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White Beam Synchrotron Topography
Using a High Resolution Digital X-Ray Imaging Detector

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Abstract

X – ray topography is a well known imaging technique to characterise strain and extended defects in single crystals. Topographs are typically collected on X-ray films. On the one hand such photographic films show a limited dynamic range and the production of films will be discontinued step by step in the near future. On the other hand new imaging detectors improved for X-ray tomography become more and more attractive even for topography because of increasing resolution, dynamic range, speed and active area. In this paper we report about the upgrade of the Topo-Tomo beamline at the synchrotron light source ANKA, Research Centre Karlsruhe, with a high resolution digital camera for the topography use.

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1. Introduction

White beam X-ray topography using synchrotron radiation is a non-destructive diffraction imaging technique. It is widely used for the characterisation of long and short range strain in single crystals which result e.g. from dopant striations and crystal defects like dislocations respectively [1]. The method is based on illuminating a single crystal with a white synchrotron X-ray beam and to record the X-rays as diffracted by the sample. During one exposure a full Laue pattern of the crystal is collected, typically on X-ray film (fig.1a). Every reflection is a topograph from the same sample position which is frequently magnified and digitized with a light microscope to analyse the number and type of dislocations.

The advantage of these films is the large field of view which is only limited by the size of the film (e.g. 13 cm x 18 cm). This allows the collection of a large number of topographs during one exposure. The high sensitivity and resolution allows a detailed analysis of defects and strain. Major drawbacks are the low duty cycles (the fraction of exposure time and read-out time compared to the whole time required for acquiring an image), poor dynamic range and the fact that high quality X-ray films are disappearing from the market [2].

One option to overcome these limitations are digital high resolution imaging detectors. Our approach focuses on indirect detection systems. They are frequently based on projecting a luminescence image magnified via microscope optics onto a CCD chip. The advantages are high duty cycles, high spatial resolution and a high dynamic range. Such a detector is well-suited to image single topographs.

In the present paper we describe the merits of the digital topography setup at the TOPO-TOMO beamline of the German synchrotron light source ANKA ("Ångströmquelle Karlsruhe") at the Research Centre Karlsruhe with a digital X-ray imaging detector, commonly used for microtomography and –radiography.

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2. Experimental setup

2.1 White beam X-ray topography

The synchrotron light source ANKA at the research centre Karlsruhe is operated with an electron energy of 2.5 GeV and beam currents of 180 – 80 mA. The dipole bending magnets are working with 1.5 T magnetic field. The resulting critical wavelength of 2 Å is perfectly suited for topography of inorganic materials. ANKA is housing several bending magnet (BM) beamlines and currently two insertion device (ID) beamlines. Two additional ID beamlines and several BM beamlines are in preparation. Since 2002 topography is performed at ANKA, located on a BM beamline which is currently dedicated to topography and tomography experiments (TOPO-TOMO) [3]. As a special feature of the TOPO-TOMO beamline there are no optical components between the bending magnet source and the experimental station, except for one Be-window directly in front of the experiment. A double slit system inside the high vacuum tube, positioned closely upstream to the Be-window, allows for a control of the irradiated area of the sample (up to 10 mm x 10 mm for large area topography as well as 15 µm x 10 mm for section topography). The sample is mounted on a 2-circle goniometer with x- and z- translation. The 30 m long source-to-sample distance and the small source size of 800 µm x 200 µm (horizontal x vertical) result in the high geometrical resolution of the topographs (about 1 µm) [3,4].

To perform topography the sample is illuminated with the white synchrotron beam. The Laue patterns to be recorded are based on the diffraction of the X-rays by the sample. Every diffraction vector $\vec{g}$, which fulfils Bragg's law, results in one topograph from the same volume of the sample during one single exposure and results in a Laue pattern of topographs [1]. Normal resolution X-ray films (Agfa D3 single coated) are used mainly for adjustment. Characterisation and values of the dynamic range of the actual X-ray films are difficult to get. The dynamic range is the saturation density over the minimum resolvable optical density. A

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recent comparison of typical X-ray films as Kodak SR45 and Agfa D4sc is given by [2]. Assuming values in between the SR45 and D4sc, the dynamic range of our applied X-ray film D3sc can be estimated in between 18.5 (SR45) and 46.3 (D4sc) gray levels.

Slavich VRP-M films (high resolution film) with a grain size of 0.05µm are used for high resolution topography. This film is originally a holographic film, optimized for green light and therefore less sensitive for X-rays. The exposure times required are about 60 times longer than the Agfa X-ray film. The observed higher dynamic range is reasonable and fits well with the result that with increasing exposure time the dynamic range of a film increases significantly [2].

Typical exposure times for a full Laue pattern of topographs depend on various parameters e.g. the absorption of the material, the structure factors of the reflections and the thickness of the crystal slices. Typical values range from about several tenths of a second for low absorbing crystals (e.g. Si) up to some tenths of minutes for highly absorbing crystals (e.g. GaSb or CdTe).

Fig 1a shows such a large area transmission pattern of highly S-doped InP (sample AMF13), grown from In-solution [5]. The crystal slice with a thickness of about 850 µm was cut along (110) parallel to the growth direction [111] and was mechano-chemically polished on both sides to eliminate any damage layer. The sample was tilted by 10° to find the 111 reflection. The wavelength of the 111 reflection under these conditions is 0.11768 nm .According to ref. [6] the linear absorption coefficent for InP at 10.536 keV is 446 cm^{-1}. For the effective thickness t_{eff} = 0.0863 cm at 10° the product $\mu \cdot t_{eff}$ becomes 38.5. The distance from the crystal to the normal resolution film was 9 cm with the screened area 5 x 5 mm^2 and an exposure time of about 8 seconds. Fig. 1b shows the corresponding simulation with program LauePT [7] which is used for indexing the Laue pattern but also for adjusting the

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crystal. Finally the topographs are magnified and digitised by light microscopy and a conventional CCD camera. The digitised topographs are image processed with respect to contrast and sharpness. The topograph shown in fig. 2 was taken with high resolution film (Slavich VRP-M) and an exposure time of about 20 minutes to resolve all the different defect structures.

2.2. X-ray imaging detector

For scanning one selected reflection of a Laue pattern with high spatial resolution we use a digital X-ray imaging detector which has been installed at the TOPO-TOMO beamline for microtomography –and radiography [8]. Its concept is based on the work of F. Busch and U. Bonse [9]: a light-microscope design is used to project the magnified luminescence image of a scintillating screen onto a CCD chip – see fig. 3. The maximal lateral resolution of this device is determined by the intrinsic pixel size of the CCD chip, the microscope optics, the scintillating screen as well as Shannon's theorem and has already been extended to the submicrometer range [10]. One consequence of the finite size of the CCD chip is the detector's limited field of view in comparison to classical X-ray films. Our microscope design is consequently a trade-off between high resolution and large field of view ("macroscope"): as tube lens a commercial photo objective Nikon "Nikkor 180/2.8 ED" (f = 180 mm) is combined with Rodenstock objectives, leading to magnification factors between 1.5x and 3.6x [11]. Typically we combine these with a CCD camera from the PCO AG which uses a Kodak KAI-11000 CCD chip (interline transfer) with 4008 x 2672 pixels, each sized 9 µm [12]: applying a Rodenstock "TV-Heliflex" (f = 50 mm, NA=0.45) leads to an effective pixel size of 2.5 µm and a 10.0 mm x 6.7 mm field of view (maximal lateral resolution is approx. 5 µm, as verified with Xradia test pattern X500-200-30). Lower magnification modes are available, e.g. with a 5.5 µm effective pixel size and a 22 mm x 14 mm field of view. Our scintillating

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screen is a 25 mm x 25 mm $CdWO_4$ (CWO) single-crystal, 300 µm thick which is polished on both sides to optical quality (by FEE GmbH, Germany) [13]. In the optical path of our detector we added a diaphragm to adjust the numerical aperture to the aimed resolution and the ratio of optical photons per converted X-ray (scintillating screen) vs. detected optical photons in our detector (CCD): in an ideal case each X-ray should lead to one digital count in our CCD in order to exploit the chips dynamic range.

The technical specifications of the CCD camera as delivered by the manufacturer claim a full well capacity of 60.000 electrons per pixel, the read-out noise is 12 e-rms (slowest readout for highest dynamic range) leading to a dynamic range of 5.000 graylevels (74 dB, 13 bit) [12].

For the digital topography in fig. 4 the 111 – reflection (compare fig. 1a) was selected and adjusted to illuminate the camera's field of view. The combination of the detector setup with the tomography sample manipulator allowed 56 cm as the shortest available distance from crystal to camera. Due to this long distance a crystal tilt of 5.4° was adjusted which results in a wave length of 0.06377 nm. The linear absorption coefficent µ at 19.442 keV reduces to 82 cm^{-1} [6]. For the effective thickness l_{eff} = 0.0854 cm at 5.4° the product $\mu \cdot t_{eff}$ is 7.0, but due to the longer distance in air a longer exposure time is needed. Most of the contrasts becomes already visible after a 10 minutes exposure time, but for a noise reduced, high contrast topograph 60 minutes exposure time are chosen.

3. Results and discussion

Figs. 2 and 4 compare the topographs taken with high resolution film and digital camera, respectively, at the same position of the InP crystal. In fig. 2 pronounced horizontal dopant

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inhomogeneities are visible in the centre of the crystal as a result of the (111) facet (compare figs. 2a and 4a) [14]. Straight V-shaped lines originating in the seed and at the interface seed – grown crystal are 60° dislocations of the type **b** = 1/2a [110]. Even at the border of the crystal with very a high dislocation density (about $10^5 cm^{-2}$) the single curved dislocations are resolved. For higher dislocation densities the local strain fields of dislocations overlapresulting in diffuse dark regions on thefilm making a discrimination of single dislocation lines impossible. Even at higher magnification (x 100) and bright illumination of the light microscope the resolution is limited and no image post processing can reveal sharp dislocation lines (fig. 2b).

The same features become visible in the topograph taken with the digital camera at the same sample position as shown in fig.4. The long term strain from dopant inhomogeneities as well as short range strain around the various types of dislocations is imaged with a equally high resolution as with high resolution film. The resolving power is the same as on a high resolution film but here combined with a much higher dynamic range. Furthermore post image processing allows to optimise either for the aspect of high dynamic range (fig.4b) or high contrast and sharpness (fig.4c) from the same set of data.

The PCO camera used delivers images with a dynamic range of up to 5.000 graylevels (13bit, 74 dB) - depending on the read-out speed [12]. In principle this means that the brightest detail in the image can be 5000 times brighter than the darkest feature. For the sake of simplicity these data are stored as 16bit TIFF files. The human eye (depending on the individual) can distinguish up to 200 different grey-levels which corresponds more or less to 8bit values. Therefore the 16bit data is reduced to 8bit for displaying purposes. Usually the reduction is done by choosing an interval in the image's histogram: values in that range are projected on the [0, 255] interval, values below the chosen greyscale interval are set to 0, values above to 255. Of course this results in a loss of information, therefore the selected greyscale interval

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has to be optimized for the corresponding image or region-of-interest chosen. As the dynamic range of our digital topography setup is much higher than for classical X-ray films we can image a Laue pattern and then decide during post-processing of our data which features are important and extract them, e.g. into different images (fig. 4b,c).

Therefore the higher dynamic range of an X-ray imaging detector allows more detailed topographs at a similar high resolution as conventional photographic films. Combined with an x-z-translation system for the sample, an automated quick and efficient mapping of wide crystal areas becomes possible [15].

The disadvantage of the actual camera is the recording of only one reflection at a time due to the finite size of the camera's CCD chip. For many standard applications, e.g. the mapping of large silicon wafers where mainly the 220 reflection is relevant for characterisation, the digital topography is a big progress. If more reflections with various diffraction vectors for a Burgers vector analysis are needed, the sample has to be tilted/turned and the camera to be moved on a x-z-stage to collect all the reflections of the Laue pattern shown in fig. 1a.

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Figure captions:

Fig. 1: Laue Pattern of InP:S slice, orientation (110), tilt 10°, distance crystal-film 9cm:
(a) Normal resolution X-ray film (Agfa D3sc) exposure time 8 seconds.
(b) Indexing of the Laue pattern by LauePT [6]

Fig. 2: (a) Topograph from InP:S, (AMF13, (110)-slice, 111 reflection) taken with high resolution film (Slavich VRP-M, inverted and image post processed for high contrast and sharpness), exposure time about 20 minutes. Intense horzontal dopant striations and various types of dislocation lines are visible (for details compare fig. 4a)
(b) Detail from (a) with higher magnification of the light microscope (x 100)

Fig.3: (a) Sketch of the detector concept: the X-rays are converted to visible light by a scintillating screen ($CdWO_4$ single crystal), this luminescence image is projected (magnified) onto a cooled CCD chip [8].
(b)– Photo of the mounted detector system at the Topo-Tomo beamline (ANKA) [10]

Fig. 4: (a) 111 - topograph from InP:S (same as in fig. 3 AMF13, (110) slice, 5.4° tilt) but taken with the digital X-ray imaging detector, exposure time 60 minutes, distance crystal – film 56 cm. Intense horizontal dopant striations and various types of dislocation lines are visible.
(b) Magnified from the original TIFF-file and image post processed for high dynamic range
(c) Magnified from the original TIFF-file and image post processed for high sharpness

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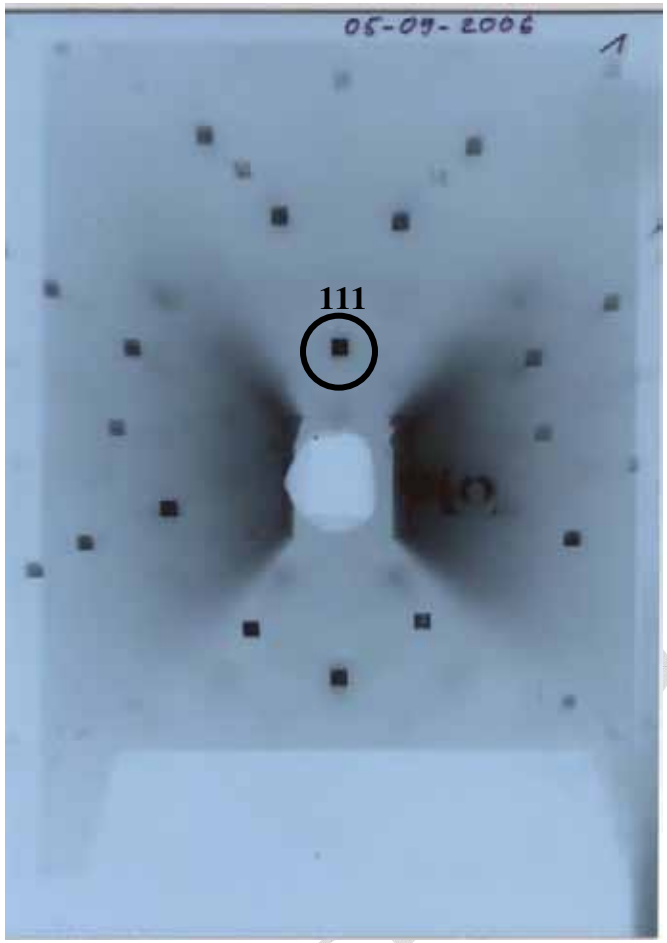
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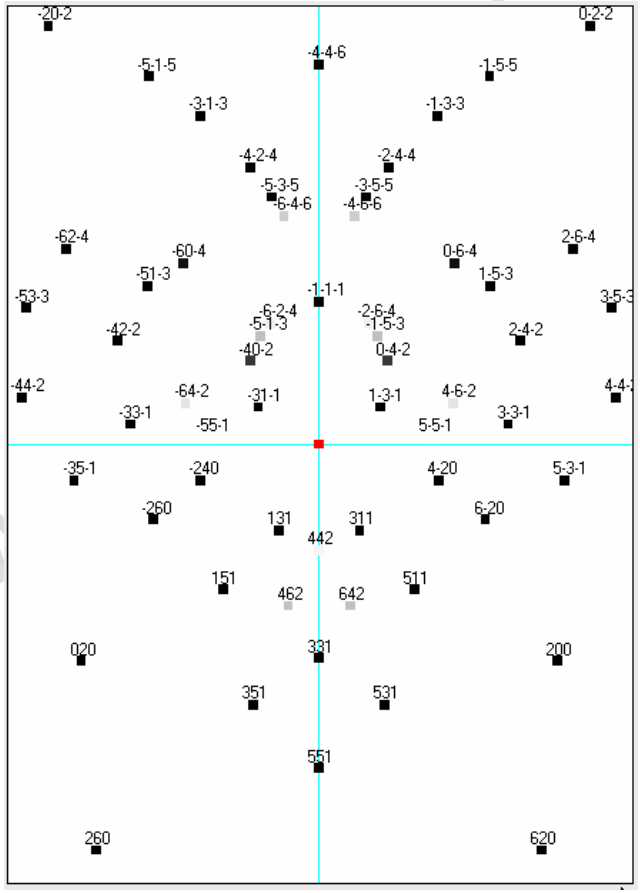
111

(a)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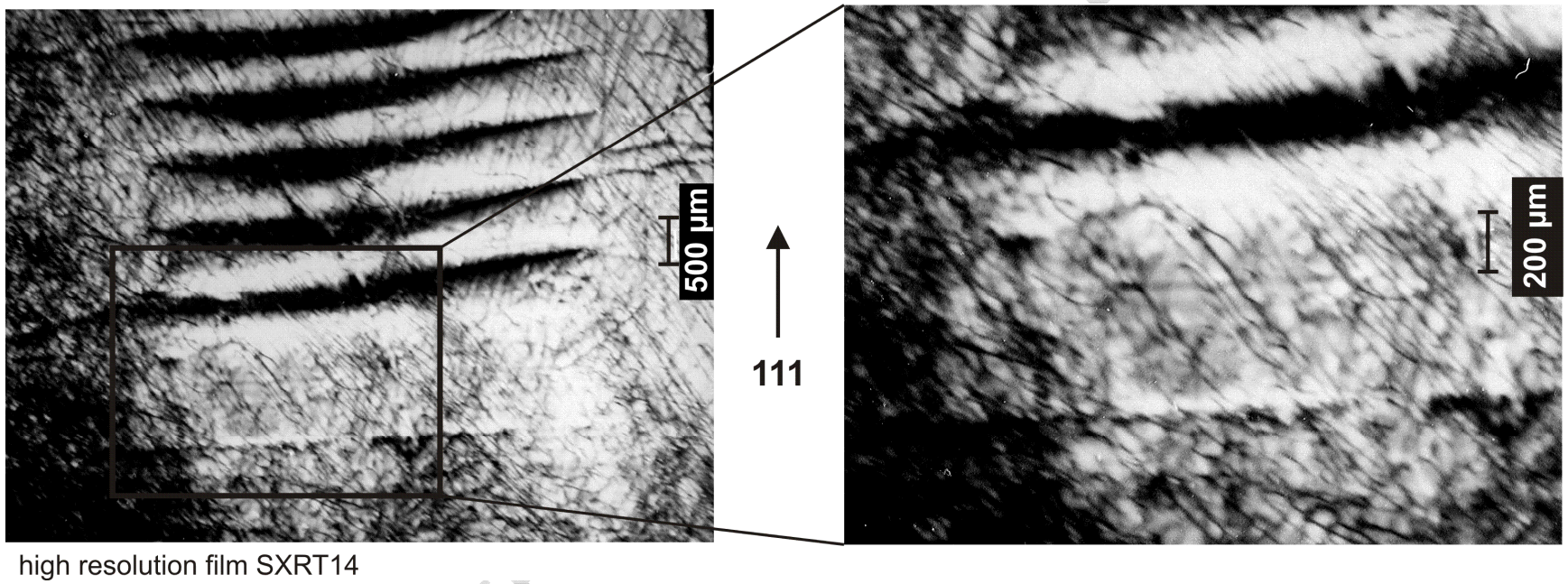
(b)

Fig.1



(a) (b)

Fig. 2

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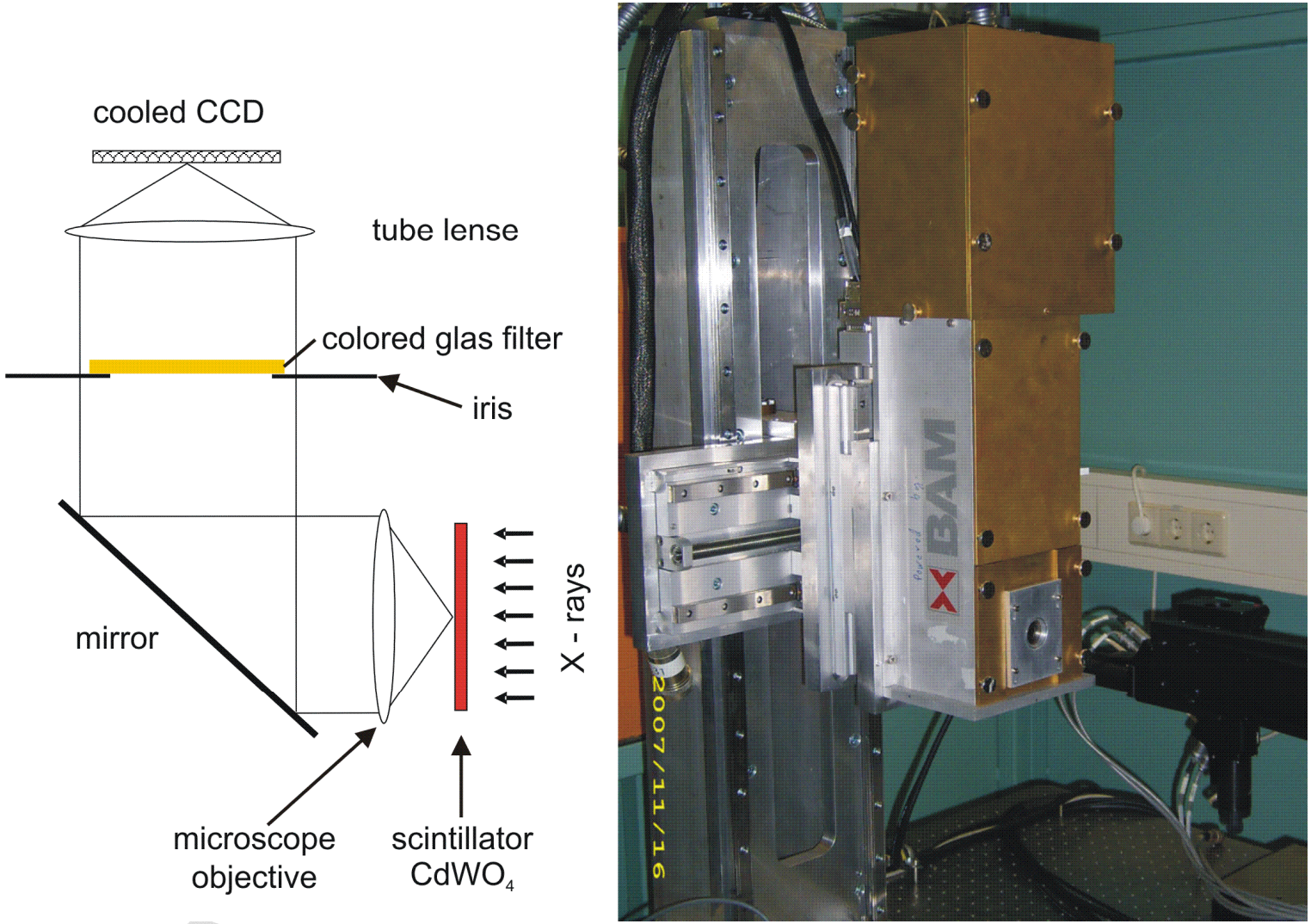
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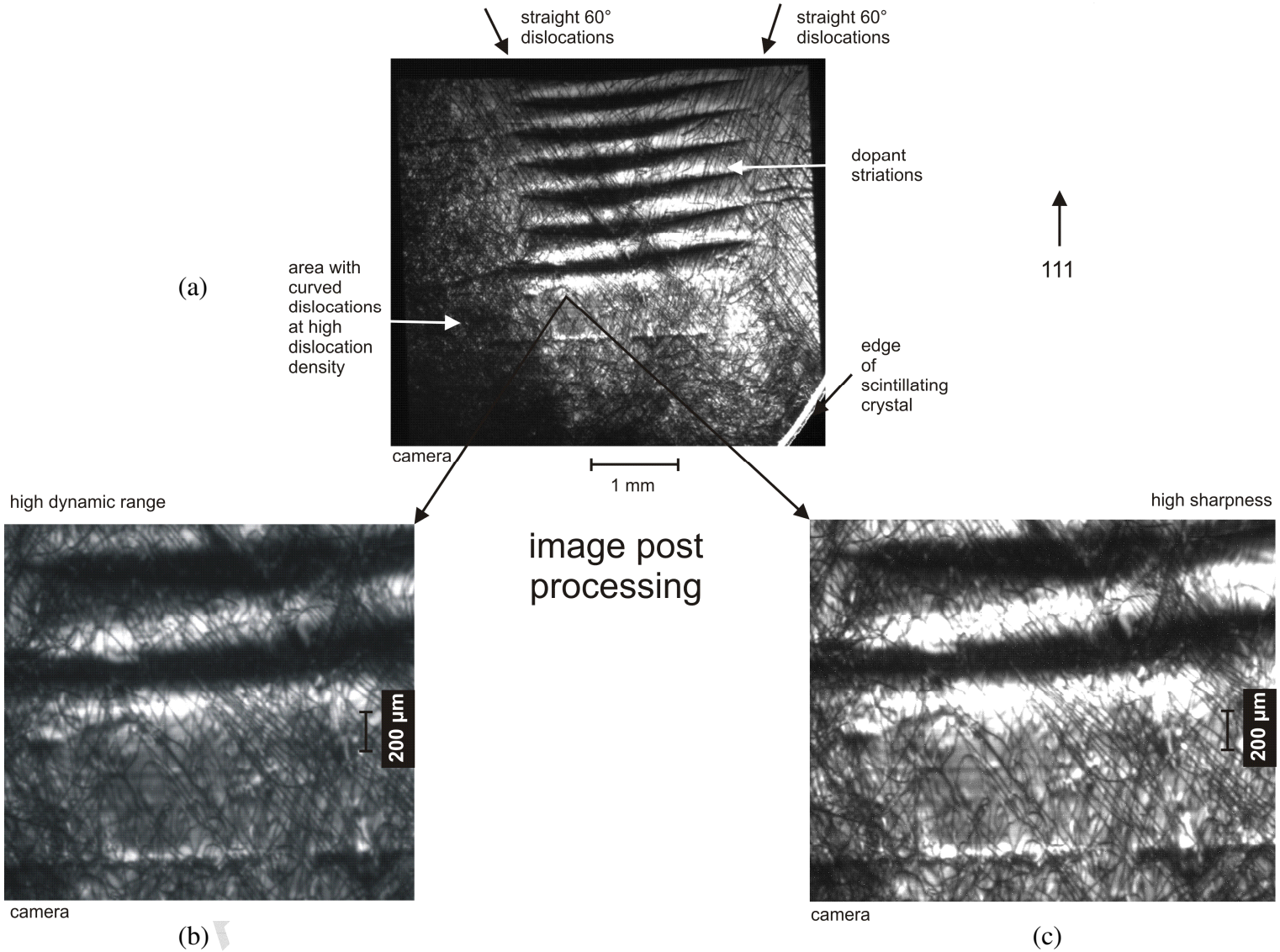
(a) (b)

Fig. 3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(b)
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Fig. 4