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Aluminum Nitride to Aluminum Nitride Direct Wafer Bonding

by Kimberley A Olver

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14. ABSTRACT
A wafer-bonding process based on plasma surface activation of sputter-deposited thin film aluminum nitride (AlN) was studied. The purpose of this investigation and process development was to eventually use AlN as a bonding medium between Metal Organic Chemical Vapor Deposition-grown gallium nitride material and diamond substrate material.

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aluminum nitride, AlN, direct wafer bonding, surface activation, Metal Organic Chemical Vapor Deposition

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1. Introduction

A method was developed for the direct wafer-to-wafer bonding of aluminum nitride (AlN) to AlN-coated wafers. Direct wafer bonding is the hybridization of two surfaces without the use of an adhesive (epoxy) medium. This type of bonding relies primarily on van Der Waals forces, as opposed to ionic or covalent bonding. Direct wafer bonding requires smooth, flat, hydrophilic surfaces that are able to be activated with appropriately charged hydrogen molecules on the prepared surfaces.

For our purpose, a 20-nm-thick film of AlN was chosen as the wafer-to-wafer intermediate bonding material due to its heat-dissipation property. The goal of the program was to directly bond a Metal Organic Chemical Vapor Deposition (MOCVD)-grown, semipolar, 369.5-nm-wavelength InGaN/AlGaN multiple quantum well (MQW) Vertical External Cavity Surface Emitting Laser (VECSEL) structure to thermally conductive and transparent diamond substrate to allow pumping with an electron beam and lasing through an external cavity. One important part of this program was to develop a fabrication process for AlN direct bonding and then to utilize this capability to bond our VECSEL GaN structures to chemical vapor deposition (CVD) diamond substrates. Thermal management is the biggest challenge for high-power lasers, and direct bonding to a CVD diamond submount would greatly improve the heat spreading associated with this structure while also maintaining UV transparency. The AlN-allowable thickness was calculated and found to be 20 nm or less (Fig. 1).

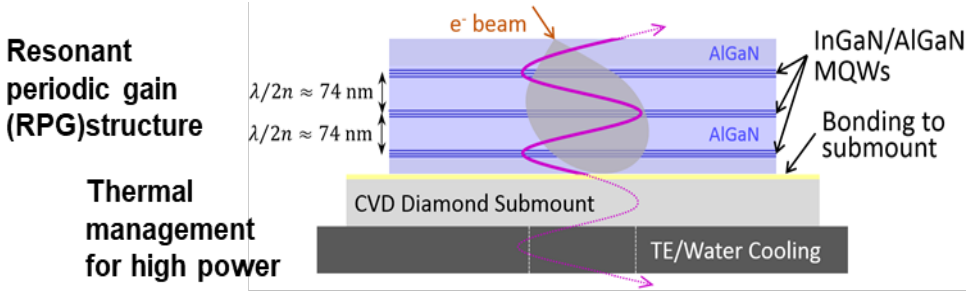


Fig. 1 Diagram of structural design

Most of the research literature found dealt with silicon (Si) and silicon dioxide (SiO_2) wafer bonding. Tong and Gösele[1] studied direct wafer bonding of Si-on-insulator (SOI) structures and the effect that wafer preparation had on bondability. They discussed hydrocarbon removal, trapped interface bubbles (voids), and whether a hydrophilic or a hydrophobic activated surface was advantageous over the other. Goetz and Dawson[2] researched low-temperature direct bonding of semiconductor materials such as Si, indium arsenide, and gallium arsenide (GaAs),

using SiO_2 or silicon nitride (Si_3N_4) as the bonding material. They studied how to enhance the "reaction bonding" by activating clean, very smooth (>1 nm) bonding coatings using varying ion-gas mixtures containing oxygen (O) and fluorine (F) molecules. They found improvement in bonding in the temperature range of 25 to 400 °C with an O-to-F activation ion ratio of 4 to 1. And, Bao et al.[3] studied AlN-to-AlN direct bonding on Si wafers, focusing on the activated AlN film stoichiometry and how the stoichiometry affects the overall bondability and interface void formation. They showed a nearly void-free, enhanced-strength bond between Si wafers by applying a 3-h, 300 °C anneal posthybridization.

2. Procedure

Initially, clean 2-inch Si wafers were used in the development of a process for AlN wafer-to-wafer bonding. After successful bonding with the Si wafers, 2-inch single-side polished sapphire wafers were then attempted. The sapphire wafers were semitransparent, allowing us to see how complete the wafer bonding was and if voids were forming. After success with sapphire wafers, and following the developed process, we attempted to bond both 1-cm^2 pieces of sapphire wafer, as well as MOCVD-grown GaN–InGaN structures to sapphire, using the same procedure (Table 1).

Table 1 Process steps for AlN-to-AlN direct wafer bonding

Step	Process	Detail
1	Initial solvent clean	Sonicate in acetone, isopropyl, methanol, rinse in DiH_2O, N_2 dry
2	RCA Clean	(RCA1; $5DiH_2O/1NH_4OH/1H_2O_2$) (BOE) (RCA2; $6DiH_2O/1HCl/1H_2O_2$)
3	AlN Sputter Deposition	500 C/ Al target/ 50sccm N_2/ 8000W bias/ no RF
4	AlN Activation	35 C, 60s 50sccm O_2+10sccm Ar, 40mT, 120W RF, 1500W ICP; 30s 5sccm SF_6, 20sccm Ar, 20mT, 120W RF, 1500W ICP; 30s 7sccm SF_6 + 10sccm Ar, 20mT, 120W RF, 1500W ICP; 30s 10sccm SF_6 + 10sccm Ar, 20mT, 120W RF, 1500W ICP
5	DiH_2O Spray Rinse	DiH_2O Spray, N_2 dry
6	Wafer Bonding	200C, N_2 gas flow, 6000Tor, 3 hrs.

2.1 Initial Solvent Cleaning

The wafers were ultrasonically cleaned in acetone followed by isopropyl and methanol, then rinsed in deionized water (DiH_2O) and blown dry using nitrogen gas (N_2). The acetone removed organic impurities, and a subsequent rinse in isopropyl removed the contaminated acetone.[4]

2.2 RCA Cleaning

The wafers were cleaned using the standard "RCA cleaning" sequence. RCA1 consisted of the removal of organic contaminants using a bath solution of 5 parts DiH_2O, 1 part ammonium hydroxide, and 1 part hydrogen peroxide. The solution was brought to 80 °C and the wafers were immersed for 10 min. The wafers were then rinsed in DiH_2O and blown dry. An optional step at this point was used for removal of thin oxides using buffered oxide etch (hydrofluoric acid: H_2O, 1:6) and immersing for 30 s to 1 min, followed by a DiH_2O rinse. RCA2 was the removal of ionic contaminants using a bath solution of 6 parts DiH_2O 1 part hydrochloric acid, and 1 part hydrogen peroxide. The solution was brought to 80 °C and the wafers were immersed for 10 min. This was followed by a DiH_2O rinse and drying with N_2.

2.3 AlN Sputter Deposition

Directly after cleaning, the wafers were placed into an Evatec Clusterline 200 Sputter System (CLC 200) for an AlN deposition. Aluminum was sputter deposited under a reactive N_2 atmosphere onto the precleaned wafer surface. Approximately 200 angstroms of AlN were deposited during a 5-s deposition using the following deposition conditions: 500 °C chamber, Al target, 50 standard cubic centimeters per minute (sccm) of N_2, 8000-watt bias power, and no RF bias.

2.4 AlN Surface Activation

Following the AlN deposition, the wafer surfaces were activated using an Oxford Plasma Lab 100 ICP Etcher. The AlN-coated wafers were placed onto a clean carrier wafer, AlN side up, and introduced into the etch chamber under vacuum. The activation recipe consisted of a total of four plasma etch steps: a 60-s oxygen plasma followed by a series of three sulfur hexafluoride (SF6) plasmas, each lasting 30 s and increasing in plasma volume. The conditions were 1) a 60-s O_2 plasma at 50 sccm mixed with 10 sccm of argon, pressure at 40 mT, 35 °C chamber, 120 watts of RF, 1500 watts of inductively coupled plasma (ICP); 2) a 30-s SF6 plasma at 5 sccm with 20 sccm of argon, at 20mT of pressure, 120 watts of RF, 1500 watts of ICP; 3) SF6 at 7 sccm mixed with 10 sccm of argon; and 4) SF6 at 10 sccm mixed with 10 sccm of argon. The temperature in the chamber remained constant at 35 °C for the entire process.

2.5 DiH_2O Spray Rinse

After activating the AlN layers, the wafers were immediately sprayed with DiH_2O to remove particulates and as a test of the hydrophilic nature of the surface. The wafers were then blown dry with N_2.

2.6 Wafer Bonding

Next, the wafers were placed into a Karl Suss SB8 Wafer Bonder, and a thermo-compression bonding mode was used. The process program was written using the following conditions: a bonding temperature ramp from room temperature to 200 °C under a N_2 gas flow, followed by a force of 6000-Tor pressure applied for 3 h while holding the elevated temperature, and finally a ramp-down in temperature to room temperature before releasing the force. The complete bonding cycle took 8 h.

The wafers were removed from the wafer bonder and inspected for voids.

3. Results and Discussion

In this fabrication development, the smoothness of the deposited ALN film was critical in avoiding void formation. A void would show up as an area that was not bonded (see Fig. 1). Voids can form from either trapped gas or trapped debris. Because the AlN film is very thin (20 nm or less) it was important to make sure the original surface was also as smooth and as defect free as possible. We were successful in bonding AlN-coated silicon wafers and AlN-coated sapphire wafers, but success on the diced 1-cm^2 pieces of AlN-coated sapphire (Fig. 2) and the AlN-coated, 0.5- × 1-mm patterned GaN structures to AlN-coated sapphire submounts proved difficult. Our original purpose in developing this direct wafer-bonding process was to fabricate 1-mm^2 circular mesas on the MOCVD-grown GaN parts by first depositing ALN on the cleaned surface, then patterning, etching, and cleaning the resultant device mesas, and finally processing them through direct wafer bonding with AlN-coated sapphire substrates (Fig. 3a). In all, we were only able to get partial bonding from these samples. This was partially attributed to particulate matter imbedded in the MOCVD-grown layers as well as debris that we could not remove post-dicing. We also concluded that for our purpose the bonding strength was not enough to hold the processed parts together due to the small contact area the 1-mm^2 mesas comprised (Fig. 3b).

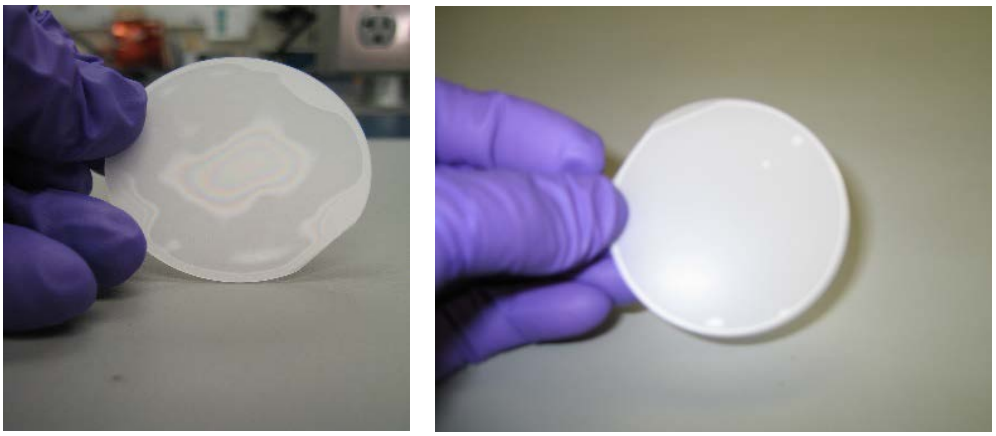
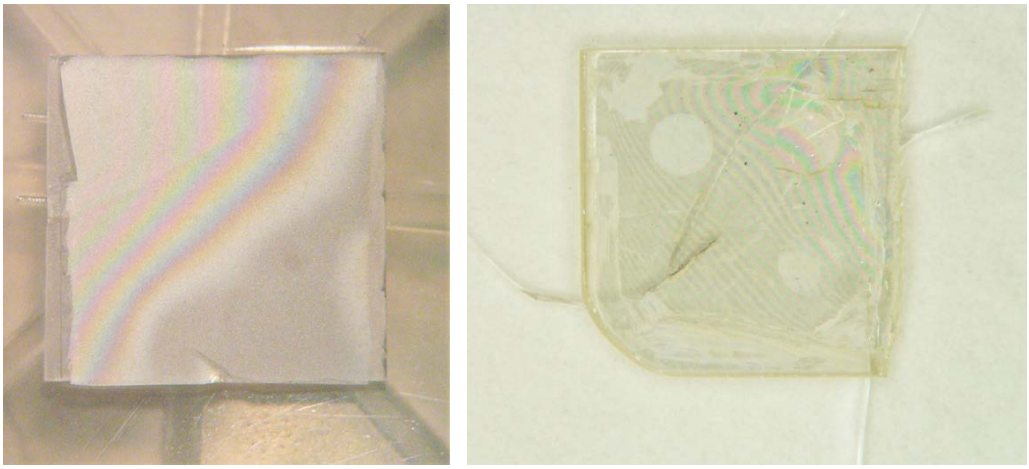


Fig. 2 Image on left shows void formation between two AlN-coated sapphire wafers postbonding; image on right shows nearly void-free, AlN-coated sapphire wafers postbonding



(a) (b)

Fig. 3 a) 1-cm^2 sapphire pieces after direct wafer bonding; b) 1-mm^2 MOCVD-grown GaN mesas partially wafer bonded to sapphire substrate

3.1 Smoothness and Cleanliness

Wafer bonding requires wafers with clean surfaces that are free of particulate, organic and metallic contaminations. This is important because the surface cleanliness has a direct effect on both the structural and electrical properties of the bonding interface, as well as on the electrical properties of the bonded materials.[1] Wafers are deformed around particles on the bonding surfaces, leaving circularly unbonded interface areas or bubbles. Our concern was ultimately the optoelectronic property of the bonded parts and not electrical properties; but, overall, the cleanliness played a major role. Atomic force microscopy (AFM) was performed on the AlN-coated sapphire as well as the AlN-coated GaN.

3.2 AlN Deposition

As important as the initial cleaning of the wafers was for particle-free bonding, the deposition of the AlN was also of concern. Initially, we used a CVC 610 Sputter System to deposit AlN onto sapphire. But after failed attempts, a closer inspection of the coated surface showed metallic particles on the surface after deposition. A change in sputter equipment to the CLC 200, and fine tuning the deposition parameters, gave a surface in which any particulates measured were less than 1 nm in height, as seen in the AFM scan in Fig. 4.

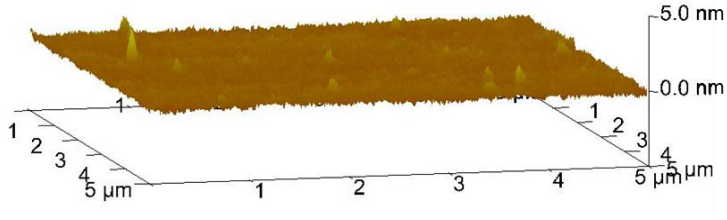


Fig. 4 AFM scan of CLC 200 sputter-deposited AlN film on clean sapphire

3.3 Plasma Activation

Surface activation is necessary for wafer bonding to occur. The surface of one wafer should be terminated by an appreciable density of electrophilic hydrogen molecules and the molecules on the mating wafer surface should have nonbonding electrons. A hydrogen (H) atom can participate in a hydrogen bond if it is bonded to oxygen, nitrogen or fluorine because H–F, H–O, and H–N bonds are strongly polarized, leaving the hydrogen atom with a positive charge. This electrophilic H has a strong affinity for nonbonding electrons so that it can form intermolecular attachments with an electronegative atom such as oxygen, nitrogen, or fluorine.[1] Goetz et al.[2] reported in their patent that the bond energy for the surfaces activated by oxygen and or fluorine ion bombardment are over three times greater than the bond energies of surfaces activated by the standard water activation. Effectively, reaction-bonded components in which surfaces were activated by bombardment with oxygen ions, fluorine ions, or a mixture of oxygen and fluorine ions may be bonded at significantly lower temperatures down to room temperature.[2]

Bao et al[3] studied the surfaces of the AlN using X-ray photoelectron spectroscopy, and concluded that after plasma activation, nitrogen bound to aluminum (Al–O and Al–OH) was found. The emergence of Al–O bonds can be attributed to two reasons: 1) surface exposure to H_2O and/or O_2 and annealing accelerated oxidation; and 2) plasma-activation-induced bond defects, which facilitate oxygen incorporation to aluminum.[3] We did not anneal our AlN thin films, but we did add SF6 into our activation plasma chemistry. The fabrication process included ion bombardment in

gas plasma to activate the AlN surface using O_2 and SF6, where the fluorine-containing ions helped to enhance the bond energy of the surface hydroxyl groups.

3.4 DiH_2O Spray Rinse

An intense spray with DiH_2O and drying with N_2 high-pressure gas just prior to placing the wafers into the Suss Wafer Bonder removed contaminates and particulates that may have been deposited during or after the AlN deposition.

Directly after activation and DiH_2O rinse, we were able to bond the silicon wafers together by hand at room temperature—without the use of the Suss Wafer Bonder. The sapphire wafers were also bonded directly after activation and DiH_2O rinse using the program developed on the Wafer Bonder. However, we had difficulty bonding either the diced 1-cm^2 pieces or the mesa structures to sapphire after the activation and bonding steps.

4. Conclusion

Sputter-deposited aluminum nitride films (20 nm) were deposited onto clean 2-inch silicon as well as 2-inch sapphire wafers for the purpose of investigating the possibility of AlN-to-AlN direct-wafer bonding. Bonding was achieved after thin film deposition, surface activation, and application of temperature and pressure in a nitrogen atmosphere for the 2-inch Si wafers as well as the 2-inch sapphire wafers. We also used this newly developed process to attempt bonding of diced sapphire pieces and smaller GaN parts with mesa structures. However, we found that smaller parts were difficult to bond.

The results of this study show that surface preparation and smoothness and the creation of an activated surface are crucial in obtaining bonded surfaces. Area of the bonding parts may also be a consideration in the activation and direct bonding of two surfaces.

5. References

1. Tong QY, Gösele U. Semiconductor wafer bonding: recent developments. Mat Chem Phys. 1994;37(2):101–127.
2. Goetz G, Dawson W, inventors. Low temperature reaction bonding. United States patent US 5,427,638. 1995 June 27.
3. Bao S, Lee KH, Chong GY, Fitzgerald EA, Tan CS. AlN-AlN layer bonding and its thermal characteristics. ECS J Sol State Sci Tech. 2015;4(7):200–205.
4. Substrate cleaning adhesion promotion. MicroChemicals Application Notes; 2013 Nov 7 [accessed 2016 Dec 1]. www.microchemicals.com.

Bibliography

Maszara WP, Goetz G, Caviglia A, McKitterick JB. Bonding of silicon wafers for silicon-on-insulator. J App Phys. 1988;64(10):4943–4950.

Schulte J, Sobe G. Magnetron sputtering of aluminum using oxygen or nitrogen as reactive gas. Thin Solid Films. 1998;324(1–2):19–24.

Takagi H, Kikuchi K, Maeda R. Surface activated bonding of silicon wafers at room temperature. Appl Phys Lett. 1996;68:2222.

Weinert A, Amirfeiz P, Bengtsson S. Plasma assisted room temperature bonding for MST. Sens Act A: Physical. 2001;92(1–3):214–222.

List of Symbols, Abbreviations, and Acronyms

AFM	atomic force microscopy
AlN	aluminum nitride
CVD	chemical vapor deposition
DiH_2O	deionized water
F	fluorine
GaAs	gallium arsenide
GaN	gallium nitride
H	hydrogen
ICP	inductively coupled plasma
MOCVD	Metal Organic Chemical Vapor Deposition
MQW	Multiple Quantum Well
N_2	nitrogen gas
nm	nanometer
O	oxygen
RF	radio frequency
sccm	standard cubic centimeters per minute
SF6	sulfur hexafluoride
Si	silicon
Si_3N_4	silicon nitride
SiO_2	silicon dioxide
SOI	Si on insulator
UV	ultraviolet
VECSEL	Vertical Cavity Surface Emitting Laser

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