



# Neutron Diffraction - Neutron Imaging Instruments and Application & An overview of the Italian Users Community

Francesco Grazzi

Consiglio Nazionale delle Ricerche Istituto dei Sistemi Complessi









$$I = I_0 e^{-\Sigma d}$$

Lambert-Beer law

$$T = \frac{I}{I_0}$$

Trasmittance

$$-\ln(T) = \Sigma d$$

& 
$$\Sigma = on$$

Contrast: material (σ) and thickness(d)







## Andor DV 436 16-bit CCD Detector

2048×2048 pixels

nominal pixel size of 13.5 µm (CCD chip size: 2.76 cm x 2.76 cm)

Lens optic system
(1:1 optic)

Sample

Pin hole

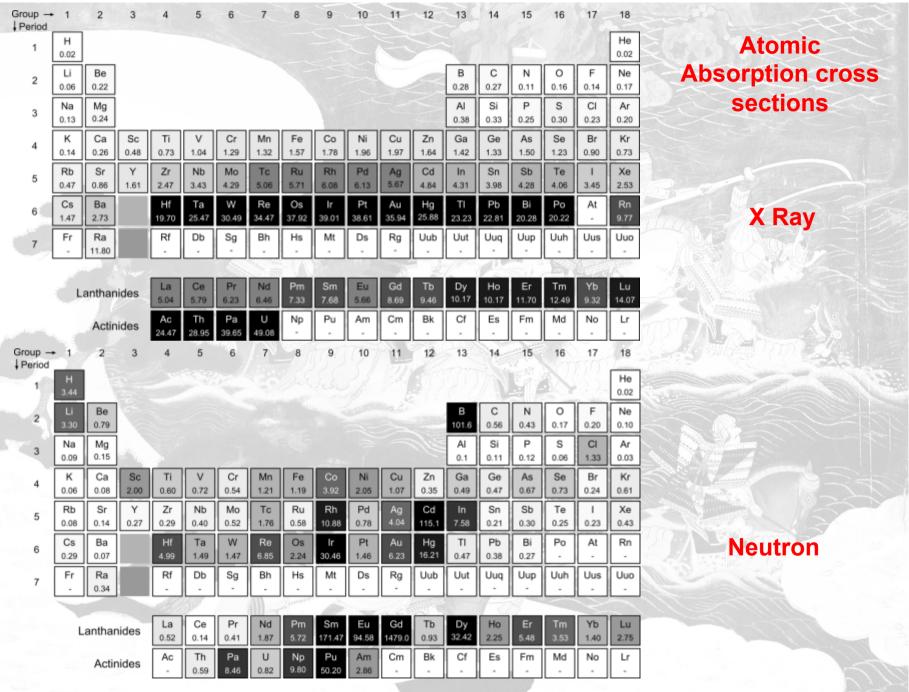
diameters: 4 cm L/D achieved: 178 Rotating table

step angle of 0.96 ° (over 360°)

exposure time for projection: range from 5 s to 60 s

<sup>6</sup>Li based Scintillator

Tickness: 100 µm

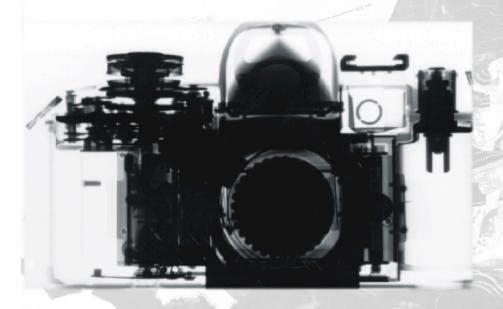


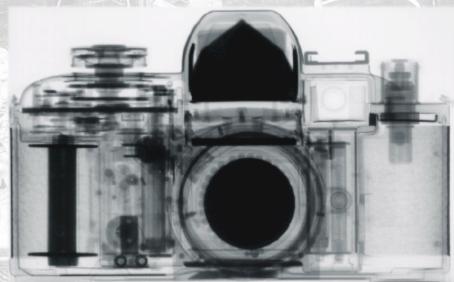
Courtesy E. Lehmann – PSI(CH)



## Complementary x-ray / neutron imaging results







X-Ray

Neutron

Neutrons are optimal for:
-light elements,
-metals

Courtesy PSI(CH)



# Neutron imaging beamlines in Europe (with open access user program)



NEUTRA thermal neutrons Paul Scherrer Institut (CH)

ICON cold neutrons Paul Scherrer Institut (CH)

CONRAD cold neutrons Helmholtz Zentrum (De)

ANTARES thermal neutrons TUM-FRM2 (De)

NECTAR epithermal neutrons TUM-FRM2 (De)

#### Neutron imaging future beamlines in Europe

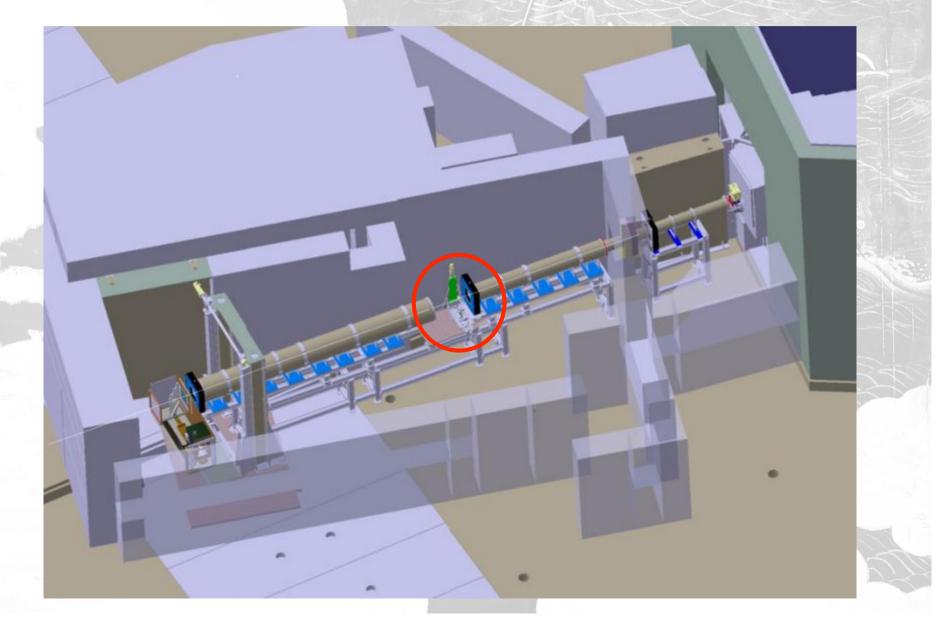
IMAT (under constr.) thermal neutrons ISIS (UK)

ODIN (approved project) cold neutrons European Spallation Source (Sw)



# An example: ICON beamline at SINQ - PSI







## Beamline components



- Moderator
- Flight tube under vacuum
- Pin hole: resolution is proportional to L/D parameter
   (L: distance diaphragm-sample, D: diaphragm aperture)
- Rotating stage (for tomographies)
- Scintillator (GdO, LiF+ZnS)
- Optics (mirror, lens)
- CCD camera
- Image processing software



### A few numbers



- **Resolution** is a function of L/D, scintillator type and thickness, CCD camera pixel size.

Typical values range from 20 to 300 μm

- <u>Brigthness</u> and <u>contrast</u> depends on moderator type, neutron flux, scintillator type and thickness, CCD camera dynamic range

Typical measuring time for a radiography ranges from 2 to 120 s

- Field of view ranges from 20x20 mm<sup>2</sup> to 350x350 mm<sup>2</sup>
- Neutron flux ranges from 1\*105 to 5\*107 n/cm2/s
- World <u>user community</u> comprehends about 2000 scientists in the last 5 years
- Proposed vs accepted experiments ratio in Europe is more than 3:1



## **Applications**



#### **Non-destructive Testing**

Traditional applications: detection of cracks, lunkers, explosives etc.

Nuclear technology: fuel inspection

#### **Scientific Applications**

Environmental research: soil physics, wood science, plant physiology ...

Electro-chemistry: fuel cell research, battery technology ...

Combustion engineering: fuel injection, exhaust gas treatment ...

Geoscience

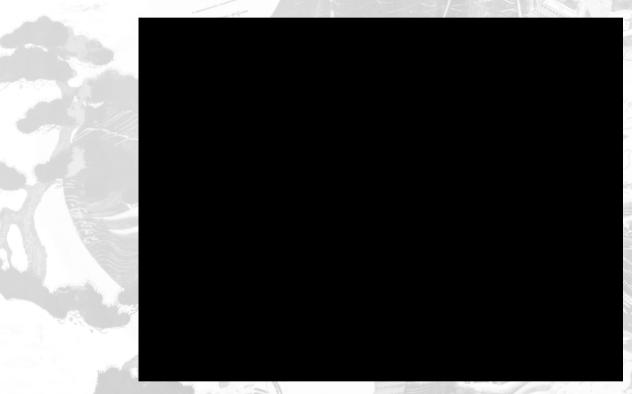
Archaeology and Cultural Heritage

Construction engineering

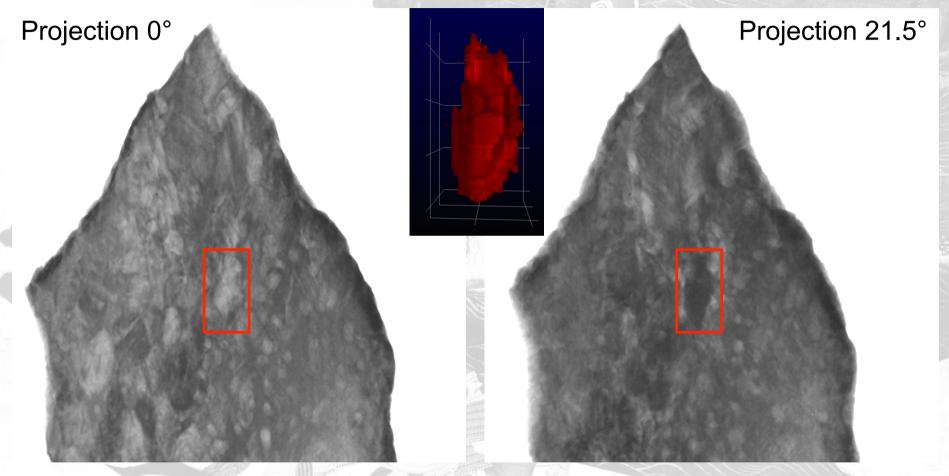


# Time resolved coffee making





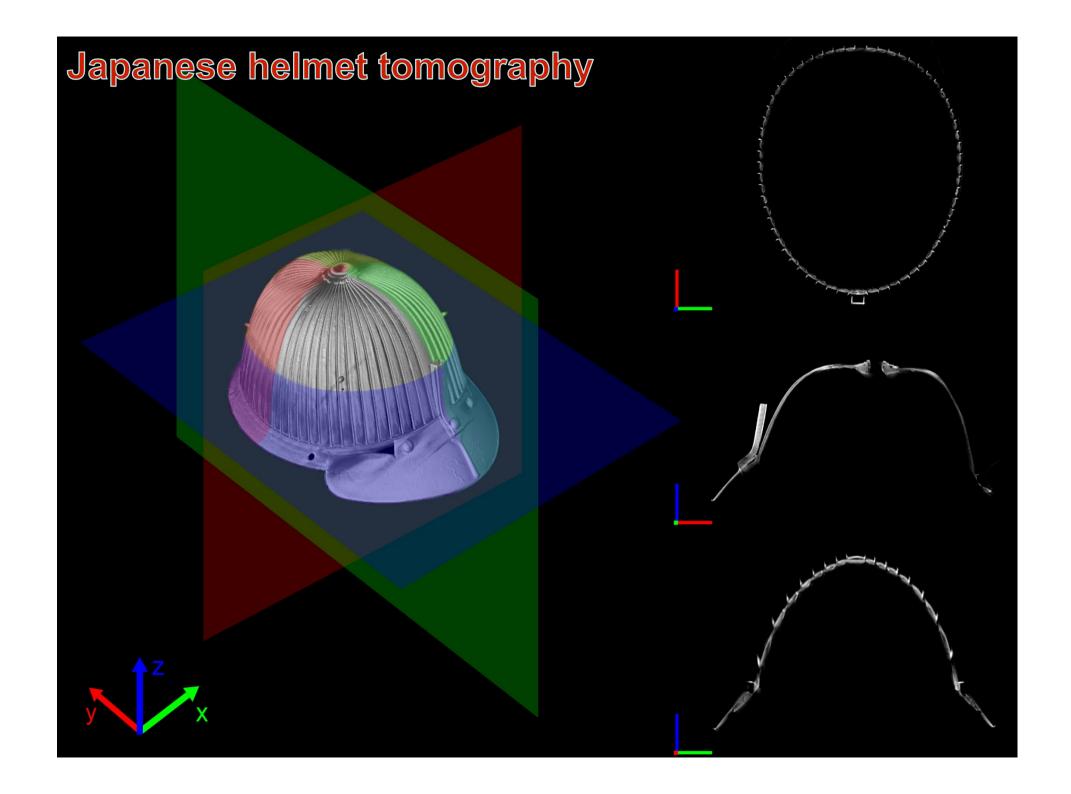
# Tomographic reconstruction of single crystal grains in meteorites using monochormatic neutron tomography





Kaczmarz method (S. Peetermans - PSI)







# Neutron Powder Diffraction: principles



Bragg law: 
$$d = \frac{\lambda}{2 \sin i\theta}$$

Constant Wavelength (CW) diffraction  $\lambda = const, d = f(\sin \theta)$ 

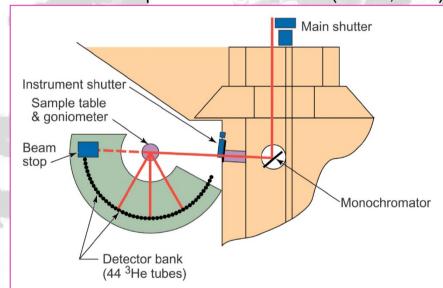
Time of Flight (ToF) diffraction  $\sin \vartheta = const$ ,  $\lambda = f(t)$ , d = g(t)



# Instrumental set-up (Constant Wavelength)



#### HB-2A neutron powder diffractometer (ORNL, USA)



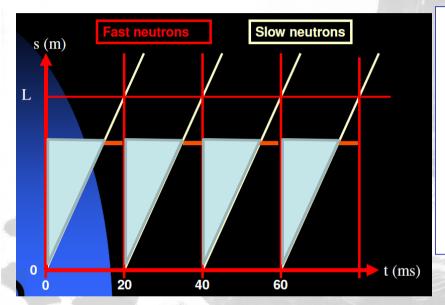
#### 2 axis instrument

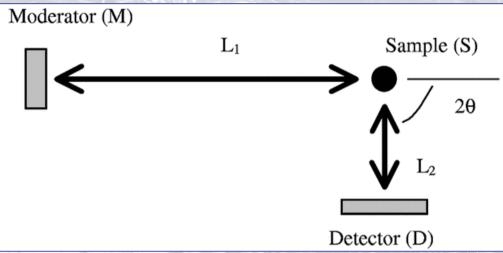
Constant
$$d = \frac{\lambda}{2\sin\vartheta}$$
Variable



# Instrumental set-up (Time of Flight)

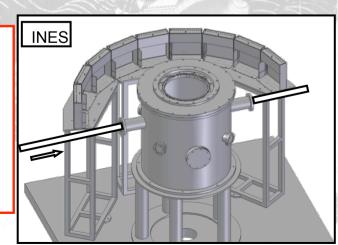






INES (ISIS, UK)

ToF Equation: 
$$d(\mathring{A}) = \frac{h}{2m_n} \frac{t(\mu s)}{L(m)\sin\Theta} = \frac{1}{505,56} \frac{t(\mu s)}{L(m)\sin\Theta}$$
 Flight Path Length Scattering Angle





# **Neutron Diffraction beamlines** in the main European Facilities



(with open access user program)

ILL (Fr) 13 diffraction beamlines (CW)

ISIS (UK) **11** diffraction beamlines (**ToF**) + **1** in construction (**ToF**)

SINQ-PSI (CH) 5 diffraction beamlines (4 CW, 1 ToF)

FRM2 (De) 6 diffraction beamlines (CW) + 2 in construction (ToF)

HZB (De) **9** diffraction beamlines (**CW**)

LLB (Fr) 9 diffraction beamlines (CW)

KFKI (Hu) 3 diffraction beamlines (2 CW, 1 ToF)

LVR15 (Cz) 4 diffraction beamlines (CW)



# Beamline components



- Moderator (all)
- Flight tubes or guides (all)
- Monochromators (CW)
- Chopper (ToF)
- Sample environment (all)
- Detectors (all)
- Data acquisition electronic (all)



### A few numbers



- **Resolution** is a function of the geometric parameters and of incident wavelength(s).

Typical  $\Delta d/d$  resolution ranges from 0.02 (low res) to 0.0005 (high res)

- <u>Brigthness</u> depends on moderator type, source neutron flux, presence of neutron guide, detector type, area and efficiency

Typical measuring time for a diffraction measurement ranges from 3 minutes to ≈10 hours

- Neutron flux ranges from 1\*104 to 5\*108 n/cm²/s
- World user community comprehends about 3000 scientists in the last 5 years
- Proposed vs accepted experiments ratio in Europe is more than 2:1



## **Applications**



- determination of the atomic and/or magnetic structure of a material
- determination of the structure of crystalline solids
- indexing of new crystalline phases
- determination of the static structure factor of gases, liquids or amorphous solids
- mapping of 2<sup>nd</sup> type residual strain in crystalline solids



# The Italian Neutron User Community (a quick overview)



- More than 500 italian users in the neutron facilities in the last 10 years
- Italian neutron users: tot pub 3684, 167 pub/y since 1990 (average last 5 years: 219 pub/y)
- There are about 340 frequent users (at least 5 papers in 10 years, 2002-2011)
- Società Italiana di Spettroscopia Neutronica (SISN) is the reference society for the largest part of the users community (about 250 associates in the last 10 years)
- -Scientific cooperation agreements with ILL and ISIS facilities (managed by CNR) to provide access for Italian users
- Well established (since  $^\prime 80s$ ) Italian expertise in beamline construction at ISIS and ILL
- -Three beamlines (BRISP, IN13 and INES) managed by CNR at ILL and ISIS



# The Italian Neutron User Community



