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Dynamic Precipitation During Cyclic Deformation in Under Aged Al-4Cu Containing θ′ (Al_2Cu) Precipitates

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The cyclic deformation behaviour of an underaged model Al-4Cu alloy has been studied using constant plastic strain amplitude tests. The cyclic stress-strain curves exhibit a rapid cyclic hardening behaviour that is absent from peak aged or overaged states of the same alloy. The cyclic hardening increment approaches 50% of the initial yield stress. The corresponding transmission electron microscopy (TEM) observations indicate that the cyclic hardening is due to the room temperature dynamic precipitation of a large number of GP zones. Both the size and number density of GP zones continually increase with the number of cycles until reaching the peak stress level. The cyclic hardening behaviour is relatively insensitive to the applied strain-rate but shows a dependence on the applied plastic strain amplitude.

Keywords: dynamic precipitation, cyclic hardening, θ′ precipitate state

1. Introduction

The fatigue life of engineering alloys depends on the cumulative effects of two processes: fatigue crack nucleation and fatigue crack growth [1]. During the initial stages of cyclic deformation, deformation structures are developed within the material and these lead to conditions that can strongly influence fatigue crack nucleation [1]. An understanding of the cyclic deformation and hardening/softening behaviour is a pre-requisite to understanding fatigue crack nucleation.

In precipitation hardened Al alloys, both cyclic hardening and cyclic softening can be observed depending of the loading conditions and the microstructural state. In general, the cyclic hardening behaviour is induced by the multiplication of dislocations and the formation of various kinds of dislocation structures, such as walls, veins or cells, depending on loading and the plastic strain amplitude [2]. Cyclic softening is most common in pre-strained metals and is usually associated with dislocation recovery, strain localisation in shear bands or the nucleation of small cracks [3,4]. Hence, cyclic softening appears as a prerequisite for a premature failure and should be avoided for extended fatigue life. In addition the cyclic behaviour can also be dramatically affected by the strain/stress induced phase transformations that can potentially occur in metastable solid solutions. In this work, the cyclic deformation behaviour of a model Al-4Cu alloy containing relatively shear resistant θ′ (Al_2Cu) precipitates has been studied using constant plastic strain amplitude tests. The cyclic stress-strain curves of the under aged alloy show a rapid cyclic hardening behaviour that is absent from peak aged or overaged states of the same alloy. The peak aged and overaged alloys typically show stable cyclic stress strain behaviour or, in some cases, slight cyclic softening. The only difference in microstructure of these aging alloys is the state of the θ′ precipitates and the amount of Cu in solid solution. The objective of this work is to uncover the origin of the rapid cyclic hardening in under aged Al-4Cu alloy.

2. Experimental procedures

An Al-4Cu alloy was solution treated at 520ºC for 1h, water quenched and then aged at 200ºC for different times in order to acquire different θ′ precipitate states (length, thickness, number density, volume fraction). After ageing, all samples were stored in a refrigerator to minimize natural aging. The monotonic deformation behaviour of the Al-4Cu alloy containing θ′ precipitates can be found in [5]. The fatigue tests were conducted using an MTS-858 table-top system at room temperature using samples with gauge dimensions of 4mm×5mm×16mm. Before the mechanical testing, the surfaces of all samples were carefully polished. The cyclic deformation tests were conducted in constant plastic strain amplitude mode, with five amplitudes: $\Delta\varepsilon_{pl}/2=1\times10^{-4}$, 5×10^{-4}, 1×10^{-3}, 2×10^{-3} and 3×10^{-3}. For the under aged Al-4Cu alloy (aging 10min at 200°C), the cyclic stress-strain curve shows a rapid cyclic hardening behaviour. In order to reveal the hardening mechanism, fatigue tests were designed to uncover the effects of plastic strain amplitude and strain-rate on the hardening behaviour.

The microstructures formed in the underaged Al-4Cu alloy during fatigue deformation were investigated by TEM and high-resolution TEM (HRTEM) using JEOL-2011 and JEOL-2100F transmission electron microscopes, respectively, both operating at 200keV. The surface deformation features of the fatigued samples were examined using optical microscopy.

3. Results and Discussion

3.1 Rapid cyclic hardening in under aged Al-4Cu alloy

During constant plastic strain controlled fatigue tests, the underaged alloy (10min at 200°C) demonstrates a rapid cyclic hardening behaviour that is absent from the other aging states, Fig. 1a. The magnitude of the cyclic hardening for the 10min aged specimen is significant: at $\Delta\varepsilon_{pl}/2=1\times10^{-3}$, the cyclic peak stress increases from 200MPa to 280MPa. The hardening increment approaches 50% of the initial yield stress of the alloy. However, the other four aging states considered show relatively flat cyclic stress-strain curves with very little hardening. Some of them even indicate some softening, such as the peak aged (1.5h) and over aged states (8h and 4 days), Fig. 1a. The rapid cyclic hardening in the 10min alloy was observed at all applied plastic strain amplitudes studied, as demonstrated in Fig. 1b. In general, the larger the imposed plastic strain amplitude, the greater the peak hardening stress reached. It is also interesting to note that the fatigue life, in terms of the cumulative plastic strain, becomes notably longer with increasing plastic strain amplitude as shown in Fig. 1b. This behaviour has to be understood from a microstructural understanding of the cyclic hardening behaviour in this state of the alloy.

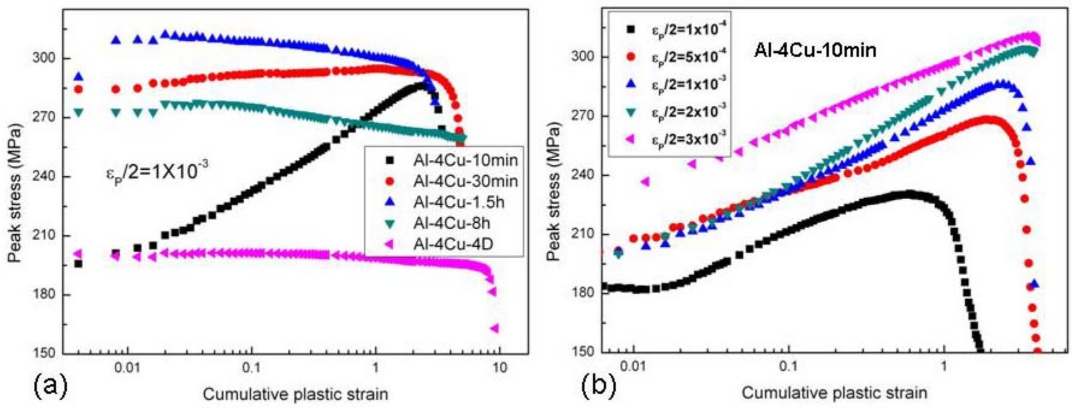

Fig. 1. (a) The cyclic stress strain curves for the five aging conditions considered tested at a constant plastic strain amplitude of $\Delta\varepsilon_p/2=1\times10^{-3}$; (b) The cyclic stress strain curves for the under aged 10min alloy tested at different plastic strain amplitudes.

Hence, the key issue is to identify the mechanism of cyclic hardening in the under aged (10min) alloy. The surface morphology of the samples after fatigue loading were examined using optical microscopy and surface deformation features of the under aged 10min sample and an over aged (aging for 4 days) sample are shown in Fig. 2. It can be clearly seen that there are dense slips bands formed on the surface of the 10min aged sample, while there are no slip bands on the surface of the over aged sample. The slip bands formed on the surface of the 10min aged sample likely mean that the θ′ precipitates in this alloy can be sheared after prolonged fatigue loading. TEM observations reveal that the θ′ precipitates have a thickness of only 1~2 nm and length of 30~40 nm [6]. For the over aged alloy, there are no shear bands inside the grains and it is likely that the θ′ precipitates remain shear-resistant in this aging state, even after prolonged fatigue loading. The θ′ precipitates for the over aged state are thicker and longer than the under aged state; ~10nm thick and lengths >100nm. Strain localisation is usually associated with cyclic softening. Hence the surface observations on the cyclic deformed underaged Al-4Cu sample would suggest softening rather than hardening. This observation indicates that the cyclic hardening in the underaged alloy has an origin other than that to do with the different state of θ′ precipitates.

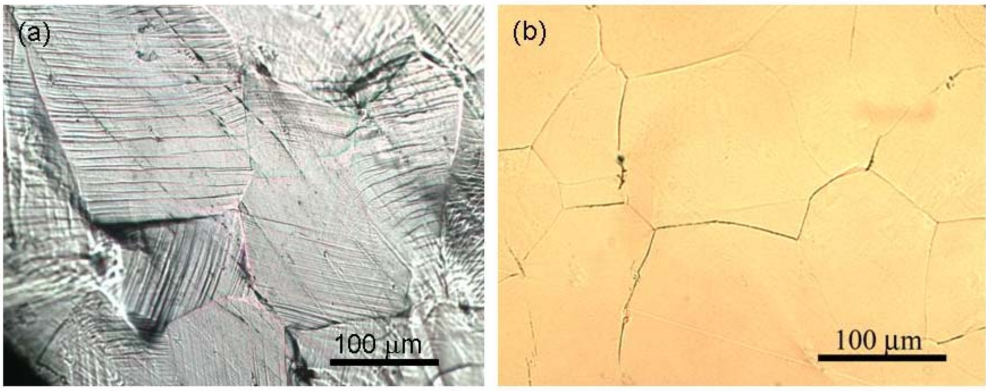

Fig. 2. Optical microscopy images of the surface morphology of the Al-4Cu alloy after fatigue deformation: (a) aging at 200°C for 10min alloy; (b) aging at 200°C for 4 days alloy.

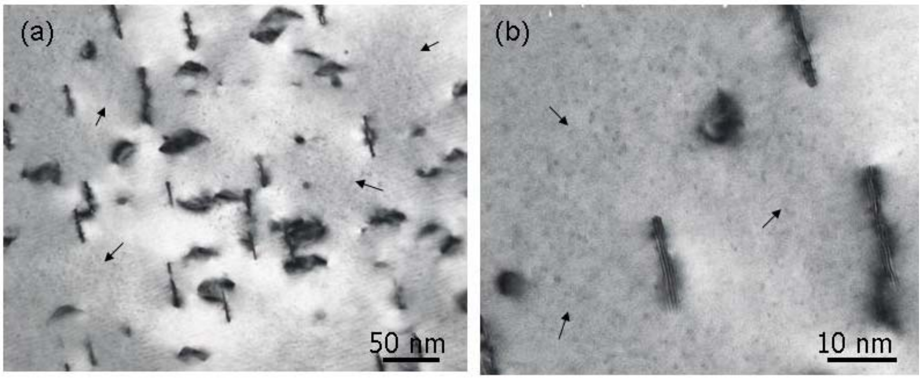

Fig. 3. Bright field TEM micrographs of the 10min alloy deformed cyclically to the peak stress level at $\Delta\varepsilon_{pl}/2 = 1\times10^{-3}$. The electron beam is close to $[011]_\alpha$.

3.2 Cyclic hardening by dynamic precipitation of GP zones

In order to help identify the cyclic hardening mechanism in the under aged alloy, samples after fatigue deformation were examined using TEM and HRTEM. The microstructures of the Al-4Cu-10min alloy before (not shown here) and after fatigue deformation were compared. The observations reveal that there are no obvious differences in the length, thickness and the number density of the θ′ precipitates. The most obvious feature is that Orowan loops are formed on some of the θ′ precipitates after fatigue loading (Fig. 3a). In addition, some very small features between the θ′ precipitates can be seen (arrowed in Fig. 3a). The higher magnification image in Fig. 3b shows that these features are a second phase, with the scale of 2-5 nm and a high number density.

Investigation using high-angle annular detector dark-field (HAADF) imaging reveals that these features are GP zones consisting of one atomic layer of Cu atoms, Fig. 4. There is no obvious preference for the variants of the GP zones that form with respect to the loading direction. GP zone formation on each of the three $\{100\}_\alpha$ planes is observed. It thus becomes evident that room temperature, fatigue induced precipitation of GP zones is the origin of the rapid cyclic hardening observed in the under aged alloy. From comparisons with the sample aged for 10min prior to fatigue deformation it can be definitively stated that the GP zones form as a result of the fatigue loading and not due to natural aging at room temperature prior to fatigue testing. It is likely that such precipitation is not observed in the other aging states because there is insufficient solute Cu in solution.

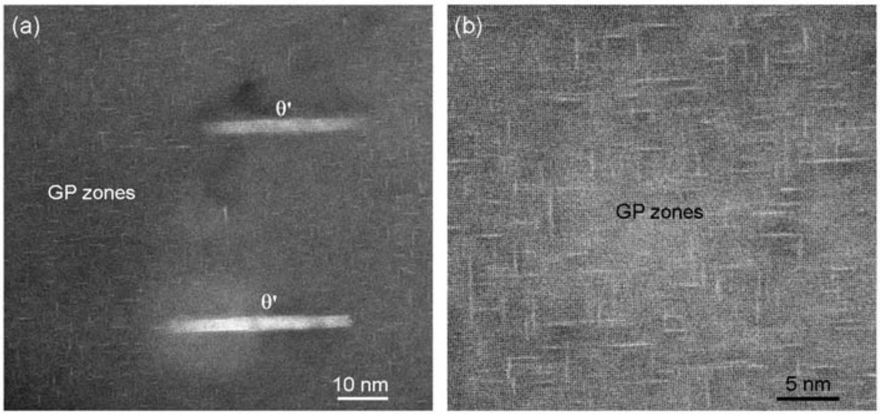

Fig. 4. High angle annular dark-field (HAADF) micrographs of the Al-4Cu-10min alloy deformed cyclically to the peak stress level at $\Delta\varepsilon_{pl}/2 = 1\times10^{-3}$. The electron beam is close to $[001]_\alpha$.

3.3 Dynamic precipitation mechanism

The observations shown in Figs. 3 and 4 suggest that the rapid cyclic hardening of the under aged alloy is due to room temperature, fatigue induced dynamic precipitation of GP zones. In order to examine the dynamic precipitation process, the fatigue test was interrupted after 70 cycles, 300 cycles and at the peak stress (~1000 cycles), and the corresponding deformation structures were investigated using TEM (Figs. 5a-c). The results indicate that both the number density and size of GP zones are continuously increasing with cumulative plastic strain.

GP zones formation requires mass transfer and this depends on the presence of vacancies. It can be readily shown that room temperature precipitation of GP zones to the extent shown in Fig. 4 requires a large excess vacancy concentration. The required excess vacancy concentration is a result of the plastic deformation and the production rate of deformation-induced vacancies is expected to be proportional to the applied strain-rate [7].

To reveal the effect of strain-rate on the cyclic hardening process, fatigue tests have been performed with different cyclic frequencies (at the same applied plastic strain amplitude $\Delta\varepsilon_p/2 = 5\times10^{-4}$). The cyclic stress strain curves of the 10min aged alloy fatigued with frequencies of: $f = 1Hz$, $f = 0.2Hz$ and $f = 0.02Hz$ are shown in Fig. 6a. Even though the strain-rates differ by approximately two orders of magnitude, the cyclic hardening behaviour of the three tests is very similar. Although the production rate of vacancies (and therefore the effective Cu solute diffusion coefficient) increases with increasing strain-rate, the time to reach a given cumulative plastic strain (and therefore the time for Cu diffusion to make GP zones) decreases with increasing strain-rate. It appears from Fig. 6a that these competing effects cancel out each other giving rise to a relatively strain-rate independent cyclic hardening behaviour.

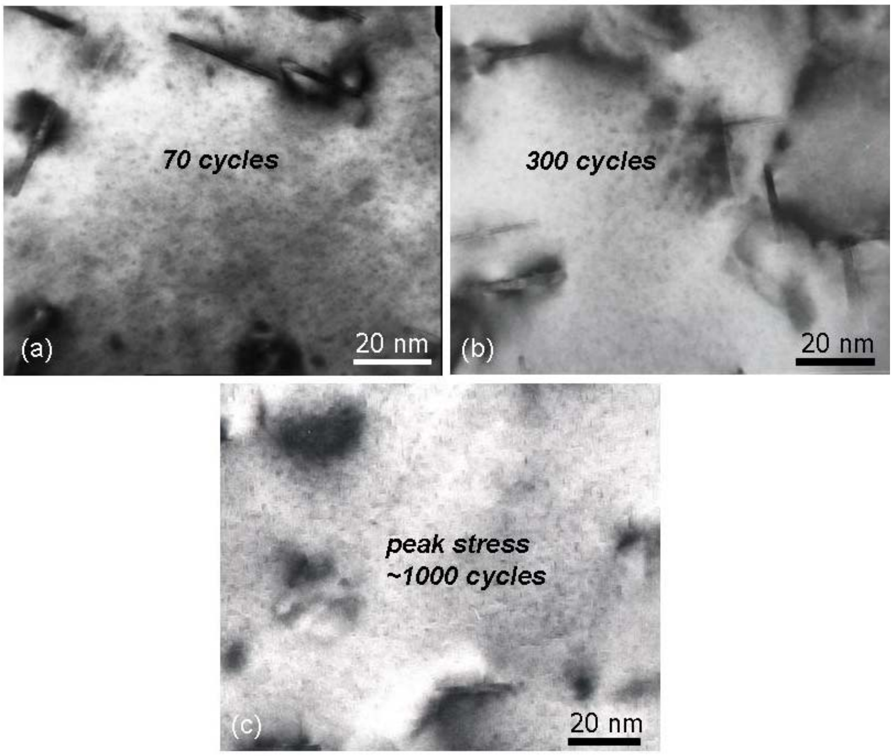

Fig. 5. The corresponding bright field TEM images of Al-4Cu-10min aged alloy after cyclic loading to (a) 70 cycles, (b) 300 cycles, and (c) peak stress state (~1000 cycles) at $\Delta\varepsilon_p/2=1\times10^{-3}$. The electron beam is close to $[001]_\alpha$. (The size and number density of GP zones increases with the number of cycles)

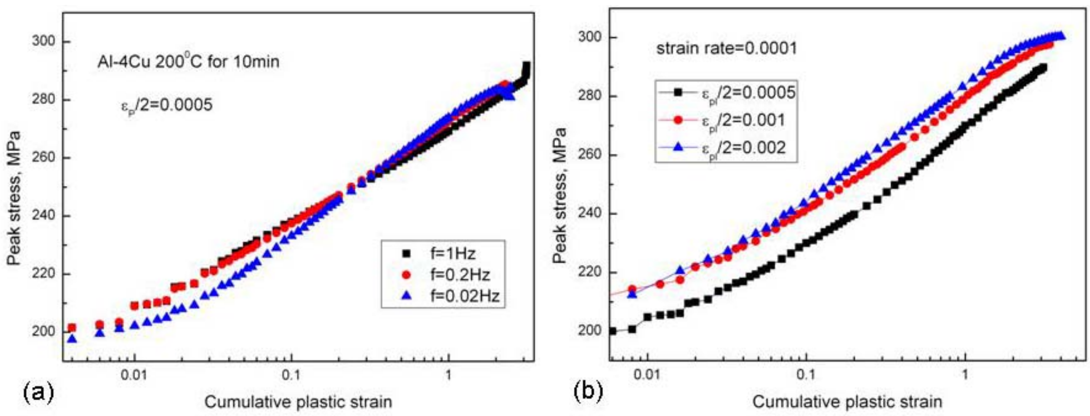

Fig. 6. (a) The cyclic stress strain curves of the 10min aged alloy deformed cyclically with different test frequencies; (b) cyclic stress strain curves of 10min alloy deformed at the same strain-rate but at different constant plastic strain amplitudes.

The production rate of deformation-induced vacancies depends not only on the applied strain-rate but also on the number of jogs on dislocations [7]. It seems plausible to expect that the number of jogs on dislocations increases with the applied plastic strain amplitude. For the larger plastic strain amplitudes, the slip distance of a mobile dislocation is longer and the chance of interacting with forest dislocations increases, resulting in greater dislocation jogs and excess vacancy production. The effect of applied plastic strain amplitude, at a constant strain-rate, is shown in Fig. 6b. It can be seen that the cyclic hardening process depends on the plastic strain amplitude and the dependence is in qualitative agreement with the expected behaviour. Further efforts are underway to establish a quantitative relationship between the amount of cyclic hardening and dynamic precipitation based on the formation of GP zones.

4. Conclusion

The cyclic stress strain behavior of an under aged Al-4Cu alloy containing θ′ precipitates shows a rapid cyclic hardening behaviour that is absent from the other aging state of the same alloy. Examination using transmission electron microscopy reveals that the rapid cyclic hardening originates from the room temperature dynamic precipitation of GP zones during fatigue deformation. The observed dynamic precipitation under cyclic loading may offer a new degree of freedom for microstructural control and materials design. It may be possible to use the service conditions to drive the microstructural changes to a state that exhibits improved fatigue properties over those that could be obtained from microstructures generated ex-service.

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