den Objektbereiche zum grössten Teil überdecken. Dann kann man nämlich entweder mathematisch die zugehörigen Bilder reproduzieren, oder – was vielleicht einfacher ist – verschiedene leicht verschobene Beugungsaufnahmen in den beiden Orientierungen herstellen. Von diesen beiden Aufnahmeserien kann man die paarweise zugehörigen Aufnahmen – die sich durch gleiche Streuintensitäten in Richtung der Schnittgeraden auszeichnen – heraussuchen. Es leuchtet ein, dass normale elektronenmikroskopische Kontrollaufnahmen sehr nützlich sind, um die wenigstens teilweise Überdeckung der vom Primärstrahlwellenfeld beleuchteten Objektbereiche zu kontrollieren.

Beim unperiodischen Objekt stellt sich erneut die schon in (I) diskutierte Frage, ob nicht die direkte Aufnahme mit einem – allerdings zunächst noch hypothetischen – Höchstauflösungsmikroskop der Zusammensetzung des Bildes aus den Beugungsdaten vorzuziehen wäre. Die dort schon diskutierten Argumente – vereinfachte und unempfindliche experimentelle Anordnung wegen Verzicht auf die Zusammenführung der Streustrahlen zum Bild, Möglichkeit der Eliminierung von Störungen durch inkohärente Streuung – bezogen sich im wesentlichen auf die zweidimensionale Abbildung. Aber ähnlich wie in der Röntgenkristallanalyse wird auch bei der Untersuchung komplizierter unperiodischer Objekte eine echte† dreidimensionale Untersuchung der Strukturen nach den in diesem Abschnitt beschriebenen Methoden nicht zu umgehen sein, da sonst Übereinanderprojektionen die Deutung unmöglich machen. Dreidimensionale Elektronenmikroskopie ist nur rechnerisch durch Zusammensetzung aus den Beugungsdaten möglich. Hier haben aber die in dieser Arbeit beschriebenen Verfahren einen grundsätzlichen Vorteil gegenüber der direkten Abbildung in einem Höchstauflösungsmikroskop: Sie liefern unmittelbar die Beugungsdaten, während die von Hoppe (1969) oder de Rosier & Klug (1968) diskutierten elektronenmikroskopischen Verfahren die Daten sekundär über Fourierzerlegung von mikroskopischen Aufnahmen gewinnen. Es leuchtet ein, dass dieser Umweg, welcher alle Fehler des Bildzusammensetz-Prozesses mitwirken lässt, unzweckmässig ist. Es mag paradox erscheinen, dass ein mittelbares Abbildungsverfahren einem unmittelbaren Verfahren überlegen sein soll. Aber man darf nicht vergessen, dass sich die Elektronenmikroskopie im Übergang zur Elektronenstrukturanalyse befindet; das einfache Prinzip der zweidimensionalen optischen Abbildung ist nicht mehr ausreichend.

Diese Arbeit wurde von der Deutschen Forschungsgemeinschaft, dem Fonds der Chemischen Industrie und der Badischen Anilin -& Soda-Fabrik unterstützt. Für diese hochherzige Förderung sprechen wir unseren besten Dank aus.

† Man darf die 'dreidimensionale' Elektronenmikroskopie nicht mit der Stereomikroskopie verwechseln, welche nur zwei Ansichten aus verschiedenen Richtungen liefert.

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Anomalies in Neutron Diffraction Intensities of KH_2PO_4 near the Phase Transition Point

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KH_2PO_4 single crystals show high extinction for Bragg reflexions, as these crystals are nearly perfect. The extinction is, however, reversibly lowered near the phase transition point. The effect was found to be due to an elevated spread in lattice distances.

When a single crystal of KH_2PO_4 is cooled down steadily, the intensities of Bragg reflexions show an excursion as the sample passes the ferroelectric phase transition. [A review article on this material is given by Jona & Shirane (1962).] This effect had already been observed for X-rays by de Quervain (1944). The same effect can also be observed in $BaTiO_3$ (Känzig, 1951) or at the phase transition of V_3Si single crystals at 21 °K, which become superconducting at lower temperatures (Batterman, 1968).

The neutron scattering experiments described in this paper were undertaken to clarify the origin of this effect.

Fig. 1 shows the time dependence of the intensity reflected by a (004) plane. The temperature was varied steadily (about 2°/10 min) and consequently a certain time interval corresponds to a certain interval in temperature. The crystal was aligned for maximal intensity at about 130°K and fixed; the counter covered an angle of 2°, whereas the primary beam had a divergence of 20′. The spectrometer which we used been described by Meister, Haas, May, Schenk & Weckermann (1968).

From several measurements concerning a possible change in the structure factor it could be concluded that a sudden decrease of extinction must be the main reason for the anomaly. This can be brought about either by a temporary increase in the mosaic spread of the crystal, or by some mechanism that introduces a spread in lattice spacings. In order to find out which of these causes predominates, the ratio of the Bragg reflected intensity at its maximum value J_{T_c}, which occurs near the Curie point, to its value at about 7°K above the Curie point J_{para} is calculated and its angular dependence is studied.

Since the mosaic spread β and the spread of lattice spacing $\Delta d/d$ are independent variables, the ratio can be deduced from momentum space as

$$\frac{J_{Tc}}{J_{\text{para}}} = \sqrt{\frac{\left(\frac{\Delta d_{Tc}}{d}\right)^2 + (\beta_{Tc}\,\text{cotg}\,\theta)^2}{\left(\frac{\Delta d_{\text{para}}}{d}\right)^2 + (\beta_{\text{para}}\,\text{cotg}\,\theta)^2}}\,.$$

Further considerations will be restricted to the 00l reflexions. For this case the variables will take the following values:

β_{para} was measured by the double crystal method in the parallel position. The rocking curve of the second crystal was found to have a half width of 6″, in agreement with recent measurements done by Sippel & Eichhorn (1968).

$\frac{\Delta d_{Tc}}{d}$ can take a value up to $5 \cdot 10^{-4}$, if one assumes temperature differences of 2°K in the sample, taking into account the anomaly in thermal expansion measured by several authors (de Quervain, 1944; Ubbelohde & Woodward, 1947; Cook, 1967).

$\frac{\Delta d_{\text{para}}}{d}$ can be assumed to be at least one order of magnitude less than $\Delta d_{Tc}/d$ as the crystals were nearly perfect.

To discuss the dependence of the ratio J_{T_c}/J_{para}, we consider the two cases:

$$(a)\quad \beta_{\text{para}} = \beta_{Tc} \qquad 10\Delta d_{\text{para}} = \Delta d_{Tc}\,,$$
$$(b)\quad 10\beta_{\text{para}} = \beta_{Tc} \qquad \Delta d_{\text{para}} = \Delta d_{Tc}\,,$$

which lead us to curves A and B in Fig. 2. From comparison with our experimental data we conclude that an increase in the spread of lattice distances is the essential source of the intensity excursions.

The assumption that the incoming neutron beam successively hits crystal sections of different temperatures which give rise to different lattice spacings was based on the following observations:

(1) If the cooling was done very slowly (1°/45 min) no excursion could be seen within the accuracy of our method.

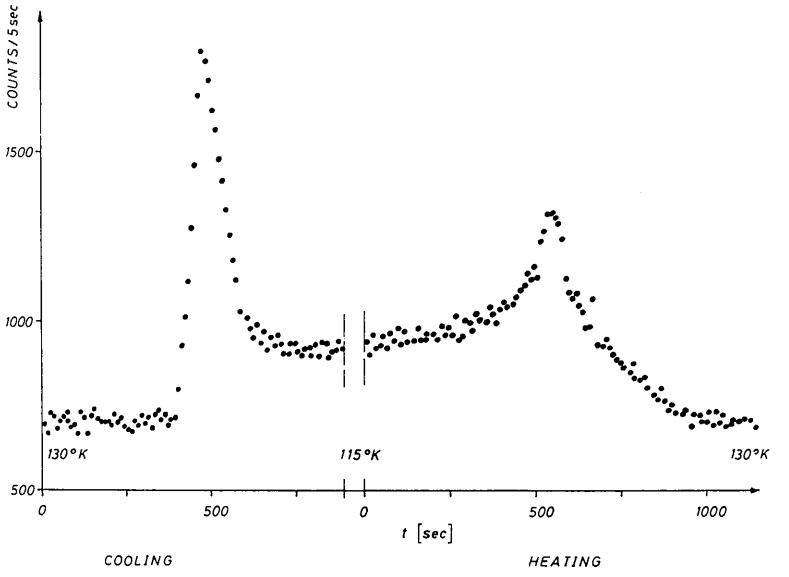

Fig. 1. Time dependence of the 004 Bragg reflected intensity of a KH_2PO_4 single crystal which undergoes a phase transition.

(2) If, by additional heating, a temperature gradient was introduced, perpendicular to the plane formed by the incoming and reflected beam, the excursion also disappeared.

Recent measurements by Umbayashi, Frazer, Shirane & Daniels (1967) which were done with a small sample and an extremely low cooling rate (1 °/200 min) still showed a small excursion. This leaves open the question whether the spread in d is only due to a temperature gradient or partly to some fluctuation mechanism that is associated with the phase transition.

As an application of our results we suggest that the reflectivity of nearly perfect crystals used as neutron monochromators may be increased by introducing large temperature gradients normal to the reflecting planes.

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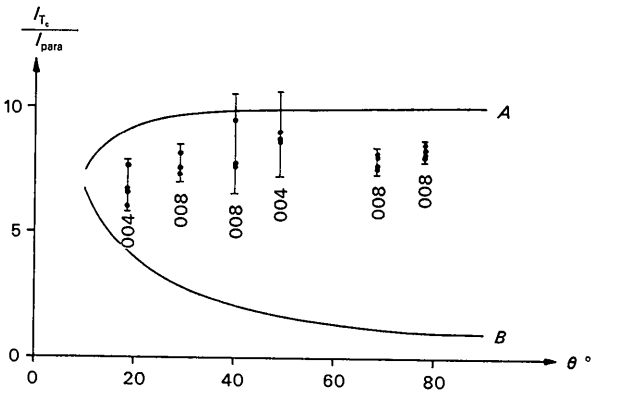

Fig. 2. Ratio J_{Tc}/J_{para} for (004) and (008) reflexions *versus* Bragg angle.

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A Method to Determine the Ratio between Lattice Parameter and Electron Wavelength from Kikuchi Line Intersections

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Three Kikuchi lines not belonging to the same zone, which nearly intersect at the same point, can be utilized for determination of the ratio between lattice parameter and electron wavelength. The method given is analogous to a method based on Kossel line intersections in the X-ray case. One example is given in which the electron wavelength is determined with an uncertainty of *ca.* 0·1%.

Determinations of lattice parameters in selected area electron diffraction necessitate careful calibrations of the camera constant λL, because neither the camera length, L, nor the wavelength, λ, can be determined separately. It is possible, however, to utilize the Kikuchi lines in analogy with the highly efficient use of Kossel lines in the X-ray case. The lattice parameter (a_0) can thus be determined, provided the wavelength is known (or *vice versa*), as shown by Uyeda, Nonoyama & Kogiso (1965).

The purpose of this note is to give an alternative method which enables a determination of the ratio a_0/λ with a relative uncertainty of *ca.* 0·1%. Only two quantities have to be measured on the photographic plate, or a reproduction at any magnification: the height of a triangle formed by three Kikuchi lines and one separation between a deficient-excess line pair.

It is assumed that diffuse scattering from the direct beam involves only negligible energy losses. Only cubic crystals are discussed, although the method can be extended to crystals with lower symmetry, as has been done in the X-ray case by Mackay (1962).

If three indexed Kikuchi lines $\mathbf{H}_i(h_i,k_i,l_i)$ intersect at the same point on the photographic plate, the fol-