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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_xN_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_xN_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_xN_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_xN_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 (AMF) scan). An AFM scan of a Si_xN_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_xN_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 $H_2SO_4/H_2O_2/H_2O$ 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 oxi-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_xN_y to Si_xN_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_xN_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_xN_y to Si_xN_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,

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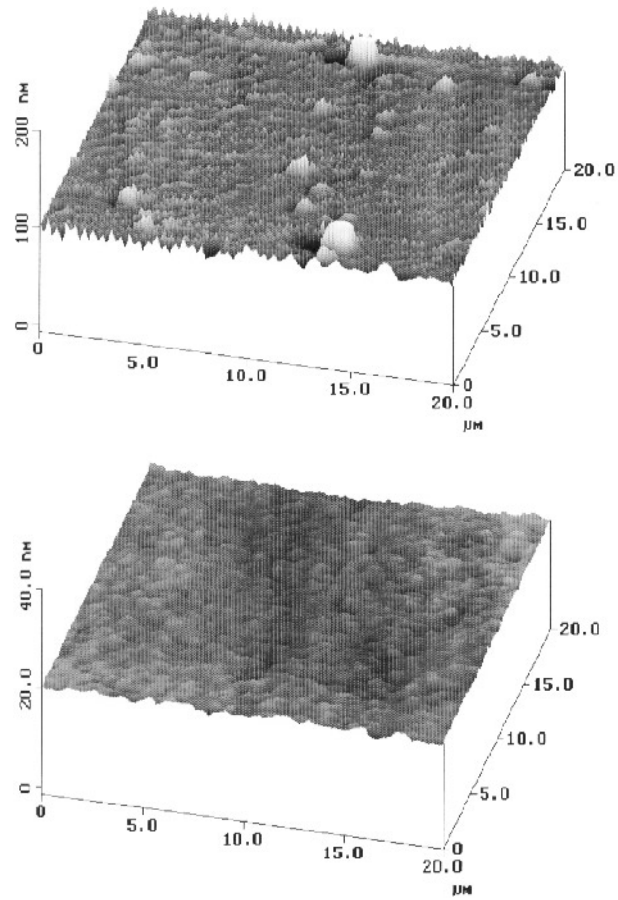

Figure 1. AFM images of LPCVD Si_xN_y surfaces before (top) and after (bottom) CMP; the R_a roughnesses are 2.9 nm and 0.3 nm respectively.

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

Bonded surface	Surface energy (J m^{-2})
Si_xN_y–Si_xN_y	0.08–0.16
Si_xN_y–Si_xN_y with HF	0.01–0.04
Si_xN_y–B-implanted Si	0.12–0.29

it should be noted that the HF dip before bonding was only performed once. On one measured sample the surface energy of the Si_xN_y to Si_xN_y 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 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

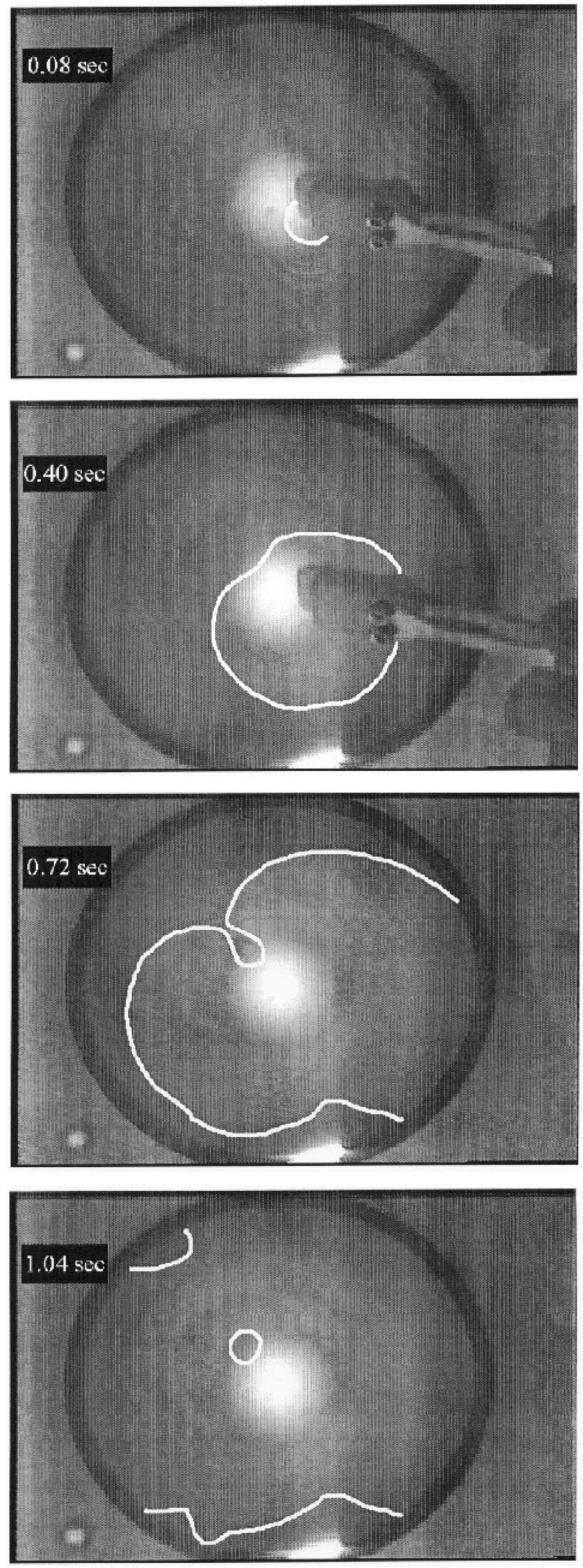

Figure 2. IR images showing the bondwave between LPCVD Si_xN_y and boron-implanted Si, initiated at the center of the 3 inch wafer; the front of the propagating bondwave has been marked because of the poor contrast in the pictures.

and 4″ wafers with a 1 μm Si_xN_y layer (after a CMP treatment). The Si top layer of the SOI wafer was

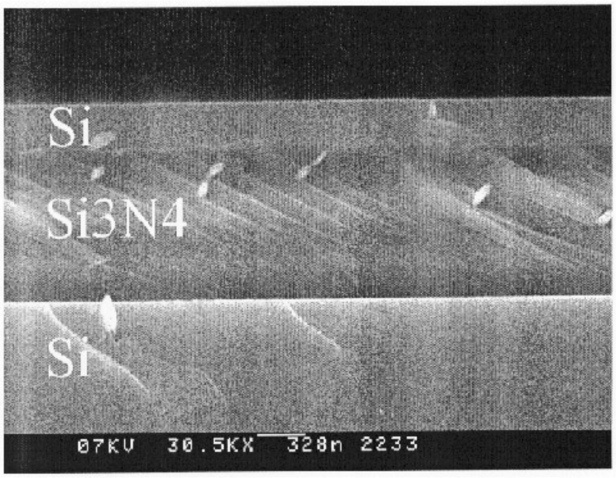

Figure 3. SEM photograph of a 0.4 μm Si layer on top of a 1 μm Si_xN_y layer using an implanted p^{++} layer.

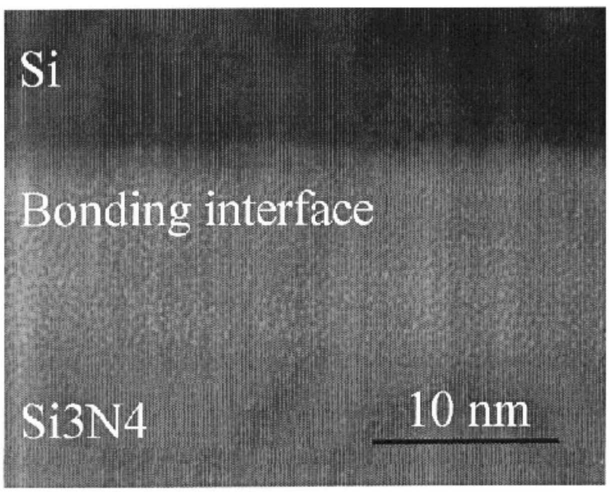

Figure 4. TEM image showing the bonding interface between boron-doped Si to Si_xN_y.

successfully transferred to the nitride wafer by bonding and etch-back. Because of the double etch stop in the SOI wafer, this route increases the thickness uniformity of the Si top layer of the obtained SON layer, which is only dependent on the SOI wafer specifications.

6. Discussion

After a standard cleaning process, the Si_xN_y surface is covered with a thin hydrophilic chemical silicon oxi-nitride layer. Therefore we suggest that the bonding mechanism of Si_xN_y–Si_xN_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_xN_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_xN_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_xN_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_xN_y–Si bond, since the Young's modulus for Si is about a factor of two lower than for Si_xN_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.

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