ESRF	**Experiment title:** Anomalous Small Angle X-ray Scattering study of the spinodal decomposition and precipitate evolution in a martensitic stainless steel during long term thermal ageing	**Experiment number**: 02-01-836
Beamline: BM02	**Date of experiment**: from: april 12th 2013 to: april 16th 2013	**Date of report**: 28/08/2013
Shifts: 12	**Local contact(s)**: Frédéric De Geuser	
Names and affiliations of applicants (* indicates experimentalists): A. Deschamps*, F. De Geuser*, L. Couturier*		

Report:

Anomalous small-angle X-ray scattering at the Cr edge has been used to characterize the nanoscale microstructure of a 15-5 PH precipitation hardened stainless steel during long term ageing. Two main characteristic features determine the properties of this steel: the presence of Cu precipitates, that form during an ageing treatment in the range 505-550°C, and the unmixing of the Cr solid solution by spinodal decomposition, that happends during long term ageing (several thousand hours) in the temperature range 290-420°C. Two sets of experiments were carried out in the reported session. First, the precipitation kinetics of Cu particles was assessed by measuring the evolution of the SAXS signal while heating in-situ samples initially in full solid solution. Second, samples aged for a long term at different temperatures following such a precipitation treatment were measured by ex-situ ASAXS at 6 energies close to the Cr edge, in order to separate (as far as possible) the signal from the spinodal decomposition from the signal of the Cu precipitates.

Description of measured samples:

- Precipitation kinetics of Cu, 4 heat treatments investigated: 505°C and 550°C, with slow and fast ramp heating to the heat treatment temperature
- Ex-situ ASAXS measurements: close to 100 samples, including the effect of temperature (range 290°C-420°C), time (up to 10000h), and stress during ageing (up to 90% of yield stress)

Results

Figure 1 below shows the evolution of precipitate size and SAXS integrated intensity (representative of precipitate volume fraction) during the heat treatments for Cu precipitation. These results show that the nucleation radius of the Cu precipitates is of the order of 1 nm and that they subsequently grow fast to a size of 3 to 4 nm in the investigated time range. As expected the precipitation kinetics is faster at the higher temperature. Changing the heating rate has no significant influence on the precipitation kinetics, which can be explained by the homogeneous nucleation mechanism of Cu precipitates in Fe (forming as BCC Cu particles).

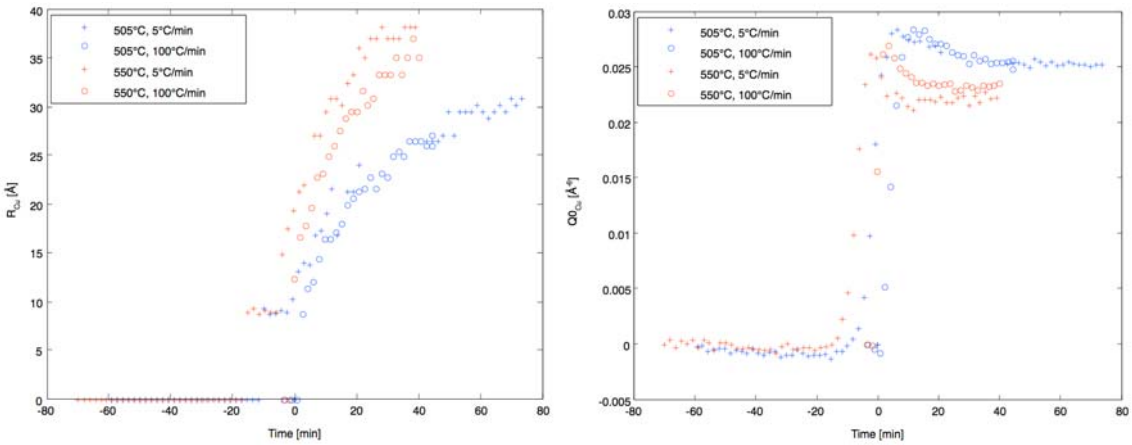

Figure 1: Evolution of precipitate size (left) and intergrated intensity (right) during heat treatment at 505°C and 550°C with two different heating rates

Figure 2 shows the scattering curves (in a Kratky plot) for the 6 energies used (the average of the 6 scattering cruevs is subtracted to all data to highlight the anomalous scatteirng effect) for 4 aging times at 350°C (from unaged to 7500h). In the unaged material, the only anomalous effect is obserbed at low scattering vectors, reflecting the lower Cr content of the Cu precipitates responsible for this signal. When ageing proceeds, a strong anomalous signal is observered to grow at relatively large scattering vectors, reflecting the appearance of spinodal decomposition with nm-range wavelength.
The full analysis of the 100 samples investigated is now under way to quantitatively characterize the influence of ageing time, ageing temperature and stress during ageing on the kinetics of spinodal decomposition.

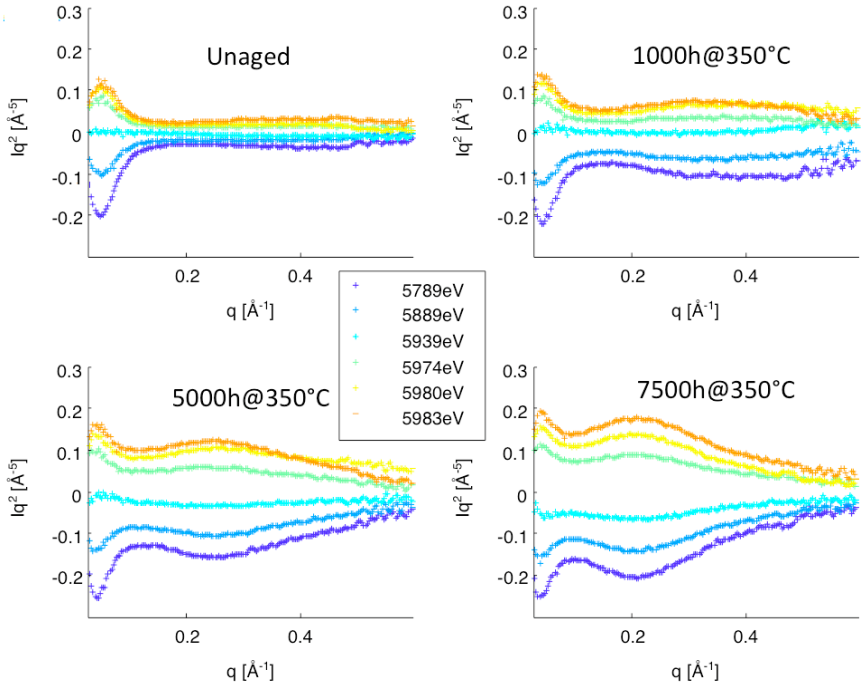

Figure 2: SAXS signal in Kratky plots for the 6 energies close to the Cr edge where the average of the 6 signals has been subtracted to all data to highlight the anomalous scattering effect.

Conclusion

This experiment has been technically extremely successful; a large dataset has been collected, covering the most complete range possible in terms of heat treatment temperatures, heat treatment times and influence of stress. The methodology for data interpretation has been optimised on a few characteristic samples, and the full analysis of the dataset is now under way.