First test of detectors at the instrument BIODIFF

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The BioDiff instrument at the FRM II has seen the installation of its two detectors. A neutron scintillator with CCD-camera detector serves as auxiliary detector to perform crystal alignment and fast measurements with smaller solid angle coverage. The main detector, a neutron image plate with online read-out covers a somewhat larger solid angle of ca. 2π. Whereas the image plate detector could not yet be tested with neutrons so far first neutron measurements conducted with the CCD-detector have been performed and will be presented here. The instrument is intended for neutron protein crystallography and is a collaboration between the Forschungszentrum Jülich (FZJ) and the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II).

In the past year the BioDiff instrument has recieved its first neutrons. This is clearly a milestone in building any new neutron instrument. This milestone could only be reached by the timely delivery of all components. For a most recent view of the instrument see Figure 1. The instrument provides a monochromatic beam port with selectable neutron wavelength between 2.4 Å and 5.6 Å. The neutron wavelength can thus be adapted to the size of the unit cell and to the required spatial resolution. Both detectors provide a spatial resolution of better than 300 µm which makes the BioDiff instrument especially adapted to crystals with large unit cells. Macromolecular crystallography of proteins will therefore be the strongest field of applications.

The workshop of the physics department of the Technische Universität München made the detector housing (see Fig.1 on the left with yellow lead shielding), including the CCD camera assembly. Both detectors the image plate and the CCD detector fit into this aluminum frame. The change over from one detector to the other can be accomplished automatically by the movement of a series of stepper motors. The detector housing arm can be swung around the monochromator axis according to the wavelength used for the actual experiment. The sample goniometer equipped with a x,y stage on top of the detector housing holds the sample in an upside down fashion with a standard goniometer head known from commercial x-ray diffractometers. The sample goniometer can carry loads up to 100 kg which allows to use cryostats as sample environment. Alternatively sample temperatures between 80 K and 500 K can be reached using a nitrogen gas stream provided by an Oxford Cryosystems Cryostream 700 Series.

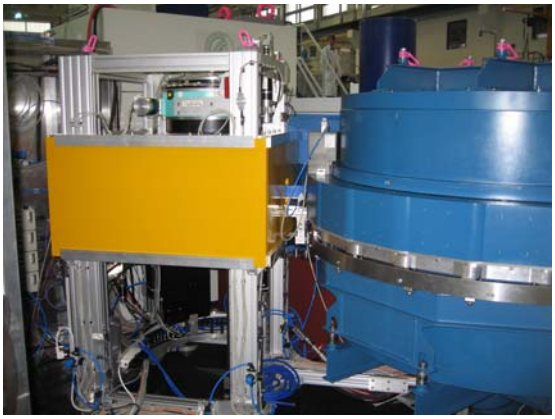

FIG. 1: Most recent side view of the instrument BioDiff at NL1 in the neutron guide hall of the FRM II in Garching. On the left: Detector housing with yellow lead shielding. The neutron image plate detector is still missing. It arrived during the long reactor shut down for maintainance. On the right: Blue lead shielding containing the monochromator crystal which later will accommodate the velocity selector for removal of λ/2 wavelength contamination of the monochromatic beam. In between detector housing and monchromator shielding the main instrument shutter, the γ-shutter is discernible.

The two instrument shutters a photo- and a γ-shutter and the assembly to move the monochromator crystal have been delivered by the Central Technology Division (ZAT) from the Forschungszentrum Jülich. The five axes assembly to move the monochromator crystal (Fig. 2 lower part) is used to move the pyrolytic graphite crystal according to Bragg's law to the right angle for the desired neutron wavelength. It also allows to lower the monochromator crystal completely out of the neutron beam. The photo-

shutter (Fig.2, upper part) is intended to provide a well defined exposure time by its fast and reproducible movement due to its light weight

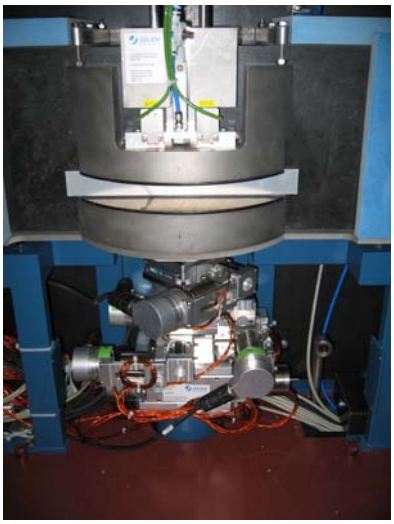

FIG. 2: The monochromator column inside the monochromator shielding (blue part on the right in Fig. 1) with the five axes assembly necessary to move the monochromator crystal according to the desired wavelength. In the upper part of the picture the first instrument shutter called photo-shutter can be seen which consists of a 4 mm B_4C ceramic plate moved by an air piston. The remaining space in front of the monochromator column is reserved for two apertures and a velocity selector.

neutron absorber and high pressure air piston.

The flux in the neutron guide gap just in front of the monochromator crystal has been measured by gold foil activation as 5.5 10^9 n/s/cm^2 for an assumed mean neutron wavelength of 5.4 Å. To measure the neutron flux at the sample position a fission chamber monitor detector was employed which measured a count rate corresponding to 1.5 10^6 n/s/cm^2 at the sample position at a wavelength of 4.77 Å for the used collimation, where the computed fraction of higher order contributions have already been subtracted. The collimation used for this measurement consisted of two boron carbide ceramic (B_4C) apertures of 3 mm diameter opening. Higher neutron flux, possibly up to 10^7 n/cm^2, may be achieved with collimation conditions allowing for larger divergence.

A sperm whale myoglobin crystal with deuterated mother liquor was used as a first test sample. With the CCD-camera detector moved to $2\Theta=0°$ resulting in the primary beam being centred in the middle of the scintillator the shadow of the sample due to scattering and some absorption is readily observed (Fig. 3). This opens the possibility to use the CCD-camera detector as a quick alignment tool to place the crystal in the beam. Moving the CCD-camera to a 2Θ value of -60° we then recorded first Bragg-reflexes from the myoglobin crystal (see Fig. 4).

First successful tests of the measurement software written by a team from the Central Institute for electronics (ZEL) have been performed and the image plate detector delivered by the company Maatel (Voreppe, France) has been installed. It is controlled by the measurement software by a TANGO client server interface.

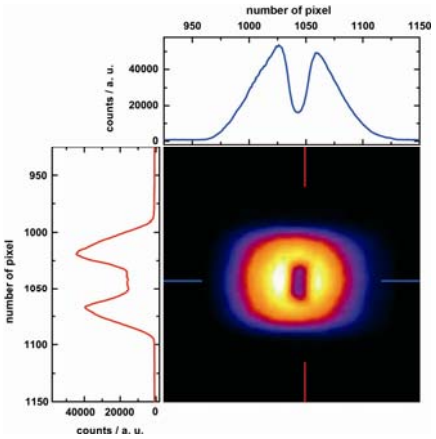

FIG. 3: Shadow of a myoglobin crystal in the primary neutron beam of the instrument BioDiff recorded with its CCD-camera detector. Cuts along the red and blue lines show the contrast reached.

Soon the image plate detector will be tested with neutrons. The complete alignment of the instrument is under way and software tests and development will have to proceed. With the advent of the next neutron beam time full data sets on test crystals will be recorded. Especially the background due to γ-radiation will be examined carefully and if necessary all feasible measures to reduce its impact on the measurement will be taken.

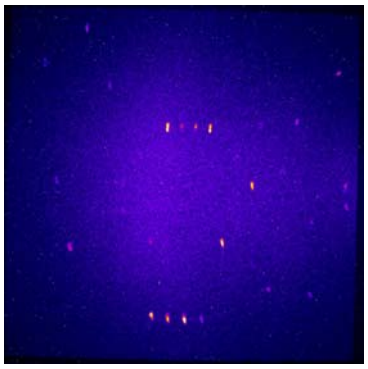

FIG.4: One of the first images recorded with the CCD-camera detector on the sample myoglobin. The exposure time was 500 s. No corrections were applied.