

2. RECIPROCAL SPACE IN CRYSTAL-STRUCTURE DETERMINATION

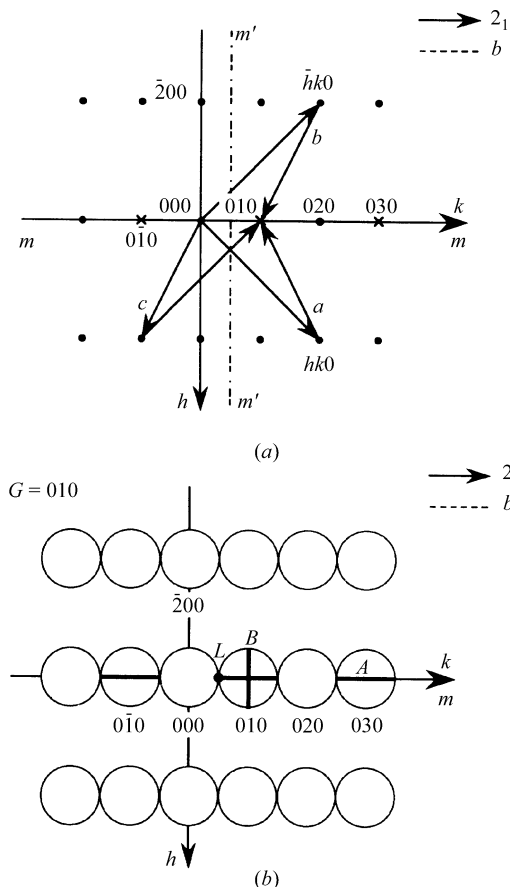


Fig. 2.5.3.10. Illustration of the production of dynamical extinction lines in kinematically forbidden reflections due to a  $b$ -glide plane and a  $2_1$  screw axis. (a) *Umweganregung* paths  $a$ ,  $b$  and  $c$ . (b) Dynamical extinction lines  $A$  are formed in forbidden reflections  $0k0$  ( $k = \text{odd}$ ). Extinction line  $B$  perpendicular to the lines  $A$  is formed in the exactly excited  $010$  reflection.

In principle, a horizontal screw axis and a vertical glide plane can be distinguished by observations of the extinction lines  $A_3$  and  $B_3$ . It is, however, not easy to observe the extinction lines  $A_3$  and  $B_3$  because broad extinction lines  $A_2$  and  $B_2$  appear at the same time. The presence of the extinction lines  $A_3$  and  $B_3$  can be revealed by inspecting the symmetries of fine defect HOLZ lines appearing in the forbidden reflections instead of by direct observation of the lines  $A_3$  and  $B_3$  (Tanaka, Sekii & Nagasawa, 1983). That is, if HOLZ lines form lines  $A_3$  and  $B_3$ , HOLZ lines are symmetric with respect to the extinction lines  $A_2$  and  $B_2$ . If HOLZ lines do not form lines  $A_3$  and  $B_3$ , HOLZ lines are asymmetric with respect to the extinction lines  $A_2$  and  $B_2$ . When the HOLZ lines are symmetric about the extinction lines  $A_2$ , the specimen crystal has a glide plane. When the HOLZ lines are symmetric with respect to lines  $B_2$ , a  $2_1$  screw axis exists. It should

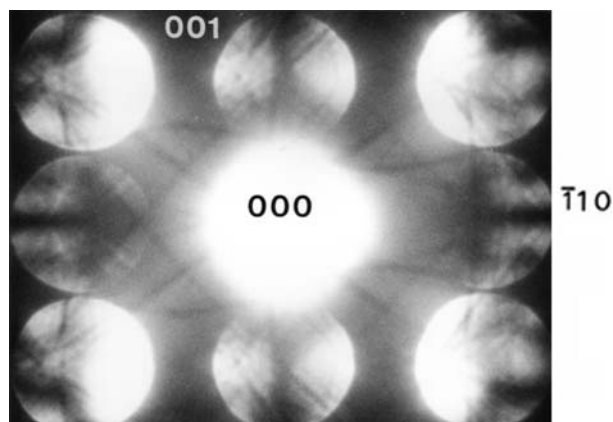


Fig. 2.5.3.12. CBED pattern of FeS<sub>2</sub> taken with the  $[110]$  electron-beam incidence. In the 001 and  $00\bar{1}$  discs, HOLZ lines are asymmetric with respect to extinction lines  $A_2$ , indicating the existence of a  $2_1$  screw axis parallel to the  $c$  axis. In the  $\bar{1}10$  and  $1\bar{1}0$  discs, HOLZ lines are symmetric with respect to extinction lines  $A_2$ , indicating existence of a glide plane perpendicular to the  $c$  axis.

be noted that a relatively thick specimen area has to be selected to observe HOLZ lines in ZOLZ reflection discs.

Fig. 2.5.3.11 shows CBED patterns taken from (a) thin and (b) thick areas of FeS<sub>2</sub>, whose space group is  $P2_1/a\bar{3}$ , at the 001 Bragg setting with the  $[100]$  electron-beam incidence. In the case of the thin specimen (Fig. 2.5.3.11a), only the broad dynamical extinction lines formed by the interaction of ZOLZ reflections are seen in the odd-order discs. On the other hand, fine HOLZ lines are clearly seen in the thick specimen (Fig. 2.5.3.11b). The HOLZ lines are symmetric with respect to both  $A_2$  and  $B_2$  extinction lines. This fact proves the presence of the extinction lines  $A_3$  and  $B_3$ , or both the  $c$  glide in the (010) plane and the  $2_1$  screw axis in the  $c$  direction, this fact being confirmed by consulting Table 2.5.3.9. Fig. 2.5.3.12 shows a  $[110]$  zone-axis CBED pattern of FeS<sub>2</sub>.  $A_2$  extinction lines are seen in both the 001 and  $\bar{1}10$  discs. Fine HOLZ lines are symmetric with respect to the  $A_2$  extinction lines in the  $\bar{1}10$  disc but asymmetric about the  $A_2$  extinction line in the 001 disc, indicating formation of the  $A_3$  extinction line only in the  $\bar{1}10$  disc. This proves the existence of a  $2_1$  screw axis in the  $[001]$  direction and an  $a$  glide in the (001) plane. The appearance of HOLZ lines is easily changed by a change of a few hundred volts in the accelerating voltage. Steeds & Evans (1980) demonstrated for spinel changes in the appearance of HOLZ lines in the ZOLZ discs at accelerating voltages around 100 kV.

Another practical method for distinguishing between glide planes and  $2_1$  screw axes is that reported by Steeds *et al.* (1978). The method is based on the fact that the extinction lines are observable even when a crystal is rotated with a glide plane kept parallel and with a  $2_1$  screw axis perpendicular to the incident

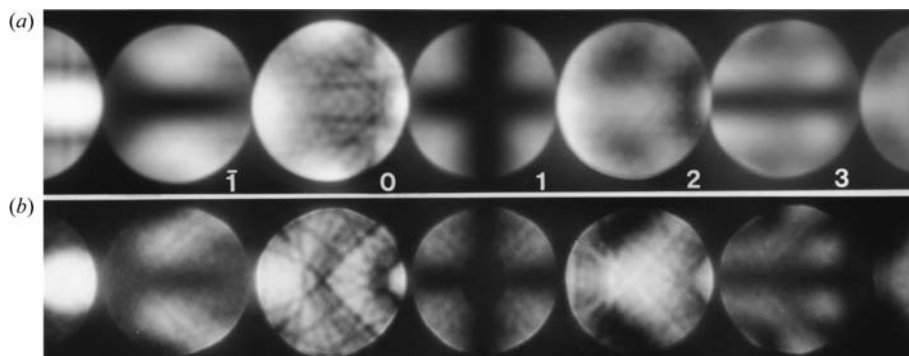


Fig. 2.5.3.11. CBED patterns obtained from (a) thin and (b) thick areas of (001) FeS<sub>2</sub>. (a) Dynamical extinction lines  $A_2$  and  $B_2$  are seen. (b) Extinction lines  $A_3$  and  $B_3$  as well as  $A_2$  and  $B_2$  are formed because HOLZ lines are symmetric about lines  $A_2$  and  $B_2$ .