das Interferenzprofil deutlich vom Untergrund ab. Im vorliegenden Fall war der Einstrahlwinkel kleiner als der Grenzwinkel der Totalreflexion, der sich zu $\psi_{tot} \simeq 32'$ berechnet. Die Oberflächenebenheit der Teilungslinien war
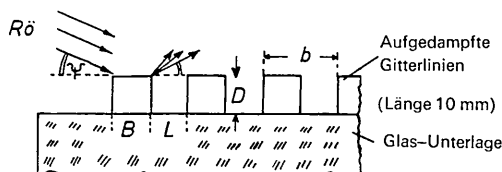

Fig. 1. Aufdampfgitter von Heidenhain: b Gitterkonstante; D Aufdampfdicke einer Teilungslinie, B ihre Breite; L Breite des unbedampften Glasstreifens. $B = L = 4$ μm. ψ ist der Einstrahlwinkel und gleichzeitig, bei regulärer Reflexion, der Austrittswinkel. Polykristalline Struktur innerhalb der Teilungslinien mit (111)-Textur parallel zur Fläche des Glasträgers.
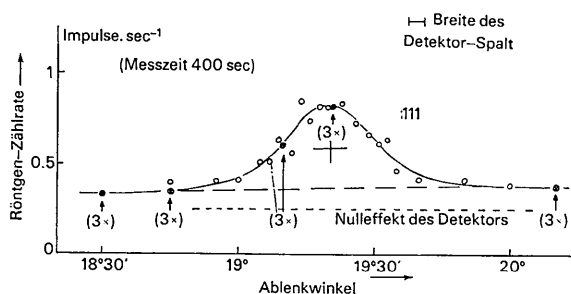

Fig. 2. (111)-Röntgen-Interferenz von den 500 Å dick und 4 μm breit auf Glas aufgedampften Teilungslinien aus Gold (Heidenhain-Gitter), erzeugt durch die unter $\psi = 15' = 0,25°$ streifend zur Gitterfläche einfallende Cu $K\alpha_1$-Strahlung (40 kV, 20 mA). Gitterkonstante 8 μm. Schrittweises Abtasten des Interferenzprofils in der Bragg-Brentano-Anordnung (Weyerer & Meierding, 1972). Szintillationszähler; Weite des Detektorspalts 0,5 mm.

bis auf ±30 Å gewährleistet; Ausbuchtungen ihrer Kanten erreichten gelegentlich 0,1 μm; einzelne Rauhigkeitszacken wurden nicht festgestellt; die Fehler der Gitterteilung überstiegen im Mittel nicht den Betrag von 0,04 μm (Weyerer & Rodemann, 1971).

Wählt man als Einstrahlwinkel $\psi = \theta_{111} = 19,15°$, so wächst die Linienintensität der (111)-Interferenz stark an; die Röntgenrate hängt ausserdem von Kristallitgrösse und Textur im Aufdampfmaterial ab. In einem einzelnen Versuch wurde an einem Goldgitter bei einem Einstrahlwinkel von $\psi = \theta_{111}$ eine 53 mal grössere Intensität der (111)-Interferenz als im Fall der Parallelanregung ($\psi \simeq 0,01$ θ_{111}) erzielt. Beim Überschreiten des Grenzwinkels der Totalreflexion für die Glasunterlage (15' bis 20') oder für das Goldmaterial ($\simeq 32'$) wurden keine schlagartigen Änderungen der Interferenzintensitäten beobachtet. Im übrigen wiesen Filmaufnahmen nach, dass alle Interferenzen des üblichen Röntgendiagramms von Gold auch bei der Parallelanregung in Erscheinung traten.

Die Parallelanregung setzt eine unvermittelte Energieumlenkung voraus, nämlich die Ablenkung aus der Richtung des einfallenden Strahls (bzw. des Oberflächenstromes) in die Richtung des reflektierten Strahles (Interferenzrichtung). Es sei deshalb auf die Beobachtung einer scharfen Umlenkung von Röntgen-Wellenfeldern hingewiesen, bei denen Krümmungsradien bis herab zu 1 μm vorkamen (Bonse, 1963).

Bevor versucht wird, diesen Effekt einer Interferenzanregung an Kristallgittern bei streifender Inzidenz in physikalischer Hinsicht besser verstehen zu lernen, müssen weitere Experimentaluntersuchungen vorgenommen und dabei einige Parameter systematisch variiert werden.

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Estimation of twinning parameter for twins with exactly superimposed reciprocal lattices. By Doyle Britton,[*]

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In twinned crystals the intensity contribution from the separate individuals can be estimated from the observed intensities without any knowledge of the actual structure, even when the two reciprocal lattices are completely superimposed. Once the separate intensity contributions are known the trial structure can be sought in the usual ways.

The following relationships for a twinned crystal are straightforward and well-known (Zalkin, Forrester & Templeton, 1964; Zachariasen & Plettinger, 1965; Grainger, 1969):

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$$I_1 = (1-\alpha)J_1 + \alpha J_2$$
$$I_2 = \alpha J_1 + (1-\alpha)J_2$$

where I_1 and I_2 are the observed intensities produced by the superposition of reflections 1 and 2, which would have intensities J_1 and J_2 in an untwinned crystal of the same total volume. If α is the fraction of the smaller individual

of the twin pair $(\alpha \leq \frac{1}{2})$, then $I_1 > I_2$ implies $J_1 > J_2$ and *vice versa*. The two equations can be solved to give

$$J_1 = \frac{1}{2} \left[I_1 + I_2 + \frac{I_1 - I_2}{1 - 2\alpha} \right] = I_1 + \frac{\alpha}{1 - 2\alpha} (I_1 - I_2) \quad (1a)$$

$$J_2 = \frac{1}{2} \left[I_1 + I_2 - \frac{I_1 - I_2}{1 - 2\alpha} \right] = I_2 - \frac{\alpha}{1 - 2\alpha} (I_1 - I_2) \quad (1b)$$

$$\frac{J_2}{J_1} = \frac{I_2 - \alpha(I_1 + I_2)}{I_1 - \alpha(I_1 + I_2)} \quad (1c)$$

so that J_1 and J_2 can be recovered from the observed intensities if α is known, except when α is too near 0·50, when $I_1 \simeq I_2$ and the corrections are indeterminate. It should be noted that, experimentally, the point at which the corrections become indeterminate is a strong function of the quality of the data.

If the two twin fragments are of quite unequal size $(\alpha \simeq 0)$, the structure can probably be solved without considering the twinning; corrections can then be introduced in the refinement stage if necessary. For examples see Smith & Alexander (1963), who detected twinning but ignored it, Zachariasen & Plettinger (1965), Zalkin, Forrester & Templeton (1964), Grainger & McConnell (1969), and Larsson & Kierkegaard (1969).* For an extensive discussion of refinement with $0 < \alpha \leq 0·50$ see Grainger (1969).

If only some pairs of reflections are superimposed by the twinning, then α can be found directly from measurements on the non-overlapped reflections, after which the overlapped reflections can be corrected. For an example of this see Sabelli, Tangocci & Zanazzi (1969).

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* The equations given by Larsson & Kierkegaard are only approximate and do not correspond exactly to those given above.

If $\alpha = 0·50$, then the structure must be solved from the experimental intensities without correction, which may or may not be possible. For an example of a successful solution of this kind of problem along with a discussion of the calculations see Wei (1969).

We wish to point out that even in cases where two reciprocal lattices are completely superimposed, but α is not 0·50, *i.e.* where intensity differences can be detected in at least some pairs of twin reflections, it is possible to estimate α from the experimental data alone, without making any structural assumptions. Suppose first that there is some pair of twin-related reflections with $J_2/J_1 = 0$. For such a pair it follows from equation (1c) that $\alpha = I_2/(I_1 + I_2)$. For all other pairs of twin-related reflections $J_2/J_1 > 0$ whence $\alpha \leq I_2/(I_1 + I_2)$. Every twin-related pair of reflections thus yields an upper estimate of α and the lowest of these upper estimates is then a reasonable guess for the actual value of α. The success of this method depends only on the condition that for some pairs of reflections $J_2 \ll J_1$ so that $I_2 \simeq \alpha J_1$.†

As an example of the validity of this estimate we show in Fig. 1 some data for diiodoacetylene (Dunitz, Gehrer & Britton, (1972). The number of reflection pairs yielding values of $I_2/(I_1 + I_2)$ in a given range is shown; from this experimental distribution we conclude that $\alpha = 0·25–0·30$. Also shown is the conventional R value as a function of α calculated from structure parameters derived from the uncorrected intensity data ($\alpha = 0$); from this curve we conclude that $\alpha = 0·28$.

As another example, using the data of Larsson & Kierkegaard (1969), we estimate α to be 0·10–0·11 in agreement with their value of 0·11 found by comparing the observed intensities with those calculated from the solved, but not refined, structure.

I would like to thank Professor J. D. Dunitz for helpful discussions.

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† Zalkin, Forrester & Templeton (1964) implicitly recognized this situation, as the following quotation will show $(x \equiv 1 - \alpha)$: 'By trial of various values of x, starting with 0·75 and with refinement with isotropic thermal parameters, we decided that 0·59 gave the optimum agreement. This value of x causes some of the corrected intensities to vanish, and a smaller value gives some corrected intensities which are negative (*i.e.* physically impossible) by amounts which exceed the estimated experimentally uncertainty'. However, they did not call attention to its use in an unsolved structure.
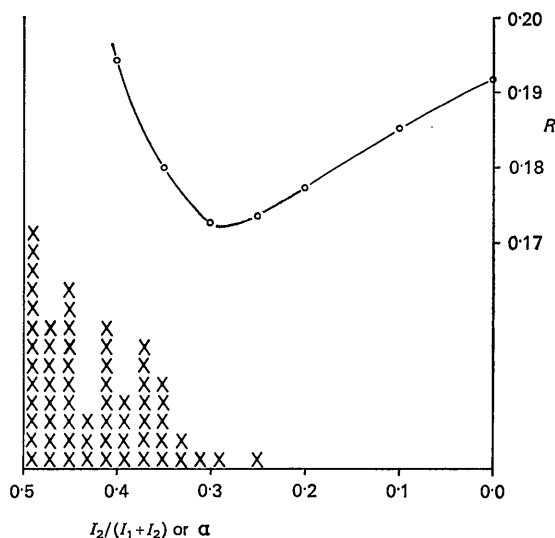

Fig. 1. Above: the conventional R value as a function of the assumed fractional size of the smaller twin fragment. Below: the number of pairs of reflections lying in different ranges of $I_2/(I_1 + I_2)$ *versus* the ratio. The minimum in the upper curve corresponds with the cut-off point in the lower distribution.

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