ESRF	**Experiment title:** Study of precipitation in Al-Zn-Mg-Cu alloys using very small angle scattering	**Experiment number:** 02.1.066
Beamline: D2AM	**Date of experiment:** from: 30/04/99 to: 03/05/99	**Date of report:** 28/07/99
Shifts: 9	**Local contact(s):** Frédéric Livet	*Received at ESRF:*

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Report:

The aim of this experiment was to characterise the precipitation microstructure in two Al-Zn-Mg-Cu alloys (7040 and 7050) subjected to various heat treatments (two different quench rates followed by various times along the ageing sequence). The purpose of this experiment was to use this microstructural characterisation to interpret and eventually model the effect of heat treatments on the toughness of these materials.

Experimental methods

The precipitation microstructure in these materials consists in two well-separated precipitate families :

- intragranular (inside the grains) precipitates, in the range 10 to 100Å
- intergranular (at the grain and subgrain boundaries) in the range 500 to 1000Å.

The energy used in this experiment was 7.8 keV.

For the measurement of the first family, a standard setup was used : relatively opend slits, sample - detector distance of about 60 cm, 1.5 mm beamstop. For the measurement of the second family, a more advanced setup was used, which has been developped by F. Livet and F. Bley : beam size on the sample was as low as 50µm x 80µm, sample-detector distance was over 2m, beamstop diameter was 1mm, resulting in the reliable measurement of scattering vectors down to 10^{-3} $Å^{-1}$.

Detection was achieved through a CCD camera. Up to now results have been interpreted by proceeding to a circular average of the signal around the beamstop and thus obtaining a single I(q) curve for each sample. Study of precipitation anisotropy with respect to the rolling direction will be carried out later.

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Results

Experiments have been performed on a rapid (cold water) and a slow (boiling water) quench, both on 7040 and 7050 materials. For each of these states, 4 heat treatments were characterised : naturally aged, under-aged, peak-aged and over-aged. On each sample 9 measurements were carried out in separate locations in order to check for reproducibility.

The influence of quench rate on the evolution of the microstructure is clearly shown in figure 1a : after a slow quench and an ageing treatment one can clearly separate the two precipitate distributions (intergranular and intragranular). The intragranular precipitation is not much affected by the quench rate, as expected, but the precipitates on grain and subgrain boundaries are much more numerous after a slow quench. From the integrated intensity one can estimate their volume fraction to 10% of the total volume fraction, or approximately 0.4%.

Figure 1b shows the influence of ageing on the two precipitate distributions in the 7050 alloy. As expected, the volume fraction and size of the intragranular precipitates increases with ageing time. The intergranular precipitation shows no strong evolution, except for the longest ageing time, which may however show problems of peak deconvolution.

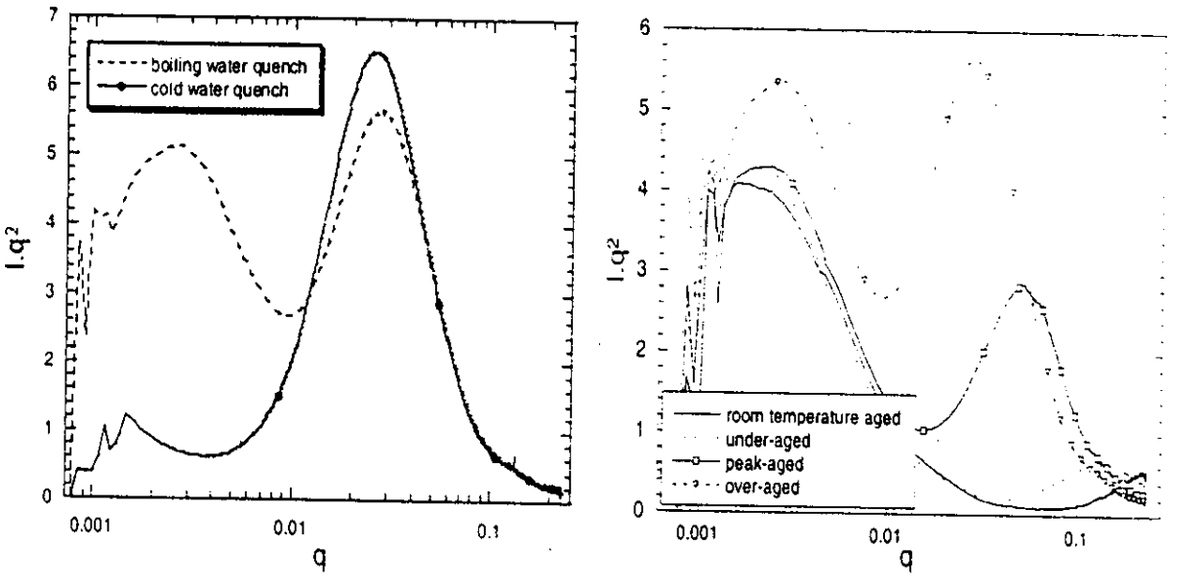

Figure 1 : X-ray spectra for the 7050 material. a) influence of quench rate on the microstructure of the overaged specimens ; b) influence of ageing time on the slowly quenched materials.

7050	Cold water quench	Boiling water quench	
	Intragranular	*Intragranular*	*Intergranular*
Under-aged	12.6	12.5	997 (0.29)
Peak-aged	30.4 (3.53)	33.9 (3.4)	757 (0.38)
Over-aged	68.2 (4.73)	61.5 (4.44)	703 (0.39)

Precipitate sizes (in Å) and volume fraction (in brackets, %) calculated from the X-ray spectra for the 7050 material

Conclusions

In this experiment we have been able to characterise precisely the precipitate microstructure in Al-Zn-Mg-Cu in the range 10Å - 1000Å, demonstrating the usefulness of a very-small angle setup at D2AM and responding to our experimental needs. We have been notably able to separate the intergranular and intragranular precipitate families in terms of size and volume fraction.

Further improvements could include measurements to even smaller angles (a few 10^{-4} $Å^{-1}$), and reducing the electronic noise of the CCD camera.