The mechanism of continuous dissolution of the lamellar structure in aged Al 22 at % Zn alloy

JAN DUTKIEWICZ, ANDRZEJ PAWŁOWSKI, PAWEŁ ZIĘBA
Institute for Metal Research, Polish Academy of Sciences 30-059 Kraków, ul. Reymonta 25, Poland

The mechanism of continuous dissolution was studied using transmission electron microscopy, X-ray diffraction and hardness measurements. The metastable α' phase was identified in the first stage of dissolution nucleating at the α–η boundaries. Interface dislocations at the α–α' boundaries forming a perpendicular net in the $\langle 1\,1\,0 \rangle$ directions were identified as of screw character and of Burgers vector $\boldsymbol{b} = \frac{1}{2}a \langle 1\,1\,0 \rangle$. During dissolution a change of shape of α' precipitates occurs leading to their fragmentation and to a change of the direction of boundaries into $\langle 1\,1\,0 \rangle$ directions.

1. Introduction

Dissolution of ageing products has recently been investigated in several works [1–6], most of which concentrated on the cellular dissolution occurring by receding of cell fronts and controlled by a diffusion at a cell boundary. However, at higher temperatures and at smaller interlamellar spacings a different mechanism of dissolution can operate [2]. It is characterized by a dissolution of individual lamellae within cells without movement of a boundary. This type of phase transformation known as continuous dissolution is controlled by a volume diffusion.

Continuous dissolution was reported by Solorzano *et al.* [4] in aluminium alloys containing 22 and 28 at % Zn and by Zięba and Pawłowski [5] in an Al 15 at % Zn alloy. In all the above papers characteristic dislocation contrast on transition phases was observed. Such a contrast cannot occur in aged aluminium–zinc alloys due to the much smaller size of metastable phases. However, a significant similarity exists to the contrast from precipitates presented by Bourchard *et al.* [7] after spinodal decomposition in Cu–Ni–Fe alloys.

The aim of the present paper is to analyse the character of interphase dislocations using transmission electron microscopy experiments and to identify the transition phases in order to describe the mechanism of continuous dissolution of the lamellar structure in aluminium–zinc alloys.

2. Experimental procedure

An alloy containing 22 at % Zn was cast from elements of purity 99.99% (Zn) and 99.7% (Al). After casting, the alloy was homogenized, then cold rolled down to 0.10 mm for transmission electron microscopy (TEM) preparation. Structure investigations were performed using Philips EM 301 operating at 100 kV and an IRYS 2 X-ray diffractometer. Sample preparation was described in the previous paper [5]. After being quenched the alloy was aged at 373 and 448 K for a period desired to obtain 60–100% zinc rich η precipitates formed in a discontinuous mode. Dissolution was performed in a salt bath furnace at 603 K or 623 K for a short period of time to obtain a continuous operating mechanism.

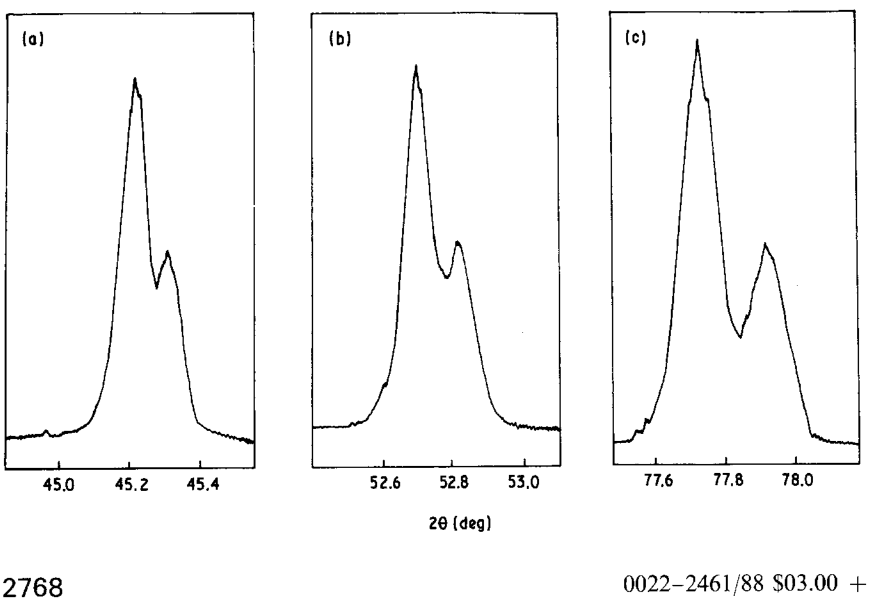

Figure 1 A fragment of the X-ray diffraction curve showing splitting of (a) {1 1 1} $\alpha = 45.21°$, $\alpha' = 45.31°$, (b) {2 0 0} $\alpha = 52.70°$, $\alpha' = 52.81°$ and (c) {2 2 0} $\alpha = 77.73°$, $\alpha' = 77.92°$ reflections in Al 22 at % Zn annealed at 603 K for 60 sec after ageing at 448 K leading to the formation of 60% η phase according to a discontinuous mode.

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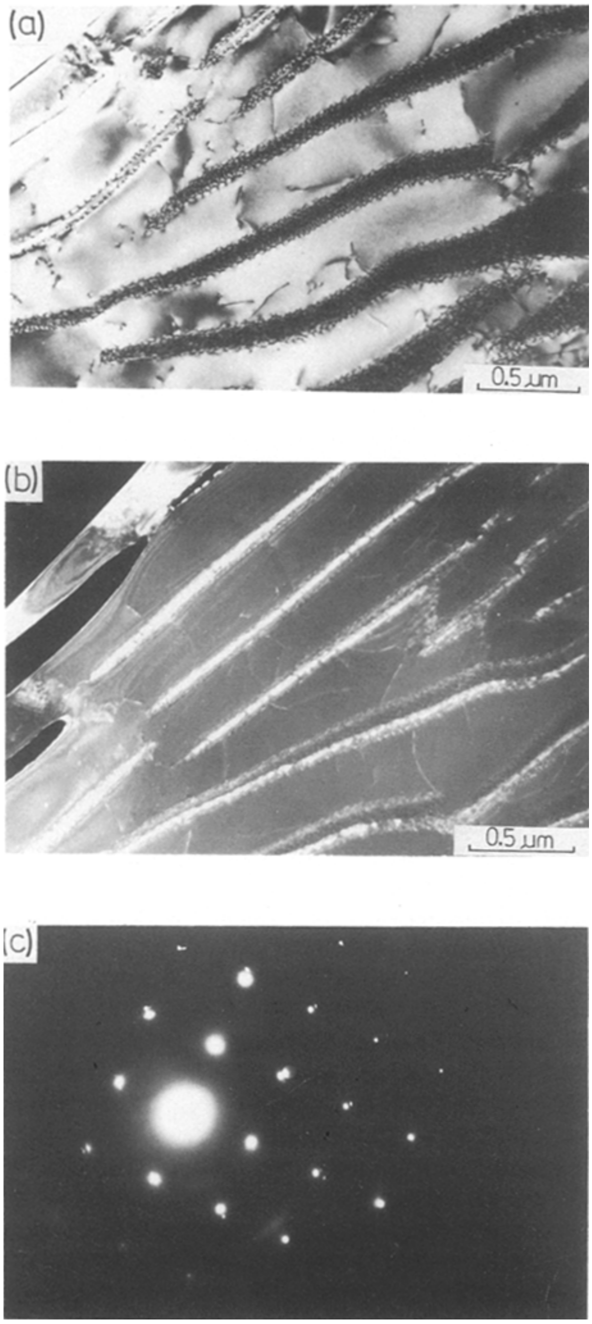

Figure 2 Set of transmission electron micrographs taken in the (a) bright field and (b) dark field using η phase relection and (c) corresponding diffraction pattern. Heat treatment is analogical to Fig. 1.

3. Results

Fig. 1 shows fragments of an X-ray diffraction record taken after 60 sec of dissolution at 603 K, where splitting of all reflections i.e. $\{1\,1\,1\}$, $\{2\,0\,0\}$, $\{2\,2\,0)$ indicates the presence of transition f c c α' phase. Its lattice parameter calculated from the presented results is equal to $a = 0.402\,55\,\mathrm{nm}$, which corresponds to the ~ 45.9 at % Zn after-calibration curve of Pawłowski and Truszkowski [2]. No splitting of basic reflections can be seen after quenching or ageing long enough to 60–80% of a discontinuous η precipitate. After a short time of dissolution, X-ray diffraction does not show the presence of the η phase.

Fig. 2 shows a microstructure typical for the first stage of dissolution. A net of dislocations visible in the micrograph is difficult to identify with respect to the

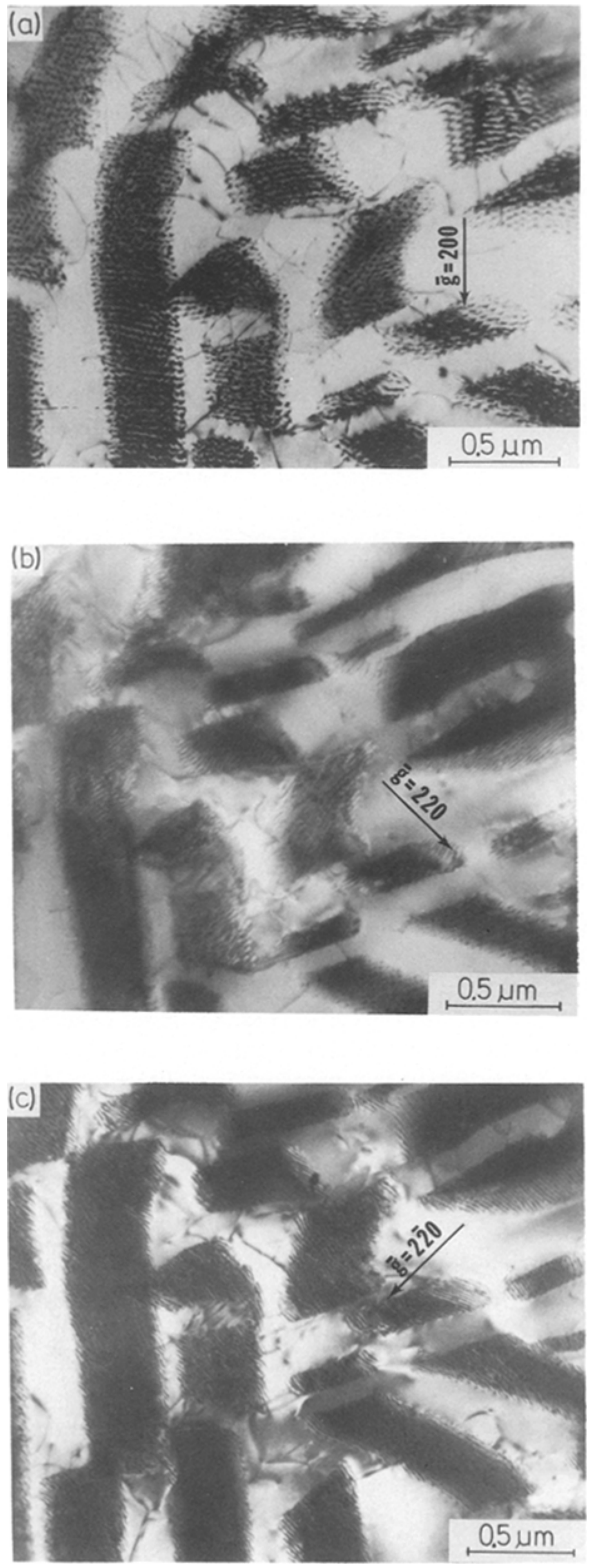

Figure 3 Set of bright field transmission electron micrographs taken at different diffraction conditions at orientation close to the [0 0 1] zone axis: (a) $\boldsymbol{g} = [2\,0\,0]$, (b) $\boldsymbol{g} = [2\,2\,0]$, (c) $\boldsymbol{g} = [2\,\bar{2}\,0]$. Heat treatment as in Fig. 1.

direction due to a high density. It exists in former places of a discontinuous η precipitate. The diffraction pattern (Fig. 2b) indicates [0 0 1] zone axis orientation and shows a significant splitting of reflections in the $\langle 1\,1\,0 \rangle$ directions. However, the splitting distance

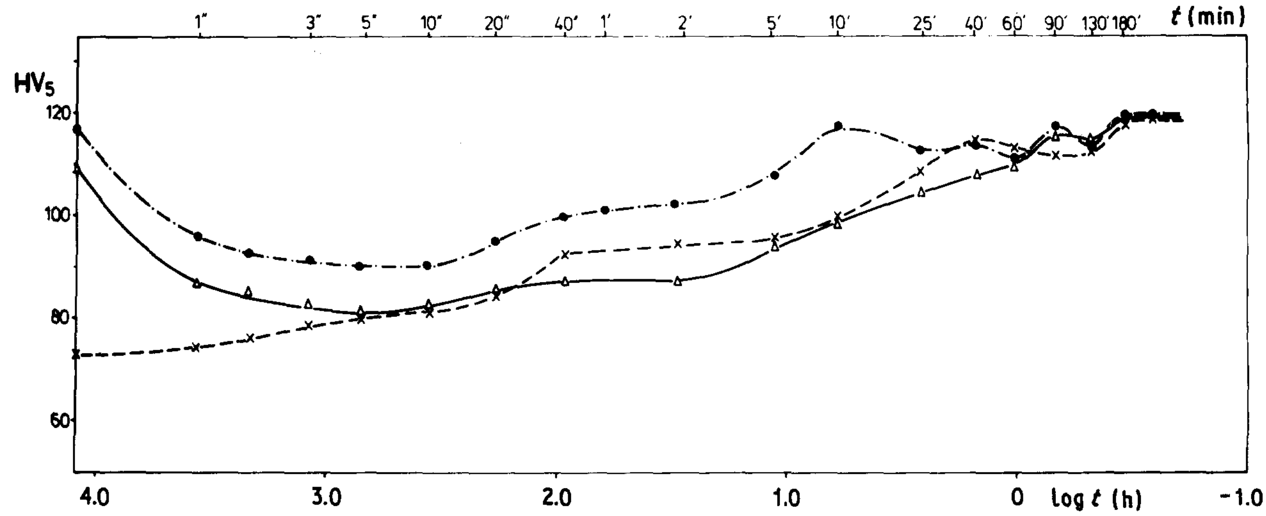

Figure 4 Hardness/dissolution curve at 603 K after ageing of sample at 373 K after different stages of decomposition according to a discontinuous mode. (● –·–·– ●) 40% η, (Δ —— Δ) 80% η, (x ---- x) 100% η.

does not correspond to the transition α' phase, but to $d_{11\bar{2}0}$ of the η phase; it can include also a transition phase reflection closer to the matrix reflection. A dark field image taken using additional reflection (Fig. 2b) marked in the diffraction pattern (Fig. 2c) shows that the thickness of the lamallae is decreasing with increasing thickness of the foil. This observation suggests that η precipitates may form in the thin areas of foil during electron microscopy observation.

Fig. 3 shows a set of microstructures of the transition phase precipitates taken at the zone axis orientation close to [0 0 1] direction, but at different diffraction conditions i.e. (a) $\boldsymbol{g} = [2\,0\,0]$, (b) $\boldsymbol{g} = [2\,2\,0]$, (c) $\boldsymbol{g} = [2\,\bar{2}\,0]$. At $\boldsymbol{g} = [2\,0\,0]$ (Fig. 3a) a net of dislocations can be seen, similar to that seen in Fig. 2, running basically in two perpendicular directions. Most of the elongated particles in the microstructure are parallel to the [1 0 0] direction but at this stage of dissolution the start of partitioning of precipitates can be seen leading to the formation of new interfaces parallel to the dislocation lines i.e. $\langle 1\,1\,0\rangle$. Dislocations lines perpendicular to the corresponding $\boldsymbol{g}$ vectors are clearly seen in Figs 3b and c. The analysis of the Burgers vectors of misfit dislocations visible in Fig. 3 according to the $\boldsymbol{g}.\boldsymbol{b} = 0$ invisibility condition indicates that there exists a net of perpendicular screw dislocations of $\boldsymbol{b} = \frac{1}{2}a\ \langle 1\,1\,0\rangle$. An additional proof supporting the screw character of misfit dislocations is that they completely disappear at the fulfilled $\boldsymbol{g}.\boldsymbol{b} = 0$ condition due to the fact that the displacement vector $\boldsymbol{R}$ is parallel to the Burgers vector only for the case of a screw dislocation, but not for an edge dislocation.

Fig. 4 shows the hardness curve for Al 22 at % Zn alloy aged at 373 K and then annealed at 623 K. Under these conditions only continuous dissolution takes place [2]. An increase in hardness after 1 min of ageing is caused (as confirmed by our structure investigation) by the formation of the semicoherent transition α' phase and generation of dislocations in the solid solution. A longer annealing time causes disappearance of the transition α' phase and the formation of a homogeneous solid solution which leads to the stabilization of hardness at the value of 120 Hv.

4. Discussion

The present investigation enables us to describe the mechanism of continuous dissolution of single plates of the η phase in the Al 22 at % Zn alloy.

In the first stage of dissolution within the η phase a transition α' phase is formed at the α–η phase boundaries. Its presence was documented by X-ray investigations where extra peaks near the $\{1\,1\,1\}$, $\{2\,0\,0\}$ and $\{2\,2\,0\}$ reflections were observed (Fig. 1). At interphase boundaries screw dislocations of Burgers vectors $\boldsymbol{b} = \frac{1}{2}a\ \langle 1\,1\,0\rangle$ are generated due to a misfit between the matrix and precipitate which is equal to 0.208% (calculated from the X-ray results). Assuming Hooke's law to be operating, the corresponding stress is given by $6 \simeq 2.08 \times 10^{-3} \times 49\,\mathrm{GPa}$ and exceeds the yield stress estimated for the investigated alloy (20 MPa). Existing dislocations running in the $\langle 1\,1\,0\rangle$ directions are paths of faster diffusion which causes the edges of precipitates to become parallel to these directions or induce fragmentation of precipitates along the dislocation lines (Fig. 3). The fragmentation during dissolution in Al–Zn alloys was reported by Pawłowski and Truszkowski [2], however without detailed explanation. Butler [8] suggested a considerable role of dislocations in the process of fragmentation of the θ phase in the Al–Cu alloys annealed above the solvus line. Solorzano *et al.* [4] observed screw dislocations during dissolution for the Al 22 at % Zn alloy, however in their opinion these resulted from misorientation occurring during the precipitation process. Bourchard *et al.* [7] observed directional fragmentation during coarsening of the spinodal structure in Cu–Ni–Fe and Cu–Al–Mn alloys and connected this phenomenon to the diffusion along misfit dislocations. The explanations of Butler [8] and Bourchard *et al.* [7] and our own observations show that the role of misfit dislocation in the directional fragmentation during dissolution seems to be the most important. The identification of η precipitates in the thin foils is in disagreement with X-ray diffraction results where this phase was not found after a certain time of dissolution. This divergence can be explained only as a fact of formation of η phase from transition α' phase during observation in the electron microscope. The transformation from α' containing more than 45 at % Zn into the η phase is very fast and the η phase lamellae were observed in very thin areas of the foil where the heating effect of the electron beam could significantly speed up the transformation $\alpha' \rightarrow \eta$.

5. Conclusions

During the first stage of continuous dissolution within η phase lamellae, the transition α' phase is formed, which nucleates at the α–η boundaries.

At α–α' boundaries a net of perpendicular misfit dislocations exists compensating for differences in lattice constants of both phases. They are of screw character, parallel to $\langle 1\,1\,0 \rangle$ directions and possess Burgers vector $\boldsymbol{b} = \frac{1}{2}a \langle 1\,1\,0 \rangle$.

During dissolution a change of shape of transition precipitates occurs often leading to their fragmentation in order to form edges parallel to the $\langle 1\,1\,0 \rangle$ directions.

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