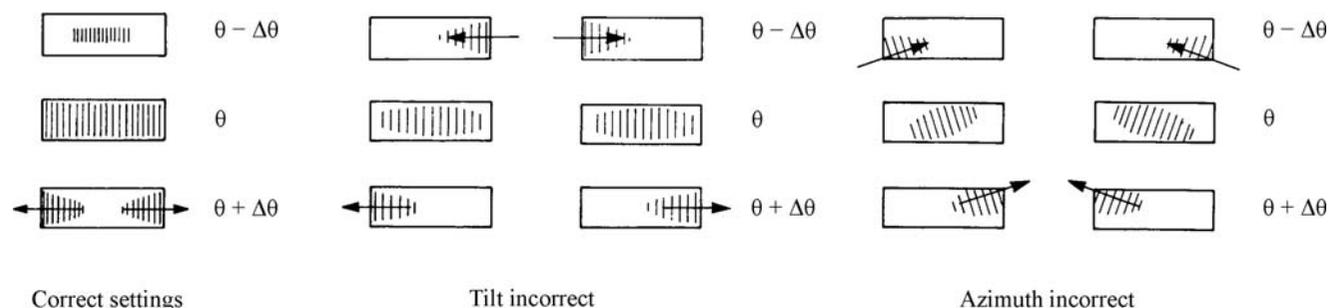


3. METHODOLOGY

**Figure 3.1.22**

Figures found within the instructions for a Siemens D500 incident-beam monochromator in a Huber 611 monochromator housing, illustrating image formation and movement for correct and incorrect settings of tilt and azimuth angles (reproduced with verbal permission from Huber).

with the line source of the tube anode, and then the tube shield/IBM assembly is aligned with the goniometer. The second stage is analogous to the instrument alignment described above, so here we will discuss only the first stage (although not exhaustively).

The alignment of the Johansson optic to the X-ray source is done largely with the X-rays present. The crystal tilt and azimuth are set by using a fluorescent screen or camera to observe the diffraction images from the optic as it is rotated through its diffraction angle. Fig. 3.1.22, which is reproduced from the instructions supplied by Siemens, shows how the images form and move, informing the operator of necessary adjustments. Initially, a set of hex-drive extensions was used to drive the optic remotely through its 2θ angle. The source was operated at full power while the movement of the image was observed through a lead-impregnated window. Later, a motor drive was installed onto the 2θ actuator of the 611 housing. In the end, the incident-beam intensity realized from the optic is dependent upon the operator's ability to discern the subtleties in the image movement (Fig. 3.1.22). Blocking the axially divergent signals from the optic with a high-resolution 0.05° Soller slit dramatically improves the sensitivity of this observation to the setting of the tilt and azimuth angles. The inclusion of the Soller slit, however, will reduce the intensity markedly. A complete darkening of the room, including blocking of the shutter lights, as well as allowing time for pupil dilation, can be helpful. However, the use of an X-ray imager or a PSD in picture-taking mode improves the quality of the alignment by allowing for a more accurate interpretation of the observations.

The goal is the formation of an image in the centre of the beam path that splits symmetrically out to the edges with increasing crystal 2θ angle (Fig. 3.1.22). The directions supplied by Siemens and Huber allude to the fine adjustment [see Huber (2014) for movies] of the tilt and azimuth by examining the structure of the diffracted beam at the optic's focal point. A fluorescent screen located at the focal point and set at a 6° angle to the beam path is used to image the beam structure. With the use of the Soller slit for coarse alignment of tilt and azimuth, the desired final image for the fine-adjustment mode was, indeed, obtained. But it was not possible, even with a deliberate mis-setting of tilt and azimuth angles, to use the defective images at the focal point as a source of feedback for correcting the settings because they were too diffuse.

The Johansson optic is supplied with a and b distances that correspond to the angle of asymmetry in the diffraction planes and the bend radius. The instructions indicate that an incorrect setting in a will cause the optic's diffraction image to move up or down in the plane of diffraction with variation of the crystal 2θ angle. Again, a lack of sensitivity prevents the use of this effect as a feedback loop to set a . Alternative experiments for the opti-

mization of the distance a of the optic were time consuming and not conclusive, so we decided to accept the supplied value for a . As before, we set the takeoff angle at 6° . A critical and quite difficult problem is the alignment of the slit located between the X-ray tube and the crystal optic (not shown in Fig. 3.1.3). This slit centres the beam onto the active area of the optic; misalignment leads to unwanted scatter from the optic's edges. It is aligned with the X-ray beam present, yielding an image of the shadow cast by the optic itself on one side, and one edge of the slit on the other. The optic is rotated in 2θ so that its surface is parallel to the X-ray beam, *i.e.* shadowing is minimized. The shadow from the second edge of the slit is obscured by the optic. Geometric considerations are used in conjunction with knowledge of the radius of curvature of the optic to obtain the correct location for the slit. A drawing is highly useful in this instance. After the installation of this slit, it is appropriate to re-check the tilt and azimuth settings, as the alignment of the optic is nearly complete.

The setting of the crystal 2θ is performed by evaluation of the direct beam, either with scans using a scintillation detector or by taking pictures with a PSD. With increasing crystal 2θ , the beam diffracted by the optic will build in the centre forming a broad profile; then the intensity on either side of the initial profile will rise, leading to the desired box form; and then intensity at the centre of the box will fall, followed lastly by the intensity at either side of the centre. This is consistent with Fig. 3.1.22. The process will repeat at half the $K\alpha_1$ intensity for the $K\alpha_2$ line. (Avoid tuning to the wrong line.) The crystal 2θ setting should be checked at regular intervals with a scan of the direct beam; this is the only setting on the IBM that has been observed to drift with time.

The final step in alignment of the IBM is the installation of the anti-scatter slit located at the focal line of the optic (Fig. 3.1.3). This is performed after the IBM assembly is aligned to the goniometer. Optimal performance of the anti-scatter slit can be expected only if it is located precisely at the focal line, which itself constitutes the smallest region within which a maximum of X-ray flux is transmitted. Therefore, the NIST alignment procedure includes an experiment using a narrow slit positioned by an x - y translator to evaluate the relative flux of the beam in the vicinity of the focal line. The y direction is parallel to the b direction (Fig. 3.1.3). A 0.05 mm slit is translated across the beam in the x direction, while intensity readings are recorded from an open detector. This process is repeated for a sequence of y distances. A plot of the recorded intensity *versus* x at a sequence of y settings will yield a set of profiles which broaden on either side of the true value of b ; the narrowest, highest-intensity profile will indicate the location of the focal line. Thus, the experiment determines both the true b distance and the location in the x direction of the focal line. Once b is known, translational adjustment of the IBM