Systematic row. Measurement of s-fringe profiles in CBED discs from strong inner reflections in systematic rows was tried by MacGillavry (1940) and developed into a method for structure-factor determination by Goodman & Lehmpfuhl (1967) and later authors. At present, this may be the commonest method for refinement of low-order structure factors from CBED. A detailed account is given in the book by Spence & Zuo (1992) and in Spence (1993). Strong non-systematic interactions should be avoided. The intensity profiles can often be approximated by the two-beam expression

$$I_g(s_g) = \frac{(U_g/k)^2}{s^2 + (U_g/k)^2} \sin^2\left[\pi t \sqrt{s_g^2 + (U_g/k)^2}\right], \qquad (8.8.6)$$

especially when U_g is substituted by an 'effective potential' which may be defined by the corresponding gap at the dispersion surface, *viz* $U_g^{\rm eff} = k(\gamma^i - \gamma^j)_{\rm min} = k/\xi^{ij}$, where ξ^{ij} is an extinction distance. The outer part of the profile (large s_g) depends mainly on the thickness, whereas the inner part is sensitive to the product tU_g. Different perturbation expressions have been proposed for the effective potential. The Bethe potential

$$U_g^{\rm eff} = U_g - \sum_h \frac{U_h U_{g-h}}{2ks_h} \qquad (8.8.7)$$

is often used, *e.g.* in the early steps of the refinement procedure (Gjønnes, Gjønnes, Zuo & Spence, 1988), and especially in order to treat weak beams beyond the typically 60–80 beams included in the Bloch-wave diagonalization (Zuo, 1993). Procedures and computer programs adapted to least-squares refinement of structure factors from energy-filtered line profiles are described by Spence (1993), Zuo (1993) and Deininger, Necker & Mayer (1994). The refinement will usually include experimental parameters (thickness, beam orientations) as well as elastic and absorptive parts of a few low-order structure factors for each profile – but not high-order structure factors and thermal parameters, which are assumed. Low-order structure factors for a number of simple substances have been determined. Errors in the best results, referred to as X-ray structure amplitudes, are of the order of 0.1% – which may be a tenth of the bonding effect in covalent compounds. See, for example, the recent study of the intermetallic compound TiAl and a variant doped with 5% Mn (Holmestad, Weickenmeier, Zuo, Spence & Horita, 1993), where the charge-density deformation distribution

$$\Delta\rho(\mathbf{r}) = \frac{1}{\Omega} \sum_g [F_g^X(\text{crystal}) - F_g^X(\text{free atom})] \exp[2\pi i \mathbf{g}\cdot\mathbf{r}] \qquad (8.8.8)$$

was constructed from nine-low-order structure factors.

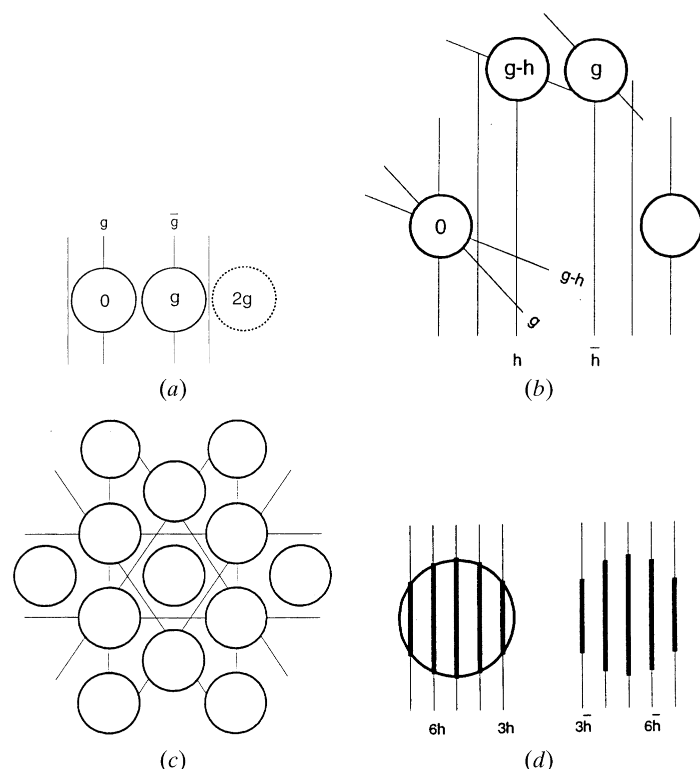

Fig. 8.8.1. Schematic representations of four convergent-beam configurations used for structure-factor determination: (*a*) intensity profile of a low-order reflection, g; (*b*) non-systematic three- or four-beam configuration with a strong coupling reflection, h; (*c*) symmetric many-beam configuration in a dense zone; (*d*) integrated intensity measurement of high-order reflections using a wide aperture (Taftø & Metzger, 1985).

Three- and four-beam, non-systematic cases. Several magnitudes can be extracted from Kikuchi or CBED patterns in such configurations, see *e.g.* Gjønnes & Høier (1971). In the non-systematic critical-voltage method, the condition for extinction of line contrast is measured; in the IKL (intersecting Kikuchi line) method, one measures the separation between line segments of the split line appearing at the intersection with a strong Kikuchi band. The precision of these methods, originally developed for Kikuchi patterns, was increased considerably when CBED was used instead (Matsuhata, Tomokiyo, Watanabe & Eguchi, 1982; Taftø & Gjønnes, 1985).

A further improvement is expected when the intensity distribution over the whole CBED discs is recorded and fitted to dynamical calculations. This has been explored recently by Høier, Bakken, Marthinsen & Holmestad (1993); the addition of non-systematic reflections in a parallel row can be seen as an extension of the systematic row configuration above. The experience so far is that the three- and four-beam configurations are very sensitive to structure-factor phases, but may not yield as accurate values for structure amplitudes as those obtained from the line profiles in the systematic row.

Zone axis CBED. Convergent-beam patterns around the axis of a dense zone contain extensive multiple-beam dynamical interaction. Bird & Saunders (1992) claim that this leads to high sensitivity as well as a high number of structure factors that can be determined from one CBED pattern. Their results from refinement of structure amplitudes in f.c.c., diamond and sphalerite structures may confirm this. From filtered intensities measured with a CCD camera, Saunders, Bird, Midgley & Vincent (1994) determined structure factors for silicon up to 331 from one pattern in the [110] zone. The point-spread function for the detector was deconvoluted from the raw data. Thickness, background level and a scaling factor were included in the refinement from a grid of 21×21 intensities in each disc. Starting values were neutral atom scattering factors, absorptive scattering amplitude calculated from TDS and a preliminary thickness determination. 121 beams were included in the diagonalization, a further 270 beams by perturbation.

Critical voltage and intersecting-Kikuchi-line (IKL) method. The above methods, based on line scans or two-dimensional intensity distributions in CBED discs, rely on extensive

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