The Skew-Symmetric Two-Crystal X-ray Interferometer

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The construction and successful operation of a Laue-case skew-symmetric two-crystal X-ray interferometer is described. The alignment is accomplished with an auxiliary X-ray beam, which is multiply reflected by both interferometer parts. As expected, the skew-symmetric two-crystal interferometer is found to be considerably less affected by vibrations than is the symmetric two-crystal interferometer. [Bonse & te Kaat (1968), Z. Phys. 214, 16–21]. The dependence of the crystal lattice moiré pattern on rotations about the $\Delta\theta$ and $\Delta\phi$ axes are investigated.

1. Introduction

In most applications of X-ray interferometry it is possible to employ a monolithic interferometer (Bonse & Hart, 1965a, b, 1966a, b, 1968; Bonse & Hellkötter, 1969; Bonse & Materlik, 1972), i.e. after its manufacture beam splitter, mirrors and analyser crystal remain connected parts of the same single-crystal block. With interferometers of this kind the necessary stability of the various instrument components with respect to each other is easily maintained.

For the task of achieving a wavelength-independent precision measurement of the interplanar spacing of a perfect crystal the first two-crystal interferometer (Bonse & te Kaat, 1968, 1971) was designed and successfully operated some years ago. It is of the symmetric triple-Laue-case (LLL) type (Fig. 1) with the analyser crystal $A$ mounted separately on a precision leaf-spring traverse.

The required positional sensitivity of the analyser crystal is essentially due to the fact that the two waves bridging the gap between the separated crystals are not parallel to each other.

Quite another stimulus to operate a two-crystal interferometer can arise from the necessity to use path lengths of the interfering beams exceeding the size of presently available perfect crystals. Thus extremely large interferometers can be useful in the case of neutrons, if experiments are intended where the neutrons are to be exposed to extended electric and/or magnetic fields or if particularly large or bulky samples are to be investigated. Contrary to the requirements of the lattice-spacing measurement positional sensitivity of the separated component crystals should be kept small or possibly be avoided altogether. This can be achieved by arranging the gap-bridging beams parallel to each other. One possible realization, the skew-symmetric two-crystal LLL interferometer (Bonse, 1969), is illustrated in Fig. 2. Its construction and successful operation are reported in the present paper. A monolithic version had been tested earlier (Bonse & Hart, 1965c).

Fig. 1. Two-crystal interferometer for precision lattice-parameter measurement.

Fig. 2. Two-crystal interferometer with reduced positional stability requirements.

Fig. 3. Skew-symmetric two-crystal X-ray interferometer; dimensions: 4.5 cm long, 1.5 cm wide, 1.8 cm high.
We also investigated the conditions of focusing and of the positional stability experimentally. We found that by meeting certain geometrical requirements of the set-up the stability may be optimized.

2. Apparatus and alignment procedure

The shape of the interferometer and the outlay of beam paths are illustrated in Fig. 3. The instrument consists of crystal C1 carrying the beam splitter S and the mirror M1, and of crystal C2 carrying the other mirror M2 and the analyser A. As usual C1 and C2 were manufactured from a perfect silicon crystal which contained no dislocations and no oxygen bands.* After cutting with a bronze-bonded diamond cutting wheel the crystals were chemically polished by etching them in a solution of 1 part 48% HF and 19 parts 65% HNO₃ for about 45 min in a rotating beaker, thereby taking off a surface layer of about 120 μm thickness, sufficient to release all elastic strain induced by the cutting process. After etching, the thickness t₁ of the individual crystal was about 500 μm, giving an overall μt ≈ 21 for Cu Kα radiation, which corresponds to the so-called thick-crystal case (μ = linear absorption coefficient).

Parallel alignment of crystals C₁ and C₂ is achieved by fine adjustments about the three rotation axes θ_0, θ₀ and θ₁ as illustrated in Figs. 3 and 4. Axes θ_0 and θ₀ consist of pairs of preloaded ball bearings, rotated via motor-driven spindles s₁ and s₂. θ_0 and θ₀ fine adjustment is performed with the help of piezoceramics P₁ and P₂ acting on the tips of spindles s₁ and s₂ respectively. For θ₁-alignment a leaf-spring rotation axis operated via the motor-driven spindle s₃ is sufficient. Adjustments about θ₀, θ₀ and θ₁ are possible in steps smaller than 10⁻⁴, 10⁻⁴ (1 step = 2·10⁻⁵) and 30° of arc respectively. In order to avoid the propagation of strains from the substrate into the crystals they are three-point supported on small steel balls glued the bases of C₁ and C₂. Sufficient stability is achieved by guiding each two of three steel balls in grooves as indicated in Fig. 4.

First the main 220-reflected interfering beams are prealigned by adjusting Δθ. Next a second (auxiliary) X-ray source is employed in Δθ and Δκ adjustment (Fig. 3). To this end crystals C₁ and C₂ have been shaped so as to provide in the centre a triple-reflexion groove with the upper (lower) wall belonging to crystal C₁ (C₂), respectively. Provided the Δκ misalignment is

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* Waso Material supplied by Wacker-Chemitronics, München, Germany (BRD).
sufficiently small, then the triply reflected auxiliary X-rays ($\Delta \theta$ beam) peak at parallel adjustment about the $\Delta \theta$ axis. With Cu Ka 333 reflexion the $\Delta \theta$ rocking curve is about 2.5'' wide; therefore, even without applying sophisticated peak-finding techniques, $\Delta \theta$ can thus be pre-adjusted to parallelism to roughly 10^{-1}''.

$\Delta \kappa$ alignment is made by first measuring the 333 reflexion with only crystal $C_2$ in place. Then crystal $C_1$ is added in such a position that the auxiliary beam impinges on the upper face of $C_1$ thereby exciting the 333 reflexion of crystal $C_1$ alone. The sideways position of the beam reflected by $C_1$ is compared with that of the beam reflected by $C_2$ and made equal by appropriate $\Delta \kappa$ alignment. If the 333 beams are measured at a distance of about 1 m, $\Delta \kappa$ can be made smaller than 10'' which is sufficient then to thread the beam through the triple-reflexion groove. Once the triply reflected 333 beam has been found, its lateral extension is increased in order to peak it by successively adjusting $\Delta \theta$ and $\Delta \kappa$ thereby improving the $\Delta \kappa$ alignment to better than about 20''. Finally the $\Delta \theta$ fine adjustment is made. The intensities of the main and of the auxiliary beam are then maximized by iteratively readjusting $\Delta \theta$ and $\Delta \theta$.

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Fig. 6. Interferogram of a wedge with its edge horizontal in one beam. Copper 'Ka radiation. X-ray tube operated at 40 kV and 30 mA. Exposure time 15 min on Ilford Industrial G X-ray film. Light areas represent higher intensity, scale mark 4 mm.

Fig. 7. Photographs of a moiré pattern. From photos 1 to 12 crystal $C_1$ was increasingly tilted with respect to crystal $C_2$. $\leftarrow \ (\leftarrow)$ means additional 0.1 (0.2)'' tilt, respectively, scale mark 4 mm.
In order to reduce the background a Si 220 fore crystal is employed between source 1 and the interferometer as shown in Fig. 5. At the same time, by using the fore crystal in asymmetric reflexion with glancing incidence, the field of view is widened to a width of approximately 4 mm. Furthermore, for adjustment with respect to the fore crystal the interferometer as a whole can be rotated about a vertical \( \theta \) axis as indicated in Fig. 4.

Included within a circulating bath thermostat, the interferometer and the fore crystal are kept to within \( \pm 0.1 \)°C of a constant temperature. In order to prevent disturbances by vibrations of the building the whole instrument rests on an antivibration mount. Later it was found that the interferometer – once aligned – could be operated without the antivibration mount.

### 3. Measurements

(a) Structure of fringe patterns

A fringe pattern obtained with a linear beryllium wedge in one of the interfering beams is reproduced in Fig. 6. It proves the proper function of the interferometer. Ideally one should observe straight horizontal fringes with homogenous spacing. The slight bending of the fringes was found to be practically independent of the adjustment state of the interferometer. It is thought to be mainly caused by the rounding-off of the edges of the crystal slabs occurring with the chemical etching. It will be referred to as a ‘built-in’ pattern in the following.

On the other hand, the inclination of the fringes is not a constant of the instrument but varies with \( \Delta \theta \) adjustment as is seen in Fig. 7 where the fringe pattern of the empty interferometer was repeatedly photographed with \( \Delta \theta \) increasing in steps of 0.1 or 0.2° as indicated. Obviously by proper \( \Delta \theta \) adjustment it is possible to see the built-in pattern alone (photo 6 in Fig. 7). It consists of a set of vertical fringes crowding to the left rounded edge of the mirror \( M_{11} \). With \( \Delta \theta \)
values below or above that of photo 6 the fringes ascend or decline respectively.

For a position-insensitive interferometer this behaviour is unexpected. Since at first sight it resembles the $\Delta Q$ dependence of the position-sensitive symmetric two-crystal interferometer with only the analyser crystal cut off (Bonse & te Kaat, 1968, 1971), where, depending on the $\Delta Q$ misalignment of the analyser, rotational moiré fringes with spacing

$$A_R = \frac{d}{\Delta Q}$$

($d$: Bragg plane spacing) are observed, and where $A_R\Delta Q = 1.92$ Å for Si 220.

However, with the skew-symmetric interferometer $A_{Rske} = \Delta Q \approx 132$ Å ± 10% was measured, which is considerably larger and reflects the expected reduction of the rotational sensitivity by a factor of 132 Å/1.92 Å ≈ 69.

The explanation of the $\Delta Q$ dependence is as follows: Suppose that a single ray $g$ from a point $P$ on source 1 is incident on the beam splitter $S$ (Fig 8). Let $g^a$ and $g^b$ be the contributions of $g$ that propagate over the separated path sections II and I into the output beam $O$. If $\Delta Q = 0$ then $g^a$ and $g^b$ coincide within $O$. If $\Delta Q \neq 0$, then $g^a$ and $g^b$, although remaining parallel, no longer coincide but propagate in $O$ with a certain separation $q$.

The same is true for the contributions of $g$ to the output beam $H$. The situation is illustrated by Fig. 8, where a vertical cut parallel to the (220) Bragg planes is shown together with the usual horizontal cut parallel to the basal plane (111) of the interferometer. Obviously $g^a$ and $g^b$ cannot interfere if $\Delta Q \neq 0$ since they do not meet in the same point of the detector, i.e. the same grain of the photographic emulsion. However, there exists a ray $g'$ which propagates over path II and meets with $g^b$ in the same detector point. The essential feature now is that the interference pattern is set up by pairs of rays of type $g'$ and $g$ which on their way to the detector are no longer parallel but form an angle $\delta_k$ with each other in the vertical plane. Consequently $g'$ and $g^b$ set up a system of horizontal fringes, the spacing of which follows from simple laws of wave propagation as

$$A_{Rske} = \frac{\lambda}{\delta_k}.$$  

From elementary geometry of mirror reflexion (1) we find for the angle of $g^a$ and $g^b$ between $M_1$ and $S$

$$\beta = 2\Delta Q \sin \theta.$$  

With the notations given in Fig. 8 we calculate

$$F\delta_k = q = \frac{x}{\cos \theta} \beta = 2x\Delta Q \tan \theta;$$

hence

$$\delta_k = 2 \frac{x}{F} \Delta Q \tan \theta.$$  

Combining (2) and (5) we obtain

$$A_{Rske} \Delta Q = \frac{\lambda F}{2x \tan \theta}. \tag{6}$$

In the experiment $\lambda = 1.54$ Å, $F \simeq 60$ cm, $x = 0.8$ cm, $\theta = 23.7^\circ$. With these values we calculate from (6) $A_{Rske} \Delta Q \simeq 131.3$ Å, which compares very well with the measured value of 132 Å.

(b) Oscillation of fringe patterns

The patterns shown in Fig. 7 not only alter their structure, $i.e.$ the number of fringes seen in the field of
view increases approximately by one with $\Delta g$ increasing by $0.15''$, but also oscillate with varying $\Delta g$ at an even faster rate. Proportional counters, with vertical slits in front about 1 mm wide and 10 mm high, in the beams $O$ and $H$ register the intensity modulation shown in Fig. 9, if $\Delta g$ is varied over a range of ca. 2''. While the number of fringes within the field of view increases by about one, roughly 2.4 oscillations are observed. The exact oscillation period was measured to $\Delta g=6.3 \times 10^{-2}$.

The origin of these faster oscillations is unknown at present. We expect to present an explanation in a forthcoming paper.

(c) $\Delta \theta$ oscillation of fringe patterns

With the same conditions the intensity modulation in Fig. 10 is registered, if now $\Delta \theta$ is varied instead of $\Delta g$ over a range of ca. 0.1''. The $\Delta \theta$ oscillation is caused by a movement analogous to the $\Delta s$ shift normal to reflecting planes known from the symmetric two-crystal interferometer (Bonse, te Kaat & Spieker, 1971) and can be explained as follows:

The mirrors $M_1$ and $M_2$ are rotated about the $\Delta \theta$ axis with radius vectors $a$ and $b$ of different length (Fig. 11). For the movements $u$ and $v$ of $M_1$ and $M_2$ respectively we get

$$u = \Delta \theta \times a \quad (7a)$$
$$v = \Delta \theta \times b. \quad (7b)$$

The movement $\Delta s$ (normal to the net planes) of $A$ relative to $M_1$ is

$$\Delta s = [(\Delta \theta \times b) - (\Delta \theta \times a)] \cdot n$$
$$= [(\Delta \theta \times (b-a)] \cdot n = [n \times \Delta \theta] \cdot (b-a). \quad (8)$$

With $b-a=s+t_{M_1}$ (Fig. 11) we obtain

$$\Delta s = \Delta \theta (x + t_{M_1}). \quad \quad (9)$$

We find the number $n'$ of fringes by dividing with the Bragg plane spacing $d$

$$n' = \frac{\Delta \theta (x + t_{M_1})}{d}. \quad \quad (10)$$

With $d=1.92$ Å and $x+t_{M_1}=0.85$ cm we obtain for $\Delta \theta(x+t_{M_1})=2.26 \times 10^{-8} \approx 4.66 \times 10^{-3''}$ per fringe, which compares very well with the experimental value of $4.55 \times 10^{-3''}$ per fringe.

(d) Fringe contrast

Fringe contrast, usually defined by the visibility

$$v = \frac{I_{max} - I_{min}}{I_{max} + I_{min}} \quad (11)$$

with $I_{max}, I_{min}$ the maximum and minimum intensity in the fringe pattern respectively, was measured with a slit 1 mm wide and high in front of detector $D_1$ (Fig. 5). A value of $v=0.8$ is typical, provided $\Delta \theta$ and $\Delta g$ are aligned properly. $v=0.5$ is observed with deviations of $\Delta \theta \approx \pm 0.13''$ or $\Delta g \approx \pm 0.17''$ from the best alignment position. On photographs the visibility is usually less (around 0.6) because during exposure the fringe pattern drifts slightly.

4. Conclusion

The feasibility of the skew-symmetric two-crystal LLL interferometer has been proved. Its expected lower sensitivity to vibrations was demonstrated. The essential feature of the instrument is that with it the overall extension of an X-ray interferometer is no longer limited by the size of crystals available. Although in the present experiment the separation of the two component crystals was still not large, it follows from this work that completely separated interfering beam paths several cm apart and 1 m or more long can be obtained if needed. Thus an interferometer of the skew-symmetric two-crystal type may become very useful in the case of thermal neutrons if, for any reason, spacious separated beam sections are required.

References