

## Research paper

Direct bonding of ALD Al<sub>2</sub>O<sub>3</sub> to silicon nitride thin films

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## ABSTRACT

Direct bonding is an advanced joining technique for bonding of silicon based surfaces at low temperature without any specific surface pretreatment. The main purpose of this work is to develop new techniques to enhance the fabrication process for nanofluidic systems for in situ transmission electron microscopy (TEM) by improving low temperature annealing bonding strength when using atomic layer deposition of aluminum oxide. We have investigated and characterized bonding of Al<sub>2</sub>O<sub>3</sub>-Si<sub>x</sub>N<sub>y</sub> (low stress silicon rich nitride) and Al<sub>2</sub>O<sub>3</sub>-Si<sub>3</sub>N<sub>4</sub> (stoichiometric nitride) thin films annealed from room temperature up to 600 °C without pretreatment prior to the pre-bonding. We find that bonding of Al<sub>2</sub>O<sub>3</sub>-Si<sub>x</sub>N<sub>y</sub> and Al<sub>2</sub>O<sub>3</sub>-Si<sub>3</sub>N<sub>4</sub> is favorable in a temperature range from room temperature to 600 °C. We report bonding strength of 1300 ± 150 mJ/m<sup>2</sup> comparable to and in some case even higher than that of other materials Al<sub>2</sub>O<sub>3</sub> can be bonded to. Preliminary tests demonstrating a well-defined nanochannel system with ~100 nm high channels successfully bonded and tests against leaks using optical fluorescence technique and transmission electron microscopy (TEM) characterization of liquid samples are also reported. Moreover, the current bonding method can be also used for further MEMS applications.

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## 1. Introduction

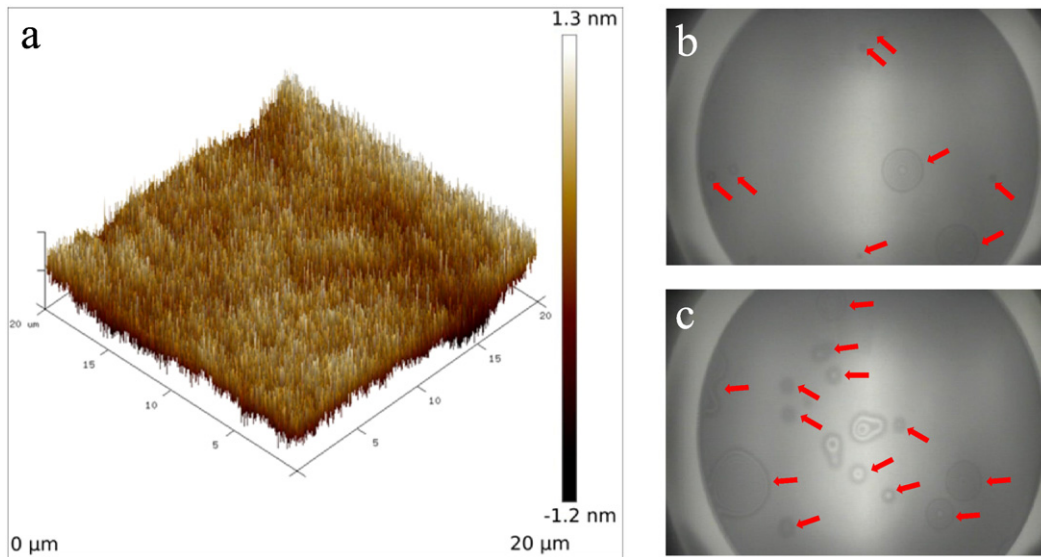
Direct bonding, also known as fusion bonding, was demonstrated in 1986 as an efficient method to bond silicon wafers [1,2]. The advantages of this technique have proven to be crucial for MEMS, microelectronics, micro/nanoreactors and optoelectronics device fabrication. Over the last decade integrating buried insulating material like SiO<sub>2</sub> in SOI substrate has been widely used and studied. However, due to the reduction in the length scale of many devices, SiO<sub>2</sub> substrates have shown some limitations such as leakage current or self-heating [3]. Thus replacement of SiO<sub>2</sub> by some other high-k dielectric material such as Al<sub>2</sub>O<sub>3</sub> and Si<sub>3</sub>N<sub>4</sub> is gaining importance [4,5]. The investigation of Si<sub>3</sub>N<sub>4</sub> as an SOI replacement has been studied since 1992 while since 2010 [6] Al<sub>2</sub>O<sub>3</sub> found some applications. Harendt et al. were the first to successfully bond silicon nitride (Si<sub>3</sub>N<sub>4</sub>) to silicon (Si) [7]. However, due to the difference in expansion coefficients between Si<sub>3</sub>N<sub>4</sub> and Si upon annealing cracks appear in the nitride layer. Fusion bonding of 1 μm thick low-stress Low Pressure Chemical Vapor Deposited (LPCVD) Si<sub>x</sub>N<sub>y</sub>-Si<sub>x</sub>N<sub>y</sub> was achieved by Sanchez et al. by polishing the surface to reduce the roughness from 2.9 nm down to 0.3 nm [8]. The bonded strength was found to be between 1.1 and 2.9 J/m<sup>2</sup> after 2 h at 1000 °C. More recently, Reck et al. have bonded Si<sub>3</sub>N<sub>4</sub>-Si<sub>3</sub>N<sub>4</sub> without any pretreatment and obtained bonding strength at 2 J/m<sup>2</sup> after several hours at 1150 °C. They also showed that exposure to buffered HF (bHF) during the pre-bonding cleaning, could lead to failure of the bonding due to absence of a thin

oxynitride layer [9]. In addition, Gui et al. have shown that the surface roughness is a critical factor to achieve a strong bond [8]. On the other hand little is presently known about atomic layer deposited (ALD) Al<sub>2</sub>O<sub>3</sub> substrate surfaces in terms of bonding performance. Suni et al. proposed for the first time bonding of ALD Al<sub>2</sub>O<sub>3</sub> with Si, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in a temperature range >400 °C [10]. They reported lower bonding strength at 900 °C due to a possible phase transition of Al<sub>2</sub>O<sub>3</sub> leading to unexpected formation of voids. Beche et al. characterized the bonding of ALD Al<sub>2</sub>O<sub>3</sub> to itself in detail highlighting de-bonding phenomena and formation of a high density of voids both at 400 °C and 800 °C [11]. ALD is a slower process than CVD, but here this is not an issue, since we need only 2, 3 or at most 10 nm thick Al<sub>2</sub>O<sub>3</sub> layers, which in a multi-wafer ALD system can be deposited almost as fast as similar CVD films, when the overhead for loading of the wafers is taken into account.

Recently, direct bonding to form nanofluidic channel devices has found several applications in electron microscopy (EM) for imaging of liquid samples thanks to the leak-proof capability offered by the bonding technique [12,13]. In this work we have investigated and characterized for the first time bonding of Al<sub>2</sub>O<sub>3</sub>-Si<sub>x</sub>N<sub>y</sub> and Al<sub>2</sub>O<sub>3</sub>-Si<sub>3</sub>N<sub>4</sub> thin films annealed from room temperature up to 600 °C without pretreatment prior to the pre-bonding. Direct bonding experiments were performed on both patterned and un-patterned wafers with a total bonded dielectric layer thickness of up to ~110 nm. Low aspect ratio nanochannels ( $h \ll w$ ) were successfully fabricated using this novel technique. The main purpose of our experiments is to develop new techniques to improve the fabrication process for nanofluidic systems for TEM technologies. ALD Al<sub>2</sub>O<sub>3</sub> improves bonding whenever the use of SiO<sub>2</sub> is not possible, for instance due to built-in compressive stress or if high anneal

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**Fig. 1.** a) AFM image of  $20\ \mu\text{m} \times 20\ \mu\text{m}$  surface area of 10 nm amorphous ALD  $\text{Al}_2\text{O}_3$  deposited on  $\sim 50\ \text{nm}$  of  $\text{Si}_3\text{N}_4$ . The RMS roughness was measured to  $R_a = 0.35\ \text{nm}$ ; b) and c) Infrared transmission image of b)  $\text{Si}_3\text{N}_4\text{-Al}_2\text{O}_3/\text{Si}_3\text{N}_4$  and c)  $\text{Si}_x\text{N}_y\text{-Al}_2\text{O}_3/\text{Si}_x\text{N}_y$ . Voids are indicated by red arrows. The height of the voids is indicated by the number of the Newtonian fringes.

temperatures should be avoided. The ALD  $\text{Al}_2\text{O}_3$  additionally offers novel surface chemistry for in situ liquid TEM experiments since it is also present in the channel and is a relevant surface for many applications such as studies of catalytic processes.

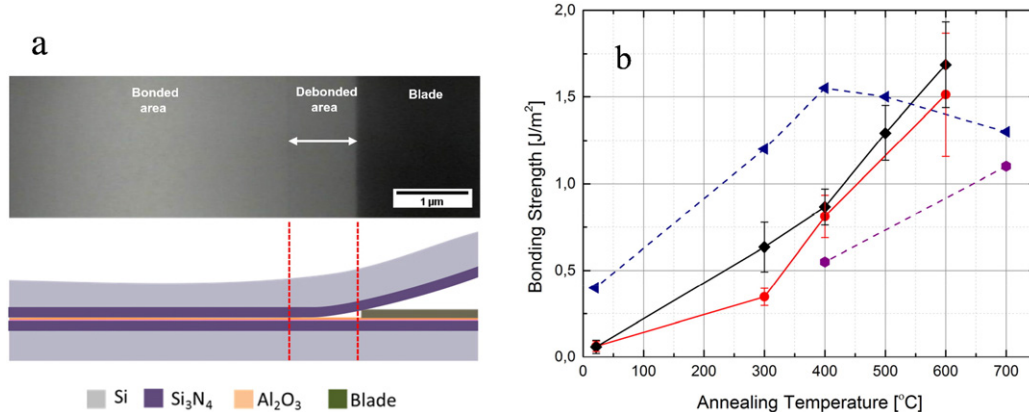
## 2. Fabrication

Different layer architectures were fabricated in order to reveal the bonding capability of the ALD  $\text{Al}_2\text{O}_3\text{-Si}_x\text{N}_y$  and ALD  $\text{Al}_2\text{O}_3\text{-Si}_3\text{N}_4$  architecture. Approximately 50 nm thick  $\text{Si}_x\text{N}_y$  (low stress silicon rich) and  $\text{Si}_3\text{N}_4$  (stoichiometric nitride) were grown by LPCVD using trichlorosilane and ammonia at  $830\ ^\circ\text{C}$  on standard boron doped double polished 100 mm,  $350 \pm 15\ \mu\text{m}$  thick (100) silicon wafers. Amorphous ALD  $\text{Al}_2\text{O}_3$  films of 10 nm were deposited at  $250\ ^\circ\text{C}$  using tri-methyl-aluminum (TMA) and water as active precursors. The surface quality of  $\text{Al}_2\text{O}_3$  and  $\text{Si}_x\text{N}_y/\text{Si}_3\text{N}_4$  were characterized as deposited using atomic force microscopy (AFM). Pre-bonding of ALD  $\text{Al}_2\text{O}_3\text{-Si}_x\text{N}_y$  and ALD  $\text{Al}_2\text{O}_3\text{-Si}_3\text{N}_4$  was carried out right after alumina deposition. Sometimes a water ultrasonic bath was applied for silicon rich nitride in order to remove particles caused by the deposition process. The pre-bonding was carried out by aligning the wafers by their main flat and dropping one on top of the other and followed by application a force ( $\sim 10\ \text{N}$ ) on the

center of the top wafer to initiate the bonding front. In order to investigate the effects of the temperature on the bonding strength, wafers were annealed in a range of temperature from RT to  $600\ ^\circ\text{C}$  for both  $\text{Si}_x\text{N}_y$  and  $\text{Si}_3\text{N}_4$  samples for 8 h in  $\text{N}_2$  atmosphere. Infrared microscopy (IR) was performed to evaluate bonding quality and bonding characterization.

## 3. Results and discussion

For spontaneous bonding, the root mean square (RMS) surface roughness has to be below 0.5 nm [14]. The surface roughnesses of  $\text{Si}_x\text{N}_y$  and  $\text{Si}_3\text{N}_4$  were measured right after deposition by atomic force microscopy (AFM) in tapping mode in air. The RMS roughness of  $1\ \mu\text{m} \times 1\ \mu\text{m}$  areas were found 0.41 nm and 0.35 nm, respectively. The values are in good agreement with those measured by Reck et al. [9] Fig. 1 shows a  $20\ \mu\text{m} \times 20\ \mu\text{m}$  square scan of 10 nm amorphous ALD  $\text{Al}_2\text{O}_3$  on top of  $\sim 50\ \text{nm}$  of  $\text{Si}_x\text{N}_y$ . The ALD RMS roughness was measured to be 0.35 nm. Thus, the roughness of both  $\text{Si}_x\text{N}_y/\text{Si}_3\text{N}_4$  and amorphous  $\text{Al}_2\text{O}_3$  is sufficiently low for spontaneous bonding, and therefore chemical mechanical polishing (CMP) is not needed. Bonding inspection was carried out using an IR camera. Fig. 1b) and c) shows IR images of bonded  $\text{Si}_3\text{N}_4\text{-Al}_2\text{O}_3/\text{Si}_3\text{N}_4$  and  $\text{Si}_x\text{N}_y\text{-Al}_2\text{O}_3/\text{Si}_x\text{N}_y$  wafers annealed at  $600\ ^\circ\text{C}$ ,



**Fig. 2.** a) Experimental bonding strength measurement using the crack propagation method. A thin blade of  $100\ \mu\text{m}$  was inserted between the bonded wafers. Then the length of the debonded area was measured; b) Trend in bonding strength for  $\text{Si}_x\text{N}_y/\text{Al}_2\text{O}_3\text{-Si}_x\text{N}_y$  (black) and  $\text{Si}_3\text{N}_4/\text{Al}_2\text{O}_3\text{-Si}_3\text{N}_4$  (red). Dotted lines results from  $\text{Si}/\text{Al}_2\text{O}_3$  [10] (dotted blue) and  $\text{SiO}_2/\text{Al}_2\text{O}_3$  [11] (dotted purple).

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