PSD neutron diffractometer (PSD) at the Budapest Neutron Centre (BNC)

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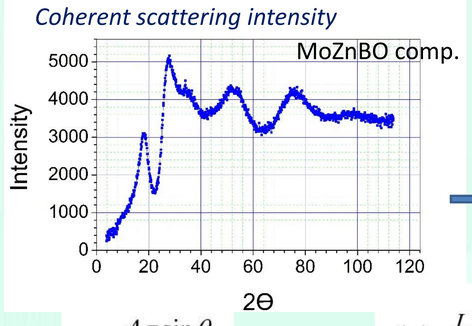
Principle of neutron powder diffraction

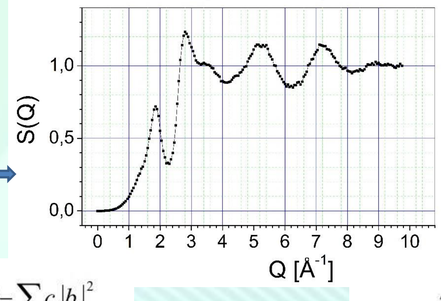
Neutron powder diffraction is particularly useful for materials with light elements in the presence of heavy ones (oxides, bores, carbides, etc.) and for magnetic materials. We present here the PSD neutron diffractometer with a position sensitive detector system mainly used for structural studies of amorphous materials.

The neutron diffraction technique can be used to measure the structure of atom clusters and molecules in disordered materials. Glasses lack long-range order, may have medium range ordering over several atomic lengths. The measured diffraction pattern is related to the positions of the atoms relative to each other in the glass. In case of amorphous material, we don't have well defined atomic positions, we refer to a distribution of atoms as a function of distance, i.e. to the probability of finding an atom at a distance *r* provided there is another atom at the origin.

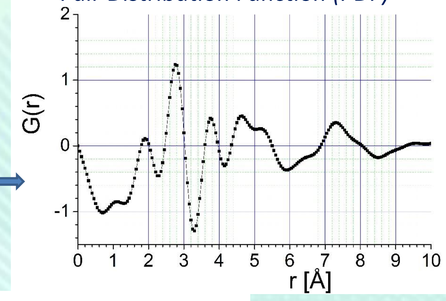
Coherent scattering intensity



Total scattering structure factor



Pair Distribution Function (PDF)



$$Q = \frac{4\pi \sin\theta}{\lambda}$$

$$S(Q) = \frac{I_{coh}(Q) - \sum c_i |b_i|^2}{\left|\sum c_i b_i\right|^2} + 1$$

$$G(r) = \frac{2}{\pi}\int_{Q_{min}}^{Q_{max}} Q[S(Q)-1]\sin(Qr)dQ$$

Source of neutrons

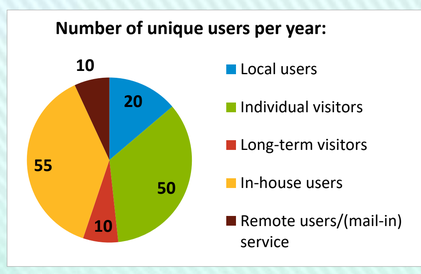
Budapest Research Reactor

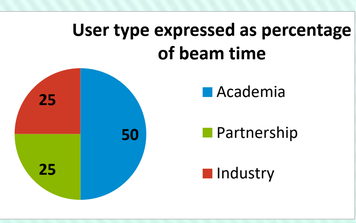
10 MW tank type reactor, moderated and cooled by light water

Thermal neutron flux: $2.5*10^{14}$ n/cm^{2}s

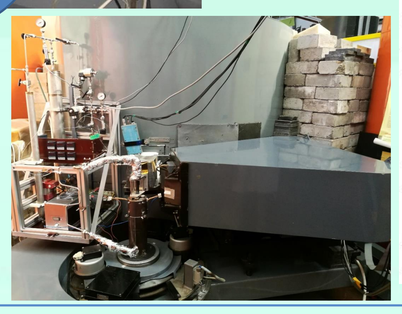
Fast neutron flux: $1*10^{14}$ n/cm^{2}s

Since 2013, the reactor has been fuelled with low enrichment (under 20%) fuel assemblies.





Instrumentation



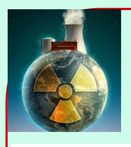
The PSD instrument is also called 'two-axis' neutron spectrometer, the two principle axes being the monochromator and the sample axis. Is placed on a tangential thermal neutron beam, the primary collimation is done before the monochromator.

+ New in-situ cell, to solve structure by direct method, in real time
Temperature and pressure interval:
TiZr cell: T_{max}: 400°C; p_{max}: 300 bar
(Ti 52.5% – Zr 47.5%, null matrix alloy)

+ Sample: 2-4 g powder, Vanadium can, Ø: 6 & 8mm

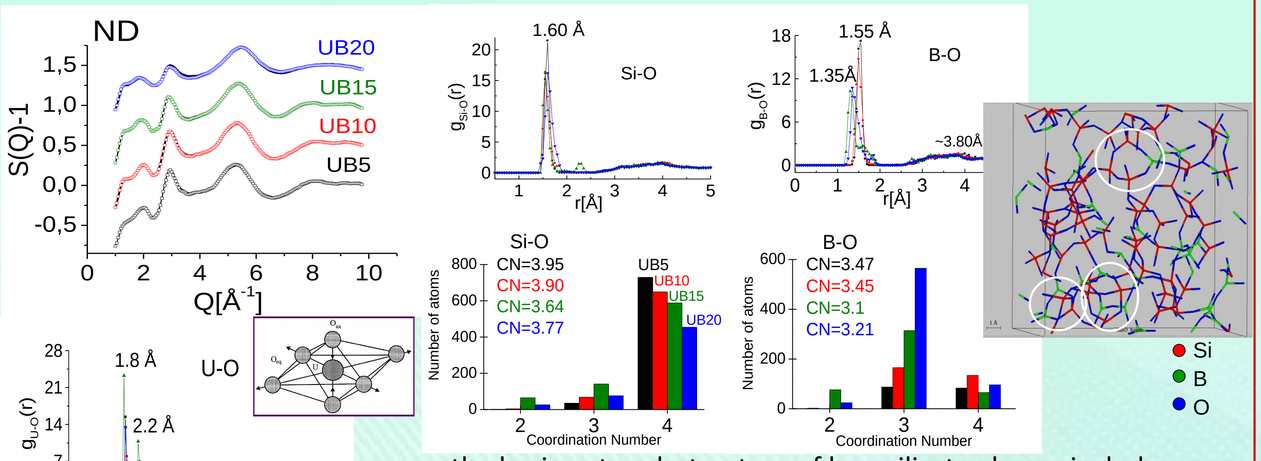
Channel	Thermal, 9T tangential
Primary collimator	Soller-type: 20'
Take-off monochromator angle facility	$-5° < 2\Theta_M < 45°$
Monochromator/Mosaicity	Cu(111) / 16'
Monochromatic wavelength	1.069 Å
Resolution, Δd/d	$1.2 \cdot 10^{-2}$
Flux at the sample position	10^6 n·cm^{-2}·s^{-1}
Beam size at the specimen	10 mm x 50 mm
Scattering angle, 2Θ	$5° < 2\Theta < 110°$
Momentum transfer interval, Q	0.6 – 9.2 Å^{-1}
Monitor counter	fission chamber
Detector system	- 3 linear position sensitive ^{3}He detectors - the detector assembly spans 25° scattering angle at a given position
Data collection	Xilinx preprogrammed unit
Data transfer and control	PC-AT with Eagle I/O card and a dedicated electronic device
Remote control and file transfer	Windows programme package

Scientific projects (examples)



Structural characteristics of borosilicate glasses doped with uranium ions

Studied compositions: 70wt%[SiO_2(65-x)B_2O_3(x)Na_2O(25%)BaO(5%)ZrO_2(5%)]+30wt%UO_3, x=5-10-15-20% like: UB5, UB10, UB15, UB20



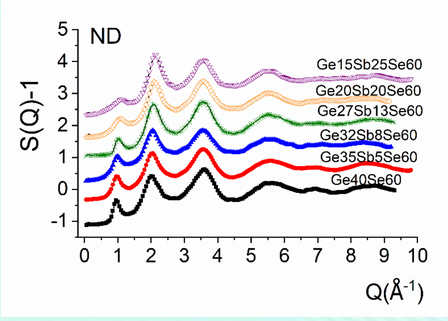
- the basic network structure of borosilicate glasses includes $^{[3]}$B-O-$^{[4]}$Si and $^{[4]}$B-O-$^{[4]}$Si linkages, the ratio of BO_3/BO_4 increases with B_2O_3 concentration

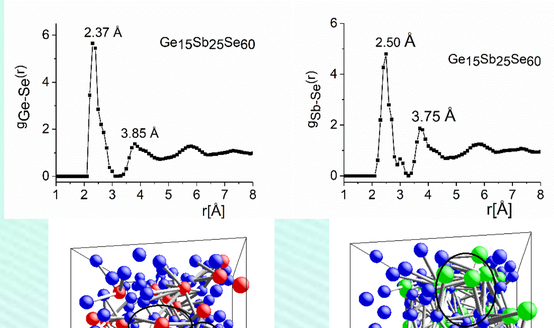
- for the U-O correlations two distinct peaks were resolved at 1.8 Å and 2.2 Å
- significant second neighbour atomic pair correlations have been revealed between uranium and the network former (Si,B) and modifier (Na) atoms
- we may conclude that for the doped glasses we have a stable basic network structure and uranium accommodates in both silicate and borate site.

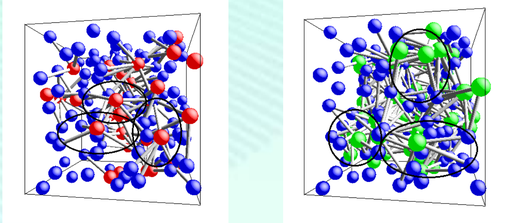
Fabian, M. et al. Structure study of new uranium loaded borosilicate glasses, J. Non-Cryst. Solids 2013, 380, 71-77

Chalcogenide glasses

Atomic-scale structure characterization of Ge-Sb-Se glasses: Neutron diffraction experiments and RMC modelling





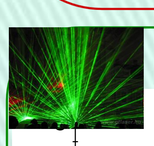


- all specimens are fully amorphous in nature as confirmed by the ND patterns.

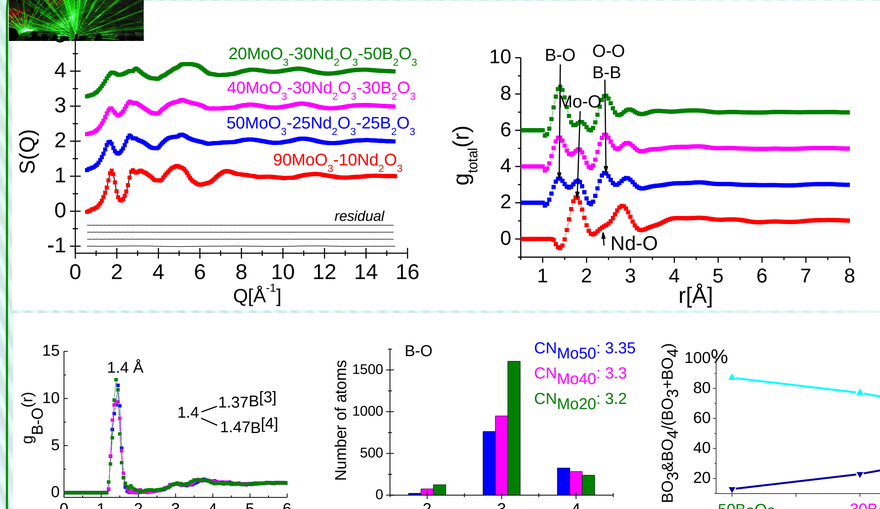
Ge-Se and Sb-Se pair distribution functions and their 3D bonds in $Ge_{15}Sb_{25}Se_{60}$ cell units. The colour represents the type of atoms: Ge-red, Se-blue, Sb-green coloured.

- addition of Sb atom to the binary Ge-Se glass does not change significantly the basic Ge-tetrahedral structural units but leads to their distortion.
- with increasing of Sb concentration, well-defined Sb-pyramidal units are formed in the ternary glass structure.
- the glassy network builds up from $GeSe_{4/2}$ tetrahedra and the $SbSe_{3/2}$ pyramid units, connected through 2-coordinated Se atoms.

Fabian, M. et al. Investigation of the Atomic Structure of Ge-Sb-Se Chalcogenide Glasses, Adv. Cond. Matter Physics 2018, 7158079



Network structure of boromolybdate glasses

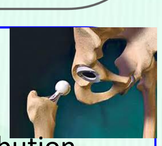


-in binary system the basic network units are MoO_4, MoO_5 and MoO_6, how in ternary glasses the main network unit is the tetrahedral MoO_4

- both trigonal BO_3 and tetrahedral BO_4 units are present

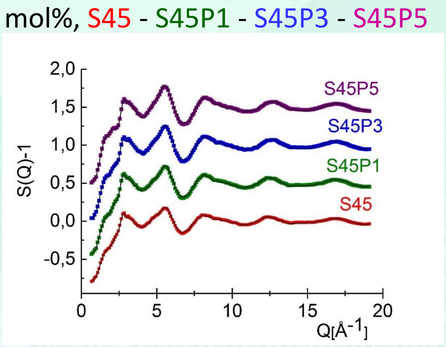
- mixed MoO_4-BO_4 & MoO_4-BO_3 linkages form intermediate range order as it was identified from pronounced second neighbour distributions and distances

Fabian, M. et al. Network structure of molybdate glasses by neutron and X-ray diffraction and reverse Monte Carlo modelling, J. Phys. Conf. Series 2016, 012068

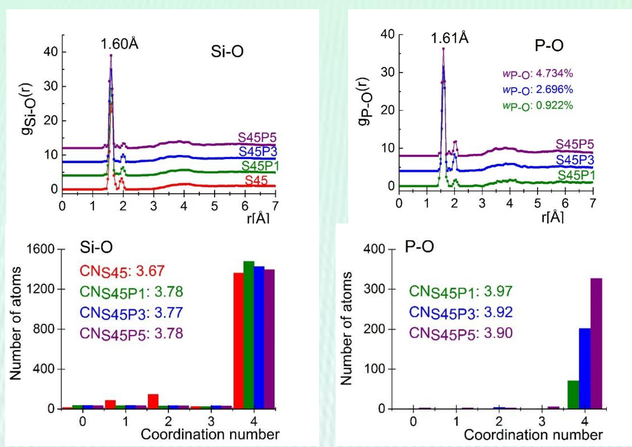


Bioactive glasses: structure characteristics and bone regeneration application

Neutron diffraction structure factors for $45SiO_2$-CaO-(30-x)Na_2O-xP_2O_5 x= 0,1,3,5 mol%, S45 - S45P1 - S45P3 - S45P5



Si-O & **P-O**: partial pair correlation functions and coordination number distribution



- we performed ND measurements and RMC simulation to determined structural parameters

- the basic network structure of glasses includes tetrahedral $^{[4]}$Si and $^{[4]}$P units
- the basic network structure of the host glasses is similar than doped with P

Fabian, M. et al. Network structure and thermal properties of bioactive (SiO_2-CaO-Na_2O-P_2O_5) glasses, J. Mat. Science 2019, submitted