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MICROSTRUCTURE OF RAPIDLY SOLIDIFIED Al-Li-Ti ALLOYS

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ABSTRACT

The annealing of the melt-spun ribbons of Al-Li-Ti alloys at 200° C reveals the cellular microstructure. Due to a partition coefficient much larger than unity and a low diffusion coefficient, the titanium atoms remain essentially inside the cells and they repel the precipitation of the $\delta'$-Al$_3$Li metastable compound in the intercellular space.

INTRODUCTION

The limited ductility and toughness of binary Al-Li alloys hardened by $\delta'$-Al$_3$Li particles is due on the one hand to the shear of the coherent metastable $\delta'$ precipitates by the dislocations and on the other hand to the occurrence of precipitate free zones along the grain boundaries (1-2). In order to reduce these limitations, a method consists in the introduction of dispersoids in the alloy. These share out the deformation on several slip systems and they harden the precipitate free zones. Furthermore, they must be thermally stable in order to avoid their coarsening during the thermal treatment which produces the precipitation of the $\delta'$ particles. The intermetallic compounds of aluminium with transition metals fulfil this requirement: the solubility of transition metals in aluminium is low, their diffusion is slow and the energy of the interface between the matrix and the precipitates is low. In order to obtain a sensible volume fraction of dispersoid, a rather high concentration of the transition metal must be used.

In this case, the classical metallurgy leads to large precipitates of the intermetallic compound and so a rapid solidification technique has to be employed. Besides the adding should not increase the density, which would be against the effect expected from the adding of the lithium.

The present work concerns the Al-Li-Ti alloys. Some results are reported in the literature about the microstructure and the mechanical properties of these alloys roller-quenched in flakes (3-4). This paper reports the microstructure of the melt-spun ribbons as-quenched and annealed at 200° C.

EXPERIMENTAL

Four alloys were prepared from aluminium-lithium and aluminium-titanium mother alloys and pure aluminium. Their composition in wt% (at%) are reported in the table:

<table>
<thead>
<tr>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
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<tbody>
<tr>
<td>Li</td>
<td>1.88 (6.92)</td>
<td>2.33 (8.48)</td>
<td>2.26 (8.29)</td>
</tr>
<tr>
<td>Ti</td>
<td>0.35 (0.18)</td>
<td>0.20 (0.10)</td>
<td>0.15 (0.08)</td>
</tr>
<tr>
<td>Al</td>
<td>BAL</td>
<td>BAL</td>
<td>BAL</td>
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The alloy is molten in a fused silica tube coated with boron nitride at a temperature 850-900°C. Then it is thrown out upon a beryllium-copper wheel, 0.28 m diameter. Its rotation speed corresponds to a linear speed equal to 44 m s⁻¹ at the periphery of the wheel. The quench of the liquid alloy occurs in helium atmosphere under a pressure equal to 0.04 hPa.

Annealing of the quenched ribbons is carried out at 200° C in an argon atmosphere for a period of time varying from 0.5 to 1000 hours. The microstructure of the as-quenched and annealed ribbons is characterized by optical metallography, X-ray diffraction and transmission electron microscopy.

RESULTS

The thickness of the melt-spun ribbons is equal to 20-40 microns. As shown on the optical micrograph of the figure 1, the solidification started by columnar formation on the wheel side and extended throughout the entire thickness. The same type of microstructure was observed in melt-spun Al-Li-X alloy ribbons (5-6).

X-ray diffraction shows that the as-quenched ribbon contents only one phase, the supersaturated lithium and titanium solid solution in α-aluminium. The annealed ribbon at 200° C for every duration, from 0.5 to 1000 hours, contents the matrix α and precipitates $\delta'$-Al₃Li. The phase $\delta'$-Al₃Li was observed only in some cases for a 1000 hours annealing. The phase $\delta$-Al₃Ti was never observed. This last observation agrees with the results of Hori et al (7) on Al-Ti alloys rapidly solidified: for a cooling rate higher than $10^4$°Cs⁻¹, the metastable Al₃Ti phase precipitates only for a concentration in titanium higher than 0.8wt%.

Transmission electron microscopy yields information on the microstructure refinement resulting from the high cooling rate associated with the melt-spinning technique. This is obvious in the very fine grains (1.5 to 3.5 microns) as displayed in figure 2, a cross section normal to a set of columnar grains, nearly at the middle of the ribbon. In few cases, some very fine $\delta'$ precipitates were detected in dark field observation.

In the first stage of annealing (0.5 H for the alloy A and up to 12 H for the alloys B, C and D) at 200°C, very fine $\delta'$ particles precipitate with an uniform distribution throughout the grains. Figure 3 reports the case of alloy B annealed for 1 hour.

In the second stage of annealing, the number and the size of the $\delta'$ particles increase and their distribution becomes less uniform. The $\delta'$ precipitation reveals a cellular microstructure inside the grains: the $\delta'$ precipitation is more abundant in the cell walls than in cell interior and the configuration of cell appears distinctly. The diameter of the cell varies from 200 to 500 nanometers. Figure 4 shows transverse sections of cells in alloy annealed for 1 hour. Figure 5 represents longitudinal sections of cells in alloy B annealed for 24 hours. The TEM micrographs of the figure 6 are associated with alloy C annealed for 48 hours. For a longer annealing duration the size of $\delta'$ particles inside the cells decreases and that of the particles in the cell walls increases. In some cases the first particles even disappear and the interior of the cells becomes free of $\delta'$ precipitates. In return, the second particles coarse and this leads to large precipitates. Micrographs of the figure 7 show the case of alloy A annealed for 500 H.

Special microstructures are observed in some grains. Figure 8 shows a gradient of particle size around precipitate free areas. Curved and elongated precipitate free zones are observed in some grains as seen in figure 9. Figure 10 shows the case of irregular cell shape and size with a wide intercellular space.

DISCUSSION

The cellular structure is associated to the solidification process: it started from the wheel side. If the partition coefficient of the solute atom is lower than unity, an enhancement of the solute concentration is expected in the intercellular space and an impoverishment in the cell interior. If the partition coefficient is higher than unity, the distribution of solute atoms is inverted. This effect is more marked, the value of the partition coefficient is more different from unity.
The theoretical value of the partition coefficient of lithium in aluminium is equal to 0.45 and its experimental value to 0.73 (8). The theoretical value of the partition coefficient of titanium in aluminium is equal to 8-13 (8). So during the solidification the cell interior enriches with titanium and impoverishes with lithium while the intercellular space enriches with lithium and impoverishes with titanium. The effect is more emphasized for titanium than for lithium.

This difference in behaviour is enhanced by the diffusion phenomenon. The values of the pre-exponential factor $D_0$ and of the activation energy $Q_0$ for diffusion in aluminium are reported in the table (8):

<table>
<thead>
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<th>$D_0$ (cm² s⁻¹)</th>
<th>$Q_0$ (ev/at)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Li</td>
<td>4.5 - 5.5</td>
<td>1.38 - 1.45</td>
</tr>
<tr>
<td>Ti</td>
<td>5*10⁻⁷</td>
<td>1.12</td>
</tr>
</tbody>
</table>

The lithium atoms move more rapidly throughout the aluminium crystal lattice and the diffusion reduces the difference in the lithium composition inside the cells and in the intercellular space. The titanium atoms move only very little in the aluminium lattice and the as-quenched distribution of titanium atoms is not changed during the annealing at 200°C.

The maximum solid solubility of lithium in aluminium is equal to 13.8±1.2 at% at 600°C (9). The rapid solidification extends the solid solubility of titanium in aluminium up to 2 at% (10). This extension produces in the as-quenched ribbon a supersaturated solid solution and the cellular structure is not revealed.

During the first stage of the annealing the $α'_1$-Al₃Li metastable phase precipitates but the size and the number of these particles are very small and the non-uniformity of their distribution does not appear clearly. During the second stage the difference in the behaviour of the cell interior and the intercellular space becomes more obvious. This difference is related to the titanium concentration: it could be expected that the titanium atoms hinder the precipitation of the metastable phase $α'_1$-Al₃Li inside the cells.

CONCLUSIONS

The addition of titanium to the binary aluminium-lithium alloy changes deeply the microstructure of the melt-spun alloy. Due to a large partition coefficient and a low diffusion coefficient, the titanium atoms remain essentially inside the cells produced during the solidification process. During the annealing at 200°C, the titanium atoms hinder the precipitation of the metastable $α'_1$-Al₃Li phase and the precipitated particles appear chiefly in the intercellular space.

REFERENCES

Fig. 1: As quenched ribbon. Alloy C. Optical micrograph of a longitudinal section.

Fig. 2: As quenched ribbon. Alloy C. TEM micrograph.

Fig. 3: Annealed ribbon for 1 H. Alloy B. TEM micrograph. Uniform distribution of $S'$ precipitates. (a) Bright field. (b) Dark field.
Fig. 4: Annealed ribbon for 18 h. Alloy A. TEM micrograph. Dark field. Cell structure with a higher density of precipitates in the walls.

Fig. 5: Annealed ribbon for 24 h. Alloy B. TEM micrograph. Elongated cellular structure. (a) Bright field. (b) Dark field.

Fig. 6: Annealed ribbon for 48 h. Alloy C. TEM micrograph. Cell structure. (a) Bright field. (b) Dark field.
Fig. 7: Annealed ribbon for 500 H. Alloy A. TEM micrograph. Cellular structure with coarsened S' precipitates in the walls. (a) Bright field. (b) Dark field.

Fig. 8: Annealed ribbon for 6 H. Alloy D. TEM micrograph. Gradient of precipitate size around depleted zone. (a) Bright field. (b) Dark field.
Fig. 9: Annealed ribbon for 96 H. Alloy C. TEM micrograph. Curved precipitate free zone inside the grain.
(a) Bright field
(b) Dark field

Fig. 10: Annealed ribbon for 12 H. Alloy A. TEM micrograph. Wide intercellular space. Dark field.