Residual stresses induced by LiH precipitation in an Al–Li alloy

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Abstract

A study by X-ray diffraction of the residual stresses induced by LiH precipitation in the aluminium matrix of an Al–2.3wt.%Li–1.2wt.%Cu–0.09wt.%Zr alloy shows (a) the induction of a compression macrostress of 60 MPa parallel to the rolling direction and (b) a change in density of the (422) planes, indicated by the fact that the density increases in the rolling direction and decreases in the perpendicular one.

1. Introduction

The characterization of possible residual stresses induced by precipitation of a dispersed hydride as the result of hydrogen reaction with lithium contained in an Al–2.3wt.%Li alloy is of double interest.

(a) It improves our knowledge about the mechanical properties of this promising material, whose behaviour in the presence of hydrogen [1] is not well known.

(b) It appears to be a good model for testing the hypothesis put forth [1] to explain the change in reactivity of hydride-forming compounds by the stress field which could result from hydride precipitation.

For such a purpose Al–Li alloys are ideal since LiH is formed after hydrogen solid diffusion [2] by disproportionation of the Al–Li solid solution. The aluminium matrix does not give any hydride in such conditions.

2. Apparatus and samples

The possible stress field developed by LiH precipitation is determined by X-ray diffraction. The method is based on the changes in position and shape of the X-ray lines of given crystallographic planes of the aluminium matrix after reaction [2, 3].

The device consists of a θ–θ goniometer equipped with a copper anticathode X-ray source. The aluminium $hkl = (422)$ planes, which give (with $\lambda = 1.54$ Å) $2\theta = 137.47°$, are explored step by step. At the same time the sample, supported by a goniometric holder, can rotate on the goniometer axis by steps of 10° from −45° to 45° to give the family of ψ angles.

Standardization is carried out on a molybdenum powder which is assumed to be free of residual stresses.

The samples consist of 1 cm^2 plates, 0.5 mm thick of an Al–2.3wt.%Li–1.2wt.%Cu–0.09wt.%Zr alloy cut from an industrial laminated rod annealed for 12 h at 135 °C. They are mechanically polished. Two directions are marked, *i.e.* parallel ($\Phi=0$) and perpendicular ($\Phi=\pi/2$) to the rolling direction.

As far as the evidence of residual stresses which result from hydridation is concerned, it is necessary to distinguish the possible effects of sample preparation and hydriding conditions from those of the actual precipitation, *i.e.* heated to 450 °C for 3 h and cooled down in 3 h, after a preliminary soaked in distilled water for 20 h [4]. For this, three samples have been prepared with the following procedure: one is polished and water pretreated; the second is polished, water pretreated and heated for 3 h at 450 °C under a vacuum of 2×10^{-4} Pa; the third is polished, water pretreated and placed under the reaction conditions defined above under an H_2 pressure of 3 MPa.

3. Results

Three quantities expressed as a function of sin^2 ψ are extracted from the experimental results obtained on a given X-ray line; then from each one a physical parameter is obtained. Respectively these functions are:

(1) the width of the X-ray line, measured by the chord at $\frac{2}{5}$ *vs.* sin^2 ψ — this gives the level of microstresses;

(2) the 2θ angular position of the X-ray line *vs.* sin^2 ψ — the slope of this curve represents the level of the macroscopic strain and stress;

(3) the integrated intensity of the X-ray line, measured by its surface after correction by a Lorentz factor and an absorption factor, *vs.* sin^2 ψ — this gives information on possible crystallographic textures.

3.1. Microstresses

Table 1 compares the chord at $\frac{2}{5}$ for molybdenum powder, polished Al–Li–Cu–Zr and polished and annealed Al–Li–Cu–Zr samples. From these results it can be noted that the microstresses introduced by mechanical polishing are released by annealing; there is no change after hydriding.

TABLE 1

Microstresses deduced from the function chord at $\frac{2}{5} = f(\sin^2\psi)$

	Mo	Al–Li–Cu–Zr (polished)	Al–Li–Cu–Zr (annealed, 3 h, 450 °C)	
Chord at $\frac{2}{5}$	0.83	1.19	0.94	($\Phi=0$)
(arbitrary units)		1.20	0.945	($\Phi=\pi/2$)

3.2. Macrostresses

Table 2 compares the samples annealed under vacuum and hydrided. Comparison between the reference, the annealed sample and the hydrided sample shows the following.

(i) A tensile macrostress of 26 MPa is replaced after hydration by a compression macrostress of -36 MPa, i.e. an absolute macrostress of 62 MPa.

(ii) The hydridation creates a notable macrostress in the aluminium lattice if the above datum is compared with the value of 200 MPa which is developed by cold working with microballs.

(iii) This result is obtained in the rolling direction ($\Phi = 0$). In contrast, in the perpendicular direction there is only a small increase in compression stress of 15 MPa. Undoubtedly there is a preferential precipitation of the LiH hydride in the rolling direction.

3.3. Change in the 422 line density

Table 3 sums up the results obtained from the integrated intensity vs. $\sin^2 \psi$. An increase clearly appears in the (422) plane density in relation to LiH precipitation in the rolling direction ($\Phi = 0$) and a decrease in the perpendicular direction.

4. Discussion

From the results of Tables 2 and 3 which compare the reference sample annealed with an identical sample hydrided, i.e. annealed under an H_2 pressure which allows hydride precipitation, a clear relationship appears between the hydride precipitation and some properties resulting in rolling. The main difference between $\Phi = 0$ and $\Phi = \pi/2$ is the existence of a tensile macrostress

TABLE 2

Macrostresses deduced from the function $2\theta = f(\sin^2 \psi)$

	Mo	Al–Li–Cu–Zr (annealed)	Al–Li–Cu–Zr (hydrided)	
Macrostress (MPa)	-2.99	26 -15	-36 -30	($\Phi = 0$) ($\Phi = \pi/2$)

TABLE 3

Change in the integrated intensity vs. $\sin^2 \psi$

	Mo	Al–Li–Cu–Zr (annealed)	Al–Li–Cu–Zr (hydrided)	
I_{max}/I_{min} (arbitrary units)	1.36	6.2 10.5	12.7 7.7	($\Phi = 0$) ($\Phi = \pi/2$)

of 26 MPa in the rolling direction and a compression macrostress of -15 MPa in the perpendicular direction. In contrast, after hydridation the result is almost identical in the two directions, *i.e.* a compression macrostress of about -30 MPa. It can be suggested that the residual tensile stress developed by rolling favours LiH nucleation by making hydrogen migration towards an octahedral interstitial site easier (according to ref. 4). The growth of LiH domains eventually produces compression stresses of the same level in the two directions.

This preference for LiH to precipitate in regions under stress is confirmed by Fig. 1, which represents a cross-section of a chip. In this photograph the LiH domains are arranged in rows. These rows do not delineate grain boundaries (as a difference with ref. 4) but more probably decorate a stress field: one can note that each row starts at the end of cracks which have been generated when the chip has been torn off. In addition, it can be noted that the cubic parameters of LiH (4.08 Å) and aluminium (4.04 Å) are so close that conditions are fulfilled to observe some coherence between the precipitate and the matrix. This observation is probably at the origin of the
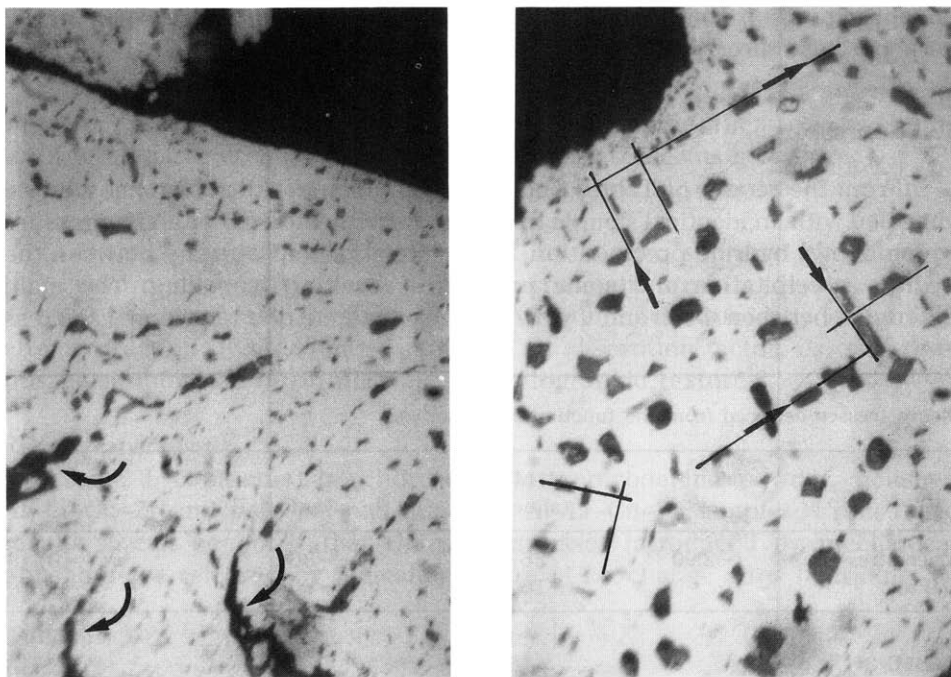

Fig. 1. Cross-section of a hydrided Al–Li–Cu–Zr chip. One can note the rows of LiH particles which decorate a stress field. The rows start on cracks, indicated by arrows, resulting from the filing (optical magnification = 800).

Fig. 2. Cross-section of a hydrided Al–Li–Cu–Zr chip. The well-defined LiH particles are in lines on two main perpendicular directions, independently of grain boundaries; this probably agrees with a coherence between the identical cubic lattices of LiH and aluminium (optical magnification = 800).

increase in density of (422) planes in the rolling direction as a consequence of the preferential precipitation of LiH.

This LiH epitaxial precipitation reinforces the intensity diffracted by the aluminium matrix in the rolling direction. Here again, the microscopic observations of Fig. 2 confirm the X-ray results in showing that LiH precipitates are in lines on two perpendicular directions, independently of the grain boundaries, as a consequence of epitaxial growth on the cubic aluminium matrix.

5. Conclusions

X-ray diffraction techniques show evidence of a residual stress field in relation to LiH precipitation in the aluminium matrix, even though the reaction temperature was 450 °C. The experimental results agree with what can be predicted, *i.e.* coherence between the matrix and the precipitate as a consequence of their almost identical crystallographic parameters and the tendency of LiH to nucleate in regions under tensile stress.

References

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