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DILATOMETRIC MEASUREMENT ON Al—Mg—Si ALLOYS

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After a treatment of homogenisation Al—Mg—Si samples were quenched into water of room temperature and placed immediately into the measuring head of a dilatometer, which measured the isothermal change of length in 30—160 °C range. The observed relative change of length within 6 hours was found to be in the order of 10^{-5}.

1. Introduction

Al—Mg—Si is an alloy of wide industrial application, where special precipitation structures are produced in order to obtain favourable macroscopic properties. The density of the solid solution of Al—Si and Al—Mg differs but slightly from the density of pure aluminium [1]. The size effect is negligible in the Al—Mg_2Si quasi-binary alloys. The lattice parameter depends on the concentration in second order only (Fig. 1) [2]. If the compound Mg_2Si is precipitated in its own crystal structure (β phase), the change of volume is considerable, as the volume/atom ratio for Mg_2Si is much larger than the volume/atom ratio of aluminium.

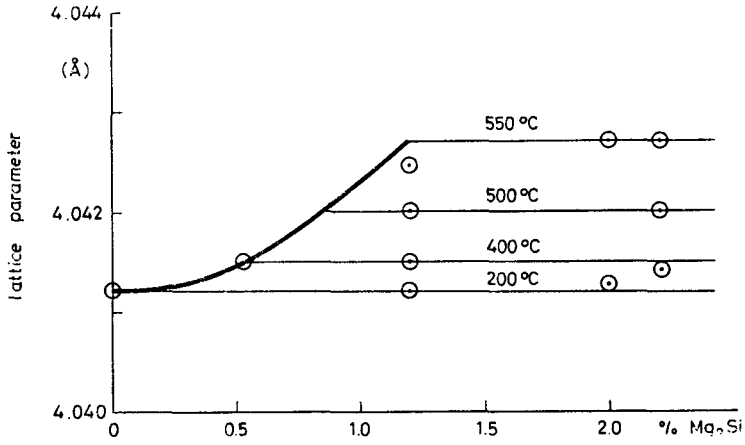

Fig. 1. X-ray measurements of the room temperature lattice parameter in Al—Mg_2Si alloys after KUZNETZOV and MAKAROV [2]. The circles show the actually measured lattice parameters on quenched sample, the full line is the solvus line

Our aim was to study the initial part of the precipitation process. From a strongly supersaturated solution the β phase does not precipitate directly, at the first stage zones do appear. This observation is due to THOMAS [3] who obtained it with transmission electron microscopy.

Quenching the Al—Mg—Si alloy to a temperature as low as 70 °C and keeping it there for one hour some weak lines appear at forbidden places in the diffraction pattern of a fcc lattice. This is the first information showing that the crystalline order has been disturbed and the process of segregation began.

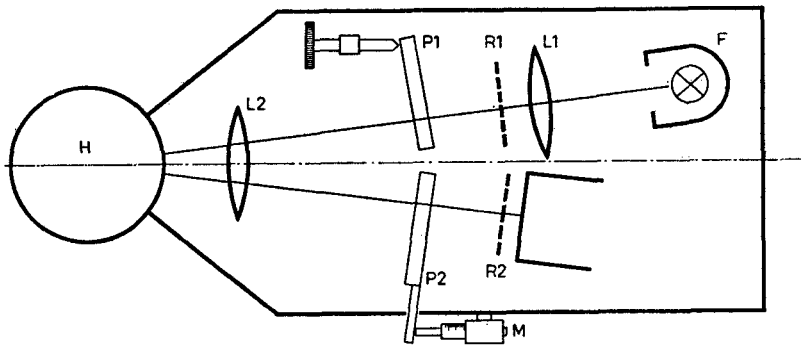

Fig. 2. Principle of the dilatometer

Increasing the temperature the incubation time before the appearance of the lines becomes shorter. According to THOMAS, zones of constant thickness form in the direction [100].

As the technique of electron microscopy is, however, of limited applicability, clear data are obtained only if the precipitates are sufficiently developed. Any rearrangement of atoms in a solid, however, may give rise to density changes. This measurement provides a single parameter only but the information is more direct since there is no developing process.

2. The dilatometer

The measurement was carried out using a dilatometer operating on the laws of geometrical optics. Fig. 2 shows the scheme of our dilatometer, working on the principle of the divided grid [4]. Lens L2 transmits the parallel beam from source F twice, projecting the image of grid R1 up to the divided grid R2 with the beam reflected on a mirror in the measuring unit of the dilatometer (H). Two samples, the one to be measured and the standard in the measuring unit are in contact with a fixed copper block at one of their ends, so when expanding differentially they rotate the mirror at their other ends. Thus the angle of rotation of the mirror is proportional to the difference of length between the two samples.

A differential photoresistor detects the difference of the light intensities passing through the two parts of the divided gird R2. This difference depends on the position of the mirror in the measuring unit. The plane parallel plate, P1, serves to adjust the beam. The deflection of the beam is compensated by plate P2 connected with a micrometer, M, and a motor. The purpose of compensation is that in the compensated state the two photosensitive elements should get the same amount of light in order to cancel the instabilities in the optical system.

The sensitivity of the dilatometer was $1.26 \cdot 10^{-6}$ rad which corresponds to $1.26 \cdot 10^{-6}$ relative change of length. The temperature of the head is controlled and adjustable in the temperature range from room temperature to 200 °C. The absolute thermal stability of the measuring head was better than 0.1 °C for short times and 0.5 °C for longer times, while the temperature difference between standard and sample is below 0.01 °C.

3. The measurement

We measured three different Al—Mg—Si alloys (in weight%):

1. Al—0.85% Mg_2Si, 0.28% Mg;
2. Al—1.42% Mg_2Si, 0.02% Si;
3. Al—1.75% Mg_2Si, 0.06% Si.

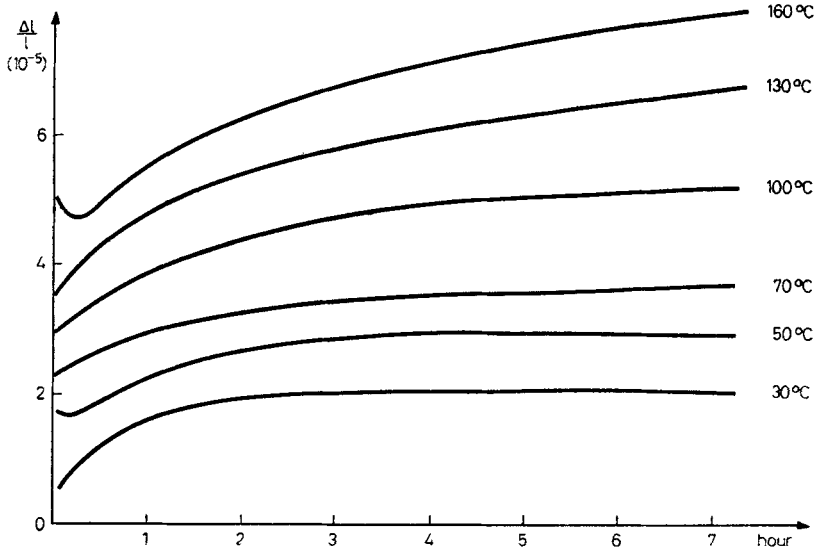

Fig. 3. Dilatometric curve of the Al-0.85% Mg_2Si-0.28% Mg

The samples of 11 mm length were cut out of wires of a diameter of 1.5 mm. The isothermal change of length compared with pure aluminium was measured. The samples were first annealed at 550 °C for 6 hours in order to remove any stresses due to casting and drawing. Before any measurement a solution treatment was performed at 550 °C for one hour and the sample immediately quenched into distilled water of room temperature.

It is known from the phase diagram [1] that after the solution treatment (550 °C) sample 1 is in a solid solution, sample 2 is just on the solvus line and sample 3 contains unsolved Mg_2Si.

As soon as possible, after quenching, the samples are placed into the dilatometer and the isothermal change of length measured at different constant temperatures from 30 °C to 160 °C. Fig. 3 shows our experimental result for the alloy 3. The other alloys show similar sets of curves but the changes of length are smaller. The data, obtained in the first 20 minutes, are not reliable because the insertion of the samples disturbed the thermal equilibrium of the measuring head.

4. Discussion

Is is seem that there is a minimum dilatation for anneals about 50—70 °C. This characteristic temperature appears in other studies too [5]. OZAWA and KIMURA [6] investigated Al—Si alloys using electron microscopy and found that the precipitation size changed at this temperature. We can conclude that probably Si is responsible for the anomaly at 50—70 °C.

At higher ageing temperatures the alloy is still supersaturated. Here the diffusion is faster leading to a greater dilatation rate.

The volume change may be interpreted either as due to phase separation or as a concentration inhomogeneity. One cannot state with certainty that Cahn's mechanism of spinodal decomposition is in fact the best description of zone formation, but the similarities are evident, the zone being a region of increased impurity concentration. In that case the change of volume is proportional to the mean square of the concentration deviation [7].

$$\frac{V - V_0}{V_0} = \left\{\left[\frac{1}{2}\left(\frac{\partial k}{\partial p} - 1\right) + \frac{2}{3}\,\frac{\mu}{k}\left(\frac{k}{\mu}\,\frac{\partial \mu}{\partial p} - 1\right)\right]\left(\frac{3k\eta_1}{3k + 4\mu}\right)^2 + \right.$$
$$\left. + \frac{k\eta_1^2}{3k + 4\mu} + \eta_2\right\} \frac{1}{V_0}\int (c(r) - c_0)^2 \, \mathrm{d}^3R \,. \tag{1}$$

Here k and μ are the bulk and the shear moduli, $\frac{\partial k}{\partial p}$ and $\frac{\partial \mu}{\partial p}$ are the quantities characterizing the pressure dependence of the appropriate elastic moduli i. e.

the second order elastic moduli. η_1 and η_2 are the parameters of the parabolic fit for the solvus line on Fig. 1.

The definition of η_1 resp. η_2 is:

$$\left(\frac{a(c)}{a_0}\right)^3 = 1 + \eta_1(c - c_0) + \eta_2(c - c_0)^2, \tag{2}$$

where $\eta_1 = 0.12$, $\eta_2 = 6.6$ at $c_0 = 0.85\%$.

The first and second terms in the formula (1) are small for Al—Mg_2Si solutions [2], the third one, η_2 is the dominating one.

Using the value of η_2, our numerical expression is:

$$\frac{V - V_0}{V_0} = 6.6 \frac{1}{V_0} \int (c - c_0)^2 \, \mathrm{d}^3R \, .$$

In our measurements a characteristic value for the relative change of length is 10^{-5}. This corresponds to a change of volume of $3 \cdot 10^{-5}$ as the process is isotropic. Using the above quantity, the root mean square of the concentration deviation is found to be

$$\left(\frac{1}{V_0} \int (c - c_0)^2 \, \mathrm{d}^3R\right)^{\frac{1}{2}} = 0.21\% \, .$$

It shows that in our alloys, containing 0.85% and 1.42% Mg_2Si, respectively, the fluctuation of concentration during low temperature annealing is an essential phenomenon.

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