Spontaneous direct bonding of thick silicon nitride

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Abstract. Wafers with 1 µm LPCVD silicon-rich nitride layers have been successfully direct bonded to silicon-rich nitride and boron-doped silicon surfaces. A chemical–mechanical polishing treatment was necessary to reduce the surface roughness of the nitride before bonding. The measured surface energies of the room-temperature bond were comparable to values found for Si–Si hydrophilic bonding. A mechanism similar to this bonding is suggested for silicon nitride bonding.

1. Introduction

Direct bonding of LPCVD Si$_x$N$_y$ has been presented in several papers [1, 2], but these works involved silicon nitride layers of a thickness of 140 nm or less. Direct bonding of Si$_x$N$_y$ layers with a thickness of 1 µm or more is nearly impossible because of the large surface roughness of such a layer.

Silicon nitride bonding is an interesting technology for the production of a high-$T_c$ superconductor bolometer, where bonding of Si$_x$N$_y$ to Si is used to obtain a thin single-crystal Si layer on top of a silicon nitride membrane [3, 4]. The use of chemical–mechanical polishing (CMP) for the reduction of the surface roughness shows its importance in silicon wafer bonding [5].

2. Wafer preparation

For the bonding experiments, 380 µm thick 3″ (100) Si wafers were selected, having a curvature of less than 10 µm across 50 mm. The used silicon nitride layers were 1 µm thick low-stress LPCVD grown Si$_x$N$_y$. Boron implantation was done at 200 keV, with a concentration of 2 × 10$^{16}$ cm$^{-2}$, followed by a 30 min anneal at 960 °C. Boron dotation in a solid source dotation (SSD) system was done at 1150 °C, leaving a surface concentration of 3 × 10$^{20}$ cm$^{-3}$.

The nitride wafers were given a CMP treatment in order to reduce the surface roughness from 2.9 to 0.3 nm ($R_a$ value, calculated from a 20 × 20 µm$^2$ atomic force microscope (AFM) scan). An AFM scan of a Si$_x$N$_y$ surface before and after CMP is shown in figure 1. Besides reducing the ‘natural’ surface roughness of the thick silicon nitride, CMP also removed particle contaminations induced by the LPCVD silicon nitride growth process. These particles are typically up to 80–100 nm in diameter. To obtain a good bondable surface an overall amount between 30 and 50 nm of Si$_x$N$_y$ was removed with CMP, with a removal rate of 2–3 nm/min.

The boron-implanted wafers were used without CMP. After removing the thin oxide layer which is used to prevent channelling, the surface roughness is comparable to that of a CMP treated wafer. Before further processing, all polished wafers were cleaned in a H$_2$SO$_4$/$\text{H}_2\text{O}_2$/$\text{H}_2\text{O}$ 1:1:5 solution at 80 °C, removing the polishing slurry contaminants.

3. Bonding process

Prior to bonding, the wafers were cleaned with fuming nitric acid (100%) and hot nitric acid (70% at 90 °C), followed by a quick dump rinse (QDR) with DI water. The wafers were kept wet in the QDR, rinsing several times, and spin dried only just before bonding. To speed up the bonding process a small pressure was applied until bonding initiated. Two wafer pairs were put in a 5% HF solution for 1 min to investigate the influence of the chemical oxid-nitride layer which is left after cleaning. An IR camera was used to monitor the initial bonding of the wafers. Annealing was done for 2 h at 800, 900 or 1000 °C in N$_2$ or O$_2$.

4. Results

Using the described wafer preparation we have achieved spontaneous room-temperature bonding of Si$_x$N$_y$ to Si$_x$N$_y$, boron-implanted and SSD doped Si. The speed of the bondwave was measured to be up to 3 cm s$^{-1}$. IR images of a typical propagating bondwave between Si$_x$N$_y$ and boron-implanted Si are shown in figure 2.

The bond strength of the room-temperature bond was measured with the crack propagation method [6]. The values for the surface energy of the room-temperature bond are given in table 1. The values for the Si$_x$N$_y$ to Si$_x$N$_y$ bond are in the same range as for Si–Si hydrophilic bonds [7]. The use of an HF dip resulted in a weaker room-temperature bond, and also in a weaker bond after annealing. However,
Figure 1. AFM images of LPCVD Si₃N₄ surfaces before (top) and after (bottom) CMP; the $R_a$ roughnesses are 2.9 nm and 0.3 nm respectively.

Table 1. Surface energies of room-temperature bonds of different bonded surfaces.

<table>
<thead>
<tr>
<th>Bonded surface</th>
<th>Surface energy (J m⁻²)</th>
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<tbody>
<tr>
<td>Si₃N₄–Si₃N₄</td>
<td>0.08–0.16</td>
</tr>
<tr>
<td>Si₃N₄–Si₃N₄ with HF</td>
<td>0.01–0.04</td>
</tr>
<tr>
<td>Si₃N₄–B-implanted Si</td>
<td>0.12–0.29</td>
</tr>
</tbody>
</table>

It should be noted that the HF dip before bonding was only performed once. On one measured sample the surface energy of the Si₃N₄ to Si₃N₄ bond after annealing at 1000 °C for 2 h was between 1.1 and 2.8 J m⁻².

5. Silicon-on-nitride

Using a bond-and-etch-back technology, silicon-on-nitride (SON) layers were produced (figure 3). Etching was done using the boron etch stop in KOH/IPA. After reaching the boron layer, the Si top layer was given a short CMP step to both reduce the thickness and the surface roughness. A Si top layer of 300 ± 50 nm was made this way, which at this moment seems to be the best obtainable result using a single etch stop. These layers will be used for the production of a high-$T_c$ superconductor bolometer [3, 4]. In recent experiments successful bonding was achieved between commercially obtained 4" SOI wafers from AT&T and 4" wafers with a 1 µm Si₃N₄ layer (after a CMP treatment). The Si top layer of the SOI wafer was
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6. Discussion

After a standard cleaning process, the Si$_3$N$_y$ surface is covered with a thin hydrophilic chemical silicon oxi-nitride layer. Therefore we suggest that the bonding mechanism of Si$_3$N$_y$–Si$_3$N$_y$ is comparable to Si–Si bonding, where bonding actually occurs between the two thin native oxide layers [8]. Using the same bonding procedure, Si–Si bonding resulted in a 2.7 nm thick bonding interface. Bonding of Si$_3$N$_y$ to Si left a 8–10 nm thick amorphous interface (figure 4), whose exact chemical composition is still under investigation.

An unsolved problem is the presence of small voids (containing gas) and unbonded areas in the bonding interface. They have a typical diameter between 50 and 400 µm, and appear particularly towards the edge of the bonded wafer pair. We believe that they are the result of remaining surface microroughness on the Si$_3$N$_y$ surface, rather than just a result of the reaction at the interface during annealing. After the CMP treatment, the surface roughness of the Si$_3$N$_y$ layer is probably somewhat higher towards the edge of the wafer. A model is currently being developed where the influence of the microroughness on the bondability is expressed as an adhesion parameter [9]. This model also takes into account the Young’s moduli of the bonded materials. This might explain the higher surface energies found for the Si$_3$N$_y$–Si bond, since the Young’s modulus for Si is about a factor of two lower than for Si$_3$N$_y$.

When making the SON layers, an additional annealing step was used following the etch-back process. After thinning the Si top layer to about 0.6 µm, a 4 h anneal at 1000°C was given, which significantly reduced the unbonded areas of the still intact Si top layer.

7. Conclusion

We have successfully bonded thick silicon nitride to silicon nitride, a boron-implanted Si surface and a SSD boron Si surface. This was only possible after CMP of silicon nitride in order to reduce the surface roughness. This process was used to produce silicon-on-nitride layers for bolometric applications.

Acknowledgments

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References

[9] Gui C to be published