Weizhong Han¹, Alexei Vinogradov² and Christopher R Hutchinson¹

¹ARC Centre of Excellence for Design in Light Metals, Department of Materials Engineering, Monash University, Clayton, 3168 Victoria, Australia

²Department of intelligent Materials Engineering, Osaka City University, 3-3-138, Sugimoto Sumiyoshi-ku, Osaka 558-8585, Japan

A method is introduced to quantify the magnitude of the internal stress during cyclic deformation based on shape changes in the hysteresis loop. Based on this method, the evolution of the internal stress as a function of precipitate state and plastic strain amplitude has been characterized in a model Al-4Cu alloy containing only the relatively shear-resistant θ' (Al₂Cu) precipitates. It is demonstrated that with increasing plastic strain amplitude, the internal stress continually increases and the magnitude of internal stress depends sensitively on the precipitate state. Recent models for the evolution of the internal stress in these alloys during monotonic deformation are currently being extended to include slip irreversibility to describe the evolution under cyclic deformation conditions.

Keywords: slip irreversibility, internal stress, cyclic deformation, θ' precipitate state

1. Introduction

Fatigue deformation involves the repeated moving backwards and forwards of dislocations. If the dislocation motion is perfectly reversible the fatigue damage done to the material is negligible and the lifetime is not affected by fatigue loading. However, dislocation motion is not perfectly reversible and a degree of irreversibility is inherent in all cyclic deformation of metallic materials [1]. It is generally thought that the greater the degree of reversibility, the better the fatigue properties of the material will be, since less damage is accumulated in the material. One measure of the reversibility of deformation is the amount of kinematic hardening that contributes to the overall work hardening process since the internal stress is an elastic stress that is recovered during reverse loading [2]. Therefore, in general, the higher the internal stress the greater the slip reversibility.

The kinematic hardening behaviour in monotonic deformation has been investigated heavily in the past few decades, and a general understanding has now been achieved, e.g. [3-7]. Building on prior works, da Costa Teixeira *et al.* [7] have recently investigated the effect of precipitate state on the magnitude of kinematic hardening in an Al-3Cu alloy containing shear-resistant θ' precipitates and showed that the precipitate state has a marked influence on the kinematic hardening behaviour. The magnitude of the internal stress is significant even for short ageing times (low volume fractions of θ' precipitates) and increases with ageing time, consistent with the increase in precipitate volume fraction. For all ageing durations, the evolution of the internal stress has two components: a stage of increase corresponding to the elastic loading of the precipitates, followed by a plateau which is thought to have its origin in plastic relaxation mechanisms. Based on those experimental results, the opportunity now exists to extend this work to attempt to understand quantitatively the evolution of the internal stress during cyclic loading with a view to better understanding the fatigue response of precipitation hardened Al alloys.

2. Experimental design and procedures

An alloy of composition Al-4Cu (wt.%) was selected for this study. This alloy offers a number of advantages that make it suitable for a study of the effect of precipitate state on the internal stress evolution during cyclic loading: i) a significant volume fraction of relatively shear-resistant

precipitates (θ') can be formed and these are the main source of kinematic hardening, ii) the precipitation process in Al-4Cu is sufficiently well understood that one can produce a microstructure containing only θ' precipitates but with different sizes and number densities, and iii) the kinematic hardening behaviour in a very similar alloy (Al-3Cu) during monotonic deformation has recently been studied in detail [7]. This is considered a pre-requisite for investigating the hardening process during cyclic deformation.

The fatigue samples of Al-4Cu alloy with gauge dimensions of $4\text{mm} \times 5\text{mm} \times 16\text{mm}$ were solution treated at 520°C for 1h, quenched into water and then aged at 200°C 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. Before the mechanical testing, the surfaces of all samples were carefully polished. The mechanical properties during monotonic deformation were characterized using tensile tests and the yield stress, ultimate tensile stress (UTS) and the uniform elongation are plotted as a function of aging time at 200°C in Fig. 1. With aging time, the yield stress of Al-4Cu alloy containing θ' precipitates continually increases, and reaches the highest yield strength of 300MPa after 1.5h, after which it decreases due to overaging (Fig. 1a). The UTS and uniform elongation of Al-4Cu alloy also change significantly with aging time, as shown in Fig 1b. The UTS has a similar trend to the yield strength. The uniform elongation continually decreases to the peak aged state, and then remains relatively constant with further aging.



Fig. 1. (a) The variation of yield stress of Al-4Cu alloy with aging time at 200°C; (b) The variation of UTS and uniform elongation of Al-4Cu alloy with aging time. At this aging temperature, only θ' precipitates are formed. (The states selected for fatigue testing are highlighted in blue in (a))

In order to study the effect of precipitate state on the kinematic hardening during cyclic deformation, five aging states were selected to conduct fatigue tests: aging for 10min (under aged), 30min (under aged), 1.5h (peak aged), 8h (over aged) and 4 days (over aged). In these states, the 10min and 4 day samples have similar yield strengths and UTS, but different uniform elongations. This is also the case for the 30min and 8h samples. The peak aged alloy (1.5h) and over aged alloy (8h and 4D) have similar uniform elongations, but different yield strengths and UTS.

The cyclic deformation tests were conducted in constant plastic strain amplitude mode, with five strain amplitudes: $\Delta \varepsilon_{pl} / 2 = 1 \times 10^{-4}$, 5×10^{-4} , 1×10^{-3} , 2×10^{-3} and 3×10^{-3} . The fatigue tests were conducted using an MTS-858 table-top system at room temperature with a frequency, f = 0.2Hz. Before fatigue testing, the microstructures of the as-aged states of the Al-4Cu alloy were characterized by TEM. Figure 2 shows an example of the precipitate states formed after aging at 200°C for 10min (under aged) and 4 days (over aged). It can be seen that the size of θ' precipitate varies greatly with aging time.



Fig. 2. The microstructure of as-aged (i.e. without deformation) Al-4Cu alloy: (a) Bright field TEM micrograph of sample aged for 10min; (b) Bright field TEM micrograph of sample aged for 4 days. In both cases the electron beam is close to [001]_α. θ' precipitates are formed on the {100}_α planes.

3. The method to quantify internal stress for cyclic deformation

During monotonic deformation, the internal stress is often measured using a Bauschinger test, e.g. [5-7]. During cyclic deformation, a number of different procedures have been proposed [8-11]. A quantitative analysis of the cyclic hardening/softening behaviour can be obtained by using the scheme suggested by Cottrell [11]. This scheme provides estimates of the friction stress, σ_f , and the effective back stress, σ_b , and it is often used to analyse plastic strain controlled fatigue data (c.f. [9]). However, the trick is in the experimental evaluation of the hysteresis yield stress σ_y , which is not straightforward. Aiming at minimising the experimental errors associated with σ_y assessment, we have modified the Cottrell's scheme to quantify reliably the internal stress in the alloys under investigation.

Consider a hypothetical metal with perfectly linear elastic and plastic deformation behaviour. During deformation, the material will initially deform elastically (line O-A, Fig. 3a) until the yield point A, after which it will deform plastically (line A-B, Fig. 3a). During reverse deformation, the material will unload along the line B-C but due to the internal stress generated during the forward deformation (which now aids reverse flow), yielding will occur at point C, which is a lower absolute stress than point D (i.e. D and A represent the same absolute stress level). Further compressive deformation follows the line C-E and at point E the contribution to the stress from the internal stress generated during the initial forward deformation has reduced to zero. The strain difference between points D and E will be referred to as the Bauschinger strain. Continued deformation will follow the line E-F and at point F the direction of straining is reversed again. The overall steady-state cyclic stress strain curve for this hypothetical material therefore consists of the curves: F-G-I-B-C-E-F. According to the analysis, in order to measure the internal stress from a real hysteresis loop, we need only identify the three lines (F-G, G-I and I-B) that best represent half of the hysteresis loop. The value of the internal stress and Bauschinger strain can then be calculated from their intersection, Fig. 3b. Figure 3b is a real hysteresis loop from the present fatigue test.

The three lines shown in Fig. 3b can be readily identified. Lines 1 and 2 are simply the tangents to the unloading (equal to the modulus of the alloy) and loading portions of the hysteresis loop. Line 3 is chosen such that the difference between the area of the real hysteresis loop and the one bounded by the three lines is minimized. Once these three lines have been identified the internal stress can be evaluated as: $\langle \sigma \rangle = (\sigma_H - \sigma_G)/2$, where σ_H and σ_G are the stresses at points G and H, respectively. To obtain an estimate of the reasonableness of approximating a real hysteresis loop by three straight lines the difference in the areas of the real loops and those represented by the three straight lines were

compared. In all cases considered in this work, the relative error is less than 2%, suggesting that approximating the real hysteresis loops by three strain lines is reasonable. Certainly this will not be true for the hysteresis loops from all materials. However, in this study the material contains only relatively shear-resistant θ' precipitates and it is known from previous investigations under monotonic loading that the hardening at small plastic strains is dominated by kinematic hardening (due to the elastic loading of the θ' precipitates by Orowan loops) and this tends to be linear in strain [7]. This is one of the reasons why the approach of fitting three lines works well in this case.



Fig. 3. (a) Schematic illustration of the method to quantify the internal stress from a hysteresis loop from a hypothetical metal exhibiting perfectly linear elastic and plastic behaviour; (b) Illustration of the fitting of three lines on a real hysteresis loop.

4. Results and discussion

Using the method shown in Fig. 3b, the internal stress evolution in the Al-4Cu alloy was measured as a function of precipitate state and applied plastic strain amplitude. Figure 4 shows the evolution of the internal stress in samples aged at 200°C for 30min, 1.5h, 8h and 4 days as a function of cumulative plastic strain. The measured internal stresses are all below 100MPa and are comparable to the data acquired in Bauschinger tests for monotonic deformation in this alloy system [7]. With increasing cumulative plastic strain, the internal stress reaches a relatively steady value for all precipitates states and imposed plastic strain amplitudes. The internal stress increases with increasing applied plastic strain amplitude for all precipitate states.

In order to compare the effect of precipitate state on the magnitude of internal stress, the plateau value of the internal stress for each aging state from Fig. 4 is summarized as a function of plastic strain amplitude in Fig. 5. It can be clearly seen that the measured internal stress depends sensitively on the θ' precipitate state. In general, the 1.5h alloy shows the highest internal stress, the 4 day alloy has the lowest internal stress, and the internal stress of 8h alloy is higher than the 30min alloy. It seems that the peak-aged state has the most obvious Bauschinger effect in cyclic deformation. In addition, the increase of internal stress with imposed plastic strain amplitudes can be divided into a quick stage and a slow stage at $\Delta \varepsilon_{pl} / 2 = 1 \times 10^{-3}$. The Al-4Cu-10min alloy shows an obvious rapid cyclic hardening during cyclic deformation that is due to dynamic precipitation [11], and due to the complexity of this case, the results of the Al-4Cu-10min alloy are not included in the comparison.

The variation of the internal stress with precipitate state and plastic strain amplitude can be qualitative understood by invoking the concept of slip irreversibility. The internal stress originates from the dislocation-precipitate interaction. During plastic deformation, Orowan loops are formed on the θ' precipitates and these load the matrix in the reverse direction giving a long-range internal stress [2-7]. During reverse loading, the recently formed Orowan loops have a certain probability to be removed by the gliding dislocations. The percentage of the Orowan loops that are removed is a

measure of the magnitude of slip reversibility. Compared to monotonic deformation, the dislocation motion during cyclic slip involves repeated backwards and forwards movement. Hence, the slip reversibility has a major influence on the internal stress development in cyclic deformation. The value of slip irreversibility is determined by the microstructures and the deformation condition [1].



Fig. 4. The measured internal stress for different precipitate states of the Al-4Cu alloy as a function of cumulative plastic strain for a range of applied plastic strain amplitudes.

For each of the precipitates states shown in Fig. 4, the internal stress increases with increasing applied plastic strain amplitude. This can be qualitatively understood by considering the average glide distance of a mobile dislocation. For greater plastic strain amplitudes, the distance traversed by a dislocation is greater and the chance of intersecting a precipitate and forming an Orowan loop (which gives rise to the internal stress) is enhanced. For greater plastic strain amplitudes, more Orowan loops are stored in each forward or reverse deformation and therefore a greater internal stress is developed.

The probability that a gliding dislocation will intersect a precipitate and form an Orowan loop also depends on the sizes of the precipitates (normal to the slip plane) and their spacing (which is related to the number density). This probability is non-monotonic with aging time. In this alloy, for aging times up until the peak yield stress the number density of θ' precipitates is relatively constant and it is mostly their size that increases. The probability of dislocation intersection with a precipitate during this regime of precipitate evolution increases with aging time. During over aging, the precipitate size increases with a concomitant decrease in the number density. The increase in size and decrease in number density have antagonistic effects on the probability of dislocation. The net result is a non-monotonic evolution of Orowan loop formation probability, and therefore the internal stress, as a function of aging time for a given plastic strain amplitude. This can be clearly seen in Fig. 4, for example, for the plastic strain amplitude of $\Delta \varepsilon_{pl}/2 = 5 \times 10^{-4}$. For the 30min aged sample, the internal stress is ~50MPa. In the 1.5h peak aged state it increases to ~65MPa. After 8h aging, the internal stress decreases to ~50MPa and then further decreases to ~25MPa for the 4 day sample. The recently developed quantitative model for the dependence of the internal stress during monotonic

loading [7] is currently being extended for application to the cyclic deformation case considered above.



Fig. 5. The internal stress variation with constant plastic strain amplitude for each of the aging states considered.

5. Conclusion

An approach to quantify the internal stress development during cyclic deformation based on the change in shape of the hysteresis loops is outlined. The evolution of the internal stress as a function of precipitate state and plastic strain amplitude has been studied in a model Al-4Cu alloy. It is demonstrated that with increasing plastic strain amplitude, the internal stress continually increases and the magnitude of internal stress depends sensitively on the precipitate state. The corresponding experimental observation can be qualitatively understood by invoking the concept of slip irreversibility.

Acknowledgement

This work is supported by the Australian Research Council (ARC) through the ARC Centre of Excellence for Design in Light Metals. CRH gratefully acknowledges the support of the ARC through the award of a Future Fellowship.

References

- [1] H. Mughrabi: Metall. Mater. Trans. A 40 (2009) 1257-1279.
- [2] L. M. Brown, W. M. Stobbs: Philos. Mag. 23 (1971) 1185-1199.
- [3] A. Abel, R. K. Ham: Acta Metall. 14 (1966) 1489-1494.
- [4] L. M. Brown, D. P. Clarke: Acta Metall. 23 (1975) 821-830.
- [5] G. D. Moan, J. D. Embury: Acta Metall. 27 (1979) 903-914.
- [6] H. Proudhon, W. J. Poole, X. Wang, Y. Brechet: Philos. Mag. 88 (2008) 621-640.
- [7] J. da Costa Teixeira, L. Bourgeois, C. W. Sinclair, C. R. Hutchinson: Acta Mater. 57 (2009) 6075-6089.
- [8] A. Abel, H. Muir: Philos. Mag. 27 (1973) 489-504.
- [9] D. Kuhlmann-Wilsdorf and C. Laird: Mater. Sci. Eng. 37 (1979) 111-120.
- [10] K. Tanaka, S Matsuoka: J Mater. Sci. 11 (1976) 656-664.
- [11] A. H. Cottrell, in: Dislocations and Plastic Flow in Crystals Oxford University Press, London (1953) p. 111-132.
- [12] W. Z. Han, A. Vinogradov, C. R. Hutchinsion: elsewhere in the Proceedings of the 12th International Conference on Aluminium Alloys, Yokohama, Japan, 2010.