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Application for beam time at ESRF – Experimental Method

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Aims of the experiment and scientific background

Study of Cu precipitation in ferritic iron is of technological importance considering its strengthening effect. Generally, the precipitation sequence of Cu can be described in terms of the formation and growth of coherent BCC Cu-rich clusters, transformation of these to transient phases with the crystallography 9R[1] and 3R[2] and finally to incoherent pure FCC-Cu precipitates. However, there are still ambiguities about the exact nature of the initial forms of Cu-precipitates (size, volume fraction, evolution of precipitate composition…), which seem the more efficient for hardening. Such a problem is more difficult to study as the nature of such precipitates is very likely to change with the alloy chemical composition. Moreover, to our knowledge there is no systematic study carried out to quantitatively characterize the effect of a single third element on the Cu precipitation. Such knowledge is indeed necessary for the identification and modelling the precipitation strengthening. Present study is a part of such a fundamental approach whose final aim is to reach to a clear understanding of the effect of an usual alloying element (C or Mn) in ferritic steels on the precipitation state of Cu and thereafter kinetics of precipitation hardening under isothermal aging conditions.

To summarize, on a scientific viewpoint, the study of Cu precipitation in steels combines a number of interesting problems:

- Ambiguities in the precipitate state and hardening mechanisms at the onset of Cu precipitation.
- No clear understanding of the effect of third elements on the precipitation kinetics of Cu.

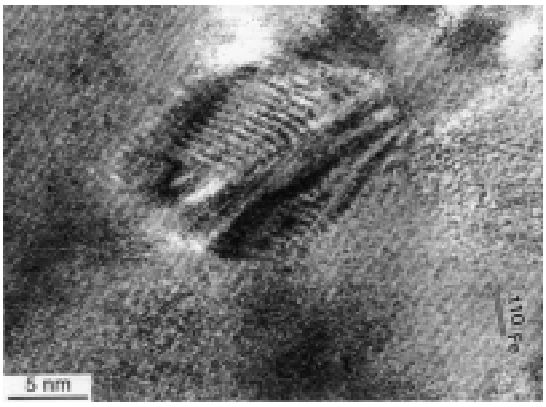

Fig. 1. HRTEM image of Cu precipitate in a complex alloy aged at 923 K for 1.8ks. [3]

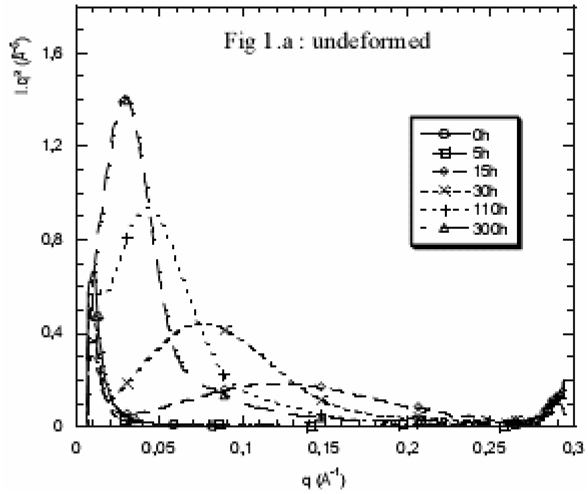

Fig. 2 : SAXS spectra obtained on Cu precipitates during ageing at 500°C for a 0.8wt%Cu alloy [4]

Precipitate Characterization

The characterization of Cu precipitation in the underaged condition is difficult because of the following reasons: the size of precipitates is very small (~2nm at peak aged condition), as well as their volume fraction (usually not more than 0.1 %). Most of the studies carried out up to now in the isothermal aging condition have relied on High Resolution Transmission Electron Microscopy (HRTEM) to characterize the precipitation of Cu (Fig.1) [3]. The use of HRTEM was justified by the lack of contrast between Cu-rich precipitates and an iron matrix in conventional TEM. Moreover, the smallness of area investigated by

HRTEM together with the heterogeneity from grain to grain have made it extremely difficult to get reliable quantitative data such as mean precipitate size, histogram of sizes average volume fraction etc. These parameters are necessary to model the precipitation hardening behaviour. Small-angle scattering techniques have demonstrated that it is indeed possible to quantify such small sized precipitates. SAXS has been used on a similar alloy at ESRF [4] during Me-254 experiments on ID01 for the study of Cu precipitation in a 0.8wt%Cu. Fig. 2 shows a small part of the results obtained by SAXS. Hence, we are expecting to obtain the mean values of both particle size together with the size distribution of the particles by means of the experiments to be carried out at ESRF. Such results will be complementary to other more local ones obtained by Tomographic Atom Probe (TAP) and HRTEM.

Aim of the experiment

The aim of the present experimentation is to determine quantitatively the effect of the concentration of either Mn or C on isothermal precipitation kinetics of Cu in ferritic iron in the range 500-650°C. The particle size is expected to be between 1 and 10nm in diameter. A special emphasis will be placed on characterizing the size distribution and volume fraction of the small particles in the under-aged samples.

Experimental method

Study Cu precipitation by scattering techniques is difficult: conventional X-ray sources are not successful because of the low signal. In the present case, a standard SAXS set-up will be used, detection will be ensured by a high quality CCD camera. In order to compensate for the low contrast between Cu precipitates and ferrite, measurements will be achieved close to the Fe K edge (at 7.104 keV). For this type of experiments, the BM2 beamline (D2AM) is particularly suitable.

The high flux available at ESRF will enable to study the precipitation processes in-situ in temperature. For this purpose, a specially designed furnace, capable of more than 800°C has been designed with a special vacuum chamber in order to reach secondary vacuum (10^{-7} torr). This equipment has been successfully used earlier at ESRF [5]. Present experiments are planned to be carried out in-situ and some in the pre-aged conditions.

Seven different alloys will be characterized including a base Fe-1.2wt%Cu alloy and others with additions of 250 to 750 wt ppm C or of 0.5 to 1.5 wt% Mn. Various states of the precipitation process at two different temperatures of aging will be investigated, with emphasis on the under-aged samples. For higher temperatures (~600^{o}C) and for shorter aging times (~10min) an insitu heating system [5] will be used. All other samples will be in the pre-aged conditions (total approximates to ~100samples). Alloys have been prepared from high purity iron in order to prevent the effect of other unwanted elements on the Cu-precipitation with the further advantages of eliminating other potential scattering sources. The SAXS signal from samples containing precipitates will be compared to the signal from references samples with the same ferritic structure.

Given the number of samples (~100) to be measured and the low signal expected, **we apply for 12 shifts.**

Results expected

We expect to get a quantitative description of the precipitation kinetics (both in terms of size and volume fraction) of Cu in ferritic steel, as a function of temperature, initial solute concentration and the presence of third element (Mn or C) in the material. These results will be compared to a detailed HRTEM study, and will be used for modelling the precipitation process in the low alloy steels.

References

1. Othen, P. J., Jenkins,M. L., and Smith, G. D.W., 1994, Phil. Mag. A, 70, 1.
2. Monzen, R., Jenkins,M. L., and Sutton, A. P., 2000, Phil. Mag. A, 80, 711.
3. Naoki Maruyama, Masaaki Sugiyama, Takuya Hara and Hiroshi Tamehiro., Materials Transactions, JIM, Vol. 40, No.4 (1999) pp. 268-277
4. F. Perrard and A. Deschamps, ESRF report no. ME254, May 2002.
5. F. Perrard and A. Deschamps, ESRF report no. 02-01-601, Oct 2003.