstitut

UHV instrumentation and alignment strategies

- XRS instrumention -

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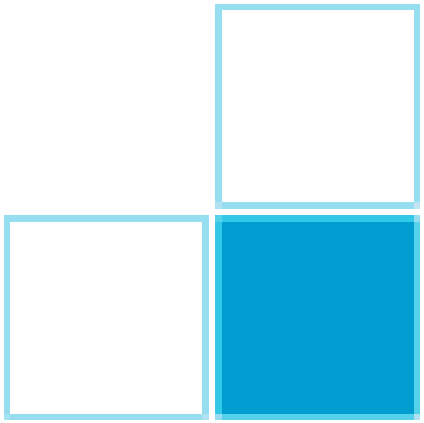
Joint ICTP-IAEA School on
Novel Experimental Methodologies for Synchrotron Radiation Applications in Nano-science and Environmental Monitoring

17 November - 28 November 2014



Outline

X-ray and IR Spectrometry

1. Motivation
2. PTB´s development of an UHV instrument for XRS/XRR nanoanalytics
3. Technology transfer to TU Berlin, CEA/LNHB and IAEA
4. IAEA measuring chamber – measurement options
5. Characterization of the IAEA measuring chamber
6. Principles of sample alignment
7. Operational recommendations
8. PTB control software

1. Motivation

- Ever decreasing sizes of components and the ever greater complexity of electronic assemblies require an analyzing method for ever decreasing mass depositions and elemental concentrations as well as ever decreasing layer thicknesses.
- Increasing demands on quality for nanoscaled materials:
  - Fast non-destructive procedure
  - High accuracy
  - High reliability

  } **X-Ray Spectrometry (XRS)**
- Lack of reference materials at the nanoscale

Calibrated instrumentation + Well-known atomic data + Well-known exciting radiation = Reference-free XRS techniques

2. PTB´s development of an UHV instrument for XRS/XRR nanoanalytics

X-ray and IR Spectrometry

E_0 = photon energy of excitation radiation

E_f = photon energy of fluorescence radiation

XSW = X-ray Standing Wave field

All methods implemented in only one instrument

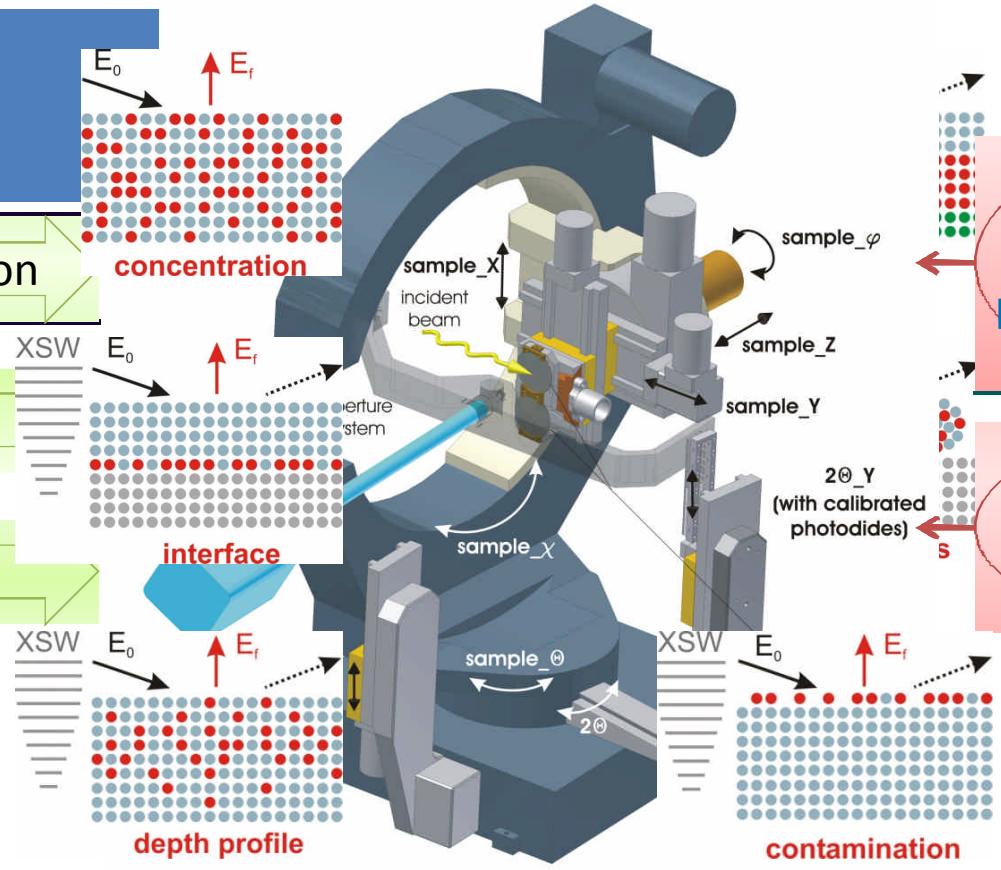
XRF

GIXRF

Synchrotron radiation

Plasma source

X-ray tube



+ Reference-free techniques with low uncertainties

+ Polarization dependent measurements

In operation in the PTB laboratory at BESSY II since January 2011.

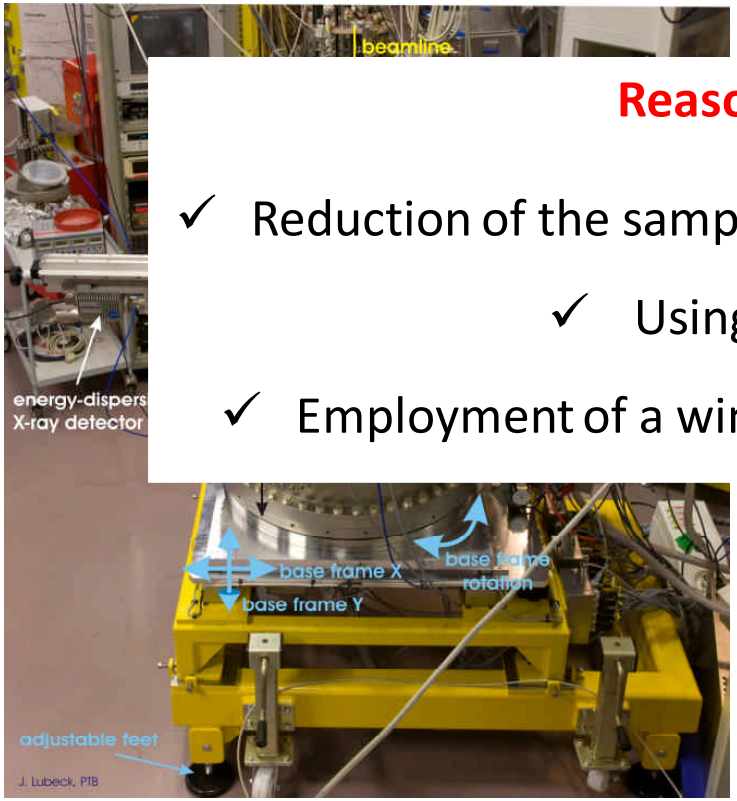
GIXRF + XRR

TXRF

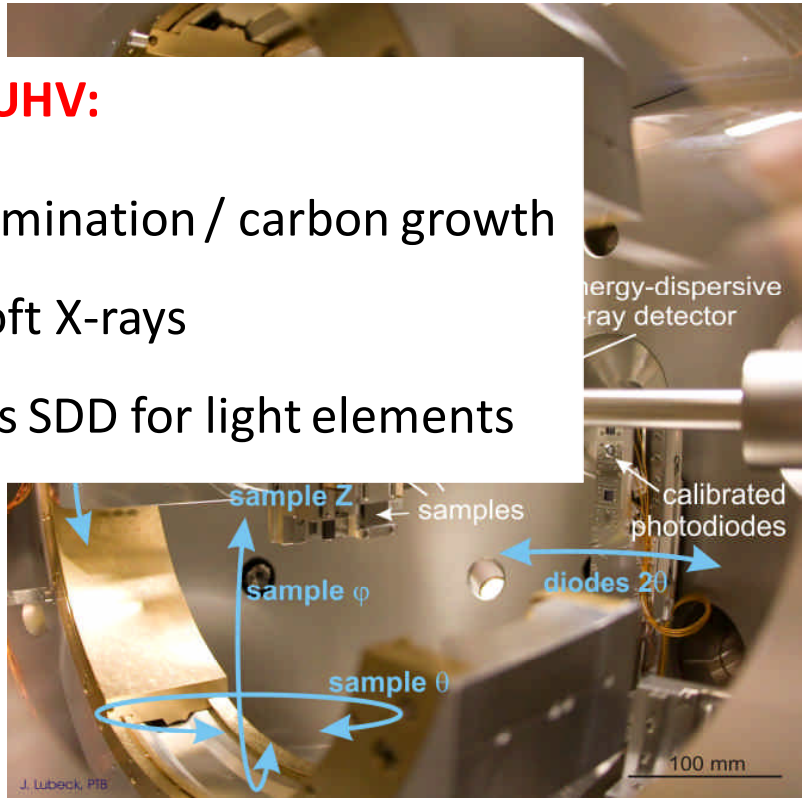
J. Lubeck et al., Rev. Sci. Instrum. 84, 045106 (2013)
JAAS 23, 845 (2008)

2. PTB´s development of an UHV instrument for XRS/XRR nanoanalytics

UHV chamber at PTB´s PGM-beamline at BESSY II



9-axis sample manipulator



Reasons for UHV:

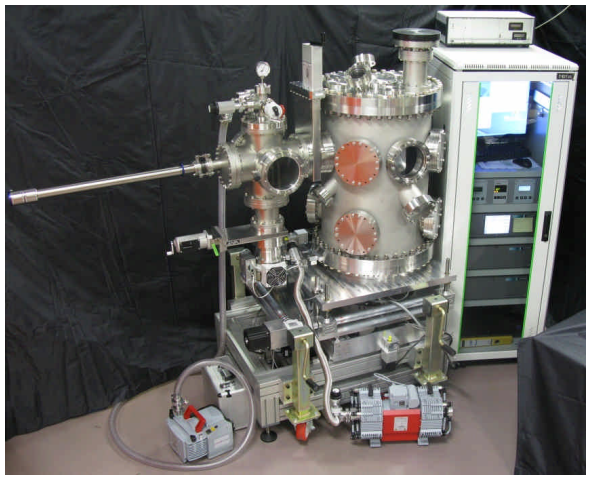
- ✓ Reduction of the sample contamination / carbon growth
- ✓ Using very soft X-rays
- ✓ Employment of a windowless SDD for light elements

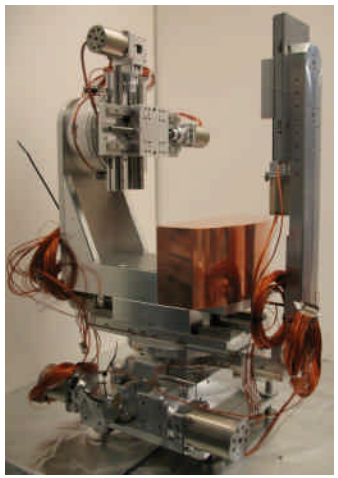
Designed by PTB to match various different project requirements with high flexibility and reliability.

3. Technology transfer to TU Berlin, CEA/LNHB and IAEA

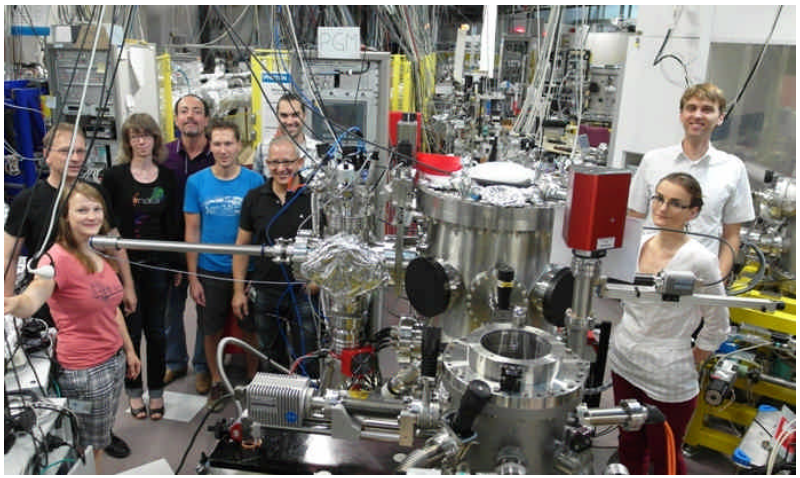
X-ray and IR Spectrometry

TU-Berlin:



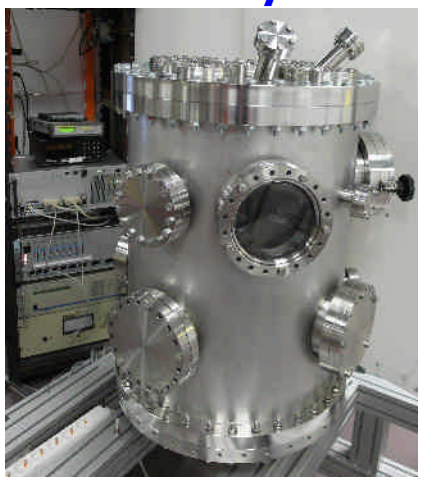


IAEA-instrument at PTB´s PGM-beamline:



- Transfer to TU Berlin and with TU Berlin to IAEA and LNE-LNHB successfully completed
- <u>Differences to PTB instrument:</u>
  - 7-axis manipulator instead of 9-axes
  - without aperture system reference-free measurements with moderate uncertainties possible
  - polarization dependent measurements not possible

LNE-LNHB at synchrotron facility SOLEIL:



3. Contact information: European metrology and XRS facilities

European Association of National Metrology Institutes (EURAMET):

- www.euramet.org/index.php?id=objectives

PTB Technical Cooperation: Europe, Asia, Latin America, Africa and Middle East

- www.ptb.de/cms/en/fachabteilungen/abtq/fb-q5.html

PTB at synchrotron radiation facility BESSY II, Berlin, Germany:

- www.ptb.de/cms/en/fachabteilungen/abt7/fb-72/ag-724.html

LNE-LNHB at synchrotron radiation facility SOLEIL, Paris, France:

- www.nucleide.org
- www.synchrotron-soleil.fr/portal/page/portal/Recherche/LignesLumiere/METROLOGIE

Synchrotron radiation facility ELETTRA, Trieste, Italy:

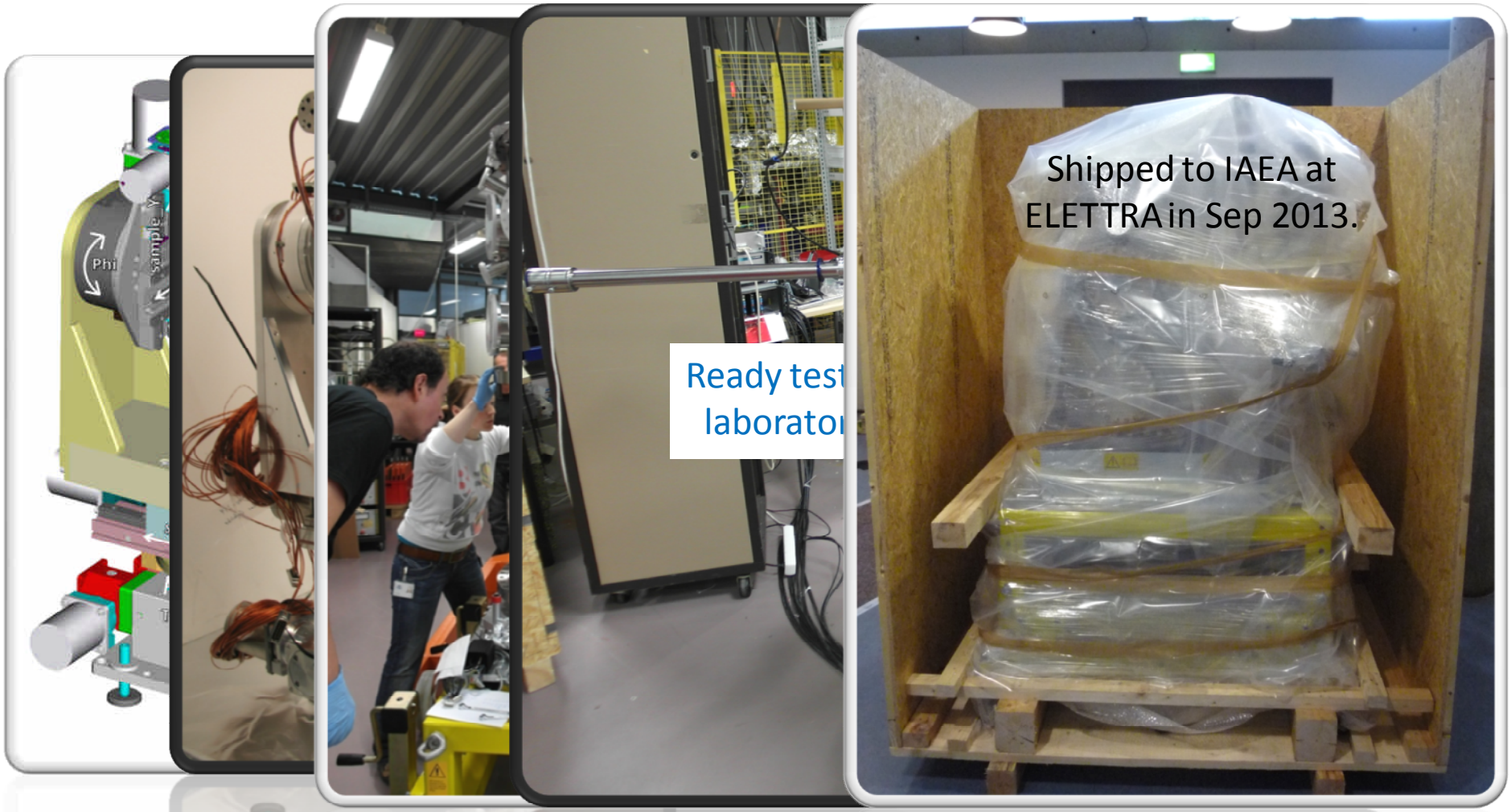
- https://www.elettra.trieste.it/lightsources/elettra/elettra-beamlines/microfluorescence/x-ray-fluorescence.html

Berlin Laboratory for Innovative X-ray Technologies, Berlin, Germany

- http://www.blix.tu-berlin.de/en/index.html

4. IAEA measuring chamber – measurement options

X-ray and IR Spectrometry



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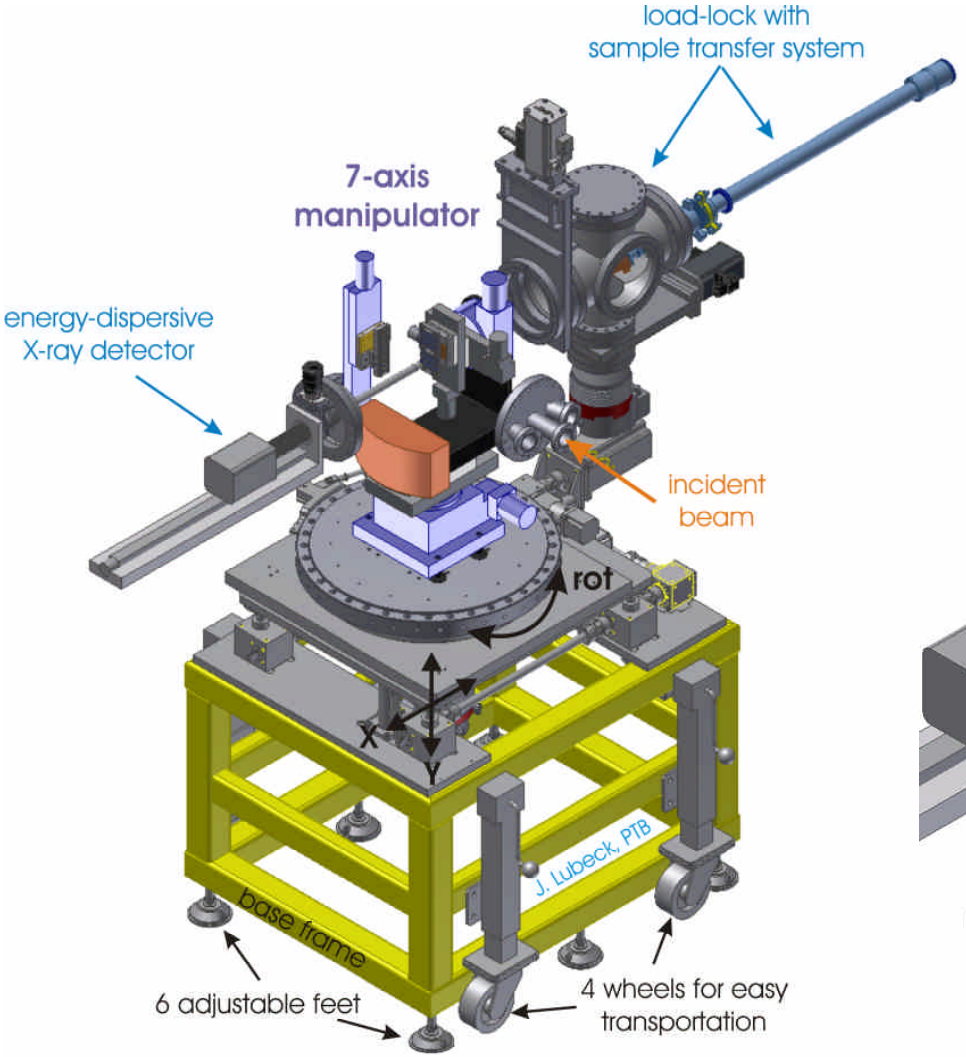
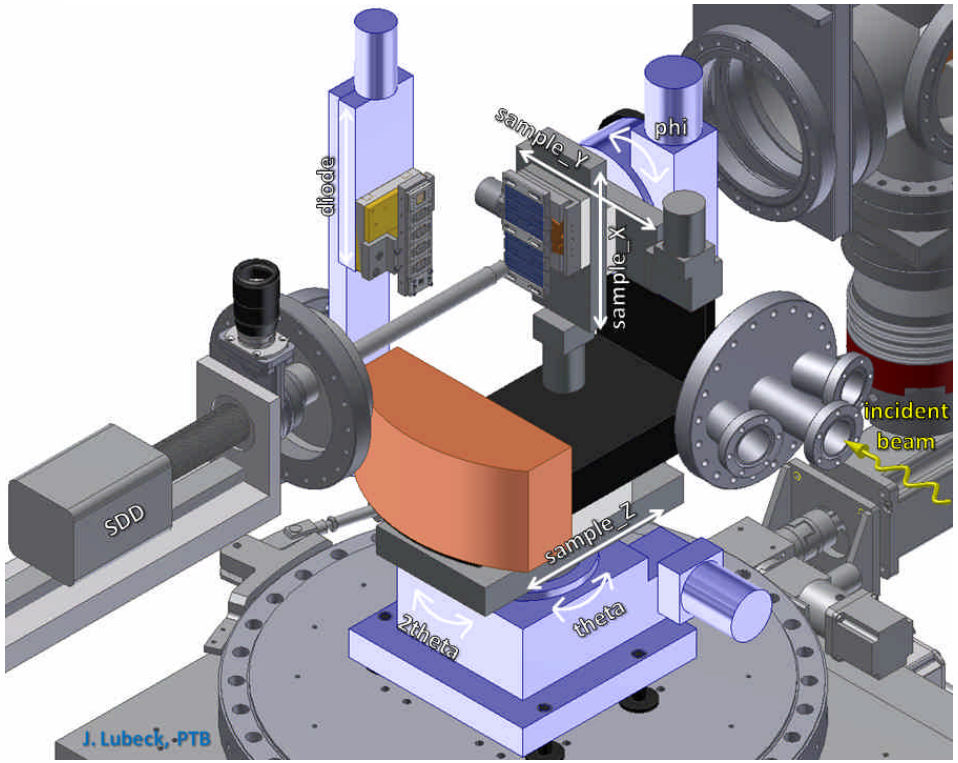
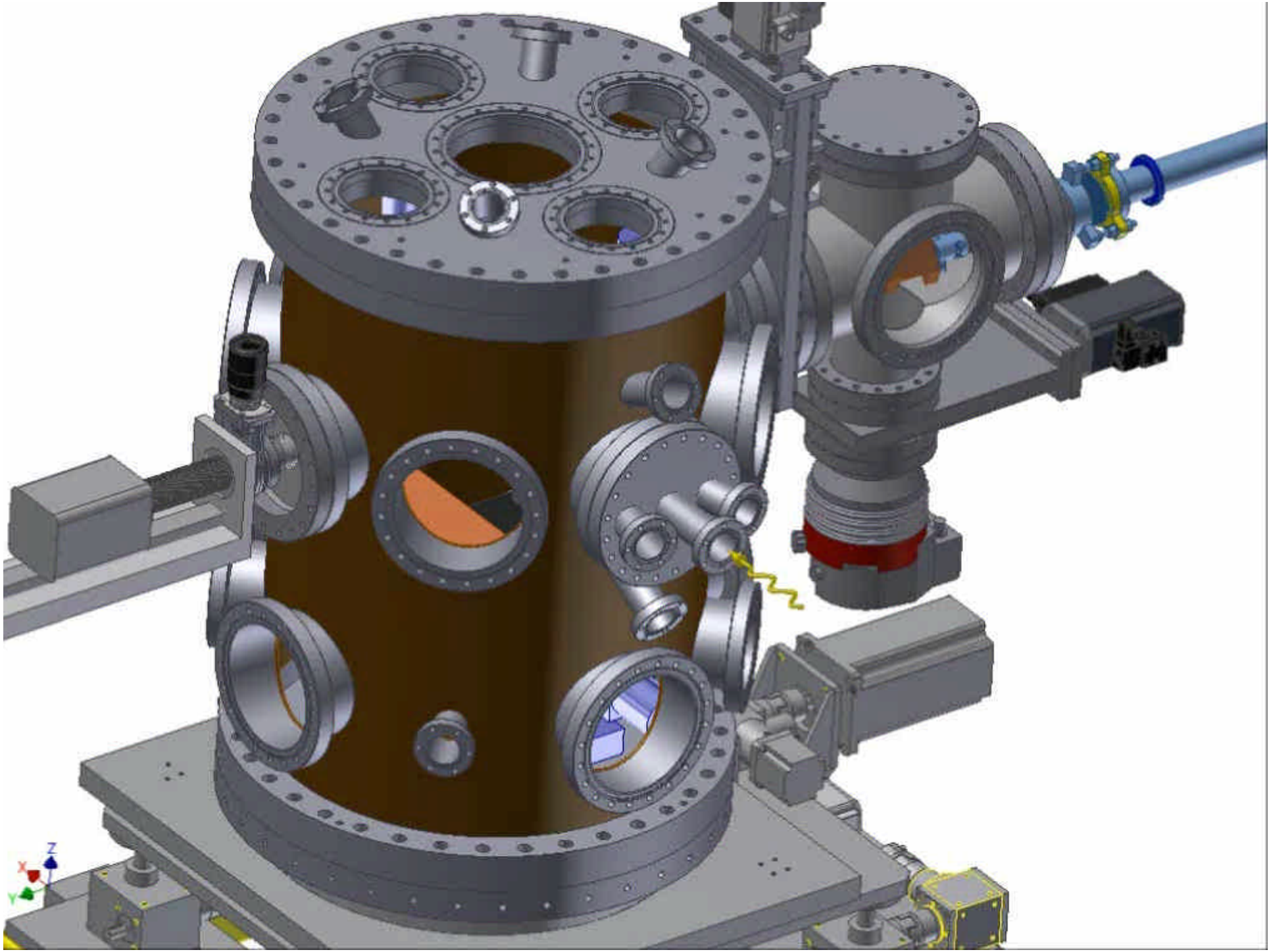
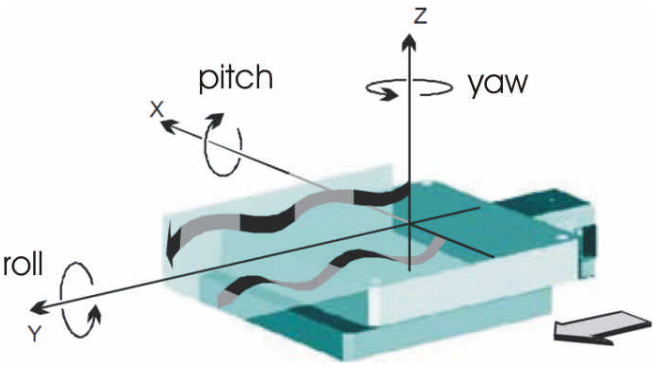




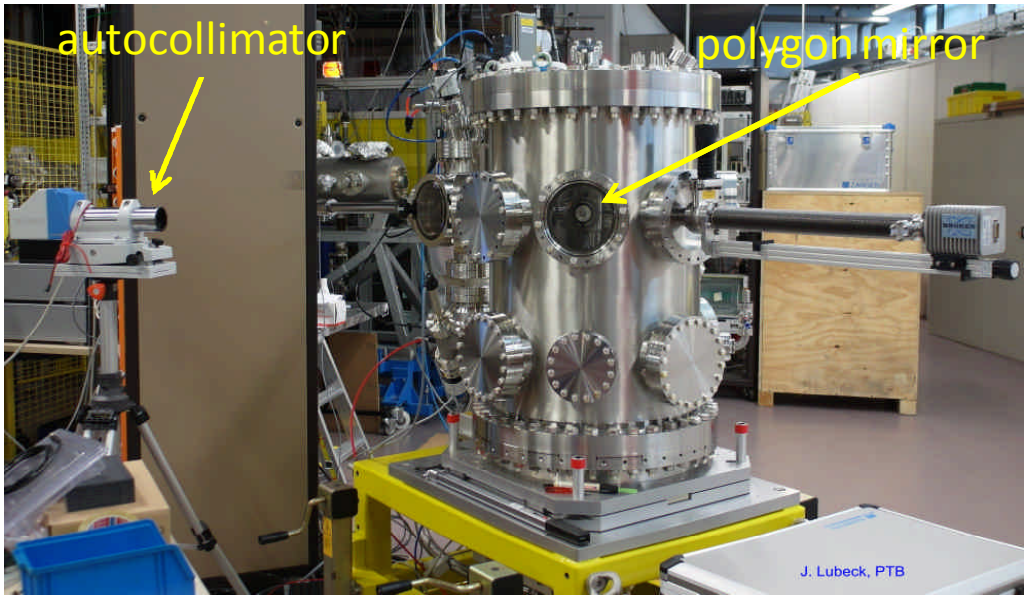

Z
X
Y

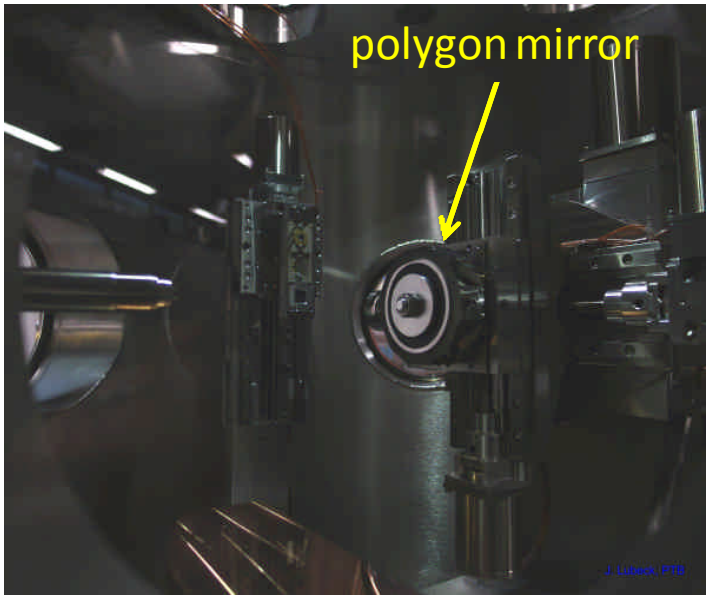
5. Characterization of the IAEA measuring chamber

- XRS or XRR results should not be affected by any artifacts of the sample movement.
- Each axis of the sample manipulator is very well characterized in experimental environment.



Courtesy of Huber Diffraktionstechnik GmbH & Co. KG



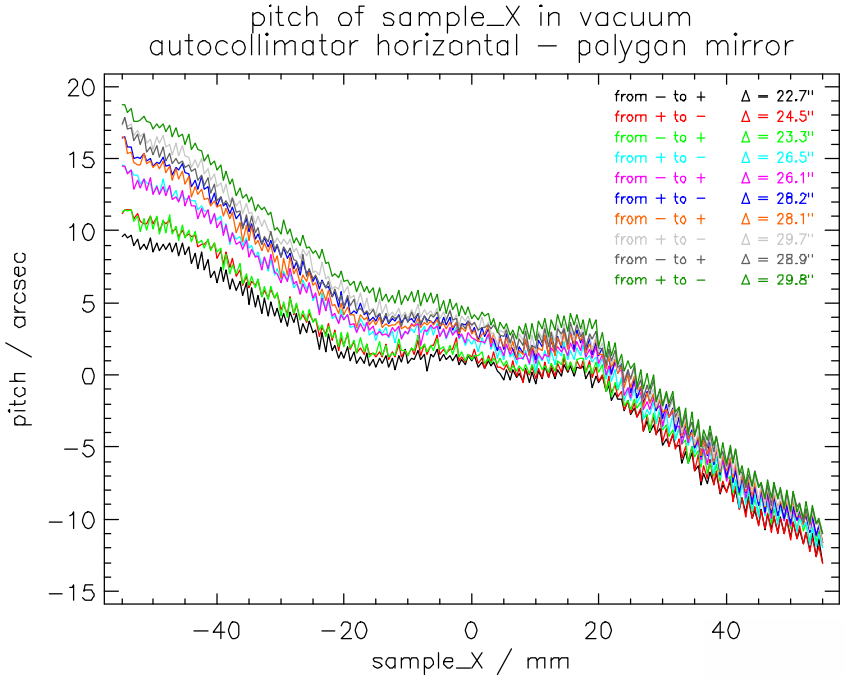


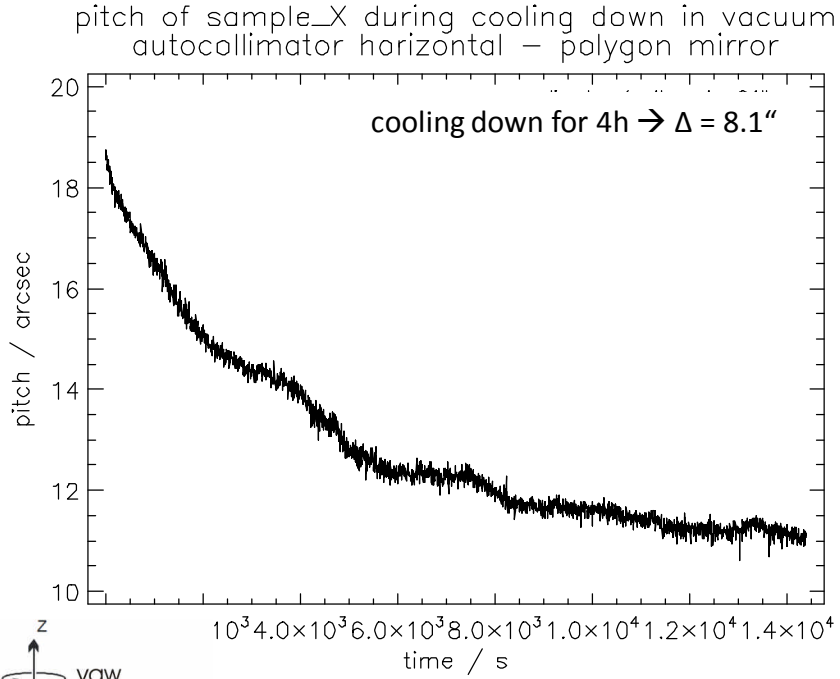
Characterization methodology as in diploma thesis of J. Lubeck, PTB.

3. Characterization of the IAEA measuring chamber

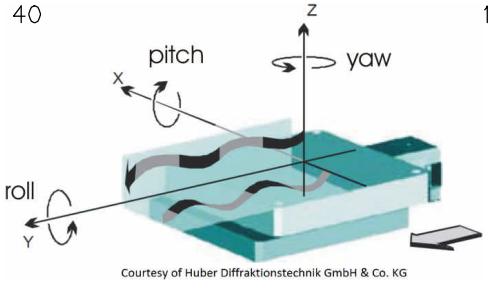
X-ray and IR Spectrometry

Sample_X axis





result = 30”



result = 16”

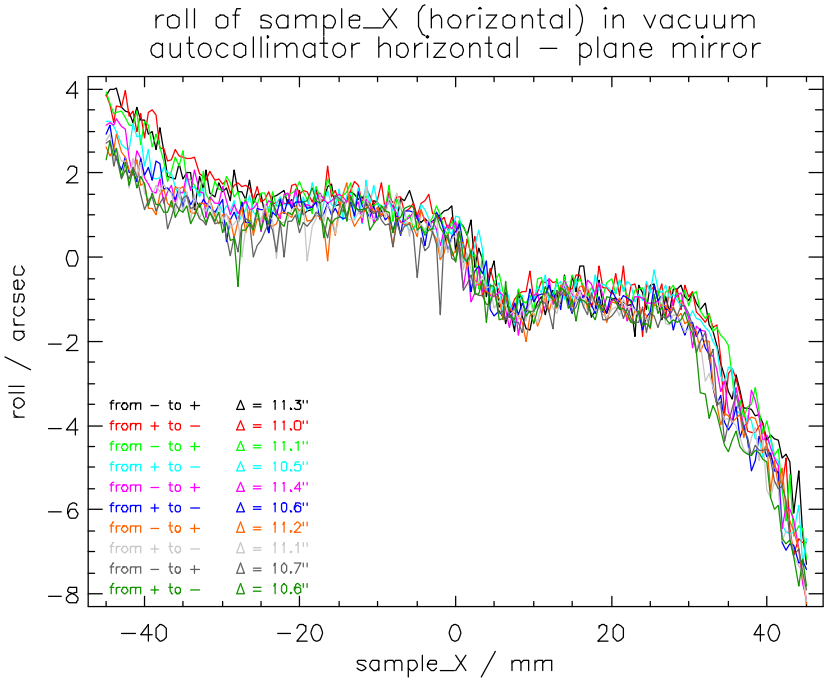
Translation of 110 mm ≙ 0.016 mm deviation

Translation of 110 mm ≙ 0.008 mm deviation

3. Characterization of the IAEA measuring chamber

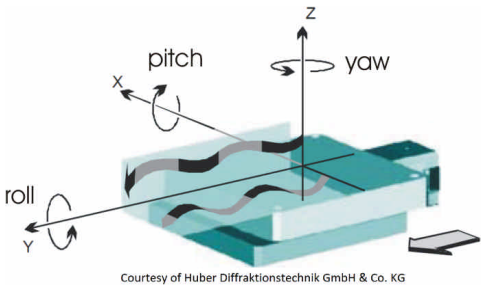
X-ray and IR Spectrometry

Sample_X axis



result = 12”

Translation of 90 mm ≙ 0.005 mm deviation



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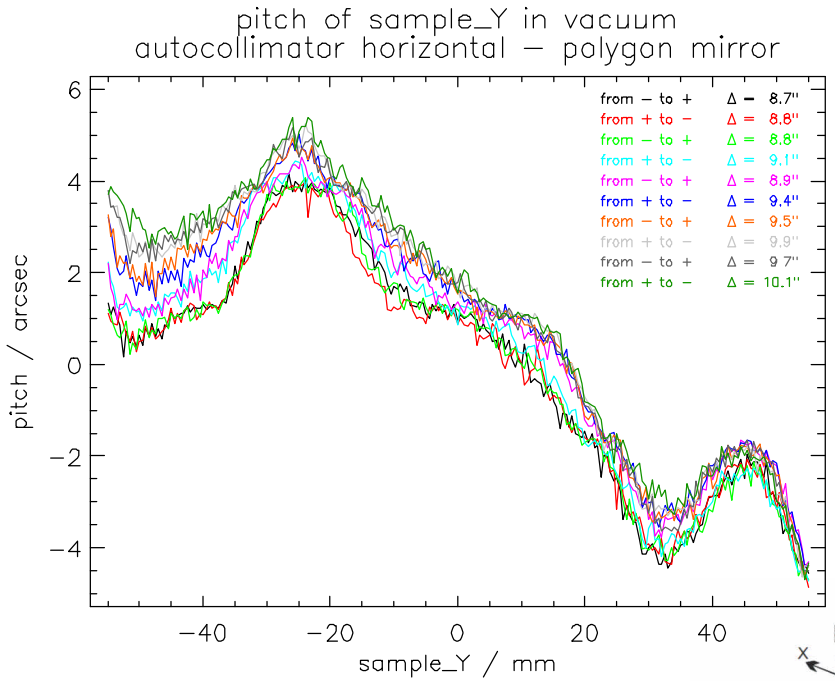
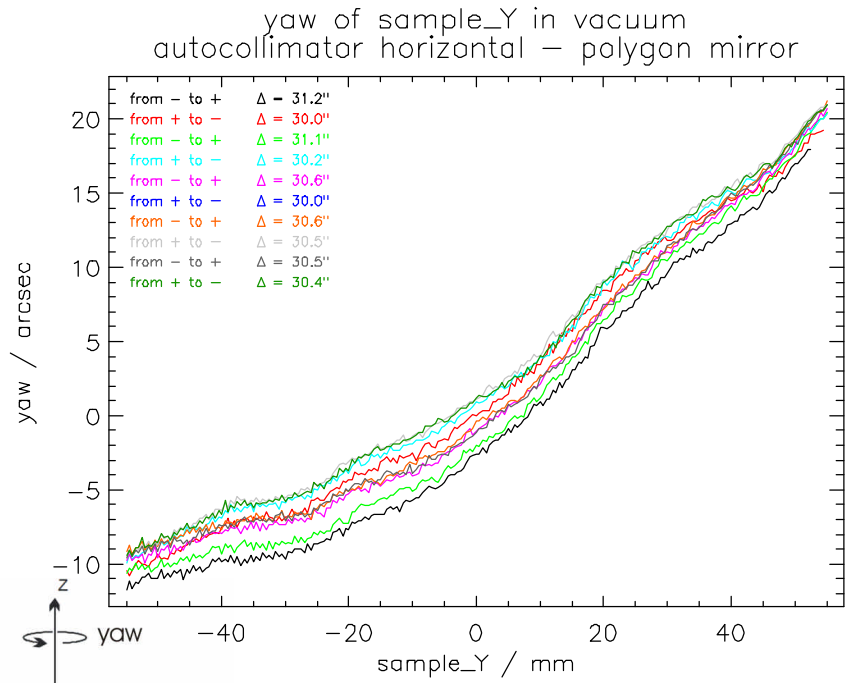
3. Characterization of the IAEA measuring chamber

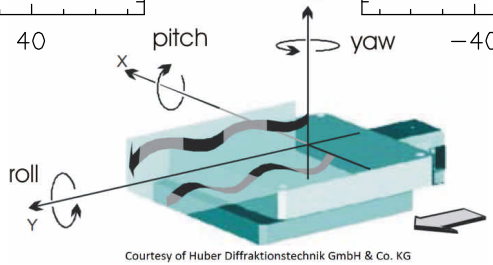
X-ray and IR Spectrometry

Sample_Y axis





result = 10”



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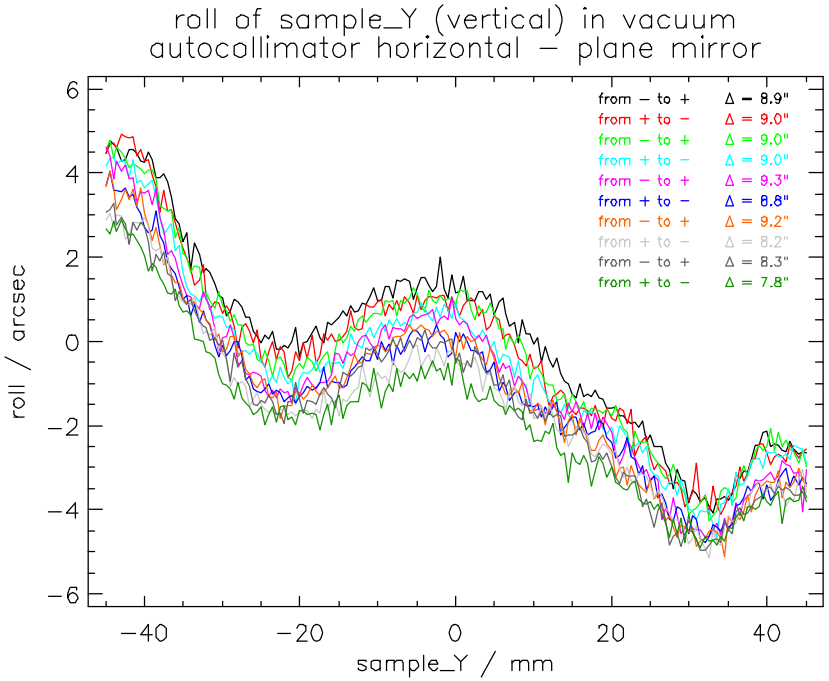
result = 31”

Translation of 110 mm ≙ 0.005 mm deviation

Translation of 110 mm ≙ 0.016 mm deviation

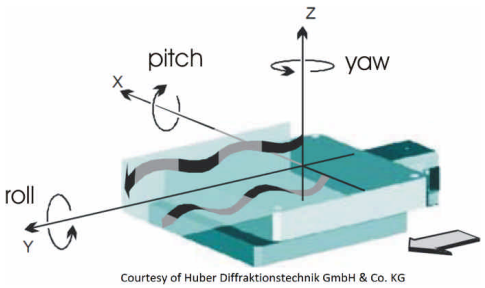
3. Characterization of the IAEA measuring chamber

Sample_Y axis



result = 10”

Translation of 90 mm ≙ 0.004 mm deviation



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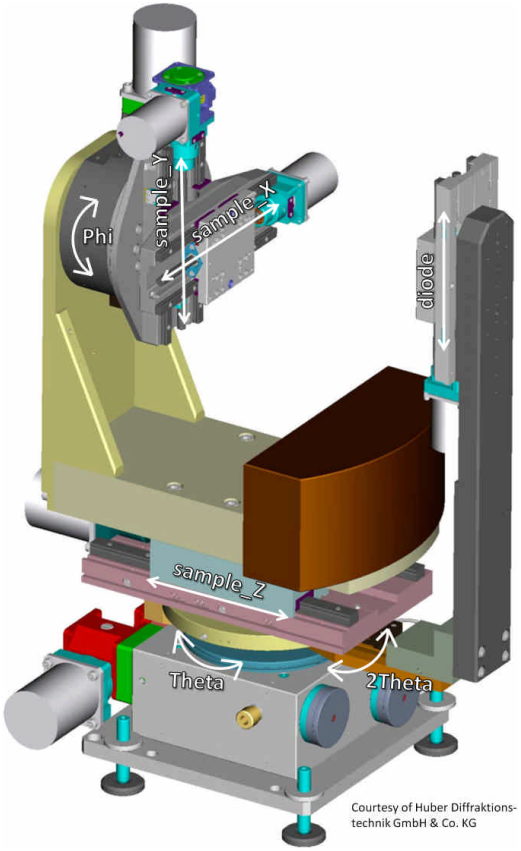
3. Characterization of the IAEA measuring chamber

X-ray and IR Spectrometry

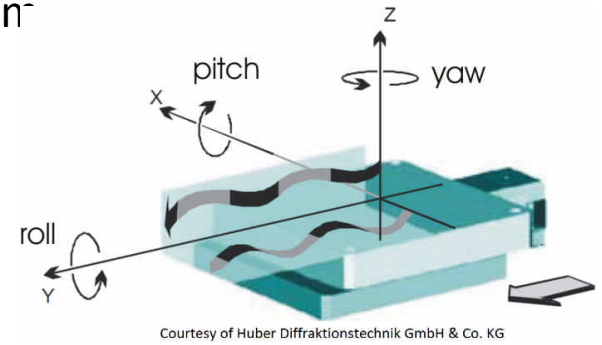
Results of the linear axes of sample movements



	Pitch / "		Yaw / "		Roll / "	
Linear axis	**Horizontal**	**Vertical**	**Horizontal**	**Vertical**	**Horizontal**	**Vertical**
sample_X	30	28	16	15	12	-
sample_Y	10	11	31	14	10	-
sample_Z	30	-	2	-	2	-

→ Characterization delivers very good results

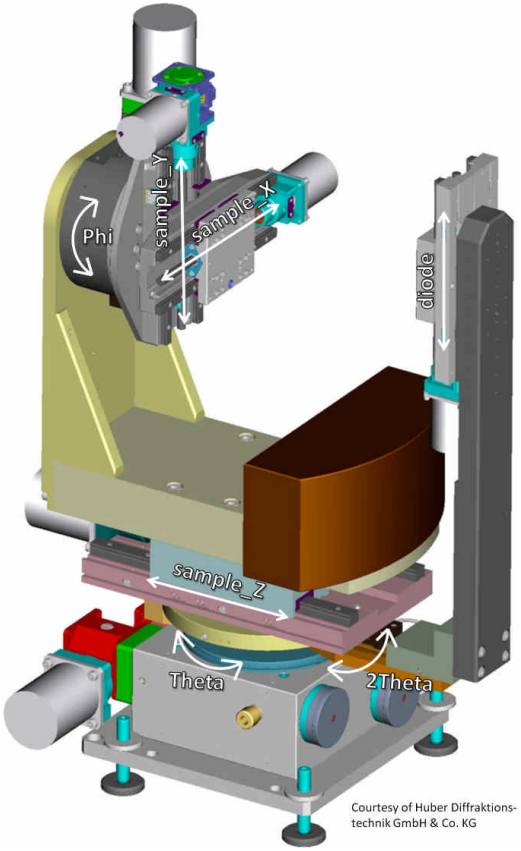
→ During mapping of 20 mm x 20 mm the maximum angle change is 6” which means 0.6 µn



3. Characterization of the IAEA measuring chamber

X-ray and IR Spectrometry

Results of the rotational axes

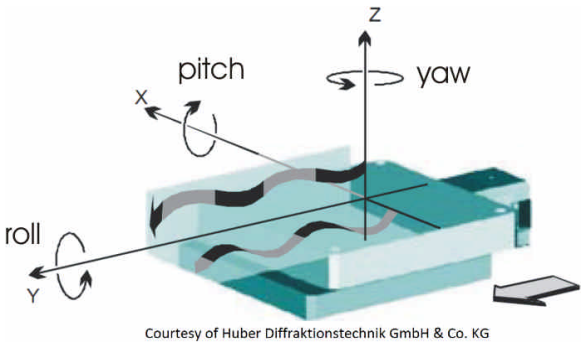


Rotational axis	Repeatability (unidir.) / “	Reversal error / “	Optimal backlash distance / °
Phi (at 0° while moving from -150° to 150°)	2.5	3.1	0.3
Theta (at 15° while moving from -105° to 75°)	2.3	4.2	0.1
2Theta (at 30° while moving from 0° to 150°)	7.0	9.4	0.1

→ Characterization delivers very good results

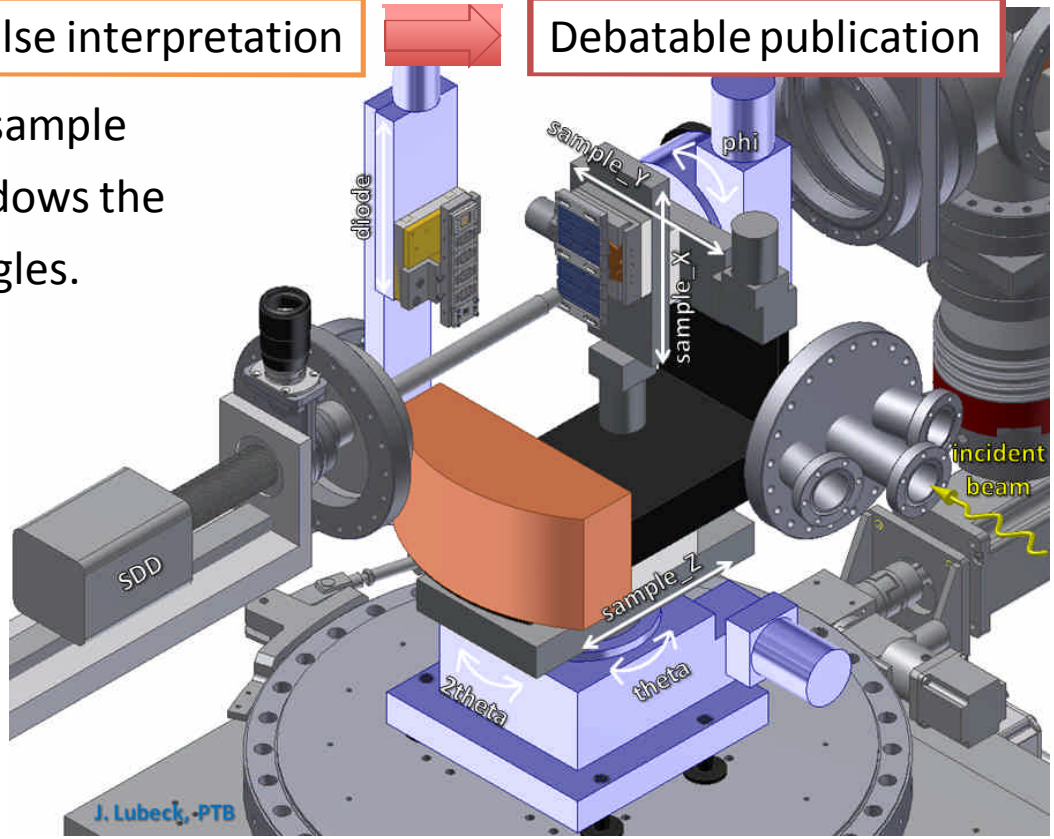
→ All measurements were performed without using encoders

→ 10 repeated runs

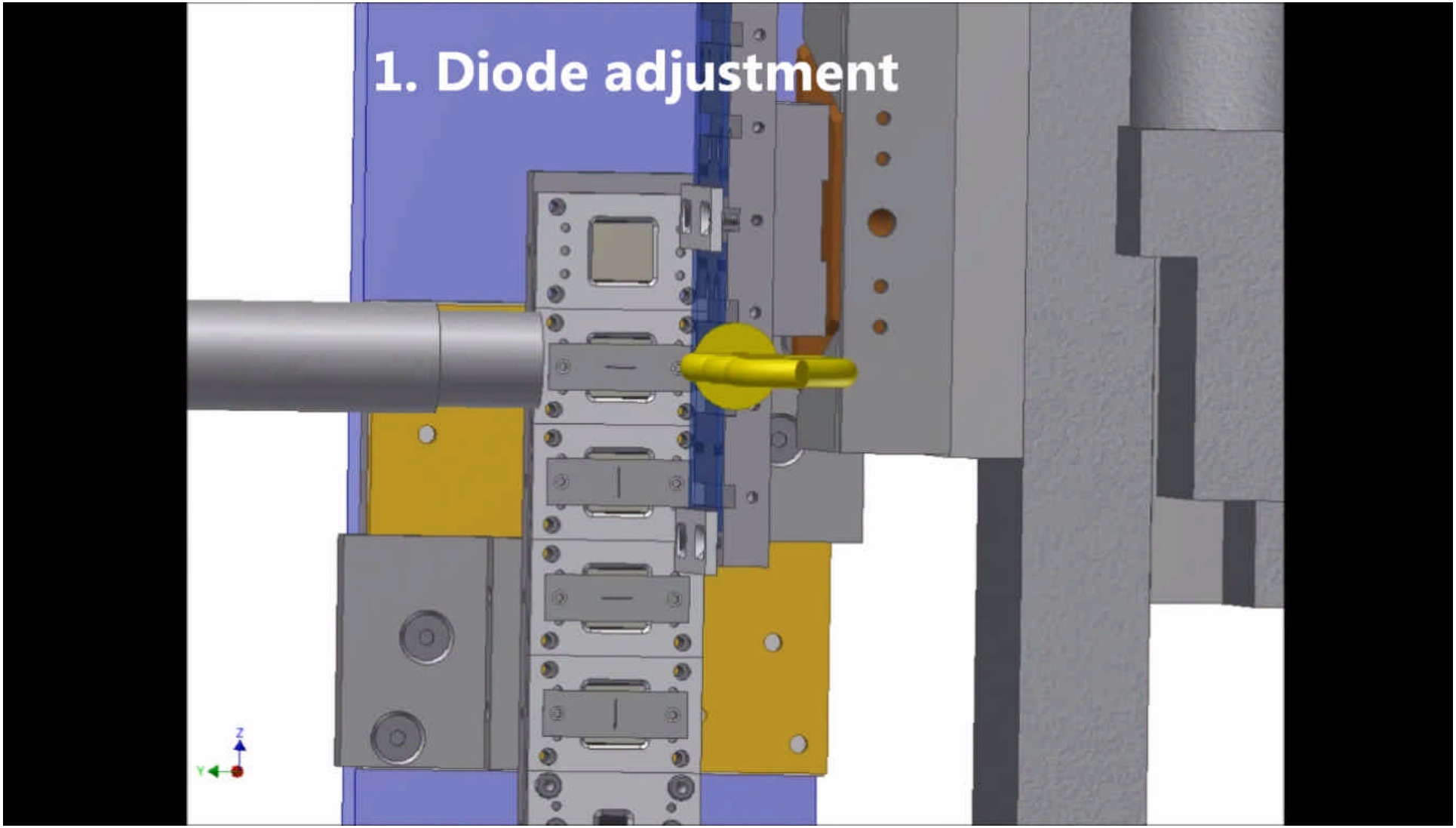


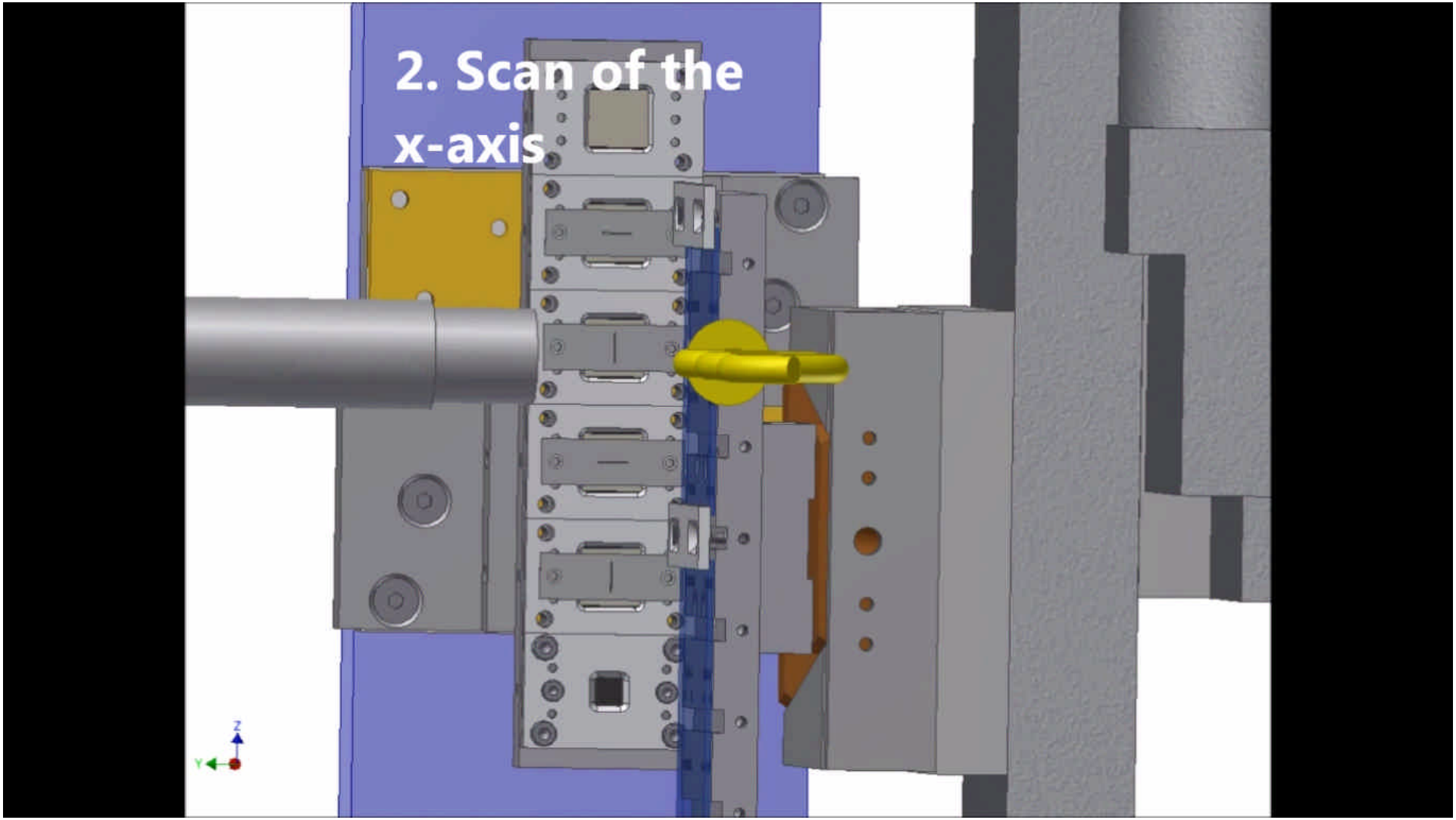
6. Principles of sample alignment

1. ~~Aligning sample_X by means of shadowing effects.~~
   Wrong aligned samples → Unusable results → Lost beamtime
2. Zero angle determination: s… e_Z scan at different theta positions to determin… the theta angle where the sample has the smallest thickn…
   OR a theta scan in the middle of the sample (sample_Y), whereas the sample shadows the photodiode at lower/higher theta angles.
   False interpretation → Debatable publication
3. Positioning of theta at the zero angle → sample_Z scan → edge
4. Aligning sample_Y by means of an SDD scan.
5. Phi only needs to be aligned for structured samples.



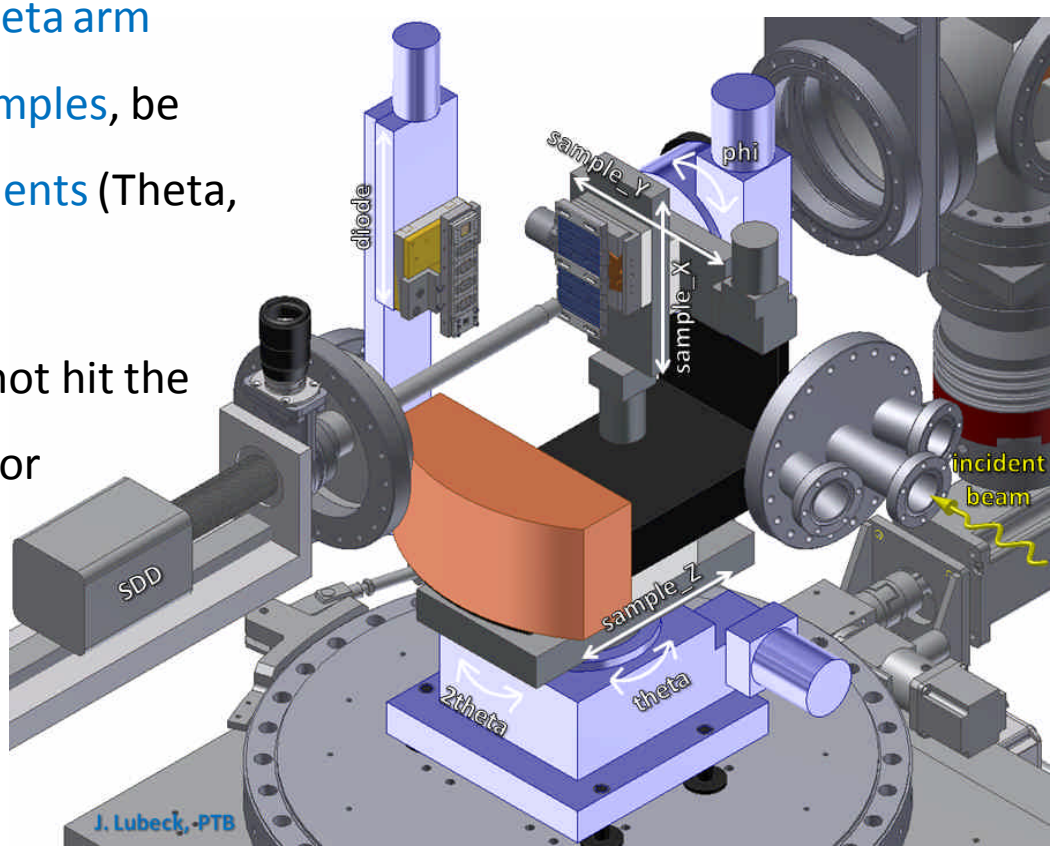
Procedure for sample aligment


1. Diode adjustment


2. Scan of the
x-axis
Z
Y

7. Operational recommendations

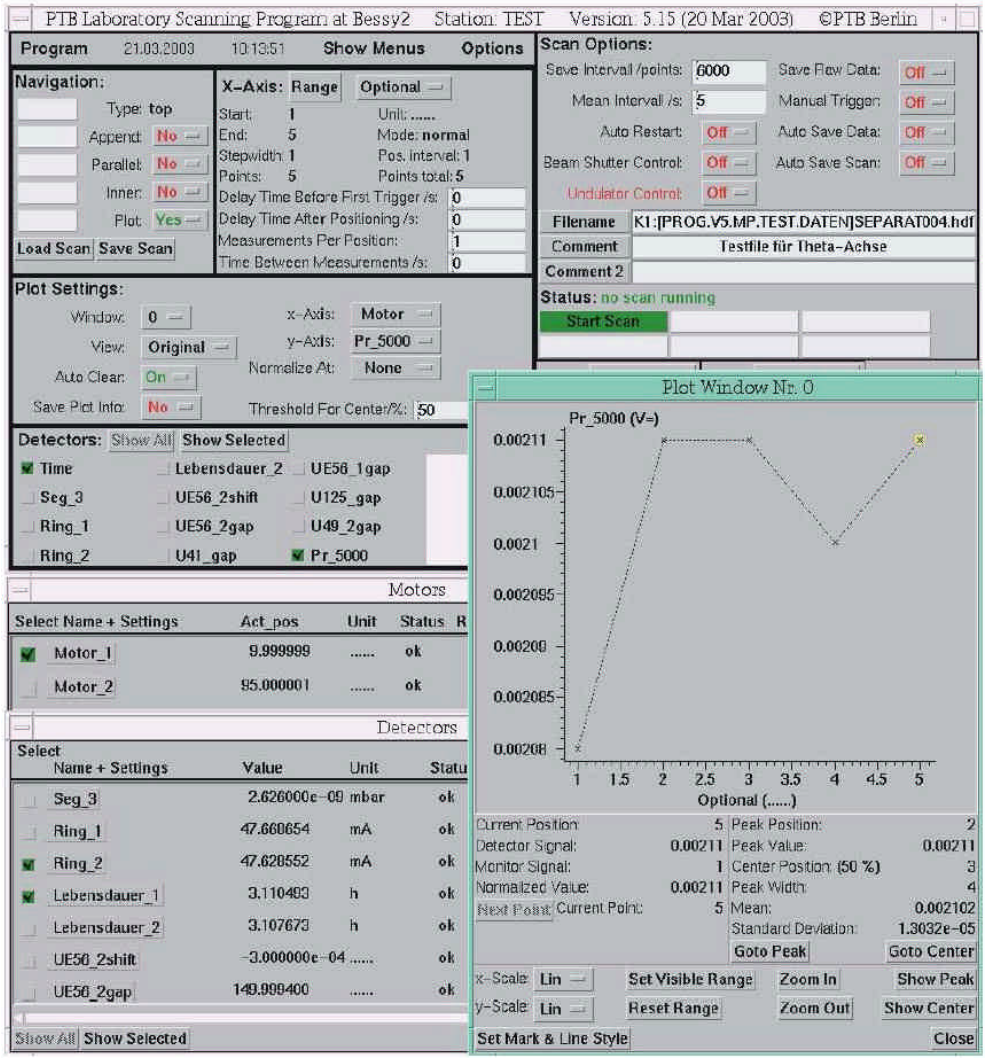
- Do not move all axes at the same time!
- Attention during measurements involving the SDD
  - Do not cut the SDD with the 2Theta arm
  - If the SDD is very close to the samples, be very careful with sample movements (Theta, X, Y, Z & Phi)
- Attention during Phi rotations → do not hit the diode arm with a motor of sample_X or sample_Y



8. PTB control software

X-ray and IR Spectrometry

- Modular structure with GUI
- Main window, windows for detectors, motors, online plot
- Saving data of storage ring, beamline and experiment
- Append, parallel and inner scans with changing detectors/motors
- “Go to” functions during and after scans
- Scan normalized on a reference scan



Thank you for your attention!

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