Sapphire direct bonding as a platform for pressure sensing at extreme high temperatures

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ABSTRACT

Direct bonding between two epitaxy-ready (EPI polished) sapphire wafers is demonstrated as the basis for an all-sapphire pressure sensor. Through chemical processing, hydrogen pre-bonding, and a final high-temperature bakeout, the two single-crystal wafers are directly bonded without the use of any adhesive or intermediate layer. Dicing across the edge of the structure and inspection of the diced pieces with a scanning electron microscope (SEM) indicates a successful direct bond. Control of the bonding wave generates an air bubble sealed between the two bonded sapphire wafers. Optical interference-based measurements of the bubble height and shape at pressures from 0 to 60psig prove that the bubble is sealed by the bonded wafers and demonstrate the potential for sapphire direct bonding as a means of constructing an all-sapphire pressure sensor. Since the structure contains no adhesives, such an all-sapphire sensor is ideal for pressure sensing in extremely harsh, high-temperature environments, potentially operating at temperatures over 1500°C.

Keywords: sapphire, direct bonding, pressure sensor, high temperature, harsh environments

1. INTRODUCTION

Pressure sensing is a key component of process control and optimization, but no sensor technology currently exists to monitor dynamic pressure in extreme high temperature environments, such as jet engines or coal gasifier furnaces. Conventional semiconductor pressure sensors are limited to low temperature ranges, and even silicon-on-insulator (SOI) designs can only operate at temperatures below 500°C [1]. Several SOI-based pressure sensors have been constructed from silicon carbide (SiC), but they are limited to the same temperature range [2-4]

Optical interrogation can extend the range of available materials to include silica glass, SiC, cubic zirconia, and sapphire. All-silica glass sensor designs avoid problems due to mismatched coefficients of thermal expansion, and have the potential for miniaturization. Such a sensor, made directly on a single-mode fiber tip, has been demonstrated at 710°C [5]. These sensors are ultimately limited to temperatures below 1000°C, at which point the glass begins to soften [6].

Advanced optical sensors, made in part with a higher-temperature material such as SiC or sapphire, have been attempted to push the thermal limit imposed by silica glass. Bonding of the high-temperature membrane, is a major challenge to the development of this type of technology; the introduction of adhesives can severely limit the thermal operating range. One solution, which relies on anodic bonding between a SiC membrane and silica glass body, has been suggested for operation up to 1100°C [7], and tested at 600°C [8]. Anodic bonding is seen as a non-ideal solution because it requires that at least one material along the interface be silica glass [9].

Sapphire is an extremely hard, corrosion resistant crystal with a melting point of over 2000°C, which makes it an ideal material for sensing in high-temperature, harsh environments. Thin, single-crystal sapphire optical fibers have shown the ability to resist plastic deformation at temperatures up to 1650°C [10]. An all-sapphire pressure sensor could therefore be expected to produce reliable pressure measurements at temperatures over 1500°C.

Sapphire direct bonding, a process by which two separate pieces of single-crystal sapphire are permanently bonded without the use of adhesives or intermediate material layers [11, 12], is demonstrated as the basis for such an all-sapphire pressure sensor. Through a process of chemical preparation and high temperature baking, a set of epitaxy-ready (EPI polished) sapphire wafers, is bonded to produce an all-sapphire pressure sensor prototype. SEM imaging of the interface shows good bond quality, and pressure testing illustrates the structure's potential as a functional sensor.

Fiber Optic Sensors and Applications VI, edited by Eric Udd, Henry H. Du, Anbo Wang, Proc. of SPIE Vol. 7316, 73160Y

2. SENSOR MATERIALS & DESIGN

2.1 Sensor Materials

This paper presents a prototype of an all-sapphire based pressure sensor, based on highly polished sapphire wafers. Intended for epitaxial growth of GaN and other thin films, these "EPI-polished" sapphire wafers are commercially available at low cost. The c-plane wafers used in this demonstration have a mean surface roughness of $R_a \leq 1$nm, which is critical to successful direct bonding. The wafers are bonded via atomic diffusion at high temperature, creating a pressure sensor prototype which contains no adhesives or intermediate material layers.

Prior to sensor construction, the wafers, which commonly come in 2" or 4" rounds, are diced into 10mm square pieces. This reduces the effect of wafer bow and warp, helping to eliminate gaps between the pieces during bonding. Ultimately a technique could be developed to bond the large wafer discs, allowing multiple sensors to be constructed prior to dicing. This potential for mass-production is seen as an additional advantage of the use of EPI sapphire wafers.

2.2 Pressure Sensor Design

The sensor prototype demonstrates the potential for sapphire direct bonding to create a functional pressure sensor from two sapphire wafers. Ultimately, a fully-developed sensor would look like the device in Fig. 1, shown to illustrate the full potential and intended use of the direct bonding technique. The all-sapphire sensor is a simple Fabry-Perot cavity that generates accurate, high-resolution pressure measurements. The key to its operation is a small cavity, sealed in a sapphire base and capped with a thin sapphire diaphragm. The diaphragm flexes in response to changes in external pressure, and optical interferometric methods are used to sense the changing depth of the cavity.

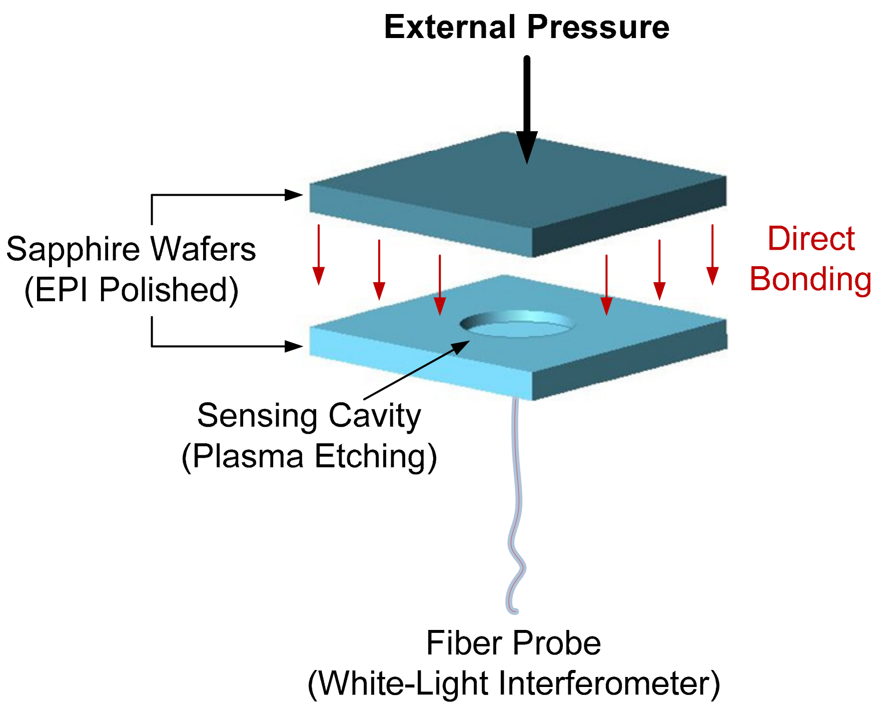

Fig. 1. The potential design for an all-sapphire pressure sensor is based on a Fabry-Perot cavity. Interrogation of the sensor could be performed using sapphire optical fiber and white-light interferometry.

White-light interferometry is suggested as the best method of interrogating such a sensor because of its absolute measurement capability, high accuracy, and relatively high speed [13, 14]. A combination of sapphire fiber, standard silica fiber, and free-space optics could be used to couple the optical signal to the sensor Fabry-Perot cavity. Sapphire fiber is an ideal coupling option because it can survive in the same harsh environment as the sensor body itself, and it can be conveniently packaged. The fiber end could be butted up against the polished base of the sensor and held in place via physical compression, fusion bonding using a CO2 laser, direct bonding, or adhesive bonding techniques.

2.3 Sensor Construction

Construction of the sensor is envisioned as a two-step process: etching of a sensing cavity in the base wafer, and direct bonding of the upper wafer to form a sealed pressure sensor. Cavity etching would likely be performed using conventional photolithography and dry etching techniques. High powered inductively-coupled plasma (ICP) etching, with Cl_2/BCl_3 or similar chemistry, can achieve etch rates of 380-550 nm/min in single-crystal sapphire [15, 16]. Because of its comparatively experimental nature, the direct bonding process is seen as the critical step in sensor construction, motivating the development of the prototype sensor presented in Section 3.

3. SAPPHIRE DIRECT BONDING

Direct bonding relies on atomic diffusion to bond two separate crystal structures without the use of adhesives. Initially developed for silicon wafer bonding [9], a unique direct bonding process has also been developed for sapphire [11, 12]. This work, originally demonstrated on large, rectangular sapphire pieces, has been adapted to bond the thin wafers required for pressure sensor construction.

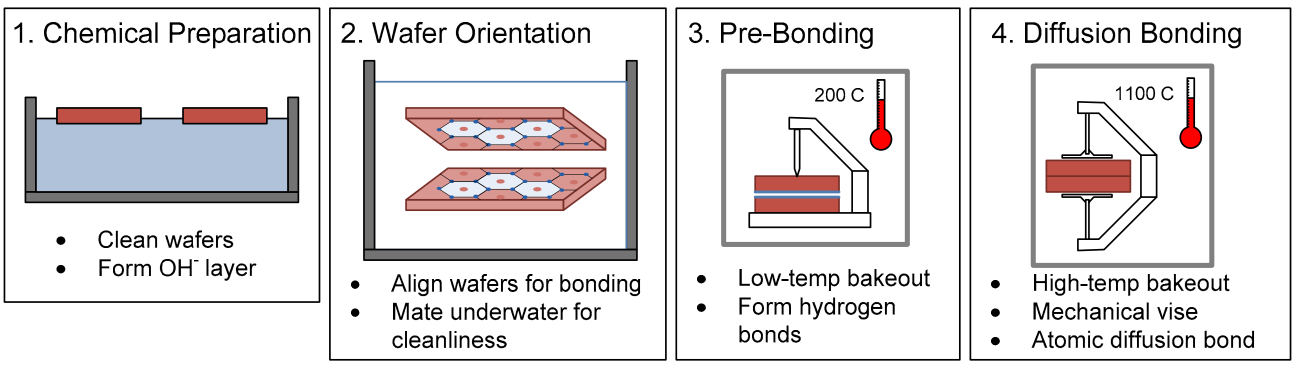

Fig. 2. Summary of sapphire direct bonding process used to build the prototype sensor. The process was adapted for thin wafer bonding.

3.1 Chemical Preparation and Pre-Bonding

A 2” round sapphire wafer is diced into 10mm square pieces to reduce the effects of wafer warp, and the pieces are thoroughly cleaned using the RCA cleaning technique. Careful dicing relative to the a-axis alignment flat ensures that the wafer pieces can be mated with their crystalline axes aligned.

The remaining steps follow the sapphire direct bonding process originally developed for creating large composite Ti:Sapphire crystals for use in high-power pulsed lasers [11]. The wafer pieces are immersed in 85% H_3PO_4, heated to 150^{o}C, for 30 minutes. This step is designed to remove any oxide layer left on the wafers after cleaning.

The final chemical preparation step is a soak in a diluted H_2SO_4, which deposits a hydrophilic OH^- layer on the wafer surfaces. Next, the wafers are immediately aligned and mated, and great care is taken not to contaminate the mating surfaces during this step. In order to ensure the cleanest-possible interface, the two wafer pieces are carefully mated underwater, directly in the H_2SO_4 solution, and removed as a pair. After removal from the solution, small adjustments are made to the alignment of the wafers by pushing them against a simple alignment plate. The entire preparation and pre-bonding process is performed in a Class-1000 clean room.

A preliminary hydrogen bond, enabled by the deposition of the OH^- layer, is generated by baking the wafer pair at 200^{o}C for 30 minutes. Such an OH^- pre-bond has also been proven beneficial in silicon wafer direct bonding [17]. The pre-bond is performed in a small clamp, which applies a small amount of pressure to the center of the wafers. This device is designed to generate an outwardly-propagating bonding wave, similar to the precautions taken in silicon direct bonding [18]. After the pre-bond is complete, the wafers are removed from the clean room environment and taken to a high-temperature furnace for final bonding.

3.2 Final Direct Bonding

The pre-bonded wafers are placed under a 2kg weight and baked at 1100°C for 50 hours. The applied mechanical pressure works to eliminate any gaps due to wafer warp, and the long process time allows atoms to break free from their locations in the lattice and migrate across the interface to the opposite wafer piece. This process is governed by the general equation of atomic diffusion (1), in which *r* represents position, and *c* and *D* are the concentration and diffusion coefficient a particular type of atom [19].

$$\frac{\partial c(r,t)}{\partial t} = D\nabla^2 c(r,t) \tag{1}$$

Assuming isotropic diffusion in three dimensions, the mean-squared diffusion distance $\langle r \rangle^2$of a particular atom can be surmised from the solution to the diffusion equation (2). Sapphire, Al_2O_3, contains aluminum and oxygen atoms, which diffuse at rates of $D \sim 10^{-20}$ m^2/s and 10^{-22} m^2/s, respectively, at 1100°C. The 50-hour bakeout is estimated to cause a mean diffusion of 100nm for aluminum atoms, enough distance to create a strong bond between the wafers [20].

$$c(r,t) = \frac{1}{\left(4\pi Dt\right)^{3/2}} \exp\left[\frac{-r^2}{4Dt}\right] \tag{2}$$

$$\left\langle r^2 \right\rangle = 6Dt \tag{3}$$

3.3 Generation of a Prototype Pressure Sensor

As discussed in Section 3.1 in reference to the hydrogen pre-bond, it is believed that the pattern of applied pressure has a significant effect on the shape and quality of the resulting bond. Fig. 3 shows a schematic of the weight, which consists of a cast alumina base and a set of steel rods inside of an alumina cup. The bottom surface of the cup is slightly concave, resulting in a ring-shaped contact area on the surface of the upper wafer.

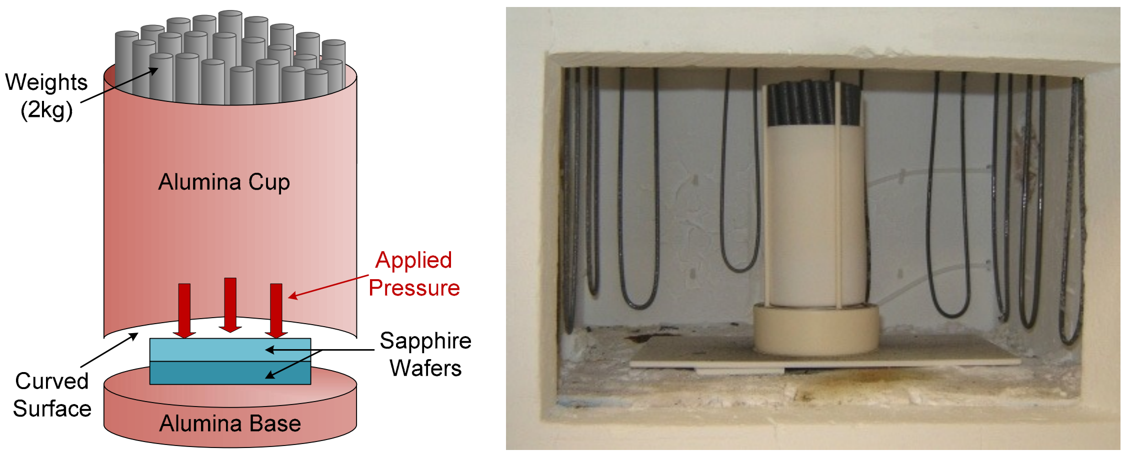

Fig. 3. Direct bonding high-temperature bakeout. (Left) schematic of bonding vise showing concave under-side of weighted cup. (Right) image of vise and sapphire wafers inside of the furnace chamber.

The resulting bonded structure (Fig. 4) contains a visible bubble in its center, which is a result of concave bottom surface of the weight. The increased mechanical pressure applied to the edges of the square wafer pieces caused the bond to form more completely, and the lack of compression in the center of the wafers left a large un-bonded void. This sealed cavity forms a prototype pressure sensor, the outer surface of which flexes in response to changes in external pressure.

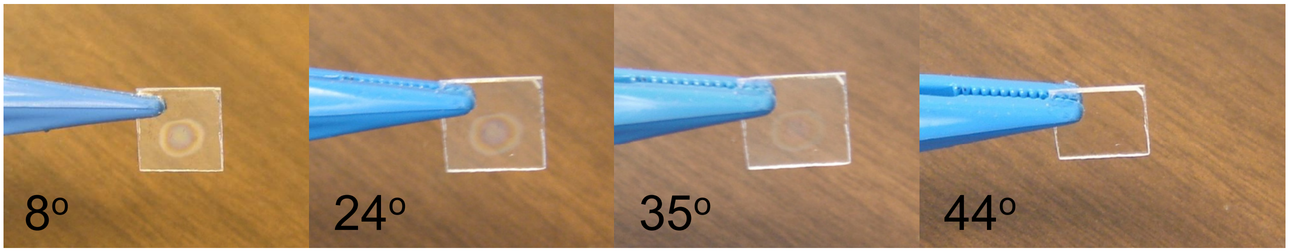

Fig. 4. Direct bonded sapphire pressure sensor prototype. The sensing bubble is a result of non-uniform mechanical pressure during bonding. Inspection of the structure from different angles indicates a slightly domed inner bubble surface.

The presence of colored interference rings, visible in the structure from several angles, indicates a slightly domed bubble shape. These Newton rings provide a clear qualitative measurement of the bubble's size and shape, roughly 6.5mm in diameter. During testing, the interference pattern is used to quantitatively map the bubble height, as well as observe the effects of changing ambient pressure on the sensing bubble.

3.4 Mapping of Prototype Sensor Bubble

Measurement and testing of the prototype sensor bubble were performed using colored Newton Rings, which are clearly visible in Fig. 4. White-light illumination was chosen over monochromatic Newton rings testing to isolate the interference between the inner surfaces of the bubble. Because of its short coherence length, the white light does not produce an interference pattern when reflected off of the top and bottom of the 660µm-thick bonded wafer pair.

Images were collected with a high-resolution B&W camera, fitted with a blue lens filter to improve fringe visibility. The effective source wavelength, determined by the light source, filter, and CCD responsivity spectra, is λ_{eff} = 468nm. The spatial optical interference pattern is demodulated to produce an accurate 3D map of the bubble height (Fig. 5), which shows a peak cavity height of 0.49µm at atmospheric pressure.

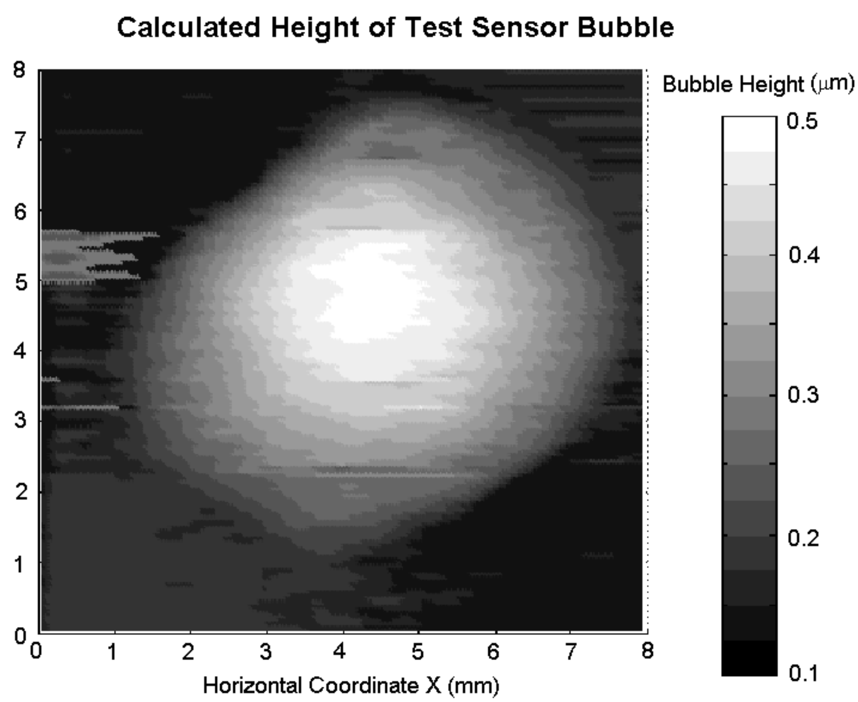

Fig. 5. Newton rings mapping of sensor bubble at atmospheric pressure. (Left) White-light interference rings, visible from filtered B&W camera system. (Right) Map of prototype sensor bubble height at atmospheric pressure. The peak bubble height is calculated to be 0.49µm.

4. PROTOTYPE SENSOR TESTING & RESULTS

4.1 Bond Quality Testing

In order to inspect the interior bond quality, the prototype sensor was placed in a dicing saw, and two cuts were made. First, the corner was cut off; next a 1.8mm-wide strip was diced off the bottom edge of the bonded wafer pair. Stresses from the dicing blade and mounting plate caused the thin strip to break during the cutting process. Visual inspection of the broken piece shows that the break occurred cleanly across the bonded interface (Fig. 6). This result agrees with Sugiyama's earlier work, in which direct-bonded sapphire pieces were purposely broken as proof of bond quality [11].

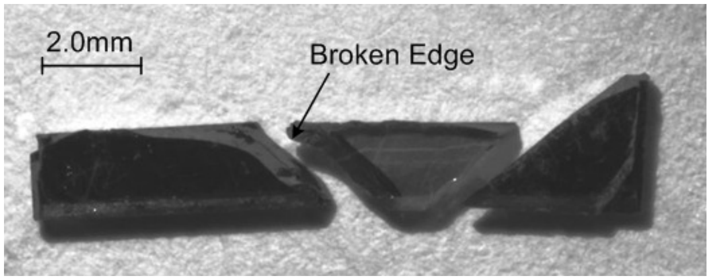

Fig. 6. Diced pieces of bonded wafer pair. The bottom corner and edge of the prototype sensor were diced to reveal the interior bond edge. During dicing, the long edge piece broke cleanly in half, indicating a strong bond.

After dicing, the interior edge was coated with a thin metallic film and imaged under the scanning electron microscope (SEM). Fig. 7 shows the diced edge of the long rectangular piece, at 50x, 3000x, and 11.6kx magnification levels. The visible edge in Fig. 7 was diced parallel to the a-axis alignment flat of the original round wafer. At 3000x magnification, the bond interface is barely visible above the roughness from the dicing saw. The wavy bond interface and lack of large void regions are both strong indicators that atomic diffusion has taken place between the two wafer surfaces. At 11.6kx, the interface is visible as a string of 200nm-scale pits. These pits may illustrate the limit of the bond quality: small voids exist between the two wafers, but these voids do not appear to be deeply inter-connected.

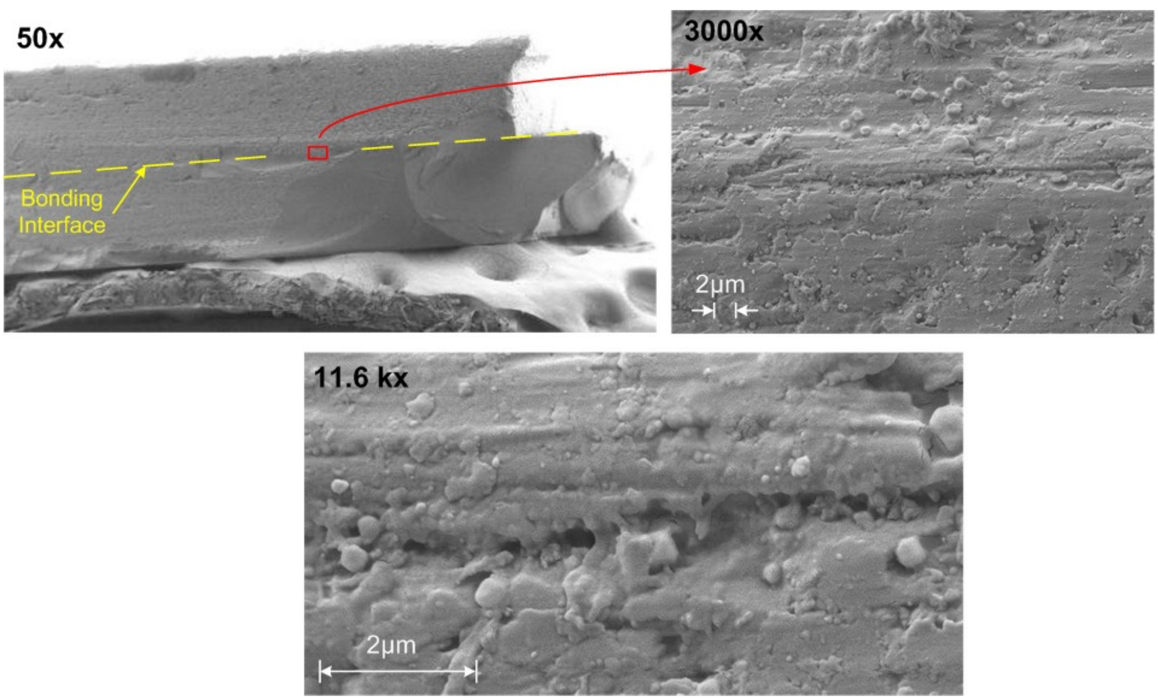

Fig. 7. SEM images of interior bonded edge. These images illustrate the bond quality along the diced a-axis edge.

In order to enable more detailed inspection, the diced edge of the triangular piece was polished with a series of diamond-grit papers, finishing with 0.5µm mean surface roughness. Shown under an optical microscope at 50x, the corner piece illustrates how the bond quality changes from the wafer's edge to its center. In Fig. 8, the bond interface is clearly visible at the left edge of the triangular piece, only to disappear toward the center.

Fig. 8. Polished interior bonded edge: this 50x microscope image clearly shows the bond interface between the two wafers, which is prominent near the left edge and becomes invisible toward the center of the cut, polished piece.

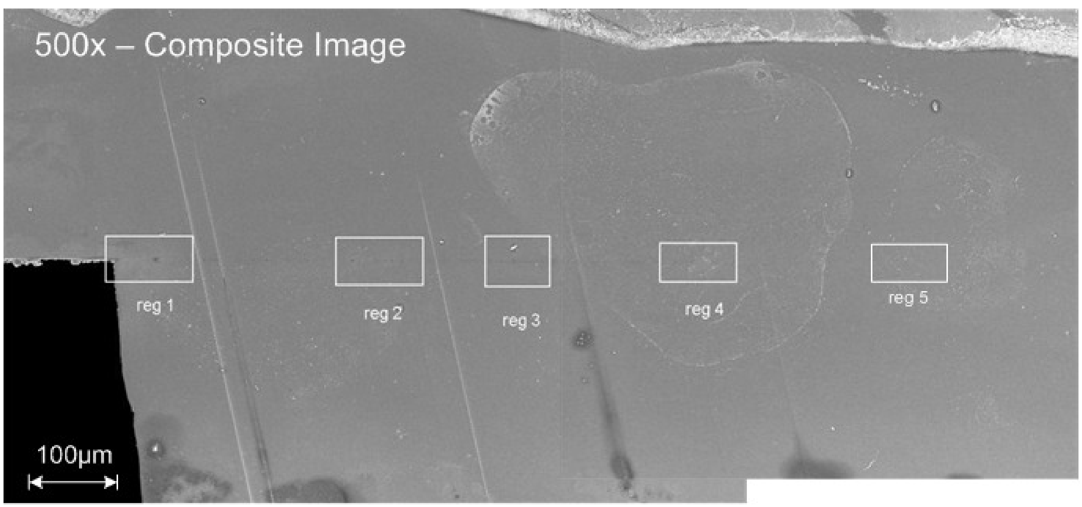

Fig. 9. SEM image of polished bonded edge. This composite image, taken at 500x magnification, shows the bond quality improving toward the center of the triangular diced edge piece.

These conclusions are confirmed in the SEM images, which show a higher quality bond toward the interior of the wafer set. Fig. 9, taken at 500x, shows the bond interface fading from left to right. Details of regions 1,2 and 4 at 20kx confirm that the bond interface does improve towards the center of the bonded wafer. The high quality bonded area is visible only by small voids of less than 100nm.

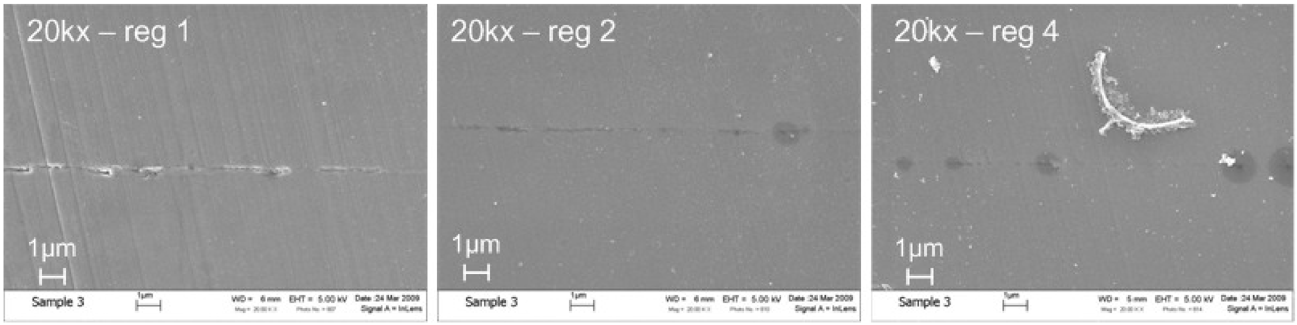

Fig. 10. High magnification SEM images of polished bonded edge. These detail images show the bond interface becoming fainter toward the center of the wafers. Most voids in the bonded region are less than 100nm in size.

4.2 Pressure Testing

The sensor was then placed in a pressure chamber and exposed to pressures from 0 to 60 psig, during which time seven Newton rings measurements were taken. Fig. 11 shows significant changes in interference pattern under increasing pressure. The slightly dimmer central spot, clearly visible in the atmospheric pressure images, disappears as the pressure is increased. At 60psig, only the outer ring is visible, indicating that the domed center of the bubble has been compressed into a relatively flat surface.

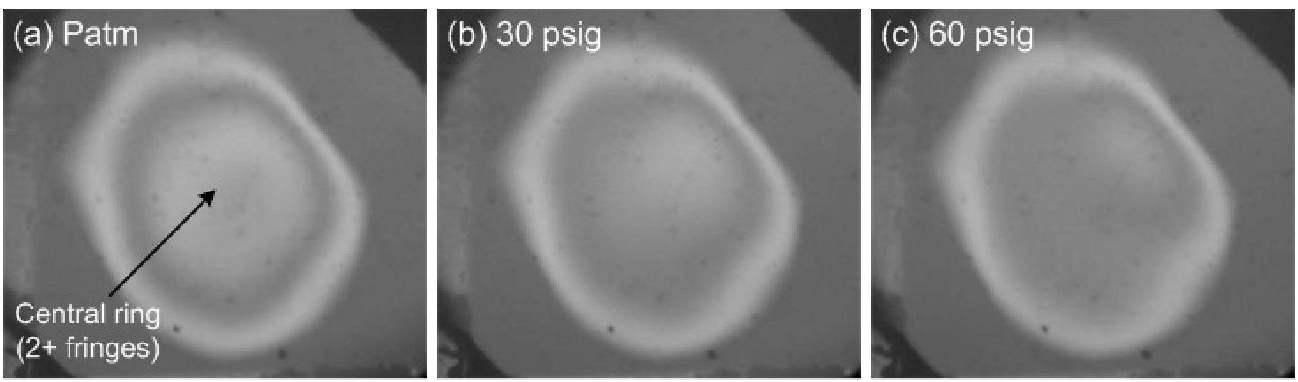

Fig. 11. Qualitative pressure test: visual inspection of Newton rings shows significant changes to the central height of the domed bubble under increasing pressure. The dim central spot in (a) indicates 2+ fringe orders at atmospheric pressure. This spot disapears in (b), leaving only a bright 1+ fringe order at 30psig. Further increases in pressure continue to reduce the height of the central dome (c) to very near 1 full fringe.

Full 3D mapping of the bubble surface was not attempted during pressure testing because of the difficulties posed by the small number of visible fringes. The presence of only one or two fringes in the pressurized images makes it difficult to accurately determine the envelope of the sinusoidal fringe pattern. At atmospheric pressure (Section 3.4), it was assumed that the bubble had a domed shape, with no change in concavity. The obvious deformations of the bubble under pressure invalidate this assumption during pressure testing. Fig. 11 is therefore presented as proof that the bubble surface deflects significantly under changes in atmospheric pressure, which indicates the presence of a sealed cavity between the two sapphire wafers.

More accurate white-light sensor interrogation requires a deeper sensing cavity to generate additional visible fringes. A suggested cavity depth of 10-20μm [5] could be achieved through ICP etching. Such an increase in cavity depth would enable accurate spatial fringe detection (the Newton rings method, above), or single-point spectral fringe detection (fiber-based white-light interferometry).

5. CONCLUSIONS

Sapphire direct bonding has been demonstrated as a viable method for construction of an all-sapphire pressure sensor from commercially-available single-crystal sapphire wafers. Given sapphire's high thermal softening point and resistance to corrosive chemicals, such an all-sapphire pressure sensor has the potential to operate in extreme harsh environments, where no current pressure sensor technology can survive.

The process of direct bonding, adapted from a technique used to construct large Ti:sapphire crystals, has been applied to the application of thin wafer bonding. Manipulation of the bonding geometry, achieved through application of non-uniform pressure during baking, has resulted in a prototype pressure sensor, with a small bubble sealed in between the two wafers.

Mechanical dicing and breaking, SEM inspection of the bond interface, and pressure testing, all verify a high quality bond and sealed inner cavity. Testing of the prototype sensor clearly demonstrates the potential for sapphire direct bonding as a basis for pressure sensing in extreme harsh environments.

Future work may include etching of a deeper sensing cavity prior to bonding, to achieve an accurately measurable linear response over a particular desired pressure range. Optical interrogation methods, possibly through white-light interferometry, could be adapted to provide accurate real-time sensor demodulation.

ACKNOWLEDGEMENTS:

The authors would like to thank Dr. Akira Sugiyama of the Japan Atomic Energy Research Institute for his expertise and generosity in sharing the details of his work on sapphire direct bonding. Dr. Sugiyama's advice was instrumental in making this work possible.

Dr. Louis Guido of the Bradley Department of Electrical and Computer Engineering, Virginia Tech, lent his expertise in semiconductor processing. Dr. Gary Pickrell of the Department of Material Science & Engineering, Virginia Tech, assisted in interpreting the direct bonding mechanism and results.

Also deserving of thanks are Don Leber, Dr. Ali Hajjiah, Syed Ali, Bassam Alfeeli, Jiajun Wang, and Brian Scott for their help in wafer processing and imaging. All SEM images were taken at the ICTAS Nanoscale Characterization and Fabrication Laboratory, Virginia Tech.

Finally, the authors would like to thank the Bradley Fellowship Program, of the Bradley Department of Electrical and Computer Engineering at Virginia Tech, for supporting Evan Lally and his exploratory work in high temperature sapphire pressure sensor development.

REFERENCES

[1] Kurtz, A. D., Ned, A. A., Goodman, S. *et al.*, "Latest ruggedized high temperature piezoresistive transducers," NASA 2003 Propulsion Measurement Sensor Development Workshop, Huntsville, Alabama (2003).

[2] Ziermann, R., von Berg, J., Obermeier, E. *et al.*, "High temperature piezoresistive beta-SiC-on-SOI pressure sensor with on chip SiC thermistor," Materials Science and Engineering B-Solid State Materials for Advanced Technology, 61-2, 576-578 (1999).

[3] Eickhoff, M., Moller, H., Kroetz, G. *et al.*, "A high temperature pressure sensor prepared by selective deposition of cubic silicon carbide on SOI substrates," Sensors and Actuators a-Physical, 74(1-3), 56-59 (1999).

[4] Okojie, R. S., Ned, A. A., and Kurtz, A. D., "Operation of alpha(GH)-SiC pressure sensor at 500 degrees C," Sensors and Actuators a-Physical, 66(1-3), 200-204 (1998).

[5] Zhu, Y. Z., Cooper, K. L., Pickrell, G. R. *et al.*, "High-temperature fiber-tip pressure sensor," Journal of Lightwave Technology, 24(2), 861-869 (2006).
[6] Wang, A., Gollapudi, S., Murphy, K. A. *et al.*, "Sapphire-Fiber-Based Intrinsic Fabry-Perot-Interferometer," Optics Letters, 17(14), 1021-1023 (1992).
[7] Pulliam, W., "Micromachined, SiC fiber optic pressure sensors for high-temperature aerospace applications," SPIE, 4202, 21-30 (2000).
[8] Pulliam, W., Russler, P., and Fielder, R., "High-Temperature, High Bandwidth, Fiber-Optic, MEMS Pressure Sensor Technology for Turbine Engine Component Testing," SPIE, 4578, 229-238 (2002).
[9] Tong, Q.-Y., [Silicon wafer bonding technology : for VLSI and MEMS applications] Institution of Electrical Engineers, London, 1 (2002).
[10] Tong, L. M., Shen, Y. H., Chen, F. M. *et al.*, "Plastic bending of sapphire fibers for infrared sensing and power-delivery applications," Applied Optics, 39(4), 494-501 (2000).
[11] Sugiyama, A., Fukuyama, H., Sasuga, T. *et al.*, "Direct bonding of Ti : sapphire laser crystals," Applied Optics, 37(12), 2407-2410 (1998).
[12] Sugiyama, A., "Feasibility study of a direct bonding technique for laser crysals," SPIE, 4231, 261-268 (2000).
[13] Yu, B., Wang, A. B., and Pickrell, G. R., "Analysis of fiber Fabry-Perot interferometric sensors using low-coherence light sources," Journal of Lightwave Technology, 24(4), 1758-1767 (2006).
[14] Yu, B., Kim, D. W., Deng, J. D. *et al.*, "Fiber Fabry-Perot sensors for detection of partial discharges in power transformers," Applied Optics, 42(16), 3241-3250 (2003).
[15] Jeong, C. H., Kim, D. W., Kim, K. N. *et al.*, "A study of sapphire etching characteristics using BCl3-based inductively coupled plasmas," Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers, 41(10), 6206-6208 (2002).
[16] Jeong, C. H., Kim, D. W., Lee, H. Y. *et al.*, "Sapphire etching with BCl3/HBr/Ar plasma," Surface & Coatings Technology, 171(1-3), 280-284 (2003).
[17] Lai, S. I., Lin, H. Y., and Hu, C. T., "Effect of surface treatment on wafer direct bonding process," Materials Chemistry and Physics, 83(2-3), 265-272 (2004).
[18] Turner, K. T., and Spearing, S. M., "Modeling of direct wafer bonding: Effect of wafer bow and etch patterns," Journal of Applied Physics, 92(12), 7658-7666 (2002).
[19] Shewmon, P. G., [Diffusion in solids] McGraw-Hill, New York,(1963).
[20] Sugiyama, A., (private communication), (2007).