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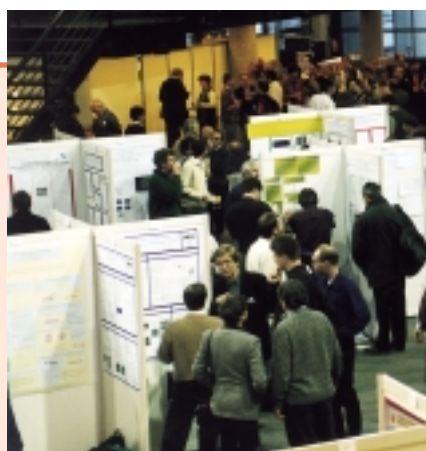
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*Photography by:*  
**G. Admans, C. Argoud  
and Jimagine.**



The tenth ESRF Users' Meeting will take place on Thursday 10 February 2000 at the Atria conference centre. As

## USERS' MEETING

10/11 February 2000

usual the sessions will comprise reports on recent activities, highlight talks, posters and a commercial exhibition. In the evening there will be a dinner on-site at the ESRF.

Three workshops are planned for the one or two following days. Details, which are yet to be finalised, will be posted on the web. There will be a

workshop on macromolecular crystallography and one on self organisation at interfaces and in thin films. The topic of the third workshop will be finalised shortly. Please consult the ESRF web pages ([www.esrf.fr](http://www.esrf.fr)) for updated information.

**M. Cooper**



# WORKSHOP ON X-RAY DAMAGE TO CRYSTALLINE BIOLOGICAL SAMPLES

9/10 June 1999

This Workshop was hosted by P. Lindley and attended by around 30 scientists from Europe and the U.S.A. The purpose of the workshop was to consider beam induced radiation damage and heating in macromolecular crystals undergoing structure determination by x-ray crystallography. The scope of the workshop, its format and the questions to be posed originated from discussions between E. Garman, C. Nave and G. Rosenbaum at conferences over the last 2 years.

With the advent of extremely intense x-ray beams from third generation synchrotron sources, observation of damage to cryo-cooled macromolecular crystals is becoming more common. In order to fully utilize the x-ray beams now available, some understanding of the processes involved in radiation damage and beam heating is required so that, if possible, evasive action to slow the damage rate can be taken.

At present, there is insufficient knowledge or understanding about radical production and diffusion in protein crystals in vitreously frozen solutions. More information is also required concerning radiation damage and beam heating and their relationship to incident dose, dose rate and incident wavelength. The workshop was convened to air all of these issues.

The workshop was informal and loosely structured to encourage maximum interchange of information and ideas. There were two main sessions, the first of which was to gather, collate and exchange information and to separate anecdotal evidence from fact. The second session was aimed at deciding on the most important questions to be answered experimentally, and then to apportion possible experiments to the various participating synchrotrons and laboratories on a complementary rather than competitive basis, since large amounts of beam time and effort are needed to obtain solid answers to the many open questions.

In the first session, two research teams [R. Ravelli (EMBL, Grenoble) and W. Burmeister (ESRF)] reported separate systematic studies on the effects of radiation decay in protein crystals. Talks followed which covered: damage in membranes and lipids [M. Caffrey (Ohio State)], current knowledge on dose rate versus overall dose effects and theoretical consideration of the temperature increase in a crystal [G. Rosenbaum (ANL)], modelling of the temperature rise in protein crystal during irradiation by finite element analysis [J. Nicolson (Daresbury)], factors to be considered when investigating radiation damage, and observations of

damage to blue tongue virus crystals under different conditions [D. Stuart (Oxford)], damage to cryogenic biological samples in x-ray microscopy [D. Weiss (Gottingen)], a general overview of radiation damage and cryo methods for biological samples [C. Jacobsen (Stony Brook)], and a consideration of whether lowering the cryo-temperature to around 40K using helium as the cryogen could be expected to alleviate radiation damage effects [E. Garman (Oxford)]. Lively, yet focused discussion took place during and after these presentations.

In the second session the issue of collecting data at different wavelengths was addressed [A. Gonzales (EMBL Hamburg)] and the information from the first session was summarized [G. Rosenbaum (ANL)]. The experiments necessary to answer the questions require careful design, and are not trivial, since there are many convoluted variables. For instance, the same sized crystals should be used within each experiment for proper comparison. Four experiments were decided upon as the highest priority, the first aim being to determine the limiting dose above which samples decay very rapidly. This limit has been reported from observations at third generation synchrotron sources but additional controlled experiments are required. Participants agreed to attempt them before the next workshop, to be held in approximately a year's time.

P. Lindley brought the Workshop to a conclusion with a plea for more attention to be focused on designing and building faster detectors. Since it is likely that radiation damage rates can only be slowed, but not made insignificant, collecting the data in a shorter time would be the best option. Investment of money and resources are needed for further detector development.

E. Garman



*A crystal of Salmonella typharium neuraminidase which has been irradiated at 100 K for 19 hours in a bright synchrotron beam. It was then allowed to warm up in cryo-buffer. The irradiated part of the crystal has completely disintegrated as a result of the released radiation damage products. The remaining L-shaped piece of crystal was re-cooled to 100 K and it still diffracted to beyond 1.0 Å.*

BIOLOGICAL CRYSTAL CHARACTERIZATION WORKSHOP:  
"IDENTIFYING WHAT A GOOD BIOLOGICAL CRYSTAL IS"

10/11 June 1999

This workshop brought together scientist from several communities, mainly concerned with macromolecular crystallography, crystal growth and crystal characterization (with special emphasis on x-ray diffraction imaging, "topography"). The convening of the workshop corresponds with the fact that, associated with the progress in crystal growth techniques, the mosaic spread of many biological crystals has decreased dramatically over the last few years: such that the measured x-ray diffraction rocking curves from lysozyme crystals are currently only a few arc seconds wide.

The topic of the workshop was introduced by P. Lindley and summarized, substantially, in the first transparency of the introductory talk by R. Fourme, who simply asked "what is a good biological crystal?"

At least two answers emerged from the contributions to the workshop:

1) from the macromolecular crystallography point of view a good crystal is the one which allows us to reach a better resolution, i.e. the smallest interreticular lattice plane distance  $d_{\min}$ , as clearly explained by R. Fourme himself.

2) from the x-ray characterization point of view a good crystal displays a small mosaic spread, and few images of defects and distorted regions in the topographs, as illustrated by the curves and images presented by M.-C. Robert, F. Otalora, A. Aubry and V. Stojanoff.

The correlations between these two approaches were discussed in a lively way. R. Fourme showed, with the help of a simple model, that  $d_{\min}$  should be proportional to  $\sigma$ , the static Debye-Waller factor which characterizes the crystal static disorder (defects, mosaic spread, etc.).

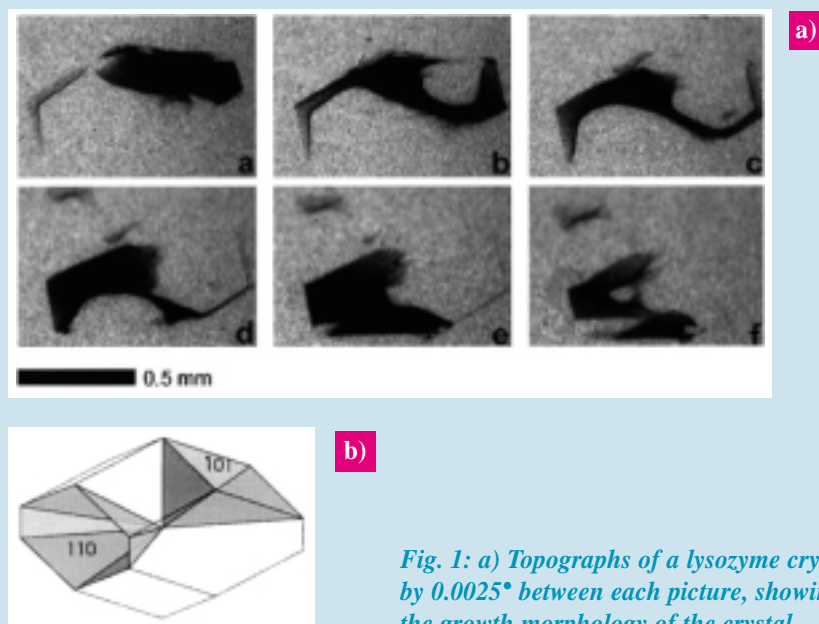
Of course the smaller  $\sigma$  is, the more perfect the corresponding crystal. The proportionality factor between  $d_{\min}$  and  $\sigma$  is a function of the experimental conditions (detector, signal-to-noise ratio, etc.) and is being decreased by the use of third generation synchrotron radiation sources.

The experiments roughly confirm this approach for relatively deformed crystals: reducing the mosaic spread of the samples, from say  $10^{-1}$  to  $10^{-2}$  degrees, entails a big improvement in the resolution. This is not so evident when dealing with the best crystals, which exhibit a mosaic spread smaller

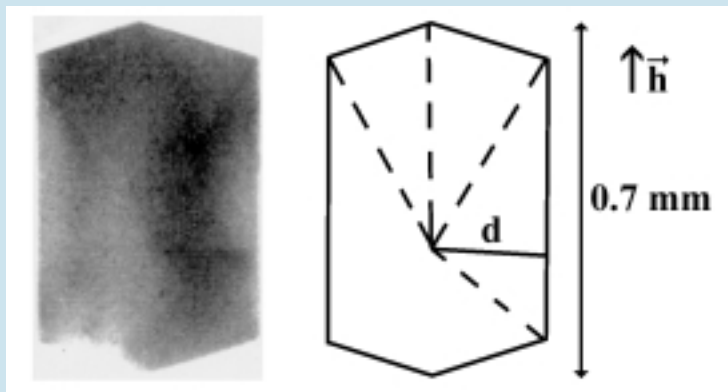
than  $10^{-2}$  degrees. However, in practice, this is not usually the case because of the damage caused by freezing and radiation.

The size and quality of the crystals results primarily, as explained by A. Tardieu, from the protein surfaces adequacy for crystallization, which varies dramatically from one protein to the other. Additionally, size and quality are a function of the growth conditions, and several new approaches allow further enhancement of these properties: growth in a gel or an electric field (A. Aubry) or under microgravity, or by reducing the growth rate. The topographic diffraction images performed on these crystals show features which look very much like the usual defects in inorganic crystals (growth striations, growth sectors and even dislocations). **Figures 1 and 2** show examples of topographs from biological crystals. Unfortunately both the very nature of these features and the mechanisms by which they produce a contrast allowing their observation (which were discussed by B. Tanner when describing the techniques allowing characterization of these crystals) are still unclear.

The participants at the Workshop agreed, after an interesting discussion, that we are still in a preliminary stage of the work in this topic. A summary of the conclusions resulting from the discussion is as follows: **a)** the current knowledge about the defects present in biological crystals is still poor and should be improved **b)** the relationship between the observed defects and the 'resolution' of the structure is not straightforward, and should be considered as a long term project and **c)** these topics are worth continued investigation, through a joint effort of the various teams concerned with and using the x-ray diffraction and imaging techniques described by



*Fig. 1: a) Topographs of a lysozyme crystal recorded at different  $\theta$  values, and rotated by  $0.0025^\circ$  between each picture, showing the different growth sectors. b) Scheme of the growth morphology of the crystal.*



*Fig. 2: Topograph of a chicken egg lysozyme crystal grown in silica showing the growth sectors.  $\vec{h}$  is the diffraction vector.*

B. Tanner. This should include systematic experiments to clearly identify the features observed on the topographs, and *in situ* experiments (growth, freezing, dehydration, radiation damage). P. Siddons (psiddons@bnl.gov) and J. Härtwig (haertwig@esrf.fr) were requested to co-ordinate this effort.

**J. Baruchel**

## WORKSHOP ON PIXEL DETECTORS

17/18 June 1999

A workshop on so-called “pixel detectors” was held on 17 and 18 June at the ESRF, bringing together 17 invitees representing the most advanced teams working on pixel detector development in Europe and the United States. Opened by Y. Petroff, this workshop organized by H. Graafsma, C. Ponchut (ESRF Instrument Support Group), P. Lindley, C. Kunz, and C. Bassani (ESRF Experiments Division) was attended by about sixty people, both from inside and outside of the ESRF. The aim of the workshop was to introduce the state of the art in pixel detector development as well as to discuss possible applications at the ESRF.

But what is a pixel detector? It can be defined roughly as a matrix of independent x-ray detection channels each one having its own signal processing circuits, all put together on one silicon chip. The presentations demonstrated that this concept could actually provide significant enhancements over currently used CCD-based x-ray detectors. For example, having more than 16 bits dynamic range and less than 10 ms readout time, they are able to meet some of the requirements expressed by S. Wakatsuki for protein crystallography and T. Narayanan for small angle scattering. Detailed



insights into detector design showed how it could also bring along several new features such as shutterless acquisition and coarse energy discrimination, thereby opening the way to a completely new field of experiments. New crystallographic data acquisition strategies such as fine phi slicing were proposed and discussed, in order to exploit these novel detector features. Examples of applications with evaluation results were presented for powder diffraction, medical imaging and spectroscopy, illustrating the number of possible implementations. Some matters of concern were not hidden but extensively discussed, such as radiation hardness and how to build large areas by stitching

together small unit modules.

As a conclusion it was agreed that several x-ray analysis techniques, among which SAXS, PX and time-resolved diffraction, could actually benefit from this technology as soon as detectors with larger area become available. Additional test experiments on unit detector modules will be carried out at the ESRF in order to fully assess pixel detectors on an experimental basis.

The organizers are specially indebted to the speakers for the quality of their twenty presentations, as well as to the ESRF staff for its active and efficient support, which resulted in a lively and successful event.

**C. Ponchut**



## 31<sup>ST</sup> COUNCIL MEETING

7 and 8 June 1999

### The Council

- discussed the future of the Multiple Wavelength Anomalous Dispersion beamline (**BM14**), in particular the options

- of maintaining BM14 in operation in view of an expected further increase in the demand for the MAD technique and
- of selling BM14 to a Collaborating Research Group or transforming it into an industrial beamline with a view to recuperating part of the ID29 expenditure for the beamline refurbishment budget,

and encouraged Management to explore further the feasibility of these options and have them discussed by the Science Advisory Committee at its next meeting;

- took note of the developing position with regard to the implementation

of the law on the reduction of working time to **35 hours** and to the revision of the *Convention d'Entreprise* (collective agreement) and discussed the aspects of funding compensatory measures in particular;

- approved (with the German delegation abstaining or dissenting for some items)

- the revision of the **1999 budget** and

- **the Medium-Term Financial Estimates (2000-2004)** including the planning figure for Members' contributions to the budget of 2000 (= 404 MFF),

as proposed by the Management;

- adopted a statement on **industrial policy**;

- noted the **scientific use** of the ESRF made by each Contracting Party from 1994 to 1998 and discussed the

reasons and the possible remedies for the imbalances found.

The council made the following appointments:

- P. Zinsli (Switzerland) as its Chairman for the period January 2000 to December 2001;
- H. Weijma (Benescync) as Chairman and B.L. Bye (Nordsync) as Vice-Chairwoman of the Administrative and Finance Committee / Audit Committee for the period July 1999 to June 2001;
- H. Fuess (TH Darmstadt) Chairman and J. Bordas (Laboratori de Leum Sincrotró, Barcelona) Vice-Chairman of the Science Advisory Committee;
- J. Bordas, G.H. Lander and D. Stuart as members to the Standing Committee on Permanent Appointments, in addition to the two *ex officio* members (Chairman of the SAC, Vice-Chairman of the Council).

**K. Witte**

## PUBLIC HEALTH MEASURES IN THE ESRF/ILL GUEST HOUSE

Two users who stayed in the Guest House during the first half of the year (one in February, the other in May) have contracted Legionnaires' Disease. Although a causal link between the stay in the Guest House and these cases of illness has not been proven, Management, after being informed of the first case, immediately undertook preventive measures and ordered tests. Legionnaires' Disease is caused by exposure to water aerosols polluted by a bacterium which may be present in water networks and air conditioning systems. Evidence of contamination has been found in some shower heads of buildings A and B of the Guest House which in itself is not too

surprising since these bacteria can be found in practically all water systems, particularly in stagnating warm water such as in shower units not regularly used.

Disinfection measures were carried out in July, which included: removal of calcium deposit from the heat exchangers, a heat shock treatment of the hot water network in buildings A and B, and a replacement of all shower heads, flexible hoses and the ceramic parts of the mixing batteries. After a series of tests for contamination had been carried out, all three buildings of the Guest House were again made available for use after the summer shut-down period (ie. from 12 August onwards).

In parallel, the ESRF has contacted all persons who arrived at the site from the 1 June onwards and who stayed in building A or B of the Guest House. They were informed of the problem and were asked whether they had any health problems subsequent to their visit to Grenoble. Fortunately no further cases have been reported.

For the future, preventive measures such as thermal shocks in all three buildings (twice a year), systematic monitoring of the legionella level and regular flushing of water in unused rooms will be implemented.

**K. Witte**