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EFFECT OF PRECIPITATION HARDENING ON SERRATED FLOW CHARACTERISTICS IN AN Al-Li ALLOY①

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ABSTRACT

The serrated flow characteristics of an 8090 type Al-Li alloy during tensile testing and the effect of age hardening on serrated flow were inverstigated. The results indicated that in the as-quenched and under-aged conditions the serrated flow occurred and in the existence of serrated flow negative strain rate sensitivity was observed. In the peak and over-aged conditions serrations were not exhibited. In addition, the predeformation prior to aging and the increase in strain rate suppressed the occurrence of serrated flow. The results are discussed in terms of the interactions of the solute atoms, dislocations and the precipitates with the mobile dislocations.

Key words: serrated flow strain aging Al-Li alloys age hardening

1 INTRODUCTION

It is well known that many alloy systems exhibit serrations on stress-strain curves in a certain range of temperature and strain rates. This phenomenon has been termed the Portevin-Le Chatelier effect. In general, the occurrence of serrated flow is explained using Cottrell' s model of solute atmospheres or modified models[1-3]. Serrated flow in Al-Li based alloys was also observed during tensile testing. However, the theoretical explanations of this effect in the Al-Li alloys are disputable. For example, Tamura *et al* reported that in the single crystal of Al-Li alloy, the serrated flow was attributed to the work softening associated with dislocation cutting of the δ′ particles[4]. Wert and Wycliffe have suggested that the serrated flow at high strains in the underaged Al-Li-Cu-Mg-Zr alloys is primarily a result of copper and magnesium in solid solution[5]. Gregson *et al.* have considered that the serrated flow is attributed to be rapid release of dislocations pinned by an atmosphere of Li, but not Cu and Mg atoms[6]. Recently, Welpmann *et al.* have suggested that the serrated flow is to be associated with the formation of GPB[7]. In additon, during the tensile testing of Al-Li alloys negative strain rate sensitivity was also observed[8,9], which seems to be always a prerequisite condition for serration. In the present work, the effect of precipitation hardening on the serrated flow phenomenon in an 8090 type Al-Li alloy was investigated. The strain rate dependence of flow stress at the occurrence of serrated flow was also studied.

2 EXPERIMENTAL METHOD

The material used in this investigation was an 8090 type Al-Li alloy in the form of a 2 mm-thick sheet. The chemical compositions were Al—2.70 Li—1.20 Cu—0.90 Mg—0.14 Zr(wt.-%). The specimens were solution treated in a salt bath at 530 ℃ for 40 min. and quenched into ice-water. After quenching some specimens were aged in an oil bath at 190 ℃ for varying times immediately, others were cold rolled by 5% prior to aging at 190 ℃ for varying times. The test pieces were ma-

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chined from the longitudinal direction of the sheet and electropolished after aging. The tensile tests were carried out using an Instron testing machine with nominal strain rate of $4.2\times10^{-3}\,s^{-1}\sim4.2\times10^{-5}\,s^{-1}$ at room temperature. The load-displacement curves of the specimens tested were recorded by a x-y recorder. The foils for TEM examination were prepared using a twin-jet electron polishing technique in a solution of 33% HNO_3+67% methanol at -20 ℃ and DC voltage of 12 V. TEM observations were carried out using H800 with an accelerating voltage of 200 kV.

3 RESULTS

3. 1 *Effect of Aging on Serrated Flow*

Fig. 1 shows the stress-strain curves of the specimens aged at 190 ℃ for various times at a strain rate of $4.2\times10^{-4}\,s^{-1}$. It can be seen that the as-quenched specimen exhibited the fine serrations. The stress drop of serrations (e. g. serration amplitude $\Delta\sigma$) was smaller, but the frequency of serrated flow was higher. With increasing aging times up to 4 h, the serrated flow became more severe, the serration amplitude $\Delta\sigma$ increased, and the frequency slightly decreased. More long aging times resulted in serrations to become less severe. At the peak aged condition (aged for 16 h) the serrations disappeared. The stress-strain curves of the over-aged specimens did not exhibit any serrations. From stress-strain curves it is also seen that the critical strain ε_c at the onset of serrated flow increased with increasing aging.

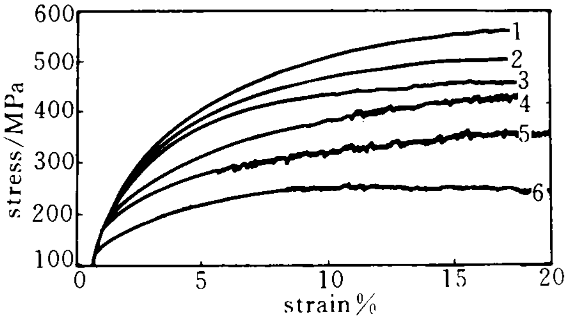

Fig. 1 The stress-strain curves of the specimens aged at 190 ℃ for various times

1—16 h; 2—32 h; 3—8 h;
4—4 h; 5—2 h; 6—as-quenched

The serration amplitude was smaller and the frequency was larger at the initial stages of the serrated flow, with increasing strain the serration amplitude increased at first and then became constant. The effect of aging on the critical strain ε_c for the onset of serrated flow and the serration amplitude $\Delta\sigma$ at the constant stages of serrated flow are shown in Fig. 2.

3. 2 *Effect of Predeformation Prior to Aging on Serrated Flow*

For the specimens cold-rolled by 5% prior to aging and then aged at 190 ℃, the serrations were less severe compared to that of the specimens which were aged for same times at 190 ℃ without predeformation. Predeformation prior to aging resulted in an increase in the critical strain and a decrease in the serration amplitude. In addition, the aging times for the serrations to disappear were shortened. Fig. 3 shows the effect of predeformation before aging on the characteristics of serrated flow. This implies that predeformation prior to aging sup-

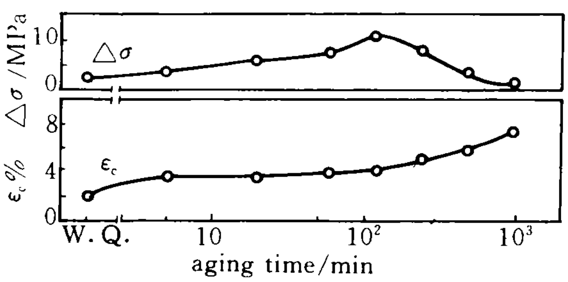

Fig. 2 The variation of the critical strain ε_c and the serration amplitude $\Delta\sigma$ with aging times

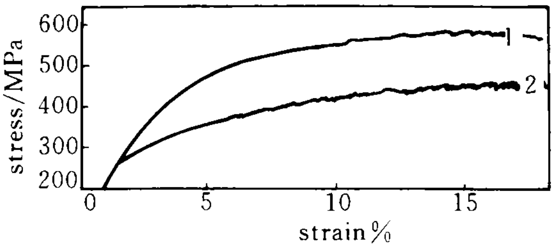

Fig. 3 Effect of predeformation prior to aging on the serrated flow

1—cold rolled by 5% and then aged at 190 ℃ for 4 h;
2—aged at 190 ℃ for 4 h

presses the occurrence of serrated flow during tensile testing.

3.3 *Effect of Strain Rate on Serrated Flow*

Fig. 4 shows the stress-strain curves of the specimens aged at 190℃ for 2h at different strain rates. From this it can be seen that at a slow strain rate the serrations were very obvious, the critical strain for the onset of serrated flow was smaller and the serration amplitude was larger. With increasing strain rate the critical strain was increased and the serration amplitude was also decreased. At a high strain rate ($4.2\times10^{-3}s^{-1}$), the stress strain curve was almost smooth, only a few serrations were visible. This indicates that the increase in strain rate suppresses the occurrence of serrated flow. These results are agreement with the results observed in the some Li-free alloys[10,11].

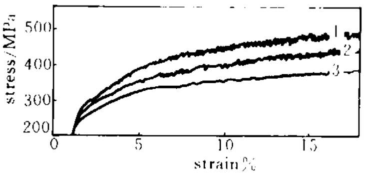

Fig. 4 Effect of strain rate on serrated flow
1—$4.2\times10^{-5}s^{-1}$; 2—$4.2\times10^{-4}s^{-1}$; 3—$4.2\times10^{-3}s^{-1}$

3.4 *Deformation Structure*

Fig. 5 shows the dislocation structure developed in a specimen during tensile deformation within the region of serrated flow. From this it can be seen that except some superdislocations, wavy single dislocations were present. The bowing of dislocations around the precipitates were also observed. This indicates that during serrated flow the dislocations move in the two ways. Some dislocations move in pairs by cutting through coherent Ll2 type δ′ precipitates, others move by passing the δ′ precipitates by the Orowan mechanism. TEM observations also indicated that δ′ particles cut by superdislocations were present in the over-aged specimens which did not exhibit any serrated flow during tensile testing (See Fig. 6). These results indicated that the occurrence of serrated folw in the Al-Li alloys tested appears not to be the results of the work softening caused by cutting of δ′ particles by superdislocations.

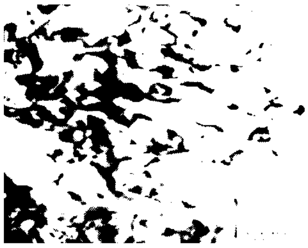

Fig. 5 The dislocation structure of the specimen when the serrared flow was present

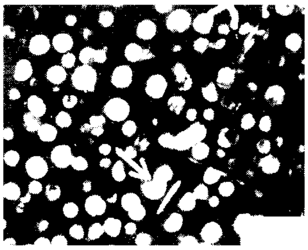

Fig. 6 Showing the δ′ particles cut by dislocations for over aged specimen

3.5 *The Strain Rate Dependence of Flow Stress During Serrated Flow*

In order to determine the strain rate dependence of flow stress during serrated flow, the strain rate change tests were carried out. During testing the strain rate was cycled between $4.2\times10^{-4}s^{-1}$ and $4.2\times10^{-3}s^{-1}$. Fig. 7 shows the results of strain-rate change tests for the specimens aged for various times at 190℃. From this it can be seen that when the strain rate increased, the sudden load drops were observed and then deformation was continued at a smaller stress level accompanying the decrease in serration amplitude. In contrast, when the strain rate decreased, the increase in the flow stress and a more large working hardening rate were observed. Here the serration amplitude increased and the frequency decreased. In addition, with increasing aging times these variations became smaller. In the over-aged condition at which serrated flow did not occur these variations were almost zero, the stress-strain curves became smooth during strain-rate change testing. These results indicated that negative strain-rate sensitivity is a characteristic feature of the alloy tested under the conditions producing serrated flow. This agrees with the results reported by others[8,9].

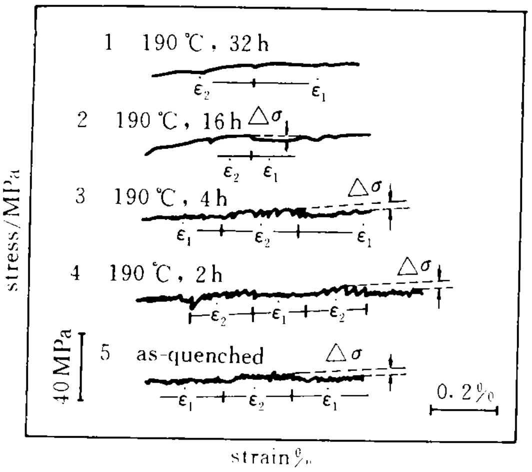

Fig. 7 The characteristics of the stress-strain curves of the specimens aged for various times during strain-rate change testing

1—32 h; 2—16 h; 3—4 h; 4—2 h; 5 as-quenched

$\dot{\varepsilon}_1 = 4.2 \times 10^{-4}\ \mathrm{s}^{-1}$; $\dot{\varepsilon}_2 = 4.2 \times 10^{-5}\ \mathrm{s}^{-1}$

4 DISCUSSION

The theoretical explanation for the phenomenon of serrated flow is based on the interaction between moving dislocations and diffusing solute atoms. In terms of the model developed by McCormick[12,13], it is assumed that the onset of serrated flow is attributed to the interaction of solute atoms and mobile dislocations temporarily arrested at obstacles in their slip path. During deformation, a mobile dislocation spends most of its time trying to surpass obstacles. The interaction between mobile dislocations and diffusing solute atoms mainly occurs duing the time the dislocation is waiting in front of the obstacles. When the average dislocation arrest time, t_w, is equal to the time, t_a, required to lock the arrested dislocation, aging of the dislocation occurs. The critical strain, ε_c, at the onset of serrated flow is expressed as[13]

$$\varepsilon_c = \left\{ \left(\frac{c_1 - c_0}{\alpha c_0} \right)^{\frac{3}{2}} \times \left[\frac{\dot{\varepsilon} \kappa T b}{L N K U_m D_0 \exp(-Q_m / KT)} \right] \right\}^{\frac{1}{m+\beta}} \qquad (1)$$

where C_l is the local solute concentration at the arrested dislocations required for locking; Co is the matrix solute concentration of the alloy; U_m is the solute-dislocation binding energy; Q_m is the activation energy for solute migration; K and m are constants in the vacancy concentration-strain relation $Cv = K\varepsilon^m$; N and β are constants in the mobile dislocation density-strain relation $\rho = N\varepsilon^{\beta}$; L is the average distance between obstacles; and D_0 is the diffusion frequency factor, $\alpha = 3$.

The effect of aging on the onset of serrated flow appears to be primarily due to changes in Co, L and ρ accompanying precipitation[12,13]. For the as-quenched specimens, solute concentration C_o in the matrix is larger compared to the aged specimens, thus from equation 1 it can be seen that ε_c will be smaller. During aging δ' particles were precipitated from the matrix. This resulted in the decrease in the Co, so, ε_c increased with increasing aging. In the over-aged condition due to accompanying the growth of δ' particles and the formation of the equilibrium phase sufficiently large decrease in C_o would cause ε_c to increase to beyond the fracture strain, thus serrated flow was not observed.

Serration amplitude $\Delta\sigma$ is proportional to the number of solute atoms which diffuse to a dislocation and the density of the aged dislocations[11,15]. The increase in vacancy concentration and mobile dislocation density with increasing strain results in the mobile dislocations becoming effectively strain aged. During the initial stages of serrated flow the concentration of the vacancies produced by deformation is lower, the solute atoms to diffuse to the dislocation and the aged dislocation density would be smaller, so, serration amplitude is smaller. With increasing strain, it would be expected that the diffusion of solute atoms becomes accelerated due to increasing vacancy concentration. This would result in the formation of a sufficiently strengthening atmosphere and large aged dislocation density, thus a sufficiently large external stress is requied to surpass obstacle. Once the obstacle is surpassed, the dislocation jumps at a high velocity to the next obstacle under a smaller level of flow stress. This implies that a large stress drop is present, e. g. $\triangle\sigma$ increases with increasing strain. However, with further increasing strain as a result of the competition between the increase in vacancy concentration and the depletion of solute atoms for locking dislocation the value of $\triangle\sigma$ becomes constant.

The fact that during the initial stages of aging (2 to 4 h) the serration amplitude was larger than that of the as-quenched specimens could not be explained using the changes in solute concentration because any age hardening always leads to the decrease in solute concentration c_o in the matrix. The changes in the obstacles controlling dislocation motion would be considered. Riley *et al* suggested that dislocation motion is limited by dislocation interactions in the as-quenched specimens and by particle interactions in the aged specimens[12,13]. In addition, the obstacle strength increases by aging[16]. During aging the strength of the δ' particles which act as the obstacles to dislocation motion increases due to the precipitation and growth of the δ' particles, thus the stress must increase to keep the dislocation moving. On further aging the decrease in solute concentration accompanying precipitation will result in an increase in the aging time required to lock the arrested dislocation provided C_1 remains constant. This decreases the dislocation density becoming effectively strain aged at waiting time; thus, long aging times cause serrarion amplitude again to decrease up to zero.

The mechanism of predeformation prior to aging to suppress serrated flow is unclear. There is not the evidence of the obvious decrease in solute concentration Co in the matrix because the precipitation of δ' phase promoted by predeformation was not observed on the initial stages of aging. The increase in ε_c can only be rationalized in terms of a decrease in obstacle spacing L. The obstacle spacing will depend on the density and distribution of the rate controlling obstacles. It would be suggested that for the shorter aging times the dislocations introduced by predeformation still remains, despite some of them were reverted. These dislocations will act as strong obstacles to dislocation motion and result in a decrease in L. In addition, for the longer aging times the precipitation of S' phase was promoted due to the dislocations acting as the sites for the nucleation of S' precipitation. This would cause L to continue to decrease as the precipitate particles become more effective obstacles to dislocation motion.

5 CONCLUSIONS

In the as-quenched and under-aged conditions the 8090 Al-Li alloy exhibites the serrated flow during tensile testing, and, negative strain rate sensitivity of flow stress is present when the serrated flow occurs. The predeformation prior to aging and the increase in strain rate suppress the occurrence of serrations. The serrated flow behaviour is influenced by the changes in the matrix solute concentration, the obstacles controlling dislocation motion and the obstacle spacing duing aging prosess.

REFERENCES

1 Cottrell, A H. Phil Mag, 1953, 44: 829.
2 Mulford, R A *et al.* Acta Metall. 1979, 27: 1125.
3 Van, Den Beukel, A. Acta Metall. 1980, 28: 965.
4 Tamura, M *et al.* Trans JIM, 1973, 14: 355.
5 Wert, J A *et al.* Scripta Metall, 1985, 19: 463.
6 Gregson, P J *et al.* Mater Sci Technol, 1988, 4: 713.
7 Welpmann, K *et al.* In: Aluminum-Lithium 6, DGM Informations gesellschaft mbH. Germary, 1992.
8 Behnood, N *et al.* Acta Metall. 1989, 37: 687.
9 Huang, J C; Gray III, G T. Scripta Metall et Mater. 1990, 24: 87.
10 King, J E *et al.* Acta Metall, 1981, 29: 1553.
11 Ling, C P; McCormick, P G. Acta Metall et Mater. 1990, 38: 2631.
12 McCormick, P G. Acta Metall, 1972, 20: 351.
13 Riley, D M *et al.* Acta Metall, 1977, 25: 181.
14 McCormick, P G. Acta Metall, 1971, 19: 463.
15 Pink, E *et al.* Acta Metall. 1982, 30: 2153.
16 Schoeck, G. Acta Metall. 1984, 32: 1229.