Nano-voids at hydrophilic, hydrophobic and UHV bonded silicon interfaces detected by TEM

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Wafers of CZ silicon were bonded hydrophilically, hydrophobically and in ultra high vacuum (UHV) at room temperature. Bonded hydrophilic wafers adhere together by hydrogen bonds, hydrophobic wafers by van-der-Waals forces and UHV-bonded wafers by covalent bonds. Annealing the pre-bonded hydrophilic and hydrophobic wafer pairs in Argon for 2 h at different temperatures increases the initially weak bonding energy. UHV-bonded wafer pairs were also annealed to compare the results.

The interfaces of bonded wafer pairs were investigated by TEM and HRTEM in cross-section and 25°-section samples.

Bonded hydrophilic wafers show a 2-3 nm thick silicon oxide layer at the interfaces. Due to the nano-roughness of the surfaces before bonding free volume is enclosed at the interface. In the oxide interlayer nano-sized voids are observed. Annealing leads to chemical reactions at the interface. Size and area density of the voids is similar after annealing at temperatures between 130°C and 950°C. At 1000°C the voids coarsen and the area density decreases. At temperatures above 1100°C the viscosity of silicon oxide decreases, the voids become closed and the oxide interlayer starts to disintegrate. Locally the oxide is dissolved and the two wafers grow together with direct lattice contact silicon to silicon.

The surface of hydrophobic wafers is terminated with hydrogen. During annealing of bonded wafer pairs to 450°C hydrogen desorbes and large bubbles are generated at the interface. Bonded areas show nano-sized shallow voids at the interface. Annealing at higher temperatures leads to diffusion of hydrogen away from the interface and to the gradual formation of a dislocation network. The interface of bonded hydrophobic and annealed silicon wafers is a direct junction from silicon to silicon without an intermediate layer, but also a high density of voids is observed. Compared to the 450°C anneal the area density of voids decreases and the shape changes at higher temperatures. By annealing at 1000°C the shallow two-dimensional voids ripen to three-dimensional facetted shape with a density of $1{,}3 \cdot 10^{10}$ cm^{-2}. Annealing at 1200°C results in precipitates of oxygen as silicon oxide with a similar area density. The former voids become filled with silicon oxide.

UHV-bonded wafers show a high bonding energy already after room temperature bonding. No intermediate layer is observed at the interface. Crystal lattice planes go across the interface over extended areas and nano-sized voids are not observed. Annealing at 800°C and above leads to the formation of a dislocation network. Because of the nano-roughness and the high bonding energy complex dislocation structures including loops due to missing or additional lattice planes are frequently observed. After annealing for 2 h up to 1200°C only a few silicon oxide precipitates are visible at the interface. Long-terme annealing at 1100°C for 24 h results in a homogeneous distribution of oxide precipitates with a density of $6{,}5 \cdot 10^{9}$ cm^{-2}.