EUROPEAN SYNCHROTRON RADIATION FACILITY

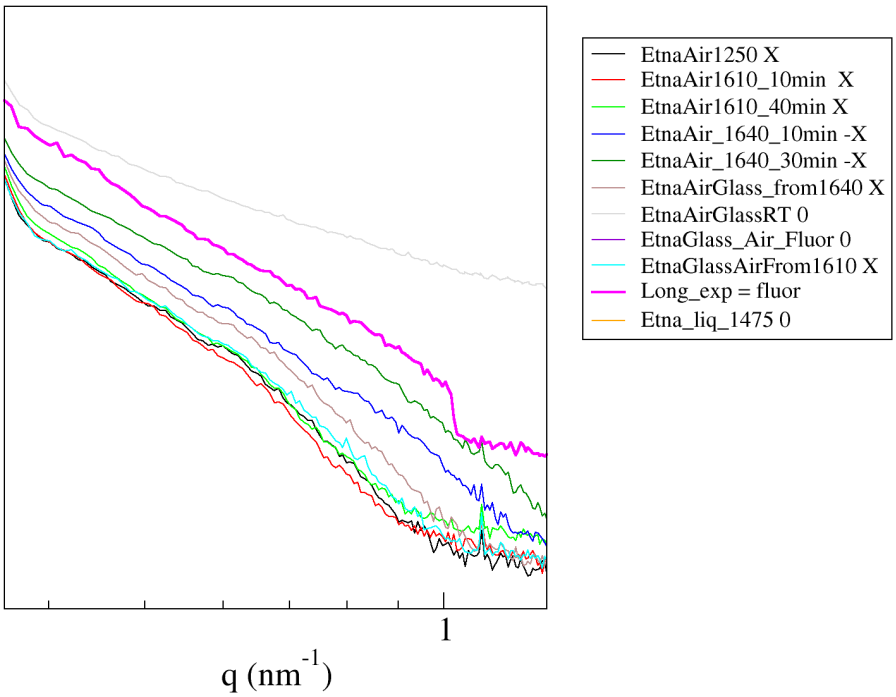
INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: In situ study of nano and micro-crystallisation in volcanic melt under different fO2	**Experiment number**: ES 793
Beamline:	**Date of experiment**: from: 12 Sep 2018 to: 12 Sep 2018	**Date of report**: 18 Sep 2023
Shifts:	**Local contact(s)**: Alessandro Longo	*Received at ESRF:*
Names and affiliations of applicants (* indicates experimentalists): Danilo Di Genova, Louis Hennet, Richard Brooker, James Drewitt and Daniel Neuville		

Report: During our experimental session, our primary focus was on acquiring SAXS data (Fig. below) to investigate the structural evolution during the crystallization of volcanic silicate melts containing iron. Our goal was to study how cooling rate and oxygen fugacity influence the nucleation of iron nano- and micro-oxides at the onset of crystallization and the evolution of the iron oxidation state (Fe2+/Fe3+ ratio) in volcanic melts.

We conducted experiments using the Mt. Etna (Italy) basalt. These experiments involved heating the samples to their liquidus temperatures and then cooling them down to eruptive conditions at oxidizing conditions (in air). Our micro-heating cell setup allowed us to control the oxygen fugacity during these experiments.



While our initial experiments provided some preliminary SAXS data under oxidized conditions, we encountered technical challenges (e.g., signal was too weak under reduced condition of argon) that prevented us from drawing conclusive findings. The absence of the complementary WAXS (Wide-Angle X-ray Scattering) signal was a limitation, as it could have provided crucial information to support our hypotheses regarding nanocrystal formation we inferred from the preliminary SAXS data acquired in air.

Recognizing the need for a more comprehensive dataset, my colleague Alessandro Longo and I prepared a new proposal for DLS, which allowed us to acquire both SAXS and WAXS signals simultaneously. These combined data from DLS were instrumental in our subsequent publication in Science Advances in 2020, authored by Di Genova et al. The publication likely provided more comprehensive insights into the structural evolution and crystallization of iron silicate melts, addressing the limitations we faced during the ESRF session.

In summary, our session at ESRF served as a starting point for our research, highlighting the importance of both cooling rate and oxygen fugacity in nanocrystal formation in volcanic melts.