Fatigue behavior and microstructure of an Al-Mg-Sc alloy at an elevated temperature

C Watanabe and R Monzen
Division of Innovative Technology and Science, Kanazawa University, Kakumamachi, Kanazawa 920-1192, Ishikawa, Japan

chihiro@t.kanazawa-u.ac.jp

Abstract. Al-Mg-Sc alloy polycrystals bearing Al$_3$Sc particles with different sizes, i.e. 4, 6 and 11 nm in diameter, have been cyclically deformed at 423 K under constant plastic-strain amplitudes, and the microstructural evolution has been investigated in relation to the stress-strain response. Cyclic softening after initial hardening is found in specimens with small particles of 4 and 6 nm, but no cyclic softening takes place in specimens with larger particles of 11 nm. These features of cyclic deformation behavior are similar to the results previously obtained at room temperature. Transmission electron microscopy observations reveal that dislocations are uniformly distributed under all applied strain amplitudes in the specimens containing large particles of 11 nm, whereas slip bands are formed in the cyclically softened specimens bearing smaller particles. The cyclic softening is explained by a loss of particle strength through particle shearing within strongly strained slip bands. The 6 and 11 nm Al$_3$Sc particles have a stronger retardation effect on the formation of fatigue-induced stable dislocation structure than 4 nm particles at 423 K.

1. Introduction
The addition of a small amount of Scandium (Sc) to Al-Mg alloys causes a significant increase in the strength of the alloys, due to the existence of coherent, finely dispersed L1$_2$ Al$_3$Sc precipitate particles [1]. Also the Al$_3$Sc particles have modest coarsening rates at elevated temperatures, leading to the effective suppression of recrystallization and the stabilization of microstructures at high temperatures. Thus Sc-containing Al-Mg alloys are expected to be used in higher-temperature application compared to conventional structural Al alloys. Although some experiments have been performed on the mechanical properties of Al-Mg-Sc ternary alloys at elevated temperatures [1], there are no investigations of the high-temperature fatigue properties of these alloys, to the best of my knowledge.

In previous studies [2, 3], we examined the cyclic deformation behavior and dislocation microstructure under plastic-strain-controlled and stress-amplitude-controlled conditions at room temperature (RT), using an aged Al-Mg-Sc alloy with Al$_3$Sc particles of different diameters, i.e. 4, 6 and 11 nm, which correspond to under-age, peak-age and over-age conditions, respectively. The over-aged alloy showed cyclic hardening to saturation despite the fact that the Al$_3$Sc particles were coherent. On the other hand, cyclic softening occurred in the under-aged and peak-aged alloys.

In this work, a polycrystalline Al-1wt%Mg-0.27wt%Sc alloy bearing Al$_3$Sc particles with different sizes has been cyclically deformed at 423 K under various constant plastic-strain amplitudes, and the microstructural evolution has been investigated in relation to the stress-strain response. The results
have also been compared with previous results obtained at room temperature. As in the previous studies [2, 3], we have selected three particle diameters, i.e. 4, 6 and 11 nm.

2. Experimental Procedure
Specimens for fatigue tests were cut from hot-rolled polycrystalline Al-1wt%Mg-0.27wt%Sc alloy plates. These specimens had the stress axis parallel to the rolling direction. The specimens were solutionized at 905 K for $7.2 \times 10^3$ s and water quenched. Transmission electron microscopy (TEM) observations revealed that no precipitates existed in the solution treated specimens. To obtain spherical and coherent Al$_3$Sc particles, one set of solutionized specimens was aged at 573 K for $9.0 \times 10^2$ s, second set was aged at 573 K for $7.2 \times 10^3$, and third set was aged at 623 K for $6.48 \times 10^4$ s, corresponding to under-ageing, peak-ageing and over-ageing conditions, respectively. Hereafter, these under-aged, peak-aged and over-aged specimens will be referred to as specimens UA, PA and OA, respectively. The average diameter of Al$_3$Sc particles were 4 nm for specimen UA, 6 nm for specimen PA and 11 nm for specimen OA. The average size was determined from over 200 particles for each taken by TEM. Judging from the Al-Sc equilibrium phase diagram [4], the volume fractions of the Al$_3$Sc particles in specimens UA, PA and OA are nearly identical, 0.007.

All mechanical tests were carried out at 423 K in air. Tensile properties were determined with an initial strain rate of $3.0 \times 10^{-3}$ s$^{-1}$. Fully-reversed tension-compression fatigue tests were performed under plastic-strain-amplitude control using a triangular command signal with a constant strain rate of $10^{-3}$ s$^{-1}$.

The fatigued specimens were sliced into 3 mm disks parallel to the stress axis and were mechanically ground down to 0.2 mm. Thin foils for TEM observations were prepared by electropolishing. Microscopy was performed with JOEL-2000EX and JOEL-2010FEF microscopes operating at 200 kV.

3. Results & Discussion
Table 1 summarizes the tensile properties of specimens UA, PA and OA tested at 423 K. For comparison, the results obtained at RT are also indicated. At 423 K, the values of 0.2% proof stress of the specimens UA and OA are almost the same and smaller than that of the specimen PA. The values of 0.2% proof stress and tensile stress of each specimen are smaller than those obtained at RT, while the values of fracture strain of each specimen show a reverse tendency.

Figure 1 shows cyclic-hardening curves of specimens containing Al$_3$Sc particles of 4, 6 and 11 nm, tested under a plastic-strain amplitude of $1.0 \times 10^{-3}$ at 423 K. The data obtained at RT are also indicated. The stress amplitude $\sigma$ is plotted against the cumulative plastic-strain $4N\epsilon_{pl}$, where $N$ is the number of fatigue cycles and $\epsilon_{pl}$ is the applied plastic-strain amplitude. The specimen OA exhibited a cyclic hardening to saturation under all applied plastic strain amplitudes, while in the specimens UA and PA a cyclic softening was observed after initial hardening. This result is similar to that obtained at RT [2], but the level of stress amplitudes at 423 K is lower than that at RT.

| Specimen | 0.2% proof stress (MPa) | Tensile stress (MPa) | Fracture elongation (%) |
|----------|------------------------|----------------------|-------------------------|
|          | 423 K | RT | 423 K | RT | 423 K | RT |
| UA       | 127   | 130 | 150   | 170 | 24    | 21 |
| PA       | 150   | 168 | 167   | 213 | 30    | 24 |
| OA       | 121   | 125 | 147   | 174 | 27    | 21 |
Figures 2(a), (b) and (c) depict the typical fatigue dislocation microstructures formed at 423K under $\varepsilon_{pl} = 1.0 \times 10^{-3}$ for specimens OA, UA and PA. In the specimen OA, dislocations were uniformly distributed under all applied plastic strain amplitudes, whereas both the specimens UA and PA, in which cyclic softening occurred, exhibited clearly developed slip bands along the trace of primary slip plane. These slip bands indicate the occurrence of very inhomogeneous deformation. In other words, strong strain localization took place within the slip bands. Such strong strain localization usually causes the destruction and re-dissolution of particles [3]. To check whether shearing of Al$_3$Sc particles occurred during fatigue tests at 423 K, the average size of the Al$_3$Sc particles within slip bands or in the matrix was measured by TEM using a specimen PA re-aged at 623K for $6.48 \times 10^4$ s following a fatigue test to failure. The resulting average diameters were about 9 nm within the slip bands and 12 nm in the matrix. This discrepancy in the particle size implies that the small Al$_3$Sc particles of 6 nm were cut by moving dislocation within the strongly strained slip bands. The cyclic softening in specimens UA and PA in figure 1 can then be explained by a decrease in particle strengthening through particle shearing or re-dissolution within the slip bands.

**Figure 1.** Cyclic-hardening curves of specimens UA, PA and OA, fatigued under a plastic-strain amplitude of $1.0 \times 10^{-3}$ at 423K. Also shown are the data obtained at RT [2].

**Figure 2.** Dislocation structures in specimens (a) OA, (b) UA and (c) PA, fatigued under $\varepsilon_{pl} = 1.0 \times 10^{-3}$ until failure at 423K. The inset in (a) is an enlarged dark-field weak beam image. The stress axis is indicated by an arrow.
It is known that second-phase particles act as barriers against the formation and development of fatigue-induced stable dislocation structure by preventing the dislocation motion and re-arrangement during cyclic deformation [5]. Since the inter-particle spacing decreases with decreasing particles size, smaller particles have more significant retardation effect on the development of the dislocation structure if volume fraction of the particles is fixed [5]. All the specimens fatigued at RT showed no geometrically regular dislocation arrangements [2], and the relatively uniform dislocation distribution were observed except the slip bands. This result shows the effectiveness of Al₃Sc particles in preventing formation of the fatigue dislocation structures at RT. However, as shown in figure 2(b), not only the slip band but also the labyrinth structure was observed in the specimens UA fatigued at 423 K. Moreover, so-called cell structure was formed with the slip bands in a specimen UA fatigued under a high plastic-strain amplitude of $1.0 \times 10^{-2}$. On the other hands, the specimens PA and OA fatigued under all applied plastic-strain amplitudes exhibit no typical fatigue dislocation structures such as labyrinth or cell structure formed in the UA specimen. This indicates that dislocation re-arrangement is more difficult in the specimen PA and OA at 423 K despite the fact that the inter-particle spacings are larger than that in the specimen UA. In the specimen UA, moving dislocations may get over relatively easily small Al₃Sc particles by climb motion and cross slip at the high temperature of 423 K, and then re-arrange themselves to form stable structures. On the other hand, larger Al₃Sc particle in the specimens PA and OA act as effective obstacles to moving dislocations even at 423 K, and thus a relatively uniform dislocation distribution is formed.

It should be noted in figure 1 that, at 423 K, the stress level of the specimen UA is smaller than that of the specimen OA, although those of both the specimens are almost the same at RT. Since recovery seems to easily take place in the specimen UA fatigued at 423 K as mentioned above, the dislocation density in the specimen UA may be lower than that in the specimen OA. This may result in the lower stress level of the specimen UA.

4. Summary
Plastic-strain controlled low-cycle fatigue tests of Al-Mg-Sc alloy polycrystals with Al₃Sc particles of 4, 6 and 11 nm in diameter were performed at 423 K. The results and conclusions are summarized as follows.

(1) Specimens UA and PA bearing 4 and 6 nm Al₃Sc particles show cyclic softening, while specimen OA with 11 nm Al₃Sc particles show cyclic hardening to saturation.
(2) In the specimens UA and PA, slip band dislocation structures are observed, and, in the specimen OA, dislocations are uniformly distributed.
(3) Fatigue behaviors and dislocation structures are similar to those obtained at room temperature [3]. However, the stress levels tend to decrease at 423 K.
(4) The cyclic softening of specimens UA and PA are caused by shearing of small Al₃Sc particles within the slip bands.
(5) The Al₃Sc particles larger than 6 nm act as strong obstacles against the dislocation motion and re-arrangement, leading to the suppression of fatigue-induced dislocation structures at 423 K.

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