ESRF	**Experiment title:** Directly bonded silicon: an investigation of the interface atomic structure by x-ray diffraction.	**Experiment number:** SI-401
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Report:

Diffraction measurements were performed to study the interface structure of directly bonded Si(001) wafer pairs. The samples had all zero tilt angles, that means parallel (001) surfaces, but various twist angles, relative rotation around the surface normal (001). Direct (fusion) bonding is a two step process. First the wafers are cleaned in a HF solution and contacted at room temperature in a high class clean room. This give a weak van der Waal bonding. Next annealing above 1000 °C produce the strong covalent bonding. In the present measurements three problems were adressed:

First: Earlier measurements have shown that as the twist angle θ gets small the interface become thick[1]. We have done new high resolution diffraction measurements of the superstructure of the interface of a bicrystal with a twist angle of only θ=0.41 degree and analyzed the line shape of the diffraction profiles. We find that the interface is accurately characterized by an exponential decay of the displacement field over long distances away from the interface.

Second: We have for the first time observed how the interface superstructure is formed as function of temperature. In this study a pair of Si(001) wafers were in advance room temperature bonded (weak van der Waal bonding) and as such carefully etched from one side so that one of the bonded crystals was 30μ thick, suitable for x-ray diffraction from the interface. A series of 1 cm^2 samples diced from these crystals were annealed at various temperatures for

[1] M. Nielsen, R. Feidenhans'l, P.B. Howes, F. Grey, K. Rasmussen, M. Benamara, and J. Vedde, to be published.

several hours in N_2 atmosphere. The interface structure of these was measured by diffraction and the figure below show that sharp peaks from the bonding superstructure grow as the annealing temperature exceeds 800 °C. This is the temperature where the protecting hydrogen layers dissolves in bulk Si and reordering occur on the clean Si surface. The bonding superstructure appears with the full range coherence length already at 800 °C.

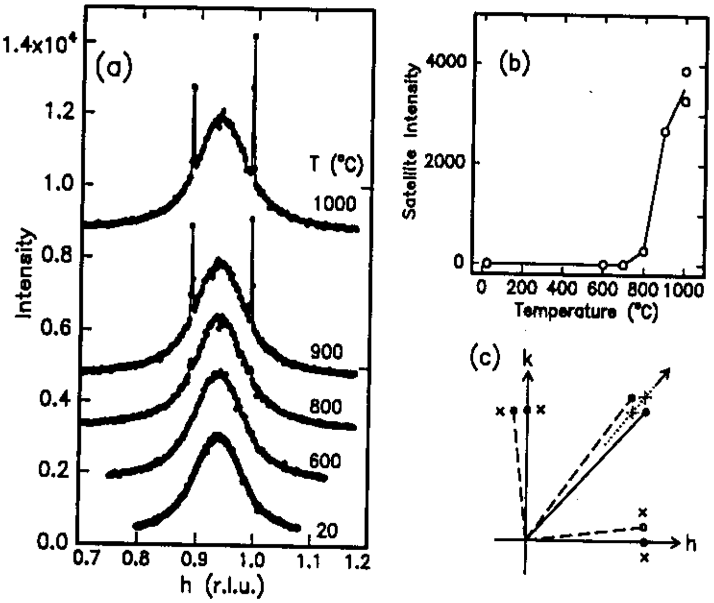

Figure 1: The figure shows in panel (a) x-ray diffraction profiles from direct-bonded Si(001) bicrystals, twist angle θ=6.5 degree, annealed at increasing temperature as given in right hand side of the panel. The profiles are measured in axial scans indicated by the arrow in panel (c). Panel (b) shows how the satellite intensity, at the positions indicated by $\times$ in (c), and signaling covalent bonding, depends on the annealing temperature. Panel (c) shows in LEED notation the reciprocal lattice plane parallel to the interface plane of the bicrystals.

Third: We extended earlier measurements on Si(001) directly bonded bicrystals to larger twist angles, θ=8-20 degree. These studies were preliminary and did not give conclusive results and they must be complemented in future measurements. However, we did observe that for $\theta > 8$ degree