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Low Temperature Direct Bonding of InP and Si_3N_4-Coated Silicon Wafers for Photonic Device Integration

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Low temperature (200°C) direct bonding of InP and Si_3N_4 coated silicon wafers using oxygen plasma surface treatment has been demonstrated, which transfers InP-based thin films onto Si substrates for heterogeneous photonic integration. Pulling test shows a high bonding energy comparable to the InP fracture energy. The high crystalline quality of the bonded thin film is preserved, as evident from photoluminescence and high resolution X-ray diffraction measurements. Cross-sectional transmission electron microscopy shows an intimately bonded interface with no observable defects generated near the bonded interface. A thin layer of amorphous oxide about 6 nm thick has been detected at the bonded interface. The hydrophilic interface obtained by oxygen plasma activation helps to achieve high quality bonding. Due to the high thermal conductivity of Si_3N_4, these high quality bonded structures of InP/Si with Si_3N_4 sandwiched in-between have potential applications in enhancing performance of high power Si photonic integrated devices.
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Recent development of silicon photonics stimulates interest in integrating III-V compound semiconductors with silicon (Si) for optoelectronic device integration.[1–4] The transfer of III-V compound semiconductor based epitaxial layers onto silicon-on-insulator (SOI) surface via wafer bonding can provide active optical functionalities such as optical gain in heterogeneous photonic integration due to the presence of direct bandgap in III-V semiconductor materials. The Si layer, on the other hand, provides a high confinement waveguide platform for passive optical wave guiding. Because of the mismatch in thermal expansion coefficients of InP and Si, development of a robust and low temperature (< 400°C) wafer bonding process is important. SiO_2 intermediate layers are commonly used in bonding wafers together because of their reactive hydrophilic surface to facilitate initial room temperature Van der Waals bonding between wafer surfaces as well as to absorb gaseous by-products (H_2 and H_2O) from the intrinsic interface polymerization reactions during wafer bonding.[5,6] Several InP bonded membrane based photonic devices such as microdisk and photonic crystal lasers have been fabricated on Si wafers using thick SiO_2 as the bonding and low-refractive-index waveguide-cladding material.[7–9] Liang et al., also reported strong low temperature III-V semiconductor and Si bonding with a very thin SiO_2 (60 nm) intermediate layer to explore the potential for realizing evanescent gain based III-V on silicon lasers.[10] However, the poor thermal conductivity of SiO_2 limits device performance and necessitates relatively high threshold pump powers.[7,9] Using alternative dielectric materials with high thermal conductivity such as silicon nitride (Si_3N_4) could lead to improved device performance (almost 20 times higher thermal conductivity as that of SiO_2 (30 $Wm^{-1}°C^{-1}$ against 1.4 $Wm^{-1}°C^{-1}$)). Si_3N_4 is also widely used in semiconductor device processing and has good physical and chemical properties such as low etch rate in HF. For example, new $Si/Si_3N_4/Si$ and $Si/SiO_2/Si_3N_4/Si$ structures have been studied and exhibit much better thermal performance as compared to conventional SOI with a buried oxide layer, which is hampered in high-power integrated circuits by the self-heating effects caused by the poor thermal conductivity of the insulating SiO_2 layer.[11,12] However, there are only few reports investigating Si_3N_4 intermediate layer wafer bonding. Ismail et al., have demonstrated Si fusing bonding to Si_3N_4 coated wafer at 1000°C in a dry oxygen (O_2) environment.[13] Bowers et al., reported GaAs-Si wafer bonding with 50 nm thick Si_3N_4 intermediate layer using NH_3 plasma activation at 300°C.[14] Hayashi et al., have studied GaAs wafer bonding with thin nitride interlayer using O_2 plasma activation.[15] However, there are no detailed investigations of III-V semiconductor thin films transferred to nitride coated Si substrates.

In this paper, we demonstrate a low-temperature (200°C) wafer bonding process to transfer InP epitaxial thin films onto Si wafers using a Si_3N_4 intermediate layer via O_2 plasma surface activation. The bonding strength of the transferred film has been characterized in detail. Although there have been reports of transferring InP epitaxial thin film onto Si wafer via Si_3N_4 interlayer bonding[16], the method of transfer using Si_3N_4 as an intermediate layer has not been fully explored.

Experimental

Low pressure chemical vapor deposition (LPCVD) was used to deposit Si_3N_4 on single-sided polished 100 mm prime-grade (100) Si wafers at a temperature of 800°C. Depending on applications, different bonding layer thicknesses are required. In applications where optical coupling between the III-V semiconductor and Si waveguide is needed such as in evanescent optical field based devices, ultrathin bonding layers below 100 nm (no bonding layer with vertical outgassing channel design in the hybrid Si devices scheme) is preferred.[10,17] Whereas in applications such as III-V semiconductor membrane photonic devices on Si, a thick bonding layer is needed to prevent optical coupling to the Si substrate. To study the influence of the interlayer thickness on the optical field leakage to the substrate, the loss coefficient for different interlayer thicknesses is calculated when the thickness of the III-V semiconductor layer is 300 nm and 500 nm, respectively. Calculation results allow us to estimate the thickness of interlayers for real applications.

In this paper, bonding of InP epi-wafers to silicon substrates with 80 nm and 500 nm thick nitride films are used as examples. The desired Si_3N_4 thickness on the Si substrate can be easily controlled by adjusting the CVD deposition time. The InP epitaxial layer was grown by metal organic chemical vapor deposition (MOCVD) on InP (100) substrate. In our experiments, no additional layer was deposited on the III-V compound semiconductor layer to simplify the fabrication process. It is also a challenge to deposit a high quality smooth dielectric layer on III-V semiconductor material. Tapping mode atomic force microscopy (AFM) characterizes 5 μm × 5 μm surface areas of both Si_3N_4/Si and InP samples. The root-mean-square surface roughness is calculated within an area of 2.5 μm × 2.5 μm in the 5 μm × 5 μm

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scan area to ensure that the surfaces are smooth enough for intimate contact between the bonding surfaces.

The samples were cleaved into ~1–4 cm^2 for the wafer bonding experiments. The samples were cleaned in organic solvents. The nitride coated substrate was further cleaned in a modified RCA1 solution ($NH_4OH:H_2O_2:H_2O$ = 1:6:30) at 75°C for 15 mins. The reduced NH_4OH in the RCA offers the effectiveness of a standard RCA clean with less surface roughness.[18] The InP wafer was cleaned with NH_4OH (25%) to remove the oxide. Before bonding, both samples were exposed to O_2 plasma in Trion Sirus plasma etching system. The chamber pressure and power were kept at 100 mTorr and 50 W with O_2 gas flow kept at 20 sccm. O_2 plasma activation of surface before bonding is one promising technology for low temperature wafer bonding since it is known to be very efficient in removing hydrocarbon and water-related species.[19] Water contact angle measurement is done to observe the wettability on the sample surface after O_2 plasma activation.

The initial bonding was done at room temperature right after plasma activation and further annealing at 200°C with 0.3 MPa pressure in the vacuum chamber for 10 hours to obtain strong bonding. Tensile pulling test is used to assess the bonding strength. To transfer epi-InP film onto Si_3N_4/Si sample, the bonded sample was etched in $HCl:H_2O$ (1:1) and $H_3PO_4:H_2O_2:H_2O$ (1:1:38) solution to selectively remove InP substrate and the InGaAs etch stop layer respectively. Only the remaining InP-based thin layer structure was bonded to the Si substrate.

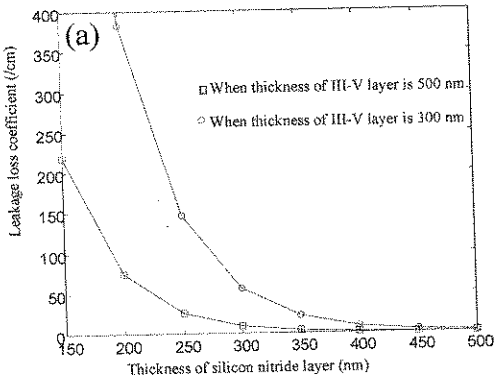
Cross sectional Transmission Electron Microscopy (TEM) images of InP thin film bonded on Si substrate with a Si_3N_4 interlayer is carried out to characterize the bonded interface. The samples for TEM are prepared by dicing, mechanical polishing and ion milling processes. Survival after the very harsh TEM sample preparation also indicates strong bonding between the InP and the Si substrate. Photoluminescence (PL) and high resolution X-ray diffraction (XRD) is further used to examine the crystalline quality of the bonded thin film.

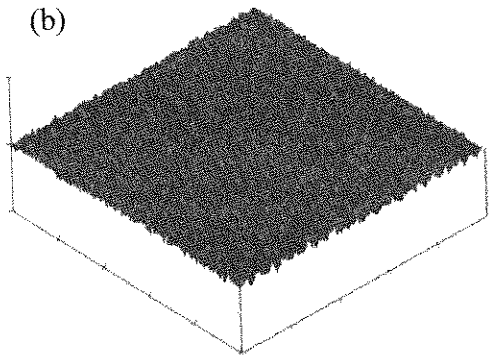
Results and Discussion

Figure 1a shows the calculated optical energy leakage loss coefficient for a beam centered at a wavelength of 1550 nm propagating in the Si guided layer leaking to the silicon substrate for different Si_3N_4 film layer thicknesses via multilayer waveguide mode analysis. Simulation results indicate that a 500 nm thick Si_3N_4 film is enough to prevent light leakage from the III-V semiconductor to the Si substrate.

Tapping mode AFM measurements show that the root-mean-square surface roughness of both Si_3N_4/Si and InP samples is less than 0.5 nm averaged over an area of 2.5 μm × 2.5 μm within a 5 μm × 5 μm scan area, which is good enough to bond the samples tightly together. The root-mean-square surface roughness of 500 nm Si_3N_4 grown on Si substrate after O_2 plasma activation is 0.35 nm as shown in Figure 1b AFM measurement. The surface after oxygen plasma exposure was seen to be very hydrophilic with a water contact angle of 7.5°, as indicated in Figure 1c water contact angle measurement. In addition, water contact angle of InP after O_2 plasma activation is measured to be 5.9°.

The bonding strength is assessed using the tensile pulling test prior to InP substrate removal. Fracture occurs at about 1.5 MPa for the bonded pair. Figure 2a shows an optical image on the fracture surface of the InP-Si bonding sample with 500 nm Si_3N_4 as the intermediate layer after the pulling test experiment. The separation did not occur at the bonded interface; instead the more brittle InP film fractured and peeled off. This indicates that the interface bonding strength is at least on the order of the bulk fracture energy of InP, which is strong enough for device fabrication. Figure 2b shows an optical image of a ~1 cm^2 2 μm-thick-InP thin film transferred to the Si_3N_4/Si substrate. O_2 plasma activation plays a very important role in the low temperature wafer bonding process for improving the bonding strength. Also it is reported that O_2 plasma surface treatment is very helpful for the suppression of interfacial bubbles generation.[19] Figure 2c shows the optical microscope image of the bubble free bonded pair. We have not





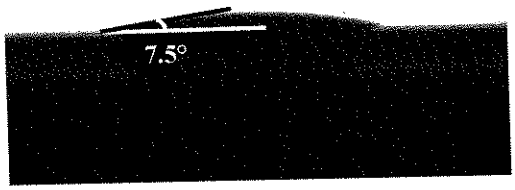


Figure 1. (a) Calculated optical energy leakage loss coefficient for 1550 nm beam propagating in the silicon guided layer leaking to the silicon substrate under different Si_3N_4 layer thickness. (b) AFM image showing morphology of Si_3N_4/Si after plasma activation. Scan area is 5 μm × 5 μm. Vertical scale is 10 nm. (c) Water contact angle measurement of 500 nm Si_3N_4/Si after O_2 plasma activation.

successfully bonded the wafers without oxygen plasma activation at this low temperature. Figures 3a and 3b show the cross sectional TEM images of InP thin film on Si substrate with a Si_3N_4 layer thickness of 80 nm and 500 nm. We can observe that InP thin films are robustly bonded to Si substrate. The bonded wafer is tightly bonded and fracture does not occur along the bonded interface.

In order to investigate the details of the bonding interface, high resolution transmission electron microscopy (HRTEM) images are taken. A smooth amorphous layer, which has obvious contrast with nitride layer, with thickness of about 6 nm at the InP-Si_3N_4 bonding interface is clearly seen in Figure 3c. This amorphous layer seen is most likely attributed to the O_2 plasma treatment. It is homogeneous and InP atoms are well aligned without showing any observable structural defects. Neither voids nor bubbles are observed. O_2 plasma treatment

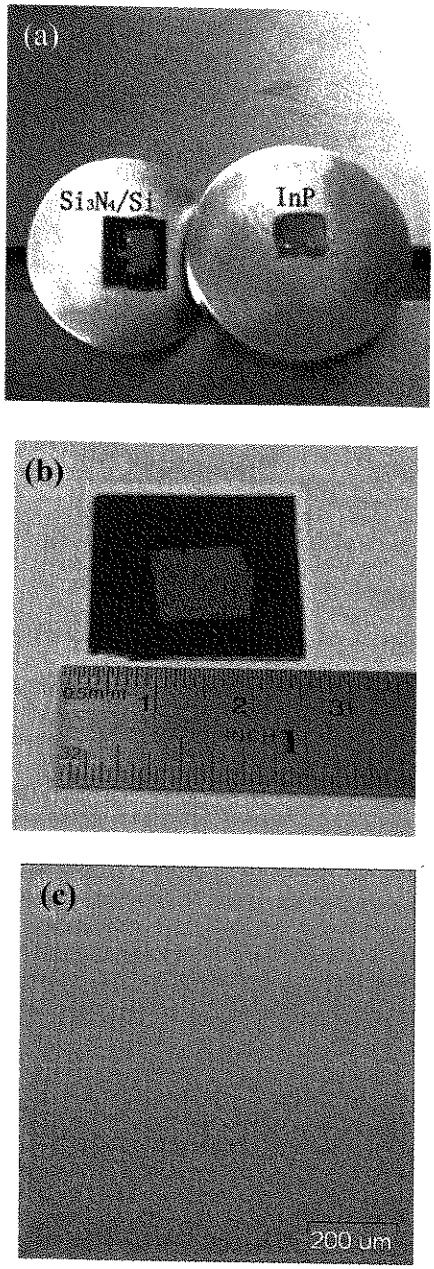


Figure 2. Optical image of (a) the fracture surface of the bonded pair after pulling test measurement and (b) ~1 cm^2 InP thin film transferred to Si_3N_4 coated Si substrates. (c) Microscope image of the bonded pair.

will create a very thin oxide layer on the surface (on the InP and Si_3N_4 surfaces in our experiment). This thin oxide layer shows a very smooth and extremely hydrophilic surface, as depicted by Figures 1b and 1c. A separate experiment indicates growth of amorphous oxide layer of about 3.2 nm thickness on the surface using the same oxygen plasma treatment conditions as measured by X-Ray Reflection (XRR), which is in agreement with our TEM results. These oxides are known to be more strained, very reactive and more likely to break and form new bonds.[19] This layer adheres well to interfaces, helps to create reliable bonding strength across the bonded interface, strengthened further by thermal treatment, thus suitable for device fabrication. We have previously demonstrated device performance of heterogeneously integrated ultra-large-angle super-compact grating (SCG) III-V-Semiconductor-on-Silicon laser by InP-Si direct wafer bonding using O_2 plasma treatment.[20,21]

The optical quality of the transferred InP thin film is further assessed by photoluminescence (PL). Figure 4a shows the room

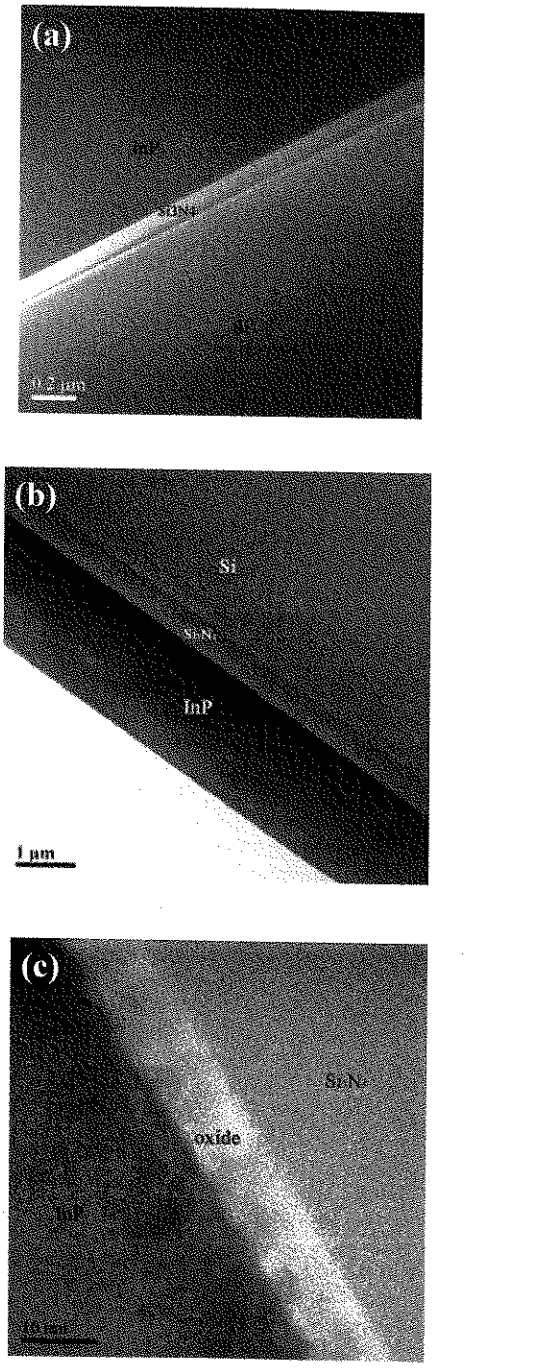


Figure 3. Cross-sectional TEM images of InP-Si_3N_4/Si bonded pair with (a) 80 nm Si_3N_4 and (b) 500 nm Si_3N_4. (c) Cross-sectional HRTEM image of the bonded interface InP-Si with 500 nm Si_3N_4.

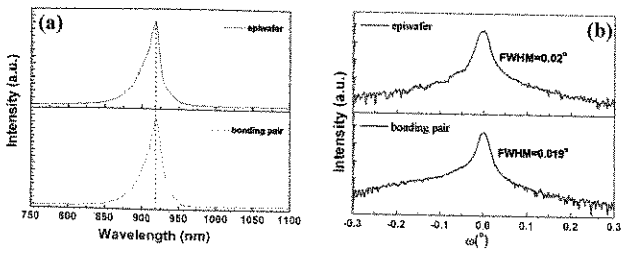


Figure 4. (a) PL and (b) XRD rocking curve of the original epi-wafer and bonded pair.

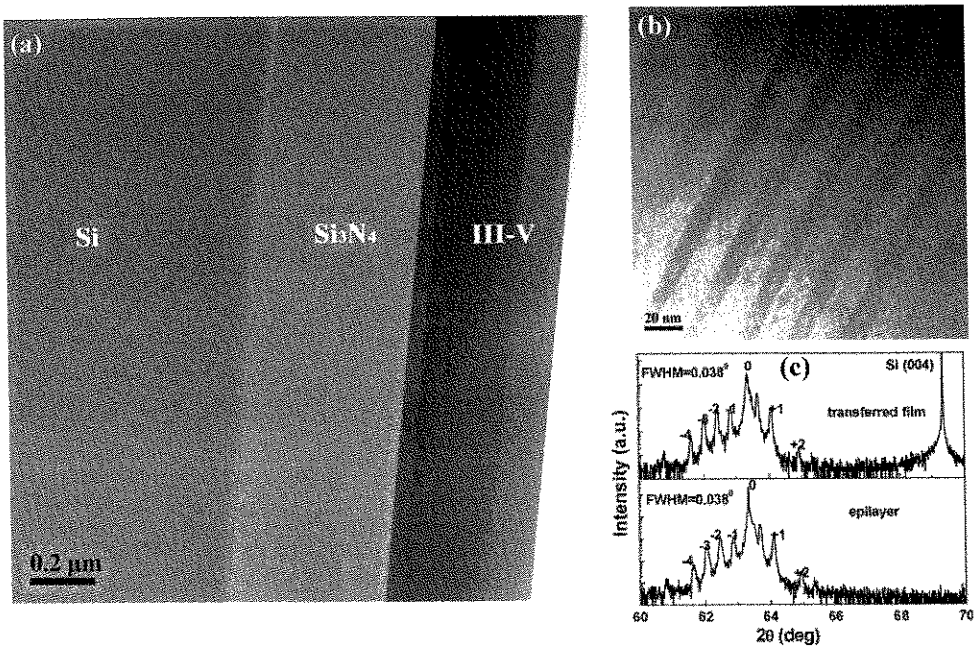


Figure 5. (a) Cross-sectional TEM image of the InP-based MQWs thin film transferred to Si_3N_4 coated Si substrate, (b) HRTEM images of MQWs, (c) XRD θ–2θ scans of as-grown and bonded epiwafer layers.

temperature PL spectra of the epilayer on InP substrate and the transferred thin film. The band edge transition is observed at around 919 nm. There is no obvious peak intensity change for the transferred film compared with the original epilayer, alluding to the high quality of the transferred film and that our process does not deteriorate the optical properties of the transferred thin film. In addition, no peak wavelength shift is observed, which suggests that the stress developed during the low temperature (200°C) bonding process is small enough not to affect the transferred InP thin film material properties. Our PL mapping results (not shown here) for the transferred film confirm very uniform peak wavelength, peak intensity and full width at half maximum (fwhm) for the entire transferred thin film, indicating the robustness and feasibility of the thin film transferred and its readiness for subsequent device fabrication. The high quality of the transferred thin film is also confirmed by the high resolution XRD InP (002) rocking curve scanning (Figure 4b), in which there is no peak broadening for the transferred film as compared to the original epi-wafer.

It is also important to investigate whether we can get high quality transferred multiple quantum well (MQW) structures since MQWs are most important for most device applications using this low temperature bonding process. About 700 nm epitaxial structure including MQWs was grown on the InP substrate and then transferred to the Si substrate with 500 nm Si_3N_4 grown on it. Figure 5a shows low magnitude TEM image of the bonded MQWs structure and (b) shows the high magnitude TEM image in the MQWs region. There is no obvious defect observed in the MQWs. XRD θ–2θ scans of the as-grown and the bonded MQWs structures are shown in Figure 5c, in which (004) peak of Si and InP substrate was set for reference. The nearly unchanged XRD satellite order and fwhm of InP (004) peak indicate that the structure integrity of MQWs is preserved and no significant defects are generated during the bonding process. This is in agreement with our TEM measurement as there is no defect propagating toward the MQWs during the bonding process.

Conclusions

In summary, we have successfully demonstrated a low temperature process to transfer the InP-based epitaxial film to the Si substrate with Si_3N_4 intermediate layer, which is a key enabling technology in heterogeneous photonic integration. The bonding energy is higher than the InP fracture energy as measured by the pulling test. The high crystalline quality of the bonded thin film is preserved, as examined by photoluminescence and high resolution XRD measurements. TEM characterization has shown an intimately bonded interface with no observable defects generated near the bonding interface. A thin layer of amorphous oxide of about 6 nm thickness has been detected at the bonding interface. This highly hydrophilic interface obtained by O_2 plasma activation helps to achieve high quality bonding.

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