ESRF	**Experiment title:** High temperature reflectivity of SiC//SiC bonding	**Experiment number**: A32-2 854
Beamline: BM32	**Date of experiment**: from: 02-12-2022 to: 06-12-2022	**Date of report**: 15 02 2023
Shifts: **9**	**Local contact(s)**: TARDIF Samuel	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

***RIEUTORD Francois, Soitec & CEA**

***Le CUNFF Maelle, Soitec & CEMES**

Report:

The experiment was carried out on samples annealed ex-situ rather than in-situ annealing, due to the difficulties to achieve the required temperatures with available beam line equipment.
As planned, we performed reflectivity on SiC SiC bondings where the bonding has been performed using a thin deposited silicon film on each of the SiC. We thus have a reflectivity of a confined Silicon film between two SiC substrates.

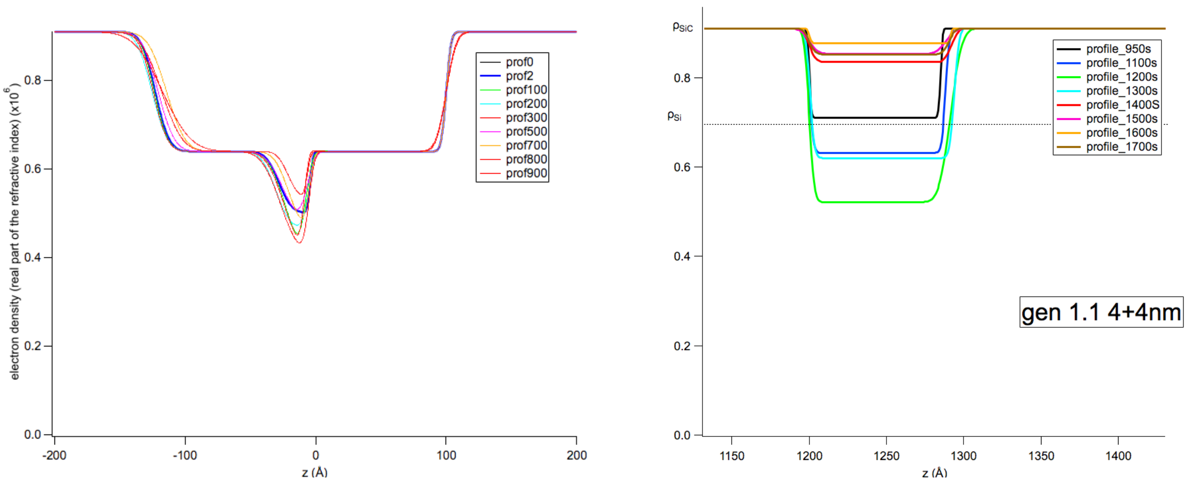

Fig.1&2 Electron density profiles extracted from XRR of a 10+10nm Si film (T<900) and (4+4nm). At low Ts the bonding interface is still visible through a small dip in the electron density in the center of the profile. At higher temperatures (right figure) the bonding interface between Si is flat (sealed) but the remaining film shows large thickness and density variations with temperature.

We could show that the density of the film reduces significantly while its thickness grows. Yet, the amount of Si is found to be roughly constant in this 950<T<1400°C range.
Complementary electron microscopy observations have confirmed that this is indicative of a dewetting process with the appearance of pits and holes in the silicon film.

Above 1400°C (i.e the melting temperature of Silicon) , the density of the interface region appears very close to that of silicon carbide, indicating that much of the silicon has been replaced in this region by silicon carbide. Again this observation has been confirmed by TEM observations which shows very important surface reconstructions mediated by liquid silicon.

In addition to low angle density measurements with XRR , we measured the resulting strain in the silicon film due to both CTE mismatch and the large volume expansion associated to liquid/solid phase transition in silicon. Thanks to the intense synchrotron beam, we could measure scattering from the polycrystalline 10nm-thick silicon film. The strain dependance obtained from different Bragg peak positions show that a strong compressive stress builds up when the silicon experiences a transition from liquid to silid state

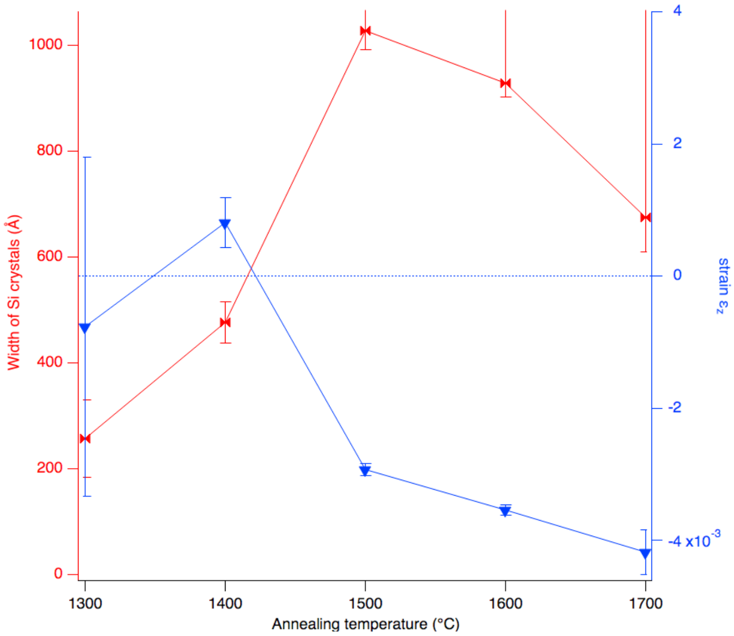

Fig.3. Normal strain (blue, right scale) and size of crystallites (red, left scale) for high temperature annealed Si between SiC. At high T, compressive strain appears while silicon crystallites grow in size.

Overall, we could collect a large amount of quantitative data on the silicon and SiC interface evolutions which now have to be matched with direct electron microscopy observations.

Complementary to the study of the silicon bonding film, we measured also the properties of the silicon carbide substrate and film. Rocking curve were performed both in plane and out of plane to measure the twist and tilt offcut of the bonding, together with the effect of the silicon "glue " layer on the top SiC film.

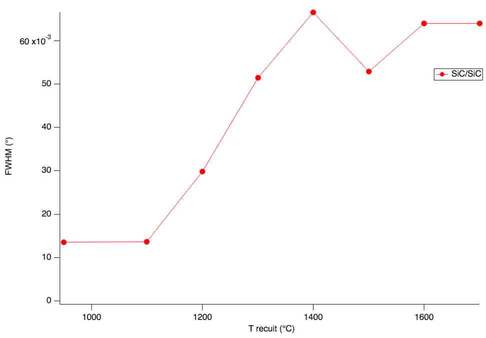

Fig.4 Rocking curve width of the SiC top film. A significant increase occurs before the silicon melting temperature, showing the impact of the interface on the film structure.