

HIGH RESOLUTION TECHNIQUE AND INSTRUMENT FOR MEASURING LATTICE PARAMETERS IN SINGLE CRYSTALS

BACKGROUND OF THE INVENTION

1. Field Of The Invention

This invention relates to an improved double-diffractometer for the precise measurement of lattice parameters in single crystal wafers, and more particularly to a technique for eliminating tilt misalignment between two independent single crystals where one crystal is misoriented in an arbitrary direction with respect to the other crystal, prior to comparing their diffraction angles.

2. Background Description

The precise measurement of lattice parameters in single crystals is important in at least two areas of research. In electronic applications, this information is used in fabricating epitaxial thin films of $\text{Ga}_{1-x}\text{Al}_x\text{As}$ on GaAs as well as $\text{Ga}_{1-x}\text{Al}_x\text{As}$ on $\text{Ga}_{1-7}\text{In}_4\text{As}$, and $\text{In}_x\text{Ga}_{1-x}\text{As}$ on InP . These formations require exact knowledge of the substrate lattice parameter, since often a comparison with the substrate yields an accurate measure of the epilayer lattice parameter and hence a measure of the alloying content. The control of alloying content is essential in preparing lattice-matched layers and thin films useful for proper operation of microelectronic devices. In materials science, on the other hand, information on lattice parameters is used for investigating the mechanical and optical properties of materials and their variation as an indicator of the defect structure and strain. A simple measurement method, sufficiently sensitive to small variation in the lattice parameter would therefore be desirable.

During the last three decades several techniques using x-ray diffraction have been introduced which provide accuracies varying from one one-hundredth of an angstrom (0.01\AA) to 0.00001\AA . Most recent applications of such measurements have required the upper levels of resolution, i.e., better than $.001\text{\AA}$, where the effects of strain and doping can be quantified as a function of minute changes in the lattice parameter. The most well known of these is the Bond method, as described in *Acta Cryst.*, 13 (1960) 814, by W. L. Bond, which permits absolute measurement of diffraction angles from which the lattice parameter may be calculated. This technique, while in principle is unsurpassed for its accuracy has nevertheless several drawbacks, one of which is the complexity and the bulk of the instrumentation which relies on a very accurate and therefore expensive theta circle for positioning the crystal. The second is the relatively long measurement time and the difficulties associated with automation which reduce this time to only about 30 minutes per measurement. The third drawback is that to attain the part-per-million accuracy the samples must be perfect over a relatively wide area. Although this last condition is necessary for all high resolution measurements, it is not a condition which can be satisfied for most crystals of interest under development today. Thus, the state of perfection of most present-day crystals, except for Si and Ge, is such that the resolution required for their evaluation need not exceed a few ten parts per million. Rather, changes in the lattice constant corresponding to the fourth decimal place ($\sim 2 \times 10^{-4}\text{\AA}$) are sought. Such variations correspond to accuracies of the order of a second of arc in the Bragg angle θ . As a result, the Bond method remains an "umpire" method with princi-

pal applications in studies involving a limited number of crystals such as Si and Ge.

In addition to the Bond method, several other techniques have been introduced, all of which require specialized instrumentation without wide applicability to different materials. For example, multiple detectors, multiple sources, or specifically shaped reference crystals are used, all of which limit the application to the measurement of the variation of the lattice parameter rather than the actual parameter itself.

FIG. 1 is a perspective view of the basic structure of a typical double crystal diffractometer, such as Blake's, having two crystal stages 10 and 12. The first crystal 10 and the second crystal 12 are the main components and each of them is very accurately aligned. X-rays come from an x-ray source 14 and become partially collimated by slits S_1 and S_2 and strike the first crystal 10. Slit S_2 is positioned between the second crystal 12 and a detector 15. Slit S_2 is sometimes used in place of slit S_2 . Both slits simply limit the beam so that it doesn't scatter. However, it is preferred to have both slit S_1 and slit S_2 near the x-ray source so that right away the total radiation is reduced to reduce the hazard of exposure. The crystals 10 and 12 have tilts δ_1 and δ_2 respectively between their normals n_1 and n_2 and a the horizontal plane, and the slant angle between the line connecting the slits S_1 and S_2 and the horizontal plane which create errors in the measurement of lattice parameters. Here the first crystal 10 is adjusted so that it can pick out diffraction angles near the characteristic radiation of the x-ray source 14—and then a characteristic radiation is reflected toward the second crystal 12. The second crystal 12 is the one which would ordinarily be in a usual double crystal diffractometer being tested. However in the method proposed below in the preferred embodiment, two independent crystals take the place of the second crystal 12, i.e. a reference and an unknown crystal—both mounted in the same general area. One is translated into the x-ray beam and then the other one is translated into the beam while looking at their different diffraction angles. And by measuring the difference in diffraction angles of these two independent crystals one can calculate, by Bragg's law, the lattice parameter of the unknown crystal.

In what follows, we will first review the method as applied to a system of two independent crystals each having only a negligible component of vertical tilt. The effects of second crystal tilt and other geometrical constraints are then discussed.

Consider two crystals, C1 and C2 (unknown and standard), mounted together on the second stage of a double crystal diffractometer as shown in FIGS. 2A and 2B. The diffracting planes are assumed to be vertical, i.e., their normals, having a small misorientation angle α between them, are directed in the horizontal plane. Monochromatic X-rays arriving from the first crystal are diffracted sequentially from crystal C1 and crystal C2 by rotating the second stage. FIGS. 2A and 2B is a sketch of such an experiment in which, for the sake of simplicity, the X-ray beam—rather than the stage—is rotated. The separation between the two X-ray beams is an angle X containing both the difference in the Bragg angles $\Theta_{B1} - \Theta_{B2} = \Theta_B$, and a misorientation angle α . In FIG. 2A, crystal C1 is shown closer to the X-ray source than crystal C2. The relationship between X , α , and Θ is thus