ESRF	**Experiment title:** In-situ study of precipitation kinetics in Fe-Cu alloys by means of Small-Angle X-ray Scattering	**Experiment number**: 02-01-601
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Report:

ESRF Small-Angle X-ray Scattering was used in order to obtain results on the evolution of the precipitation state during In-Situ aging of Fe-1.4 wt%Cu and Fe-0.8 wt%Cu alloys. The aim of this experiment was to monitor the volume fraction and Guinier radius of particles during isothermal and anisothermal agings as a function of time.

Numerous results were obtained, and we will only present selected results for Fe-1.4wt%Cu alloy.

Details of the experiment :

Several heat treatments were performed :

For Isothermal In-Situ agings, samples were annealed at 845°C for 5 hours and water quenched.

For Ex-Situ characterizations, samples were annealed at 845°C for 5 hours and water quenched, followed by aging at different temperatures for a range of times. (see table 1).

For non-isothermal In-Situ agings, we have selected one of the precipitation states in table 1, and treated the sample at a higher temperature.

Table 1 : Times of aging for each temperatures

500°C	2h	20h	200h	1000h

A small angle setup was chosen to well characterize particles from 5Å to 100Å, corresponding to scattering vectors from 10^{-2} to 0.2$Å^{-1}$. For each measurement, the volume fraction and Guinier radius of the particles were determined.

Results :

Isothermal agings

We can see in fig. 1 and 2 the evolution of particle radius and volume fraction for three different temperatures.

In-Situ aging at 500°C allows us to deduce from the very first time of the heat treatment the nucleation radius. We can also notice that the results of the In-Situ and Ex-Situ experiments are in very good agreement at 500°C. The combination of In-Situ and Ex-Situ aging gives access to the major part of the precipitation kinetics, from nucleation to coarsening.

The comparion between the three aging temperatures let us know that the nose of the Temperature-Time-Transformation curve seems to be located between 600 and 700°C for this alloy.

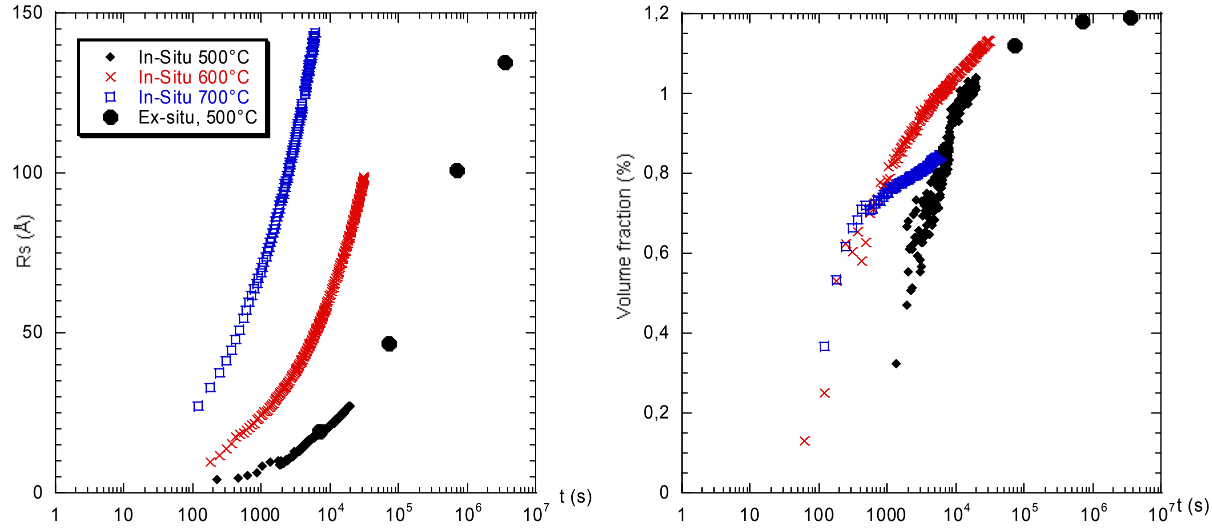

Fig. 1 and 2 : Evolution of particle radius and volume fraction vs. time for isothermal In-situ aging at 500°C, 600°C and 700°C, and Ex-Situ aging at 500°C.

Non-isothermal agings

The In-Situ experiment is a very good tool for the study of non-isothermal aging. In this case, we have taken a sample aged 20 hours at 500°C and held it at 675, 700 and 725°C. (fig. 3 and 4).

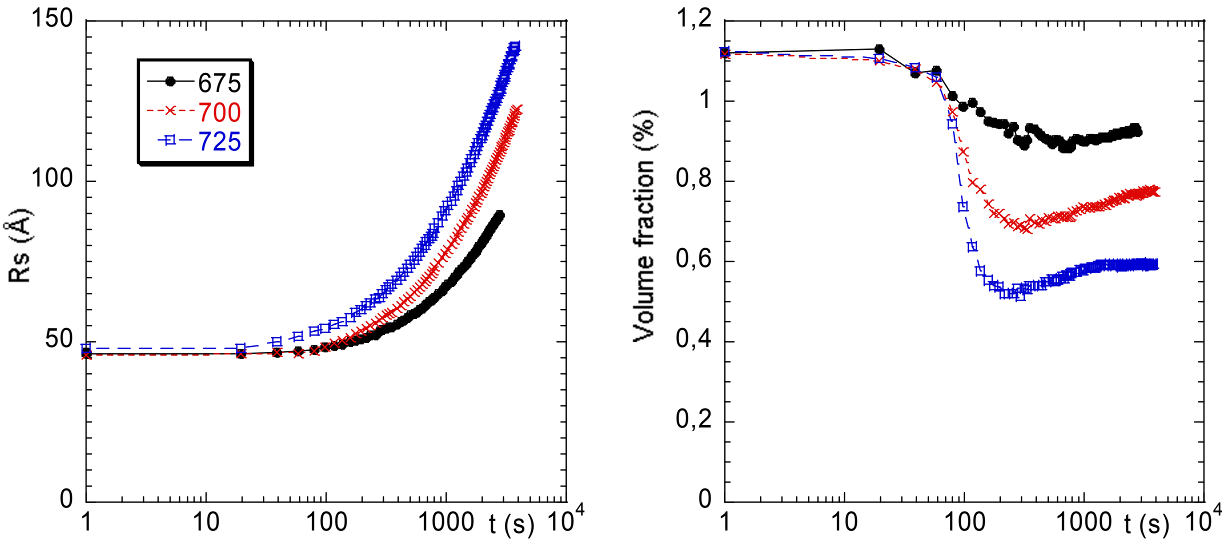

Fig. 3 and 4 : Evolution of particle radius and volume fraction vs. time for anisothermal In-situ agings at 675°C, 700°C and 725°C, based on a 500°C, 20h precipitation state.

Those experiments can be divided into two parts : first, a dissolution of the precipitates at a constant mean radius (from 1 to ≈100s), and secondly growth and coarsening, where the radius is increasing and the volume fraction is reaching the equilibrium volume fraction (for long times).

It is clearly shown here that the higher the aging temperature, the greater the dissolution.

<u>Conclusions :</u>

This experiments has been very usefull and a lot of information has been recorded. We are grateful to the ESRF staff, and especially to the BM02 beam line for their help.