Characterization of InGaAsP surface corrugation used for distributed feedback lasers by means of Raman spectroscopy

Minoru Kubo, Keisuke Koga, and Mototsugu Ogura
Semiconductor Research Center, Matsushita Electric Industrial Company, Ltd., 3-15 Yagumo-nakamachi, Moriguchi-shi, Osaka 570, Japan
Shigemi Kohiki
Matsushita Technoresearch, Inc., 3-15 Yagumo-Nakamachi, Moriguchi-shi, Osaka 570, Japan

(Received 17 March 1986; accepted for publication 16 June 1986)

Thermally deformed surface corrugations on both an InP substrate and an InGaAsP layer have been analyzed by means of x-ray photoelectron spectroscopy and laser Raman spectroscopy. From the spectra of the deformed surface corrugations on an InP substrate on which a GaAs wafer was placed during its heat treatment, it has been found that material formed in the grooves is an InGaAsP alloy single crystal.

InGaAsP/InP distributed feedback (DFB) lasers are widely used in optical communication systems because of their finely controlled and highly stable single longitudinal mode operation.[1,2]

Many authors have reported that, when these DFB laser structures are grown in a liquid phase epitaxy (LPE)[3–6] furnace, thermal deformation of the surface corrugations on an InP substrate or on an InGaAsP layer occurred during the soaking period just before the LPE growth. Recent studies have proposed various means for suppression: to cover an InP substrate with a GaAs wafer instead of an InP wafer,[3,6] to introduce PH_3 gas into a H_2-filled ambient,[4,5] or to employ a (Sn + InP) saturation chamber.[7,8]

In this letter, we present for the first time the characterization of the thermal deformation of surface corrugations on both an InP substrate and an InGaAsP ($\lambda_g = 1.1\,\mu$m) layer using x-ray photoelectron spectroscopy (XPS) and laser Raman spectroscopy. The surface corrugations are fabricated in uniform within a depth of 800–1200 Å at a pitch of 3950 Å. They are formed on both a (100) Sn-doped InP substrate and an InGaAsP ($\lambda_g = 1.1\,\mu$m) LPE-grown epilayer in holographic photolithography technology, followed by wet chemical etching. The LPE growth and heat treatment were carried out in a conventional LPE furnace. To avoid thermal deformation of the surface corrugations, we adopted two methods: placing a GaAs wafer on the sample to cover the surface corrugations, and placing whole samples in a (Sn + InP) saturation chamber. After heat treatment the deformation of the surface corrugations was analyzed by XPS and/or laser Raman spectroscopy.

Figure 1 shows the scanning electron microscope (SEM) view of the stained-etched epilayer on which the surface corrugations were formed. This sample was covered with a GaAs wafer during the soaking period of LPE growth. It has been found that on the surface corrugations etching and deposition simultaneously appear at the convex and the concave, respectively. This phenomenon can be compared to the deformation of the *V* groove, i.e., etching at the shoulder and deposition at the bottom after heat treatment in an ambient of H_2 and PH_3.[9] Tanahashi *et al.*[10] have reported the possibility of surface migration of indium (In) and evaporation condensation of phosphorus (P) in the deformation process of *V* grooves on the surface of an InP substrate, and have concluded from energy dispersive x-ray spectroscopy (EDX) analysis that the material in the grooves is probably an InP crystal. Similarly, the experiment leads us to assume that both arsenic (As) and P atoms probably vaporize, and that In and gallium (Ga) atoms can transport by migration.

The surface corrugations, which were covered with a GaAs wafer, show deformation with the heat treatment at 600 °C for 60 min. We have analyzed the deformation by XPS. Figure 2 shows both Ga $2p_{3/2}$ and As $2p_{3/2}$ spectra at a depth of 10 Å from the surface and Fig. 3 In 4*d*, Ga 3*d*, and As 3*d* spectra at a depth of 20 Å from the surface. Within the depth of 10–20 Å, Ga atoms are transported from the GaAs wafer to the concave of the surface corrugations on an InP substrate. As $2p_{3/2}$ and As 3*d* signals also arise at 10 and 20 Å below the surface, respectively. At 10 Å in depth, the intensities of As_2O_3 and As_2O_5 signals are much larger than those from GaAs or As. At 20 Å in depth, the As_2O_3 and As_2O_5 signals decrease and the main signal comes from GaAs and As. It has been found that the material in the grooves of the thermally deformed surface corrugations, when a GaAs wafer was placed during heat treatment, is composed of Ga, As, and In atoms. From these results, however, we cannot conclude whether this material corresponds to a binary, ternary, or quaternary alloy and whether it is made of a single crystal or not.

Raman spectra of variously heat-treated surface corrugations on both an InP substrate and an InGaAsP ($\lambda_g = 1.1\,\mu$m) layer are shown in Figs. 4 and 5, respectively. Spectra

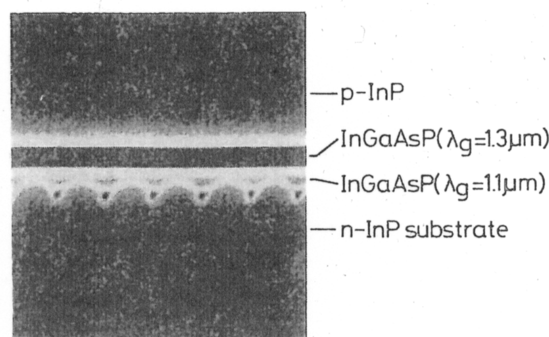

FIG. 1. Cross-sectional view of a stained-etched epilayer structure on an InP substrate whose surface corrugations are covered with a GaAs wafer.

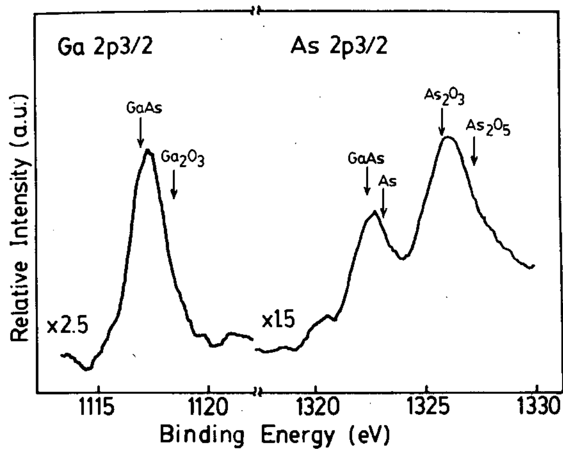

FIG. 2. XPS spectra of Ga $2p_{3/2}$ and As $2p_{3/2}$ for heat-treated surface corrugations on an InP substrate covered with a GaAs wafer.

(a) and (b) in Fig. 4 show relative intensity of a Sn-doped InP substrate and of surface corrugations fabricated on an InP substrate, respectively. Spectrum (c) in the same figure represents relative intensity with thermally treated surface corrugations on an InP substrate in a (Sn + InP) saturation chamber, and the spectrum (d) with the corrugations treated with a GaAs wafer covered. Peak *A* for an InP substrate is related to longitudinal optical (LO) phonon in InP and peak *B*, to coupled plasmon LO phonon (L^-),[11] because the carrier concentration in a Sn-doped InP substrate is $\sim 2\times 10^{18}$ cm^{-3}. The peak of spectrum (b) in Fig. 4, corresponding to peak *A* of spectrum (a), becomes smaller than peak *C* after the fabrication of surface corrugations on a Sn-doped (100) oriented substrate. This is because the corrugations probably lie along a (111) *A* oriented surface. According to the selection rules on the first order Raman scattering, LO phonon should be observed on (100) oriented surface, and both transverse optical (TO) and LO phonon be seen on (111) *A* oriented surface. As a result, the spectrum (b) presents enhanced peak *C* relating to TO phonon and L^-. Raman spectrum (c) with the heat-treated InP corrugations in a (Sn + InP) saturation chamber is in fairly good agreement with spectrum (b). In spectrum (d) measured with InP corrugations which were heat treated with a GaAs wafer cover, peak *D* appears. We speculate that this peak was caused by

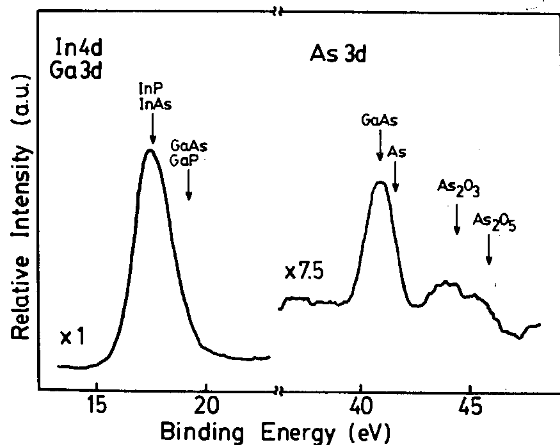

FIG. 3. XPS spectra of In 4*d*, Ga 3*d*, and As 3*d* for heat-treated surface corrugations on an InP substrate covered with a GaAs wafer.

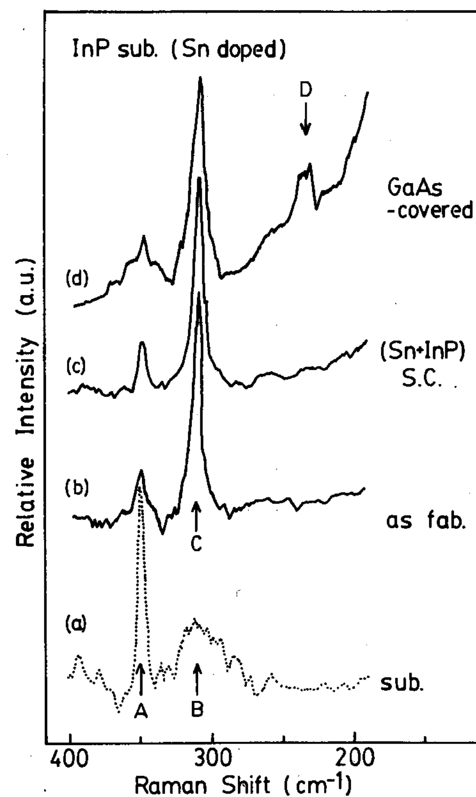

FIG. 4. Raman spectra of heat-treated surface corrugations on InP substrates. (a) Sn-doped InP substrate; (b) surface corrugations fabricated on InP substrate; (c) surface corrugations on InP substrate heat-treated in a (Sn + InP) saturation chamber; (d) heat-treated surface corrugations on an InP substrate covered with a GaAs wafer.

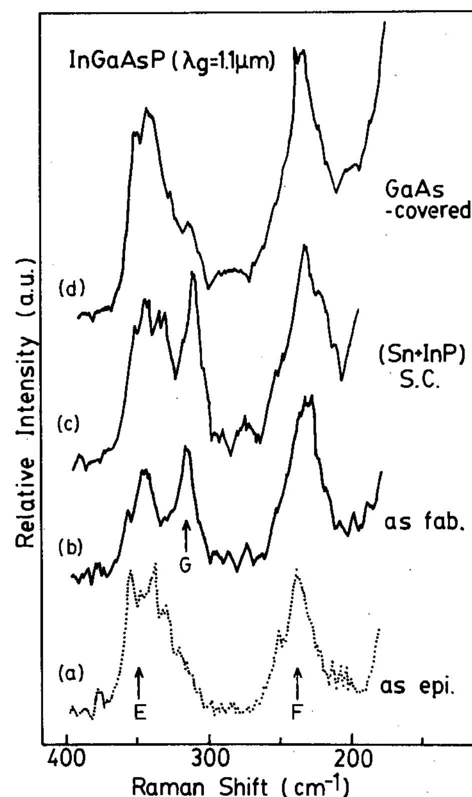

FIG. 5. Raman spectra of heat-treated surface corrugations on InGaAsP ($\lambda_g = 1.1\,\mu m$). (a) InGaAsP ($\lambda_g = 1.1\,\mu m$) epilayer; (b) surface corrugation fabricated on an InGaAsP epilayer; (c) surface corrugations on an InGaAsP epilayer heat-treated in a (Sn + InP) saturation chamber; (d) heat-treated surface corrugations on an InGaAsP epilayer covered with a GaAs wafer.

some materials that appeared in the grooves during heat treatment. Raman spectra of InGaAsP ($\lambda_g = 1.1\ \mu$m) layers are shown in Fig. 5. Peak F on its spectrum (a) corresponds to peak D on the spectrum (d) in Fig. 4. From these results it seems that peak D on spectrum (d) in Fig. 4 arises from an InGaAsP quaternary alloy single crystal which was formed in the grooves during heat treatment. Peak E on the spectrum (a) in Fig. 5 is related to LO phonon in InP; peak G, to the TO phonon. These peaks, E and G, seem to be similar to peaks A and C in Fig. 4, respectively. Spectra (c) and (d) are for the surface corrugations on an InGaAsP epilayer which was heat treated both in a (Sn + InP) saturation chamber and with a GaAs wafer cover, respectively. Spectrum (c) represents fairly good agreement with spectrum (b) with surface corrugations fabricated on an InGaAsP epilayer. This means that vaporized P atoms in a (Sn + InP) saturation chamber can greatly suppress the thermal deformation of the surface corrugations. From the fact that there is a similarity between spectrum (d) and spectrum (a), it is understood that vaporized Ga and As atoms are transported from the GaAs wafer to the surface corrugations on the InGaAsP ($\lambda_g = 1.1\ \mu$m) epilayer, changing the surface corrugations to erosion surfacelike.

In summary, we have characterized thermally deformed surface corrugations on both an InP substrate and an InGaAsP ($\lambda_g = 1.1\ \mu$m) layer by means of XPS and laser Raman spectroscopy. From the XPS analysis, it has been found that the material in the concave bottom of the thermally deformed InP surface corrugations is composed of In, P, Ga, and As atoms. The analysis through the Raman spectra has also verified that the material appeared in the grooves is apparently an InGaAsP single crystal. In addition, it has been confirmed that a sufficiently vaporized phosphorus pressure can greatly suppress the thermal deformation of surface corrugations on both an InP substrate and an InGaAsP ($\lambda_g = 1.1\ \mu$m) layer. These results suggest that if our saturation chamber technique is applied to DFB laser fabrication, it appears that anyone can easily fabricate high-performance DFB lasers. Further development of this fabrication process is now under way.

The authors would like to thank Dr. H. Mizuno and T. Kajiwara for their continuous encouragement.

[1]S. Akiba, X. Utaka, K. Sakai, and Y. Matsushita, Electron. Lett. **18**, 77 (1982).
[2]T. Matsuoka, H. Nagai, Y. Itaya, Y. Noguchi, Y. Suzuki, and T. Ikegami, Electron. Lett. **18**, 27 (1982).
[3]Y. Uematsu, H. Okuda, and J. Kinoshita, Electron. Lett. **18**, 857 (1982).
[4]H. Nagai, Y. Noguchi, T. Matsuoka, and Y. Suzuki, Jpn. J. Appl. Phys. **22**, L291 (1983).
[5]H. Nagai, Y. Noguchi, and T. Matsuoka, J. Cryst. Growth **71**, 225 (1985).
[6]J. Kinoshita, H. Okuda, and Y. Uematsu, Electron. Lett. **19**, 215 (1983).
[7]H. Temkin, G. J. Dolan, N. A. Olsson, C. H. Henry, R. A. Logan, R. F. Kazarinov, and L. F. Johnson, Appl. Phys. Lett. **45**, 1178 (1984).
[8]P. Besomi, R. B. Wilson, W. R. Wagner, and R. J. Nelson, J. Appl. Phys. **54**, 535 (1983).
[9]M. Ayabe, H. Nagasawa, and K. Kaneko, J. Cryst. Growth **58**, 1980 (1982).
[10]T. Tanahashi, I. Ushijima, I. Umebu, S. Nakai, Y. Nishitani, and K. Akita, J. Cryst. Growth **64**, 492 (1983).
[11]G. Abstreiter, R. Trommer, and M. Cardona, Solid State Commun. **30**, 703 (1979).