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DYNAMIC PRECIPITATION IN Al-4%Cu

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Abstract

Hot compression tests were performed on a previously solution treated and aged Al-4%Cu alloy. The effect of the precipitation occurring during hot deformation and the flow stress behaviour were investigated by means of scanning and transmission electron microscopy and electrical conductivity and hardness measurements. It was found a very heterogeneous microstructure with the development of regions of strain induced Al_2Cu growth and coarsening. In these regions, the coarsening produces flow localization and flow softening.

Introduction

Recovery, recrystallization and precipitation reactions have been under continuous investigation during many decades and even though they are all in general very complicated solid state reactions, they are now considered to be conceptually fairly well understood. It must, however, be admitted that the nucleation stage during recrystallization and precipitation is rather incompletely developed.
During the last decade it has been demonstrated repeatedly that these reactions can, under some conditions, take place under the influence of plastic straining. The terms dynamic recovery, recrystallization and precipitation have thus been introduced to distinguish these reactions from the corresponding static ones that take place without any applied plastic straining.
The basic understanding of the dynamic reactions is much less than that of the static ones, even though much progress has been made during the last years. The least investigated and least understood of the dynamic reactions is dynamic precipitation. Apart from extensive studies of dynamic precipitation in steels [1] and particularly in HSLA-steels, very little work has been done on other alloy systems. Quite recently Blaz and co-workers have investigated dynamic precipitation in an Cu-Si alloy [2] and Evangelista and co-workers have made similar studied in some Al alloys [3].
When precipitation takes place during plastic straining, the straining will affect all the three stages of the precipitation process: nucleation, growth and coarsening. In order to characterize the dynamic precipitation process it seems reasonable to try to investigate each of these reactions separately. The simplest stage to investigate seems to be the coarsening because the straining will affect the reaction mainly through the effect on the diffusion constants. The present investigation reports some (preliminary) results on the effect of plastic straining on the precipitation in an Al-Cu alloy.

Experimental

The alloy was produced from 99.997% Al and a 99.99% Cu alloy and grain refined by means of Al-5%Ti-1%B hardener before casting. The composition of the alloy is given in Table 1. Following an homogenization heat treatment at 540° C for 24 h the casting was broken down by rolling and intermediate anneal also at 540°C, to slabs of 15 mm thickness. To avoid a strong rolling texture in the final specimens, cross-rolling was applied during the last passes. The cylindrical compression specimens, 10 mm in diameter and 15 mm high, were machined from the slabs. The age hardening of the alloy was followed by hardness and electrical conductivity measurements. The electrical conductivity was obtained by a Fisher Sigmascope. The solution heat treatment of the compression specimens was carried out in salt bath at 540°C for only 60 s to avoid extensive grain growth in the specimens during this heat treatment (grain size 250 μm). A preliminary investigation demonstrated that full solubilization was obtained after this solution heat treatment. The specimens were subsequently quenched into water at room temperature and immediately transferred to liquid nitrogen to prevent pre-precipitation at room temperature during storage. The compression tests were carried out with a MTS-machine equipped with a heating chamber. The heating up period of the specimens from the liquid nitrogen to the testing temperature of 200°C was measured to be less than 60 s and the compression testing started when this temperature was reached. Several strain rates, included changes of the rates during the run were used. Here only the results from the series with $\dot{\varepsilon} =$ 5 10^{-5}, 10^{-4} and 10^{-3} s^{-1} will be presented. A more complete presentation will be given elsewhere. After the compression the specimen was immediately quenched in water and stored in liquid nitrogen. The metallographic investigation was carried out by means of the scanning and transmission electron microscope.

Table I: Chemical Composition in weight %.

Al	Cu	Ti	B	Si	Fe
bal.	4	0.01	0.005	0.01	0.004

Results and Discussion

The variation in hardness and electrical conductivity during ageing at 200°C is shown in Figure 1. The hardness increase passes through a maximum after approximately 24 h. The electrical conductivity increases parallel with the hardness until the maximum hardness is obtained and then remains constant on continued ageing.

Figure 2 shows the TEM-micrographs of the structure after 24 h ageing. A uniform distribution of θ'-plates, with dimensions of 10 nm in width and 200 nm in length, is seen in the matrix while, bulky θ-particles of 300 nm in diameter have formed on the grain boundaries. The hardness increase is due to the nucleation and growth of θ'-plates and the maximum hardness occurs at critical precipitate size and spacing of these plates. The increase in conductivity is due to the draining away of the Cu-atoms from the matrix during the growth stage of the θ'-plates and remains constant during the subsequent coarsening stage.
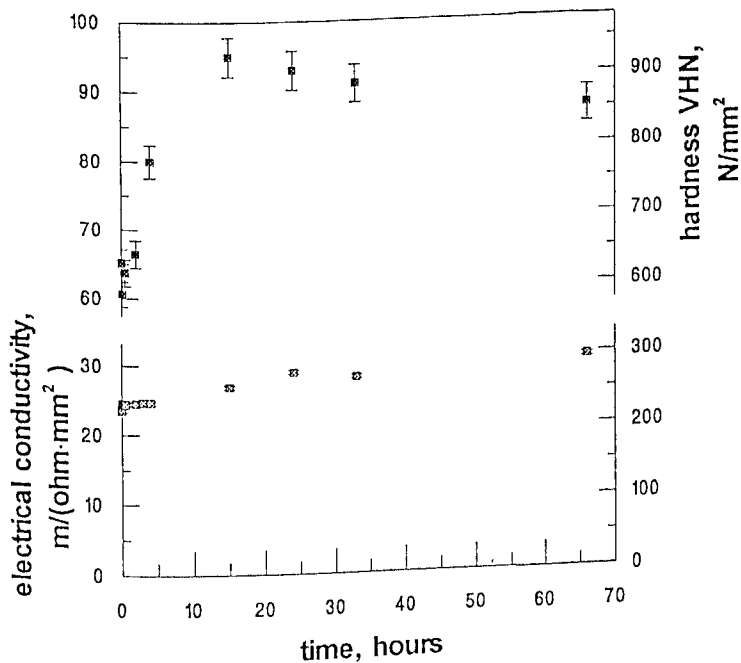
Figure 1. The variation in VHN and electrical conductivity during ageing at 200°C.

The results of the compression tests are shown in Figure 3. For all the strain rates used the σ-ε curves go through a fairly sharp maximum. The comparatively rapid softening that takes place
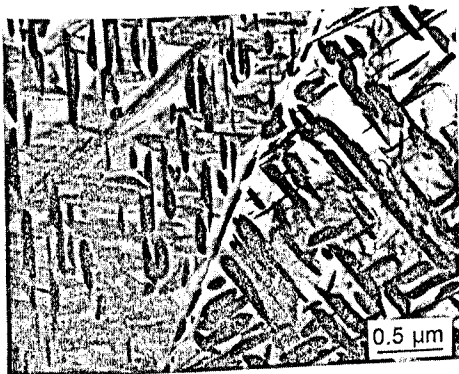

Figure 2. Microstructure after 24h at 200°C.

for equivalent strains larger than approximately 0.05 reflects an instability of the precipitation hardening structure with respect to the plastic straining. As a comparison, the σ-ε curve for pure Al is also shown in Figure 3. No similar softening takes place at this strain level in this case. The nature of this structural instability was investigated metallographically and some of the observations are reported here.
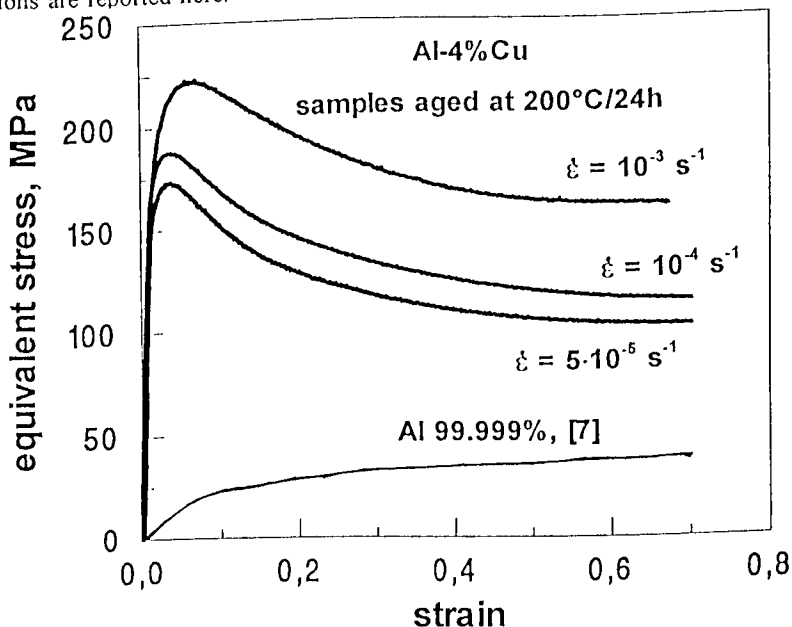

Figure 3. True stress-true strain curves obtained at three different strain rates after ageing at 200°C/24h ($\dot{\varepsilon}=10^{-4}$ s^{-1}).

The microstructure of specimens strained different amounts is shown in Figure 4. At ε = 0 (Fig. 4a) only θ-precipitates on the grain boundaries are visible. At ε = 0.17 (Fig. 4b) the particles within the grain boundary region seem to have substantially increased in size while the precipitates within the matrix are still too small to be seen. At ε = 0.7 (Fig. 4c) broad bands along the grain boundaries are seen, filled up with large particles. These bands are also seen locally to penetrate into the grain interior and also within these bands large particles are present. In between the bands the precipitates are still too small to be revealed. This microstructure was investigated in greater details by TEM. Particular attention was payed to the banded regions. Figure 5 shows a typical example of such a structure. The matrix region has been deformed rather uniformly and the contrast is blurred due to the overlapping of contrast from both θ'-precipitates and dislocations introduced during straining. A detailed dark field investigation showed the θ'-precipitates to be uniformly distributed in the matrix. In between two grains, a broad region has developed with a coarse subgrain structure and with very large θ-phase particles rather uniformly distributed. As it can be seen, the border between this band and the matrix is very sharp. However, at some locations, a penetration of the coarse structure into the grain interior has occurred. These TEM-observations of the microstructure were further extended by means of the SEM operating in the channelling contrast mode, Figure 6.
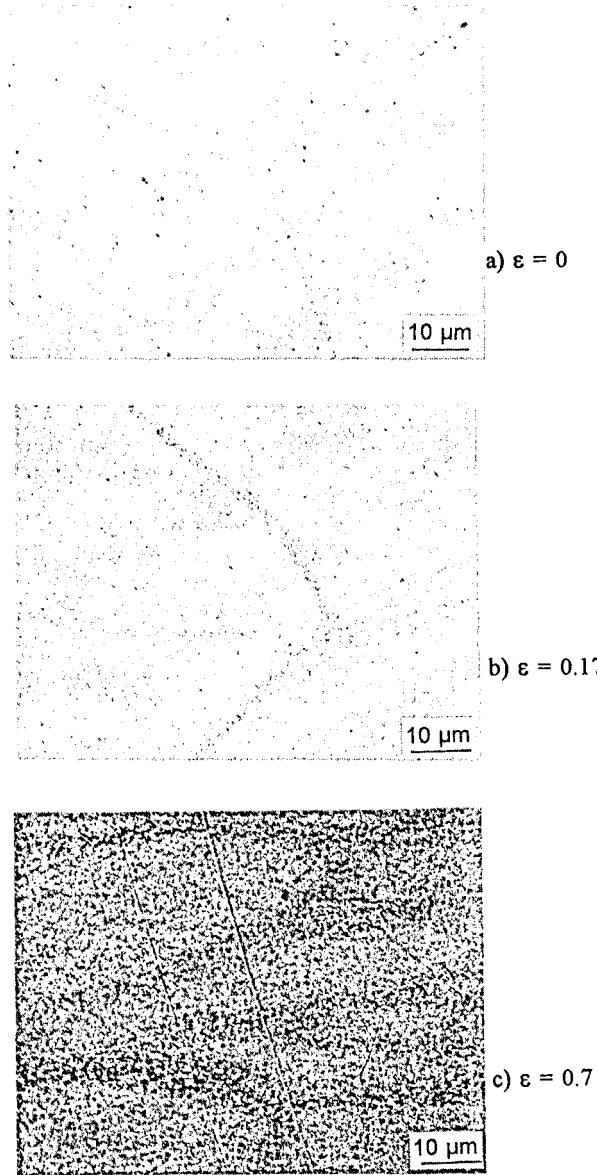
a) ε = 0

10 μm

b) ε = 0.17

10 μm

c) ε = 0.7

10 μm

Figure 4. Scanning electron micrographs showing the microstructure after different amounts of straining ($\dot{\varepsilon} = 10^{-4} \text{ s}^{-1}$).
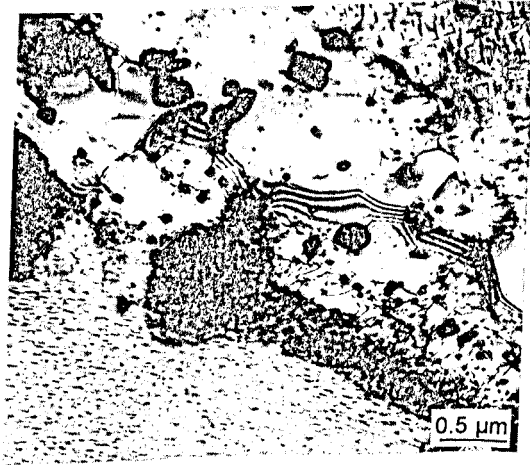
Figure 5. TEM micrograph showing the grain boundary region.

The grain interior seems to have been plastically deformed without forming a well defined subgrain structure, while in the banded regions rather large, regular subgrains can be seen. A more detailed presentations of the SEM results including also EBSP characterization of the deformed structure is given elsewhere.
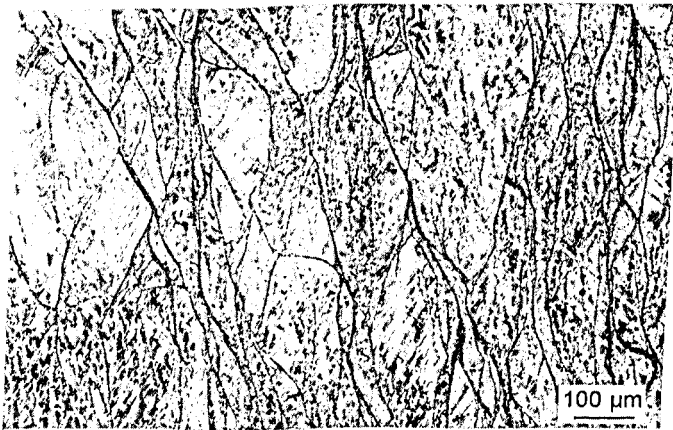

Figure 6. Channeling contrast micrograph showing microstructure at $\varepsilon = 0.7$.

These results demonstrate that a uniform, strain-induced coarsening of the θ'-precipitates does not take place under these experimental conditions. Instead, the microstructure has become exceedingly heterogeneous and regions of coarse θ-particles have developed. Similar structures have been reported by Blaz et al. [2] and Evangelista et al. [3]. The explanation to this phenomenon can be as follows: the distribution of strain in policrystals is known to be inhomogeneous during plastic straining. Due to the requirement of compatibility of strain across

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the grain boundaries, a particular turbulent deformation pattern is known to develop in these regions [4]. This strain localization during straining will increase the rate of diffusion in these region. It is well known that pipe diffusion along stationary dislocation lines substantially increases the diffusivity, particularly at low temperatures [5]. The mechanism by which moving dislocations increase the diffusivity has so far not been considered in details, but the effect is most probably even stronger than for stationary dislocations. The θ-particles will start to grow on the expense of the θ'-precipitates in the matrix. This coarsening will reduce the flow stress in these regions which amplifies the localization of the strain even further in the same region. A coarse substructure is thus developed within these regions and new θ-particles can form on these subboundaries.

As it can be seen the θ-particles reach quite large dimensions during this reaction and the diffusion coefficient D must have a correspondingly high value. A very simple estimate of the D can be made in the following way. The mean size r of the Al_2Cu precipitates in the coarse bands, after straining at 200°C for a time t of 3000 s, is approximately 100 nm ($\dot{\epsilon}=10^{-4}$ s^{-1}). As a first approximation, we write:

$$ r \approx \sqrt{D\,t} \qquad\qquad (1) $$

which gives a diffusion coefficient D for Cu in Al of approximately 3 10^{-14} cm^2/s. The diffusion coefficient of Cu in Al at the same temperature, determined from the data in the Metals Handbook [6] is 3.6 10^{-20} cm^2/s. Thus the straining has increased in this case the magnitude of the diffusion coefficient at 200°C by a factor of approximately 10^6.

Conclusions

The microstructural investigation performed on the hot deformed Al-4%Cu demonstrated the development of a very heterogeneous microstructure. In particular, coarse θ particle regions developed due to strain localization and the following increase of Cu diffusion coefficient in Al. As a consequence, there was a rapid softening in the equivalent stress-equivalent strain curves that took place at true strains larger than 0.05.

References

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