ESRF	**Experiment title:** Secondary Ageing Study in Al-Cu-Mg alloys by Small Angle X-Ray Scattering	**Experiment number**: ME-504
Beamline: BM26B	**Date of experiment**: from: 05 February 2003 to: 08 February 2003	**Date of report**: 27 February 2004
Shifts: 9	**Local contact(s)**: **Dr. Sven HOFFMANN (e-mail: hoffmann@esrf.fr)**	*Received at ESRF:*

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Report:

1. Aim of the experiment

Our work addressed secondary ageing, i.e. the structural re-organisation that may occur in precipitation-hardenable alloys near to or at room temperature after a thermal treatment at a considerably higher temperature [1]. Scientific attention to secondary ageing phenomena is rather recent, ensuing the discovery of the beneficial effects that they may have on the mechanical properties of alloys [2, 3]. Much has still to be understood on the nanostructural aspects of the problem: morphology (size, crystal structure, spatial dispersion) and chemical composition (chemical internal order) of the precipitates, kinetics of precipitation, etc. All this can be addressed only by using a multiplicity of experimental techniques, among which SAXS plays a very important role.

Our original proposal addressed Al-Cu-Mg alloys, where secondary ageing had been observed for the first time [4]; in the mean time, however, it was discovered by Positron Annihilation Spectroscopy (PAS) that spectacular secondary ageing phenomena occur also in Al-Zn-Mg alloys [5]. We thus decided to focus our attention to these materials, in the framework of an ambitious research programme that combines SAXS with PAS, TEM, differential scanning calorimetry (DSC), electrical and mechanical properties measurements.

2. SAXS measurements

The material chosen for SAXS experiments was a laboratory system obtained from pure elements; we adopted the same basic composition (Al- 4.8 wt. % Zn – 1.3 wt. Mg) of the commercial alloy AA7020, which is a typical high-strength material for automotive and aeronautical applications. The samples were shaped and prepared in Turin in the form of supersaturated solid solution (SSSS), then brought to Grenoble in a liquid nitrogen Dewar flask. The SAXS experiments were performed on the beamline BM26B at the fixed energy of 12 keV. The scattered radiation was detected by a 2D multiwire chamber. The following heat treatments were studied:

a) RT evolution (natural ageing)
b) Evolution at 150°C
c) RT evolution after 30 min at 150°C
d) Evolution at 95°C after 4 days at RT

The results were analysed according to standard procedures [6] to obtain the Guinier radius and the volume fraction of the precipitates.

3. Results and publications

So far, the results of the SAXS experiments on Al-Zn-Mg have been partially published in two papers [7,8] where they have been read in the light of other data coming from DSC, PAS, TEM and microhardness measurements. The main information specifically given by SAXS can be summarised in the following points:

a) the Guinier radius of GP zones formed at RT during secondary ageing is about 10% smaller than those obtained with natural ageing; much bigger GP zones are formed at 95°C (see Fig. 1, upper panel)
b) the number density of GP zones (proportional to the parameter ρ shown in Fig. 1, lower panel) saturates at approximately the same level with primary and secondary ageing at RT; it decreases, as expected to occur for a coalescence process, at 95°C
c) secondary ageing at RT after preliminary ageing at 150°C for 30 min gives rise to a bimodal distribution (see Fig.2).

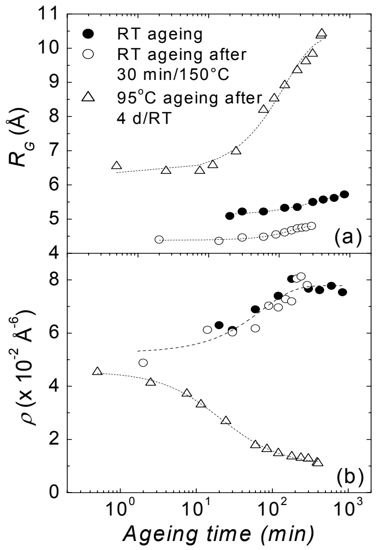

Figure 1: Evolution of the Guinier radius and of the number density of GP zones

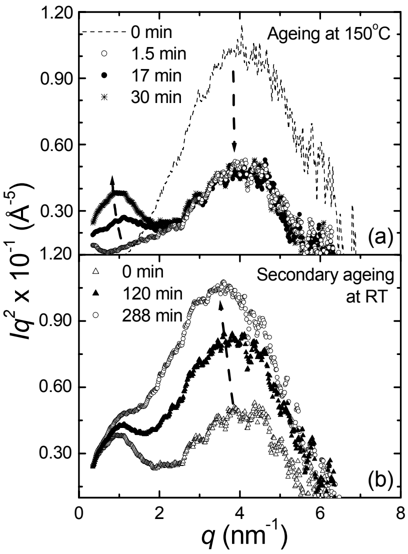

Figure 2: Evolution of the SAXS distribution during primary and secondary ageing

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