ESRF	**Experiment title:** In situ observation of phase-separation in a molten glass	**Experiment number**: SC-3724
Beamline: ID19	**Date of experiment**: from: 09 nov. 2014 to: 11 nov. 2013	**Date of report**: 1st march 2014
Shifts: 6	**Local contact(s)**: Elodie Boller, Anne Bonnin, Alexander Rack	*Received at ESRF:*

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Report:

Introduction

The aim of this proposal was to continue the investigation of phase-separation in a molten glass by X-Ray tomography, started with proposal HD501 [HD501]. The first experiments allowed us to caracterise the geometry of the growing microstructure by *in situ* observation. We could successfully recover theoretical predictions of the growth law of the domains, and observed a progressive departure from this growth due to a fragmentation of an initially percolating domain [Bouttes14]. In order to explore in more details this fragmentation, and assess the effect of the important viscosity contrast between the two phases, we prepared new compositions to vary the volume ratio of the phases, to perform new experiments in the scope of the proposal SC3724.

Rescheduling and troubleshoot

Initially scheduled on 26-27-28 october 2013, we could not perform any experiment due to vacuum loss in the beamline. This could not be fixed during the initial slot (no vacuum support during weekends and long time required to obtain vacuum again). Fortunately ID19 beamline could provide new beamtime shortly after. The furnace used during the experiment (Ecole des Mines furnace) had an issue during the experiment (electric connection lost between the resistors) but a quick response of ID19 technical suppport allowed us to restart after a few hours without any further problem.

Experiments
Twelve samples with different compositions were observed *in situ* at temperatures from 1180°C to 1400°C, using the “Ecole des Mines” furnace. Most of the samples were treated with at constant temperature; two were first held above the demixion temperature (1380°C), then quenched in situ. The temperature of these samples were monitored during the experiment with an additional thermocouple, to check the heat gradiants within the durnace. The scan times were chosen in order to be shorter than the growth rate, and varied between 6 seconds to 30 seconds, using the PCO Dimax camera and the fast Leuven rotation stage.

The 3D images were reconstructed with ESRF software, then recovered for further treatment and analysis : segmentation, measures of connectivity, volumes, specific surfaces, curvatures, etc.

Preliminary results
Thanks to these expeiments with various compositions, we can demonstrate the effect of the volume ratio of the two phases. There is a clear difference between system were the majority phase is the most viscous one and one it is the less viscous one. On the figure below, we show the minority phase of two system will sumilar volume fraction, but inverting which phase is the minority phase; when it is the most viscous one, we don't observe any fragmentation, whereas when it is the less viscous we observe a fragmentation that will ultimately stop the coarsening, as the hydrodynamic regime the growth requires a percolating morphology.

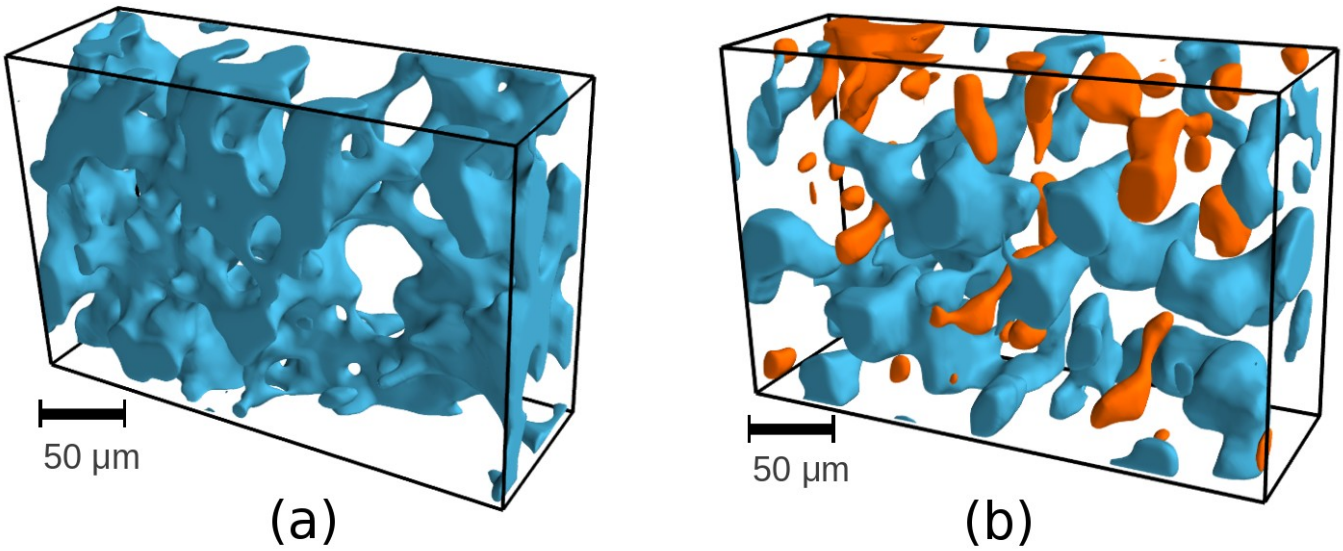

Figure : 3D visualisation of the minority phase after 20 minutes at 1150°C. In both cases the volume fraction is 26%, but in (a) the minority phase is the most viscous one, whereas in (b) it is the less viscous one. The blue domain is percolating, orange domains are isolated (only a fraction of the total image is represented here).

The tomographic experiment performed gives us not only such images, but also the full dynamics of the domains, and after a detailed analysis of the data we expect to be able to give the fundamental ingredients that cause these stricking differences.

References
[HD501] HD501 Experiment report.
[Bouttes14] D. Bouttes et al, arXiv:1309.1724, (2014).