Applications of micro-diffraction to the study of fibres

Christian Riekel

European Synchrotron radiation facility, B.P. 220, F-38043 Grenoble Cedex

Résumé – The talk will discuss recent developments of micro- and nanodiffraction at the European Synchrotron Radiation Facility (ESRF). X-ray beam sizes available for user experiments range currently from 5 microns to about 300 nm. Beam sizes down to about 30 nm can be expected in the future. Examples for instrumentation, sample environments and applications will be given. The scientific examples will be taken mostly from the area of fibrous polysaccharides such as starch and cotton.

1. Introduction

The talk will review progress in instrumentation and scientific applications leading to routine use of synchrotron radiation micro- and nanobeams for SAXS/WAXS experiments.[1] The ESRF-ID13 beamline has been developing these techniques since the onset of operation of the ESRF in 1994 with an emphasis on polymeric and biological samples. At the current stage, beam sizes between 5 microns and 300 nm are routinely available for user experiments. A dedicated end station for nanobeam experiments has been commissioned beginning of 2007 and a beam size of less than 90 nm has been demonstrated for user experiments. It is expected that focal spot sizes of about 30 nm will be attainable. I will provide some examples for the main X-ray optical systems used, which are mirrors, refractive lenses and Fresnel lenses. I will also show progress in the development of sample environments and in particular approaches taken for developing a goniometer for nanobeam scanning diffractometry.

Scientific applications of micro- and nanobeams range from scanning diffraction on heterogeneous materials to single crystal diffraction.[1,2] I will show a number of examples for fibrous biopolymers such as polysaccharides. Thus the local structure of starch granules has been studied by groups from CERMAV and INRA-Nantes.[3] These studies have been recently extended to the high-pressure gelatinisation of single starch granules. In a complimentary study the unit cell of A-type amylose has been refined.[4] The atomic structure of A-amylose is thus accessible to single crystal diffraction techniques.

X-ray micro- and nanobeams allow the development of special sample environments and sample manipulation techniques for in-situ studies such as microfluidics. An example for the use of inkjet systems is the reaction of a single cotton fibre with aequous NaOH microdrops. Microdrop diameters in the range of a few 10th of microns allow inducing a highly local reaction.

2. Références

- [1] Riekel, C., New Avenues in X-ray microbeam experiments. Rep. Prog. Phys., 2000. 63: p. 233-262.
- [2] Riekel, C., M. Burghammer, and G. Schertler, *Protein crystallography microdiffraction*. Current Opinion in Structural Biology, 2005. **15**(5): p. 556-562.
- [3] Buléon, A., et al., Crystalline Ultrastructure of Starch Granules Revealed by Synchrotron Radiation Microdiffraction Mapping. Macromolecules, 1997. **30**(13): p. 3952-3954.
- [4] Popov, D., et al., A-Amylose Single Crystals: Unit Cell Refinement from Synchrotron Radiation Microdiffraction Data. Macromolecules, 2006. **39**(10): p. 3704-3706.