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Fusion bonding of silicon nitride surfaces

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Abstract

While silicon nitride surfaces are widely used in many micro electrical mechanical system devices, e.g. for chemical passivation, electrical isolation or environmental protection, studies on fusion bonding of two silicon nitride surfaces (Si_3N_4 – Si_3N_4 bonding) are very few and highly application specific. Often fusion bonding of silicon nitride surfaces to silicon or silicon dioxide to silicon surfaces is preferred, though Si_3N_4 – Si_3N_4 bonding is indeed possible and practical for many devices as will be shown in this paper. We present an overview of existing knowledge on Si_3N_4 – Si_3N_4 bonding and new results on bonding of thin and thick Si_3N_4 layers. The new results include high temperature bonding without any pretreatment, along with improved bonding ability achieved by thermal oxidation and chemical pretreatment. The bonded wafers include both unprocessed and processed wafers with a total silicon nitride thickness of up to 440 nm. Measurements of bonding strength, void characterization, oxidation rate and surface roughness are also presented. Bonding strengths for stoichiometric low pressure chemical vapor deposition Si_3N_4 – Si_3N_4 direct fusion bonding in excess of 2 J cm^{-2} are found. The stoichiometry is verified indirectly through refractive index and intrinsic stress measurements. The importance of surface oxide in Si_3N_4 – Si_3N_4 fusion bonding is investigated by x-ray photoelectron spectroscopy measurements.

1. Introduction

While fusion bonding of silicon to silicon dioxide has been investigated in detail in the literature, silicon to silicon nitride fusion bonding [1–4] has only received little attention and Si_3N_4 – Si_3N_4 bonding even less. Since silicon nitride is widely used as a mechanical and electrical material in many MEMS and complementary metal oxide semiconductor (CMOS) processes, fusion bonding of two silicon nitride surfaces has a large number of potential applications in electronics, MEMS and optics. In this paper, we will focus on stoichiometric low pressure chemical vapor deposition (LPCVD) Si_3N_4 – Si_3N_4 fusion bonding.

Bonding of silicon nitride surfaces has previously been thought unobtainable [5]. Direct bonding of hydrophilic LPCVD Si_3N_4 – Si_3N_4 surfaces with a total thickness of 64 nm has however been reported by Ismail *et al* [6]. They also showed that even smooth hydrophobic silicon nitride surfaces might require an oxidation pretreatment in order to bond. Spontaneous direct bonding of 1 μm thick silicon rich LPCVD

Si_xN_y – Si_xN_y surfaces has been accomplished by Sánchez *et al* [7]. This bonding was only achievable when the surface roughness was reduced by chemical mechanical polishing (CMP) prior to the bonding. These experiments showed a decreased bonding strength for hydrophobic surfaces and that surface roughness should be on the order of $R_a \simeq 0.3 \text{ nm}$ (for a $20 \mu\text{m} \times 20 \mu\text{m}$ area) for spontaneous bonding to occur. Similar results have been obtained in [8], where CMP was also applied to reduce surface roughness from $R_a = 1.4 \text{ nm}$ to $R_a = 0.4 \text{ nm}$. Anodic bonding of silicon nitride covered wafers to glass substrates has been reported by Weichel *et al* [9], where both thermal oxidation and exposure to an oxygen plasma prior to the bonding was shown to improve bonding strength. Also bonding of PECVD silicon nitride surfaces has been reported [10, 3], offering process temperatures below $300 \text{ }^\circ\text{C}$. A summary of the literature results on Si_3N_4 – Si_3N_4 fusion bonding is shown in table 1. Examples of fusion bonding of silicon nitride to silicon or to glass are also shown.

As seen from table 1, the knowledge on Si_3N_4 – Si_3N_4 fusion bonding is rather limited and incomplete. For instance, the bonding strengths have not been measured either for thick stoichiometric silicon nitride, for thin stoichiometric LPCVD silicon nitride or for thin silicon-rich silicon nitride bonded

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Table 1. Summary of literature results on LPCVD Si₃N₄/Si₃N₄ fusion bonding as well as examples on silicon nitride bonding to other surfaces. For entries marked with an asterisk, the Si₃N₄ fabrication method is unknown. Pretreatment refers to one or more of the following pre-bonding treatments: none (a), CMP (b), thermal oxidation (c), chemical oxidation (d) and oxygen plasma exposure (e). Bonding strengths marked with a dagger are from pull tests, while all other bonding strengths are obtained using the crack propagation method [11]. Fracture instead of delamination typically occurs at a bonding strength of 2.5 J m⁻² or above.

Surfaces	Thickness (nm)	Pretreatment	Bonding strength	References
LPCVD Si ₃ N ₄ /Si ₃ N ₄	32	a/c,d	–	[6]
LPCVD Si ₃ N ₄ /Si ₃ N ₄	–	e	2.2 J m ⁻²	[12]
PECVD Si ₃ N ₄ /Si ₃ N ₄	50	d,e	Fracture	[10]
LPCVD Si _x N _y /Si _x N _y	1000	b,d	0.01 – 0.16 J m ⁻²	[7]
LPCVD Si _x N _y /Si _x N _y	260/400	b,d,e	–	[8]
Si/Si ₃ N ₄ *	140	a	Fracture	[1]
Si/Si ₃ N ₄ *	300	a	Fracture	[1]
LPCVD Si ₃ N ₄ /Glass	100	a/c/e	5.5/23 N mm ⁻² †	[9]

interfaces. Furthermore, it is not clear whether pretreatment is necessary for bonding to occur or not. Stoichiometric LPCVD silicon nitride is widely used in MEMS and CMOS fabrication due to its mechanical, electrical and optical properties. Fusion bonding of stoichiometric LPCVD Si₃N₄/Si₃N₄ surfaces are therefore of interest to these fields and will potentially allow for novel chip and process designs. We will in the following therefore focus on fusion bonding of stoichiometric LPCVD Si₃N₄–Si₃N₄ surfaces. Though the stoichiometry of the fabricated thin films is not verified directly, it is common in MEMS related and semiconductor technology literature to refer to LPCVD silicon nitride deposited at high NH₃/SiH₂Cl₂ flow rate ratios as stoichiometric [13], while LPCVD silicon nitride deposited at low NH₃/SiH₂Cl₂ flow rate ratios is referred to as low-stress silicon rich. The stoichiometry of the fabricated thin films can, however, be inferred indirectly from the refractive index and thin film stress measurements [14, 15].

2. Fabrication

In order to investigate the bonding potential of Si₃N₄–Si₃N₄, the following experiment was conducted. The LPCVD silicon nitride was deposited on boron-doped (1–20 Ω cm) double-sided polished 100 mm diameter (0 0 1) silicon wafers at a deposition temperature of 790 °C, using 100 sccm NH₃ and 25 sccm SiCl₂H₂ (DCS) gas flows at a pressure of 200 mTorr. Layers with thicknesses of 24 nm, 45 nm and 75 nm, respectively, were deposited. The refractive index of silicon nitride was found to $n = 2.0$ at a wavelength of 830 nm and the intrinsic stress was 1012 MPa. This is in accordance with the expected values of stoichiometric silicon nitride [15]. The fusion pre-bonding was carried out by simply aligning and placing the two wafers on top of each other, i.e. no force or heat treatment was applied. After pre-bonding, the wafers were annealed for 8 h at 1150 °C and 4000 sccm N₂ flow to consolidate the bond.

In order to investigate the effect of hydrophilic surfaces on bonding ability, a number of wafers with fresh silicon nitride surfaces were briefly dipped in bHF for approximately 10 s prior to bonding. The contact angles were measured using a Krüss DSA100 water drop shape analyzer. Part of

the bHF treated wafers were re-oxidized for 1 h in a wet thermal oxidation process at 1000 °C and 500 sccm O₂ gas flow where approximately 13 nm of SiON was grown followed by contact angle measurements. The measured oxidation rate of silicon nitride was, in agreement literature [16, 17], found to be diffusion limited. The oxidation was followed by a treatment in a 1:3 solution of H₂O₂:H₂SO₄ (Piranha). On a number of wafers, conventional MEMS fabrication steps including UV-lithography, deep reactive ion etch and thermal oxidation processes were carried out prior to LPCVD silicon nitride deposition. These processes reduced the effective bonding area by approximately 5%. On these wafers, the LPCVD silicon nitride was deposited on top of a thermal silicon dioxide layer, as is often done in real devices.

3. Results

Figure 1(a) shows an atomic force microscopy (AFM) scan of the surface of a 75 nm thick LPCVD silicon nitride film. From the AFM scan, an average roughness of $R_a = 0.16$ nm was deduced. This roughness is so low that a roughness-reducing CMP is not needed, and, indeed, spontaneous bonding was observed on wafers with such surfaces, even when the surfaces had been structured prior to LPCVD nitride growth. The strength of the prebond has been measured to approximately 0.03 J m⁻². While this is a very low bonding strength if used for real applications, it is sufficient for wafer handling and comparable to the bonding strength of thick Si_xN_y [7]. Figure 1(b) shows an infrared image of two bonded silicon nitride wafers. In the image shown, particles formed during handling and silicon nitride deposition cause a number of relatively large voids. It is thus critical that wafers are kept clean both before and during silicon nitride deposition. When taking special care to reduce particle contamination of the wafers, the relative void area to total wafer area is on the order of 1% or less.

After annealing, the bonding strength was measured using the crack propagation method [11]; for 24 nm thick silicon nitride, the average bonding strength measured was 2.2 J m⁻² with a standard deviation of 0.8 J m⁻². For 45 nm thick silicon nitride, the measured average bonding strength was 2.7 J m⁻² with a standard deviation of 1.1 J m⁻².

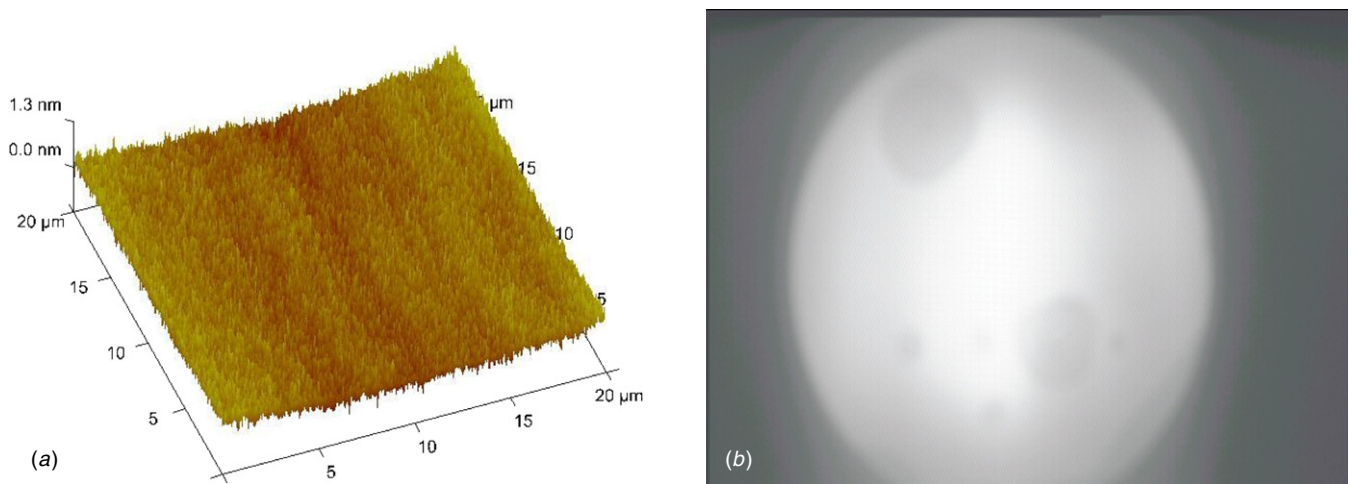


Figure 1. (a) AFM image of a $20\ \mu\text{m} \times 20\ \mu\text{m}$ surface area of a 75 nm thick silicon nitride. The average roughness was measured to $R_a = 0.16\ \text{nm}$. (b) Infrared transmission image of thin silicon nitride bonded wafers. While a number of voids are seen in this image (dark spots) due to particles formed during handling and silicon nitride deposition, the void area relative to the total wafer area amounts to less than 1% if care is taken to keep wafers clean and particle free prior to bonding.

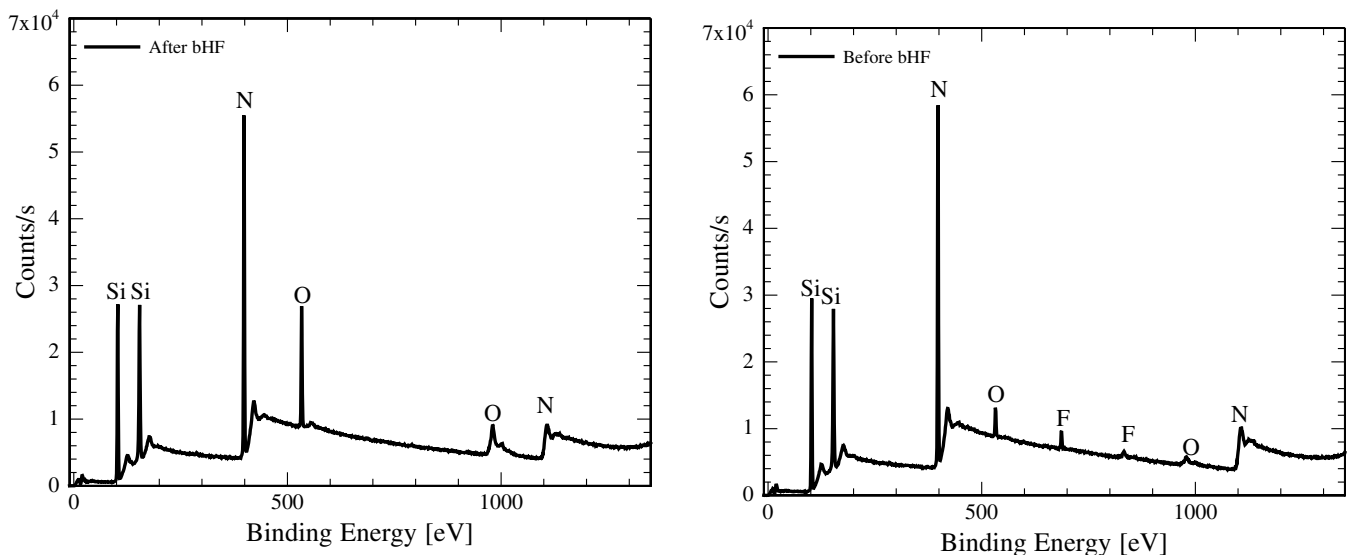


Figure 2. XPS spectra of LPCVD silicon nitride samples. Left: XPS spectrum of the as-deposited sample measured immediately after deposition. Right: XPS spectrum of the bHF-treated sample measured immediately after a bHF dip. The bHF treatment clearly reduces (by a factor of 3.5) the amount of oxygen in the surface of the bHF-treated sample compared to that of the as-deposited sample. A small amount of fluorine remains on the sample surface after the bHF treatment. Apart from the fluorine on the bHF-treated sample, both samples are remarkably clean.

These bonding strengths are comparable or even superior to the bonding strengths of conventional silicon to silicon dioxide fusion bonding [11].

If bHF is used in the prebonding process, subsequent bonding failed. A possible cause for the bonding failure may be that a thin native oxide on the silicon nitride surfaces is essential for its bonding ability. Even though silicon nitride is considered highly resistant to oxidation, the elevated temperatures during unloading of the wafers from the LPCVD furnace could cause a mild oxidation of the silicon nitride from the ambient air. To verify this, x-ray photoelectron spectroscopy (XPS) measurements were carried out (Thermo Scientific K-alpha XPS system with an Al $K\alpha$ source at

1486.68 eV) on a sample taken directly from the silicon nitride furnace as well as a sample that was subsequently treated with bHF. The XPS spectra are shown in figure 2. It is clear that the surface contains oxygen immediately after silicon nitride deposition. Treatment with bHF reduces the number of oxygen atoms present near the surface by approximately a factor of 3.5 (from 11.11% to 3.25% of the total number of near surface atoms), while the change in silicon and nitrogen concentrations is insignificant. A small amount of fluorine remains on the surface after the bHF treatment, but otherwise the sample surfaces are free from measurable contaminants. Close-ups of the nitrogen 1s and silicon 2p XPS peaks are shown in figure 3, where peaks from the as-deposited sample

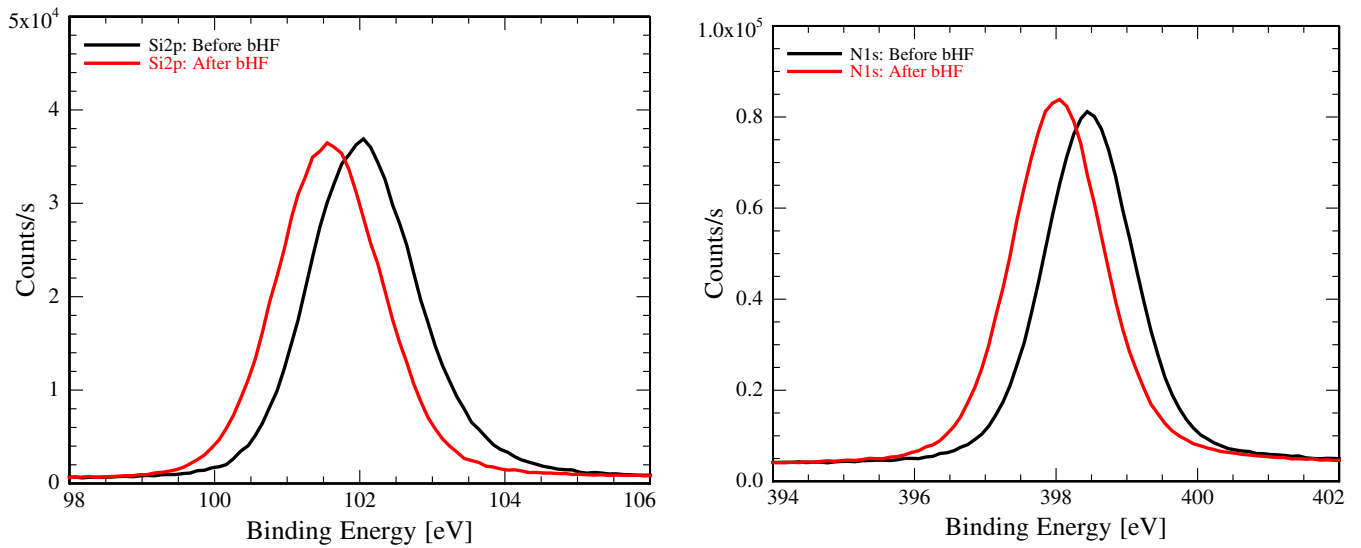


Figure 3. The 2p XPS peaks of silicon (left) and 1s XPS peaks of nitrogen (right) for the as-deposited sample (black curve) and bHF-treated sample (red curve). Both peaks show a shift in the binding energy of 0.5 and 0.4 eV, respectively, between samples; this shift is consistent with a reduction of the SiO₂ to Si₃N₄ concentrations in the bHF-treated sample.

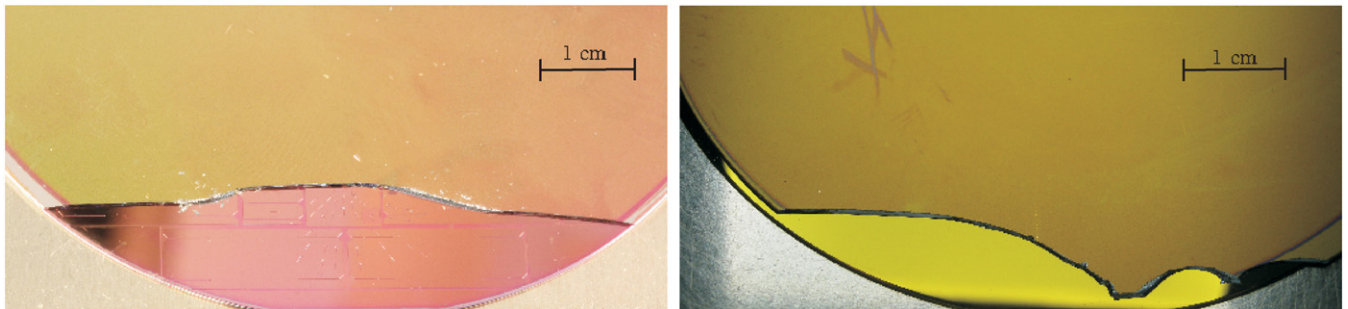


Figure 4. Fracture images of bonded wafers with as-grown Si₃N₄ surfaces (left) and bHF-treated, re-oxidized and piranha-treated Si₃N₄ surfaces (right). Both show fracture-limited bonding strengths after annealing; however, fragments are generally larger for as-grown Si₃N₄ than for re-oxidized and piranha-treated Si₃N₄ surfaces.

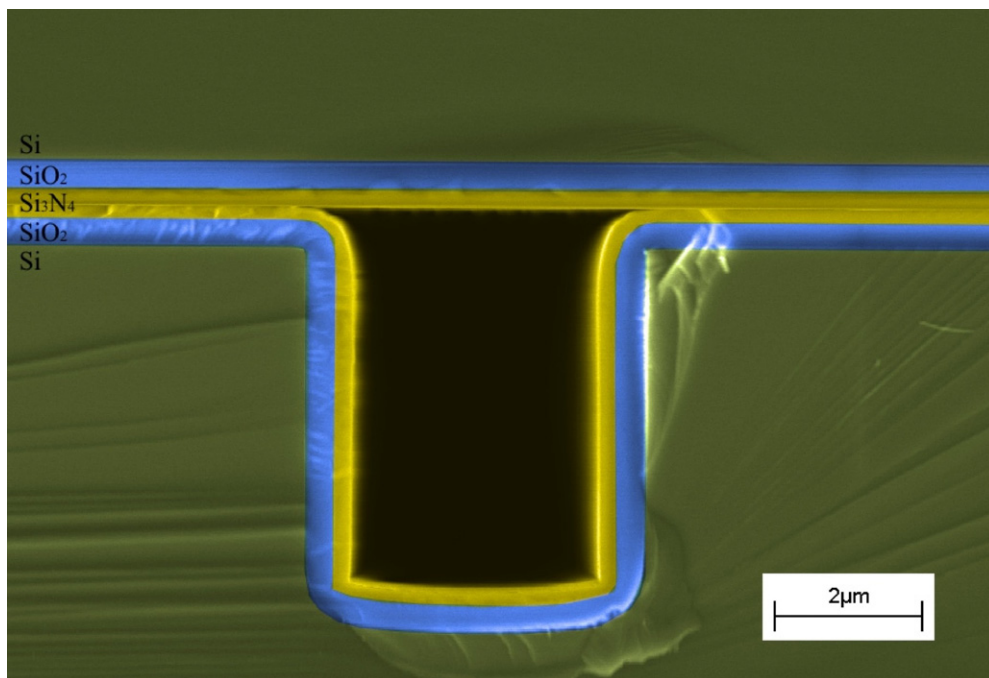


Figure 5. SEM image of a structured sample cross-section showing the bonding interface near an etched trench.

are shown as full black curves, while peaks for the bHF-treated sample are shown as full red curves; both peaks are shifted toward lower binding energy in the bHF-treated sample. The decrease in Si 2p binding energy is consistent with a reduction in the amount of SiO₂ relative to Si₃N₄ [18] (note the Si 2p peak in SiO₂ is expected at 103.4 eV, while an elemental Si 2p peak is expected at 99.8 eV); also the reduction in N 1s binding energy is consistent with a loss of oxygen bonds [19]. The contact angle measurements show that the silicon nitride surface is hydrophilic both before and after bHF treatment with water contact angles of approximately 20° or less. Hence, a hydrophilic silicon nitride surface is by itself not a useful indicator for the surfaces' ability to bond. On the other hand, the importance of a surface oxide in nitride bonding is further substantiated by the observation that bHF-treated wafers that were re-oxidized by thermal oxidation followed by a Piranha treatment were again bondable with a fracture-limited bonding strength. Fracture images are shown in figure 4.

If the Piranha treatment is omitted and only a very short thermal wet oxidation for 20 min at 1000 °C is conducted (approximately 5 nm SiON), 180 nm thick silicon nitride layers that are otherwise not able to bond will bond. While the bonding strength also in this case is fracture limited, the wafer debris are generally larger than if a Piranha treatment is included, indicating that the average size of voids is reduced if the wafers are also treated with Piranha prior to bonding.

The combination of thermal and chemical bonding enhancements makes Si₃N₄-Si₃N₄ bonding applicable in more general MEMS processing. The SEM image in figure 5 shows a hollow core optical waveguide fabricated by fusion bonding of two 220 nm thick Si₃N₄ surfaces. The processed wafers have been subjected to various MEMS processing technologies such as plasma-assisted etching, chemical etching and photolithography. The bonding strength of the processed wafers proved to be fracture limited. For thick silicon nitride layers (i.e. > 300 nm), CMP is usually necessary in order to reduce surface roughness and facilitate bonding.

4. Conclusion

In summary, LPCVD Si₃N₄-Si₃N₄ direct fusion bonding gives bonding strengths in excess of 2 J cm⁻² which is comparable to or larger than what is found for conventional silicon bonding in the literature. The bonding ability is limited primarily by surface roughness, which should be less than 0.3 nm for bonding to occur, and the presence of oxygen in the surface layer. It is critical that a high wafer cleanliness is maintained before and during silicon nitride deposition if such low roughness is to be obtained. For moderate surface roughnesses and/or wafers without oxygen present in the surface layer (e.g. due to bHF treatment), the bonding ability is improved by thermal wet oxidation. This is the case even when the resulting SiON is only few nanometers thick. Further improvement in bonding ability and reduction of void size can be obtained by combining thermal oxidation with a Piranha treatment. Using these methods, Si₃N₄-Si₃N₄ fusion bonding is applicable to MEMS fabrication in general.

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