Status & Developments at the NIST Center for Neutron Research

May 2017

Rob Dimeo, Director

Promoting U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve our quality of life.

NIST LABORATORIES

Physical Measurements Laboratory	Material Measurements Laboratory	Engineering Laboratory	Information Technology Laboratory	Communication Technology Laboratory	Center for Nanoscale Science and Technology	NCNR

MEASUREMENT LABORATORIES

TECHNOLOGY LABORATORIES

USER FACILITIES

NCNR MISSION

…to ensure the availability of neutron measurement capabilities to meet the needs of U.S. researchers from industry, university and other Government agencies.

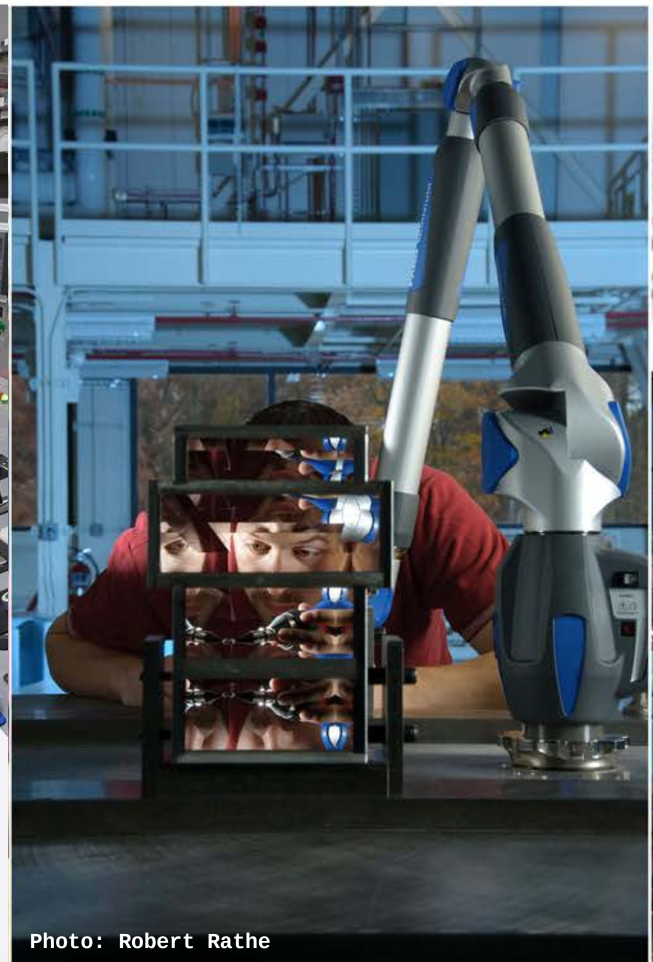
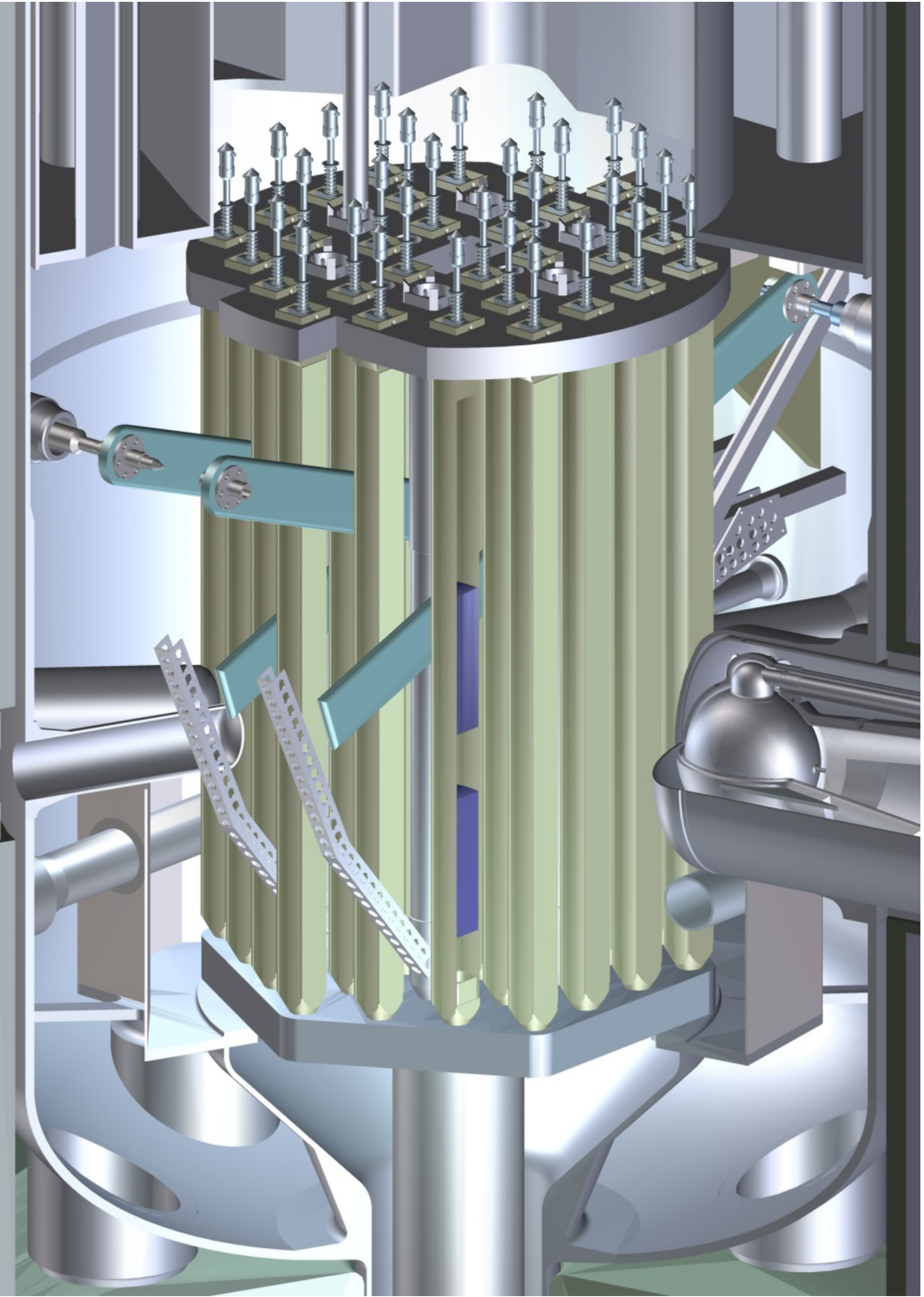


Photo: Robert Rathe



NBSR

Neutron Beam Split-core Reactor

20 MW / D_2O moderated

$\Phi = 1.5 \times 10^{14}$ n/cm^2/s
at mid-plane
(un-fueled region)

7 cycles/year
38 day cycles
~240 days/year

50
years young in December

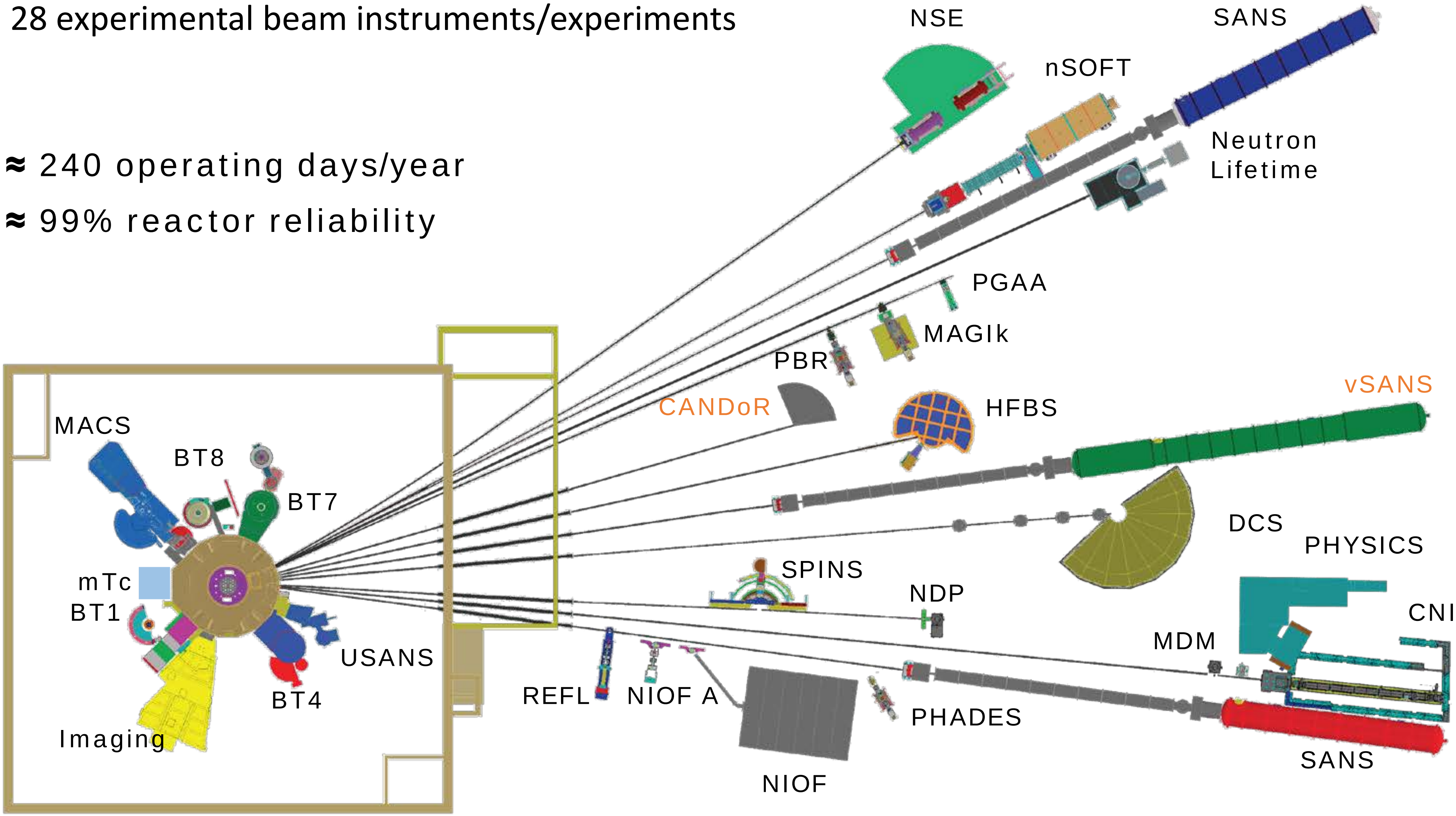
Licensed through 2029

NCNR Overview

28 experimental beam instruments/experiments

≈ 240 operating days/year

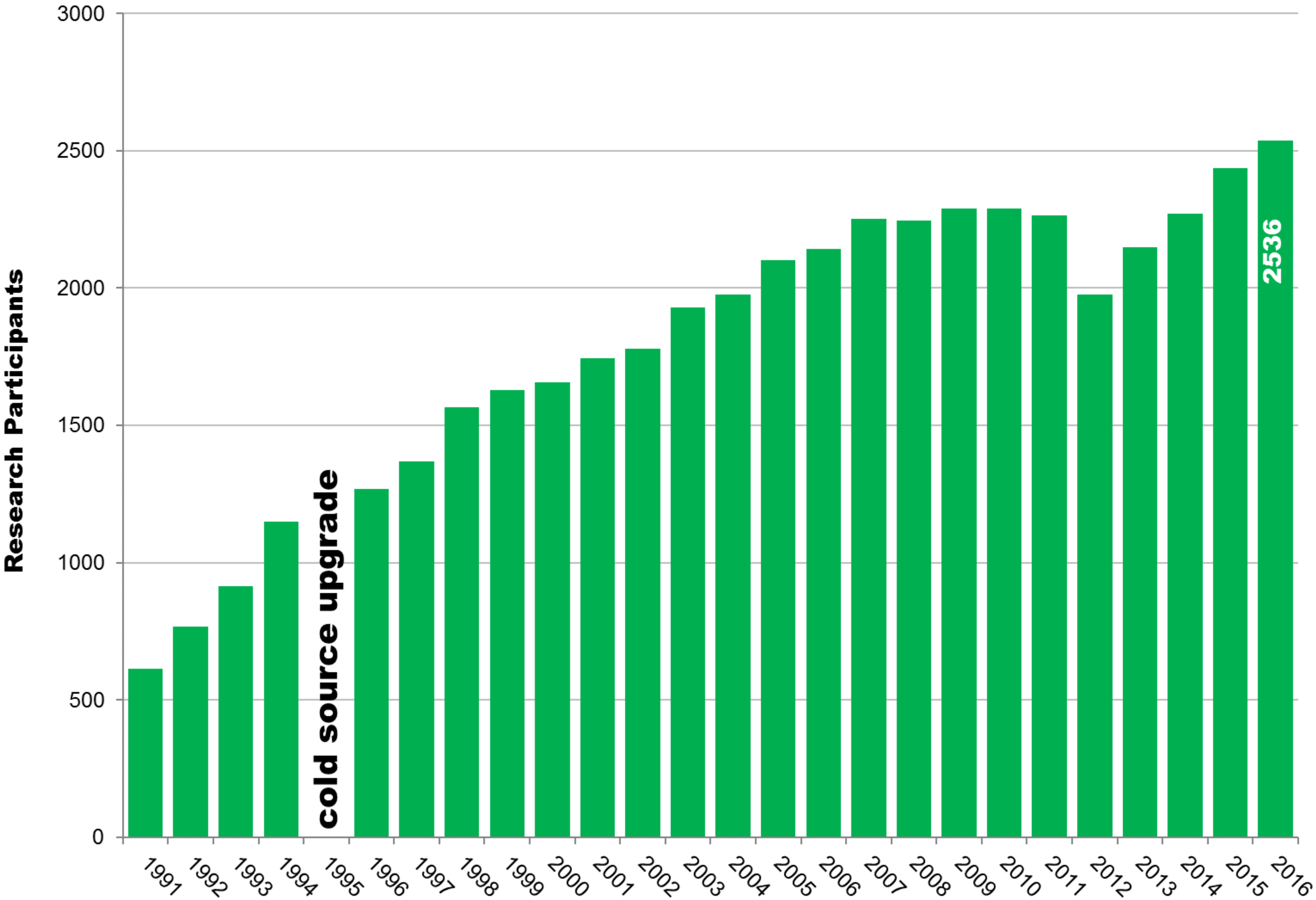
≈ 99% reactor reliability

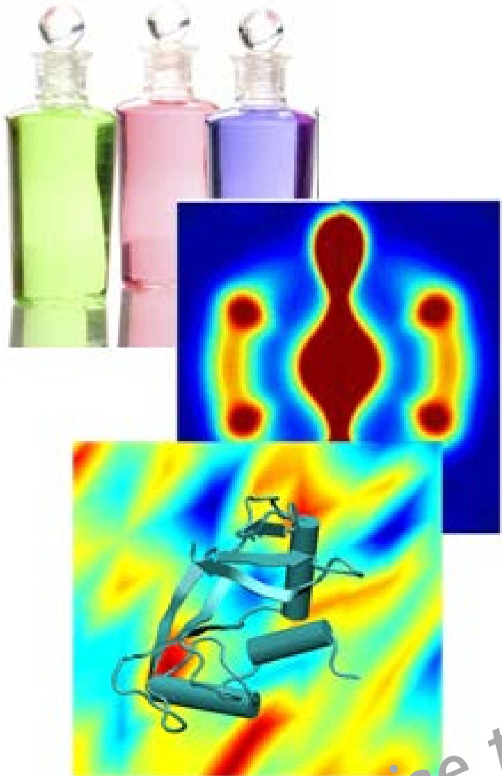


≈ 2500 research participants/year

≈ 300 pubs/year

RESEARCH PARTICIPANTS





nSoft

A consortium for the advancement of neutron-based measurements for manufacturing of soft materials.

nSoft Model

Members identify key problems

NIST develops sample environments, data analysis packages, neutron measurement methods

nSoft transfers expertise to members

Members use expertise in proprietary access mode

Membership = $25k/year

Proprietary access purchased separately

Member Benefits

Tailored measurement techniques for show stopper problems

Training in the use of those techniques

Unprecedented access to NIST staff, programs, and resources

Ron Jones, *nSoft Director*

Amgen – Aramco – Chevron Phillips – Dow Chemical – Genetech
L'Oreal – Pfizer – Proctor & Gamble – Toyota Research

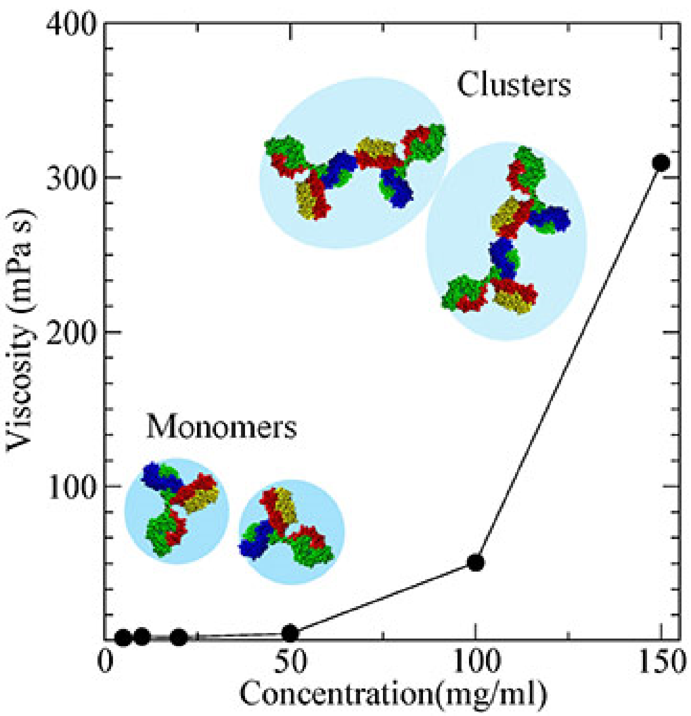
Scientists Discover Cause of Unwanted Viscosity Increase in Monoclonal Antibody Solutions (mAbs)

nSoft
A consortium for the advancement of neutron-based measurements for manufacturing of soft materials.

mAbs widely used to treat cancer and infectious diseases

Drugs would be easier and cheaper to administer subcutaneously via thin needles

Some mAb solutions show unwanted increases in viscosity as a function of concentration and shear experienced in needles making this difficult



Neutron experiments revealed the role of clustering responsible for this unwanted behavior

Discovery may lead pharmaceutical companies to create a variety of cancer and autoimmune disease treatments based on mAbs that can be delivered via thin needles

Genentech
A Member of the Roche Group
University of Delaware
NIST
ILL

Yearley et. al., *Biophysical Journal*, April 16, 2014.

USER CAPABILITIES

Image used with the kind permission of @lehrerboys

By staying still and blending in with his surroundings, Mel once again avoids helping with the dishes.

BL^2 Biomolecular Labeling Laboratory

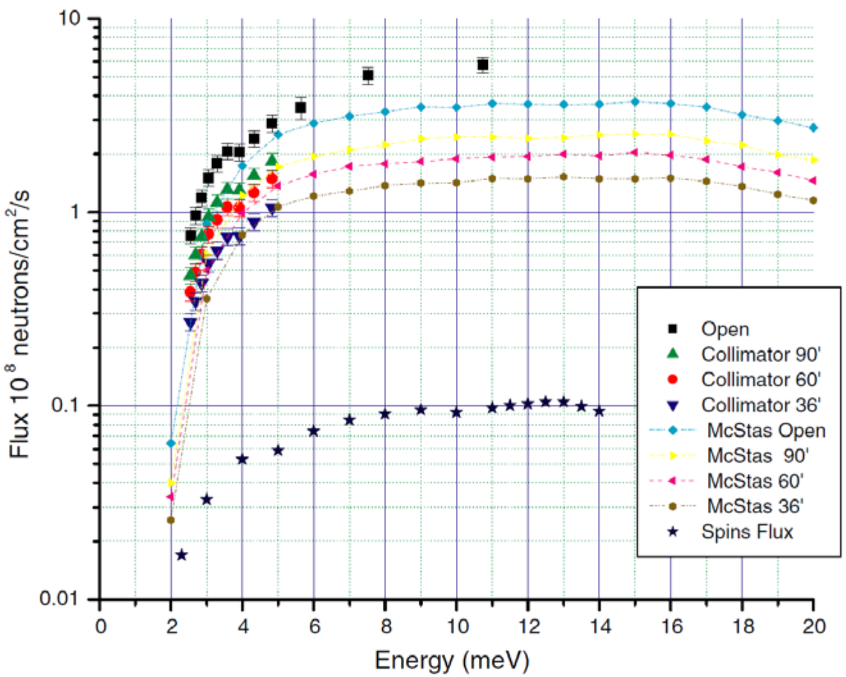
MULTI-AXIS CRYSTAL SPECTROMETER

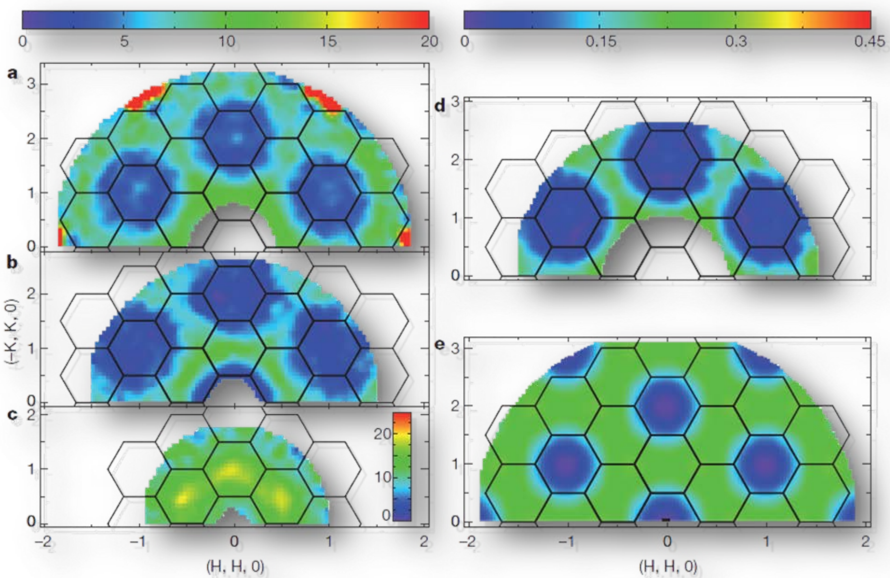
DFM: 17x21 array of PG, 1428 cm^2

$2.2 < E_i$ (meV) < 17

$\Phi = 5.0 \times 10^8$ cm^{-2}s^{-1} @ 7.5 meV

Detector: 20 x 8 degree coverage, total 160 degrees



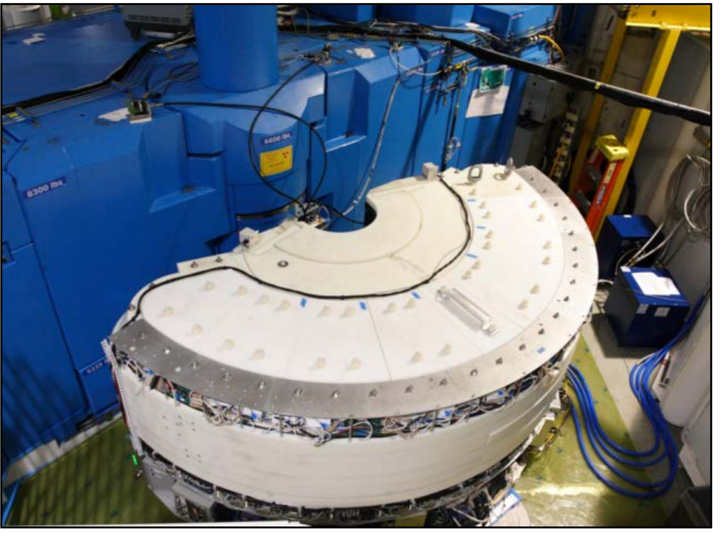


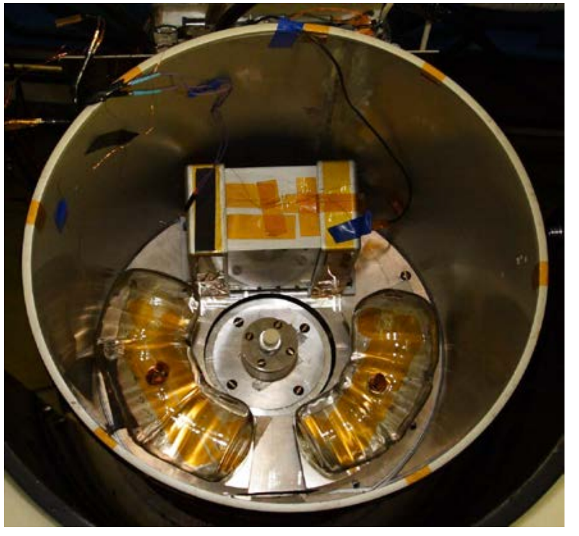
J.A. Rodriguez et al, Meas. Sci. Technol. 19, 034023 (2008)

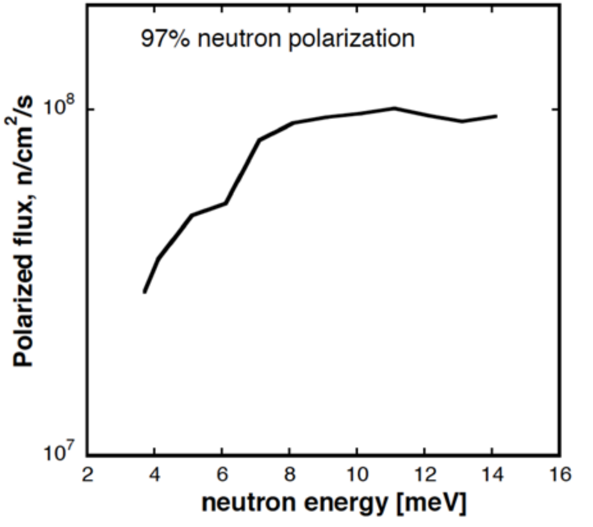
T.-H. Han et. al., Nature 492 11659 (2012)

POLARIZED MACS

MACS allows for high sensitivity to access physics in small samples and is ideal for probing slowly propagating excitations in hard condensed matter







$2.2 < E_i$ (meV) < 17
$2.5 < E_f$ (meV) < 5

220°
Total angular coverage for polarization analysis

~10^8 n/cm²/s @ 10 meV

40
Initial flipping ratio

HORSESHOE-SHAPED CELL DEVELOPMENT

Advantages over current 110 degree three section cells

Fully blown → better relaxation times

Rectangular cross section and circular shape → more uniform analyzing power.

Polarization efficiency correction → more straightforward

9 cm ID - 22 cm OD - 8 cm tall

260°

angular coverage for Polarization analysis

350 h

Relaxation time

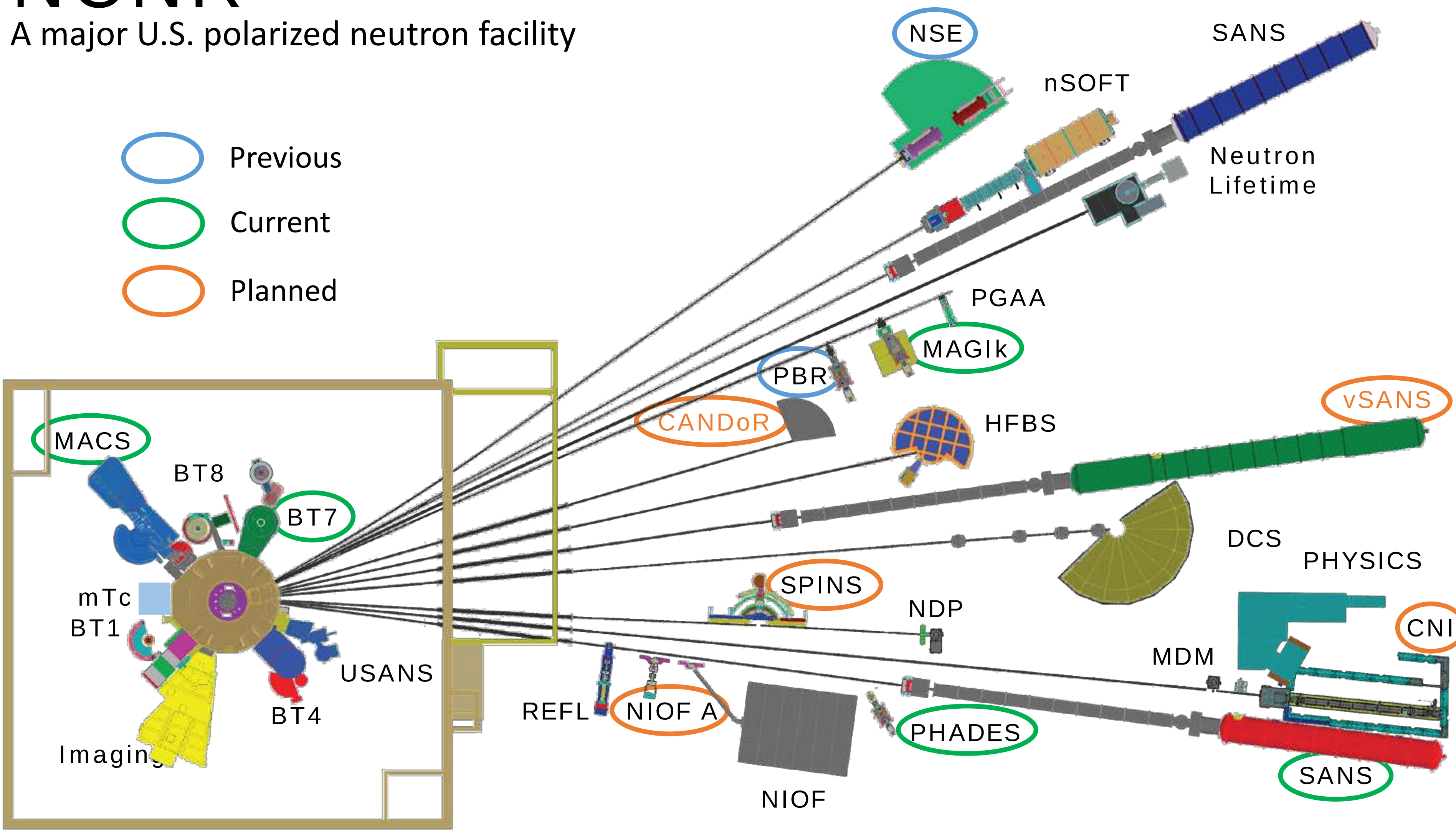
80%, to date

Max. ^3He polarization to date

W.C. Chen *et al*, J. Phys. Conf. Ser. 746, 012016 (2016)

NCNR

A major U.S. polarized neutron facility

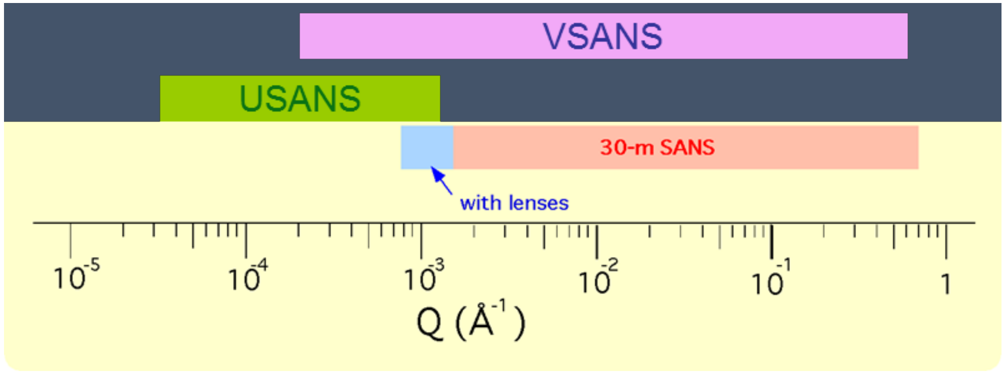


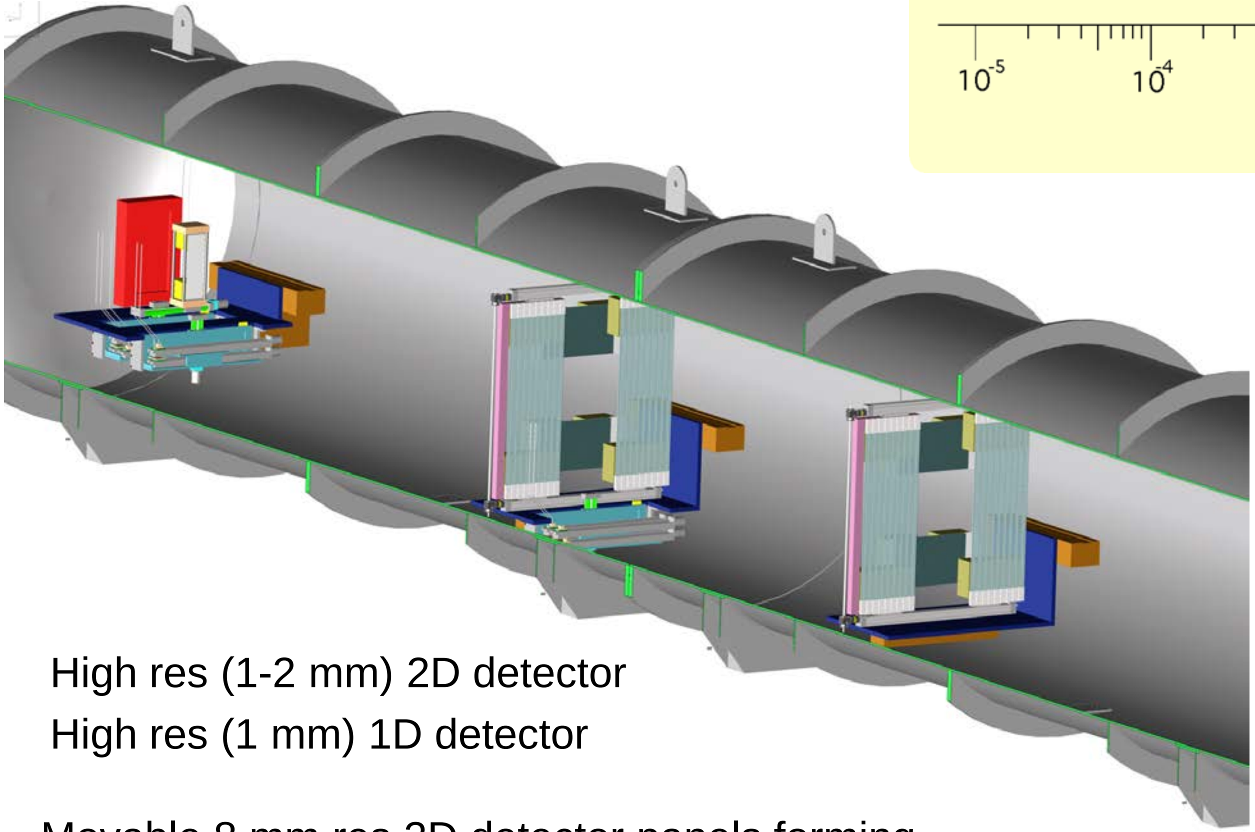
6 years | 150 experiments
540 beam days
930 bar-liters ^{3}He

FACILITY DEVELOPMENTS

vSANS

Data collection rate:
~10x SANS & ~300x uSANS





Extended Q-range
$2×10^{-4} \rightarrow 1$ $Å^{-1}$

Flexibility
2% - 13.5% - 30%
wavelength band

High res (1-2 mm) 2D detector
High res (1 mm) 1D detector

Movable 8 mm res 2D detector panels forming
1 m^2 area on front and middle carriage

Installation is nearly complete




vSANS SCHEDULE

First Neutrons on Detector: June 2017

Delivery and fit out of detector vessel
Install/testing of detector carriages
Install/testing of eight detector panels

First SANS experiment: August 2017

Sample area installed
‘Basic’ data acquisition software tested
Tube detector NISTO software tested
‘Basic’ data reduction software tested

Full Polarized Beam Operation: January 2018

Polarizer installed
RF Flipper installed
Guide Fields installed
NICE software polarized beam option tested
Data reduction software for polarized beam tested

vSANS SCHEDULE

Kinetic SANS: March 2018

Event mode data output (software) from tube detectors (built/tested)
Event mode option in NICE software (built/tested)
Event mode data reduction in IGOR software (built/tested)

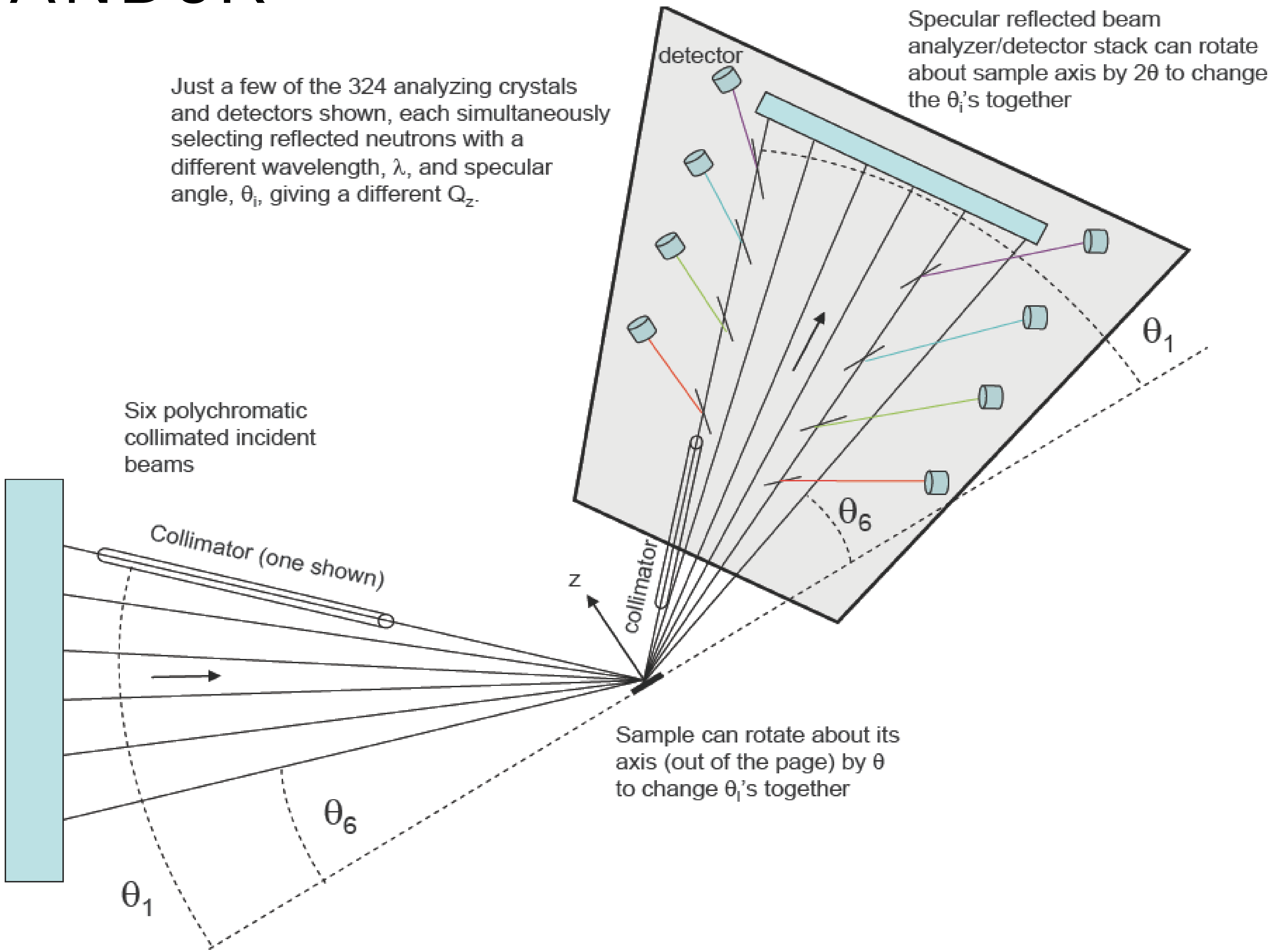
Very Small Q: High resolution mode: January 2018

Install/test high resolution detector
Build/install/test rear carriage
New NISTO software to handle the detector
New NICE software to handle the detector
Data reduction software to handle new detector histogram
Procure/install chiller for MgF_2 prisms and lenses
Build/install/align converging beam apertures

Graphite Monochromator: January 2018

Procure/deliver HOPG graphite
Install graphite

CANDoR


Specular reflected beam analyzer/detector stack can rotate about sample axis by 2θ to change the θi's together
detector
Just a few of the 324 analyzing crystals and detectors shown, each simultaneously selecting reflected neutrons with a different wavelength, λ, and specular angle, θi, giving a different Qz.
θ1
θ6
Six polychromatic collimated incident beams
Collimator (one shown)
z
collimator
Sample can rotate about its axis (out of the page) by θ to change θi's together
θ6
θ1

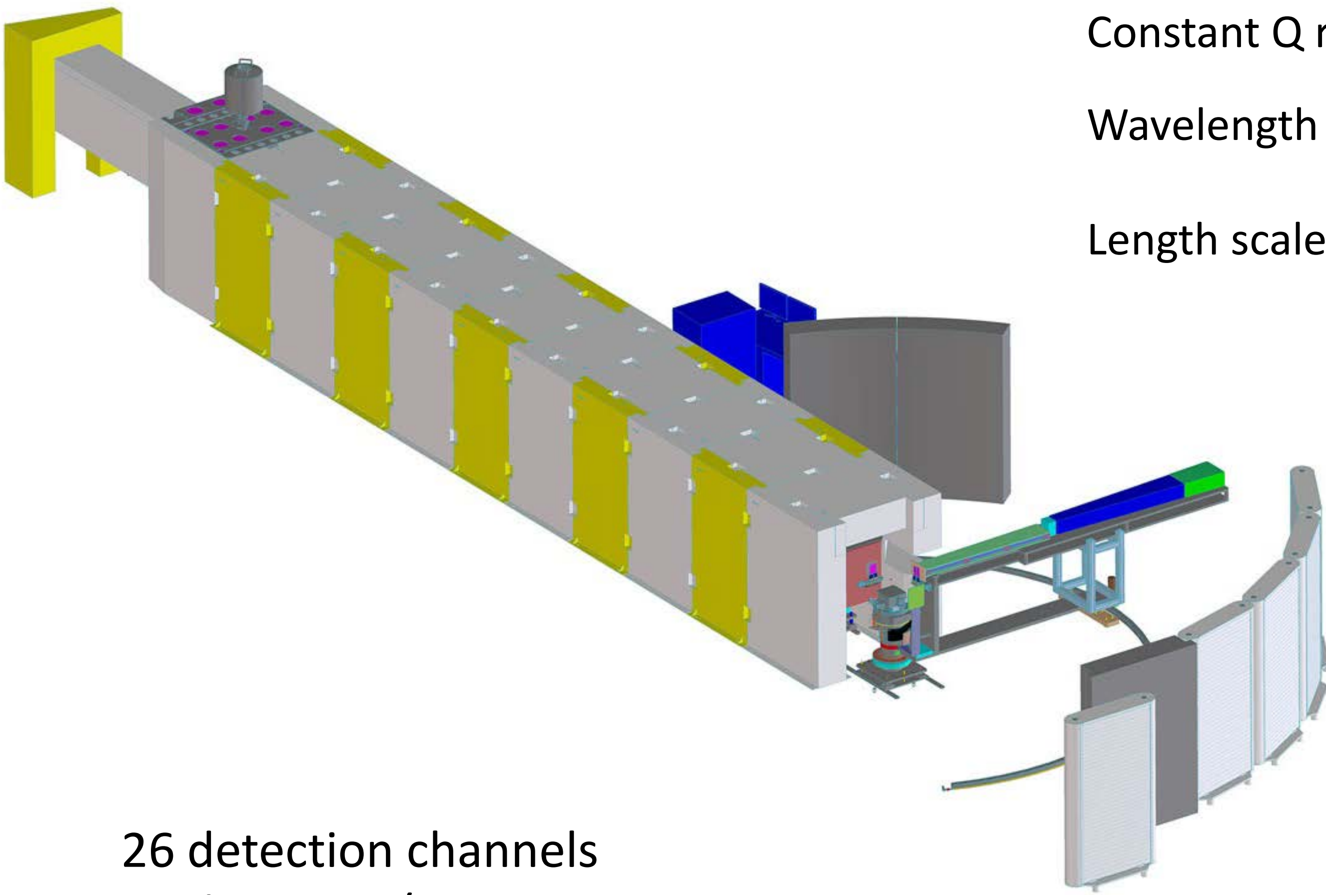
CANDoR

Data collection rate > 10x our current reflectometers

Constant Q resolution: $\delta Q/Q \approx 0.025$

Wavelength resolution: $\delta\lambda/\lambda \approx 0.015$

Length scales: 0.3 nm → 10 μm



26 detection channels
54 detectors/array
1400 detectors total

CANDoR

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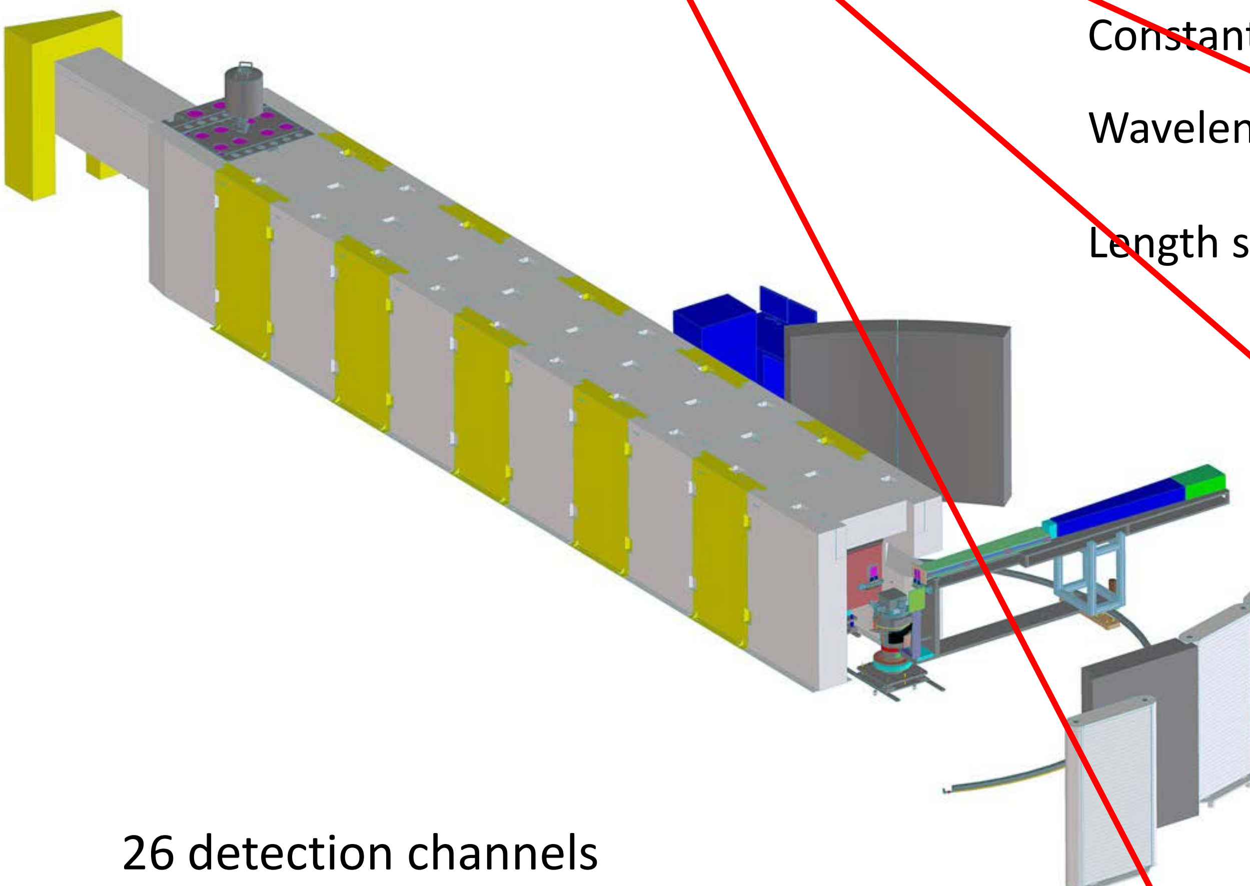
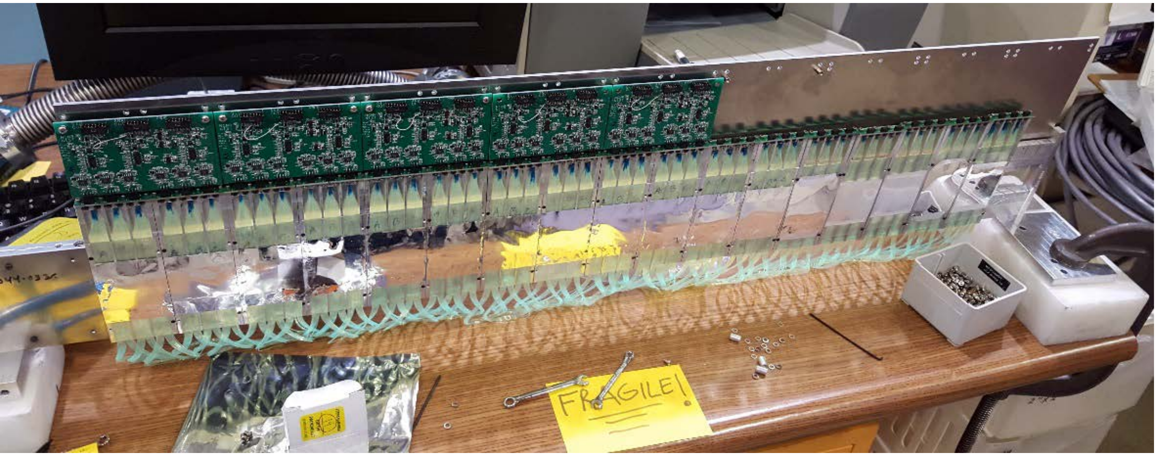


26 detection channels
54 detectors/array
1400 detectors total

The Farago upgrade criteria

CANDoR DETECTOR TESTS

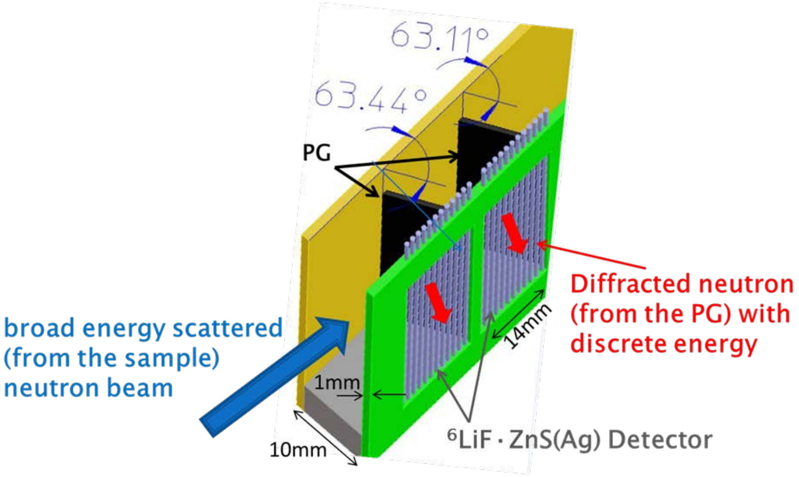


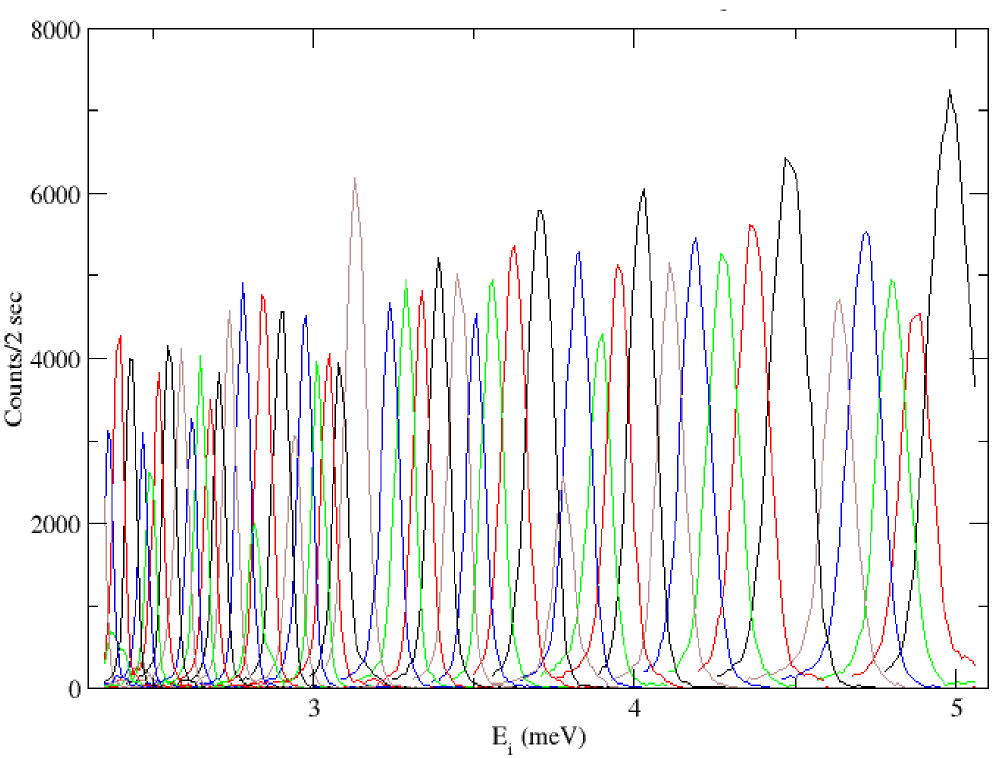
Full Working Prototype

54 energy channels

pulse-shape analysis

Active area 1 cm × 3 cm
Total thickness ~ 1.5 mm
Absorption > 95% at 3.27 meV
Absolute neutron sensitivity > 93% at 3.27 meV
Pulse shape discrimination
Gamma rejection ~10^{-7}
Handles 10 kcps with minimal deadtime (4 µs)





CANDoR SCHEDULE

First Neutrons on Detector: June 2018

Scintillator detector production/repeatability

Data acquisition electronics

Installation/testing of detector

First Specular Reflection Experiment: October 2018

Sample area installed

'Basic" NICE software features tested

'Basic' data reduction software (built/tested)

Full Polarized Beam Operation: October 2018

Polarizer installed

RF Flipper installed

Non Specular Capability Available: October 2018

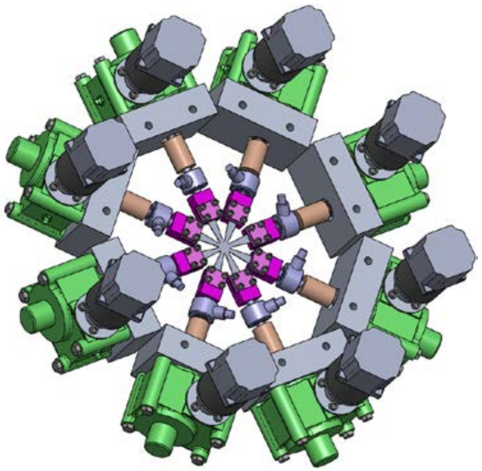
Data reduction software (built/tested)

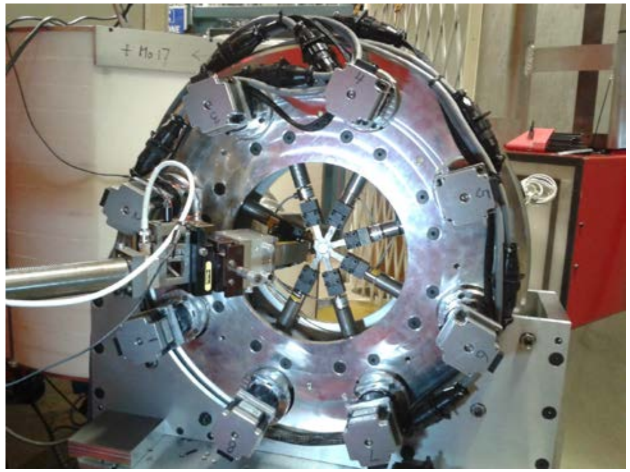
Event Mode Available: October 2018

Event mode option in NICE software (built/tested)

Event mode data reduction software (built/tested)

OCTO-STRAIN: A NOVEL MULTI-AXIAL LOADING DEVICE FOR IN-SITU STRESS MEASUREMENTS





Eight arm multi-directional straining device with 10 kN load capacity.

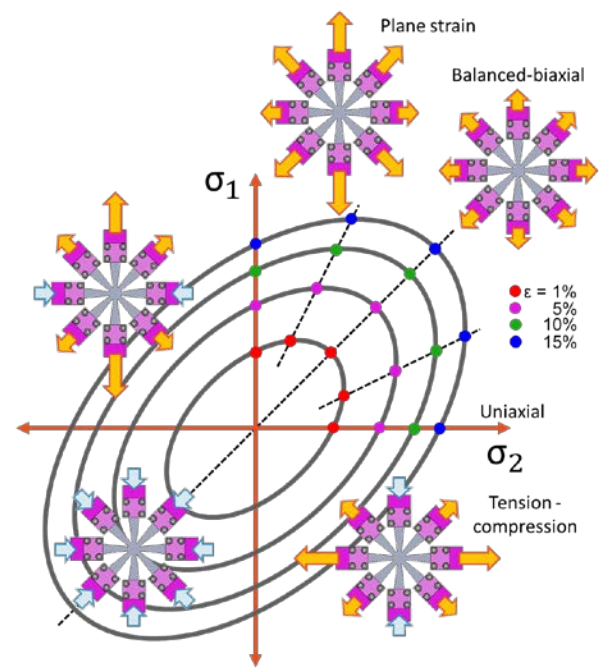
Individual control of each loading direction.

Ability to define various strain paths, such as: uniaxial, plain strain, balanced-biaxial and tension-compression.

With addition of anti-buckling device can achieve strain paths over entire yield surface.

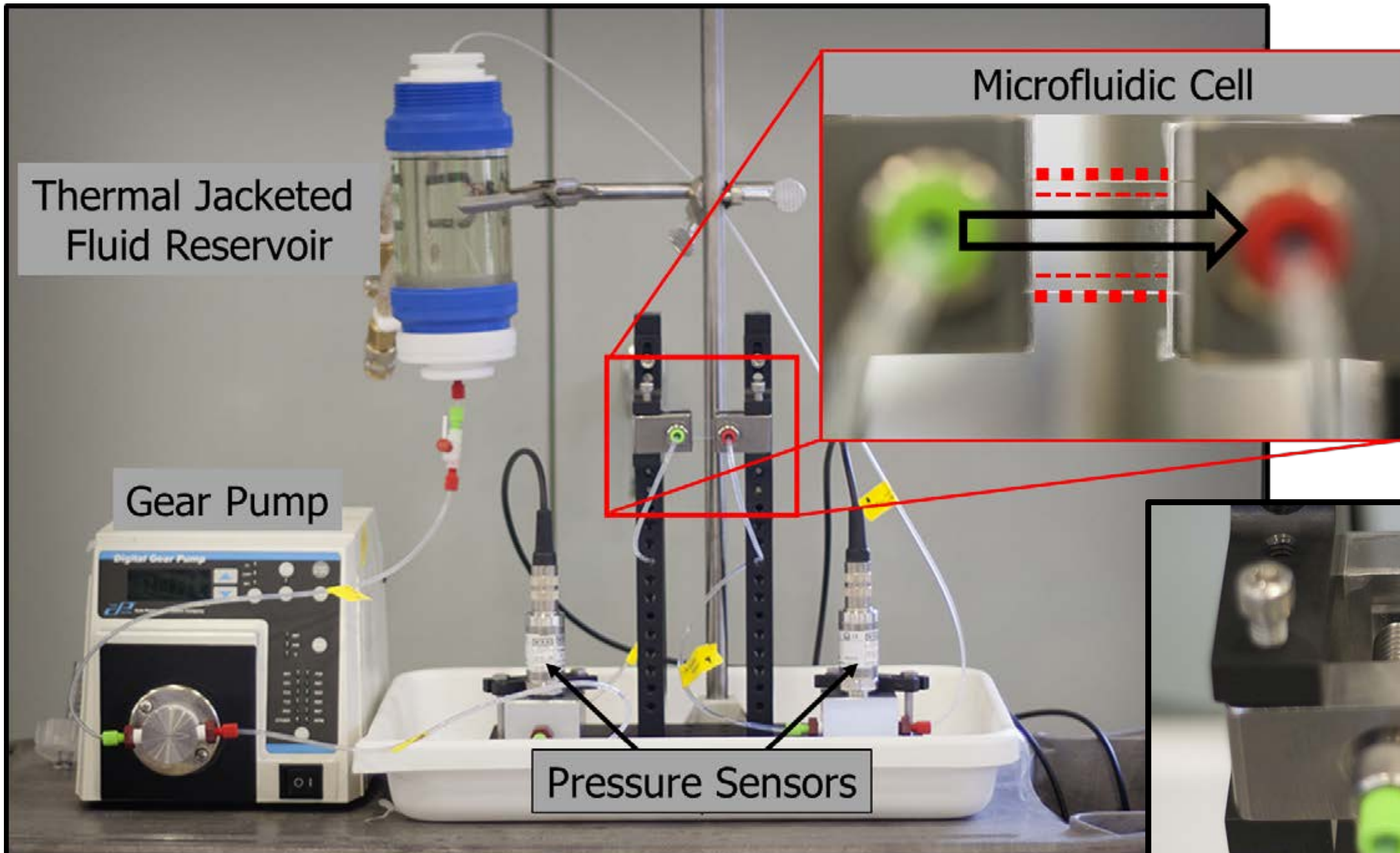
Neutron diffraction is the only method where an anti-buckling device can be used and measurements within the material is still possible.

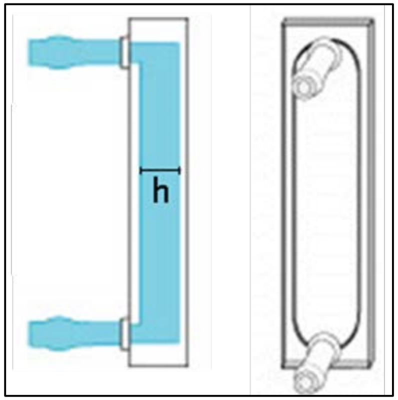
Future work will include addition of an actuator in the σ_3- direction to produce three-dimensional yield surfaces.



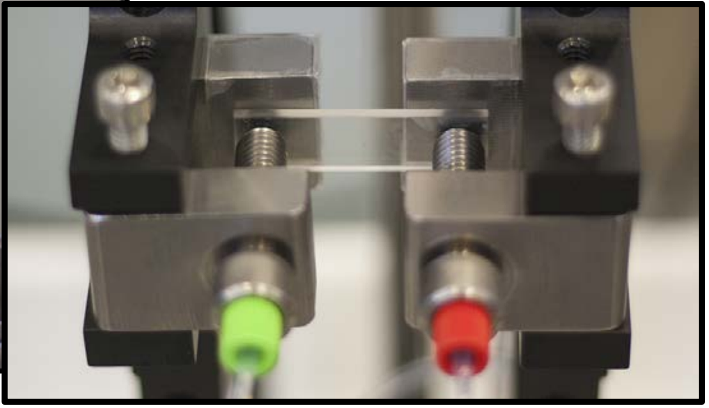
Yield Surface (Stress Space)

μRHEO SANS



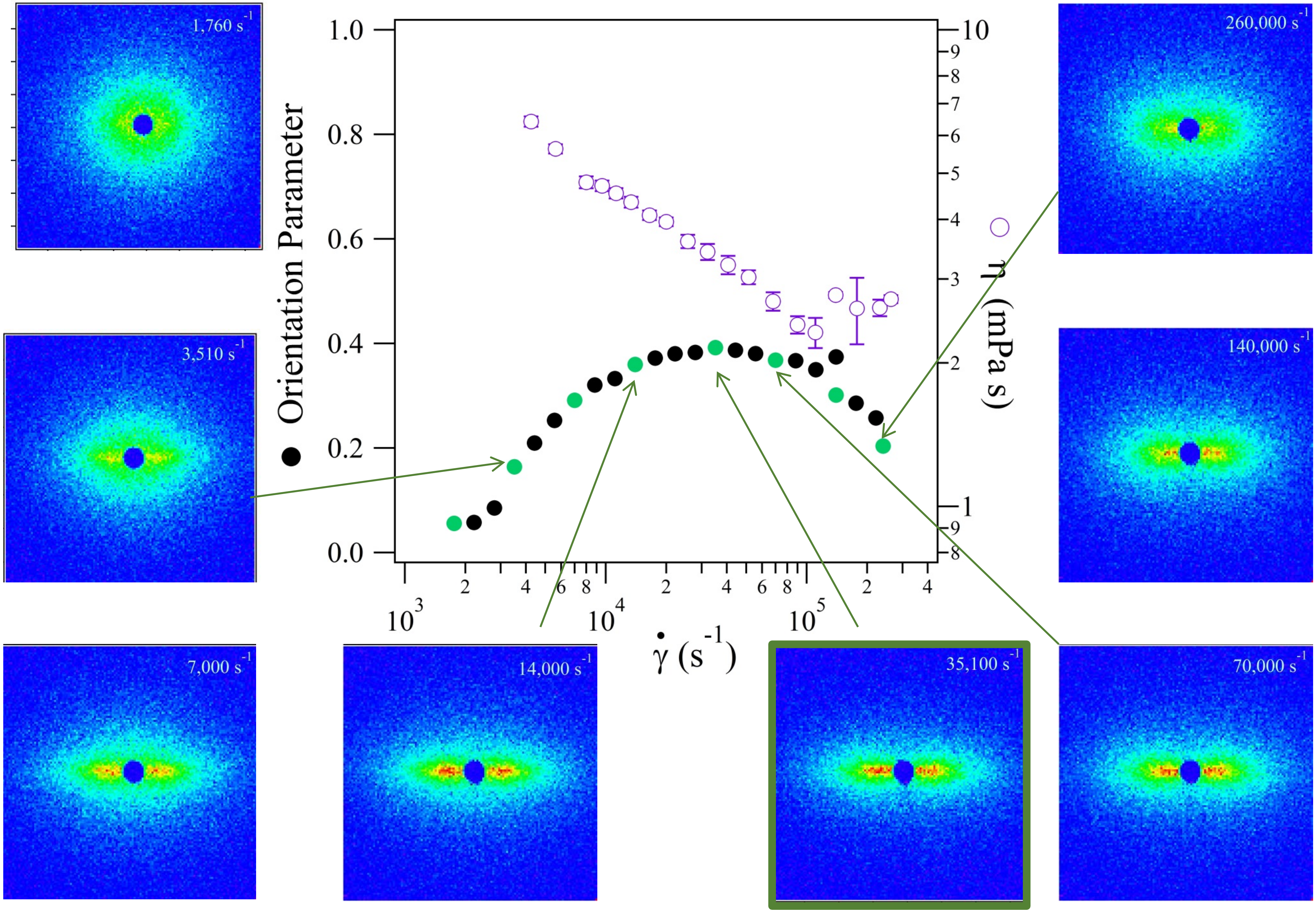


**UV/Vis Flow Cell
From Starna**



μRHEO SANS

Alignment Factor for CPCl Wormlike Micelles in Poiseuille Flow



NEUTRON MICROSCOPE

Wolter Optics

Faint x-ray sources (nebula, *etc*.) need to be focused for good imaging

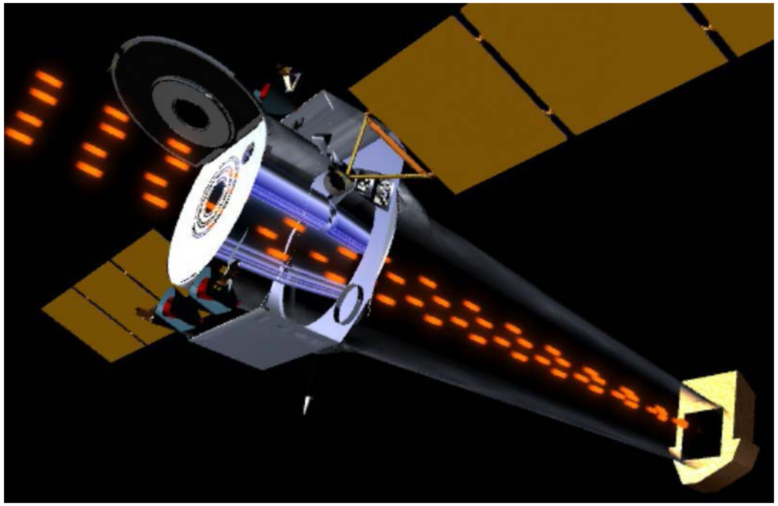
CHANDRA mirrors are coated on 2 cm thick glass substrates

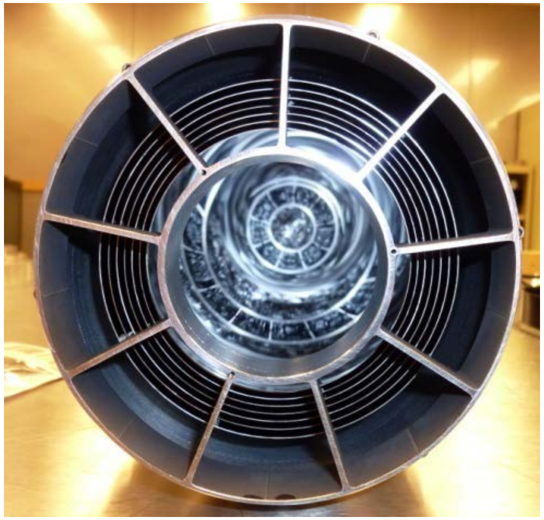
NASA has developed a new fabrication technique to create Wolter Optics from nested Ni-foil mirrors – light for space telescopes and *perfect for neutrons*

Reflection is achromatic, Wolter Optics have reasonable off-axis properties

Resolution from the lens not collimation

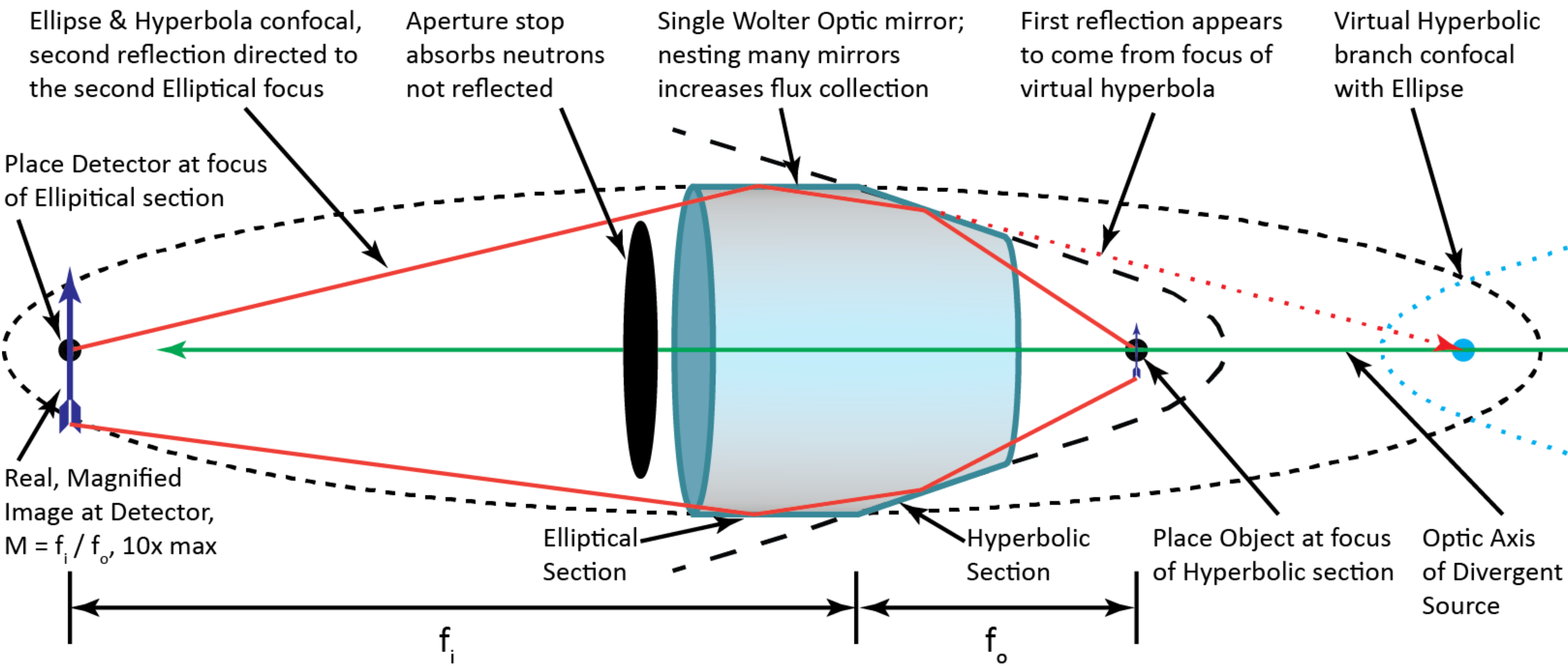
Focusing can yield ***100x gain*** for imaging





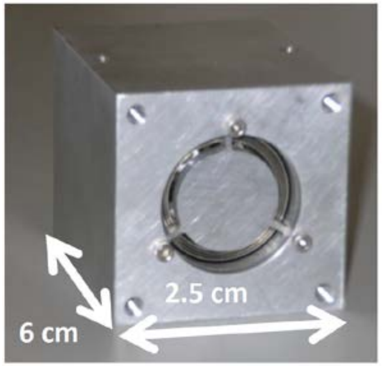
NEUTRON MICROSCOPE

WOLTER OPTICS



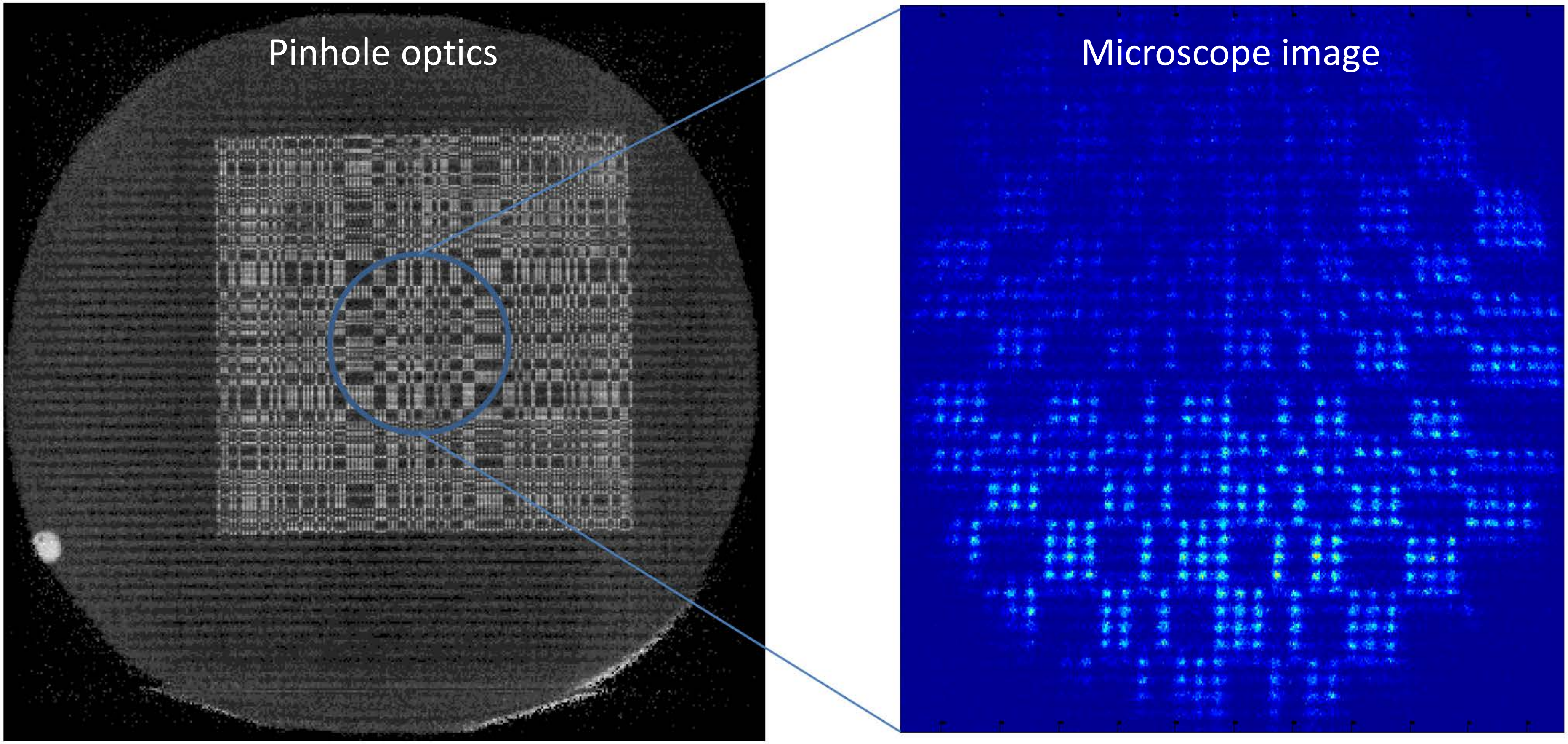
NEUTRON MICROSCOPE

WOLTER OPTICS



An early prototype
3 nested Ni foil mirrors
75 μm resolution, 110 μrad res
5x intensity gain

2 cm x 2 cm pinhole mask with 0.1 mm diameters on 0.2 mm centers



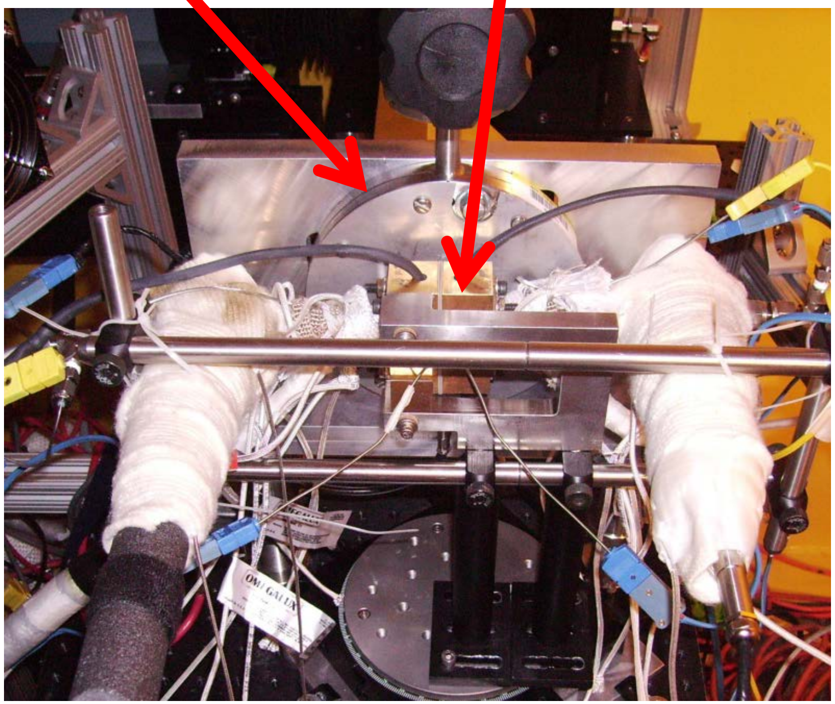
→ Engineering optic tested in July 2016

→ Addressing imperfections from step fabrication process

NEUTRON MICROSCOPE

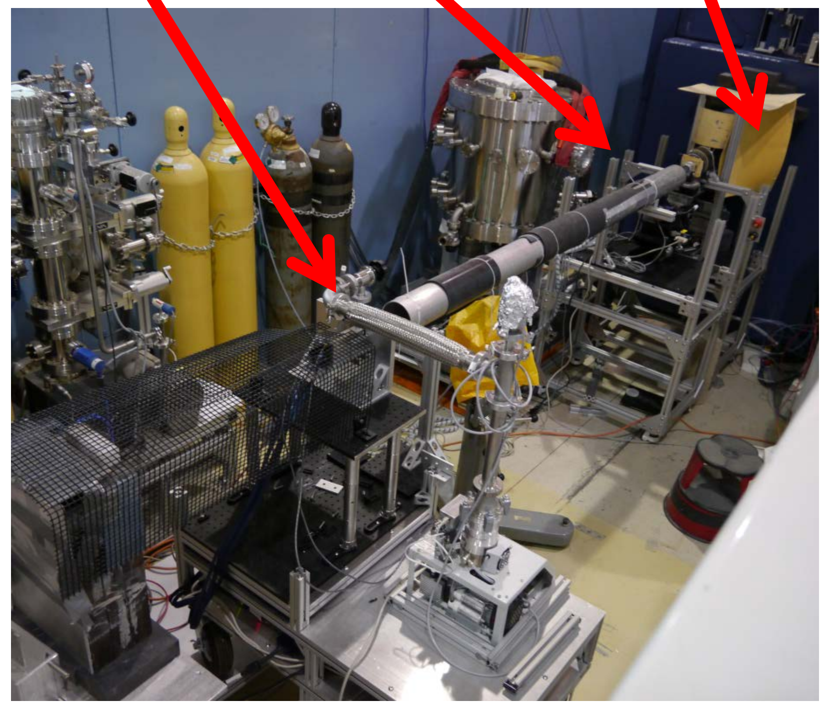
Pinhole Optics vs. Wolter Optics layout

Detector Fuel cell



Pinhole Imaging Setup: 1 cm between sample and detector

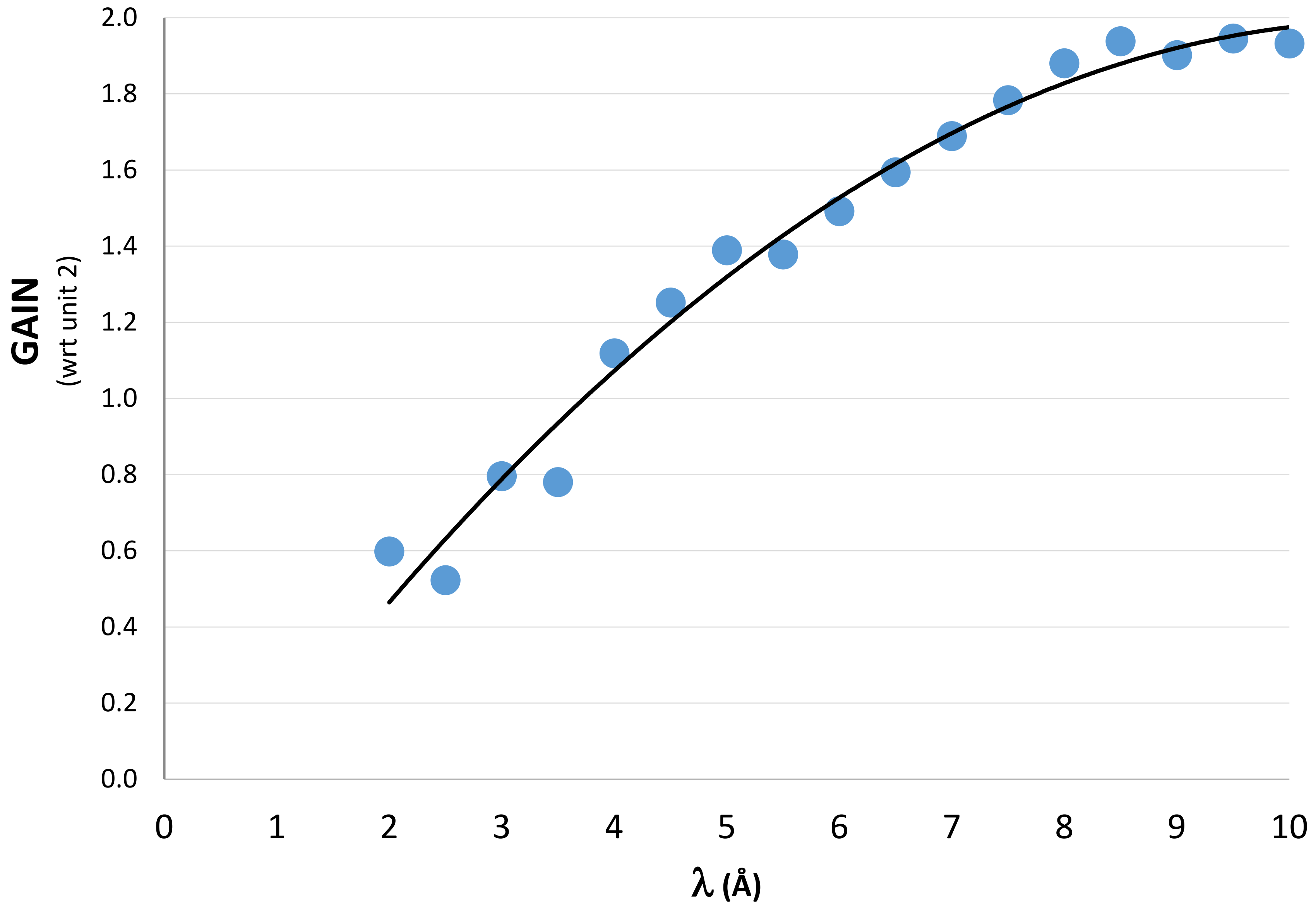
Detector Lens Fuel cell



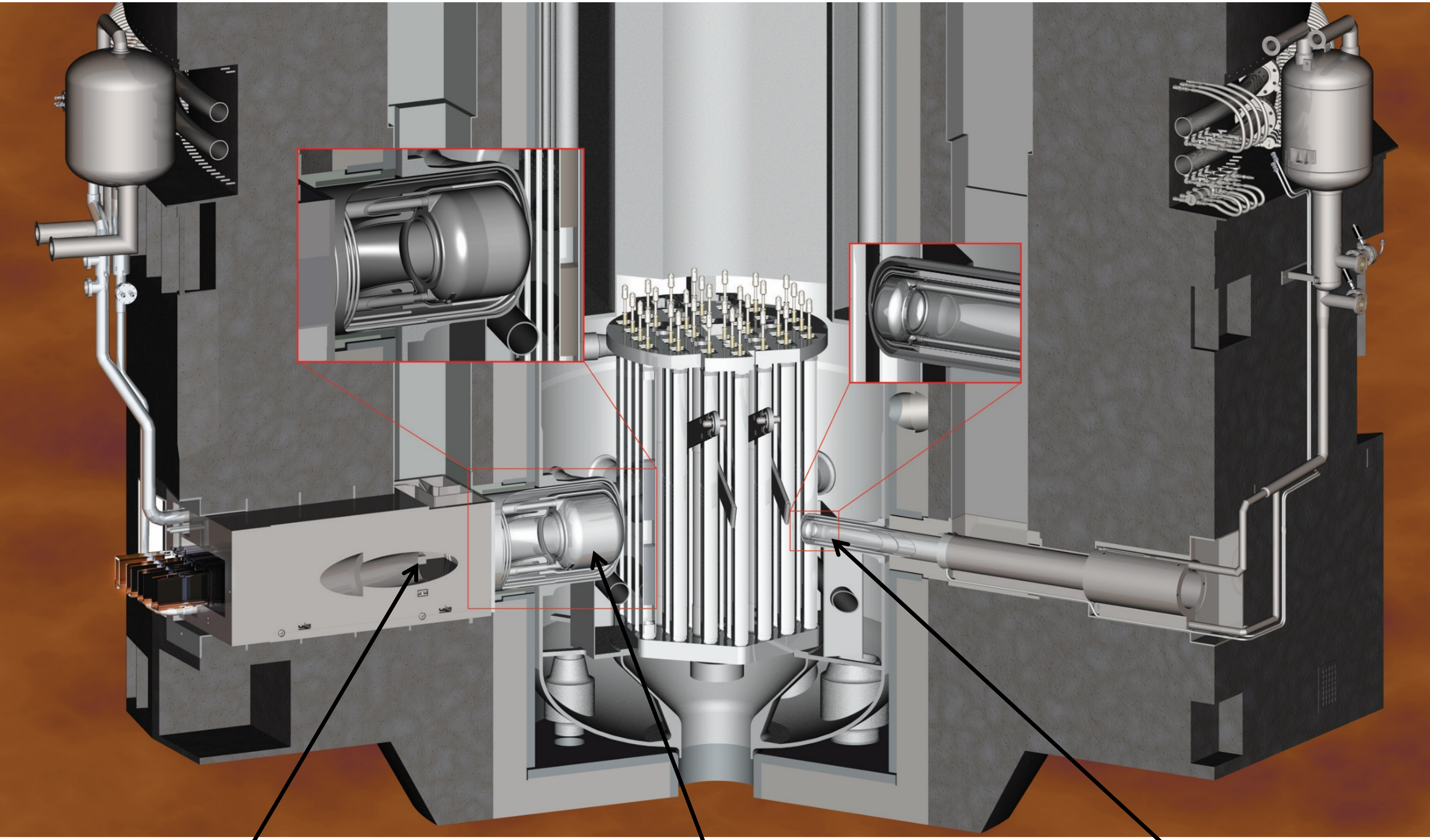
Wolter Optics Setup: 60 cm between sample and lens, 2.5 m between lens and detector

FUTURE

D_2 COLD SOURCE



D_2 COLD SOURCE



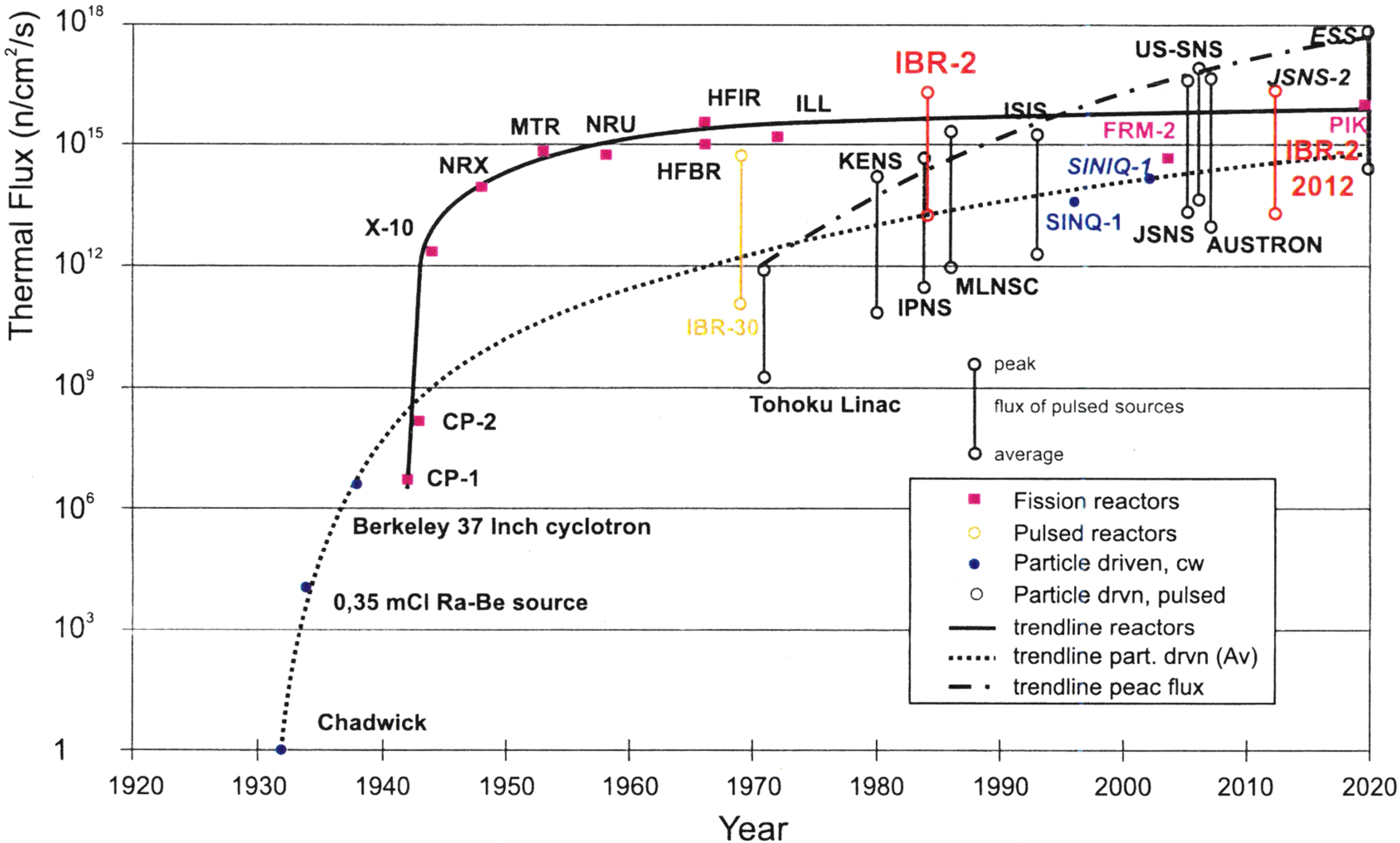
Cutout for new guides

LD_2 source

MACS LH_2 source

FUTURE

Recent neutron source improvements *evolutionary* not *revolutionary*



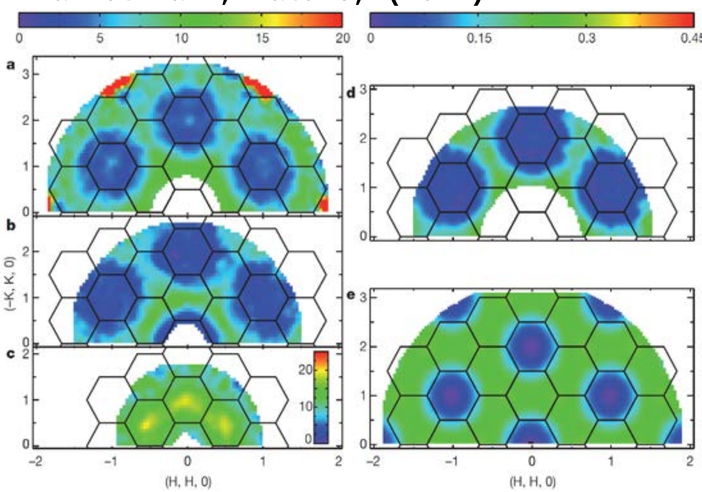
Graphic based on figure from the 2015 PNPI Scientific Highlights, p. 122.

FUTURE

Revolutionary advances in science with neutrons come from instrument advances and novel sample environments

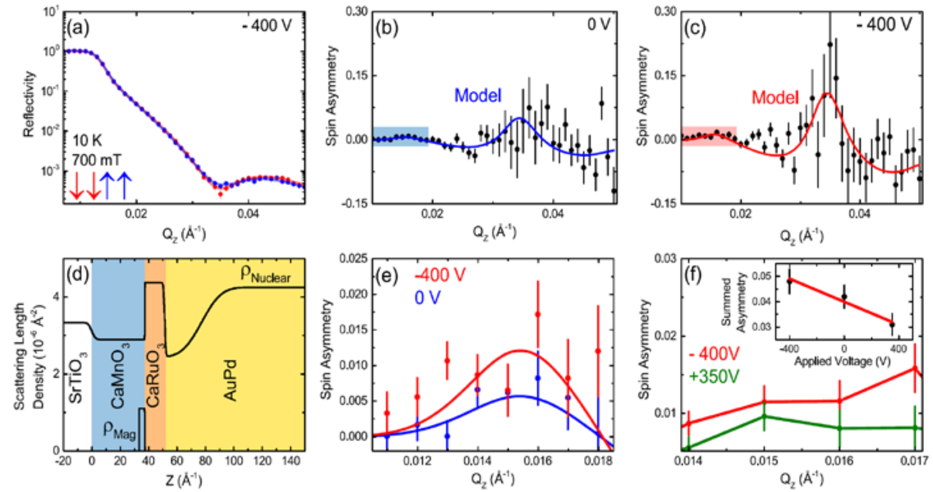
s = 1/2

Han et. al., Nature, (2012)



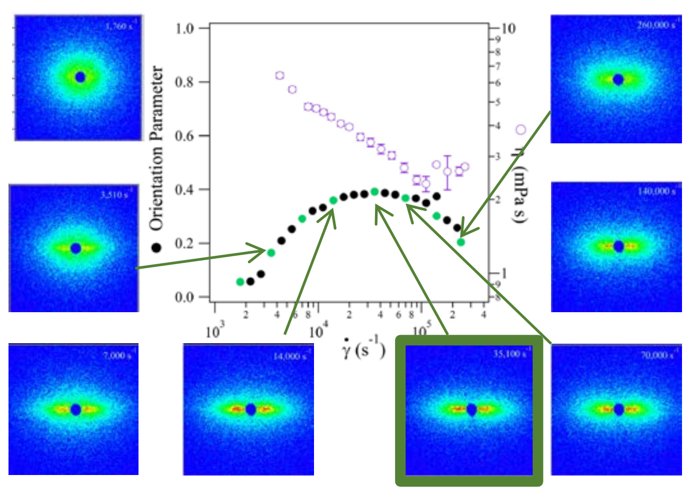
Interfacial magnetism

Grutter et. al., PRL, (2015)



High flow

Weigandt et. al., (2016)



FUTURE

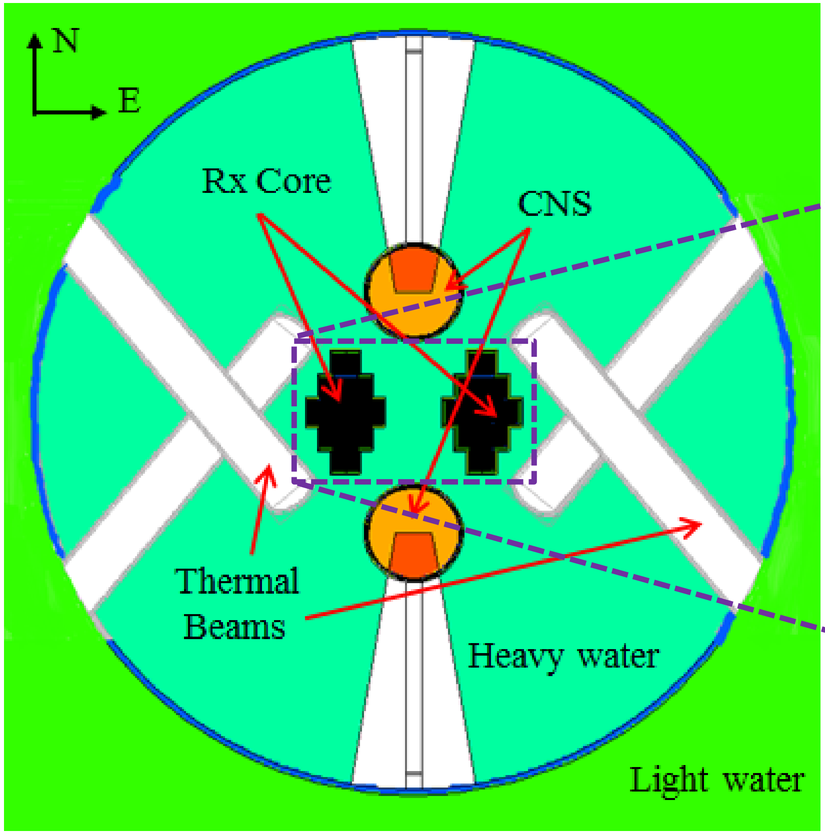
Future NIST Neutron Source: options under consideration

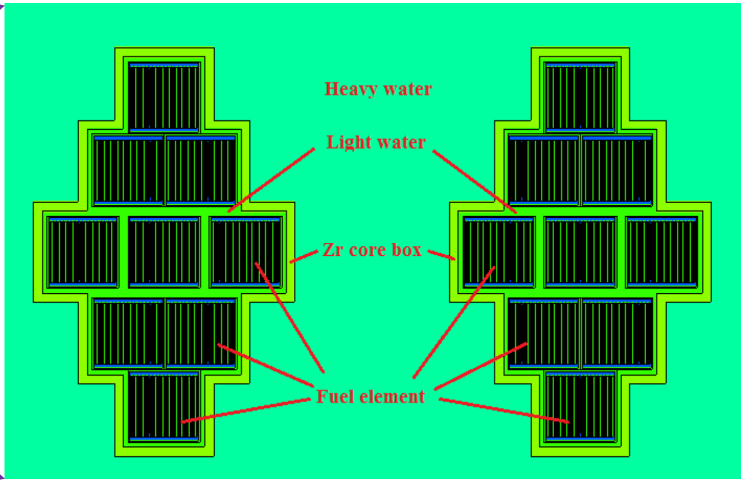
Upgrade NBSR for higher performance with LEU

Report from ROE team expected in 2017

or

New LEU-based reactor

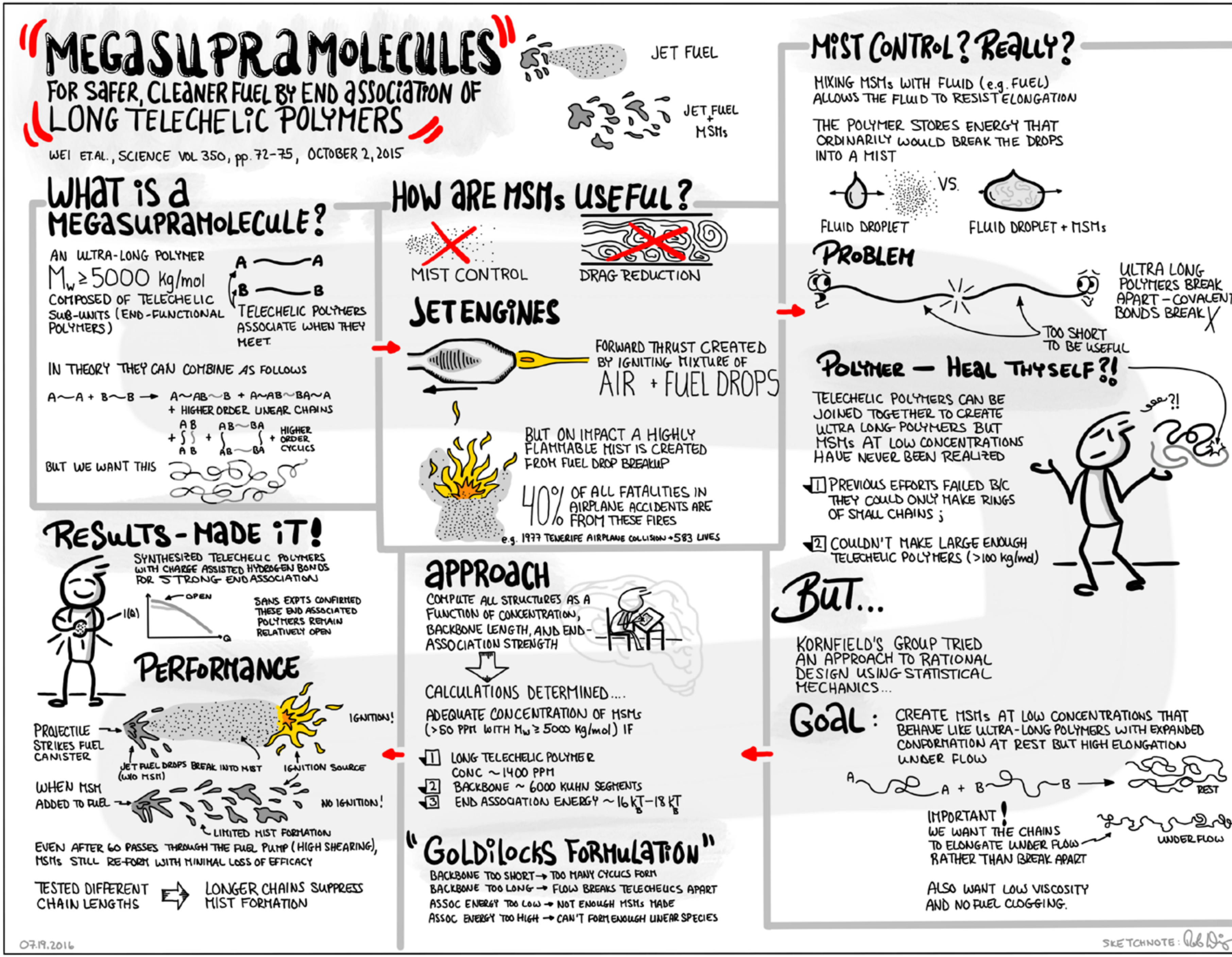




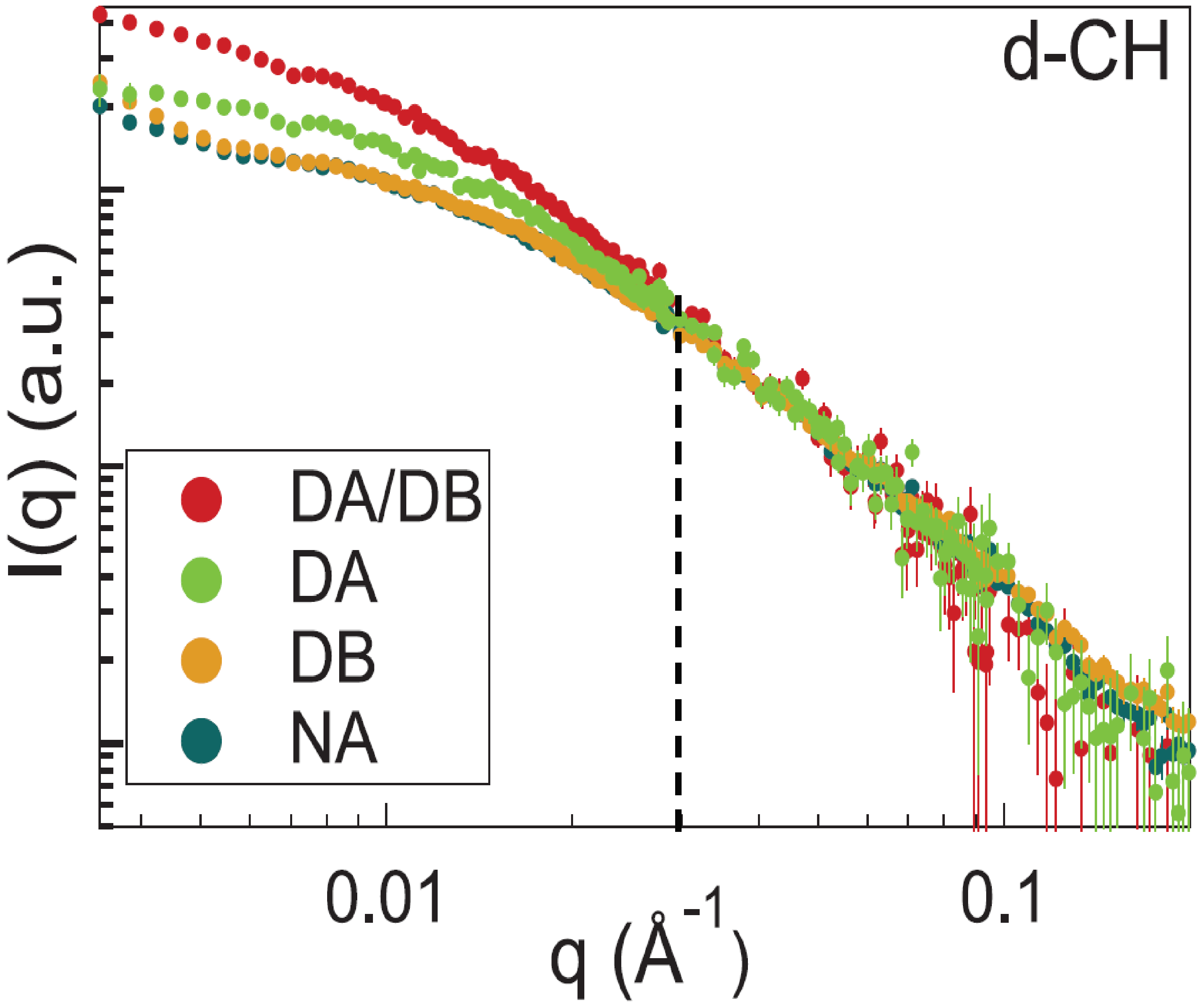
The core consists of total 18 fuel elements which are evenly distributed into two horizontal split regions.

MIST PREVENTION IN JET FUEL

VIDEO: JET FUEL


"MEGASUPRAMOLECULES"
FOR SAFER, CLEANER FUEL BY END ASSOCIATION OF LONG TELECHELIC POLYMERS
WEI ET.AL., SCIENCE VOL 350, pp. 72-75, OCTOBER 2, 2015
JET FUEL
JET FUEL + MSMs
WHAT IS A MEGASUPRAMOLECULE?
AN ULTRA-LONG POLYMER
Mw ≥ 5000 kg/mol
COMPOSED OF TELECHELIC SUB-UNITS (END-FUNCTIONAL POLYMERS)
A ~ A
B ~ B
TELECHELIC POLYMERS ASSOCIATE WHEN THEY MEET
IN THEORY THEY CAN COMBINE AS FOLLOWS
A~A + B~B → A~AB~B + A~AB~BA~A
+ HIGHER ORDER LINEAR CHAINS
+ HIGHER ORDER CYCLICS
BUT WE WANT THIS
HOW ARE MSMs USEFUL?
MIST CONTROL
DRAG REDUCTION
JET ENGINES
FORWARD THRUST CREATED BY IGNITING MIXTURE OF AIR + FUEL DROPS
BUT ON IMPACT A HIGHLY FLAMMABLE MIST IS CREATED FROM FUEL DROP BREAKUP
40% OF ALL FATALITIES IN AIRPLANE ACCIDENTS ARE FROM THESE FIRES
e.g. 1977 TENERIFE AIRPLANE COLLISION → 583 LIVES
MIST CONTROL? REALLY?
MIXING MSMs WITH FLUID (e.g. FUEL) ALLOWS THE FLUID TO RESIST ELONGATION
THE POLYMER STORES ENERGY THAT ORDINARILY WOULD BREAK THE DROPS INTO A MIST
VS.
FLUID DROPLET
FLUID DROPLET + MSMs
PROBLEM
ULTRA LONG POLYMERS BREAK APART - COVALENT BONDS BREAK
TOO SHORT TO BE USEFUL
POLYMER - HEAL THYSELF?!
TELECHELIC POLYMERS CAN BE JOINED TOGETHER TO CREATE ULTRA LONG POLYMERS BUT MSMs AT LOW CONCENTRATIONS HAVE NEVER BEEN REALIZED
1 PREVIOUS EFFORTS FAILED B/C THEY COULD ONLY MAKE RINGS OF SMALL CHAINS;
2 COULDN'T MAKE LARGE ENOUGH TELECHELIC POLYMERS (>100 kg/mol)
BUT...
KORNFIELD'S GROUP TRIED AN APPROACH TO RATIONAL DESIGN USING STATISTICAL MECHANICS...
GOAL: CREATE MSMs AT LOW CONCENTRATIONS THAT BEHAVE LIKE ULTRA-LONG POLYMERS WITH EXPANDED CONFORMATION AT REST BUT HIGH ELONGATION UNDER FLOW
A ~ A + B ~ B → REST
IMPORTANT! WE WANT THE CHAINS TO ELONGATE UNDER FLOW RATHER THAN BREAK APART
UNDER FLOW
ALSO WANT LOW VISCOSITY AND NO FUEL CLOGGING.
APPROACH
COMPUTE ALL STRUCTURES AS A FUNCTION OF CONCENTRATION, BACKBONE LENGTH, AND END-ASSOCIATION STRENGTH
CALCULATIONS DETERMINED....
ADEQUATE CONCENTRATION OF MSMs (>50 PPM WITH Mw ≥ 5000 kg/mol) IF
1 LONG TELECHELIC POLYMER CONC ~1400 PPM
2 BACKBONE ~6000 KUHN SEGMENTS
3 END ASSOCIATION ENERGY ~16 kBT-18 kBT
"GOLDILOCKS FORMULATION"
BACKBONE TOO SHORT → TOO MANY CYCLICS FORM
BACKBONE TOO LONG → FLOW BREAKS TELECHELICS APART
ASSOC ENERGY TOO LOW → NOT ENOUGH MSMs MADE
ASSOC ENERGY TOO HIGH → CAN'T FORM ENOUGH LINEAR SPECIES
RESULTS - MADE IT!
SYNTHESIZED TELECHELIC POLYMERS WITH CHARGE ASSISTED HYDROGEN BONDS FOR STRONG END ASSOCIATION
OPEN
I(Q)
Q
SANS EXPTS CONFIRMED THESE END ASSOCIATED POLYMERS REMAIN RELATIVELY OPEN
PERFORMANCE
PROJECTILE STRIKES FUEL CANISTER
IGNITION!
JET FUEL DROPS BREAK INTO MIST (W/O MSM)
IGNITION SOURCE
WHEN MSM ADDED TO FUEL
NO IGNITION!
LIMITED MIST FORMATION
EVEN AFTER 60 PASSES THROUGH THE FUEL PUMP (HIGH SHEARING), MSMs STILL RE-FORM WITH MINIMAL LOSS OF EFFICACY
TESTED DIFFERENT CHAIN LENGTHS ⇒ LONGER CHAINS SUPPRESS MIST FORMATION
07.19.2016
SKETCHNOTE:

MIST PREVENTION IN JET FUEL



MIST PREVENTION IN JET FUEL

VIDEO: JET FUEL W/MSM UNSHEARED

MIST PREVENTION IN JET FUEL

VIDEO: JET FUEL W/MSM SHEARED