Capturing rapid phase transformations in glass melts thanks to 3-D tomographic imaging

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Materials often undergo rapid phase and microstructural transformations during elaboration or in service conditions. While X-ray tomography is a tool of choice to characterize the 3-D microstructure of materials, in-situ 3-D imaging is a unique way to witness from the inside turning points of such transformations.

I will first describe the development of in-situ tomography on the ID19 beamline of the ESRF, and mention some of the technical challenges that have been successfully overcome. Beamline staff have leveraged several technical developments to provide 3-D imaging with a time resolution that can be as good as one second, while keeping an excellent spatial resolution down to the micron. Moreover, a variety of dedicated set-ups are now available on the beamline, for in-situ conditions such as as heating, mechanical loading, freezing, etc. Such set-ups are nowadays used on a regular basis on ID19, for diverse applications such as food engineering, metallurgy, ceramics processing, etc.

Thanks to the various furnaces available on the beamline, we have been using in-situ tomography at high temperature for the study of silicate melts, both for industrial glasses and model systems. Soda-lime window glass melting is an energy-intensive process, where granular raw materials react at high temperature to form silicate minerals, which then turn into melts. The efficiency of silica conversion depends crucially on the geometry of reactions during decarbonation reactions, that occur in a few seconds in the case of liquid carbonates. I will show how ultrafast tomographic imaging of glass batch reactions resulted in new insights about the geometry of reactions, and the origin of crystalline defects that are hard to dissolve in the industrial process.

As a second application, we studied in-situ the evolution of phases during coarsening of phase-separated silicate liquids. Once quenched, these composite glasses can be transformed into porous materials with a tailored microstructure. In-situ imaging brought to light the critical role of asymmetric viscosity for break-up and fragmentation of liquid bridges, resulting in a complex microstructure with a wide range of scales. Recently, we have started using nanotomography on ID16-A, in order to investigate on quenched samples how such mechanisms transpose at the nanoscale.

Finally, the acquisition of terabytes of image data, often with a challenging speed vs. quality trade-off, calls for efficient and advanced image processing tools. I will present how some algorithms implemented to process ID19 tomography data have been integrated into scikit-image, a general-purpose image processing library for the Python language, that is now used by several groups of users and beamline staff.