ESRF	**Experiment title:** Study of precipitation kinetics in Fe- 0.8wt%Cu alloy by means of Small-Angle X-ray Scattering	**Experiment number**: ME254
Beamline: ID01	**Date of experiment**: from: 12.12.01 to: 16.12.01	**Date of report**: 23.05.02
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Report:

ESRF Small-Angle X-ray Scattering was used in order to get results on the evolution of the precipitation state during aging of Fe- 0.8wt%Cu alloy in terms of volume fraction and Guinier radius of particles.
This alloy was used in order to understand the influence of heterogeneous nucleation sites (dislocations) on the kinetics of precipitation.

It is now generally accepted that the following sequence is characteristic of precipitation in this system :

$$\alpha \text{ (supersaturated solid solution)} \rightarrow \text{BCC copper} \rightarrow \text{9R copper} \rightarrow \text{FCC } \varepsilon$$

Initially, metastable body centered cubic (BCC) precipitates which are fully coherent with the matrix are observed. When the precipitates reach a critical size; i.e. a radius in the range 2.3nm to 3nm, the coherency strain energy becomes too large and a martensitic transformation to the 9R structure occurs. The 9R structure has a face centered cubic lattice with a high density of twins, which help to minimize the misfit with the iron matrix. Finally, at larger precipitate sizes, the twins disappear and the precipitates attain the equilibirum ε phase; i.e. face centered cubic.

It is also known that dislocations can have a strong effect on the nucleation sites for copper precipitates

Details of the experiment :

Three different initial states of an Fe- 0.8wt%Cu alloy were prerared :

Simple solution treatment at 845°C for 5 hours and water quench

Simple solution treatment at 845°C for 5 hours and water quench followed by a 1% deformation (tensile deformation)

Simple solution treatment at 845°C for 5 hours and water quench followed by a 10% deformation (cold rolling)

After that, different aging temperatures and times were applied for the three initial states and were investigated at ESRF (see table 1):

Table 1 : Times of aging for each temperatures

400°C	50h	150h	300h	600h	
500°C	5h	15h	30h	110h	300h
600°C	2h	6h	12h	44h	120h

A small angle setup was chosen to well characterize particles from 5Å to 100Å, corresponding to scattering vectors from 10^{-2} to $0.3Å^{-1}$. For each measurement, volume fraction and Guinier radius of the particles were determined.

Results :

Numerous results were obtained, and we will only present results at 500°C.

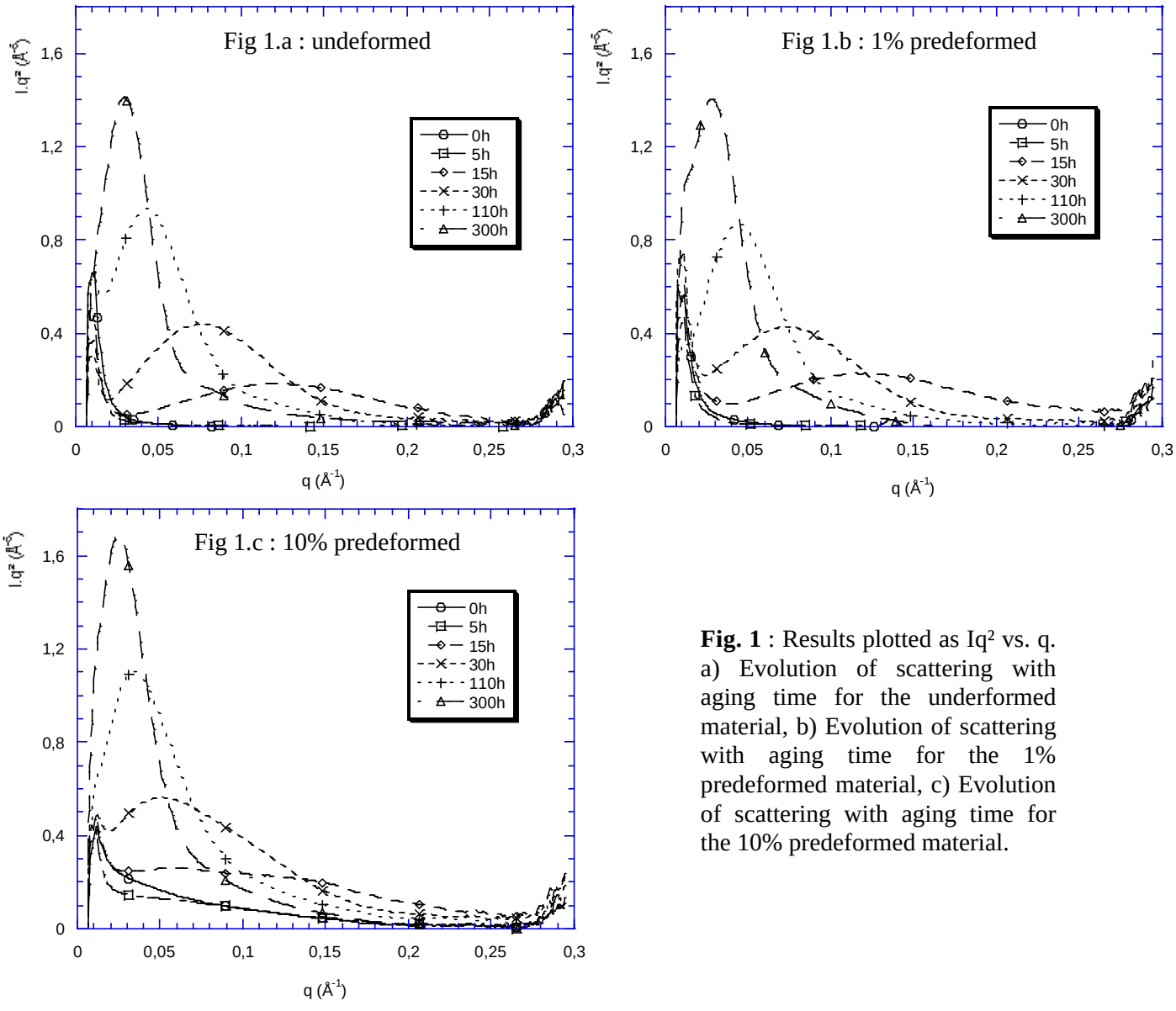

Fig. 1 : Results plotted as Iq^2 vs. q. a) Evolution of scattering with aging time for the underformed material, b) Evolution of scattering with aging time for the 1% predeformed material, c) Evolution of scattering with aging time for the 10% predeformed material.

We can see in fig. 1 the evolution of scattering amplitude with aging time at 500°C for the three different deformations. The difference between 0 and 1% predeformation is not very important (the predeformation is not sufficient to have a strong influence on precipitation state). This difference is stronger after 10% predeformation : the Iq^2 vs. q curves are shifted to the small q-range, and the integrated intensity is more important than for samples without predeformation.

It is also usefull to notice that a 10% predeformation gives double diffraction for small q. It is very difficult to deal with this double diffraction because all samples are different and we cannot substract this background easily. An in situ set of experiments could be usefull in order to avoid this kind of problems.

More quantitatively, fig. 2 and 3 show the evolution of particle radius and invariant Q0 vs. time.

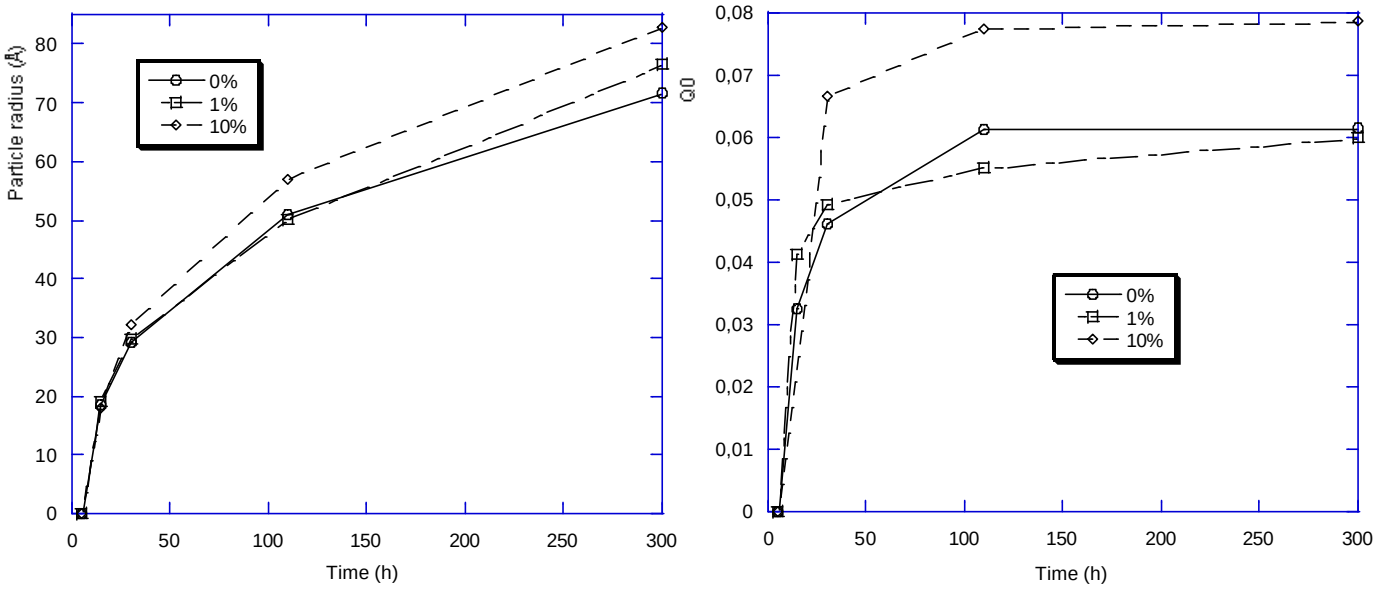

Fig. 2 and 3 : Evolution of particle radius and invariant Q0 vs. time for aging at 500°C.

It is clearly shown here that the differences between 0 and 1% predeformation are very small. But for the 10% predefomation samples, particle radius and integrated intesity are bigger at a given time.

Conclusions :

This experiments has been very usefull and a lot of informations has been recorded. We are grateful to the ESRF staff, and especially on the ID01 beam line for their help.