ESRF	**Experiment title:** Combined Small-Angle Scattering and powder diffraction in-situ study or precipitation in the Fe-Si-Ti system	**Experiment number**: MA-699
Beamline: BM02	**Date of experiment**: from: 23-04-09 to: 28-04-09	**Date of report**: 23-07-09
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Characterization of precipitates in the Iron-Silicon-Titanium system by SAXS: in-situ and ex-situ observations

Introduction:

ESRF Small Angle X-ray Scattering was used to study the precipitation kinetics in the Fe-Si-Ti system. In fact, the formation of Fe2SiTi precipitates is interesting to follow, since they are responsible for the increase in hardness and mechanical properties in general of this alloy.

Theses precipitates grow at temperatures between 450°C and 800°C, having radius values between 2nm and 1µm. They are spherical, coherent and ordered which makes them ideal to model their growth kinetics.

The aim of this experiment was to follow, thanks to in situ measurements during heat treatments, the radius and volume fraction evolution in order to use them for precipitation modelling and compare them with ex-situ samples so as to see the influence of ageing conditions.

During this experiment, it was initially planned to study the precipitation kinetics in parallel by in-situ Small-Angle X-Ray scattering and powder diffraction. However, detailed preliminary diffraction experiments in the laboratory showed that due to the relatively low volume fraction of the precipitates and to the pronounced crystalographic texture of the steel, it was extremely difficult to extract information from diffraction experiments. Therefore it was decided to concentrate for this experiment on the small angle scattering method.

Experimental conditions:

The studied alloy is of the composition Fe-2.5wt%Si-1wt%Ti. Iron and Titanium having close absorption energies (7.1keV and 5keV), it was thought of interest to work at a much higher energy in order to improve the contrast between the precipitates and the Fe matrix. Therefore the energy used for the experiment was 17keV on the beamline BM02.

In order to have the best transmission as possible, samples were polished mechanically until reaching a thickness close to 40µm (the ideal being 20µm, though very difficult to reach).

During the experiment two kinds of initial conditions were observed:

- ex-situ using samples which had been heat-treated prior to the experiment

- in-situ for temperatures comprised between 450°C and 600°C using samples which had been either simply quenched at 900°C for total dissolution of particles or aged at 900°C followed by a pre-precipitation treatment at temperatures between 450°C-650°C during 10minutes.

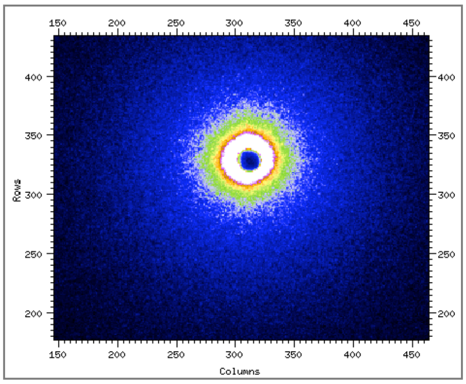

Figure 0: scattering image due to the spherical precipitates obtained by

Ex-situ measures were performed using a grid to position the samples. Most of them had sufficient transmission to provide a good signal (see Figure 0). Hence, even samples aged at low temperatures, thus having with very small precipitates (~2-3nm) could be analysed

The furnace used for in-situ experiment is the SIMAP furnace developed internally. However, due to problems encountered during the heating of the furnace using the beamline power supply, it had to be replaced by another one, much less powerful. The maximal theoretical temperature for the use of the second furnace was 500°C, which we exceeded for the interest of the experiment. Thus the temperatures reached during the four days of experimentation might have been not very precise, due to the overheating of the elements of the furnace.

As a consequence, for each temperature several samples were used in order to make reproducibility tests. An example is the In-situ tests performed for samples which had been aged at 550°C. The analysis of the signals showed a good reproducibility between each sample, and an effect of the pre-precipitation treatment prior to in-situ observation (Figure 1). In fact, Figure 1 shows that the volume fractions are all consistent with each other while the radius seems influenced by the pre-precipitation. However, these observations were not confirmed by SANS experiments at ILL performed one month later on equivalent samples, which tend either to show no effect of pre-precipitation. Comparison with ex-situ samples (see Figure 1) shows a good compatibility of radius values with the samples which were pre-precipitated but not for the volume fraction.

Another reason for questioning is the analysis of samples at 520°C. They are not at all consistent with those at 550°C or 580°C (Figure 2). It could be explained by a bad control of the furnace at temperatures which were not in its design range.

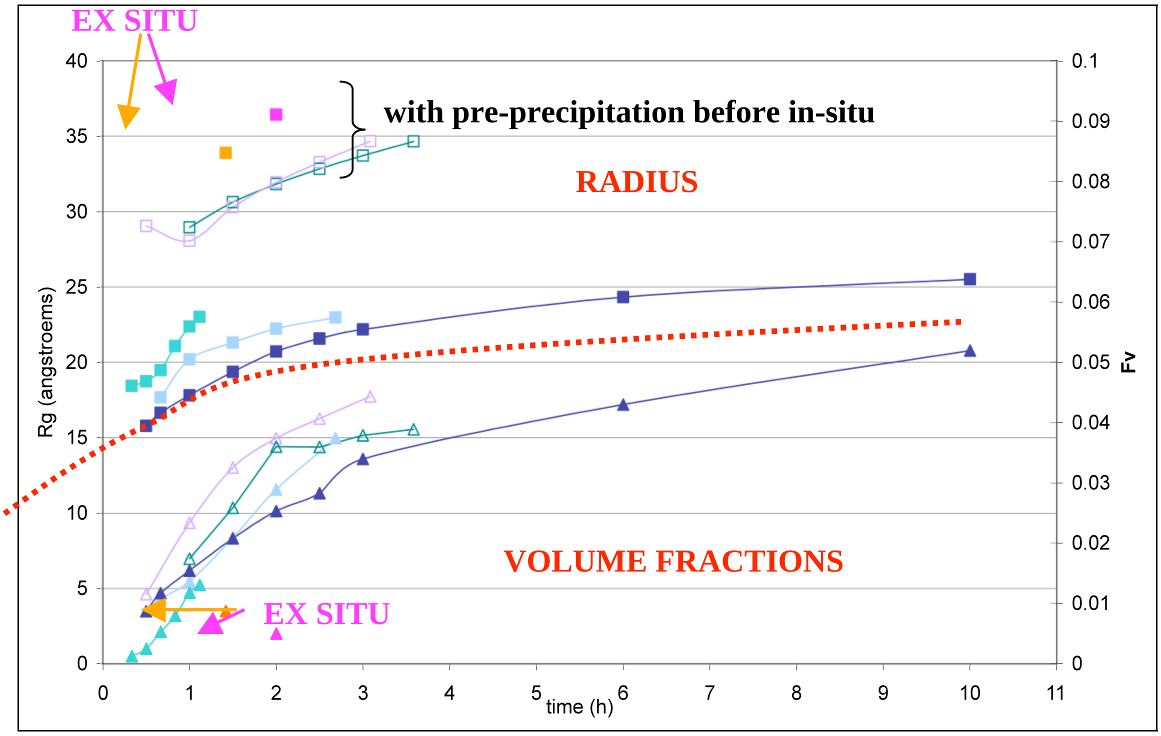

Figure 1: influence of the pre-precipitation treatment prior to the in-situ SAXS experiment for samples aged at 550°C

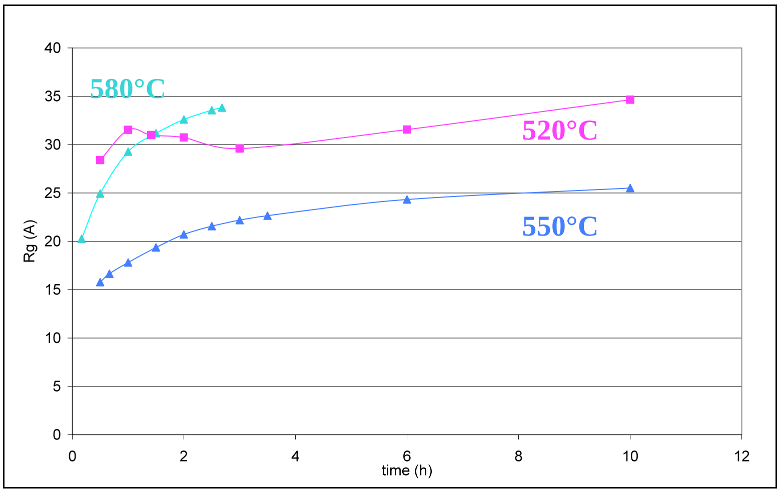

Figure 2: influence of temperature on radius evolution as a function of time

Conclusion

Ex-situ experiments worked out very well, and the choice of the energy was appropriate, as we can see from the very good contrasts of the signals. Concerning the in-situ tests, the bad working of the furnace leaves a doubt on the credibility of the results, especially since they were not confirmed by parallel SANS experiments.