The Influence of Stress Reversals on the Structure of Initially Cold-Rolled Copper

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Received 26 June 1967, and in revised form 20 January 1968

An electron microscope study of initially cold-rolled copper has revealed that cyclic stressing, in the long life region of the *S*/log (*N*) curve, can promote room temperature recovery in isolated regions. Examples of polygonisation, subgrain growth and even a recrystallised grain have been detected in specimens possessing predominantly the typical cold-rolled structure. This is envisaged as a preliminary stage allowing eventually the formation of fatigue damage in certain regions.

1. Introduction

Previous studies of the influence of stress or strain cycling on cold-worked metals at room temperature, for reviews see [1, 2], have shown that softening can occur, accompanied by a reduction in internal stresses as revealed by X-rays. The structural changes associated with this "fatigue softening" in the case of cold-rolled aluminium were shown to involve polygonisation [3], and even recrystallisation [4].

More recently, thin film electron microscopy has been used to study the effects of fatigue upon cold-worked copper, a higher melting point metal. It has been shown [5, 6] that at high strain amplitudes, giving lives generally less than 10^5 cycles, the cold-worked structure is replaced by an equiaxed cell structure whose size is independent of stress or strain history, but is directly related to the current strain amplitude. The final structure is comparable to that developed in a fully annealed specimen subjected to high strain amplitude cycling and one would expect the failure mechanism to be similar for both cases.

The behaviour at low strain amplitudes, giving lives in excess of 10^6 cycles, is not so well documented, mainly because fatigue-induced damage is not uniformly distributed throughout the entire structure, as in the case of high strain fatigue, and also because a low strain fatigue specimen after failure will contain damage produced by the high strains existing near a crack front. Thus, as with the fatigue failure of fully annealed specimens, the surface grains are the only part of the fractured specimen containing low strain fatigue damage.

Some low strain amplitude tests [7] have reported the formation of equiaxed cells but in these tests a mean stress was present, which gave appreciable creep, and this may have influenced cell formation as discussed elsewhere [8]. Some work on a swaged Cu/Ni/Fe alloy [9], also reported this type of structural change. In this case the original "woolly cell structure" was found to change to a "sharp cell structure" in specimens which failed in $\sim 4 \times 10^5$ cycles; however, this alloy also displayed complex changes in precipitate structure. These results appear to indicate that low amplitude fatigue failure of cold-worked copper is similar to the high strain mode. This is in sharp contrast to the fully annealed copper behaviour, where at low strain amplitudes cell formation has not been observed [8, 10, 11].

In order to avoid the above complications, the present electron microscope study of the structural changes occurring during the low amplitude fatigue of cold-rolled copper has been made at zero mean stress and strain.

2. Experimental Procedure

Electrolytic copper sheet, 0.5 mm thick and nominally annealed, was cold-rolled down to 0.15 mm prior to testing.

Several samples were stress cycled in a Weidemann Baldwin reversed plane bending

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fatigue machine; the method used being that of cementing the thin sheet to a normal copper specimen. The thin sheet specimens were subsequently removed by immersion in a bath of toluene and thinned for examination electrolytically. Examination was conducted in a Siemens Elmiskop I at 100 kV.

3. Results

Samples of cold-rolled copper thinned prior to the application of stress cycles could be seen to possess a very high dislocation density arranged in what is generally termed a cloudy distribution [12].

The first sample was tested at 7.8 kg/mm^2 for 6.8×10^5 cycles, the test being interrupted prior to failure. Typical examples of the structure found in this sample are shown in figs. 1 and 2 and reveal a striking difference from those which had not been fatigued. The difference is in the presence of clearly defined boundaries between the regions of high and low dislocation density. In fig. 1 a completely enclosed dislocation-free region can clearly be seen whilst fig. 2 shows a more loosely defined structure which would appear to be an early form of polygonisation.

Figure 1 Dislocation-free region seen in a fatigued cold-rolled copper sample (80% red.; $S = 7.8$ kg/mm^2; $N = 6.8 \times 10^5$; $\times 36\,000$).

A further test conducted at 10.1 kg/mm^2 for 13.67×10^5 cycles, showed, on examination of the attached sheet sample, an even greater degree of recovery.

Fig. 3 shows a region of this sample containing a completely recrystallised grain surrounded by the originally highly worked structure.

Fig. 4 shows a partial stage in the process for it shows an example of a subgrain undergoing

Figure 2 Early form of polygonisation seen in a fatigued cold-rolled copper sample (80% red.; $S = 7.8$ kg/mm^2; $N = 6.8 \times 10^5$; $\times 18\,000$).

growth. One side of the subgrain is probably the side of low misorientation for one can still see the dislocation tangles there whilst the other is a very clearly marked boundary and is the boundary undergoing movement during the subgrain growth.

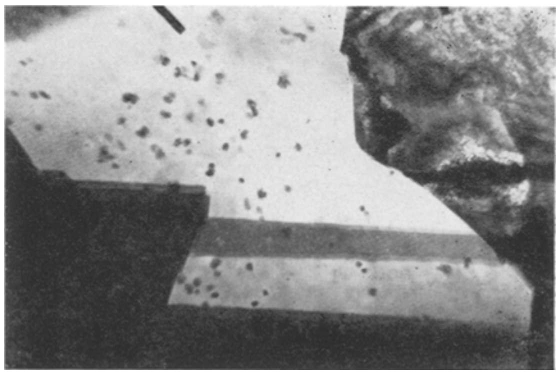

Figure 3 Recrystallised grain seen in a fatigued cold-rolled copper sample. (80% red.; $S = 10.1$ kg/mm^2; $N = 13.67 \times 10^5$, $\times 36\,000$).

These features were not typical of all the specimen regions observed and an example of the most commonly found structure is shown in fig. 5.

It is of importance at this stage to establish whether the deformation seen in specimens of only 0.15 mm in thickness is truly representative of deformation in much larger specimens. The order of magnitude of the specimens examined here lies between that necessary for examination in the electron microscope and that of bulk specimens. These two extremes have been found

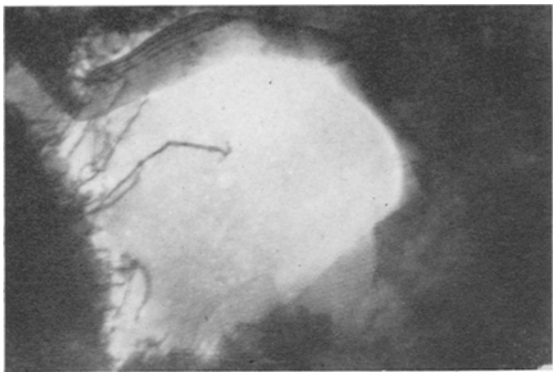

Figure 4 Subgrain undergoing growth in a fatigued cold-rolled copper sample. (80% red.; $S = 10.1$ kg/mm^2 $N = 13.67 \times 10^5$; $\times 36\,000$).

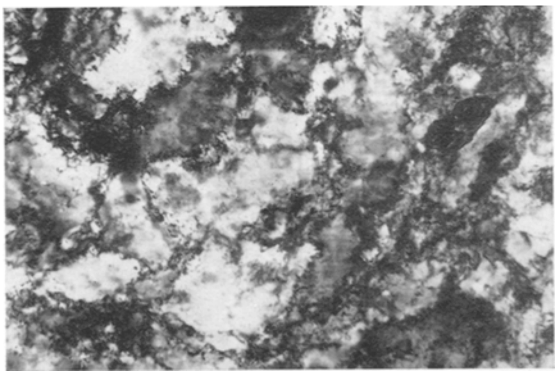

Figure 5 Polygonisation seen in a fatigued cold-rolled copper sample. (80% red.; $S = 10.1$ kg/mm^2; $N = 13.67 \times 10^5$; $\times 18\,000$).

to exhibit quite different deformation characteristics, see for example [13] on the use of a straining device within the electron microscope, probably because in the case of the thin foil there are two free surfaces in close proximity.

Previous work [10] under identical conditions to those used for the results presented here showed that the fatigue damage produced in a sheet specimen of 0.15 mm in thickness was truly representative of the fatigue damage found in bulk specimens. The observations presented earlier in this section on recovery processes occurring during cyclic loading also appear to be representative of bulk specimen recovery but as a precaution identical specimens were thinned prior to load cycling and the recovery of these specimens was observed directly in the electron microscope through the use of a heating stage. The results were found to be quite different from those given earlier and it is considered that the observations on recrystallisation given here are typical of the behaviour of the bulk material.

4. Discussion

Cold-rolling of copper is characterised by an ill-defined cell structure at low deformations gradually changing into what has been termed a cloudy dislocation structure at higher deformations. The most significant features of this latter type are the extremely high dislocation density and the absence of clearly defined boundaries between regions of high and low dislocation density.

Recovery, at an elevated temperature, occurs by gradual polygonisation, mainly in the regions of high misorientation, followed by recrystallisation, a process involving the production of dislocation-free grains by the migration of the high angle grain boundaries formed.

The recrystallisation temperature is known to be dependent on the degree of preliminary cold-working, but the influence of cyclic stressing on this temperature has not been clearly established.

It has been shown [14], that the recrystallisation temperature of electrodeposited copper was influenced by stress cycling and a relationship was derived between the recrystallisation temperature and the stress amplitude. The occurrence of recrystallisation was determined in these tests by an optical microscope examination.

The mechanical system of testing used in the present work is analogous to the copper plate test conditions and the results are complementary. The stress levels used here, in the range of 8 to 10 kg/mm^2, on the basis of the results from [14] would be expected to produce recrystallisation at room temperature, especially when one takes into account the more precise determination of recrystallisation used in the present case.

The structure seen after stress cycling the heavily cold-rolled copper at room temperature clearly shows evidence of polygonisation and the beginning of recrystallisation. Fig. 2 shows a region where only some polygonisation had occurred, and this was presumably initially a region of low misorientation. Figs. 1, 3, and 4 show the later stages in the process where complete low dislocation density grains have been formed.

It would appear from this that stress cycling of initially cold-worked copper can cause some regions to recrystallise even at room temperature, depending upon the stress amplitude and this is the probable explanation for the fatigue softening phenomenon displayed by work-hardened copper during a fatigue test at low amplitude. It is possible that a network of recrystallised regions is formed in the material, leaving islands containing the original structure, and that these softened regions are the sites of fatigue damage. No sign of fatigue damage was detected in the present work, and it is presumed that this is because the tests were interrupted prior to the formation of the fatigue damage and indeed probably before the completed network of softened or recrystallised regions had been achieved.

These observations have a parallel in an earlier study [15] of the release of stored energy from a fatigued material during subsequent annealing. The authors deduced from results showing that a smaller energy release occurred after stress cycling that regions of recovery existed in the originally work-hardened structure. This was considered to be due to the mode of stressing, i.e. in a rotating bending test only the surface grains experience the maximum stress and one would expect recovery to be limited to this surface region. The present results indicate that this partial recovery is not necessarily dependent on the mode of stressing but could be due to the formation of a network of softened regions, which can eventually form regions of fatigue damage, leaving islands of material still in the initial work-hardened state.

The physical reasons for this reversion of state at room temperature are associated with the influence of stress cycling on the initial stored energy and the energy input during a test. The latter quantity is probably associated with the high concentration of vacancies known to be produced by stress cycling which in turn could facilitate dislocation climb, resulting in a limited degree of polygonisation [16]. Recrystallisation is probably dependent on both the high vacancy concentration, to assist nucleation, and the stored energy, to assist grain growth. If this were the case one would expect a similar process to occur at high stress levels, i.e. recrystallisation prior to cell structure formation. This has not been observed possibly because the process occurs very rapidly at high stresses and is completed within a relatively few cycles.

5. Conclusions

(i) Primary recrystallisation has been seen to occur during the stress cycling of heavily cold-rolled copper at room temperature.

(ii) Regions of polygonised and recrystallised structure were found; the presence of either of these probably being determined by the degree of misorientation in the structure.

(iii) The drop in flow stress during the stress cycling of work-hardened copper is due to the presence of partial recrystallisation.

(iv) Recrystallisation forms an intermediate stage in the change from a cold-worked structure to the fatigue-hardened state, and is probably limited to only certain regions of the material.

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