ESRF	**Experiment title:** Time resolved scannning SAXS during in situ heating of Cu/Cu-Co diffusion couples to study the effect of supersaturation on precipitation	**Experiment number**: MA1443
Beamline: BM02	**Date of experiment**: from: may 10th 2012 to: may 14th 2012	**Date of report**: 28/08/2013
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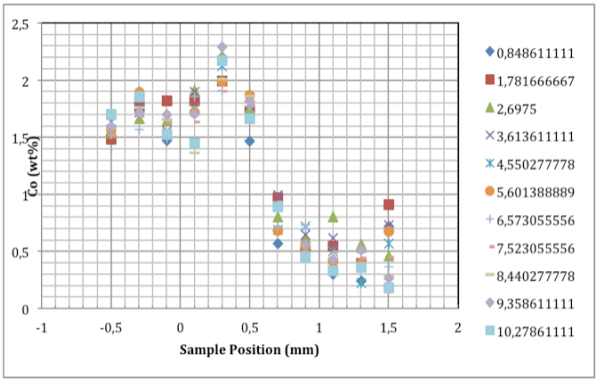
Report:

Diffusion couples were realised between pure Cu and a Cu-2%Co alloy in order to investigate the effect of Co composition on the precipitation of this phase. After processing and homogenisation of the couples a suitable dimension of the diffusion profile of Co was obtained on about 2 mm. The diffusion couples were then solutionized and quenched into water to obtain a full solid solution of Co. Thin slices of final thickness about 40 µm were prepared from these diffusion couples by carefull cutting and polishing.

These slices were then heated in-situ in the temperature range (XXX) in a dedicated furnace while measuring continuously the SAXS signal at several positions along a line-scan on the sample. In this way, the kinetics of precipitation at one temperature becomes available for all compositions present in the diffusion couple in only one experiment.

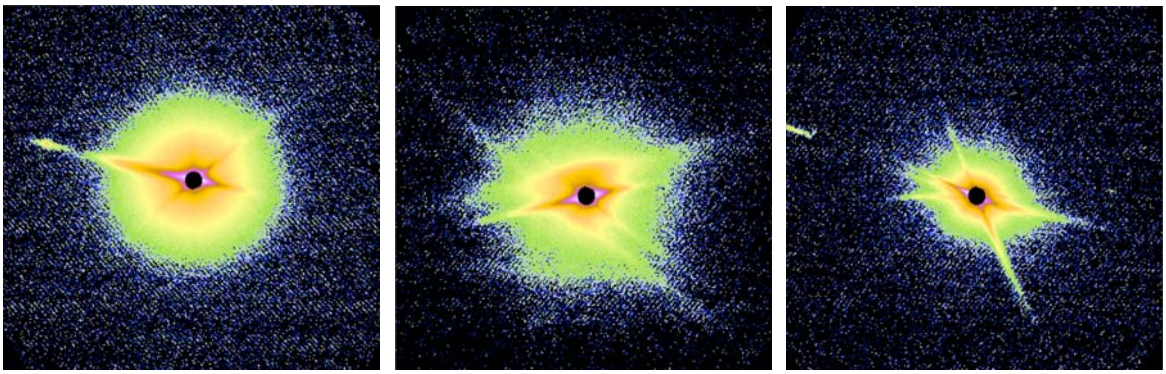
It is obviously extremely difficult to locate precisely the position of the diffusion couple within the furnace with respect tp the X-ray beam with sufficient precision to determine the exact local composition where measurements are done. In order to know precisely the diffusion couple location, the following method was applied: a line scan of the sample was realised while measuring the sample transmission of the sample at two X-ray energies, one below the Co edge and one above. The ratio between the two transmissions can be quantitatively related to the Co composition, since the absorption of the sample above the Co edge is highly dependent on the composition in this element. Such measurements were repeated sevefral times during the heat treatment top make sure that the sample did not shift. The figure below shows that the coiuple location could in this way be located precisely and that the sample did not shift with time.



A very large number of data have been recorded:

- X heat treatments at different temperatures
- 11 locations of measurement along the diffusion couples for each heat treatment
- about 100 measuements for each kinetics at one location

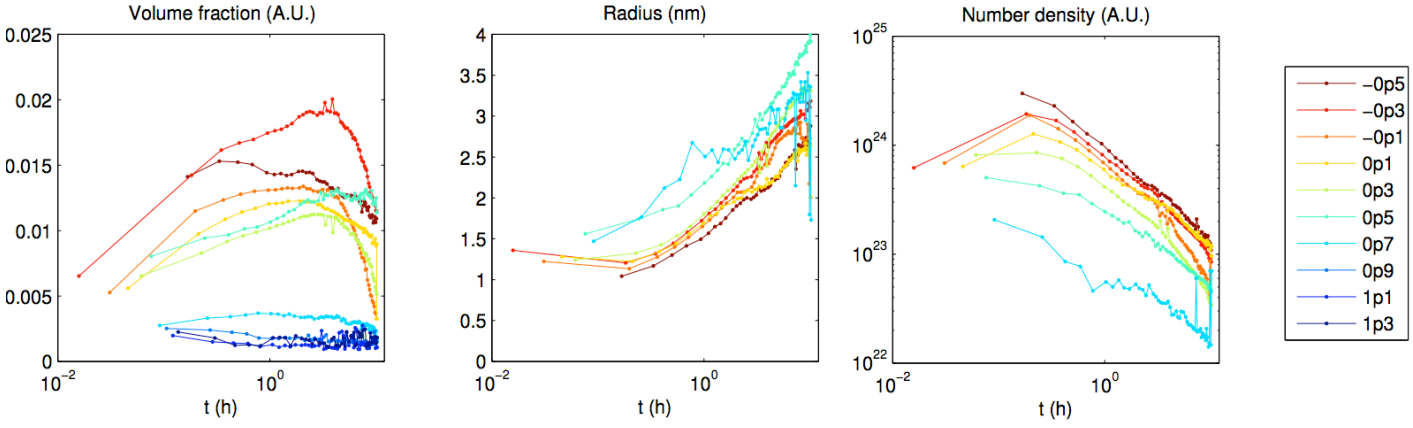
Therefore the analysis of the data needed to be fully automatic in terms of determination of precipitate size and volume fraction. In principle, precipitation in the Cu-Co system is a very simple case as far as data analysis is concerned, because precipitates are spherical. Actually the analysis proved to be much less simple than expected due to unexpected parasitic streaking undoubtedly due to multiple Bragg diffraction and the particular mosaicity within the large grains fo the samples. The series of SAXS images below, at one heat treatment time across the diffusion couple, illustrate both the variation in precipitate scattering (isotropic signal) and the presence of the anisotropic streaking.



Heat treatment 1h at 550°C ; from left to right : Cu-2%Co ; Cu-1%Co ; pure Cu

Therefore, a specific procedure needed to be devised to correct for the streaks and extract the isotropic scattering, which is representative of the presence of the sperical Co precipitates. After the application of this procedure, the evolution of all parameters of the precipitates can be followed for all positions in the diffusion couple and all times of the heat treatment. The results show very interesting features, such as the evoluttion of the maximum volume fraction with solute content, of the precipitate number density and the effect of solute content on the evolution of precipitate radius.

Now that the procedure has been applied to one heat treatment (550°C), ongoing work is in progress to apply it to all heat treatments investigated. The nbext step will ten be to apply a precipitation model to this large dataset.



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 and solute contents. Despite the fact that data analysis has proved to be more complicated than expected, a specific procedure has been divised and the final analysis is no in progress.