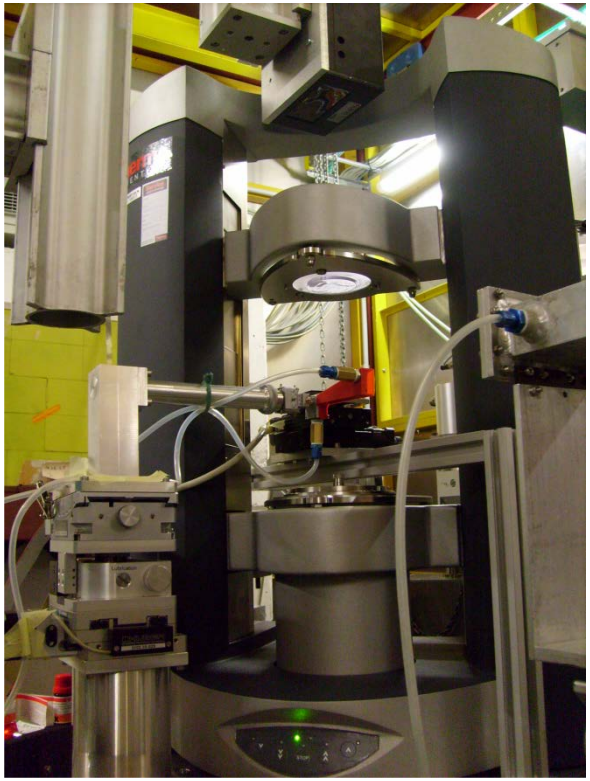
# A new approach to rheology and X-ray scattering at DESY

Bernd Struth



Haake Mars II rheometer as installed at beamline BW1, Doris III

## Why rheology with X-rays

There is lack of knowledge about microstructures of fluid materials when rheology is applied

Could open a new market for DESY as a user facility?

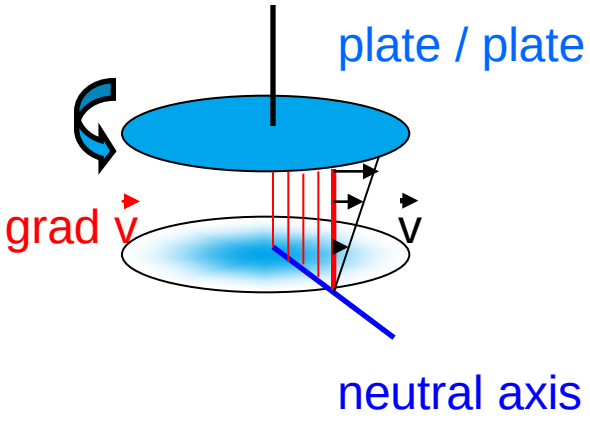
~ 80 universities in Germany

~ 40 ➡ rheology

~ 40 % of MPI‘s

~ 75 % of Fraunhofer

In industrial processing

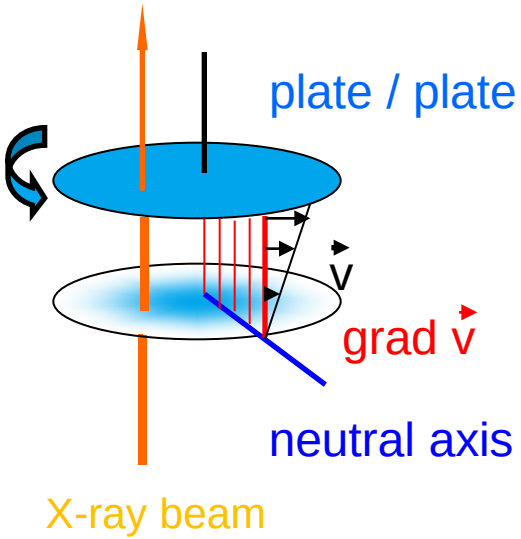


## 2) cone / plate, plate / plate with vertical X – ray beam

Advantage:

Apply the full range of rheological tests available for plate / plate and cone / plate and other possible geometries

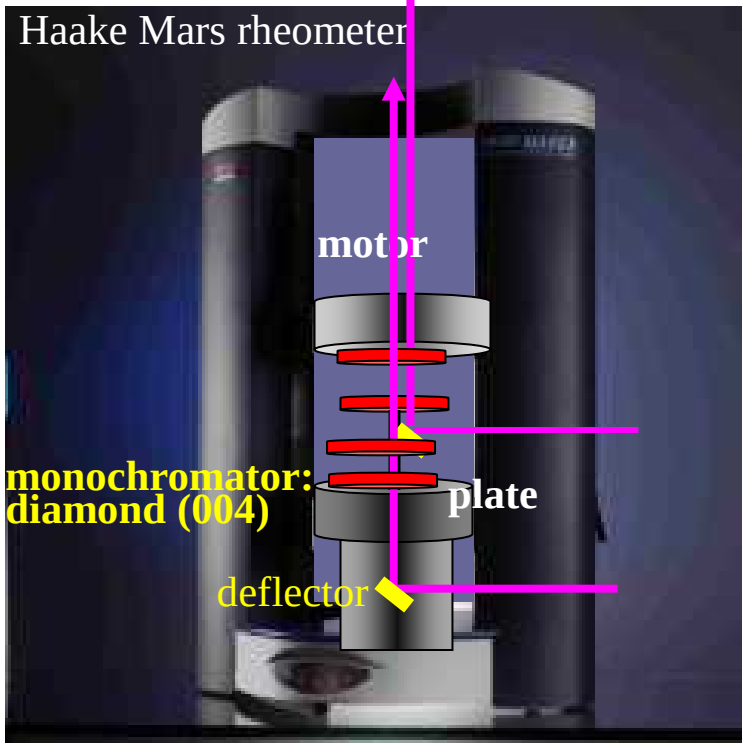
The vertical X-ray beam allows to probe the sample in a very well defined way along the velocity gradient



# *Vertical beam +*

## *Plate / Plate*

2d detector

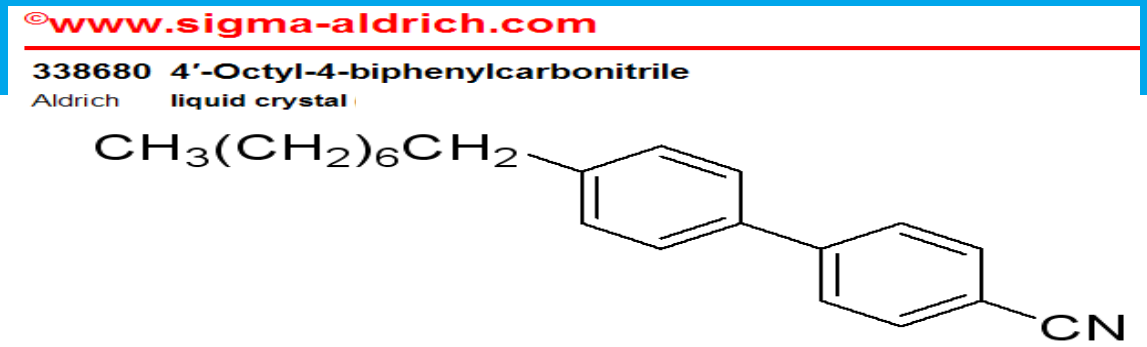



motor blocks access of beam

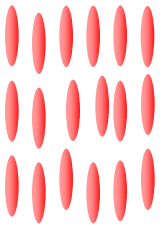
inverted geometry

# Liquid Crystal

(8CB)




Smectic 21.5 C – 33.6 C



Our plan: 2 experiments

*in situ*

Constant shear rate
(alignment
under shear)

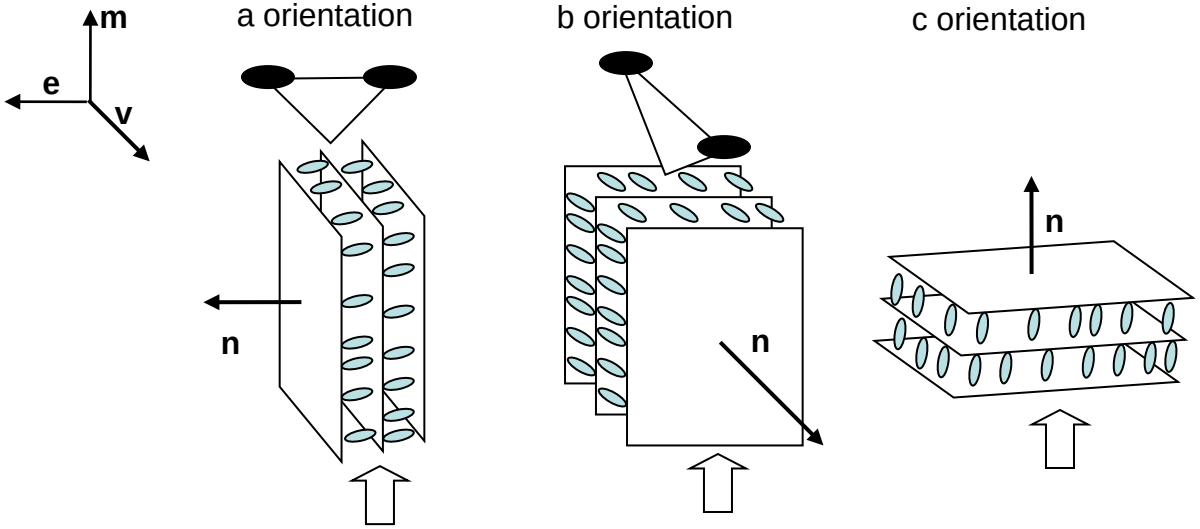
Large amplitude oscillatory shear (LAOS)

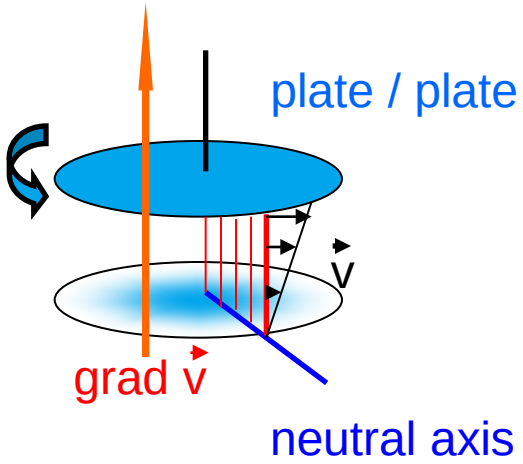
(prediction:
alignment due to shear)

HELMHOLTZ GEMEINSCHAFT

DESY

Smectic A phase of 8CB under steady shear:

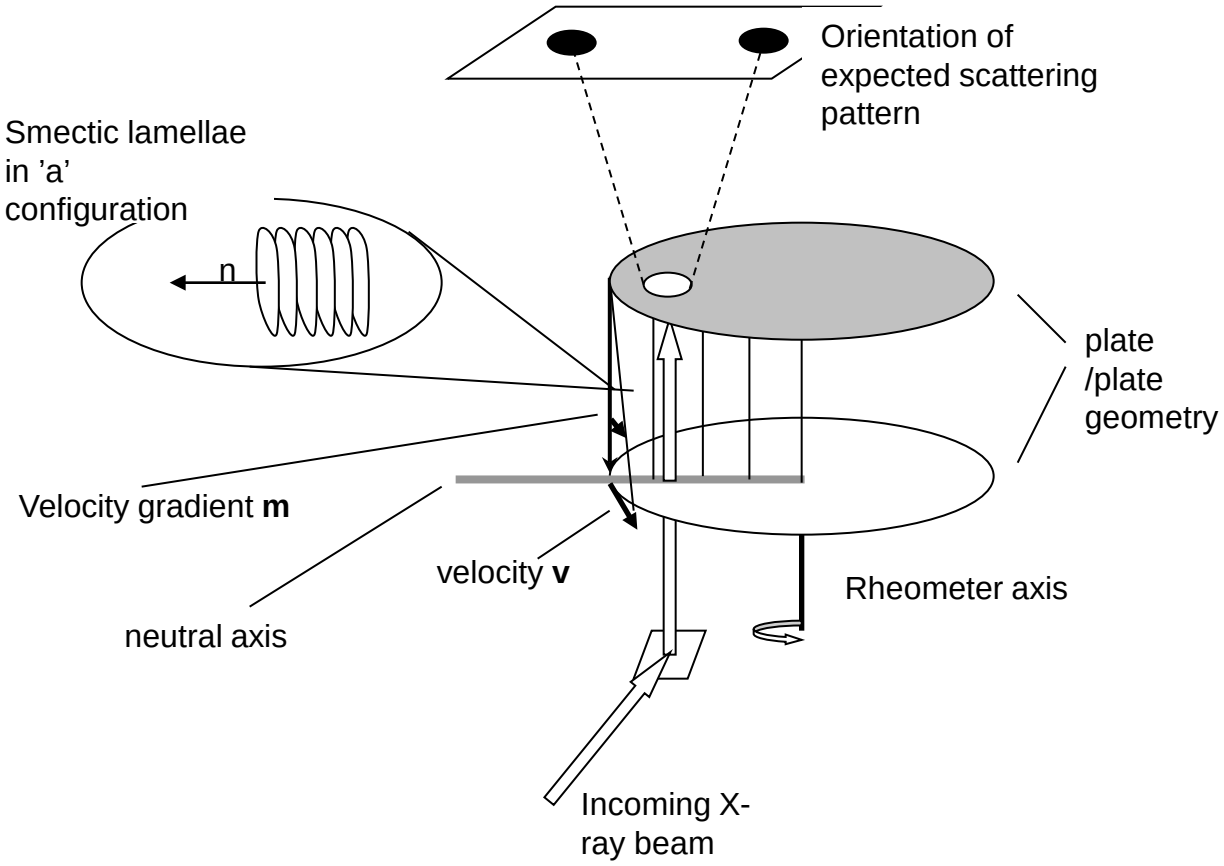



Under steady shear: A orientation was observed
in couette cell (Safinya et.al.)

In plate / plate geometry: C orientation **not** visible in vertical geometry
(Laue equations)

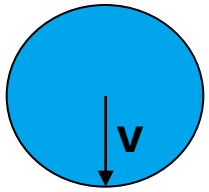
# Scattering geometry for 8CB



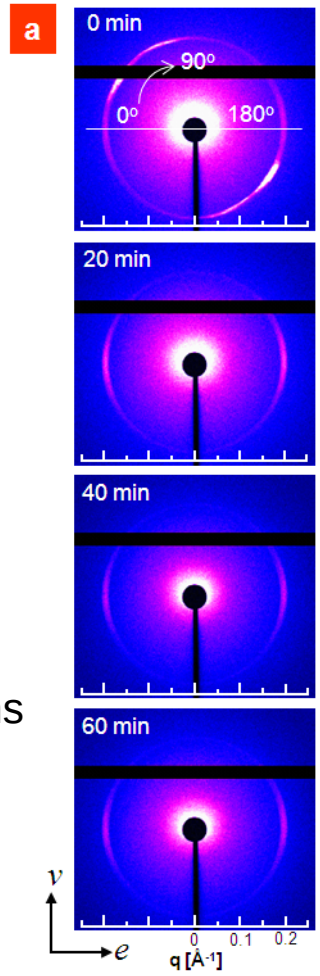
# Results: steady shear

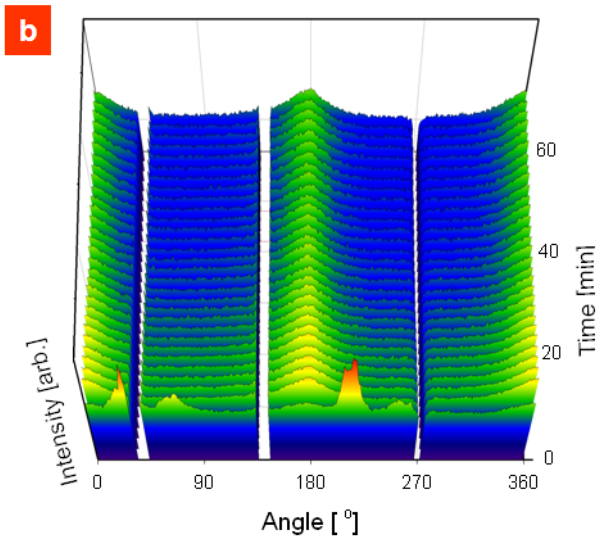
After quenching from isotropic phase
Sample is prealigned

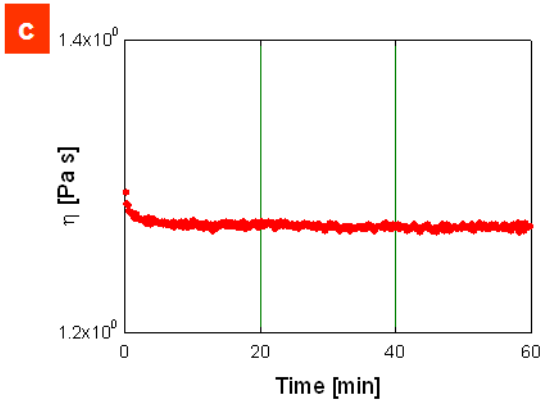
due to squeezing during the loading process (A/B orientation)

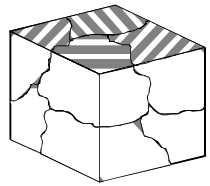



Beam probes few domains (domain size)

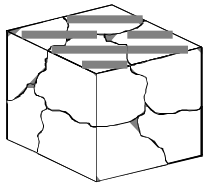


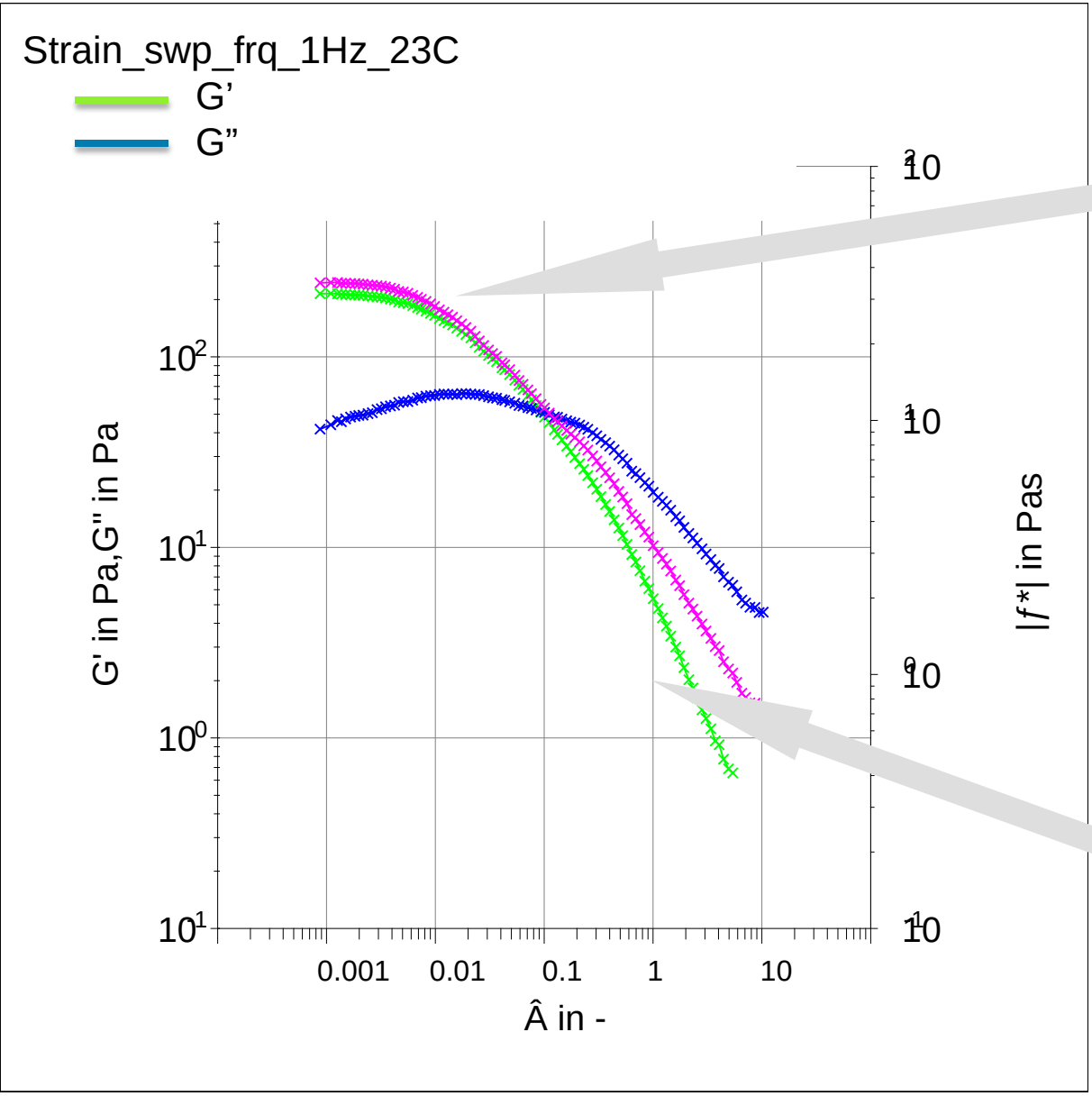



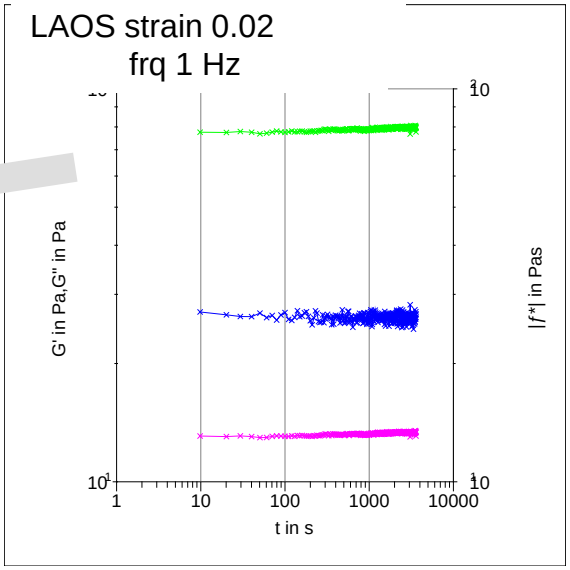



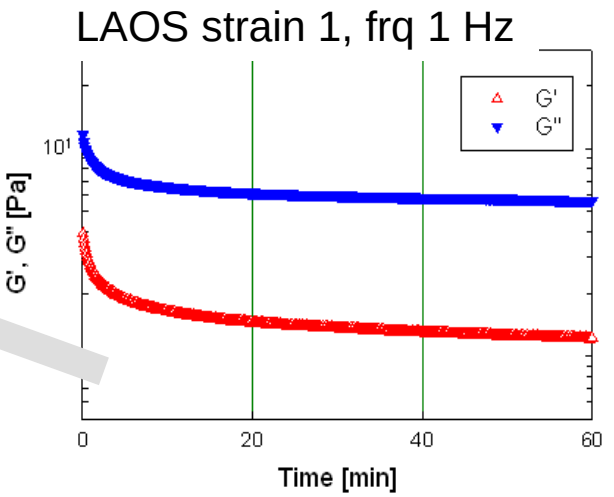
shear



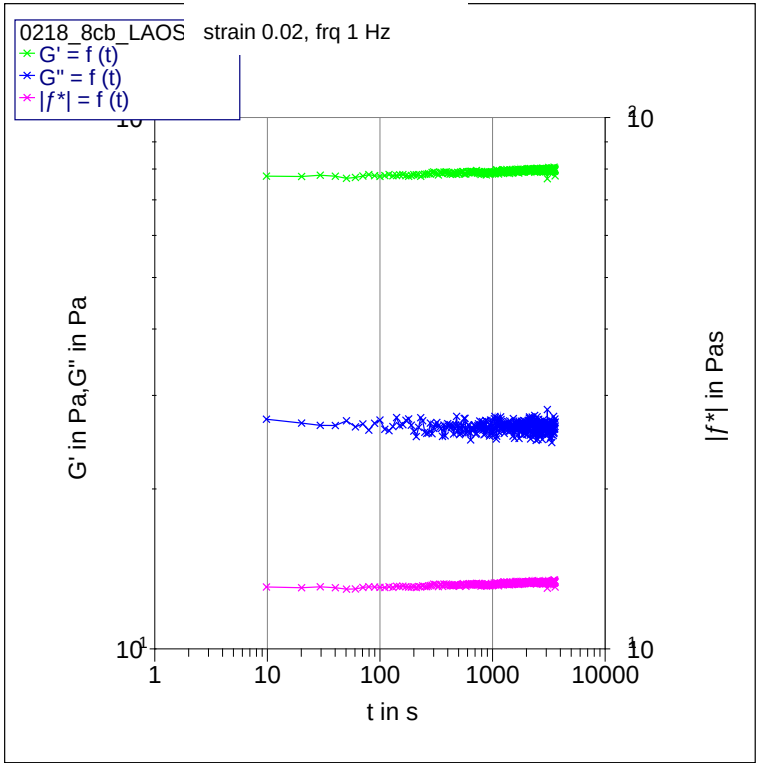
# Large amplitude oscillatory shear (LAOS)

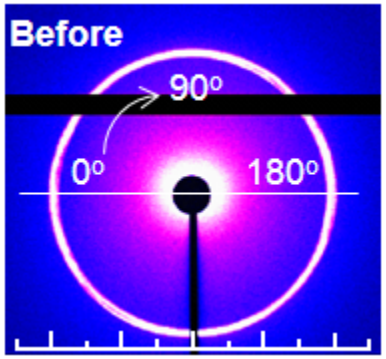


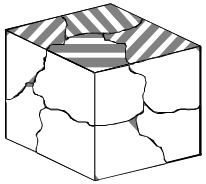



# Frequency 1 Hz, strain 0.02



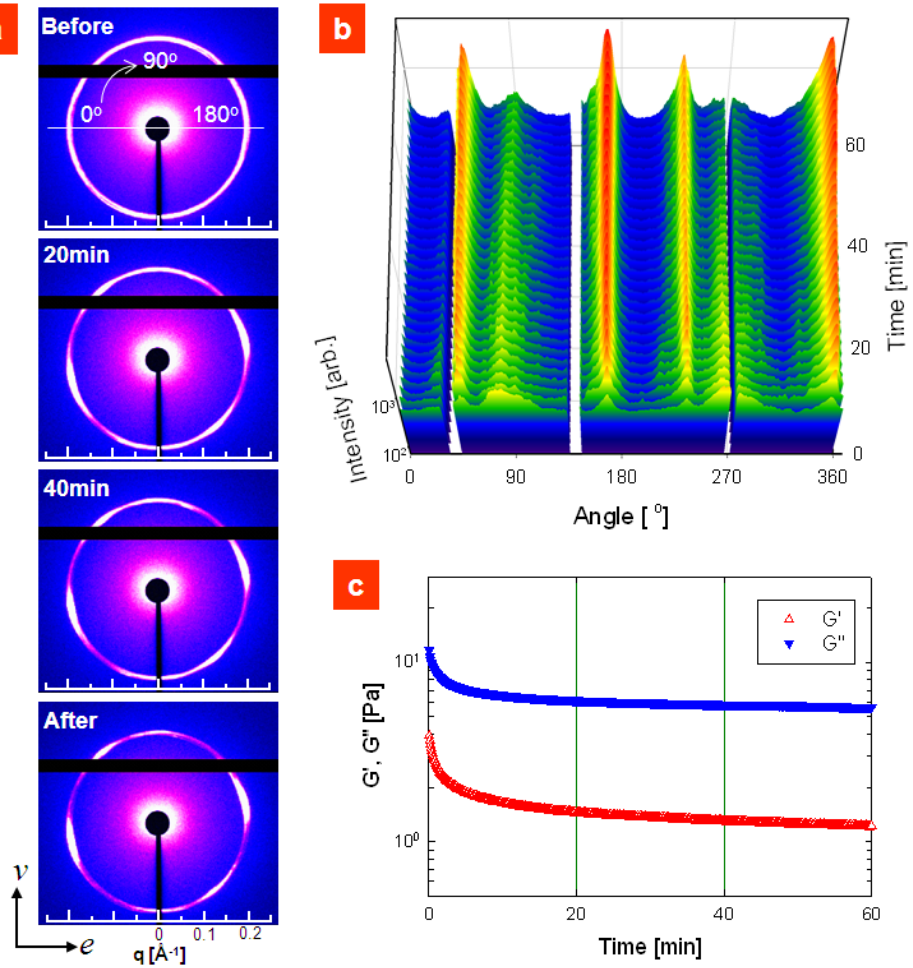




No orientation occured during the experiment



Lamellae stay randomly distributed

# Results: Large amplitude oscillatory shear (LAOS)




Frequency : 1 Hz

Strain amplitude: 1

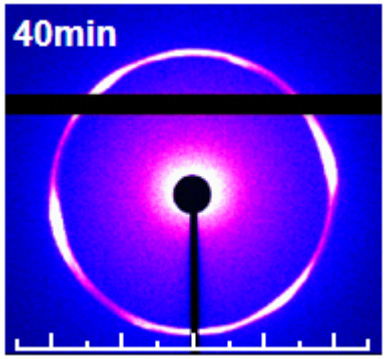
**Non – linear regime**

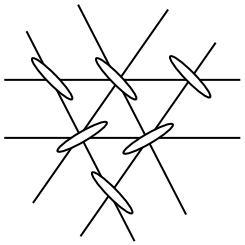
*Struth B, Hyun K, Kats E, Meins T, Walther M, Wilhelm M, Gruebel G*
*LANGMUIR 27, 2880-2887, 2011*

Creation of a sixth order orientational structure
Reorientation only in non linear regime
Stable hours after stopping the experiment

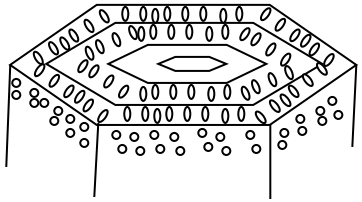
DESY

# Possible scenarios



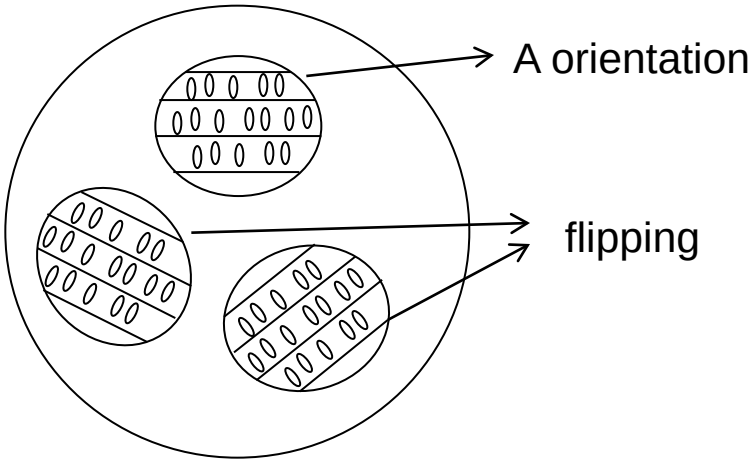
distorted herringbone



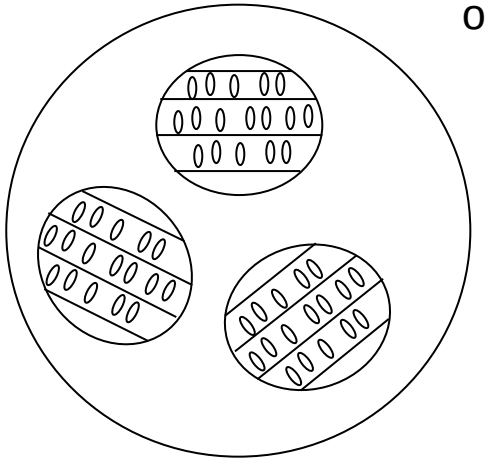
chevron like

Experiments at Petra III:

Flipping and oriented domains




oriented domains



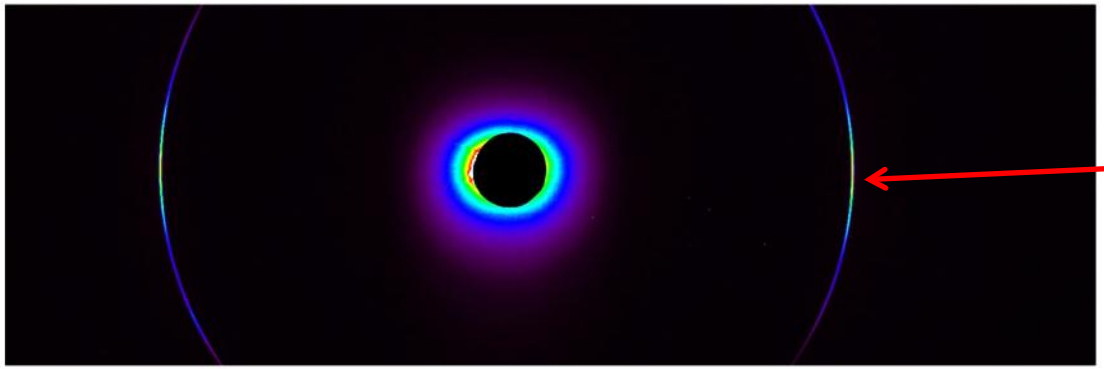
# Towards better spatial and time resolution

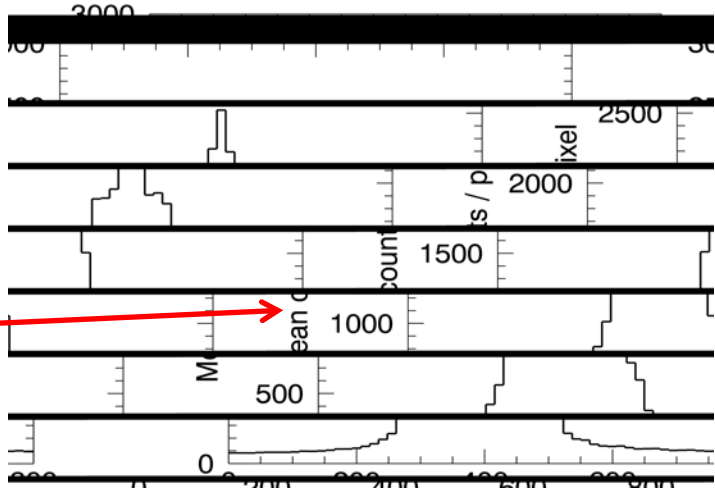
Since september 2012: Member of FS-DS (detector group) at DESY (H. Graafsma)

60% for detectors, 40% for rheology

Diffraction from liquid crystal 8CB under strain (Rheo/SAXS setup at P10, Petra III)

- First beamline experiment with LAMBDA detector
- Pixel size enables line shape analysis





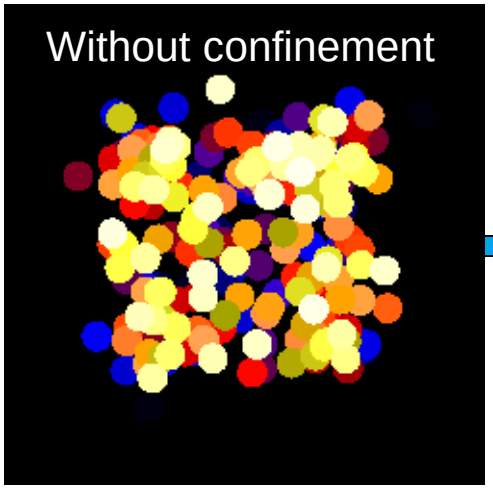

8CB produces very sharp reflections, line shape analysis impossible with standard detectors

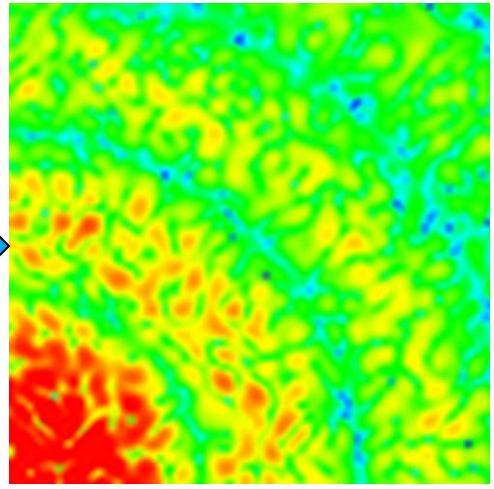
Pixel size of LAMBDA detector allows for line shape analysis when rheology setup is used

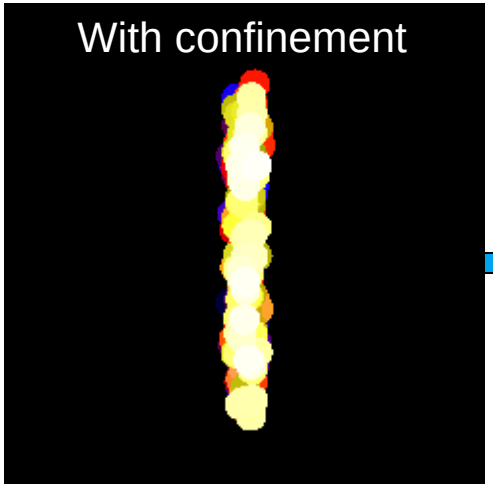
David Pennicard, Bernd Struth, Heinz Graafsma

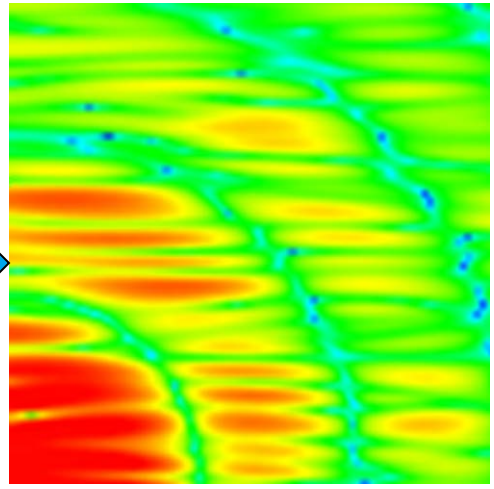
# Comparison of expected speckle shapes and sizes for a system of charge stabilized spheres with and without lateral confinement.

MC – Simulation: System parameters are identical.
Only confinement force along the x-Axis increased by a factor of 10.
Both systems start from completely random particle distribution and evolve into the two states:





Important result for experiments with coherent X-ray beams

Speckles are often too small for detection

In confined systems, speckle size increases mostly in one dimension

This increase could allow detection of ultra fast processes with a completely new generation of ultra fast detectors

Possible application for XFEL???

J. Becker, B. Struth, H. Graafsma

DESY

# Conclusion:

Installation of the modified Mars rheometer successfull at DESY

First in situ experiments with vertical beam geometry at Doris III

28 user experiments already performed at DESY

5 publications (2011-2012)

(1) Observation of New States of Liquid Crystal 8CB under Nonlinear Shear Conditions as Observed via a Novel and Unique Rheology/Small-Angle X-ray Scattering Combination
Struth B., Hyun K., Kats E., Meins T., Walther M., Wilhelm M.,Gruebel G
LANGMUIR 27, 2880-2887, 2011

(2) New Insight to the Mechanism of the Shear-Induced Macroscopic Alignment of Diblock Copolymer Melts by a Unique and Newly Developed Rheo-SAXS Combination
Meins T, Hyun K, Dingenouts N, Ardakani MF, Struth B, Wilhelm M
MACROMOLECULES 45, 1, 455-472 2012

(3) Shear induced structure orientation in norbornene block copolymers: In situ Rheo-SAXS investigations
Pulamagatta B, Ostas E, Herbst F, Struth B, Binder W.H.
EUROPEAN POLYMER JOURNAL 48 1127–1134 2012

(4) Influence of shear-induced crystallization on the electrical conductivity of high density polyethylene carbon nanotube nanocomposites
Tao F, Bonnaud L, Auhl D, Struth B, Dubois P, Bailly C
POLYMER 53 25 5909-5916 2012

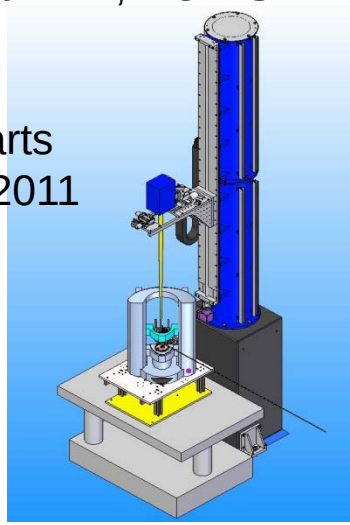
(5) Nonlinear behavior of nematic platelet dispersions in shear flow
Lettinga M.P., Holmqvist P., Ballesta P., Rogers S., Kleshchanok D., Struth B.
PHYS. REV. LETT. 109 2012

Transfer of setup to Petra III, P10 successfull in december 2010

New powerful detectors will push limits of spatial and time resolution

# Time schedule, 2003 – 2013, acknowlegement

2003 idea for a rheology experiment with vertical X-ray beam (B. Struth, ESRF, France)
2004-2005 (B. Struth) first contact with Haake, Karlsruhe, C. Kuechenmeister
2005 idea for modification of the Haake Mars platform to inverted geometry (Haake, B. Struth)
scientific support from M. Wilhelm, Karlsruhe university
2006 move to DESY, Hamburg (Bernd Struth), responsibility: Langmuir and Rheology at DORIS III
2007 delivery of modified rheometer Mars II to DESY
2008 first installation of rheometer with vertical beam geometry at Doris III, BW1
postdoc: K. Hyun, engineers: M. Walther, H. Schultz-Ritter, scientific support M. Wilhelm
first experiments on 8CB, steady shear and LAOS
2009 shutdown of DORIS III, development of heating chamber, 10C – 270C (M. Walther, B. Struth)
2010 first satisfying results at DORIS III: 8CB and Polymers, setup in user mode at BW1, DORIS III
Transfer of experiment to Petra III in december, successful commissioning
2011 design of a developed vertical beam environment at Petra III, P10
(D. Meissner, R. Heitmann, B. Struth), june: design finished, production of parts
first publication: B. Struth et.al., Struth et al, LANGMUIR, 27, 6, 2880-2887, 2011
September: new postdoc, Eric Stellamanns
October / November: installation of vertical beam setup at P10
(L. Klein, S. Bondarenko, E. Stellamanns, B. Struth)
2012 Rheology with vertical X-ray beam in user mode at beamline P10, Petra III
3 papers (results from Doris III), 1 paper (result from Petra III)
shutdown of Doris III
2013 Setup is integrated part of beamline P10, Petra III (beamline responsible M. Sprung)
Instrument responsible: E. Stellamanns



Special thanks to: J. Schneider, E. Weckert, W. Drube, G. Gruebel, H. Franz, H. Graafsma