boundaries (Perreault et al 1992).

iv) In the plan-view images some of the samples showed linear structures running at an angle of about 30° to a <110> direction. Their spacing is about 50 nm. Diffraction contrast experiments proved that these structures are not related to 60° dislocations. Cross-sectional samples prepared from different {110}- and {100} planes showed a zigzag-like structure in one {100} plane with the same periodicity (50 nm). Samples of all the other planes only show the dislocation network ({110} samples) or dot-like contrasts referring to oxide islands (the other {100} plane). As shown in Fig. 3, a terrace-like structure is reconstructed from all these images. Their nature is not yet known. Terraces may be caused by stress relaxation of the rotational misorientation.

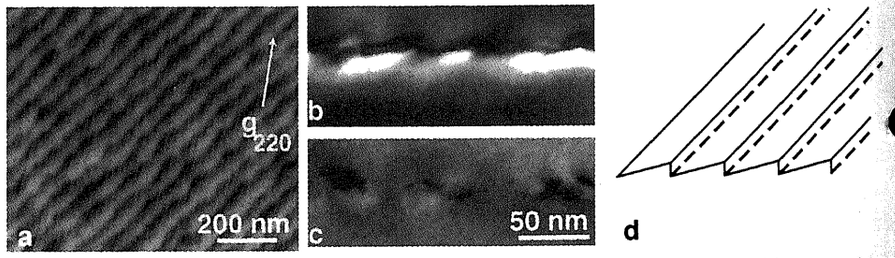

Fig. 3: Terrace-like structure in the interface. TEM-plan-view (a) and cross-sectional images of 2 different {100} planes (b,c). Schema of the terrace structure (d).

CONCLUSIONS

It is shown that the interfaces of bonded hydrophobic wafer pairs and such pairs bonded under UHV conditions correspond to grain boundaries having a twist and a tilt component. MD-simulations for clean and reconstructed surfaces (as obtained under UHV conditions) explain the generation of dislocation networks during bonding by the relaxation of interfacial atoms and the formation of covalent bonds. Furthermore, the defect structure in interfaces of wafers bonded under atmospheric conditions is more complex. Besides the screw dislocation network, interactions of screw dislocations with 60° dislocations, resulting from the miscut of the samples used, are observed more frequently. These interactions may be caused by the high annealing temperatures applied. In some cases, instead of the screw dislocation network, a terrace-like structure forms which may constitute a form of adapting the rotational misorientation, at least partially.

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Paper presented at Microsc. Semicond. Mater. Conf., Oxford, 7–10 April 1997

Dislocation structure in interfaces of bonded hydrophobic silicon wafers: experiment and molecular dynamics

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ABSTRACT: The defect structure in interfaces of directly bonded hydrophobic silicon wafers is studied by HVEM and HREM. It is shown that the twist component of the boundary causes a screw dislocation network, while the tilt component or a miscut are compensated by arrays of 60° dislocations. Interactions occur between both especially during annealing at higher temperatures. For annealing at temperatures below 1000°C the structure of the interface is more or less analogous to that of wafer pairs bonded under UHV conditions at room temperature without any subsequent heat treatment. Molecular dynamics simulations of the latter revealed that the screw dislocation network is caused by the relaxation of atoms into a mosaic-like structure with the formation of covalent bonds via the interface.

1. INTRODUCTION

Silicon wafer direct bonding has become of increasing interest for silicon-on-insulator (SOI) and power devices as well as for micromechanics and sensors. Here, wafers used for wafer bonding are covered with either a thin native oxide or a purposely grown thermal oxide which has been rendered hydrophilic by cleaning procedures. At room temperature, the initial bonds are hydrogen bonds caused by the presence of OH-groups at the interface, while at higher temperatures they are transformed into stable Si-O-Si bonds. The interfaces are atomically flat without any defects. Alternatively, silicon surfaces may be treated with diluted HF, which removes the native oxide layers and directly covers the silicon surface with hydrogen, thus rendering the surfaces hydrophobic. Here, van der Waals forces are generally assumed to be the origin of the attractive force at room temperature. At higher temperatures, they are transformed into Si-Si bonds. Depending on the misorientation between both wafers, dislocation networks are generated in the interface: the twist component causes a network of pure screw dislocations, while the tilt component is compensated by a periodic array of 60° dislocations. Both dislocation fractions were extensively investigated after annealing at $T \geq 1100°C$ (e.g. Gafiteanu et al 1993, Benamara et al 1994, 1995).

The present paper analyzes the interfacial defects of bonded hydrophobic wafer pairs occurring at lower temperatures (> 800°C). Furthermore, these interfacial defects are compared to the interfaces of wafer pairs bonded under ultra-high vacuum (UHV) conditions at room temperature without any subsequent heat treatment.

2. EXPERIMENTAL

Czochralski-grown silicon wafers (diameter 4 in., (100) orientation, p-type) were used for the experiments. The tilt component (cut-off) of all wafers is below ± 1°. After cleaning in standard

RCA 1,2 solutions they were dipped into ~ 1% HF for 2 minutes in order to make the surfaces perfectly hydrophobic. Without subsequent water rinsing the wafers were bonded in a micro-cleanroom set-up (Stengl et al 1988) under normal atmospheric conditions. After bonding one group of wafer pairs was annealed at temperatures between 800°C and 1200°C under inert or oxidizing conditions. Another group was transferred in a UHV chamber, separated, and bonded again at room temperature after annealing at 600°C < T < 800°C in order to desorb the hydrogen from the wafer surfaces before bonding (Gösele et al 1995). These UHV room temperature bonded wafers did not receive any subequent heat treatment.

After thinning down one of the wafers of a pair, samples for electron microscopy were prepared by chemical thinning in HF/HNO_3 solutions (plan-view or oblique cut) and by ion beam milling (cross-sectional samples). Both, high-voltage and high resolution electron microscopy were applied. The formation of interfacial defects for the case of UHV bonding at room temperature was simulated by molecular dynamics (Scheerschmidt et al 1996).

3. INTERFACES OF UHV-BONDED WAFER PAIRS

If two clean silicon surfaces meet (as under UHV conditions) strong covalent bonds develop already at room-temperature between the surfaces (Gösele et al 1995). HREM images show that the interface is not perfect. Instead, defects occur caused by the unavoidable misorientations between the two wafers. Analyzing such samples without any additional heat treatment reveals an analogous interface defect structure generally known from interfaces of wafer pairs bonded under atmospheric conditions (Benamara et al 1994, 1995). There is a 2-dimensional dislocation network having square-like meshes (Fig. 1). The dislocations forming the network are screw dislocations (**b** = a/2<110>), with spacings of about 66 nm, corresponding to a twist angle Θ ~ 0.3°. Furthermore, there are irregularities in the network suggesting a superposition of 60° dislocations. Molecular dynamics (MD) simulations were carried out starting with two perfect Si blocks

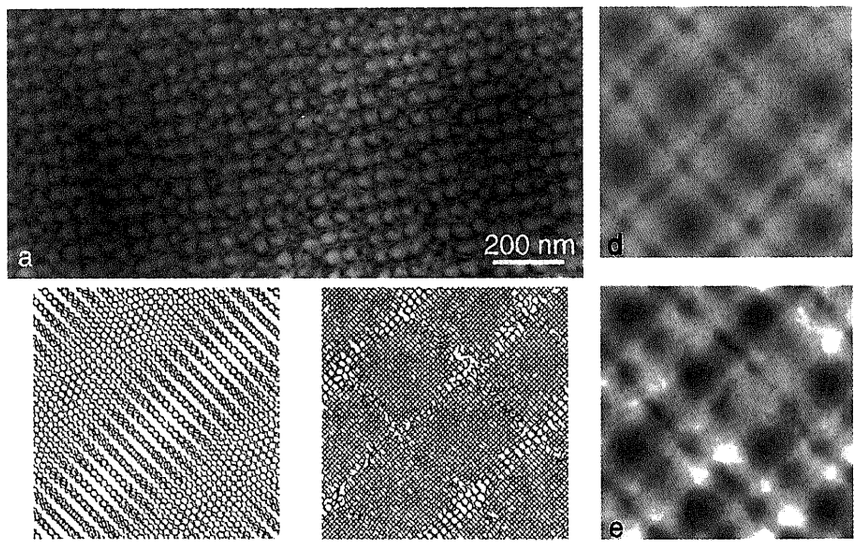

Fig.1: Formation of a screw dislocation network in the interface of bonded silicon wafers. The wafers were bonded under UHV conditions. TEM image of a bonded {100} interface (a). MD-simulation of rotationally misoriented wafers (b,c). Starting configuration (dimer rows at interface) with 4.58° misorientation (b) and after relaxation (annealing at 900 K for 1.25 ps (c)). Simulated TEM bright field images from (c) with $\mathbf{g}_{220}$ excited (thickness 4.3nm (d),12.9nm (e)).

of 2 x 1 reconstructed (100) surfaces. A small twist angle is assumed as a rotational misorientation without other defects (Fig. 1b). After sufficient relaxation a mosaic-like interface structure (size 7 nm) occurs where almost all atoms have a bulk-like environment separated by screw dislocations (Fig. 1c). The dislocations show irregularities due to randomly started bonding processes. Image simulations (TEM bright field contrast) based on the MD-model showed the square-like arrangement of dislocations as shown in the experimental images (Figs. 1d,e). The dark contrast effects between the dislocation lines are caused by a lattice plane deformation. Recently, MD-models on the basis of double-layer steps on the surfaces have also been developed for simulating the nature of 60° dislocations (Belov et al 1997).

4.INTERFACES OF WAFER PAIRS BONDED UNDER ATMOSPHERIC CONDITIONS

The hydrogen passivation of the wafer surfaces causes only van der Waals forces as the attractive force at room temperature. Therefore the bonding strength is low, requiring additional annealing at higher temperatures. Here wafers were bonded with a rotational misfit up to 5°. Interfaces of wafer pairs annealed at temperatures between 800°C and 1200°C show 4 different features:

i) There is a more or less periodical network of 60° dislocations originating from the tilt component. In most samples analyzed the spacing of these dislocations is of the order of several micrometers corresponding to tilt angles of about 0.02°.

ii) At higher magnifications an array of screw dislocations occurs respresenting the twist component or rotational misfit (Fig. 2a). At lower temperatures (T < 1000°C) the network is more regular than at higher temperatures. Here, the screw dislocations are displaced along almost straight lines running at an angle to the <110> directions. The displacements are due to interactions with the 60° dislocations described by Gafiteanu et al (1993) and Benamara et al (1995) for bonded wafers, and by Föll and Ast (1979) for grain boundaries in sintered silicon. The latter showed that the interaction causes *extraneous* dislocations lying in their {111} glide plane and resulting in a step in the boundary plane. This may be the reason for the high dislocation density (Fig. 2b).

iii) In some areas the dislocation network abruptly discontinues, with only Moiré fringes appearing in the images (Fig. 2c). They may represent bonded areas in which the misorientation is not adapted by misfit dislocations but by an adaption arrangement distributed over all interface atoms via slightly changed bond lengths and angles, or are due to interfacial defects filled with residual oxide (SiO_2) not removed by the HF dip. Such areas would correspond to the oxide-welded

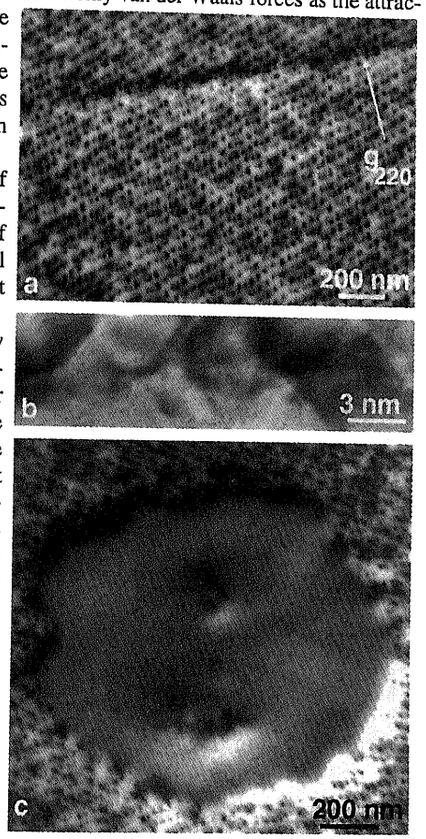

Fig. 2: Interfacial defects in wafer pairs bonded under atmospheric conditions.
a) Screw dislocation network superimposed with 60° dislocations (plan-view image).
b) Formation of *extraneous* dislocations due to interactions (cross-sectional image).
c) Interfacial area without the dislocation network (plan-view image).