Extended Abstracts of the 19th Conference on Solid State Devices and Materials, Tokyo, 1987, pp. 119-122

Absolute Lattice Parameter Measurement of GaAs Crystals Using Monochromatic Synchrotron Radiation

K.Usuda, S.Yasuami, Y.Higashi*, H.Kawata* and M.Ando*

Research and Development Center, Toshiba Corporation
1, Komukai Toshiba-cho Saiwai-ku, Kawasaki 210, Japan

*Photon Factory, National Laboratory for High Energy Physics
Ohomachi, Tsukubagun, Ibaraki 305, Japan

A high resolution diffractometer with a monolithic Si monochromator was developed for absolute lattice parameter measurements using synchrotron radiation. Accuracy in measurement of the lattice parameter, better than $\Delta d/d < 4 \times 10^{-6}$ was attained. This degree of accuracy enables us to discuss composition dependence of the lattice parameters for GaAs crystals.

1. Introduction

III-V compound crystals, in particular GaAs, are attractive for the fabrication of high speed integrated circuits (IC), because of high mobility and low power dissipation. Contrary to these advantages, it is difficult to obtain single crystals with uniform properties. Crystal characteristics vary with composition shift from stoichiometry, and with defects and impurities induced during crystal growth. There are several reports regrarding correlations of the lattice parameter with stoichiometry [1-4], and with threshold voltages: (Vth) of field effect transistors (FET) [5]. However these results are not thoroughly consistent.

Since the variation in the lattice parameter ($\Delta d/d$) seems to be of 10^{-5} order, an accuracy of 10^{-6} order is required for the measurement. To date, the lattice parameter of GaAs has been measured by Bond's method [5]. Its accuracy is limited by the angular divergence ($\sim 10^{-3}$ rad) and wavelength dispersion ($\Delta\lambda / \lambda \sim 10^{-4}$ order). To achieve further improvement, the (+,+) arrangement is required for the monochromater crystals.

One of the difficulties met in this set-up using a conventional X-ray source is a decrease in the X-ray intensity. The other is limitation in available wavelengths. We thus developed a precise diffractometer using monochromated synchrotron radiation (SR) at the National Laboratory for High Energy Physics (KEK).

2. Experimental

2.1 Diffractometer

Figure 1 shows a plan view of the diffractometer set-up for this study. X-ray beams run through in the horizontal plane. The first (111) channel-cut Si crystal is used for roughly selecting an energy range including the energy finally extracted for the lattice parameter measurement. The 2nd crystal is a monolithic monochromator, which has two reflection planes composing (+,+) setting. In this experiment, a combiantion of the 335 and $\bar{5}3\bar{5}$ reflections was selected. An X-ray beam with λ centered on 1.3536 Å wavelength impinges on the thrid crystal (sample). This combination of reflection planes was selected for the 800 reflection of

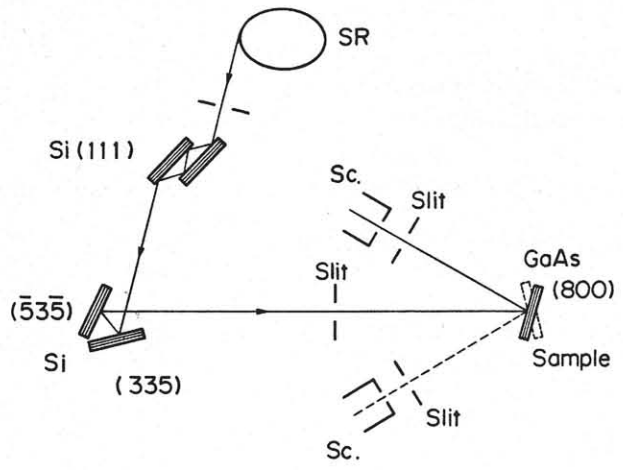


Fig.1 Experimental arrangement for lattice parameter measurement.

GaAs samples and 444 reflection of a Si reference crystal to be detected for one wavelength. The reference Si crystal was used to calibrate the X-ray wavelength, regarding its lattice parameter as being d_{100}= 5.4310652 Å, as derived by Desllates et al [7]. The resultant angular divergence and wavelength dispersion were $\Delta\theta < 7\times10^{-6}$ rad, and $\Delta\lambda < 3\times10^{-6}$ Å, respectively, and hence the lattice parameter was determined with an accuracy better than $\Delta d/d \sim 4\times10^{-6}$,

Each crystal was mounted on a goniometer, whose minimum angular step is 1/10000 degree of arc. The angular positions of these goniometers were monitored by rotary encoders which read 1/10000 degree of arc for the second and third goniometers, and 1/100 for the first one. This diffractometer was in a room where temperature fluctuations were kept within ±0.1℃ during measurement for one sample.

The entire measuring procedures were monitored with a computer, which drives pulse motors for the goniometers, counts X-ray intensities and saves data in memory. After all measurements were finished, the peak positions for rocking curves were obtained through curve fitting procedures, assuming the Cauchy distribution as a theoretical rocking curve. Corrections for the Lorentz-polarization and refraction effects were made afterwards. Finally, all lattice parameters thus obtain were normalized to their values at 298 K, using a linear thermal expansion coefficient α of $5.8\times10^{-6}(K^{-1})$.

Each topograph was taken at one of peak positions of respective rocking curves used for the lattice parameter determination.

2.2 Samples

Samples were prepared from undoped GaAs crystals with varied compositions, In-doped and the so called As-injection crystals. They were grown in the [100] direction by the MLEC or LEC methods. (100) oriented wafers were obtained either perpendicular or parallel to the pulling direction. Diced samples measured 7.5 by 7.5 mm.

3. Results and Discussion

Figure 2(a) shows variations in the lattice parameter across an undoped, 3 inch diameter MLEC crystal wafer. The lattice parameter is larger in the central part than in peripheral regions. After this wafer was diced into small pieces, the lattice parameters obtained from the same points as before agree within a fluctuation of 3×10^{-5} Å,

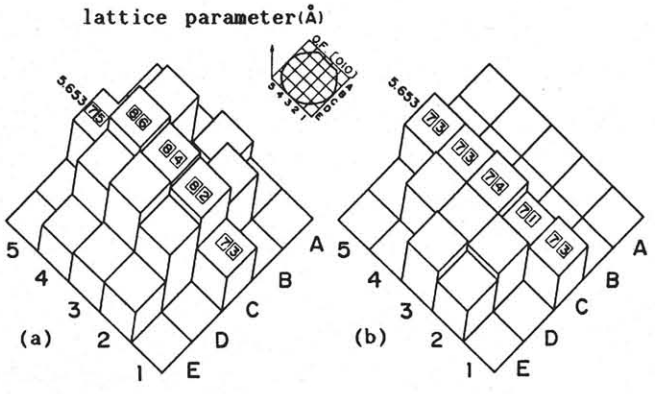
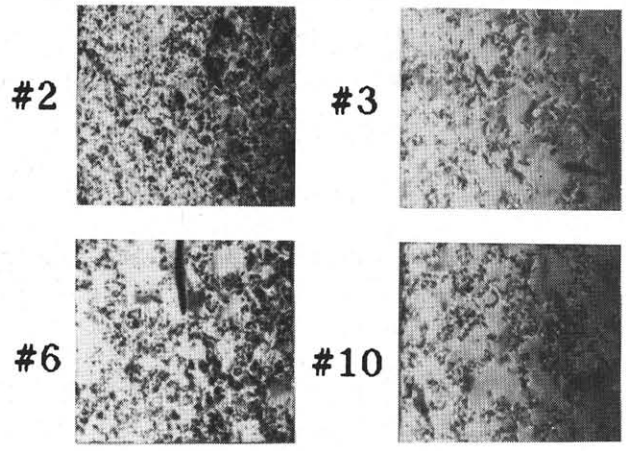


Fig.2 Lattice parameter variation across an undoped 3 inch diameter MLEC wafer. (a) shows data obtained for the wafer before dicing and (b) for the diced wafer.

as shown in Fig. 2(b). This fact implies that the stress, induced while the crystal was grown, and remaining in the wafer, was relaxed by dicing.

Figure 3 shows variations in the lattice parameter along the pulling direction of an undoped GaAs crystal, grown from nearly stoichiometric melts using As-injection technique. The open and closed circles represent data for a wafer and for chips after separating, respectively. Except that the lattice parameter increases at the initial stage of the crystal growth, it decreases toward the tail of the crystal. When this wafer was separated into chips, the lattice parameter value lowered as a whole.



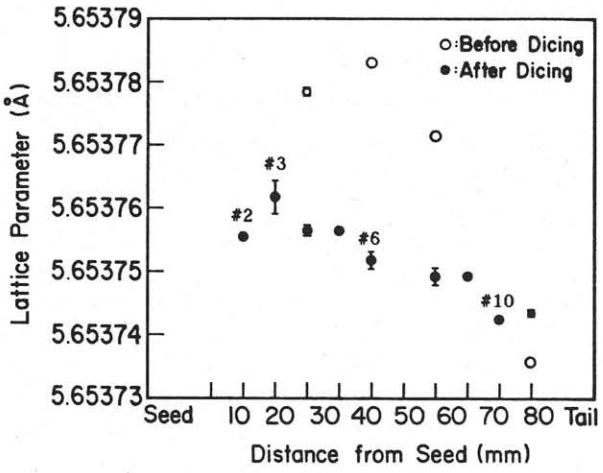


Fig.3 Lattice parameter variation along the growth axis of an As-injection wafer. Open circles for the wafer before dicing ; closed circles for the diced wafer after dicing. Each topograph was taken at one of peaks of respective rocking curves.

From Figs.2 and 3, it was revealed that stress induced during the crystal growth remains after fabricated in wafers. This strain is appreciably relaxed by dicing wafers into small pieces. Accordingly, the data in Figs. 4 and 5 were taken only for small samples to eliminate this strain effect.

The variation in the lattice parameter still left, is considered due to composition, because the As fraction in the crystal is believed to become lower as the crystal grows, in this case. However, a high density of dislocations might have raised the lattice parameter values for #3, and #6.

Figure 4 shows the lattice parameter variation along the pulling direction of an In-doped crystal grown by the LEC technique. The lattice parameter values are larger than those for the undoped crystal shown in Fig.2(a). The linear increase of the lattice parameter towards the tail corresponds to increasing In segregation into the crystal as the growth proceeds.

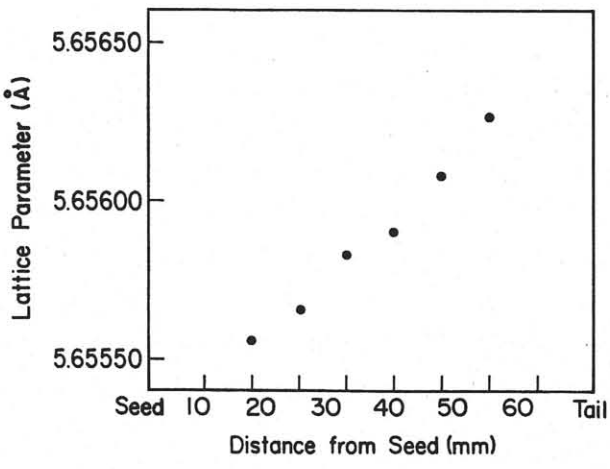
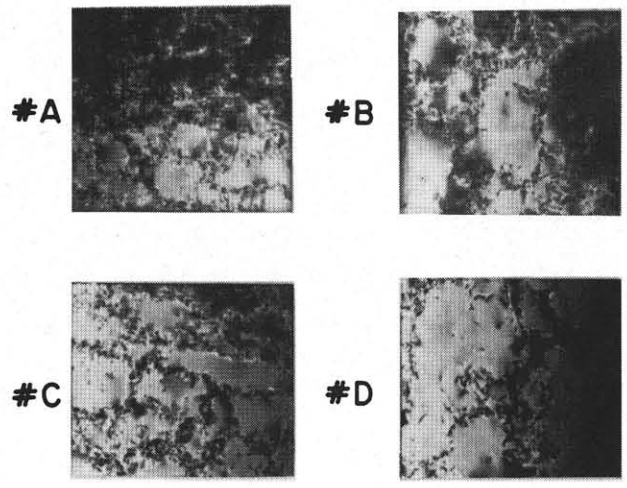


Fig.4 Lattice parameter variation along the growth axis of an In-doped wafer.



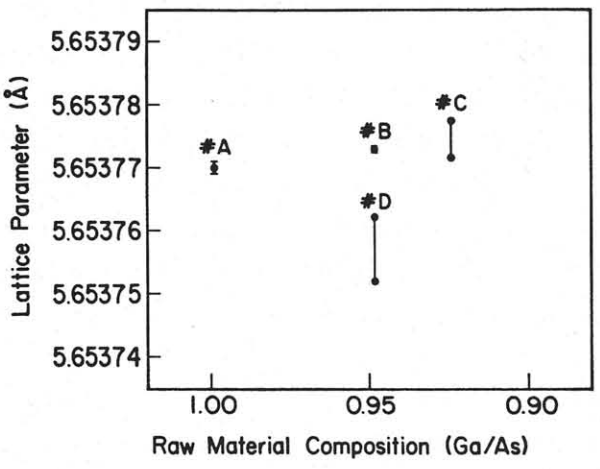


Fig. 5 Lattice parameter variation obtained for crystals growth from varied raw composition. Each topograph was taken at one of peaks of respective rocking curves.

In Fig. 5, we compare the lattice parameters obtained for crystals with varied raw material compositions. All samples were obtained from undoped, 3 inch diameter crystals grown by the MLEC method. The same field was applied to samples A,B and C, but not to sample D. For crystals grown under the same magnetic field, the lattice parameter seems to increase as the As fraction increases. On the other hand, the lattice parameter difference among the crystals with the same mateiral composition (B and D) is larger than that among the sample A, B, and C. Incidentally, the topographs for the sample B and D show that the sample B contains a higher density of dislocations than the sample D.

We hence conclude that it is too early to extract meaningful results on the effects of composition on the lattice parameter, unless strain effects are completely eliminated.

Acknowledgements

The authors would like to thank Drs. K.Ozeki, A.Hojo and T.Nakanisi for their encouragement during this study.

They also thank Dr.K.Terashima and Mr. T.Fujii for supplying the samples.

References

1) Y.Okada et al.,Appl.Phys.Lett.48,(1986)975
2) M.Nakajima etal.,Appl.Phys.Lett.49,(1986) 1253
3) K.Terashima et al.,4th Conference on Semi Insulating Materials
4) H.Kuwamoto et al.,J.Appl.Phys.59,(1986)656
5) Y.Takano et al.,Jap.J.Appl.Phys.24,(1985) L239
6) W.L.Bond,Acta.Crystallogr.13,(1960) 814
7) R.D.Deslattes et al.,Phys.Rev.Lett, 31, (1972) 16