Wafer fusion technique features for near-IR laser sources

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Abstract. The paper presents the results of studies of the conditions for the formation of A3B5 compound semiconductors heterointerfaces including InP, InGaAsP and GaAs layers. The heterostructures were grown by molecular-beam epitaxy and were fused by wafer fusion technique. Improvement of planarity and homogeneity over the thickness of heterointerface due to using optimized preliminary preparation of semiconductor wafer surfaces was demonstrated. No additional extended defects such as dislocations were found.

1. Introduction
Nowadays, there are various techniques for a semiconductor wafers bonding, which can be divided in general into the direct wafer bonding technology and the wafer bonding with inter-mediate layer technology. The wafer bonding technology with an inter-mediate layer includes many bonding methods such as adhesive, thermocompression, metal, transient liquid phase and a few others wafer bonding techniques. These methods have found their application in CMOS technologies and hybrid integration in silicon photonics [1, 2], but they are not suitable for a near-IR laser structures based on A3B5 compound semiconductors, due to huge losses inside the formed layers. Here in, an absorption of light and a light scattering on layers defects occur, and the resonant wavelength shifts as well. On the other hand, an alternative approach is the direct wafer bonding technology which includes the molecular wafer bonding (wafer fusion) and is suitable for bonding of silicon, quartz, and also for hetero materials such as GaAs and InP [3]. This method is based on a high temperature annealing of the wafers at a certain orientation relative to each other and under significant pressure. One of the important features for a successful intermolecular bonding of wafers by van der Waals forces with the wafer fusion is a necessity of elimination of void spaces fulfilled with an air. Thus, the preliminary preparation for a removal of organic contaminants and oxides from surfaces of wafers is an important task.

The wafer fusion of GaAs and InP heterostructures with a typical pressure of 3 kPa – 3 MPa and temperatures of 550–650 °C can be carried out both in a chemically active liquid medium (liquid bonding) and in an oxygen-free medium (dry bonding) [4]. In turn, the use of negligible internal pressure (<10⁻⁵ mbar) with a dry bonding promotes the effective removal of volatile compounds formed on the surface of structures during heating [5].
In this paper we present the results of a dry wafer fusion of GaAs and InP, GaAs and InGaAsP epitaxial layers grown on GaAs and InP substrates.

2. Method details
The studied epitaxial layers were grown by a molecular-beam epitaxy (MBE) using of Riber 49 machine. Wafer fusion was carried out with an EVG 510 bonding machine with a pneumatic press under a following conditions: temperature was ~ 600 °C, press pressure was ~ 35 kPa, time was 12 minutes. System of grooves for the removal of volatile compounds was not used because the fusion was carried out under a high vacuum condition which was 10^-6 mbar.

Due to the significant difference in the coefficient of thermal expansion of GaAs and InP, the fused structure undergoes elastic deformations, and upon rapid cooling the destruction of the fused wafers is possible [6]. Thus, we have chosen a slow cooling mode of the sample with a cooling rate not exceeding 10 °C/min. It is also possible to use a wafer fusion technique at lower temperatures (~ 350 °C) in a vacuum, but the probability of delamination of a fused sample is rather high [7].

The removal of organic impurities and oxides from the surfaces of epitaxial heterostructures was carried out using isopropanol, acetone and trichlorethylene. A deionized water was used for washing for 5 minutes, and subsequent drying was carried out in a stream of dry pure nitrogen. A removal of absorbed water from the surface of the wafers was carried out by a heating tile.

The fusion interfaces were analyzed using transmission electron microscopy (TEM). To carry out studies by TEM it is required to carry out a special preparation of the samples to make samples transparent enough for electrons. Thus, the structures were prepared in the cross-sectional configuration using preliminary mechanical thinning and following sputtering with 4-keV Ar+ ions at a glancing angle to the surface.

3. Experiment
At the first stage, the analysis of TEM images of the fused GaAs/InGaAsP interfaces demonstrated a presence of a large number of void spaces (Figure 1) in the amorphous layer at the fused interface both from the InGaAsP side and from the GaAs side. We assume that this amorphous layer consists of oxides of A3B5 compounds on the fused surface and consists of adsorbed water molecules. The thickness of the planar part of the GaAs/InGaAsP fused interface was about 3 nm and in case of void spaces was up to 10 nm. Following optimization of the preliminary preparation of the fused surfaces was aimed to eliminating voids and achieving a uniform amorphous layer.

![Figure 1. TEM images of fused GaAs/InGaAsP heterointerfaces with void spaces](image)

We have optimized the preliminary preparation of the surfaces of the wafers in an oxygen plasma and in etchants HCl:H2O for InP (InGaAsP) layers and HF:H2O for GaAs layers to remove surface oxides from these wafers. Also, we have increased the temperature of the preliminary surface annealing of wafers at 200 °C and the duration up to 5 minutes for a more effective removal of
adsorbed water molecules. The adjustments made it possible to ensure high planarity of the boundaries and uniformity in thickness in the region of the fused interface of GaAs/InP (Figure 2). The fused interface of GaAs/InP was about 5 nm. Also, TEM studies did not reveal any misorientation of the GaAs and InP lattices in the (110) cross-sectional plane.

![Figure 2](image2.png)

**Figure 2.** TEM images of fused heterointerfaces with optimized preliminary preparation GaAs/InP

It is known that due to the difference in lattice parameters, the fused interface must contain edge misfit dislocations [5]. In our case, we can assume that the dislocations lie in an amorphous layer, which removes the fields of elastic stresses from these dislocations. For this reason, the misfit dislocations will not be visible in the TEM images of the fused interface.

It is also known that the fused interface is not always an amorphous layer, but also superimposed crystal lattices with multiple dislocations. Furthermore, an increase in fusion temperature leads to a mutual diffusion and a formation of an intermediate InGaAsP layer which is about ~10 nm thick [8]. Also, it should be mentioned that the addition of an InGaAsP layer to a n-InP layer in terms of fabrication of near-IR laser sources decreases optical losses [8] and makes it possible to simplify the formation of a low-resistance ohmic contact to such a layer.

We have overgrown a n-InP layer with a InGaAsP layer using a MBE technique for a subsequent annealing in order to effectively remove oxides from the overgrown InGaAsP surface. The temperature of annealing has been lowered to 525 °C due to optimized etchant HCl:H2O. The temperature of 540 °C, which was previously used to annealing in MBE setup, leads to an overheating of the sample surface and to an increase in the roughness of the overgrown surface. The resulting GaAs/InGaAsP fused interface demonstrated in figure 3 had a thickness of 1-2 nm and a uniform amorphous boundary.

![Figure 3](image3.png)

**Figure 3.** TEM images of fused GaAs/InGaAsP heterointerface with optimized preliminary preparation
4. Conclusion
We have demonstrated an improvement in planarity and uniformity over the thickness of the boundaries of heterointerfaces using optimized preliminary preparation of surfaces of semiconductor wafers. It was achieved due to an effective removal of oxide films and adsorbed water molecules from wafers’ surfaces. TEM studies have proved that extended defects, such as dislocations, do not propagate into the bulk of the fused wafers. We assume that optimization of preliminary wafer preparation for a subsequent fusion makes it possible to create near-IR laser sources based on A3B5 material systems using only MBE and dry wafer fusion technique. We believe that obtained results is of interest to create hybrid structures Si/A3B5 which can be used in hybrid integration silicon photonics.

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