

Wafer bonding of different III–V compound semiconductors by atomic hydrogen surface cleaning

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Large-area wafer bonding of different III–V compound semiconductors in an ultrahigh vacuum background is demonstrated. The bonding procedure, the microstructure, and the mechanical strength of the bonded GaAs/InP and GaAs/GaP interfaces were studied. The cleaning procedure and the bonding were separated in order to avoid undesired artifacts and thermal stress at the interface. First, thermally generated atomic hydrogen was employed to clean the surfaces. Then, the wafers were brought into contact below 150 °C. At contact, the interface formed spontaneously over the whole wafer area without application of a mechanical load. Transmission electron microscopy showed the formation of atomically direct interfaces and misfit dislocation networks. The fracture surface energy was measured as being comparable to that of respective bulk materials. Heat treatments of the bonded GaAs/InP samples led to relaxation of the interfaces but also to the formation of nanoscopic voids in the interface plane and volume dislocations. © 2001 American Institute of Physics. [DOI: 10.1063/1.1403684]

I. INTRODUCTION

Direct wafer bonding (DWB) of III–V semiconductors has become increasingly important as a key technique for fabricating optoelectronic devices. It enables interface formation between different materials, which, due to large lattice mismatch (see Table I), cannot be epitaxially grown on one another without the formation of a high density of threading dislocations. The applied fields involve manufacturing of high brightness light emitting diodes (LED),¹ vertical cavity surface emitting lasers (VCSELs),^{2–9} distributed feedback lasers,¹⁰ optic coupling,¹¹ second harmonic generators (SHGs),¹² waveguide arrangements, stacked cascade solar cells, and field effect transistors. Thus, the necessity of understanding and controlling wafer bonding of compound semiconductors will further increase in the near future.¹³

In most fabrication procedures, the wafers to be bonded are first placed into contact, face to face, after the necessary chemical cleaning procedures to eliminate particle and other contaminants. This generally leads to relatively weak van der Waals type bonding between the wafers. Subsequently, some heating steps are carried out in reducing atmospheres under a mechanical load to maintain good contact, thereby achieving the formation of stronger chemical bonds. Typically, heating temperatures of 600–800 °C and mechanical pressure of a few GPa have been reported. However, these high temperature and high mechanical load processes encompass a series of disadvantages and restrictions such as the degradation of electronic characteristics, interdiffusion of individual layers or smearing out of doping profiles, incongruent decomposition, and thermomechanical strain due to different thermal expansion coefficients (see Table I). Technologically, the last

factor makes it difficult to scale up the wafer size for bonding. The area of the samples for bonding is typically a couple of cm². In fact, wafer-size bonding of 75 mm GaP to AlGaInP–GaP was reported.¹⁴ However, wafer-size bonding of thermally mismatched materials remains a challenging process. The microstructure of the GaAs/InP interfaces formed by the aforementioned methods has been studied in detail,^{15,16} where not only the dislocations related to the lattice mismatch and the geometry at the surfaces such as twist and tilt angles between the bonded crystals were found, but also dislocations outside of the interface plane caused by thermal mismatch during cooling.

In addition, the gaseous products from the reduction of oxides, decomposition of contaminants, and outgassing are enclosed at the interface, leading to the formation of different size bubbles or voids. In order to avoid void formation, one of the surfaces to be bonded is often patterned with a grid of channels a few to 100 μm in width and a pitch of a few 100 μm.¹⁷

Recently, uniform direct wafer bonding of whole GaAs wafers was achieved by two methods.^{18–20} In both cases, the wafer surfaces are cleaned *in situ*, and then contacted, or bonded, without any mechanical load. Accordingly, this enables one to avoid enclosing undesirable released surface contamination during heating and to conduct bonding at low temperatures.

The bonding method proposed in Ref. 18 is carried out all the way through in an ultrahigh vacuum (UHV) background. The wafer bonding utilizing UHV techniques is often called UHV bonding, the most significant merit of which is a bonding temperature which can be as low as room temperature.^{21–24} It was shown that a covalently bonded atomically abrupt interface could form^{21,22} in the case of Si/Si. In Ref. 18, GaAs/GaAs was bonded in the temperature range of 150–400 °C by surface cleaning using thermally generated atomic hydrogen. In this article, this cleaning

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TABLE I. Lattice constants and linear thermal expansion coefficients of GaAs, InP, and GaP.

Material	Lattice constant (nm)	Linear thermal expansion coefficient ($10^{-6}/\text{K}$)
GaAs	0.565	5.5
InP	0.587	4.75
GaP	0.545	5.8

method is applied to surfaces of different III–V materials such as GaAs, InP, and GaP to enable large-area DWB at low temperatures without any mechanical load.

II. EXPERIMENT

Semi-insulating GaAs (001) wafers of 50 and 75 mm and 50 mm InP (001) wafers doped with Zn were used, both of which were liquid encapsulated Czochralski (LEC) grown and had “epi-ready” surface quality. The surface of the 50 mm GaP wafers used was epi ready as well, with a miscut of approximately 2° in the $\langle 100 \rangle$ direction. As a protective first step, immediately after unpacking the wafers in a cleanroom, the wafers were put together face to face to avoid contamination of the surfaces and to reversibly bond them [Fig. 1(a)].²⁵ No wet chemical cleaning was done at all. Wet steps are often a source of contamination such as particles and organic substances. Therefore, it is more desirable to avoid them unless it is necessary, as will be shown later. The pre-bonded wafer pair was inserted vertically into the UHV system with background pressure of less than 5×10^{-11} Torr, and was separated by ~ 1.5 cm using sharp blades. Then the separated wafers were heated from both sides first up to $\sim 350^\circ\text{C}$. After confirming the decrease of water and other contaminants desorbing from the wafers by a mass spectrometer, typically after 10–15 min, an atomic hydrogen beam was applied to the opening between the two wafers with the temperature held constant [Fig. 1(b)]. The cleaning conditions for each bonding experiment are shown in Table II. Atomic hydrogen flow was produced by feeding hydrogen gas through a tantalum capillary heated by electron bombardment to 2100 K.^{26–28} The temperature of the capillary was measured by a pyrometer. During the cleaning process, the hydrogen flow rate was controlled so that the pressure of the atmosphere was maintained at 1.0×10^{-6} Torr.

It has been found that, when using atomic hydrogen as a reducing agent, atomically clean compound semiconductor surfaces can be prepared at significantly lower temperatures than when using only thermal decomposition of surface oxides. The reduction in cleaning temperature permitted by the use of atomic hydrogen is of great importance for avoiding thermal decomposition of near-surface layers of the crystals themselves. The reactions reported between atomic hydrogen ($\text{H}\cdot$) and the oxides^{29–35} can be summarized as follows. For GaAs:

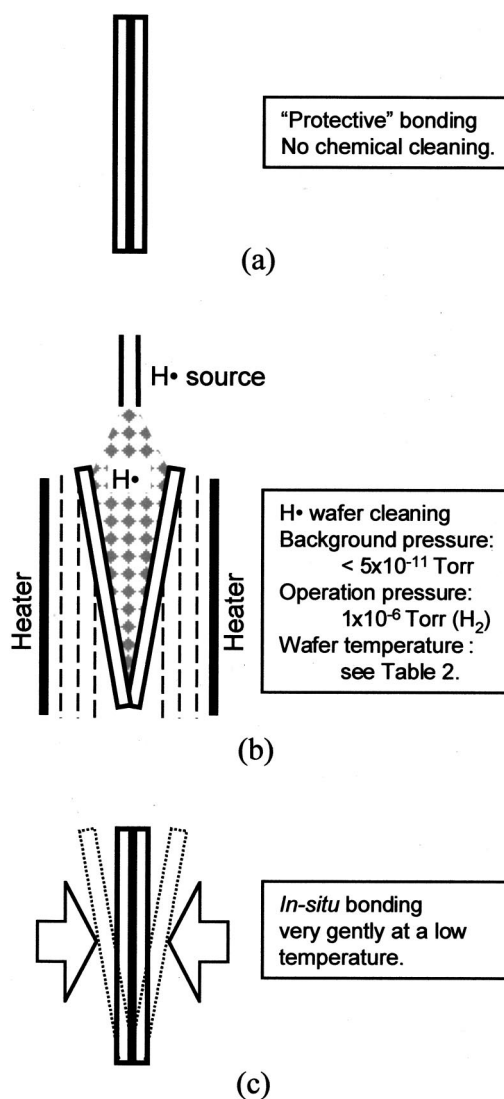
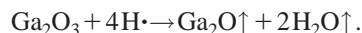
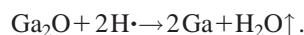


FIG. 1. Schematic of the bonding procedure.

where $x=1, 3$, or 5 stands for the various oxides of arsenic. At above 200°C ,



At temperatures lower than 200°C , a Ga-rich surface may form by the following reaction:

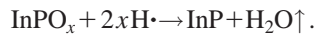


Consequently, the temperature used should be higher than 200°C . For InP,

TABLE II. Surface cleaning and bonding conditions.

Combination	Cleaning temperature ($^\circ\text{C}$)	Cleaning time (min)	Bonding temperature ($^\circ\text{C}$)
InP/InP	360	60	$\sim 300^a$
GaAs/InP	300–350	60	< 150
GaP/GaP	500	30	$\sim 400^a$
GaAs/GaP	500	30	< 150

^aImmediately after the cleaning procedure.



Further exposure to atomic hydrogen might lead to an In-rich surface. Therefore, care must be taken to prepare a stoichiometric surface. The reaction of atomic hydrogen with the oxides on GaP surfaces has not been studied to the best of the authors' knowledge. However, the surface oxides of GaP are considered to be a mixture of gallium oxides and phosphorus oxides and, therefore, should be able to be removed by similar chemical reactions with atomic hydrogen.

Following the cleaning and some cooling, the wafers were brought into contact by rods from both sides. No intentional mechanical load was applied, only a gentle force just large enough for the wafers to come into contact [Fig. 1(c)]. For all the bonding experiments, the (001) surfaces were bonded. The [110] directions of both wafers were aligned parallel using the orientation flats of the wafers. Since almost always there is inaccuracy in the crystallographic orientation of the flats, rotational misorientation between the two wafer crystals of less than 1° (for details, see Ref. 18) is often present.

As mentioned above, the bonding temperature has to be almost at room temperature to avoid thermal stress in the case of bonding wafers of different thermal expansion coefficients (see Table II). Otherwise, the wafers may fracture right after bonding. In the UHV system used, the cleaned wafer surfaces are exposed to residual gases, while the wafer temperature decreases to a low bonding temperature. In a previous study, it was shown for the case of GaAs that the cleaned surface can be bonded at room temperature. The effect of recontamination during cooling was observed as a slight decrease in the bond strength. For the present experiments, the UHV bonding system was equipped with a nitrogen trap to minimize exposure of the cleaned surface to residual gases. Therefore, in this study, pairs of the same materials, InP and GaP, were bonded immediately after the cleaning procedure to investigate the bondability. The bonding of dissimilar materials was carried out below 150°C .

The interface microstructure was analyzed by transmission electron microscopy (TEM). The TEM specimens were taken from the center part of the bonded areas. They were cut either at 90° (for a cross-sectional view) or at 25° (for plan view) with respect to the interface plane, mechanically dimpled, and ion thinned. A Philips CM20T microscope was used for plan-view and cross-sectional observations at 200 kV, and JEOL 4000EX and 4010 microscopes were used for cross-sectional high-resolution observations at 400 kV. The fracture energy was measured by double cantilever beam (DCB) tests. For the DCB tests, samples of ~ 1 cm in width and a few cm in length in the [110] direction were cut from the bonded wafer pairs. The crack length was measured by using infrared transmission light, as a blade of $100\ \mu\text{m}$ in thickness was gradually inserted into the interface plane.

III. RESULTS AND DISCUSSION

A. *In situ* infrared observation

As listed in Table II, the wafers were brought into contact, or bonded, immediately after the $\text{H}\cdot$ cleaning for com-

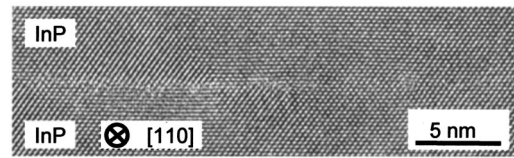


FIG. 2. Cross-sectional TEM of the InP/InP interface.

binations of the same materials, or below 150°C for combinations of different materials. For all the cases shown here, the bonding occurred spontaneously over almost all of the wafer area as soon as the wafers came into contact with each other. The interface formation occurred so fast that observation with the infrared camera could not follow the propagation of the interface front quickly enough. This clearly indicates that the bonding occurs by minimizing surface energy, with practically no load applied. In practice, therefore, this method does not limit the bonding size and should be applicable to any wafer diameter.

B. GaAs/InP interface

In order to find proper cleaning conditions for oxide removal from InP and to confirm the bondability of the cleaned surfaces, InP/InP was bonded. During $\text{H}\cdot$ cleaning of the InP surfaces, the cleaning temperature was maintained at $\sim 350^\circ\text{C}$ to avoid incongruent decomposition of the crystal (Table II), and the wafers were then bonded immediately, i.e., at $\sim 300^\circ\text{C}$. Even at this temperature, too long a cleaning time leads to an excess of In. In fact, although they were partial, In islands were observed at the bonded InP/InP interface, even for a cleaning time of only 30 min. Figure 2 shows the InP/InP interface that was bonded immediately after the cleaning process. As can be seen, native oxides were removed, and an atomically abrupt interface formed to avoid thermal stress.

It was found that the GaAs surface as well can be cleaned under the same condition as that for InP. Thus, GaAs and InP wafers can simultaneously undergo the cleaning process in the same experimental setup. After this $\text{H}\cdot$ cleaning, the wafers were bonded below 150°C . As was observed by IR transmission photography, almost the whole wafer was bonded [Fig. 3(a)]. No wafer bowing was visible for this sample. On the other hand, the wafers that were put into contact at $\sim 300^\circ\text{C}$ were bonded over the whole wafer area, but, during cooling, fractured along cleavage planes into a few bond pieces due to thermal stress [Fig. 3(b)]. However, no fracture along the bond interface was observed.

Figure 4(a) shows a cross-sectional view of the bonded GaAs/InP interface. Figure 4(b) is a magnified view of Fig. 4(a). As can be seen, the two crystals are bonded directly. It is worth noting that, even at this bonding temperature, misfit dislocations with a spacing of 8–12 nm have formed. This spacing corresponds to the lattice mismatch between GaAs and InP (see Table I). Also, an atomic half plane in the interface can be seen in Fig. 4, which may result from a surface step that existed on a wafer surface before bonding. The spacing of two misfit dislocations with a 60° dislocation between them is approximately 1.5 times as large as the spacing of two misfit dislocations without a 60° dislocation be-

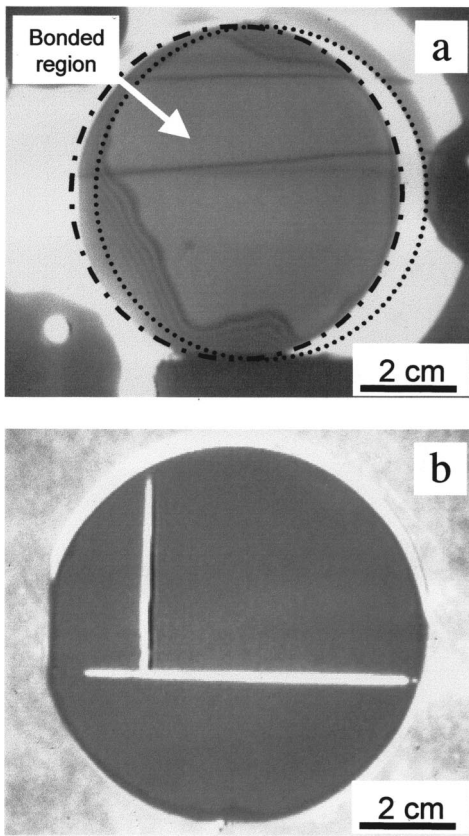


FIG. 3. (a) *In situ* infrared photograph of 2 in GaAs (dashed circle)/InP (dotted circle) wafers bonded below 150 °C. The bond area extends over almost the whole wafer area. Interference fringes at some edge regions indicate a gap, possibly due to contaminants such as particles. (b) Infrared photograph of GaAs/InP bonded at approximately 300 °C (right after cleaning). The bonded wafer pair fractured due to thermal stress.

tween them. This observation is identical to that reported in Refs. 15 and 16. In the plan view of this sample, the misfit dislocations can be observed as a two-dimensional network [Fig. 5(a)]. The moiré patterns overlap. The twist angle of this sample was measured as being near zero from the diffraction pattern of a cross-sectional sample. Accordingly, any twist component is negligible. The periodicity of the misfit

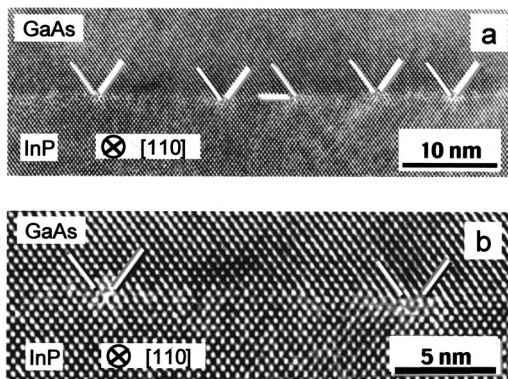


FIG. 4. (a) Cross-sectional TEM of the GaAs/InP interface bonded below 150 °C. Terminating lattice planes of misfit dislocations and of a 60° dislocation are indicated. (b) Magnified view of (a). An atomically direct interface is confirmed, with two misfit dislocations due to the lattice mismatch between GaAs and InP.

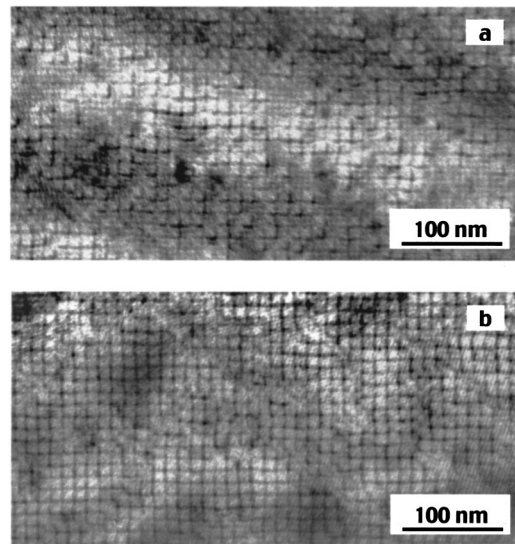


FIG. 5. (a) Plan-view TEM of the GaAs/InP interface showing the misfit dislocation network. It clearly shows that covalent bonds form even at low temperatures. (b) Plan-view TEM of the GaAs/InP interface after heat treatment at 650 °C for 30 min. The misfit dislocation network exhibits a more regular structure, including reactions with 60° dislocations, indicating that the interface is relaxed.

dislocations measured from the plan-view picture is ~11 nm, which agrees with the value from the cross-sectional picture (Fig. 4) and the crystallographic calculation.

Heat treatment of the bonded wafers was found to enhance the appearance of the dislocation networks. Figure 5(b) is a plan-view micrograph after heat treatment at 650 °C for 30 min, in which the network presents a more continuous structure. A half-period shift due to the 60° dislocations can be seen as a structure made up of much clearer lines. Thus, the heat treatment caused relaxation and probably diffusion of atoms at the interface. However, some “nanovoids”³⁶ have formed as can be seen in the cross section at lower magnifications after heat treatment at 350 °C for 1 h [Fig. 6(a)]. Ripening of the nanovoids was observed after heat treatment of 650 °C for 30 min [Fig. 6(b)]. The voids formed symmetrical to the interface with the borders preferably parallel to the {111} planes. The size of the nanovoids is found

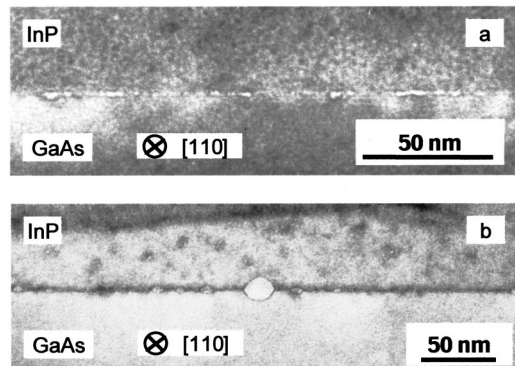


FIG. 6. (a) Cross-sectional TEM of the GaAs/InP interface after heat treatment at 350 °C for 1 h. (b) Cross-sectional TEM of GaAs/InP interface after heat treatment at 650 °C for 30 min. Voids imaged by Fresnel defocus contrast can be seen.

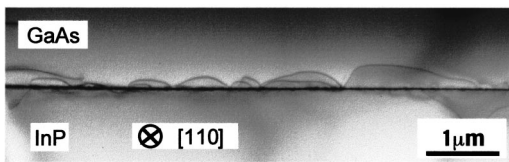


FIG. 7. Cross-sectional TEM of the GaAs/InP interface after heat treatment at 650 °C for 30 min. Volume dislocations formed in the crystal can be seen.

to be approximately a few nm in the direction of the interface with some very rare cases of 10 nm, seen in the middle of Fig. 8(b). The voids are thought to form due to aggregation of defects at surface steps on the microscopic surface.³⁶ Moreover, some volume dislocations were found near the interface (Fig. 7). These volume dislocations appear to form half loops with both ends at the interface. They were found on both sides of the interface but more so in GaAs. The volume dislocations were not found in the as-bonded samples. It is clear that they formed during the postbonding heat treatments. Such volume dislocations had been found in GaAs/InP interfaces.^{15,16} It was suggested that the volume dislocations might form during cooling from the bonding temperatures of 630 °C to room temperature. It can, therefore, be concluded that the volume dislocations formed due to thermal stress. In the current case, the dislocation half loops appear to end in the interface plane, indicating that the source may have been interfacial defects. However, our observations are not complete enough to measure the distribution or to gain statistical information on the characteristics of these volume dislocations.

C. GaAs/GaP interface

First, a pair of GaP wafers was bonded in order to test the H₂ cleaning procedure for GaP. Cleaning at as low as 350 °C, however, did not lead to even adhesion of the wafer pair, presumably due to insufficient removal of oxides from the surface. Therefore, the H₂ cleaning for GaP surfaces was conducted at 500 °C for 30 min (see Table II for the bonding condition), and the wafers were bonded immediately after cleaning. Figure 8 shows a cross-sectional view of the GaP/GaP interface. Again, the native oxide was removed, and an atomically abrupt interface formed. The twist misalignment of these two wafer crystals was measured to be less than 0.05°. However, a distinct tilt angle is observed here by viewing along the interface. The tilt angle measured from a cross-sectional picture is approximately 3°. This tilt angle was confirmed in the selected area diffraction pattern (SAD) of the cross section. SAD revealed that the tilt axis is $\langle 110 \rangle$ direction, which is parallel to the viewing direction of Fig. 8

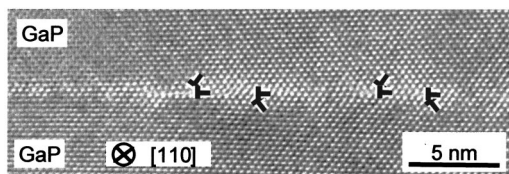


FIG. 8. Cross-sectional TEM of the GaP/GaP interface.

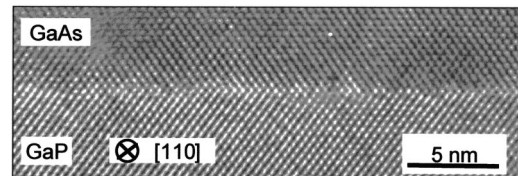


FIG. 9. Cross-sectional TEM of the GaAs/GaP interface bonded below 150 °C. An atomically direct interface has formed.

in the interface plane. The atomical steps at the original GaP surfaces due to the miscut were aligned in a $\langle 100 \rangle$ direction. At the bonded interface, the miscut $[100]$ directions of both crystals were perpendicular to each other. The resulting tilt angle of 3° and its $[110]$ direction can be explained by adding these two $[100]$ vectors and angles of 2° in its direction. Correspondingly, terminating lattice fringes were observed (see Fig. 8). The Burgers vectors of these 60° dislocations were often found to alternate. This alternating configuration of the Burgers vectors is considered to give a tilt boundary of a $\langle 110 \rangle$ rotation axis when consisting of only 60° dislocations.

Using the same cleaning condition, a GaP wafer of the same kind was bonded to GaAs below 150 °C. As shown in Fig. 9, an atomically abrupt interface formed between the GaAs and GaP crystals, as well. A tilt angle of about 1.5° introduced by the miscut of the GaP is visible again. The tilt axis lies inclined about 45° toward the transmission direction in Fig. 9, which complicates interpretation of the image with respect to terminating dislocations along the interface. The miscut of about 2° of the GaP surface corresponds to a mean periodicity of atomic surface steps of about 8 nm. Therefore, for the case of near-zero twist alignment detected, an appropriate high density of 60° dislocations should cross a square network of misfit dislocations with a period of 10.7 nm in an ideally relaxed interface of this kind. In addition, the tilt orientation relationship of this bonded wafer pair of 2° did not allow us to attain the usual conditions for imaging the misfit dislocation network in the plan-view direction. Due to these factors, the formation of misfit dislocations could not be confirmed by TEM.

D. Mechanical properties of the bonded interfaces

During the DCB test, GaAs/InP samples exhibited brittle fracturing only inside the InP crystal as a blade was inserted into the interface. Since the interface did not open up, the surface energy could not be estimated. Nevertheless, this result surely indicates a strong interface. In addition, in order to assure the strength of the interface, a quarter of the 2 in. bonded wafers underwent heat treatment at 450 °C. This procedure did not cause any failure of the sample. These facts indicate that the mechanical stability of the bonded GaAs/InP interface is high enough to withstand any typical technological procedures.

The fracture surface energy, γ , at a grain boundary between crystals 1 and 2 can generally be expressed, including the interface energy γ_{int} associated with structural relaxation, as

$$\gamma = \frac{1}{2}(\gamma_1 + \gamma_2 - \gamma_{\text{int}}). \quad (1)$$

In cases of strong bonding at the interface such as covalent bonding, γ_{int} can be assumed to be small compared to the surface energy γ_1 and γ_2 , giving

$$\gamma \approx \frac{1}{2}(\gamma_1 + \gamma_2). \quad (2)$$

For the GaAs/GaP interface on which a crack at the interface could be formed, the fracture surface energy γ of the bonded interfaces was estimated using

$$\gamma = \frac{1}{2}(\gamma_1 + \gamma_2) = \frac{3t_b^2 E_1 t_{w1}^3 E_2 t_{w2}^3}{16L^4 (E_1 t_{w1}^3 + E_2 t_{w2}^3)}, \quad (3)$$

where t_b is the thickness of the blade, L is the crack length, and E_1 , t_{w1} , E_2 , and t_{w2} are the Young's moduli and thickness of wafers 1 and 2, respectively. The crack length was measured by IR transmission spectroscopy. Errors in the measured values due to the resolution limit of the IR transmission were corrected.³⁷ The thickness of the blade was 100 μm .

The Young's moduli of GaAs and GaP are 121.3 and 144.7 GPa, and the thicknesses of the wafers used are 535–540 and 265–270 μm , respectively. By substituting these values with the parameters in the Eq. (1), the fracture surface energy of the bonded GaAs/GaP is estimated to be

$$\gamma(\text{GaAs/GaP}) = 0.84 \pm 0.14 \text{ J/m}^2.$$

In the case of bonded pairs of identical materials,

$$\gamma = \frac{3t_b^2 E t_w^3}{32L^4}. \quad (4)$$

Likewise, the fracture surface energy of the bonded GaP/GaP interface can be estimated to be

$$\gamma(\text{GaP/GaP}) = 1.17 \pm 0.12 \text{ J/m}^2.$$

The fracture surface energies of GaAs/GaAs interfaces formed using the identical bonding technique were estimated in Ref. 18 as

$$\gamma(\text{GaAs/GaAs}) = 0.9 \pm 0.1 \text{ J/m}^2,$$

for samples bonded at $\sim 400^\circ\text{C}$

$$= 0.7 \pm 0.05 \text{ J/m}^2,$$

for samples bonded at 150°C .

The fracture surface energy of the interface formed at 150°C was found to be lower than that of the interface formed at 400°C . The difference can be attributed to the surface recontamination that occurred during the cooling time from the cleaning temperature of 500°C to the bonding temperature. Or it could be due to less structural relaxation in low temperature bonding, i.e., γ_{int} is not negligible.

The fracture surface energy of GaAs and GaP has been predicted from various theoretical models by different groups, where scattering in the values obtained can be seen from 0.91 to 3.17 for GaAs (100) and from 1.04 to 4.00 for GaP (100).^{38–40} However, a tendency $\gamma(\text{GaP}) > \gamma(\text{Si})$

$> \gamma(\text{GaAs})$ for low index planes exists and was confirmed by measured values.⁴¹ In the current experiments, we obtained values for bonding temperature of 400°C of

$$\gamma(\text{GaP/GaP}) = 1.17 \pm 0.12 \text{ J/m}^2$$

and

$$\gamma(\text{GaAs/GaAs}) = 0.9 \pm 0.1 \text{ J/m}^2,$$

and for bonding temperature of 150°C values of

$$\gamma(\text{GaAs/GaP}) = 0.8 \pm 0.14 \text{ J/m}^2$$

and

$$\gamma(\text{GaAs/GaAs}) = 0.7 \pm 0.05 \text{ J/m}^2.$$

Thus, with the same tendency and almost similar values to the values reported, it can be concluded that the values of the fracture surface energy of the bonded interfaces are as high as those of bulk materials.

IV. CONCLUSION

Thermally generated atomic hydrogen was employed to clean various III–V compound semiconductor wafers in an UHV background. Using this method, it was demonstrated that different III–V wafers such as GaAs/InP and GaAs/GaP can be bonded at temperatures below 150°C without application of mechanical loads, and form wafer-size bond areas. TEM observations showed that atomically abrupt interfaces result in misfit dislocation networks even at the interfaces that formed below 150°C . The interface structure and adhesion, comparable to the cohesion of bulk materials, clearly demonstrate that low temperature covalent bonding of different III–V compound semiconductors is possible. The following merits underscore the high manufacturability of this bonding technique. (1) No pretreatment using chemicals is necessary. (2) Whole wafer areas can be bonded without application of a mechanical load. (3) Extension of the bonding procedure to wafer dimensions of any size appears to be possible. (4) Bonding at low temperatures may enable integration of many other different materials combinations. The electronic properties of the interfaces formed are now under investigation.

ACKNOWLEDGMENTS

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