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Low temperature direct wafer bonding of GaAs to Si via plasma activation

C. Y. Yeo,¹ D. W. Xu,¹ S. F. Yoon,¹ and E. A. Fitzgerald²

¹*School of Electrical and Electronic Engineering, Nanyang Technological University, Singapore 639798, Republic of Singapore*

²*Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, USA*

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The present work seeks to demonstrate the elegance and simplicity of monolithic integration via plasma-activated direct wafer bonding. Two-inch gallium arsenide and silicon wafers were directly bonded through argon plasma activation. The highest specific bond energy was found for plasma conditions of 30 s, 120 mTorr, and 200 W, followed by low temperature annealing at 140 °C, and was 478 mJ/m². Through this process, a processed silicon integrated circuit could be integrated with optoelectronics gallium arsenide on a wafer scale. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4791584>]

Unlike heterogeneous growth, monolithic integration *via* wafer bonding technology does not experience lattice and crystal mismatch issues. Hence, different types of materials can be monolithically integrated using various forms¹ of this technology, and high performance devices can be realized.² Most of the pioneering work on wafer bonding was based on silicon-to-silicon (Si-to-Si). It has been demonstrated that annealing temperatures in excess of 800 °C under a normal ambient condition are required in the conventional wet activation bonding of Si-to-Si to achieve strong, covalently bonded interfaces.³ However, the bonding of a pair of thick dissimilar materials substrates through high temperature annealing to achieve strong bonds is not feasible because of the existence of an unavoidable thermal mismatch barrier, especially between III-V compounds and Si. The annealing of such materials at temperatures exceeding 100 °C often results in debonding and/or fracture.⁴

Fortunately, several techniques are available that achieve a high bond energy at low temperature such as plasma activated wafer bonding,^{5,6} bonding under vacuum,^{7,8} and ultraviolet (UV) activated wafer bonding.^{9,10} By far, direct wafer bonding carried out under ultra-high vacuum (UHV) has proven to be the technique most able to achieve an extremely high bond energy, even at room temperature.¹¹ However, the implementation of this sophisticated system with a long duration to reach vacuum levels low enough to obtain absorbates-free bonding surfaces so that a covalently bonded interface can be directly achieved seems impractical from a manufacturing perspective. Alternatively, wafer bonding *via* plasma activation can achieve a relatively high bond energy at a low temperature. Tan *et al.*¹² attributed the enhanced bonding achieved in plasma activation to surface roughening caused by ion bombardment that increased the surface area and, hence, the number of silanol (Si-OH) groups. Moreover, because of the accompanying effect of increasing porosity in the oxide layers, the out-diffusion of water molecules (H₂O) was facilitated. This out-diffusion promoted Si polymerization reactions according to the equation

Most of the research on wafer bonding has been devoted to homogenous Si-to-Si systems, and the reported work on the monolithic integration of gallium arsenide (GaAs) with Si through direct wafer bonding is often on a small scale of dimension in thickness or size.^{13,14} Lehmann *et al.*⁴ directly bonded 3-in. GaAs to 4-in. Si and reported a low bond energy of 200 mJ/m² after annealing at 140 °C. In the present work, a high specific bond energy of thick GaAs-to-Si bonded substrates on a wafer scale was achieved through argon (Ar) plasma activation and parameter optimization without the addition of any intermediate layer/s. The current effort seeks to provide an inexpensive, simple, and robust solution for the monolithic integration of GaAs and Si.

In this study, 2-in. (100) P-type Si wafers with a thickness of $275 \pm 25 \mu\text{m}$ were directly bonded to 2-in. (100) N-type GaAs wafers with a thickness of $450 \pm 25 \mu\text{m}$. The Si wafers were first cleaned with a solution containing NH₄OH:H₂O₂:H₂O in a ratio of 0.25:1:5 by parts at 70 °C for 10 min,¹⁵ rinsed thoroughly in deionized water, and dried under a flow of nitrogen gas. The GaAs wafers were pre-cleaned and individually packaged by the manufacturer (AXT, Inc.). Pairs of GaAs and Si wafers were then treated with plasma in an AST Products Inc. Descum-Clen100 plasma system with Ar. The chamber was first purged down to less than 25 mTorr before Ar gas was introduced. Subsequently, the desired pressure was stabilized for 1 min before being discharged. After the plasma treatment, the wafer pairs were rinsed thoroughly in deionized water and dried under a flow of nitrogen gas. Next, they were bonded at room temperature with point loads at their centers. Finally, all the bonded wafers were annealed at 140 °C for 24 h in a convection oven.

A statistical approach of a full 2³ (2 levels of low and high associate with each of 3 factors in consideration) factorial with two replicates was employed to assess the influences of three plasma parameters on the wafer pairs: (1) exposure time, (2) pressure, and (3) power. These parameters were abbreviated as factors A, B, and C respectively. The low level (−) values of these parameters were taken as 5 s, 120 mTorr, and 100 W, respectively, while the high level (+) values were taken as 30 s, 200 mTorr, and 200 W, respectively. These values are presented in Table I.

TABLE I. The values of plasma parameters investigated.

Level	Factor code		
	A exposure time	B pressure	C power
Low (–)	5	120	100
High (+)	30	200	200

Table II presents the factorial design matrix, which consisted of 8 different plasma conditions runs. Each run involved a particular combination of the low and high levels of the three plasma factors (see Table I). Two replicates were performed for each run. For example, in Run 5 of Table II, 2 GaAs wafers and 2 Si wafers were each subjected to the same plasma conditions of a low level of exposure time and pressure (5 s and 120 mTorr, respectively) and a high level of power (200 W) to obtain two bonded pairs (Replicate 1 and 2).

The post-annealing specific bond energy was determined using the crack opening method¹⁶ as

$$\gamma = \frac{3t_b^2 E_{Si} t_{Si}^3 E_{GaAs} t_{GaAs}^3}{16l_c^4 (E_{Si} t_{Si}^3 + E_{GaAs} t_{GaAs}^3)}, \quad (2)$$

where E_{Si} and E_{GaAs} denote Young's moduli and t_{Si} and t_{GaAs} denote the thicknesses of the Si and GaAs wafers, respectively. The thickness of the blade t_b was measured to be 106 μm , and the crack length l_c was determined from infrared (IR) optical transmission images. Contact angle measurement through a video contact analyzer and an atomic force microscope (AFM) were used to characterize the plasma-treated Si and GaAs surfaces.

The post-annealing specific bond energy results for the bonded wafers are presented in Table III. The average post-annealing specific bond energy of each experimental run in Table III was determined by taking the average of the results of Replicate 1 and 2. The table shows that most of the high bond energy results were obtained using a power of 200 W. Pairs from Run 1 to 4 that were subjected to a plasma power of 100 W debonded after annealing at 140 °C for 24 h. The analysis of variance (ANOVA) was computed,¹⁷ and the results revealed that all three plasma parameters affected the bond energy significantly with plasma power having the largest influence (see Table IV). Moreover, all factor interaction, especially those which involved plasma power, had

TABLE II. The design matrix of the 2³ factorial with two replicates.

Run	Factor level		
	A exposure time	B pressure	C power
1	–	–	–
2	+	–	–
3	–	+	–
4	+	+	–
5	–	–	+
6	+	–	+
7	–	+	+
8	+	+	+

TABLE III. The post-annealing specific bond energies of the bonded wafers.

Run	Post-annealing specific bond energy (mJ/m ²)		
	Replicate 1	Replicate 2	Average
1	66	0 (debond)	33
2	0 (debond)	0 (debond)	0 (debond)
3	0 (debond)	0 (debond)	0 (debond)
4	0 (debond)	0 (debond)	0 (debond)
5	371	337	354
6	369	478	424
7	0 (debond)	0 (debond)	0 (debond)
8	422	387	405

significant effects on the bond energy. The collective average post-annealing specific bond energy was subsequently determined for each of the levels of each of the plasma factors to reflect each degree of effect. For example, Run 1 to 4 were associated with a low level of power while Run 5 to 8 were associated with a high level of power (Table II). The collective averages of the power factor were then determined from the results listed in Table III of Replicate 1 and 2 in Run 1 to 4 (a low power level) and of Replicate 1 and 2 in Run 5 to 8 (a high power level). These averages were 8 mJ/m² for the replicates in Run 1 to 4, and 296 mJ/m² for the replicates in Run 5 to 8. The same was done for exposure time and pressure factor. The results are shown in Fig. 1.

As observed in Fig. 1, the specific bond energy increased significantly when the plasma power was changed from 100 W to 200 W. Table III shows that nearly all debond failures were derived from surfaces subjected to the low power level of 100 W in Run 1 to 4. In contrast, high bond energy results were found on surfaces subjected to the high power level of 200 W in Run 5 to 8.

In addition, the contact angle measurements revealed that Si and GaAs surfaces subjected to a plasma power of 100 W were not as hydrophilic as those exposed to 200 W. The water droplet silhouettes on Si and GaAs surfaces that were subjected to a power of 100 W reflected a relatively poor surface hydrophilicity as a result of a low number of surface hydroxyl (–OH) groups (similar to the example shown in Fig. 2(b)). Contact angle measurements could not be made on the surfaces treated with a power of 200 W

TABLE IV. The analysis of variance (ANOVA) results.

Source of variation	F _o ^a	P-value ^b	Significance
Exposure time (A)	41.71	0.0002	Extreme
Pressure (B)	35.19	0.0003	Extreme
Power (C)	282.41	<0.0001	Extreme
Exposure time-pressure (AB) factor interaction	28.94	0.0007	Extreme
Exposure time-power (AC) factor interaction	55.09	<0.0001	Extreme
Pressure-power (BC) factor interaction	24.68	0.0011	Extreme
Exposure time-pressure-power (ABC) factor interaction	19.49	0.0022	Average

^aF_o is the F-test statistic.

^bP-value is the probability that a result changes due to experimental noise.

FIG. 1. The collective average post-annealing specific bond energy associated with each plasma parameter.

because water droplets wetted the surfaces to such large extents that their silhouettes could not be observed (similar to the examples shown in Figs. 2(c) and 2(d)). The extremely low wetting angles of less than 5° indicated that the surfaces were highly saturated with $-\text{OH}$ groups. The results clearly show that a critical minimum plasma power is required before surfaces are sufficiently activated to achieve good bonding.

In conjunction with the work of Michalske and Bunker¹⁸ and Tan *et al.*,¹² it is highly possible that a high plasma power of 200 W is capable of inducing damages to the native oxides of Si and GaAs to deem them highly strained and reactive. This may result in a high number of surface gallium arsenide hydroxyl (GaAs-OH) and Si-OH groups. Upon annealing, they could then polymerize in a similar fashion as shown in Eq. (1) as

and result in a high number of interfacial covalent oxygen bridges between GaAs and Si (GaAs-O-Si).³ Moreover, the

FIG. 2. The silhouette images and contact angles of water droplets on GaAs surfaces with (a) untreated reference, and plasma treatments associated with experimental (b) Run 1, where Run 2, 3, and 4 had similar results, (c) Run 5, where Run 6 had a similar result, and (d) Run 7, where Run 8 had a similar result. The silhouette images and the contact angles of the water droplets on the Si surfaces were similar to those shown here, respectively.

high energetics of the Ar species of 200 W could also increase the porosity in the native oxides that will aid in H_2O out-diffusion and enhance the polymerization reaction of Eq. (3) further to yield a higher number of GaAs-O-Si bridges. A high number of interfacial GaAs-O-Si bridges is desired, especially in a highly thermal mismatch bonding system, to restrain peeling stress during annealing and prevent debond failure.¹⁶ On the other hand, when bonding surfaces are subjected to 100 W of plasma power, most of the Ar species possess energies that are too low to damage and alter the surface chemistry of Si and GaAs surfaces. This results in inadequate activation. Consequently, a lower number of surface GaAs-OH and Si-OH groups and inefficient H_2O out-diffusion occur, and the number of interfacial GaAs-O-Si is reduced. Thus, the bond energy is too low to restrain peeling stress during annealing at 140°C , and it leads to debond failure.

These conjectures agree with the statistical results in Table IV. These results also indicate that exposure time-power and pressure-power factor interaction affect the bond energy significantly. Run 1 to 4 in Table III show that changes in exposure time and pressure did not affect the bond energy when the plasma power was at its low level of 100 W. This was due to the low energetics of the Ar species at this low level of power that was insufficient to bombard surfaces to a large degree. Therefore, exposure time and pressure did not have any further influences on the surfaces. In Run 5 to 8 in Table III, where Si and GaAs surfaces were exposed to high plasma power level of 200 W, the energetics of the Ar species was high enough to cause surface damage. Consequently, the effects of exposure time and pressure could take place to affect the surface morphology and chemistry and result in bond energy changes. Hence, it is necessary for the plasma power to be raised to a sufficiently high level before adequate activation, and the effects of exposure time and pressure can start to take place.

Fig. 1 reveals that the specific bond energy decreased when the plasma pressure increased from 120 mTorr to 200 mTorr. This result seems inconsistent with the intuition that a stronger plasma power achieved through a higher pressure should yield a higher bond energy. The contact angle measurements revealed that superhydrophilic states were observed for Si and GaAs surfaces treated under the pressures of 120 mTorr and 200 mTorr at a power of 200 W. These were similar to the examples shown in Figs. 2(c) and 2(d), which indicated a high number of surface GaAs-OH and Si-OH groups. However, the AFM 3D images of Si and GaAs showed that the condition of 200 mTorr yielded rougher surfaces (like those shown in Fig. 3) than the condition of 120 mTorr, especially for GaAs surfaces.

These findings indicated that although a stronger plasma effect through higher pressure will induce a high number of surface GaAs-OH and Si-OH groups, which could potentially be polymerized into GaAs-O-Si bridges as per Eq. (3), the bond energy will not necessarily be high because the joining surfaces will be atomically rougher. Consequently, most of these oppositely GaAs-OH and Si-OH groups could not come in close proximity enough to allow polymerization reactions to occur. With a lower number of interfacial GaAs-O-Si bridges to hold GaAs and Si bonding pair together,

FIG. 3. The AFM 3D images of (a) and (b) Si surfaces and (c) and (d) GaAs surfaces. In (a) and (c), the surfaces were treated with plasma as per experimental Run 7 with an exposure time of 5 s. In (b) and (d), the surfaces were treated with plasma as per experimental Run 8 with an exposure time of 30 s. All runs used a pressure level of 200 mTorr and a power level of 200 W.

peeling stress will dominate during annealing at 140 °C and result in a lower bond energy.

Fig. 1 shows that the specific bond energy increased when the plasma exposure time increased from 5 s to 30 s. The effect of the plasma exposure time is further demonstrated by comparing the results from Run 7 and 8 in Table III. These two runs had exposures of 5 s and 30 s, respectively, at 200 mTorr and 200 W. The former wafers debonded while the latter exhibited a much higher bond energy. Superhydrophilic states, as shown in Figs. 2(c) and 2(d), were associated with Si and GaAs surfaces subjected to these two sets of plasma conditions. These superhydrophilic states implied that the surfaces were equally strongly activated and that they were highly saturated with $-OH$ groups. However, the AFM 3D images (Fig. 3) revealed that rough surfaces formed under high plasma pressure could be smoothed to a certain extent through a prolonged exposure time.

These findings suggest that a prolonged plasma exposure time could compensate for the negative effect of surface roughening induced by exposure to a higher plasma pressure. Consequently, a larger number of oppositely GaAs-OH and Si-OH groups can then come in close proximity to polymerize and yield a higher number of interfacial GaAs-O-Si bridges that hold GaAs and Si bonding pair tightly. This would result in a higher bond energy and prevent debond failure caused by high peeling stress during annealing at 140 °C. Hence, it is essential that the plasma pressure and exposure time be at low and high levels, respectively, to prevent extensive surface roughening, which will lead to bond energy deterioration.

In sum, the plasma conditions of 30 s, 120 mTorr, and 200 W were found to yield the highest bond energy of GaAs-to-Si bonded wafers among the combinations of parameters investigated. The associated highest specific bond energy was 478 mJ/m², which is double the energy level reported elsewhere.⁴ It is recommended that the approach outlined in this study be extended to lower and higher values of plasma

exposure time, pressure, and power, so as to obtain a full understanding of the effects of these parameters on monolithic integration through plasma activation wafer bonding.

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