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X-RAY INTERFEROMETRY AND LATTICE PARAMETER INVESTIGATION

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Résumé. — L'essentiel de l'interférométrie des rayons X et ses applications principales sont revus. Des variations très petites de la constante réticulaire, qui se trouvent même dans des cristaux de la meilleure qualité, ont été étudiées à l'aide de topographie ordinaire et celle à franges moirées. Des déformations correspondantes aux variations de $\Delta d/d \approx 10^{-7}$ s'observent dans des grands cristaux parfaits.

Abstract. — The essentials of X-ray interferometry and its principal application fields are reviewed. Very small lattice disturbances which are present even in highest quality crystals of silicon have been investigated by ordinary and moiré topography. Strains corresponding to $\Delta d/d$ variations of 10^{-7} are found in large perfect crystals.

The principles of X-ray interferometry [1, 2, 3, 4, 5] are best and in a most illustrative way explained by means of the moiré model [4] which, although simplifying, describes the essentials of interferometric defect study correctly.

We consider the triple Laue case (LLL) interferometer of figure 1 with the diffracting net planes normal

FIG. 1. — LLL-interferometer, see text.

to the wafer surfaces as indicated. X-rays of some wavelength λ are Laue case diffracted, i. e. the reflected wave H propagates behind the crystal, by the three crystals named wave splitter S, mirror M and wave analyser A. Ideally S, M and A are all perfect crystals which have exact parallel orientation. The X-rays are coherently split by S, conducted over two widely separated beam paths I and II and superimposed in

either outgoing beam O and H by diffraction from A. The outwardly reflected beams behind M are not used and can be eliminated by beam stops if necessary.

We assume $\mu t \gg 1$, where μ is the normal absorption coefficient and t the thickness of the crystal wafers which for ease of discussion we assume to be equal for S, M and A.

The boundary conditions for the electromagnetic field [6, 7, 8] when applied to the Laue case diffraction require the excitation of either kind of the two wave-field modes possible in the crystal: type 1 wavefield with its nodal planes between the planes of lattice atoms and type 2 wavefield with its nodes on the atoms. (If both σ - and π -polarization are considered then there are two modes for each state. For reasons of simplicity we neglect the π -state.)

The type 1 field experiences anomalous absorption and the type 2 field anomalous transmission [9]. With $\mu t \gg 1$ the type 1 field is totally absorbed in the lattice and only the type 2 field emerges from the exit surface of the crystal. Here the boundary conditions require the continuity of the electric induction D inside and outside the crystal so that the nodal planes of the standing wave pattern which is set up by the transmitted O and H wave behind S (P_S in Fig. 1) point necessarily on the atomic planes of S. By means of M the image pattern P_A of P_S in front of A is formed. If the atomic planes of S, M and A are all perfectly « in line » then, for reasons of symmetry, the nodal planes of P_A will also point on the atomic planes of A. Continuity of D requires that in A solely the type 2 field is excited. It traverses A with little attenuation and emits one of its plane wave components into beam O and the other into beam H. Thus both beams have equal intensity which in addition is constant over the cross sections of O and H.

The situation described changes drastically if now A is shifted in a direction normal to the Bragg planes by half a spacing of these planes. The wave pattern P_A enforces the excitation of the type 1 field in A which however is completely absorbed by the atoms of A. Consequently there is zero intensity in beams O and H.

Moreover, if A is continuously traversed along a direction x normal to its Bragg planes, the intensity of O and H is modulated proportional to $\cos^2 \pi x/d$. Hence from the number of intensity modulations we can directly count the number N of Bragg plane passages occurring over a given total shift x_0 . Thus we find $d = x_0/N$ the Bragg plane spacing. The great importance of this method lies in the fact that contrary to conventional lattice parameter determinations no other number enters the measurement of d [3]. The method is being used therefore for the precision measurement of the absolute value of the lattice parameter of a perfect crystal [10, 11, 12].

x_0 is measured either with a multiple beam interferometer of the Fabry-Perot type with extra optical mirrors [13, 14] or with a two-beam interferometer and silicon mirrors polished directly onto the interferometer crystal [15]. First results with the aim of a new determination of the Avogadro constant have been reported by Deslattes *et al.* [16].

It is very informative and also convenient to look at the interaction of the wave pattern P_A with A as of the superposition of two grids with spacings d_p and d_A which set up a moiré pattern. As is well-known, if

$$\Delta d := d_A - d_p \neq 0,$$

a dilatational moiré pattern with fringes parallel to the net planes and with a spacing

$$A_D = \bar{d}/(\Delta d/\bar{d})^{-1}$$

with

$$\Delta \bar{d} := (d_A + d_p)/2$$

is formed.

Similarly, if $d_A = d_p$ and the grids are rotated with respect to each other through an angle φ , a rotational moiré pattern with fringes orthogonal to a line bisecting and spacing $A_R = d\varphi^{-1}$ is formed. With both $\Delta d \neq 0$ and $\varphi \neq 0$ inclined fringes are obtained and the values of Δd and φ can be determined from the fringe periodicities measured along a direction normal and parallel to the net planes respectively.

Traversing the mirror M instead of the analyser A causes the same intensity modulations of the beams O and H except that a shift of M by an amount x is equivalent to a shift of A by $-2x$. Similarly, in the generation of moiré patterns the sensitivity is doubled if M is moved instead of A. Relative movements of S are equivalent to equal movements of A.

It is obvious from the extreme positional sensitivity of the interferometer that very good mechanical and thermal stability is a necessary working condition. The

easiest way to guaranty stability is to use monolithic interferometers. However, in some applications the positional responsivity is a goal in itself and in these cases the outermost of thermal and vibrational shielding is needed.

Examples. — Rotational moiré fringes obtained with X-rays are shown in figure 2. In the experiment a

FIG. 2. — Rotational moiré fringes obtained with X-rays. Tilt angle $\varphi = 0.085^\circ$ (a) ; 0.15° (b) ; 0.26° (c) ; 0.36° (d).

LLL interferometer as illustrated in figure 3 was used. Three saw cuts as indicated isolated the analyser from the rest of the interferometer so that the only connecting link was a small support about 5 mm by 0.2 mm

FIG. 3. — LLL-interferometer for the observation of moiré fringes of figure 2. The analyser A may be rotated about the ρ -axis by elastic deformation of the small supporting ridge.

cross section which could be elastically deformed in order to rotate the analyser by a small couple. The rotation angle varied between 0.085 and 0.36 s of arc. As is seen from figure 2 it is not difficult to arrange patterns with quite small fringe spacings and yet very good contrast. For phase shift measurements performed to the end of scattering factor determinations [17, 18, 19, 20, 21] rotational moiré fringes are favourably used

since fringes produced with a wedge in one of the interfering beams usually suffer a loss in contrast because of absorption in the wedge.

If the analyser A (or any of the other interferometer components) contains defects such as dislocations, stacking faults, twin boundaries or variations of the d-spacing, a characteristic moiré pattern is observed [4, 22, 23, 24]. Irradiation damage was investigated by Gerward, Christiansen and Lindegaard Andersen [25, 26].

In order to obtain a large field moiré topograph the imaging techniques known from conventional X-ray

topography like Lang scanning [27] or double crystal setting with asymmetric fore crystal [28, 29] may be applied.

As an example which demonstrates nicely the extreme sensitivity of the method we discuss the investigation of Δd -variations in large dislocation free silicon crystals of highest quality. Figure 4 shows the moiré pattern of an empty interferometer cut from dislocation free medium quality silicon. The interferometer is about 3.5 cm long and 3.0 cm wide with 111 wafer surfaces. The Bragg planes used are $\bar{2}20$ planes parallel to the growth direction [111]. The fringe pattern seen is mainly caused by Δd changes arising from small differences of impurity incorporation due to periodic temperature fluctuations occurring correlated with the crystal rotation in the growing procedure. The $\Delta d/d$ changes are of the order of 10^{-6} . The bending of the fringes in the upper left corner of the topograph is probably due to some stress concentration not completely etched out after the mechanical cutting of the interferometer.

With increasing dimensions of the interferometer the Δd fluctuations show up more seriously. Therefore, if (as it is the case with neutron interferometry [30, 31, 32]) crystals some 8 cm long and 6 cm wide are used, a sensitive sample selecting procedure is desirable.

From the bulk crystal of about 8 cm diameter a slice

FIG. 4. — Moiré pattern of empty interferometer mainly due to lattice parameter fluctuations.

FIG. 5. — Silicon sample interferometer cut from slice 1 cm thick and 8 cm diameter.

FIG. 6. — Section moiré topograph of interferometer of figure 5. Note irregular pattern and the bending of the originally straight vertical section lines.

FIG. 7. — As figure 6 but other region of the topograph.

is cut roughly 1 cm thick. By grinding two grooves into it the sample interferometer imaged in figure 5 is made. Two regions of double crystal moiré topographs are shown in figures 6 and 7. (The topographs have been taken in vertical sections about 0.5 mm wide and the regularly arranged vertical lines are due to this kind of sectioning technique.) From the fringe structure we see that the crystal has a rather irregular lattice disturbance and conclude that it is most likely unsuited for quantitative interferometry with a large interferometer.

The Δd disturbance can also be seen on ordinary high-sensitivity double crystal reflection topographs. An example is shown in figure 8 which was taken with the silicon $\bar{6}60$ reflection and MoK_α radiation. It was found that the striations seen on the topograph are correlated with the growth striations observed on the cylindrical surface of the as-grown crystal.

It is worth pointing out that further information about the nature of the deformation may be drawn from the very obvious bending of the originally straight sectioning lines on figures 6 and 7, which is in accor-

FIG. 8. — Topograph of a silicon crystal, obtained with a double crystal arrangement, $\bar{6}60$ -reflection, MoK_α radiation.

dance with the moiré fringe structure. We believe that the bending is caused by the influence of the Δd fluctuations on the directions of the local energy flow within the Borrmann fans of the crystal wafers.

Summarising we may say that X-ray interferometry is a method very suitable for the detection of small and extremely small lattice disturbances in almost perfect crystals. As such it may also be applied for instance to the investigation of irradiation damage at low doses and of strains exerted by thin films on the substrate they have been deposited on [33, 34]. Other applications of crystal diffraction interferometry are possibly phase contrast micrography [35] and Fourier transform spectroscopy in the angström range. For further aspects see also the reviews on this subject [34, 36, 37, 38].

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