



DRACULA and EXED

Alan Hewat, Diffraction Group, ILL Grenoble



Diffractometer for
*RA*_{pid}
Collection over
Ultra
Large
Areas



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Comparison of TOF & CW Diffractometers

Jorgensen, J.D., Cox, D.E., Hewat, A.W., Yelon, W.B.

“Scientific opportunities with advanced facilities for neutron scattering”

Shelter Island Workshop, 1984

Nuclear Instruments and Methods in Physics Research B12 (1985) 525-561

Efficiency for a given resolution = time averaged flux on the sample
* sample volume
* detector solid angle

Large **detectors** + high **flux on the sample**



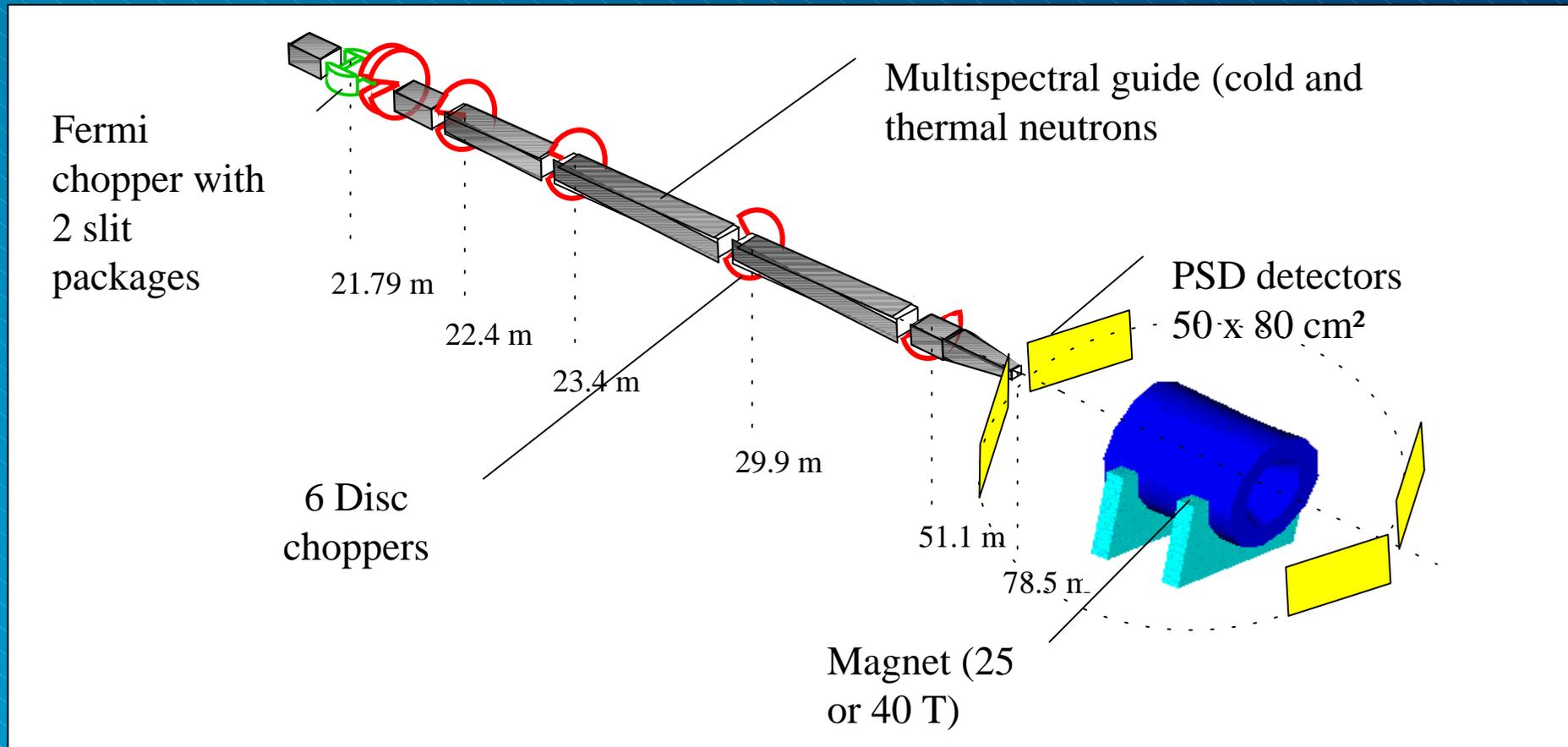
DRACULA and EXED

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Q: Why is the sample flux so high with TOF (EXED) ?

A: A relatively wide band of wavelengths is used with a converging guide



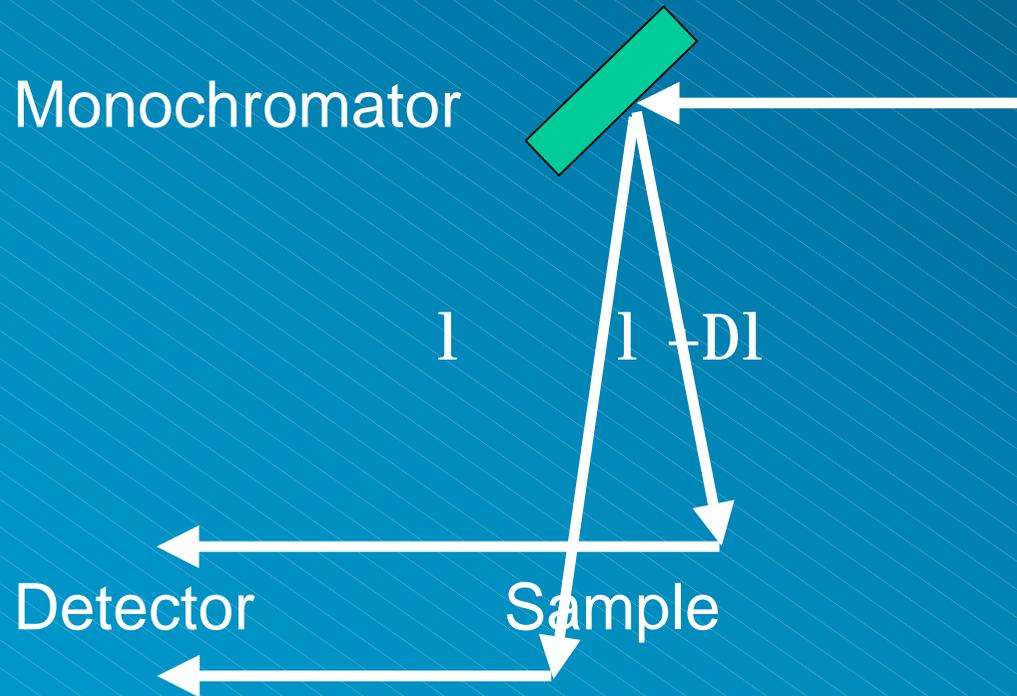
(Wavelength band 0.7 -> 1.8Å ultimately limited by frame overlap)

High Flux on DRACULA

Q: Why is the sample flux so high from a reactor

A: A relatively wide band of wavelengths is used (1% for 0.1% resolution)

Large Focussing Monochromators



Alan Hewat, EXED Workshop, May 2004, HMI Berlin

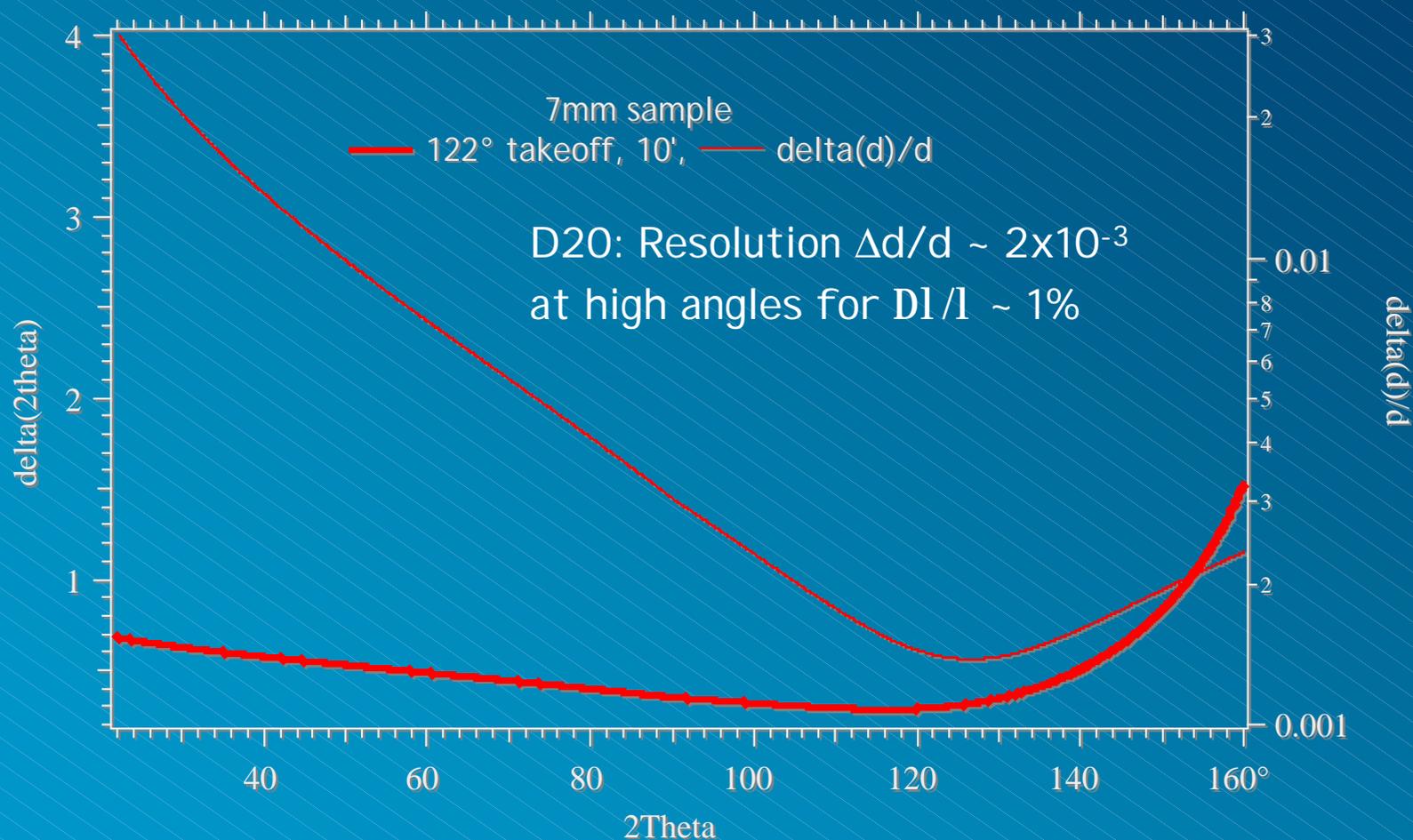


High Flux AND High Resolution



Q: Is High Resolution compatible with large DI/l ?

A: Yes. Resolution is INDEPENDENT of DI/l at the focussing (take-off) angle.



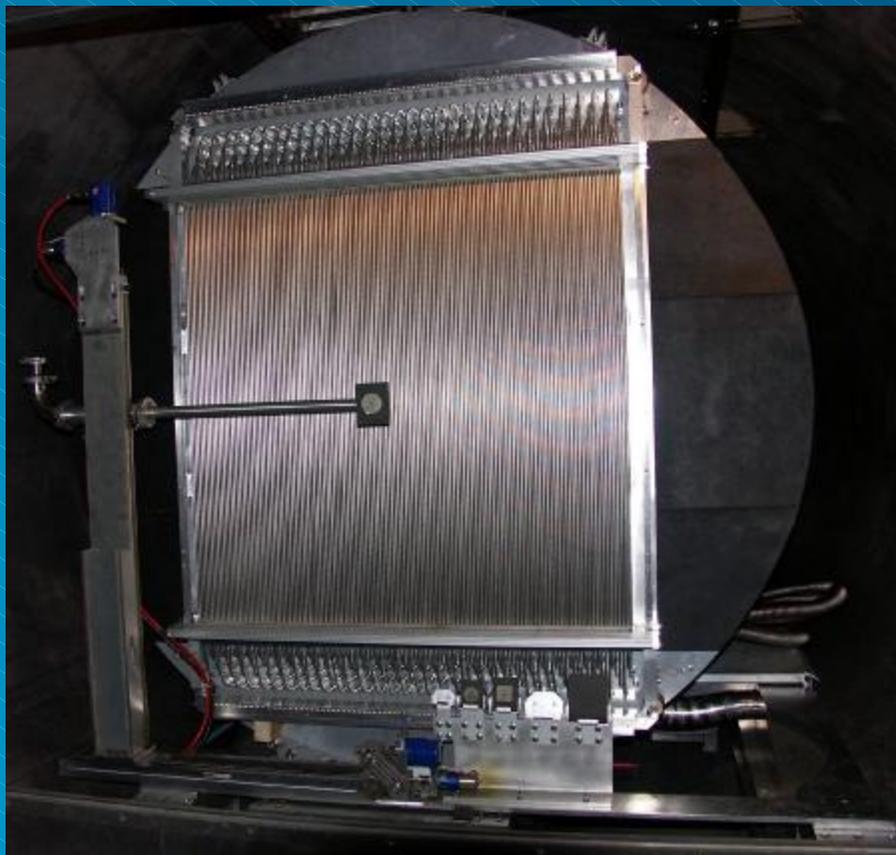


Array of linear wire PSD detectors for EXED



Q: How do we build a very large detector for EXED ?

A: An array of linear wire detectors c.f. Super-D2B and D22 at ILL



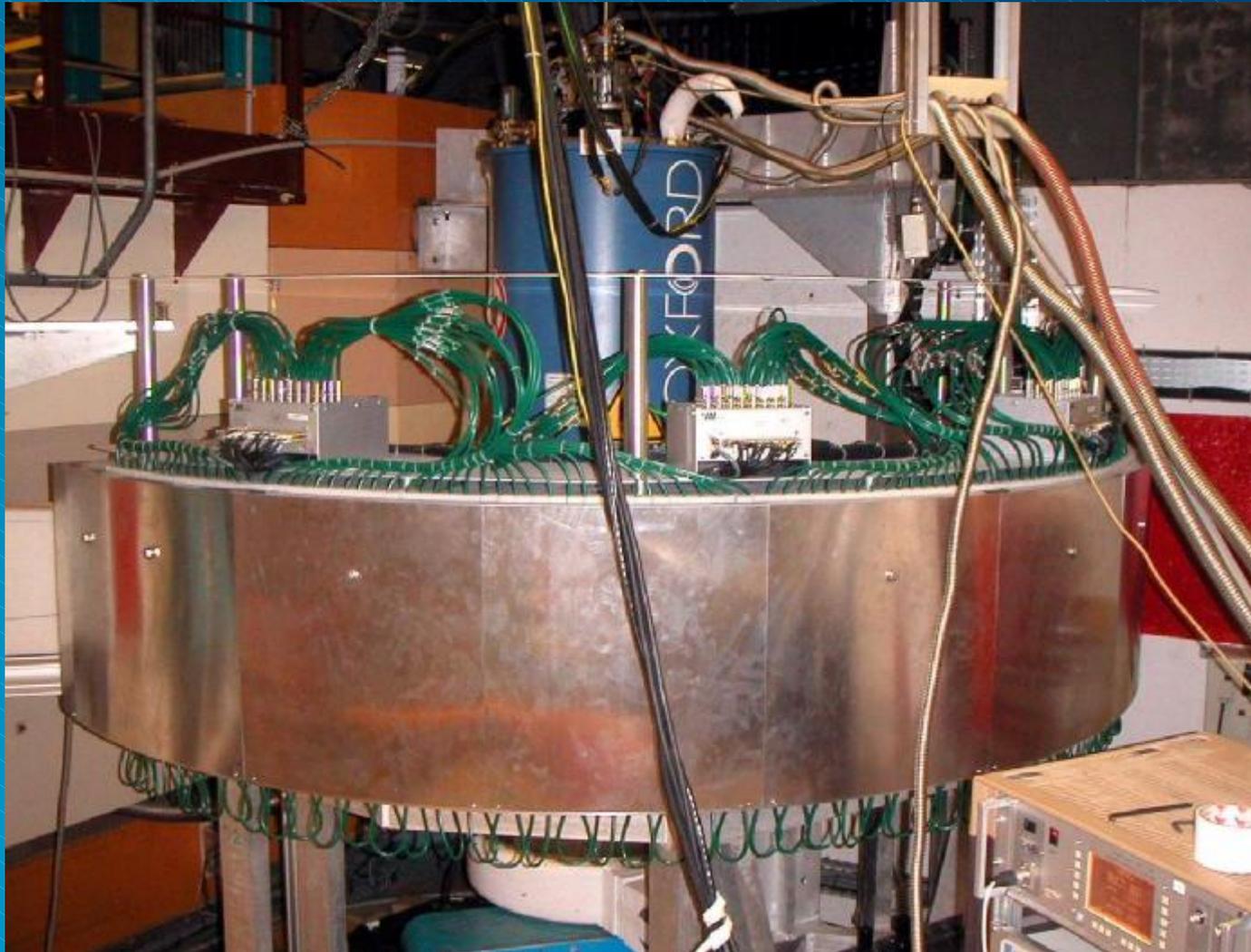
Array of 8mm x 1000mm linear wire detectors, D22



Array of linear wire PSD detectors on D2B



Array of linear wire PSD-tubes on Super-D2B at ILL



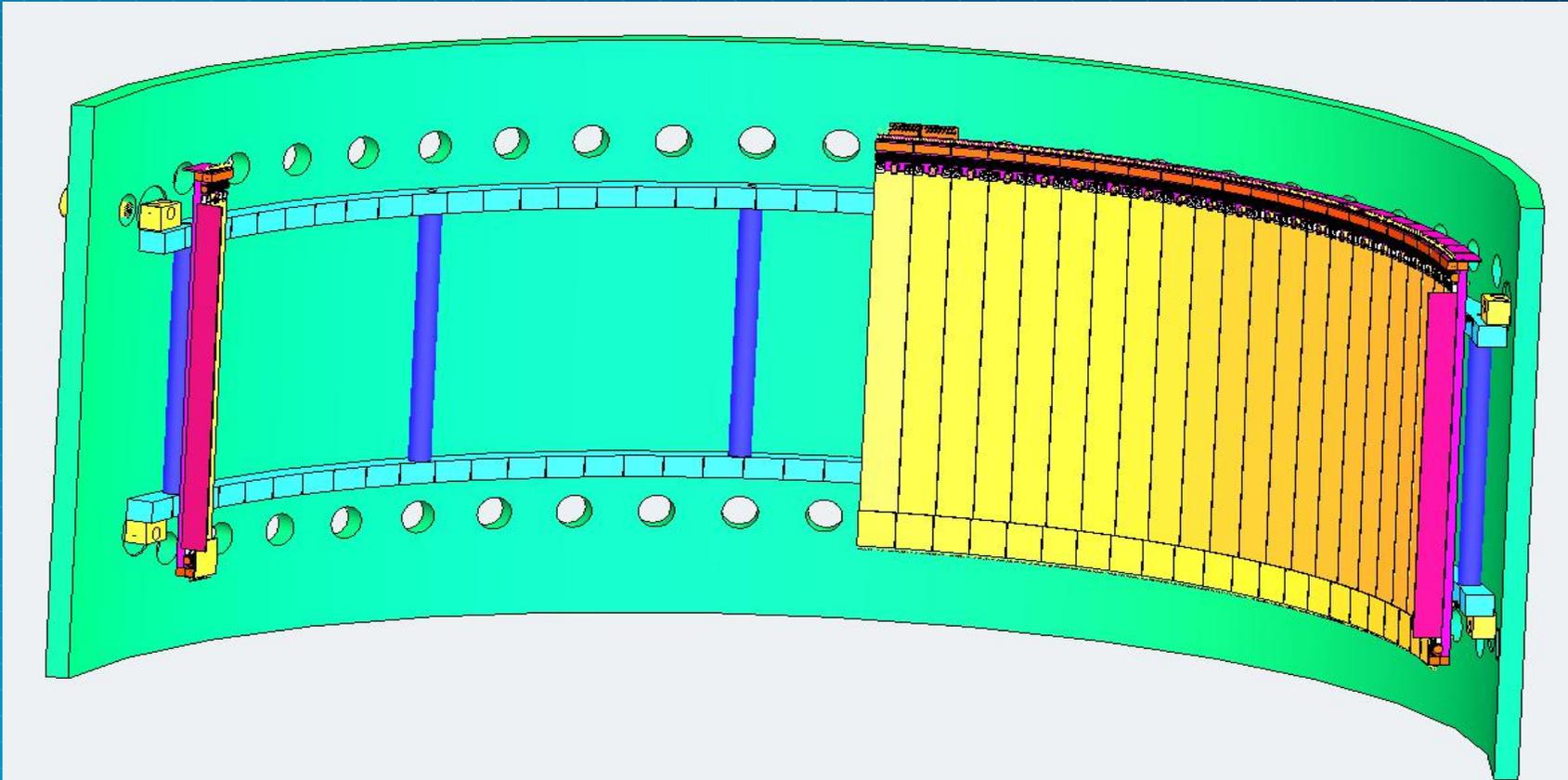


Linear-wire PSD detector for DRACULA



Q: How do we build a very large detector for Dracula ?

A: A new 2D linear wire He3-gas gas detectors developed at ILL & BNL



2D with Solid Angle > 1 steradian c.f. 0.27 on D20



2D linear-wire detector for DRACULA



D19 Millennium - A Revolution in large 2D Gas Detectors



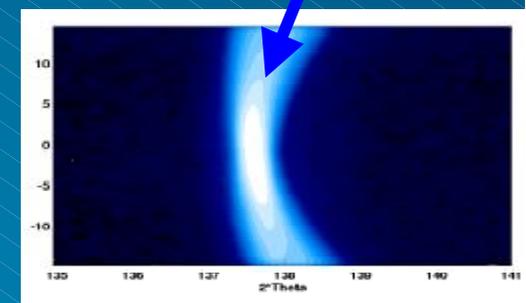
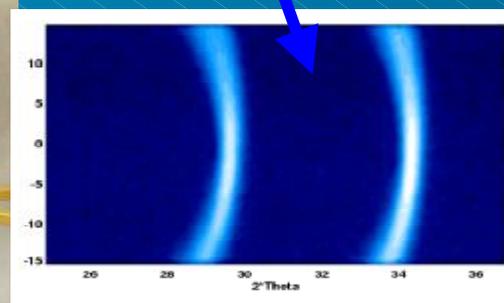
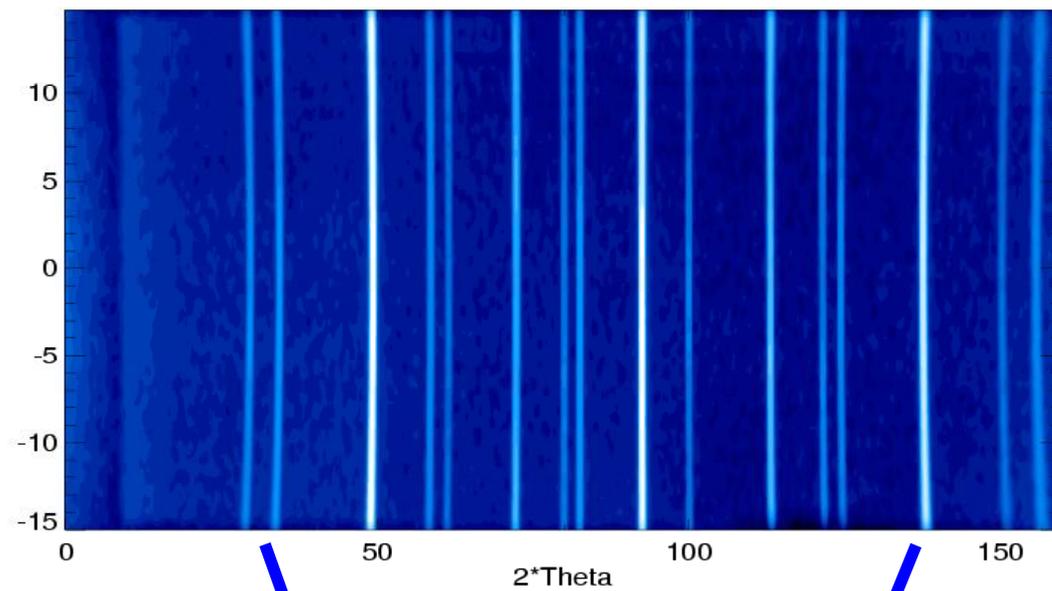


2D detectors for Powder Diffraction

How do they work in practice ?



UK-EPSC Super-D2B project at ILL



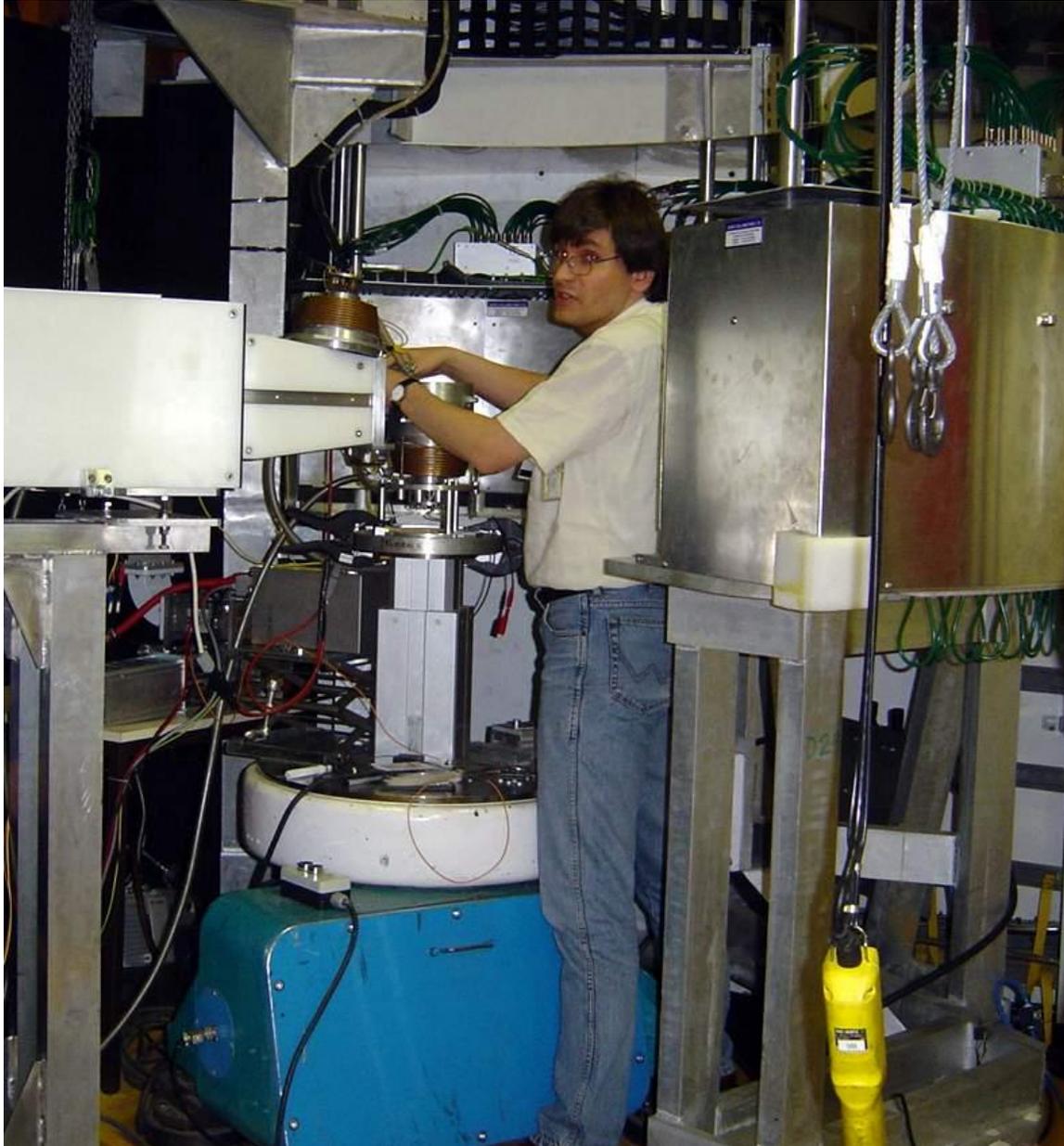
E.Suard, C.Ritter, A.Hewat, P.Attfield... (Edin.)

Alan Hewat, EXED Workshop, May 2004, HMI Berlin



Super-D2B at ILL

Very high temperatures on small samples



High T Microwave Furnace
on D2B (Boysen et al.)

...with Carsten Korte from Giessen



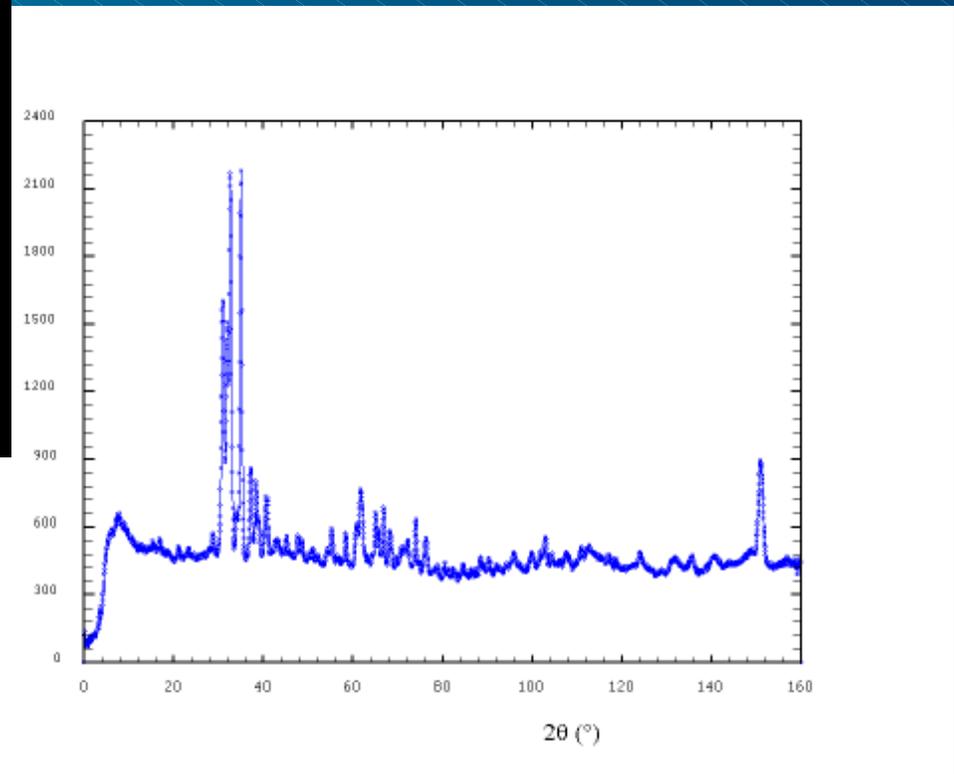
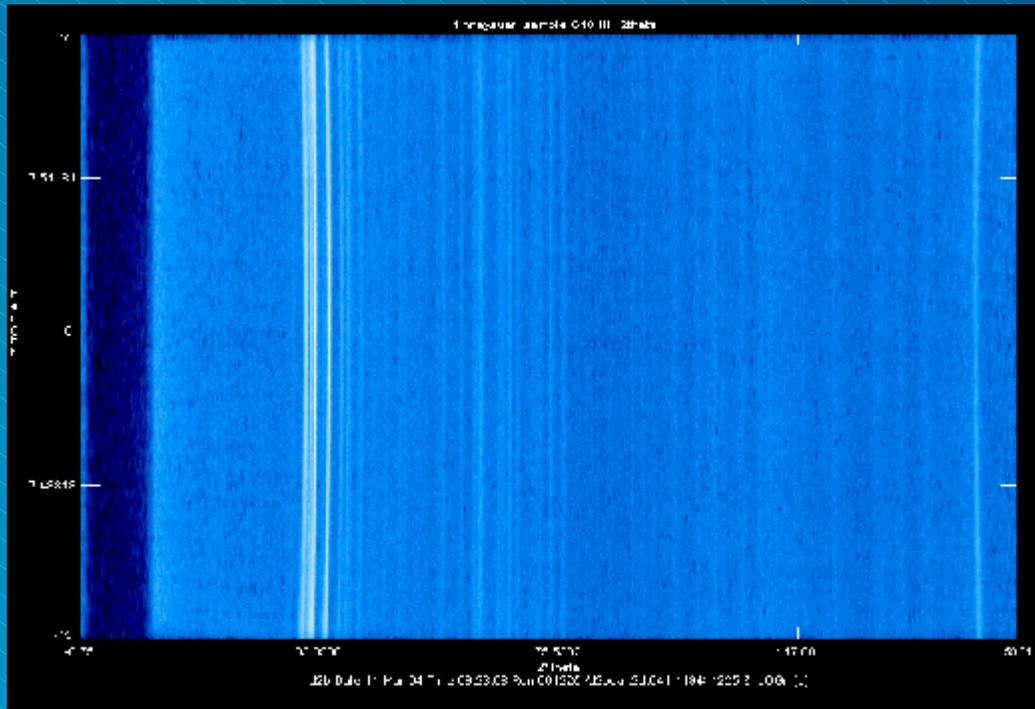


Super D2B at ILL

Very small samples with very low B/G



180 mg of HP Ice on Super D2B (J.Finney, E.Suard)



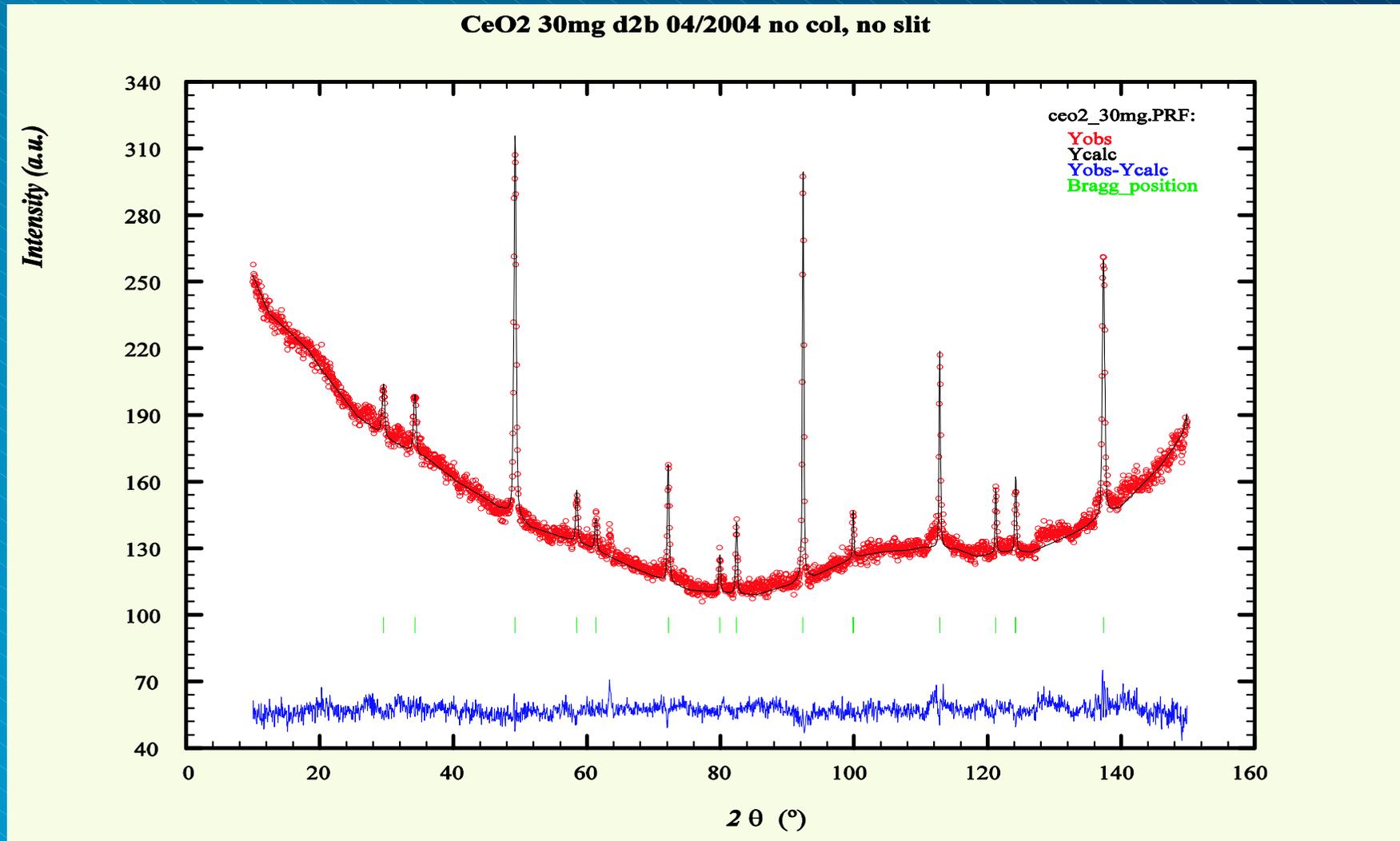


Super D2B at ILL

Very small samples with very low B/G



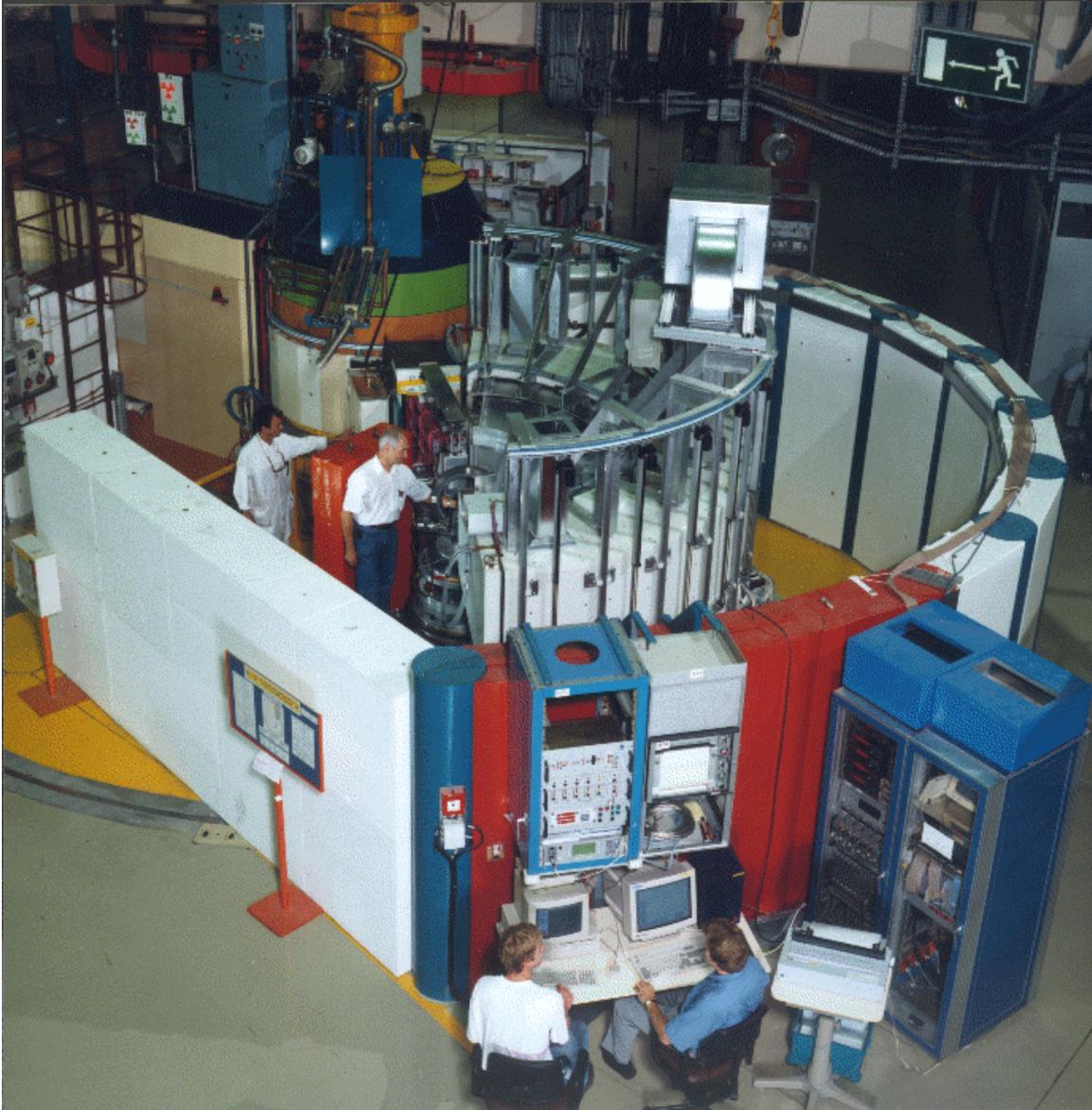
30 mg sample on Super D2B (E. Suard & C. Ritter, ILL)





D20 at ILL

Fast PSD Detector for very small samples



Large Microstrip Detector

A.Oed, P.Convert, T. Hansen, et al...





D20 at ILL

Applications of large fast detectors



New ceramics to replace metals in engineering components



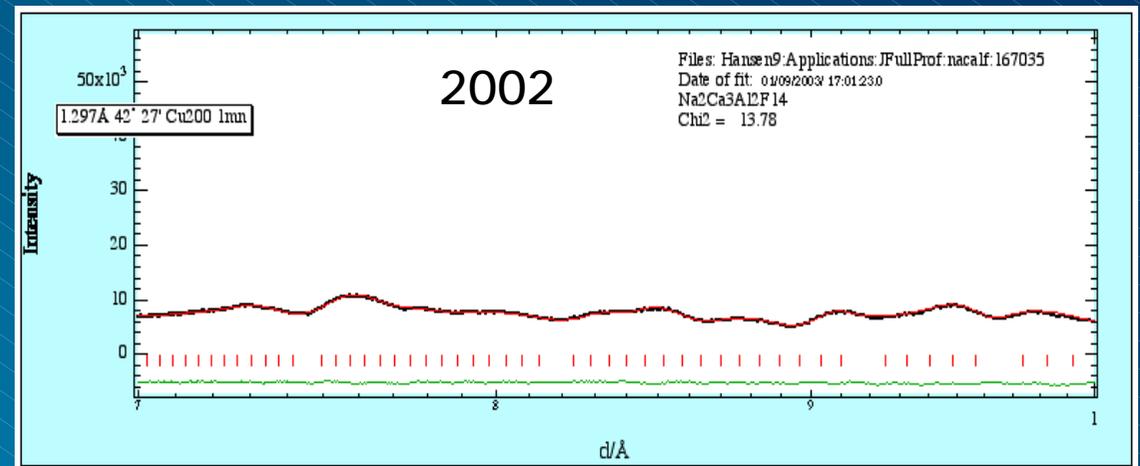
- | The explosive SHS reaction was studied in real time with neutrons
- | The reaction is exothermic, & heats the sample to 2200°C in <1 sec
- | The complete diffraction pattern (left) is collected at 300 ms intervals
A World Record
- | Knowledge of the SHS process allows us to prepare a pure Ti_3SiC_2 product

D.Riley, E.Kisi, T.Hansen, A.Hewat

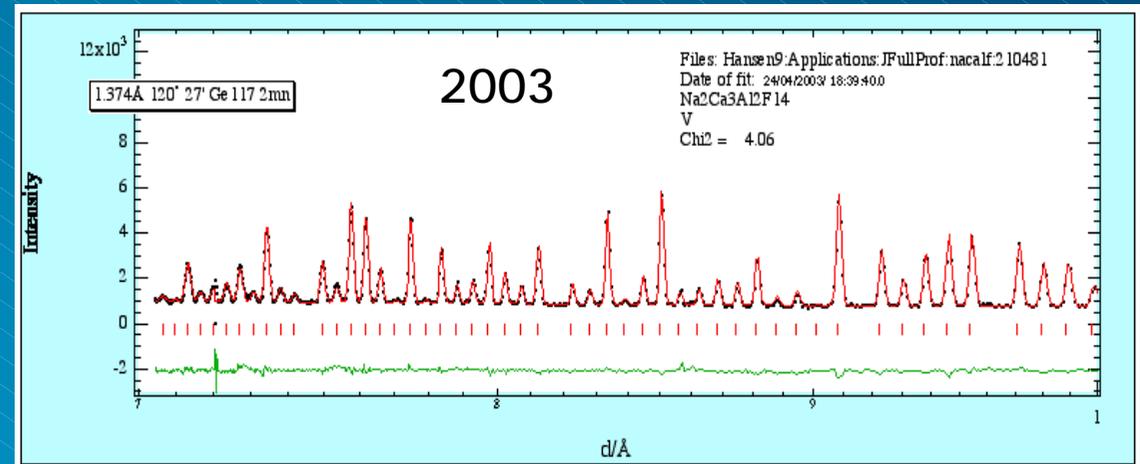
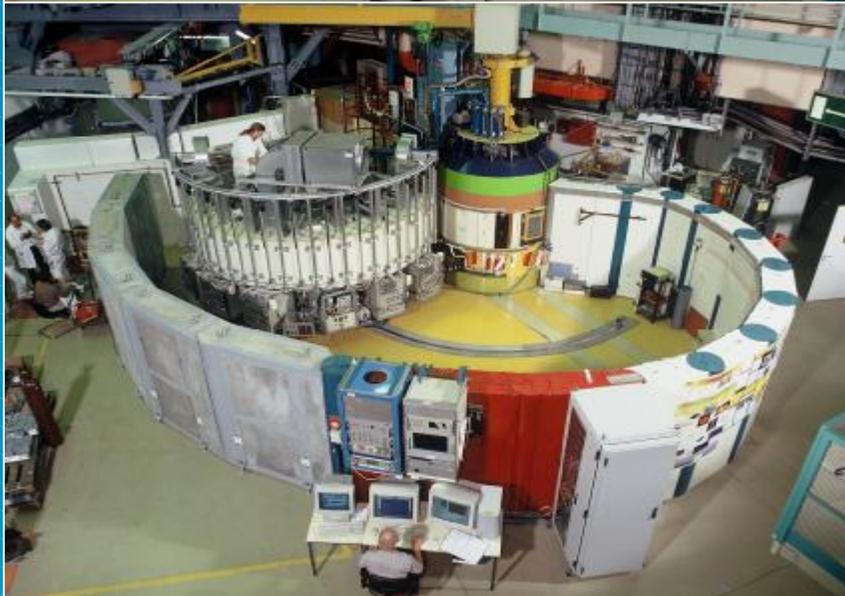


D20 – High Resolution but still very Fast

T.Hansen, P.Henry, P.Fischer



Before and After (data in 2 min.)



Higher D20 resolution since 2003



D20 – High Resolution but still very Fast

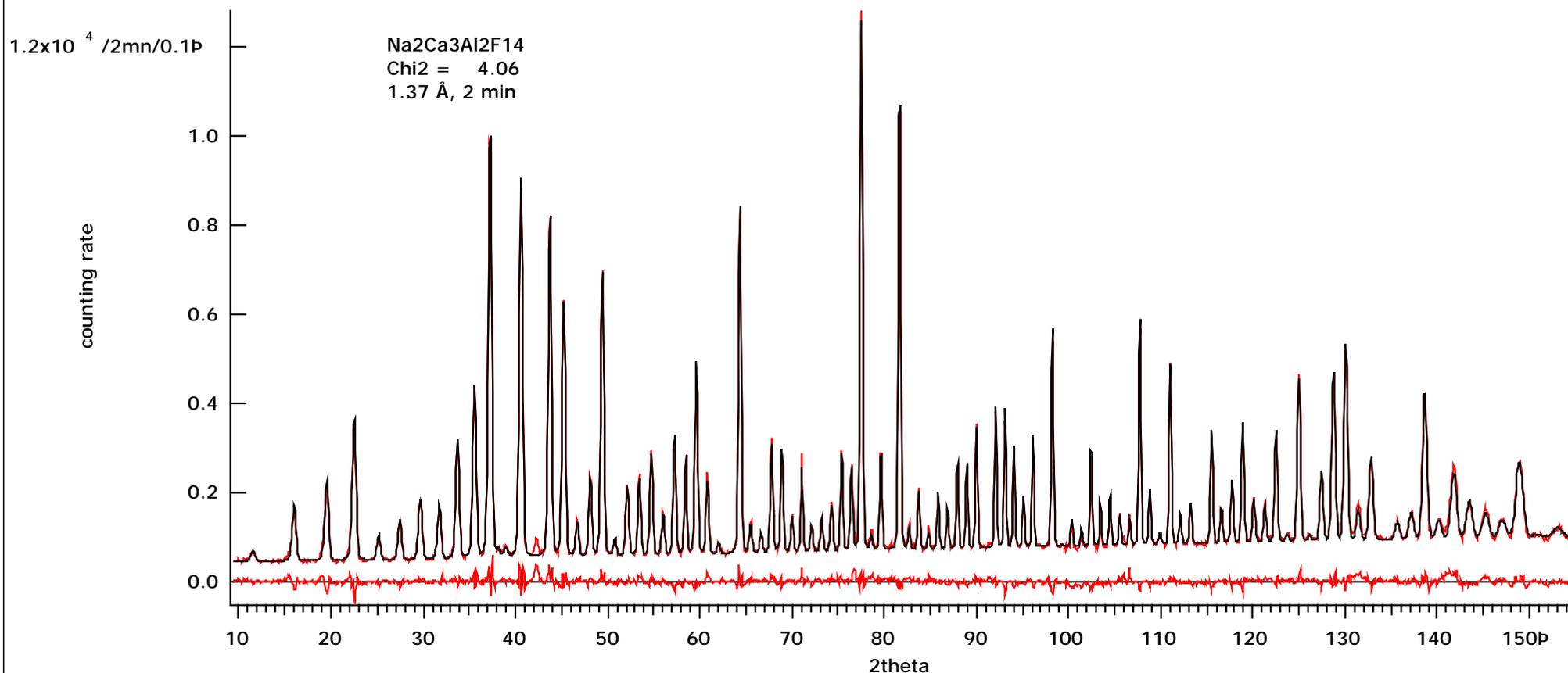
T.Hansen, P.Henry, P.Fischer



D20 is very fast (chemical kinetics)

Thomas Hansen (2003) ILL News, June 2003

2 minute D20 data for a $\sim 700 \text{ mm}^3$ sample of $\text{Na}_2\text{Ca}_3\text{Al}_2\text{F}_{14}$





GEM - High Resolution, Small Samples

P.Radaelli, A.Hannon, L.Chapon



GEM can measure very small samples

Radaelli, Hammon & Chapon (2003) Neutroni e Luce di Sincrotrone

~700 minute GEM data for a 2mm³ sample of Y₃Al₅O₁₂

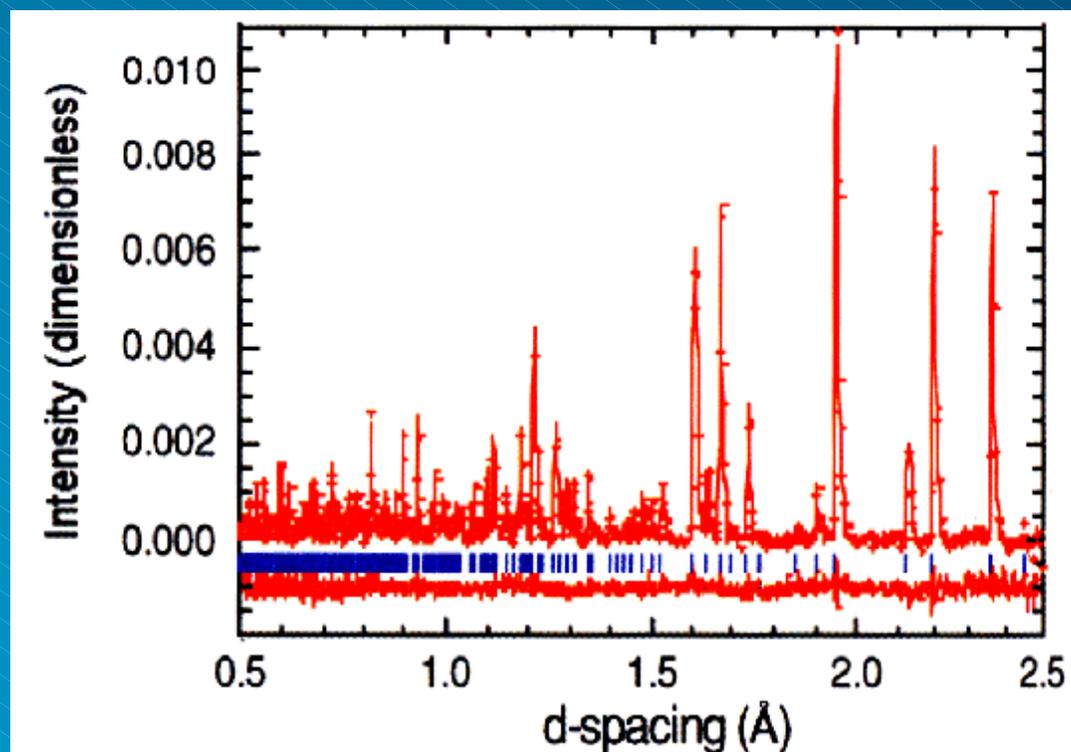


Fig. 7. Rietveld Refinement plot for a 2 mm² sample of Yttrium Iron Garnet (YAG), after an overnight data collection.



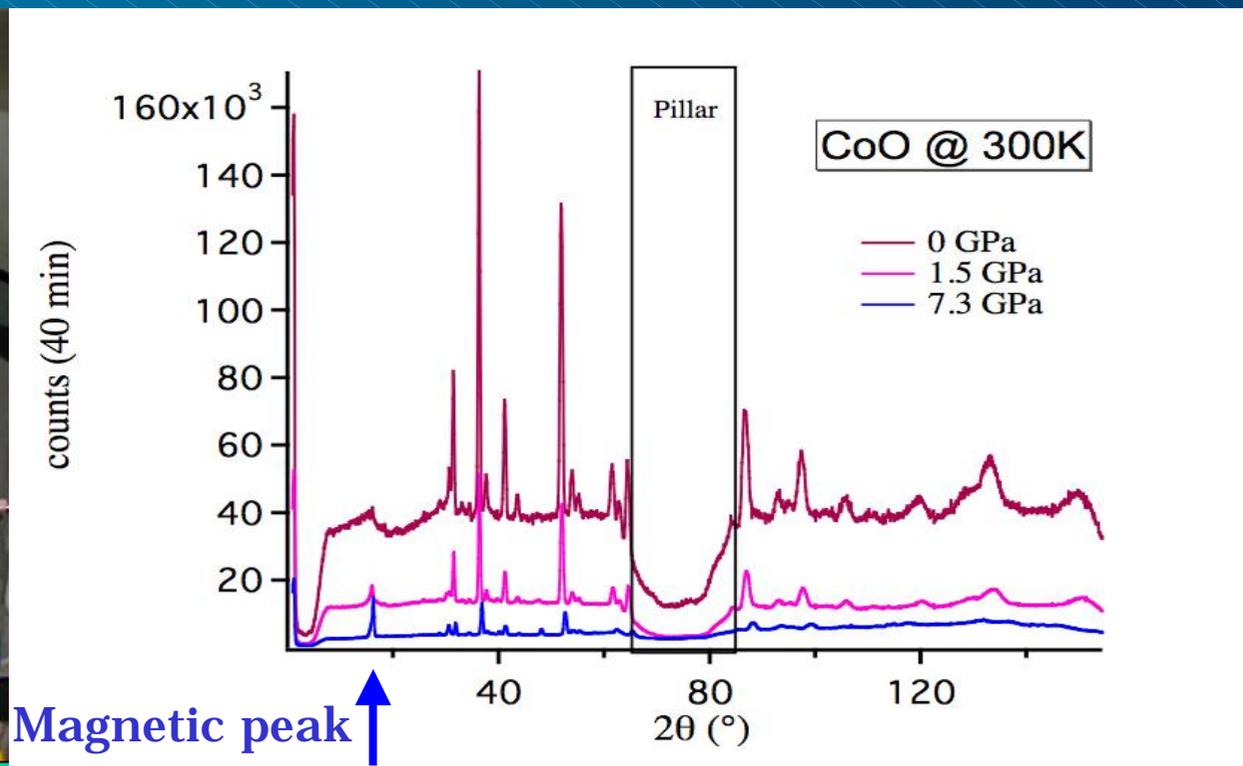
D2O - High Pressures, Small Samples



D2O with "large" Paris-Edinburgh Pressure Cell (50 Kg)

Kernavanois et al. (2003) Advanced Millennium Pressure Project

40 minute D2O data for a 100 mm³ sample of CO at 7.3 GPa



BUT low temperatures -> smaller cells -> 1-10 mm³ samples



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* sample volume
* detector solid angle

Large **detectors** + high **flux on the sample**



DRACULA and EXED

Comparison of TOF & CW Diffractometers

	D20	GEM
time averaged sample flux	5×10^7	$\sim 2 \times 10^6$
detector solid angle	0.27 sr	4.0 sr
efficiency	1.7	1



DRACULA and EXED

Comparison of TOF & CW Diffractometers

	D20	GEM	DRACULA
time averaged sample flux	5×10^7	$\sim 2 \times 10^6$	$\sim 10^8$
detector solid angle	0.27 sr	4.0 sr	1.5 sr*
efficiency	1.7	1	18

* Based on new D19 detector: R=760 mm, h=400 mm, 800 linear resistive wires covering 30°x160°



DRACULA and EXED

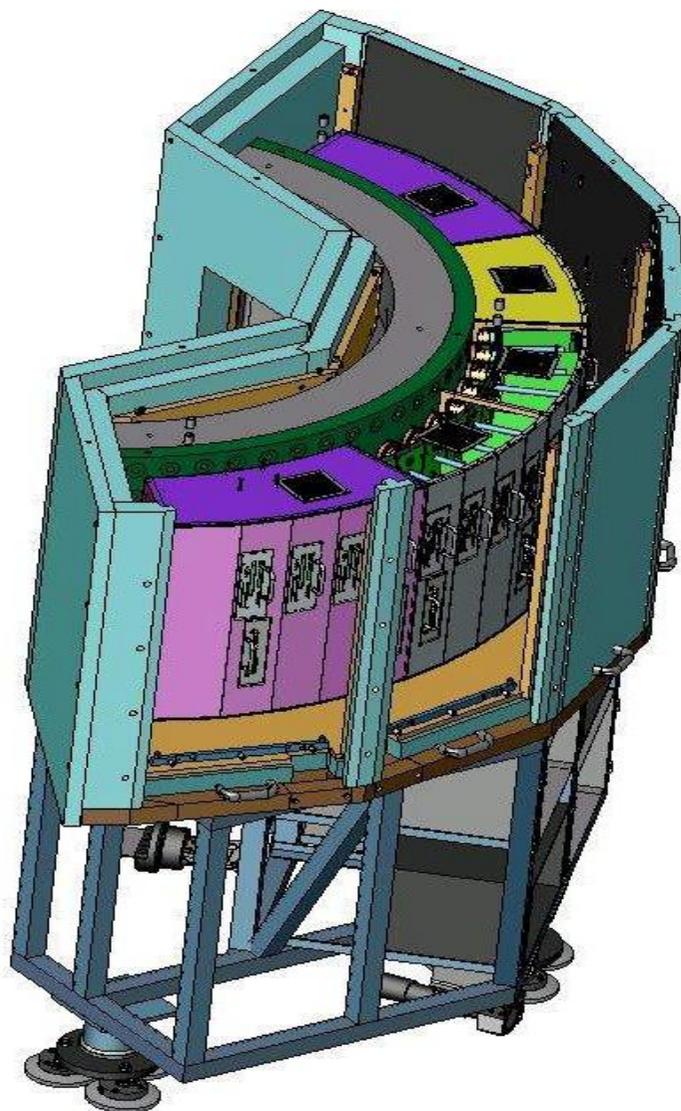
Comparison of TOF & CW Diffractometers

	D20	GEM	DRACULA	US-SNS
time averaged sample flux	5×10^7	$\sim 2 \times 10^6$	$\sim 10^8$	$\sim 2.5 \times 10^7$
detector solid angle	0.27 sr	4.0 sr	1.5 sr*	3.0 sr
efficiency	1.7	1	18	9

* Based on new D19 detector: R=760 mm, h=400 mm, 800 linear resistive wires covering 30°x160°

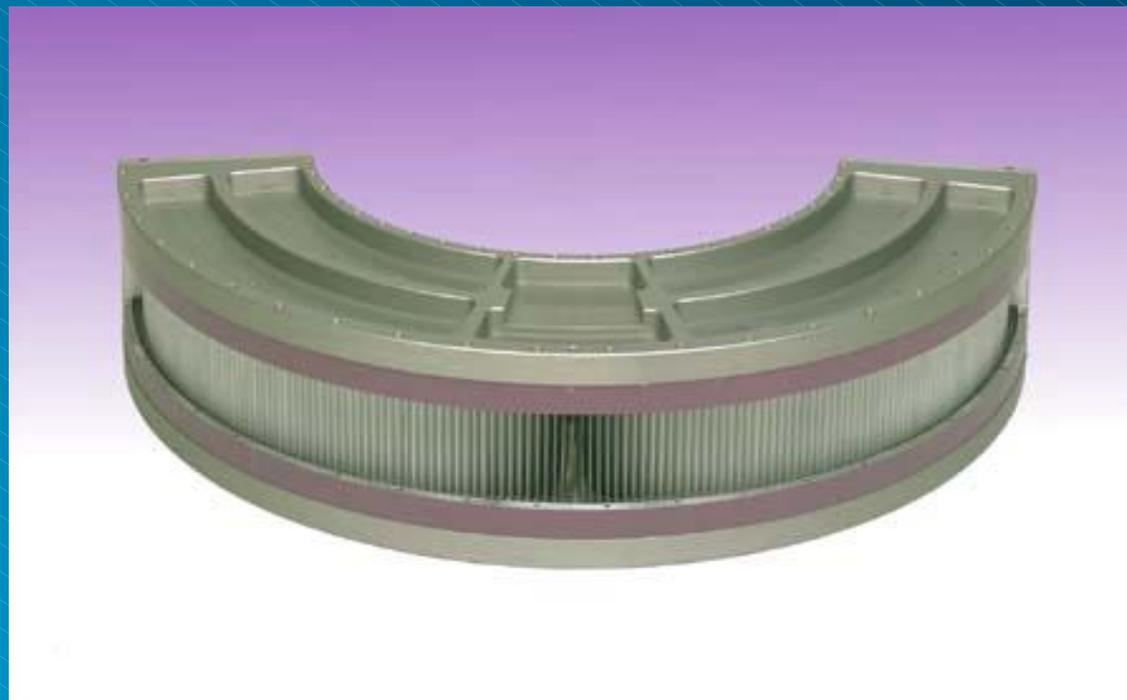


DRACULA - An ILL Project for A Small Sample, High Pressure Diffractometer



What do we want to do ?

- Order of magnitude smaller samples than D20
- Low background (pressure cell)
- Large, compact 2D area detector (D19 model)
- Radial collimator





DRACULA

A Small Sample, High Pressure Diffractometer



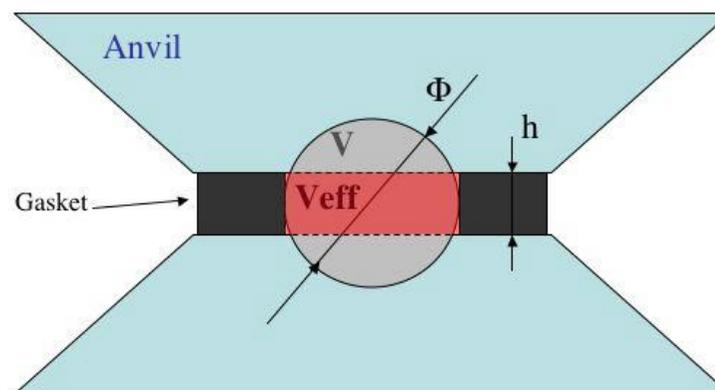
Very High Pressure with Small Samples at Low Temperature

LLB Kurtchatov sapphire/diamond cell 10+ GPa
50mm diameter cell, Useful sample $\ll 1\text{mm}^3$

ILL Compact Paris-Edinburgh cell 10 GPa
180mm diameter cell. Useful sample $\sim 4\text{mm}^3$



Powder sample



V = Total sample volume

V_{eff} = Effective sample volume



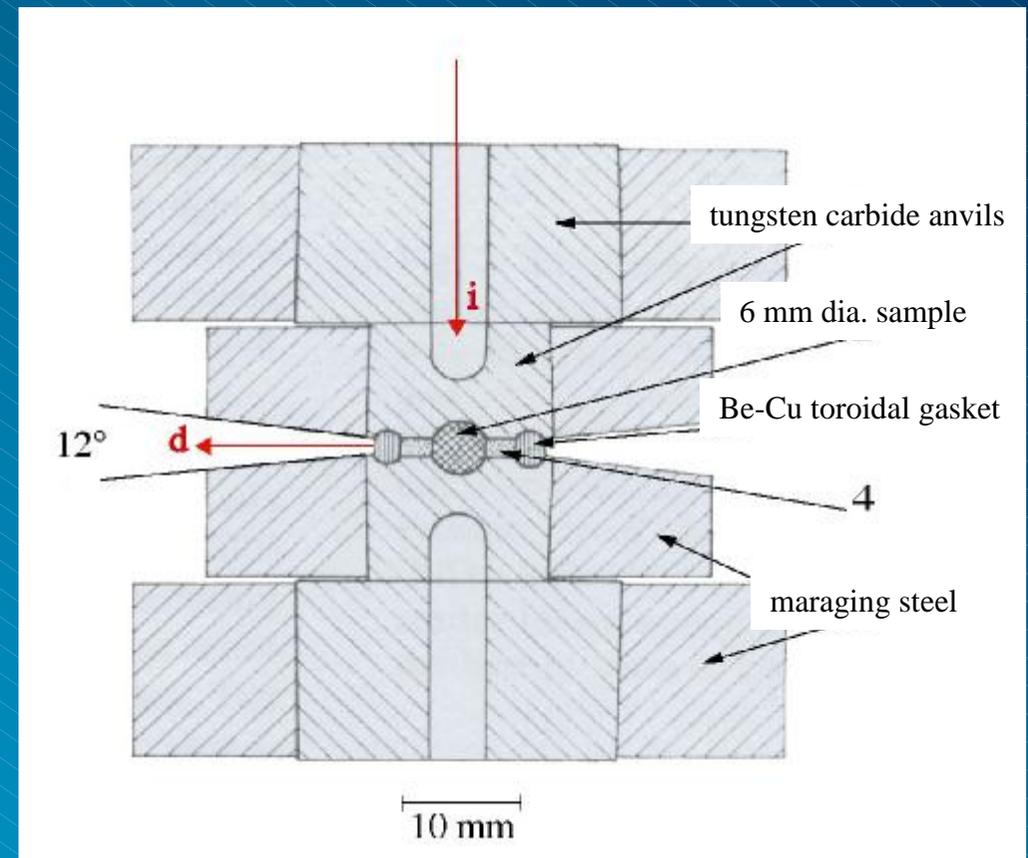
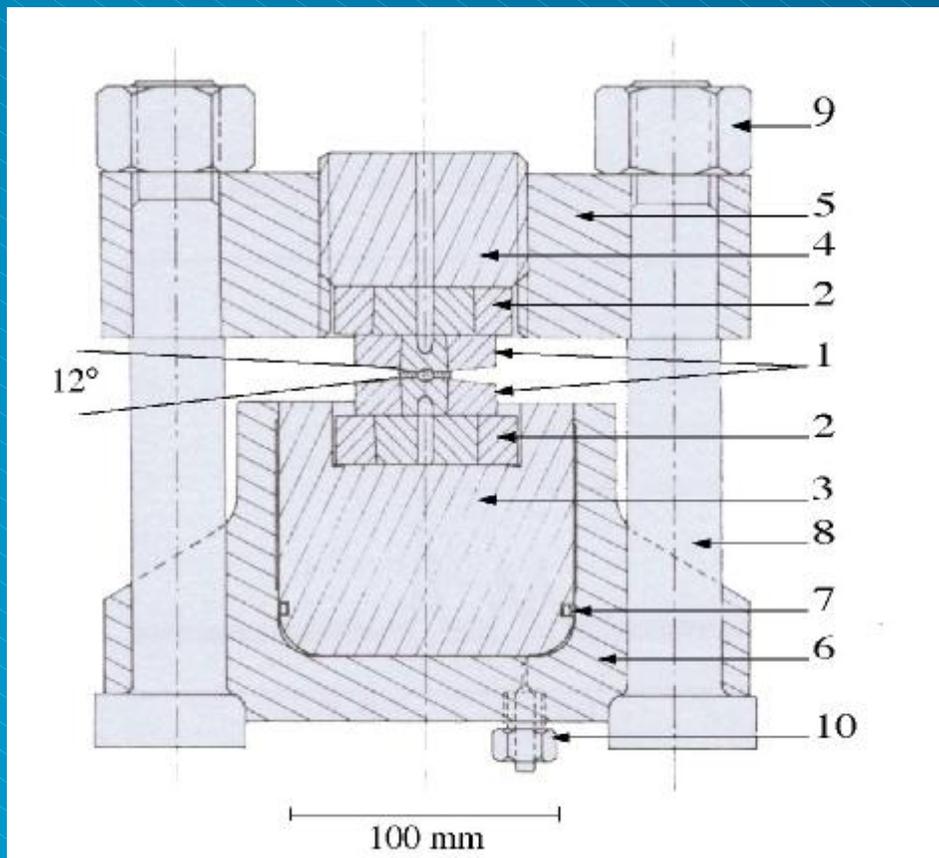
DRACULA

A Small Sample, High Pressure Diffractometer



Very High Pressure with Small Samples at Low Temperature

ILL Compact Paris-Edinburgh cell to 10 GPa. 180mm diameter cell. Useful sample $\sim 4\text{mm}^3$





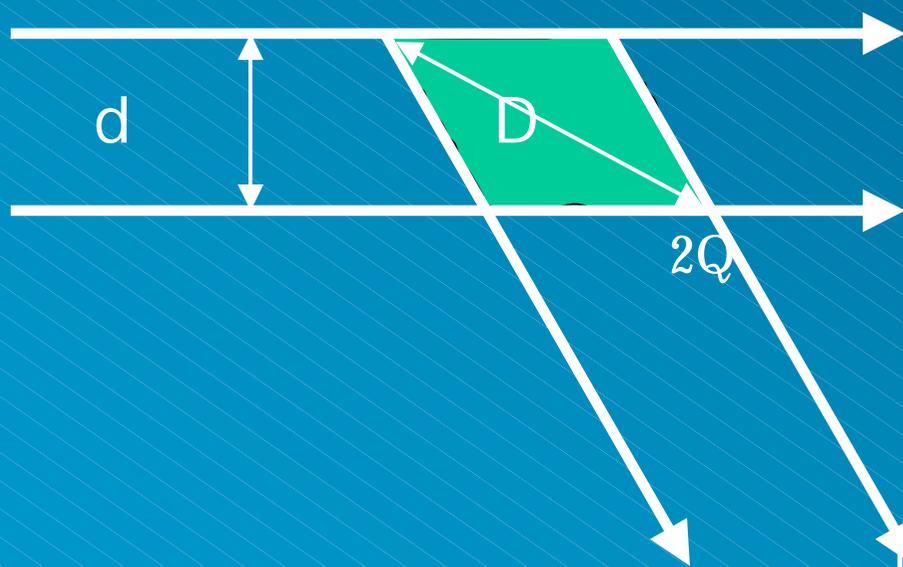
DRACULA

A Small Sample, High Pressure Diffractometer



Large detector on a reactor near 90 degree scattering

$\pm 15^\circ$ vertical as for the new D19 detector cf $\pm 6^\circ$ for new Paris-Edinburgh cell
 $\pm 30^\circ$ horizontal ie $2Q = 60^\circ - 120^\circ$ (maximum range of scattering angles)



d = diameter of the incident beam

D = diameter of scattering volume

$$= d / \sin Q$$

$$= d \sqrt{2} \text{ minimum at } 2Q = 90^\circ$$

$$= 2d \text{ maximum at } 2Q = 60^\circ \text{ \& } 120^\circ$$

$$D = 5\text{mm} - 7\text{mm for } 2Q = 60^\circ - 120^\circ$$



DRACULA

A Small Sample, High Pressure Diffractometer



Can we obtain all d-spacings with a 2θ range of 60° - 120° ?

Use a large focusing Ge monochromator near 90° take-off to obtain several λ

[115] \rightarrow 1.54Å; $d = 0.889\text{Å} - 1.54\text{Å}$

[113] \rightarrow 2.44Å; (graphite filter) $d = 1.39\text{Å} - 2.44\text{Å}$

[111] \rightarrow 4.61Å; (beryllium filter) $d = 2.66\text{Å} - 4.61\text{Å}$

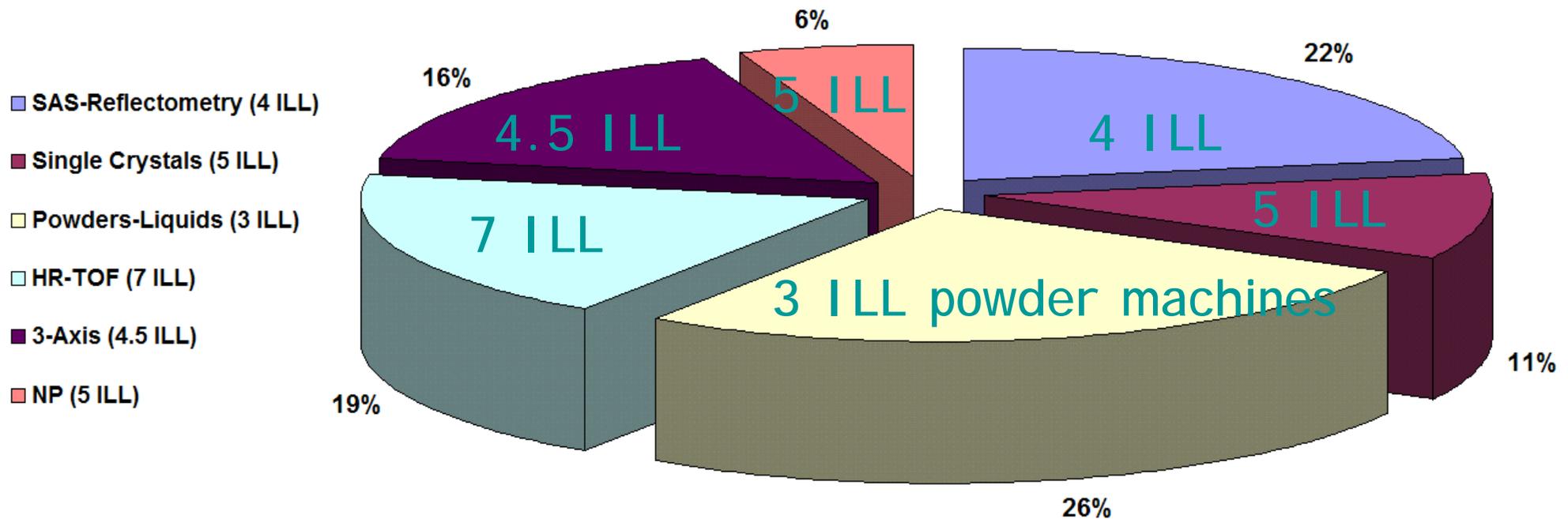


DRACULA

A Small Sample, High Pressure Diffractometer



At present only 3 ILL machines for more than ¼ of all proposals





DRACULA

A Small Sample, High Pressure Diffractometer



Can we compete with the Americans while waiting for ESS ?

Yes, but we must...

- I Use our natural advantage – time average flux on sample
- I Use big detectors, as on pulsed neutron sources
- I Do not assume that the SNS will be a long time coming
- I Do not wait until the SNS is operational before reacting