Lectures - Tuesday, June 23

L3

Coherent diffraction
KOHERENTNÍ DIFRAKCE

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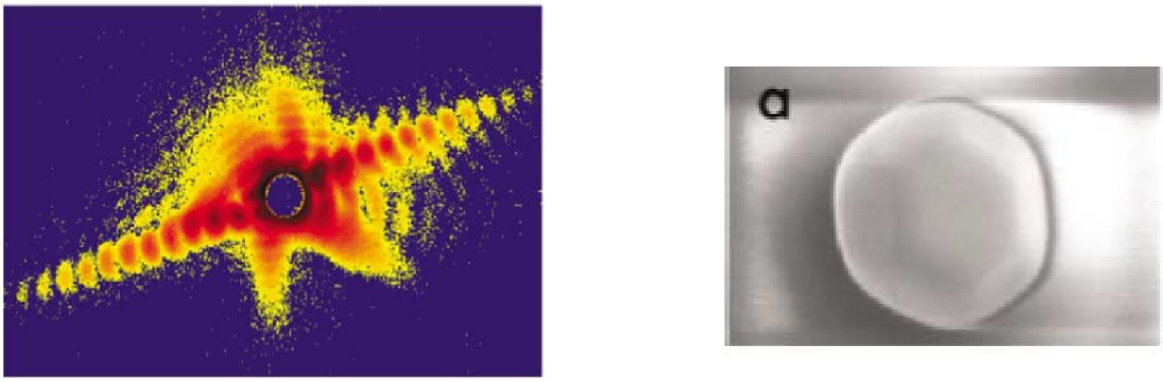
S fázovým problémem strukturní analýzy se setkal každý “rentgenář” během základních kurzů strukturní analýzy. Měřením intenzity, a nikoliv amplitudy, difraktovaného záření ztrácíme informaci o fázi rozptýlené vlny. Tím jsou poněkud komplikovány úlohy řešení struktury z difrakčního záznamu.

Moderní zdroje synchrotronového záření poskytují záření s vysokou úrovní koherence, tj. fáze dopadajících fotonů je shodná. Vysoký stupeň koherence je příčinou pozorování ohybových jevů jako důsledek interference rozptýlených vln, viz. Obr.1. V angličtině se pro tento jev ujal termín “diffraction speckles”. Právě použití koherentního záření pro difrakční experimenty usnadňuje řešení fázového problému, tj. nalezení fáze difraktující vlny.

Fázi rozptýlené vlny lze z difrakčního záznamu určit pomocí takzvaných *phase-retrieval* algoritmů [2, 3]. Tyto algoritmy porovnávají Fourierův obraz modelu rozložení elektronové hustoty s naměřenými absolutními velikostmi strukturních faktorů. Aplikací různých podmínek v přímém i reciprokém prostoru (constrains) lze urychlit konvergenci výpočtu.

V příspěvku budou prezentovány, kromě popisu experimentálního zařízení, výsledky simulací *phase-retrieval* algoritmů a experimetů na zdrojích SR.

1. Robinson, I.K., Vartanyants I.A., Williams G.J., Peiffer, M.A., Pitney, J.A., Phys.Rev.Letters **87**, 195505 (2001).
2. Gerchberg, R.W., Saxton, W. O., Optik (Stuttgart) **35**, 237–246 (1972).
3. Fienup, J. R., Appl. Opt. **21**, 2758–2369 (1982).



Obrázek 1. Rozptyl koherentního rtg. záření na zlaté nanočástici. Jsou patrné interfereční kroužky (“speckles”) . Napravo zrekonstruovaný tvar rozptylující částice Au. Převzato z [1].

L4

TUNABLE LABORATORY X-RAY SOURCES

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The choice of wavelength of X-ray radiation is critical for many diffraction and non-diffraction experiments. The traditional laboratory X-ray sources are in large based on generation of radiation characteristic for the element of the source anode, i.e. monochromatic or close-to-monochromatic radiation. Therefore experiments relying on changing properties of samples with varied energy of the primary X-ray beam have so far been performed mostly at sources of synchrotron radiation. Synchrotron radiation of much higher flux densities, lower divergence and tunable energy produced by bending magnets or insertion devices fully satisfies requirements of many types of experiments.

Because of intrinsically weak diffraction of crystals of biological molecules higher intensity sources of radiation have always been sought. Sources of synchrotron radiation of the 3rd generation brought a substantial improvement of the methods and experiments of bio-crystallography, namely an easier implementation of multiple wavelength anomalous dispersion experiments used widely for solving the phase problem. Intensive and tunable laboratory sources of X-ray radiation would offer a long-wanted tool not only for bio-crystallographic applications.

In the last ten years several research groups have aimed at development of laboratory-size tunable X-ray sources. One direction of these efforts utilizes the phenomenon of inverse Compton scattering. Interaction of electrons accelerated to energies of tens of MeV with photons of electromagnetic radiation of micrometer wavelength leads to generation of X-ray photons. The Compact Light Source (CLS) of Lyncean Technologies, inc., Palo Alto, California has been developed with the aim of application of this phenomenon in generation of an X-ray beam suitable for various types of experiments, which could be housed within a larger laboratory. Some recent results indicate that CLS can be used both for X-ray imaging and diffraction experiments [1].

Another application of the inverse Compton scattering mechanism is applied in the newly designed type of an X-ray source - Compact X-ray Source (CXS) designed at the Massachusetts Institute of Technology/Nuclear Reactor Laboratory. In this case technology of superconductors should be applied to achieve electron acceleration with a finely controlled time structure, high repetition rate and leading to highly brilliant x-ray beam.

Another direction of these developments relies mainly on generation of Brehmsstrahlung by high-energy electrons. Large currents of electrons with energy of 25 MeV interacting with a solid target such as carbon wire generate a wide spectrum of X-ray and gamma radiation. The Photon Production Laboratory, Ltd., Shiga, Japan has designed a single-magnet “synchrotron” setup with selection of a given wavelength of radiation by a system of monochromators - “Mirrorcle”. X-ray radiation is generated by two different mechanisms - Brehmsstrahlung from a micrometer target or so called “parametric” radiation produced by interaction of the intensive and energetic electron beam with a single crystal periodic lattice. The Mirrorcle design is being optimised to become an X-ray source usable for diffraction experiments [2].

While the CXS source is only in the stage of planning CLS and MIRRORCLE RAY 20 ST systems have been built and partially optimised. Both produce x-ray radiation of variable energies. Developments to provide high intensity monochromatic beams for a broad range of experiments are under way.

1. M. Bech, O. Bunk, C. David, R. Ruth, J. Rifkin, R. Loewen, R. Feidenhans’l, F. Pfeiffer, *J. Synchrotron Rad.,* **16**, (2009), 43.
2. H. Yamada, T. Hiraia, M. Morita, D. Hasegawa, M. Hanashima, In *Developments in x-ray tomography VI,Proceedings of the Society of photo-optical instrumentation engineers (SPIE),* **7078,** (2008)**,** P780.

CL3

NEW POSSIBILITIES FOR X-RAY DIFFRACTOMETRY: BRIGHTEN UP YOUR HOME-LAB

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The latest developments in new X-ray microfocus sources lead to new possibilities in X-ray diffractometry. Using the new Incoatec Microfocus Source (IµS^{TM}) with the latest kind of two dimensional focussing Montel optics, the so called Quazar^{TM} optics, in combination with a two dimensional detector allows high quality diffractometry measurements with very good resolution of very small and weakly scattering samples in the home-lab in short time.

The advantages of the new 30 W air cooled IµS^{TM} with a focal spot size below 50 µm are presented. The use of IµS^{TM} is as easy as for all sealed tube systems and the performance exceeds typical combinations of traditional rotating anodes with multilayer optics. With a focussing optics IµS^{TM} achieves in a single crystal diffraction set-up a flux above $3\cdot10^8$ cps in a 250 µm spot with Cu-K or a flux above 10^7 cps in an 110 µm spot for Mo-K radiation. For SAXS experiments there is also an IµS^{TM} available with a 2 dimensional collimating optics.

Various examples of new application measurements on different X-ray samples are shown, like proteins, small molecules and SAXS, proving the broad field of applicability of the IµS. The figure shows the X-ray powder diffraction patterns of ibuprofen recorded with a typical parallel beam sealed tube set-up (left) and with IµS^{TM} (right) in transmission setup.

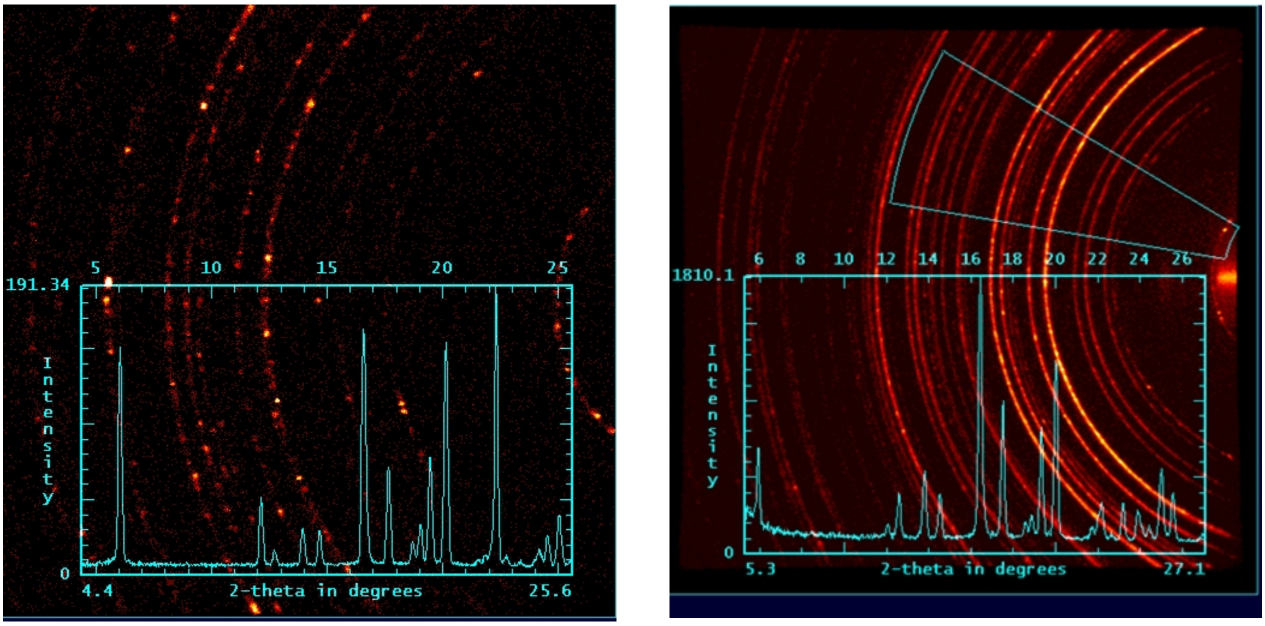

Figure 1: Diffraction pattern of ibuprofen recorded with a parallel beam sealed-tube set-up (left) and an IµS^{TM} (right), both with a Bruker D8 GADDS. The exposure times were 120 sec (left) and 15 sec (right) respectively.