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Influence of Cold-work on Subsequent Precipitation in an Al–20 wt% Ag Alloy

Precipitation in a supersaturated alloy is known to be altered considerably by a plastic deformation after solution-annealing and before ageing. This effect has been investigated particularly in several aluminium alloys by means of transmission electron microscopy (TEM) in the last few years (e.g. [1-5]). In the case of Al–Ag alloys however, no TEM-studies of precipitation after cold-work (especially heavy cold-work up to 99% thickness reduction) have been done to the authors' knowledge. Recently, Krause and Laird [6] examined an Al–15 wt % Ag alloy cold-rolled 50% and subsequently aged 1 day at 160° within the framework of an investigation of fatigue properties of aluminium alloys.

It is the purpose of this paper to present some results of an Al–20 wt % Ag alloy (prepared from 99.99% Al) in the following conditions: solution annealed 1 day 540° C (quenched in water of 15° C), then cold-worked by rolling with 10, 25, 50, 80, 90 or 99% thickness reduction and aged at 200, 250 or 300° C between 1 min and 1 day. Thin foils transparent for 100 kV electrons were prepared by the standard window technique using an electrolyte of 20% $HClO_4$ + 80% C_2H_5OH at 4° C and 17 V.

Without deformation, the decomposition follows the usual, well-known sequence [7] of spherical GP-zones, plate-like metastable γ' precipitates on {111}-matrix planes and the equilibrium phase γ, grown usually in a lamellar, discontinuous manner starting from grain-boundaries (fig. 1). However, according to Laird and Aaronson [8], γ may also grow continuously by a direct transition of γ' plates into the γ crystal structure by means of misfit dislocations. In this case, which was also observed in the present study in the case of undeformed specimens, the γ' and γ plates may not be distinguished easily.

After deformation in the solution-annealed state, the dislocation structure observed was similar to that described earlier [2] in the case of Al–4 wt% Cu, i.e., it consists of short, irregularly shaped dislocation lines and loops more or less randomly distributed after low or medium degrees of deformation (up to 50%). After

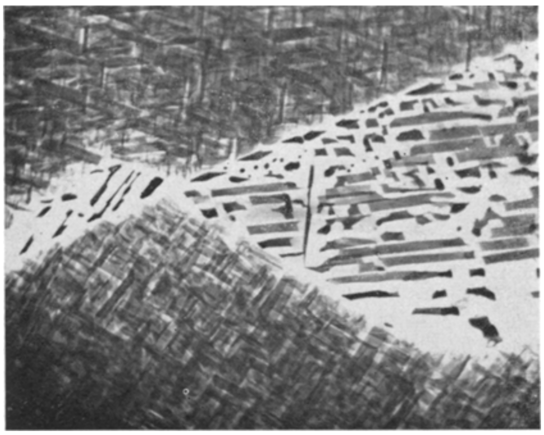

Figure 1 Al–20% Ag, 10 h 300° C. γ' in the grain interior, γ discontinuously grown from grain-boundaries (undeformed specimen) (× 2600).

higher deformation there develops a kind of cell structure, with "thick walls" (see fig. 1 in [1]). The cell diameter was 1 to 2 μm and the wall thickness about 0.5 μm.

The results of precipitation after deformation may be described as follows. After 10% cold-rolling, γ' formation was observed to be accelerated drastically on account of suppression of the GP-zones, in accordance with Murakami and Kawano [9] (mechanical, electrical and X-ray studies). This is due to the easier nucleation of γ' on dislocations introduced by the deformation. The γ' plates are much more numerous and much smaller than without deformation. Also the discontinuous γ formation is accelerated.

At medium degrees of deformation, 25 and 50 %, the same effects occur and, in addition, a new γ morphology appears sporadically: at some highly distorted lattice sites γ crystals grow, not as lamellae which is the usual habit originated discontinuously, but in an *isometric habit*, i.e. having a shape approximately equally extended in all three dimensions (nearly equiaxed) as seen in fig. 2 at A. This observation is in agreement with Krause and Laird [6] who also observed isometric γ crystals after 50 % coldwork (fig. 3 in [6]). The occurrence of the equilibrium phase in isometric habit (which is believed to grow in a continuous rather than a discontinuous manner) is similarly observed in the Al–Cu alloys: here, from a heavily cold-worked supersaturated matrix the θ phase grows in an isometric habit only [1, 2, 4]. Apart from isolated isometric crystals they sometimes appear in banded groups (as in fig. 6c in [2] or in fig. 6 in [6]). This is probably due to a corresponding banded dislocation structure before precipitation.

The most important modifications occur at large amounts of deformation (80 to 99 %), however. Firstly, the effect of "time-reversal" reported earlier for Al–Cu alloys [1, 10] was found here, too. It means that the equilibrium phase θ (or γ) appears *first* and the metastable (transition-) phase θ' (or γ') grows *later* if observed by an integrating method such as X-ray diffraction. This time-reversal may be explained as follows [1]. After severe deformation there are on a *submicroscopic scale* areas having a very high local dislocation density. Here the equilibrium phase grows first because the nucleation of its incoherent lattice is favoured by the strong local lattice distortions. The γ crystals growing in this way are moreover of the isometric habit. A typical example of this early state is seen in fig. 3. Obviously these γ particles grow in a continuous rather than a discontinuous manner. The semi-coherent transition phases, on the other hand, cannot nucleate in an extremely distorted lattice. Therefore they nucleate later in other matrix regions after the lattice there has become relatively perfect as a result of incipient recovery, provided the matrix has not yet decomposed. This is seen, after further ageing, in region B of fig. 4: the metastable γ' phase precipitates in the well-known habit of plates on {111} matrix planes, closely packed in a Widmannstätten

Figure 2 50% cold-rolled + 10 min 300° C. γ' precipitation and isometric γ crystals at "A" (× 20 000).

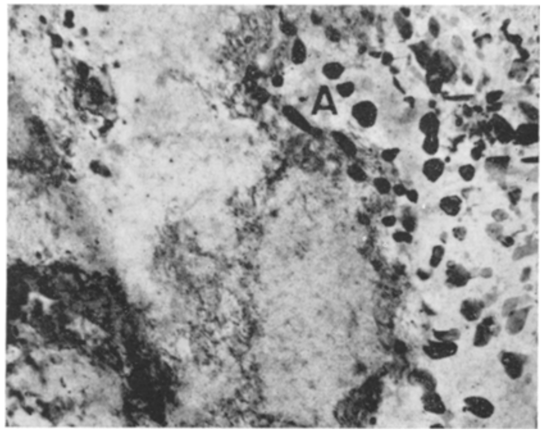

Figure 3 99% cold-rolled + 1 min 200° C. Precipitation starts in heavily distorted lattice regions (isometric γ at "A") (× 24 000).

manner. These precipitates form sharply bounded "islands", corresponding to the prior cells of the dislocation structure. Furthermore, in fig. 4 there are seen regions C of lamellar γ, formed obviously in the conventional (discontinuous) manner starting either from grain-boundaries or – more probably – from what were highly distorted spots in the grain interiors of the original cold-worked material. A typical *triplex-structure* is formed this way, consisting of the three components (fig. 4) A – isometric γ, continuously grown; B – plate-like γ', continuously grown; C – lamellar γ, discontinuously grown.

Decomposition starts in the mode A in heavily distorted lattice regions. Later on B follows in

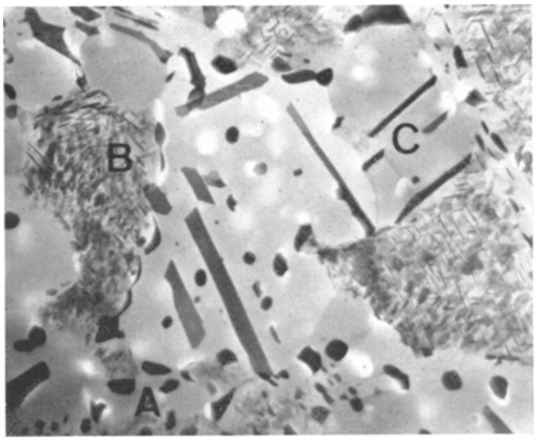

Figure 4 99% cold-rolled + 10 min 300° C. Typical triplex-structure of isometric γ (A), plate-like γ' (B) and lamellar γ (C) (× 8000).

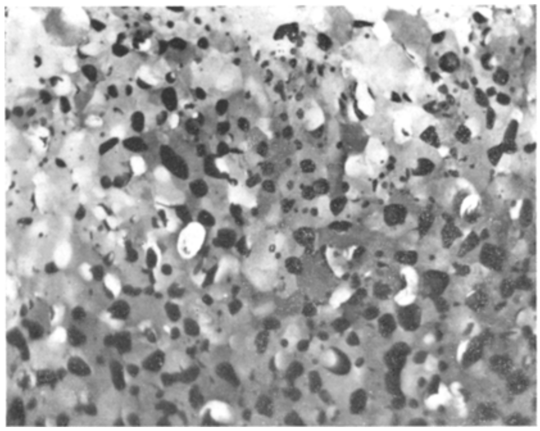

Figure 5 99% cold-rolled + 8 h 300° C. Stable type of precipitation after long ageing: only isometric γ remains × 2600).

regions having obtained a moderately low dislocation density, as explained above. Simultaneously, type C develops in other lattice regions. Generally, however, the amount of C is lower than A and B.

In the course of further ageing, it is remarkable that the equilibrium phase type A – which was the first to appear, locally – turns out to be the most stable type. Extended ageing after the highest degree of deformation (99%) produces *only* the isometric type (fig. 5). This extreme "single structure" appearance resulted only, however, after 99% deformation. Ageing following 90% or less deformation left some regions of type B and C. Obviously the driving force was not great enough in these cases to transform all parts into A which from a thermodynamical point of view also is considered to be the most stable type because of its equiaxed (as opposed to lamellar) morphology.

The $\gamma' \rightarrow \gamma$ transformation (B → A) was also examined by heating thin films containing a mixture A + B + C in the same manner as described earlier [11]. Series of micrographs were taken to record the changes of the same specimen section. Here the transformation occurred by dissolution of B and re-precipitation as A. Usually, existing isometric crystals nearby were the nucleation points. The fact of this $\gamma' \rightarrow \gamma$ transformation by dissolution is noticeable in so far as in undeformed specimens the γ' platelets transform directly into the γ structure [8]. This may be caused by the much bigger size of the γ' plates grown in undeformed samples (figs. 1, 4; note the relative magnifications).

Finally, the orientation relationship of the hexagonal γ crystals in the new modification A was investigated by means of selected area diffraction. In twenty-three of thirty analysed cases it was the same as the known relationship for cellular (lamellar) γ, namely $(1\,1\,1)_{\mathrm{Matrix}}//(00.1)_{\gamma}$; $[1\,1\,0]_{\mathrm{Matrix}}//[11.0]_{\gamma}$.

In the remaining seven cases no unambiguous analysis was possible owing to the difficulty that most of the isometric γ crystals are surrounded by several subgrains having slightly different orientations. (All treatments reported here were below the starting point of any recrystallisation.)

The partial or total suppression of the discontinuous precipitation C in favour of a continuous decomposition A as a result of prior deformation is to a certian degree in agreement with analogous effects in some copper alloys [12]. On the other hand, the morphology of the cellular reaction C, i.e. the lamellar form of γ, in Al–Ag alloys is not basically changed by prior deformation, in contrast with a Cu–15% In alloy [13]. Generally, the influence of deformation on the discontinuous decomposition in particular is a very complex matter, as reviewed recently by Borchers *et al* [14]. Current theories for discontinuous precipitation (Turnbull, Cahn) are difficult to test since they are based on the diffusion constants D_{B}, D_{V}, for diffusion along cell boundaries and within the matrix respectively. These diffusion constants are not safe to apply in the case of heavily cold-worked structures, since short-circuit diffusion along dislocations will play a major role. More work is needed therefore in order to elucidate results of the kind described here from a more analytical or theoretical point of view.

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Book Reviews

The Mechanism of Phase Transformation in Crystalline Solids

Pp, 324 (Institute of Metals Monogram no. 33, 1969) £6

This monograph which records the contributed papers, as well as oral and written discussion at an International Symposium held at the University of Manchester 3–5 July 1968, represents a very definite advance in the synthesis of the various aspects of phase transformations. This was the first international conference for more than 10 years in which the mechanisms of solid state phase transformations were considered. In the interim, many new aspects of the subject have been developed and enormous strides made in the experimental techniques used. At last we are beginning to see that the fundamental changes that occur in the structure, say of a steel, are paralleled in both non-ferrous and non-metallic systems. Another important point that has emerged from this conference is the ability to classify experimental observations and their associated theories into general schemes, which enable the fundamental mechanisms of these transformations to be more clearly understood.

As may be expected the conference was dominated by papers dealing with precipitation hardening, interface controlled transformations and martensitic reactions.

In the field of precipitation hardening, it is interesting to notice the accent on studies of what happens at the grain-boundaries. This change of emphasis has no doubt been prompted by the current interest in the fracture toughness of high strength precipitation hardening materials, the properties of which are strongly influenced by grain-boundary precipitates and trace elements.

Nicholson and Lorimer introduced a new theory of precipitation hardening which is concerned with the initial growth of the G P zones at low ageing temperatures and their subsequent transformation at higher temperatures. This paper provoked a good deal of discussion about the changes which occur in multi-stage heat-treatments and the formation of precipitate free zones at the grain-boundaries. It is perhaps pertinent to ask at this point if these multi-stage heat-treatments which appear to produce good properties under laboratory conditions are a viable proposition under industrial production conditions.

A second important subject which emerged at this session was the influence of trace impurity elements on the ageing behaviour. Papers by Brooks and Hatt, and also by Wilson, emphasised that these impurities tend to slow down the ageing reaction, probably by combining with the excess vacancies in the matrix, and hence favouring the formation of the semicoherent θ'—type