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DECOMPOSITION IN BINARY Al-Li ALLOYS

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Abstract:

We have investigated the influence of aging on the morphology and phase composition of annealed Al-Li binary alloys by means of an atom probe with a multi-detection system. The influence of artifacts such as the preferential evaporation of the Li-atoms and the pile-up effects for the Al-atoms were analysed as well.

Specimens of Al - 2.0 wt% Li and Al - 2.9 wt% Li were solution treated and aged at 190°C and 150°C for several times. δ'-precipitates were observed by FIM and their composition as well as the Li-content of the matrix were obtained by performing AP analysis for the different heat treatments.

I. INTRODUCTION

Lithium is the lightest element which has a solubility of more than 1 at% in Aluminium and a great influence on the E-modulus and the density of Al in alloying. Not only improving the E-modulus but also precipitation of the δ'-phase in the Al-Li alloys leads to the particular hardness of these alloys. This makes such alloys of a great interest in the industry and was therefore the reason of intense research/1/. In many overviews the properties of the Al-Li alloys are presented/2/.

Although these alloys were known since 1924/3/ and named as scleron-alloys still the composition of the δ'-precipitate is an open question because of many difficulties in determining the Li concentration by most of the analytical methods. Only a few AP-investigations about these alloys have been reported/4/, /5/. In a previous paper/6/, we have shown the ability of the AP to perform best analysis of these alloys. Using a new detection system, we have tried to analyse and to assure the Li-concentration of the δ'-precipitates.

II. EXPERIMENTAL

Al- 2.0 wt%(7.35 at%) Li was supplied by PECHINEY, and an Al- 2.9 wt%(10.4 at%) Li alloy by VAW( Vereinigte Aluminum Werke, Bonn). The ingots were drawn to 0.2 mm wires, which were
solution treated at 500°C and 550°C respectively, and iced-water quenched. These wires were further aged at 190°C and 150°C for different times.

FIM tips were prepared using an electrolyte of methanol and nitric-acid in a proportion of 2:1 at 5-7 Volts DC and a temperature of −40°C. A circulation system was used as in /7/ to cool the electrolyte. The atom probe used in this study was described elsewhere/8/, with a multidetection system mentioned in /9/. The detector is divided into four independent sectors with equal areas leading to a four times faster analysis with same systematic error rate. This detection system has the advantage of discriminating the ions having the same time of flight, which helps to reduce the influence of the pile-up effects on our measurements to about 0.3 at% to 0.5 at% if applying the multidetection system.

III. RESULTS AND DISCUSSION

Fig.1 shows an FIM-image of an Al-2.0 wt% Li tip, aged for 4h at 190°C using Neon as imaging gas of 5.10−5 mbar pressure and a tip temperature of 20 K. Even at this temperature δ' precipitate (dark regions) image darkly due to the different height of the evaporation field in both phases. A plane by plane analysis was performed to scale the AP. All measurements in this study were undertaken with a DC high voltage between 5.0 and 7.5 kV. In this range the (111)-pole was calibrated. Fig.2 shows e.g. such a calibration of the (111)-pole in which the AP aperture was directly located on it. As described by Miller/10/, it is known that local magnifications and trajectory aberrations have an influence on the size estimation in these phases imaging with different contrast. The change of the number of atoms per layer in- and outside of the decomposed region must be taken into account. Taking a mean number of atoms per layer for both regions, it is clear that this calibration gives only an approximation of the size of the darkly imaged phase. Nevertheless the influence of magnification in our measurements was only of importance for large precipitates.

The best conditions to perform AP analysis were achieved in our previous publication/6/, such as a tip temperature between 20K and 30 K, a high vacuum of < 10−10 mbar, an average ion rate between 0.02 and 0.03 ion per pulse and a pulse ratio of 0.2.

With these investigation conditions concentration profiles were obtained from the different specimens. In fig. 3(a−d) some of them are shown. Fig. 3-a is a concentration profile of an Al-2.0 wt% Li tip aged for 3h at 190°C showing δ'-precipitates with diameter between 120−180 Å. Increasing the aging time slightly, the diameter of the precipitates increases after aging for 4h at this temperature (fig. 3-b). If the supersaturation is changed, e.g. by changing the nominal concentration of the tips from 2.0 wt% to 2.9 wt% Li, specimens aged for 4h at 190°C show an increasing number density of precipitates, and the diameter of the precipitates increases slightly in fig. 3-c. After an annealing time of 24h at 190°C the diameter of the precipitates reaches already sizes over 500 Å (fig. 3-d) for the Al-2.9 wt%Li specimens. Table 1 summarizes the results obtained for these aging conditions.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Aging condition</th>
<th>$D(\text{Å})$</th>
<th>$C_p(\text{at%})$</th>
<th>$C_m(\text{at%})$</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0 wt%Li</td>
<td>3h/190°C</td>
<td>120−180</td>
<td>21.5 ±1.6</td>
<td>5.8 ±0.4</td>
</tr>
<tr>
<td>2.0 wt%Li</td>
<td>4h/190°C</td>
<td>200−250</td>
<td>19.5 ±2.1</td>
<td>5.7 ±0.4</td>
</tr>
<tr>
<td>2.9 wt%Li</td>
<td>4h/190°C</td>
<td>200−280</td>
<td>20.5 ±2.1</td>
<td>6.0 ±0.5</td>
</tr>
<tr>
<td>2.9 wt%Li</td>
<td>24h/190°C</td>
<td>&gt;500</td>
<td>19.7 ±2.0</td>
<td>5.7 ±0.3</td>
</tr>
<tr>
<td>2.9 wt%Li</td>
<td>10h/150°C</td>
<td>100</td>
<td>21.6 ±2.2</td>
<td>6.0 ±0.4</td>
</tr>
</tbody>
</table>

Table 1 The results as obtained with the AP for the different aging conditions. $C_m =$ Li−concentration of the matrix and $C_p =$ Li−concentration of the precipitates.
The results obtained by the AP point out that the Li concentration of the $\gamma'$-precipitates must lie between 20 at% and 22 at% at these aging temperatures taking the error bars into account. If no further loss of Lithium is assumed, the concentration of the $\gamma'$-precipitate we measured is in a good agreement with the phase diagram calculated by Gayle/12/. The matrix concentration reaches a level of $C_m = 6$ at%Li, again nearly the same concentration assumed by Gayle.

In the case of Al-2.0 wt%Li aged for 4h at 190$^\circ$C a precise size measurement of the diameter of the precipitates was done, by counting the evaporated (111)-planes. The mean diameter was found to be $D \approx 150 \, \AA$, whereas AP- estimation gives a diameter between 200 $\AA$ and 250 $\AA$. This show that the size of the precipitates is overestimated in our measurements, due to the local magnification effects mentioned above.

In a previous paper /6/, Al-3.5 wt%Li-3.6 wt%Mg was studied. After aging for 24h at 190$^\circ$C, $\gamma'$-precipitates were observed with a concentration of $C_p = 17.5 \pm 0.5$ at%Li and $C_m = 4.9 \pm 0.3$ at%Mg. The concentration of the matrix was measured to be $C_m = 6.3 \pm 0.3$ at%Li. There we made the assumption that the Magnesium atoms may occupy the Li sites in the L1$_2$ ordered structure to complete the stoichiometry. Our new results point out that the Li concentration of the $\gamma'$-precipitate in the Al-2.9 wt%Li alloy aged for 24h at 190$^\circ$C, which has a similar supersaturation, is higher than the one measured for the ternary system. This is consistent with our assumption that the addition of Mg in the Al-Li system decreases the concentration of Li in the $\gamma'$-precipitates.

Furthermore a tip aged for 10h at 150$^\circ$C shown in fig.4-a including many precipitates cut by the analysis cylinder. The maximum plateau of the concentration is again nearly 22 at%. From the autocorrelation profile (fig.4-b) the mean diameter of the precipitates was estimated for this aging condition. In addition the number density of the precipitates was calculated using a statistical model, which was proposed by Blavette et al./11/ leading to $N_v = 5 \times 10^{23}$ (m$^{-3}$) and a volume fraction of $f_v \approx 26\%$. Calculating the volume fraction from the measured matrix and precipitate concentrations gives $f_v \approx 28\%$, which agrees well with the results obtained from the model. This aging temperature indicates that the diameter and the number density of precipitates could be sufficient to study the kinetics in the Al-Li system.

**IV. CONCLUSION**

Specimen of Al-Li alloys were investigated by means of the atom probe. It is clearly shown that aging at 190$^\circ$C and 150$^\circ$C leads to the $\gamma'$-precipitation. The Li concentration in these enriched regions is found to be $C_p \approx 22$ at%, which is nearly the same concentration as Gayle et al. have assumed and calculated in their phase diagram. A matrix concentration of $C_m \approx 6.0$ at% was determined at these aging conditions. Preliminary observations of the time evolution of the diameter and the number density of precipitates were achieved. The specimen aged at 150$^\circ$C for 10h had a sufficient number density of precipitates that may allow an investigation of the kinetics in these alloys. Further investigations are required to optimize the parameters needed to follow the time evolution of the precipitates, especially a number density of precipitates $> 10^{23}$ (m$^{-3}$).

**V. ACKNOWLEDGEMENT**

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VI. REFERENCES

Fig. 1 FIM-image of an Al-2.0 wt%Li tip aged for 4h at 190°C at 12.9 kV using Neon as imaging gas, P = 5.10^-5 mbar, tip temp. = 20K. S- precipitates are darkly imaged.

Fig. 2 Plane by plane analysis of the (111)-pole to calibrate the AP.
Fig. 3 Concentration profiles as measured with the AP:

- a) Al- 2.0 wt\%Li aged for 3h at 190\(^\circ\)C.
- b) Al- 2.0 wt\%Li aged for 4h at 190\(^\circ\)C.
- c) Al- 2.9 wt\%Li aged for 4h at 190\(^\circ\)C.
- d) Al- 2.9 wt\%Li aged for 24h at 190\(^\circ\)C.
Fig. 4 - a) Concentration profile of an Al-2.9 wt%Li aged for 10h at 150°C showing more than one precipitate cut by the analysis cylinder.
- b) Autocorrelation of this profile.