Table 2. *Experimental and theoretical B_k values for* RbCl *at* 300°K (Å^2)

B_{Rb}		B_{Cl}		
Theoretical	Experimental	Theoretical	Experimental	Reference
2·31	2·28 ± 0·1	2·38	2·44 ± 0·1	Jarvinen & Inkinen (1967)

temperatures in steps of 20°K from 20 to 300°K. In order to check the accuracy of the values we have calculated, comparison is made in Table 2 between the theoretical and the available experimental value of B_k (Jarvinen & Inkinen, 1967) for RbCl at 300°K; the good agreement is highly encouraging. The errors involved and their effect on $\langle u^2\rangle_k$ values (possibly 2%) have been adequately dealt with (Reid & Smith, 1970) and hence no detailed discussion need be made here.

In conclusion, our results are in conformity with the predictions (Scheringer, 1973) namely that for a given fixed number of atoms in the unit cell, the bigger the difference between the masses, the greater the tendency for the heavy (light) atom to have the smaller(larger) amplitude.

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Optimum scanning ratio in neutron diffraction.

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The formula for the optimum scanning ratio, *g* [Werner, S. A. (1972). *Acta Cryst.* A**27**, 665–669] in equatorial-plane, neutron-diffraction experiments has been experimentally verified. This ratio gives the correct coupling between the detector and crystal motions such that the diffracted beam always enters the detector on its centerline.

Introduction

In the course of measuring the Debye–Waller factor of potassium metal at various temperatures, we have encountered difficulties in isolating the elastic Bragg scattering from the thermal diffuse scattering (TDS). Even at liquid-helium temperatures, the contribution of TDS to the integrated intensities of high-index reflections is substantial owing to the large zero-point energy in potassium. From the slope of a plot of $\ln[I_{hkl}\,.\,\sin 2\theta_{hkl}]$ *vs.* $(h^2+k^2+l^2)$ (where I_{hkl} is the integrated intensity, and $2\theta_{hkl}$ is the scattering angle corresponding to the *hkl* reflection) our initial measurements at 7·2 °K made with a large detector aperture gave an equivalent Debye temperature about 10 degrees higher than that obtained from the measured frequency distribution (Cowley, Woods & Dolling, 1966).* We believe this discrepancy is primarily due to the increasing contribution of TDS to the integrated intensities with increasing scattering vector.†

The effects of TDS can be reduced by narrowing the detector aperture. However, it is necessary that the detector accept all of the Bragg-scattered neutrons for every angular setting of the crystal as it is rotated through a given reflection. In order for this to be accomplished the detector and crystal motions must be coupled together in such a way that the centerline of the diffracted beam remains aligned with the centerline of the detector.

A formula for this optimum coupling was given in a paper by one of the present authors (Werner, 1971). Although we have not yet fully resolved the problem of the contribution of TDS to the measurement of the Debye–Waller factor in °K, we have verified that the suggested optimum-scanning prescription is correct.

Under the assumption that the mosaic distributions of both the monochromator (M) and the sample (S) are Gaussian, and the transmission of the collimator between M and S is also Gaussian, the optimum scanning ratio is:

$$g=\frac{\alpha_1^2[1+3a+2a^2]+2\eta_M^2a^2}{\eta_S^2+\alpha_1^2(1+a)^2+a^2\eta_M^2}\,, \tag{1}$$

and the width of the rocking curve (FWHM = 2·36σ) is given by

$$\sigma=[\eta_S^2+\alpha_1^2(1+a)^2+a^2\eta_M^2]^{1/2}. \tag{2}$$

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* The calculated Debye temperatures based on the neutron measurements of the phonon dispersion in K (Cowley *et al.*, 1966) agree with the specific heat capacity measurements (Martin, 1965) to within about 1·5 degrees at 7·2°K where $\Theta_D \simeq 85$°K.

† This effect, of course, is not easily isolated from problems connected with extinction.

Here η_S and η_M are the mosaic spread parameters of the sample and monochromator respectively, α_1 is the Gaussian width parameter for the collimator, and

$$a = \pm \frac{\tan \theta_S}{\tan \theta_M}\,. \tag{3}$$

θ_S and θ_M are the nominal Bragg angles of the sample and monochromator; the '$-$' sign is appropriate for the parallel side and the '$+$' sign for the antiparallel side of the incident beam. The expression for g given by equation (1) is only strictly valid for a point sample since spatial effects can become important for large crystals [see Fig. 6 and the discussion of it by Werner (1972)].

Experimental results

The expression for g requires that if the crystal is rotated by an angle $\Delta\varphi$ in sweeping a Bragg reflection, the detector should be moved by the angle $\Delta\gamma = g\Delta\varphi$, and that this coupling depends on the scattering angle through equation (3). This optimum coupling can be experimentally determined in the following way: the sample is rotated to a Bragg reflection such that the crystal angle is say φ_1. The detector, with a very narrow aperture, is swept through the diffracted beam and the location of the maximum is determined to be say γ_1. The crystal is then rotated off the center of the reflection to an angle φ_2 (where the intensity with the detector wide open has fallen to about $\frac{1}{2}$ of its peak value). The detector with a narrow aperture is then again swept through the diffracted beam, and the location of the maximum is determined to be γ_2. The optimum scanning ratio for this reflection is then

$$g = \frac{\gamma_2 - \gamma_1}{\varphi_2 - \varphi_1} = \frac{\Delta\gamma}{\Delta\varphi}\,. \tag{4}$$

This procedure is then repeated at a series of reflections. The results of such a series of measurements on potassium at 77°K are shown in Fig. 1. The parameters η_S, η_M, and α_1 were determined by fitting equation (2) to the width of the rocking curves (taken with a wide-open detector) for various reflections as shown in Fig. 2. The solid curve shown in Fig. 1 is the result of using these parameters and plotting g *vs.* a as given by equation (1).

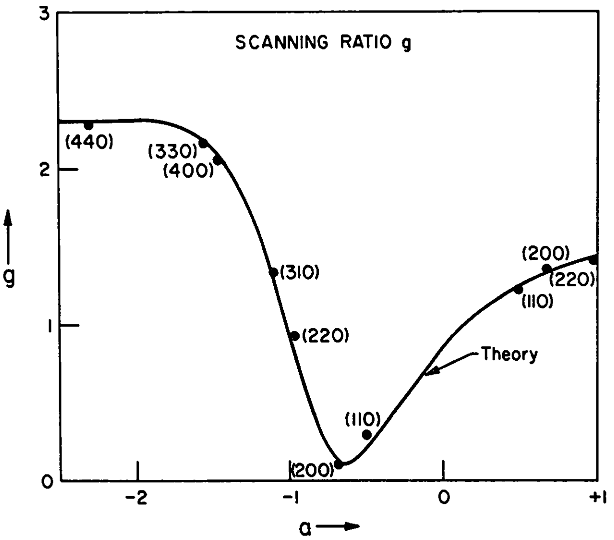

Fig. 1. A comparison of the measured optimum scanning ratio g with the expression given in equation (1) for various reflections in a single crystal of potassium at 77°K. The scattering angle is given by $a = \pm\tan\theta_S/\tan\theta_M$. A copper (200) monochromator was used with $\theta_M = 17{\cdot}4°$.

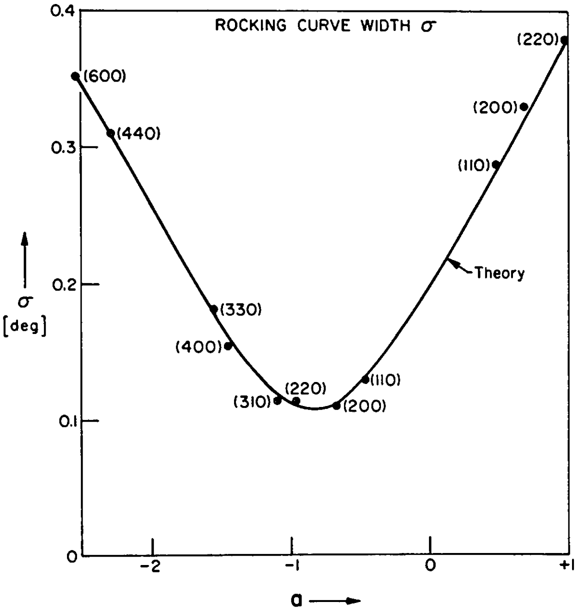

Fig. 2. Result of a least-squares fit of the expression for the width of a rocking curve σ given by equation (2) to the measured widths of various reflections in a single crystal of potassium at 77°K. The parameters obtained from this fitting process and used to calculate the solid curve of Fig. 1 are: $\eta_M = 0{\cdot}0785$ deg., $\eta_S = 0{\cdot}0709$ deg., and $\alpha_1 = 0{\cdot}1833$ deg.

Conclusion

The agreement between the measured scanning ratio g and the calculation are excellent. These measurements were done on a cylindrical sample (0·8 cm diameter with [001] vertical) placed in an incident beam of 1·2 cm width; consequently, the point-sample assumption under which equation (1) was derived does not limit its usefulness. Measurements on other crystals (Fe and Si) have been carried out with results similar to the ones reported here. The prediction that the optimum mode of scanning is seldom an ω scan or a θ–2θ scan is borne out by these results.

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