Neutron Small-Angle Scattering in Aged Al–Mg

BY J. M. RAYNAL

Faculté des Sciences, Université de Rouen, 76130 Mont-Saint-Aignan, France

AND M. ROTH

Institut Laue-Langevin, B.P. 156, 38042 Grenoble Cedex, France

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Neutron small-angle scattering in polycrystalline Al–Mg alloys aged nine months at 0°C has revealed the existence of Guinier-Preston zones in an Al–11.5% Mg alloy, but no G.P. zones in an Al–7% Mg alloy. The evaluation of the data for the 11.5% Mg alloy gives the following parametric values: mean radius of the zones, 19 Å; mean distance between them, 56 Å; Mg concentration in the zones, about 19%; in the matrix, about 8%. A qualitative model is proposed for the process of dissolution of the zones by annealing at 100°C.

Introduction

Previous results of neutron small-angle scattering on Guinier–Preston zones in aged Al–Mg alloys (Roth & Raynal, 1974) concerned G.P. zones in alloys aged at 40°C and room temperature. The present work deals with alloys aged at 0°C. All the neutron small-angle scattering measurements were made on the spectrometer D11 at the Institut Laue-Langevin in Grenoble. In each case the small-angle scattering cross sections were evaluated from the scattered intensities by comparison with the incoherent scattering of a vanadium sample, measured under the same experimental conditions.

Neutron small-angle scattering by Guinier–Preston zones in polycrystalline Al–Mg alloys aged at 0°C

The polycrystalline Al–Mg samples (batches of 1 mm thick platelets) used were aged nine months at 0°C. The neutron small-angle scattering measurements were made at room temperature; the samples were heated a short time (a few hours) at room temperature before and for the measurements. The neutron wavelength distribution in the incident beam was quasi-triangular with a mean wavelength value of 7.4 Å and a half-height width of 48%. The measurements were made in the 0.07 to 0.25 Å⁻¹ scattering-vector range.

As in the first series of measurements (Roth & Raynal, 1974), we obtained the intensity scattered by G.P. zones alone, by subtracting from the intensity measured with the sample containing the zones the intensity scattered by the same sample after an anneal at 100°C long enough to dissolve the zones. For the present case, we used a 10 min anneal; indeed, after 10 min at 100°C, the scattering ring characteristic of scattering by GP zones completely disappeared.

Experimental results and interpretation

As was the case for the Al–7% Mg aged at 40°C or at room temperature (Roth & Raynal, 1974), no evidence was found for the existence of G.P. zones in this alloy after aging at 0°C (in particular, the scattered intensity does not vary after annealing at 100°C). The results obtained with an Al–11.5% Mg alloy are shown in Fig. 1. The curves b to d represent the small-angle scattering cross section of the G.P. zones in this alloy after 0, 1 and 3 min annealing at 100°C. We used the same method as in our previous paper (Roth & Raynal, 1974) to analyse quantitatively these results, i.e. we assumed that the G.P. zones constitute a system of identical, spherical particles correlated in position by a rather smooth step-like correlation function. A good fit with the experimental results is obtained (Fig. 2) with a zone radius of 19 Å and a mean distance between zones of 56 Å for the sample before annealing at 100°C. The corresponding fraction β of volume occupied by the zones is found to be about 31.5%. With this value of β and with the computed value of the integrated cross section, we find a difference of 11% in Mg concentrations between the zones and the matrix. Hence, taking into account the mean Mg concentration of the alloy, one finds for the matrix an Mg concentration of about 8% and for the zones, 19%.

The dissolution of the zones by annealing at 100°C

The evolution of some of these parameters by annealing at 100°C is shown in Fig. 3: the most striking aspect of the process of dissolution of the zones is the decrease of the Mg concentration in the zones. This result suggests the following model for the dissolution of the zones: by interdiffusion of the Al and Mg atoms, the sharp concentration gradient of the interface zones–matrix is smoothed out and each zone is thus transformed into a continuous local concentration fluctuation of the solid solution. The amplitude of these fluctuations decreases down to zero as a function of the aging time. Indeed, the decrease of the corresponding scattered intensity I(h,t) with time t can be computed very simply as a function of the initial intensity I(h,0), if one assumes that the diffusion constant of the atoms is unique and independent of the concentration. One
where $h$ is the scattering vector.

However no quantitative agreement is found by analysing the experimental data with the relation (1): $D$ appears to be a function of $h$ and $t$ which means in fact that $D$ is a function of the concentration. This is indeed not surprising. The concentration fluctuations resulting from the smoothing out of the zones are rather large in amplitude, except at the end of the dissolution, so that the assumption on $D$ is too crude. A better quantitative description of the phenomenon would be obtained with a diffusion equation very similar to those given for the theory of spinodal decomposition (Cahn, 1961; Tomozawa, MacCrone & Herman, 1970), with the variation of $D$ with concentration and the effect of the concentration gradient taken into account. But this would be sensible only with much more experimental data than we have at the moment. Nevertheless, one can remark a general qualitative agreement between the present experimental data and equation (1). In effect, equation (1) predicts qualitatively the apparent increase of the radius of the zones shown by the experimental results, and also the apparent decrease of the total number of zones (which results from the interpretation, in the frame of a model with discrete particles, of the shift with $t$ of the position of the peak of the curve $I(h,t)$ towards lower values of $h$). This model also predicts that the overall speed of dissolution of the zones is larger for small zones (since they produce small-angle scattering in relatively high ranges of $h$) than for large zones. It thus accounts for the fact that the dissolution of the zones by annealing at $100^\circ$C is faster in an alloy aged at $0^\circ$C than in an alloy aged at room temperature or at $40^\circ$C.

**Conclusion**

The results of neutron small-angle scattering in the Al-11.5\% Mg alloy aged at $0^\circ$C confirm the results obtained with the same alloy aged at room temperature and at $40^\circ$C: the Mg concentration in the zones amounts to 20–25\% and in the matrix to 8–10\%. These concentration determinations are not very accurate because they are not obtained directly, but by means of an approximate theoretical model. The radius and the mean distance between zones increase with increasing aging temperature; one finds radii of 19, 33 and 56 Å, and mean distances of 56, 130 and 200 Å for aging temperatures of 0, 25 and $40^\circ$C, respectively. The results show that the zones are formed during the aging either by a homogeneous nucleation and growth process, or more likely (because nucleation phenomena are generally heterogeneous) by an initial
spinodal decomposition. The increase of the mean size of the zones and of the mean distance between them with the aging temperature can be explained by both models. The dissolution of the zones by annealing at 100°C seems to proceed as a smoothing out of concentration fluctuations by atom interdiffusion in the solid solution, rather than as a shrinking of the zones by 'evaporation' of Mg in the matrix.

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References