

Report:



	Experiment title: Study of high temperature SiO ₂ //SiO ₂ interface sealing	Experiment number: 32-2-781
Beamline: BM32	Date of experiment: from: 02/09/2015 to: 08/09/2015	Date of report: 25/02/2016
Shifts: 9	Local contact(s): RIEUTORD	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): *Rieutord F *Tardif Samuel Oleg Kononchuk Frank Fournel		

Objective & expected results (less than 10 lines):

The purpose of the experiment was to follow as a function of temperature T and time t the evolution of the interfacial structure of a direct bonding interface, in a temperature range (1100°C) where full sealing of the interface is expected. The kinetics of this sealing was to be measured at different temperatures, in order to possibly extract an activation energy.

Results and the conclusions of the study (main part):

We could perform the experiment. On fig 1&2 we show the evolution of the reflectivity curves as a function of rising temperatures.

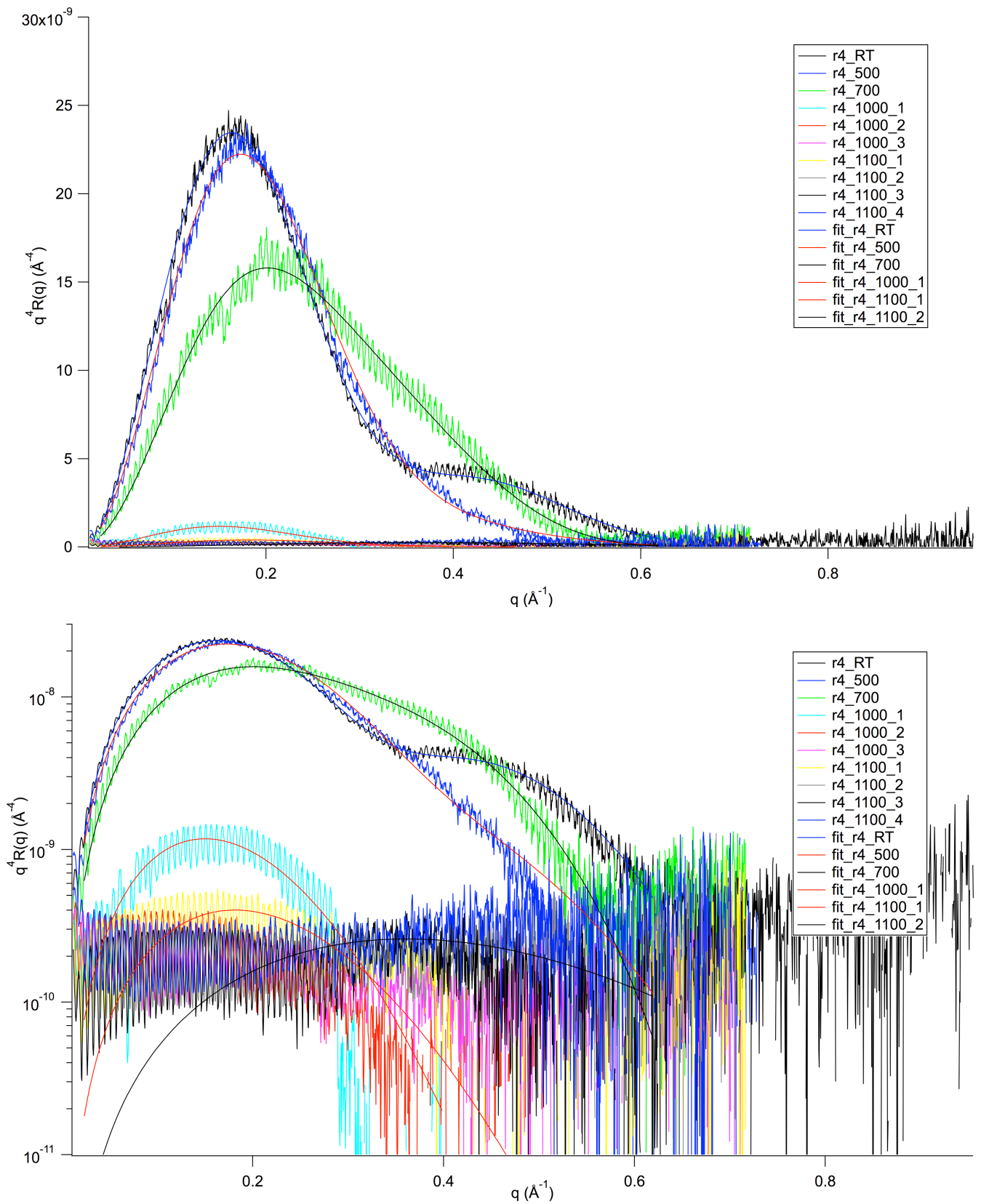


Fig.1 & 2: Evolution of reflectivity of a SiO₂/Si bonding as a function fo temperature and timè (linear and logarithmic scales). The reflectivity decreases globally with increasing temperature or time, indicating a sealing of the interface. The curves are fitted to a model profile (fig3).

The curves can be fitted to model density profiles

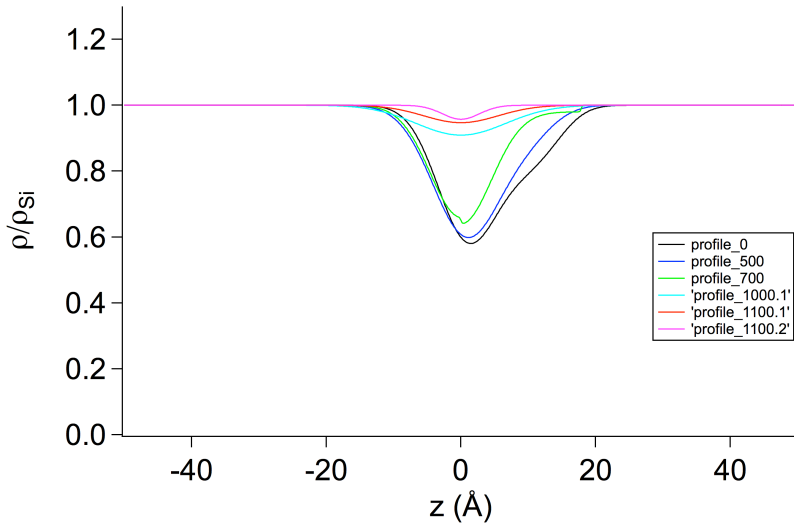


Fig.3 Density profiles as obtained from fig1 & 2 curves showing the sealing of the interface. To model the kinetics the surface area of the profile has been used.

Performing time-dependent experiments allowed one to extract some time constants for the sealing kinetics and demonstrated the absence of full sealing at temperatures < 1050 °C.

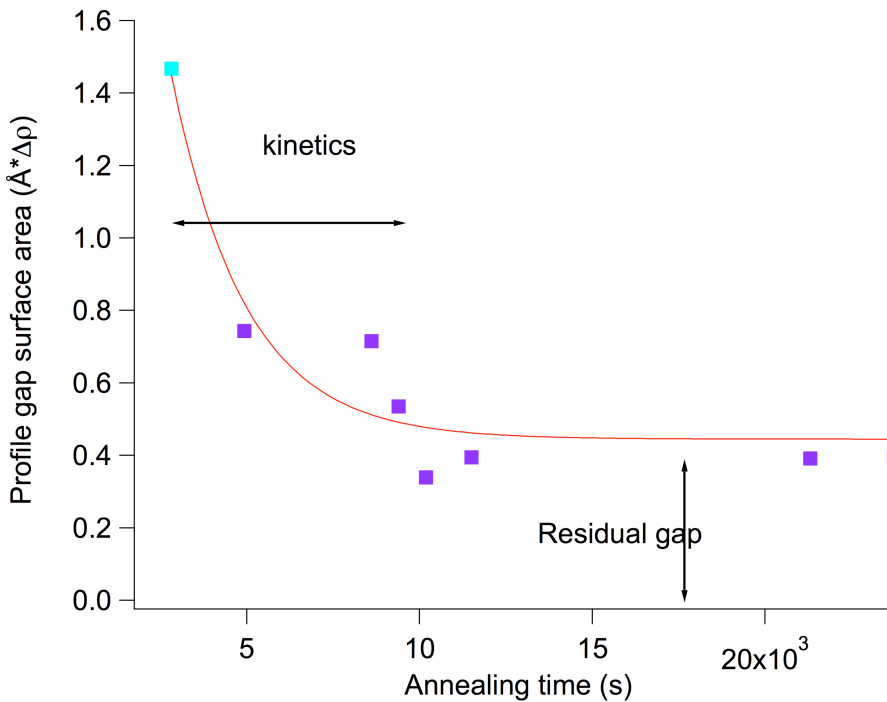


Fig.4. Evolution of the gap profile surface area with time.

Justification and comments about the use of beam time (5 lines max.):

Most of the beamtime has been used despite some difficulties controlling temperature and alignment for these sensitive experiments. The main troubles associated with high temperature were:

- a lack of stability of the furnace when regulating temperature
- a huge temperature difference (>200 °C) between the setpoint temperature of the furnace and the actual temperature (measure from the silicon lattice parameter) whatever the furnaces used.
- A large stress induced by the CTE difference between silicon and the alumina ceramic sample holder resulting in curvatures of the sample

These difficulties were identified and next experiment will take countermeasures:

- reduction of sensitivity to alignment
- use of a furnace capable of 1500°C
- use of clamps to mount the sample