The NIST Wolter optic neutron microscope



Daniel S. Hussey MC Daugherty, Y Kim, JM LaManna, DL Jacobson Physical Measurement Laboratory National Institute of Standards and Technology

B Khaykovich, MIT

Workshop on Neutron Focusing Optics – NFO Mar 2 – 3, 2023 Paul Scherrer Institute, Villigen, CH

Limbo photo credit: By Endlisnis - Street limbo 3, CC BY 2.0, https://commons.wikimedia.org/w/index.php?curid=45772088



Timeline Spatial Resolution Development at NIST



The real limbo competition has been to try and see water inside the catalyst layers of PEMFC during operation



- 250 μm (1-30 Hz frame rate): In-plane studies of total water content and manifold.
- 25 μm (20 minute): Through plane water distribution to begin GDL transport studies.
- 10 μm (20 minute): More accurate measurement of diffusion media as well as temperature driven phase change flow and thermal osmosis, studies of PGM-free catalyst layers.
- 1.5 μm (2 hours): GadOx centroiding improves resolution but requires long exposure times.
- Thinner granular scintillators yield resolution on order of thickness, Gd-157 enables few microns
- Wolter Optics 3 μm (1 s): A neutron lens will improve both spatial and time resolution; spatial resolution is easier with magnification; time resolution improves with focusing



Real MEA's aren't flat -> need 3D





The Limits of Pinhole Optics and Conventional Neutron Imaging

- Poke hole in reactor wall, form image of core at detector
- Best resolution when object *contacts* detector due to ~cm sized apertures
 - No geometric magnification
- Resolution derived from collimation, producing geometric blur:

 $\lambda_g \approx z d / L$

- Flux goes as (d/L)², Small d and/or large L → small Flux → Ø





If only we had a neutron imaging lens ...

But:

- $n \sim 1 10^{-6} \lambda^2$ (similar to x-rays)
- Neutrons are neutral and neutron beams are large, not points, many electron and x-ray tricks don't translate



Transforming x-ray telescopes into neutron microscopes



Wolter Optics power CHANDRA



NiCo-foil Focused X-ray Solar Imager



The neutron lens is based on mirror foil Wolter Optics:

- Need to realize 1 arcsec angular resolution
- ➤ X1000+ flux
- Image magnification for spatial resolution of 3 μm
- Achromatic lens
- ~1 m separation between lens, object, and detector

Win-win over pinhole cameras: boosts <u>time</u> and <u>spatial</u> resolution

The First Neutron Microscope images



Detector, 2.6 m from Optic, 3.2 m from sample



a)

Pinhole Grid Conventional Image



Prototype optic



Pinhole Grid Microscope Image





D. Liu et al, Appl. Phys. Lett. 102, 183508 (2013); doi: 10.1063/1.4804178

Field curvature

- Ray-tracing results of concentric rings for a parabolic-parabolic lens with M=1x, 8m focal length, 6 cm diameter
- The best focal surface is not planar but has curvature
- Resolution degrades with increasing offset from the center



NIST National Institute of Standards and Technology U.S. Department of Commerce

Field Curvature

• vs. Mirror Radius (L = 20 cm) • vs. Mirror Length (R = 3.5 cm)



- Paraboloid-Paraboloid configuration, M = 1, 8 m focal length
- Field curvature is determined by quadratic fit
- Curvature ~ L / \sqrt{R}
- Solution: Many short mirrors as radius is optimized for flux collection



- Plot of the standard deviations along the best focus lines (field curves)
- The decay rate of resolution is seen to depend mostly on radius rather than mirror length



Wolter Optics: Mapping Inhomogenities in Quantum Magnets



- $\mbox{-} \mbox{Quantum magnet } \mbox{T}_{\mbox{C}}$ can depend on composition
- and on pressure
- $\,{}^{\bullet}\,T_{\rm C}$ measures of samples in clamp cells in cryostats can suffer from poor SNR
- Neutron depolarization imaging is a robust probe, but poor image resolution
- Wolter optics are a neutron image-forming lens
- A lens preserves resolution even with:
 - Bulky environments
 - Spin polarization







Neutron Depolarization Imaging with a Wolter Optic



Depolarization vs temperature



10 FEB 2017: Photo at ANTARES beam, He-3 holding field not shown

P. Jorba et al. JMMM 2019

Mapping Inhomogenities in Quantum Magnets



- Factor of 10 improvement in spatial resolution over standard (100 μm vs 1 mm)
- Factor of 2 improvement in time resolution over standard (3 min. vs 6 min.)
- Distribution in $T_{\rm C}$ likely due to freezing of pressure medium

Prototype optic used for measurements at ANTARES at FRM2



P. Jorba et al. JMMM 2019

The mirrors image the intensity pattern at the focal plane



National Institute of Standards and Technology U.S. Department of Commerce

P. Jorba et al. JMMM 2019

Wolter mirrors

We intend to create the first neutron analog of Hooke's microscope



Sketch of parameters optimized through ray tracing with the constraints:

- minimum ri = 2 cm
- figure error is 10 μrad, so maximum object focal length is 75 cm to have 3 μm resolution
- Objective optic length limited to 20 cm to reduce field curvature
- See:
 - M Abir et al, J. Imaging (2020), 6(10), 100; doi: 10.3390/jimaging6100100
 - DS Hussey et al NIMA (2021) 987, 164813, doi: 10.1016/j.nima.2020.164813.



Some comments on NIST ray tracing – Monte Carlo

- Written in MATLAB to take advantage of CUDA capabilities (arrayfun is fast)
 - Also independent of the effort at MIT using McSTAS
 - Random particle generation using neutron guide spectrum and divergence
 - Includes roughness, expected mirror figure error, gravity
 - Not particularly well documented ...
- Main code about 850 lines, plus definitions of guide, optics, sample(s), aperture(s), detector(s)
- Instrument "designed" using an Excel spreadsheet, specifying order and geometry of each optical component
- With no sample, two Wolter optics, 1 s exposure (10^11 neutrons) requires 1 h on an nVIDIA TI-1085 GPU
- With a sample, a fair bit longer (depends on # slices, etc.), waiting on an A100 to arrive to load sample volume into GRAM



Defining an "Instrument"

Ŀ	5 •∂-∓				oldNIST_W	/olterOptic.x	dsm [Read-On	oft Excel			⊡	≣ চিশ ঔশ ∓							oldNIST_WolterC		
Fil	le Home	Insert Pag	e Layout	Formulas	Data	Review	View Q	Tell me wha	it you war	nt to do			File	Hom	e Ins	sert	Page Layout	Formulas	Data	Review	
D14	4 👻 :	$\times \checkmark$	f _x										B10	Ŧ		x	f _x tr	ansverse			
		Α				В			c	D	E						,				
1	Name		Va	lue											Α				В		
2 (Output Path		F:\	WolterOptics	5\								1 Na	me			Value				
3 (Output Prefix		sir	n									2 Sa	mple Labe			Rings				
4 5	Simulation Event	3.3	31E+11									3 Sample Size				20	20				
5 I	Incldue Gravity		ye	s							Ray Trace (W	/olter	4 Tra	nslation			5				
6 E	6 Element 1			guide										nnle Type			Volume	Volume			
7 6	Element 2			optic									6 Lin	o Width			0.05	0.05			
8 E	Element 3			aperture													0.05	0.05			
9 E	Element 4		sa	mples									7 Lin	e Spacing			0.2				
10 E	10 Element 5			otic							8 Sample Pixel Pitch						0.005	0.005			
11 E	1 Element 6			detector							9 Volume Directory					F:\Wolt	F:\WolterOptics\				
12													10 Slic	e Orienta	ation		transve	rse			
13									ľ				11 Att	enuation	Length	Units	cm^-1				
14									ļ	Ł			12								
15													12								
17													14								
17					D			o lite i l				-	14								
	Ger	neralInput	Aperture	Objectiv	eRadiiScra	tchSheet	Condense	RadiiScratch	nsheet	Sample5	Sample6	Sam									
Read	dy									E											



Defining an optic

H	5 -∂	÷			oldNIST_WolterOptic.xIsm [Read-Only] - Microsoft Excel										G S C → oldNIST_W						
File	Hon	ne	Insert Page	e Layout	Formulas	Data	Review	View	♀ Tell i	me what you	want to do			File	Home	Insert	Page Layo	ut Formu	las Data	Revie	
			_																		
	-		XV	<i>f_x</i> =H\$:	12* SQRT(12*12/(H\$14	4*H\$14) - 1)						16	-	: ×	$\checkmark f_x$				
	A		В	с		D	E	:	F	G	н	I.	J		А			В			
Sł	nell num R	adii		zlens	Paramete	r Definitio	ns		Z H1	Shell 1		Z H2	Shell 2	Name	2		Value				
	1		59	9 0	fi			750	1106.433	53.25167		1106.841	47.16104	Optic	Label		condenser				
	2		52.25167397	7 1	fs			7500	1107.433	53.30963		1107.841	47.21237	Optic	Туре		ph				
	3		46.16103728	3 2	rimax			59	1108.433	53.36759	52.25167397	1108.841	47.26369	Focal	Lengths		1000, 1000, 1	1000, 1000, 10	000, 1000		
	4		40.66392721	L 3	thetaH1		0.0078	66504	1109.433	53.42552	0.006966777	1109.841	47.315	Inters	ection Ra	adii	26.5, 29.417,	32.545, 35.8	99, 39.496, 4	43.353	
	5		35.70245882	2 4	thetaE1		0.0785	04992	1110.433	53.48345	0.069556507	1110.841	47.3663	Sectio	on Length	s	300, 300				
	6		31.22440567	7 5	theta1		0.0215	92874	1111.433	53.54137	0.019130821	1111.841	47.41759	Trans	lation		4000				
	7		27.18264302	2 6	cH1		456.43	28148	1112.433	53.59928	456.8407884	1112.841	47.46888	Trans	lation Fro	m	Nearest Ups	tream			
	8		23.53464683	3 7	cE1		4581.4	32815	1113.433	53.65718	4581.840788	1113.841	47.52015	Surfa	ce Roughi	ness	3				
	9		20.24204266	58	betaH1		12506	30.222	1114.433	53.71506	1250491.42	1114.841	47.57141	Angu	lar Resolu	ition	0.0001				
	10		() 9	betaE1		12468	011.22	1115.433	53.77294	12474881.59	1115.841	47.62267	Outp	ut Mirror		yes				
	11		(0 10	bH		24.074	86161	1116.433	53.83081	21.34251715	1116.841	47.67392	Tag S	nell		no				
	12		() 11	bE		76.533	70725	1117.433	53.88866	67.7707129	1117.841	47.72515	Mirro	r M-Value	2	1.2				
	13		() 12	аН		455.	79745	1118.433	53.94651	456.3419802	1118.841	47.77638								
	14		(13	aE		4582.0	72025	1119.433	54.00434	4582.341964	1119.841	47.8276								
	15		() 14	ZOH		8706.4	32815	1120.433	54.06217		1120.841	47.87881								
	16		() 15	ZOE		2918.5	67185	1121.433	54.11998		1121.841	47.93001								
	17		() 16	length			100	1122.433	54.17779		1122.841	47.9812								
	18		() 17	wall			1	1123.433	54.23558		1123.841	48.03238								
	19		() 18	radii				1124.433	54.29337		1124.841	48.08356								
	20		() 19	magnifica	tion		10	1125.433	54.35114		1125.841	48.13472								
				20					1126.433	54.4089		1126.841	48.18587								
	•	Ger	neralInput 🛛	Aperture1	Objectiv	veRadiiScra	tchSheet	Con	denseRadi	iScratchShe	et Sample	25 Samp	ole6 Sam	• •	s	ample5	Sample6	Sample1	Sample2	Samp	
ady			- Nunonar	mamore or								E		ady							



A few of the ray tracing studies







Looking at which mirrors in the Objective optic are "active", funds support 5 shells

The Final Optics Design: Expect 1000x gain in time resolution, magnification of 10, spatial resolution of 3 μ m



Detector Position: 11800 mm

> Perspective: Conventional Pinhole would need 3.5 mm diameter, and

- the beam would diverge:
- Pinhole flux: 5 × 10⁵
- Optics flux: 1×10^{10}

- <u>A gain of 2x10⁴!!!</u>



DS Hussey et al NIMA (2021) 987, 164813, doi: 10.1016/j.nima.2020.164813

Liquid water uncertainty for various methods/detectors



- Time and Spatial Resolution would approach a conventional synchrotron imaging beamline
- Sample environments like Furnaces, Griggs Rigs, Pressurized Fluid Flow Cells, Magnets, Cryostats,... will be straightforward to incorporate on the beamline
- Can improve quantitative analysis using a velocity selector to coarsely define the wavelength band $\Delta\lambda/\lambda \sim 10-15\%$



The Condenser optic nearly forms a point source -> Tomography only





NCNR Upgrade: Source

The NIST reactor is planning on a major upgrade of its cold source.

Current: Liquid Hydrogen *Future:* Liquid Deuterium (lower absorption) Estimate 10- 12 months for installation

Colder spectrum will benefit Wolter optics







New Liquid Deuterium Cold Source



NCNR Upgrade: Guides



Straight NG-6 guide will be replaced with a new curved guide

- This reduces guide failure risks (guides are 30 years old)
- Curvature removes reactor core fast neutron and gamma ray background
- Factor 3 gain in intensity



Thanks for your attention

- Neutron imaging is a valuable tool for water management studies in PEM FC&WE systems
- The Wolter optic neutron microscope will be a huge gain in time resolution at the finest spatial resolution currently achievable
- Tomography simulations are underway, but are time consuming
- It's hoped this new tool is ready for users in 2027 ... maybe with a CLS ...
- The Microscope is funded by a NIST Innovation in Measurement Science project
- Thanks to Boris Khaykovich (MIT) for the ongoing collaboration on Wolter optics implementation and the NASA MSFC X-ray optics group



Mandrels and hardware for diamond turning ... lathe under repair currently ...



Imaging Mandrel being turned at long last (02 MAR 2023)

