

A l - L i 系合金のアコースティック・エミッション
による変形特性

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研究成果報告書

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研究代表者 古 川 稔
(福岡教育大学)

はしがき

リチウム (Li: 比重0.53) を10mol%程度含むアルミニウム合金はアルミニウム合金の中で比重が小さく、また $L1_2$ 型規則相による析出強化により高い強度が得られる。そのため宇宙・航空機用材料として注目され、活発な研究が行われている。なかでも現在、Al-Li-Cu-Mg-Zr合金が最も有望とされている。

これまでに、著者らはAl-11.1mol%Li合金およびAl-Li-Cu-Mg-Zr合金の室温での強化と変形機構、77K～523Kにおける変形機構の温度依存性および歪速度依存性を明らかにした。またAl-Li 2元合金のヤング率の温度依存性および熱処理中の脱リチウムについても検討した。

本系合金の実用化に対する最も大きな障害は、低靱性の性質にある。著者らは、これまでにTEMによる変形転位の観察に基づいて、Al-Li系合金の変形特性を検討してきたが、本研究ではアコースティック・エミッション(AE)法により、Al-Li-Cu-Mg-Zr合金の変形特性を調べ、他の実用アルミニウム合金(Al-Cu系、Al-Mg系など)と比較検討することにより、低靱性とAEによる変形特性を明らかにする。

本研究の成果は、新しい高比強度・高比弾性率金属材料開発のための不可欠の基礎資料となることが期待される。

研究組織

研究代表者： 古川 稔 (福岡教育大学教育学部 助教授)

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Formation and Stability of Orowan Loops in Al-Li Single Crystals.

5th International Aluminium-Lithium Conference, March 1989

研究成果

- (1) 3 ~ 4.5mass%リチウムを含むAl-Li 2元合金において、 δ' (Al_3Li) $L1_2$ 規則相が転位により剪断される不完全時効状態から完全時効状態においては、応力-歪曲線の降伏点近傍に大きなA Eが伴う。A Eは転位が δ' 相をバイパスする過時効段階では減少する。
- (2) Al-Li合金のA Eは、他のアルミニウム合金 (Al-4mass%Cu および7050合金) のA Eより著しく大きく、Al-Li合金における粗大な集中すべりに対応する。
- (3) Al-Li合金の低靱性は粗大すべりのみでは説明できず、無析出物帯 (PFZ) の形成や不純物元素の偏析などとの重畳効果に起因する。
- (4) Al-3mass%Li合金に0.1mass%以上のジルコニウムを添加することにより、降伏点近傍でのA Eは著しく減少し、破断伸びは増大する。
- (5) Al-Li合金へ0.1~0.2mass%のジルコニウムを添加すると、結晶粒が微細化され、 δ' 相と同じ $L1_2$ 規則相である微細な整合 β' (Al_3Zr)相が分散し、時効の初期段階から転位のバイパス機構が導入され、すべりの集中が抑制される。微量のジルコニウムの添加は靱性の改善にきわめて有効である。

さらに、学会誌、研究紀要等に発表した論文の別刷を以下に示し、研究成果とする。

Dislocations in Al-Li Single Crystals Deformed at Different Temperatures

Y. Miura,* K. Yusu,** M. Furukawa,*** and M. Nemoto*

* Department of Metallurgy, Faculty of Engineering, Kyushu University.

** Graduate School, Kyushu University.

*** Department of Technology, Fukuoka University of Education.

I. Introduction

Compressive strength of single crystals, containing 2-3mass% Li, in underaged and overaged conditions are measured in the temperature range from 77 to 523K and deformation-induced dislocation structure is observed by transmission electron microscopy(TEM). The observation is necessary for the discussion of temperature dependent deformation mechanisms in terms of dislocation-precipitate interaction.

II. Experimental Procedures

Single crystals were grown from rectangular ingots($6 \times 6 \times 100 \text{mm}^3$) of Al-Li binary alloy by the modified Bridgman method in an argon atmosphere with an alumina soft mold. The growth rate was $5 \times 10^{-3} \text{mms}^{-1}$. The grown single crystal rods were solution treated at 823K for $1.08 \times 10^4 \text{s}$ in glass capsules filled with argon, followed by quenching into iced water. Specimens($2.5 \text{mm} \times 2.5 \text{mm} \times 5.0 \text{mm}$) for compressive test were cut out of the rods and they were sealed in capsules with argon and aged at 473K for various periods of time up to $1.2 \times 10^6 \text{s}$. An Instron-type machine was utilized for the compressive deformation in the temperature range from 77 to 523K. The initial strain rate was $3.3 \times 10^{-4} \text{s}^{-1}$. An optical interference microscope was used for slip trace analysis. Most of the TEM observation was made of the thin foils that were cut parallel to the primary (111) slip plane. The Al_3Li precipitates were imaged with a 110 dark field reflection and dislocations were with a 220 dark field reflection. An electron microscope, JEM-1000 of the HVEM Laboratory of Kyushu University, was used.

III. Results

Analysis of slip lines in the underaged specimen revealed exclusive operation of octahedral slip for the whole temperature range tested. No trace of cubic slip was detected. Slip lines appeared to be coarser as temperature increased, being accompanied with occasional {111} cross slips. The TEM micrographs of slip plane sections of the underaged specimen A are shown in Photo.1. At lower temperatures, dislocations are all in pair. They tend to lie along certain crystallographic orientation. The distance between the dislocations in a pair becomes slightly larger as temperature increases, and pairs are no longer observed at temperatures higher than 423K. Dislocation loops as well as unit dislocations are observed at 423 and 473K. The size of the loops is comparable to the Al_3Li precipitates ($\sim 40 \text{nm}$). Above 523K random arrays of unit dislocations are observed. In the case of the overaged specimen B(Photo.2), dislocation loops and random unit dislocations are seen in the whole temperature range except at 77K, where a number of dislocation pairs are observed, indicating that shearing of the particles occurred. Those loops are approximately of the size of the precipitates in this aging condition ($\sim 150 \text{nm}$), implying that dislocations bypassed the precipitates by the Orowan mechanism. The densities of the Orowan loop and dislocations tend to decrease with increasing temperature. TEM observation was also made of the underaged crystals subjected to the strain rate change test. From these crystals, thin foils were cut perpendicular to the primary octahedral plane and parallel to the primary Burgers vector. The sheared Al_3Li precipitates were observed to be of oval shape. The amount of shear could be simply estimated by assuming that the Al_3Li precipitates were uniformly sheared by the primary dislocations along the (111) slip plane. The average taken with several precipitates is ~ 0.3 , being in good agreement with the given shear strain.

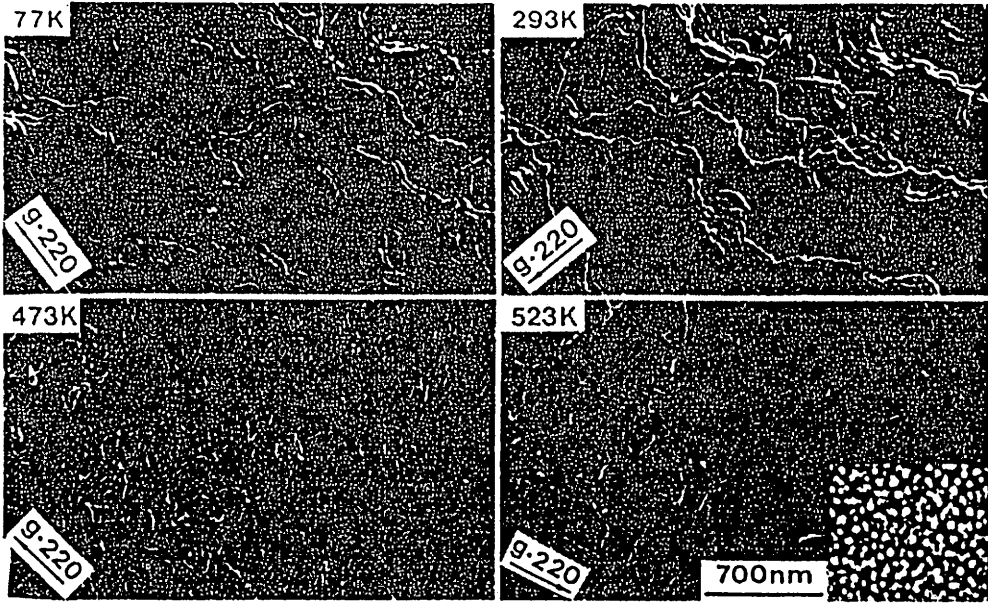


Photo.1. 220 dark field electron micrographs of (111) slip plane sections, showing the deformation-induced dislocations. A 110 dark field micrograph of δ' - Al_3Li precipitates is shown in the bottom right corner. (underaged crystal A)

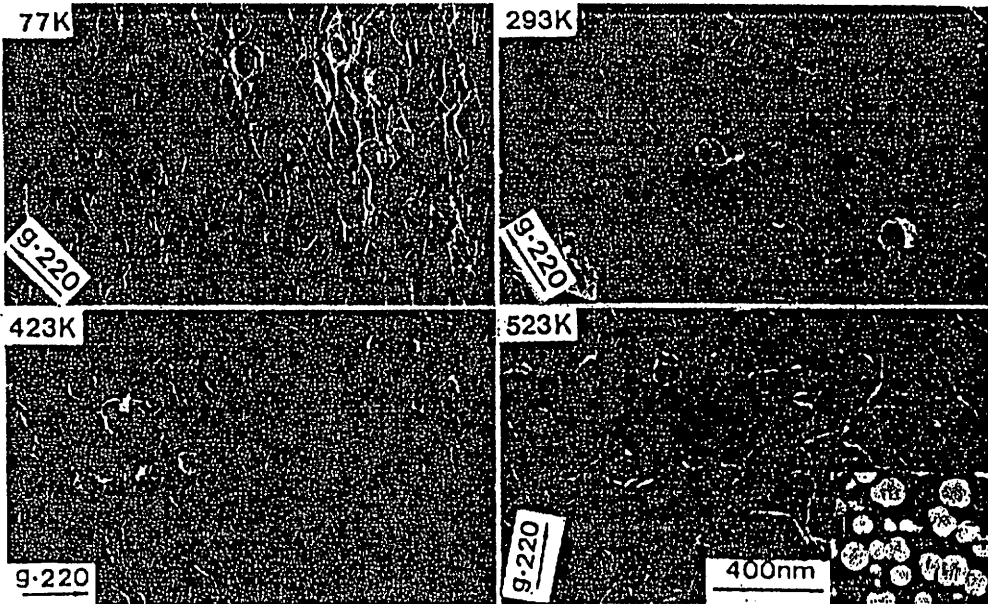


Photo.2. 220 dark field electron micrographs of (111) slip plane sections, showing the deformation-induced dislocations. A 110 dark field micrograph of δ' - Al_3Li precipitates is shown in the bottom right corner. (overaged crystal B)

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Yield Strength of Al-Li Solid Solution Alloys at High Temperatures

Y. Miura^{*}, S. Nishitani^{}, M. Furukawa^{***} and M. Nemoto^{*}**

^{*}*Department of Metallurgy, Kyushu University, 6-10-1 Hakozaki, Higashi-ku, Fukuoka, 812 Japan*

^{**}*Graduate School, Kyushu University*

^{***}*Department of Technology, Fukuoka University of Education, Munakata, 811-41 Japan*

ABSTRACT

Compressive yield strength of Al-Li binary solid solution was measured as a function of temperature, using single crystals and polycrystals. The temperature range and strain rate employed were 77-823K and $1.4-6.6 \times 10^{-4} \text{ s}^{-1}$ respectively. CRSS of the primary octahedral slip system of single crystals (5.3at%Li) varied with temperature in three steps: a large increase in the low temperature range below 200K; a gradual increase to a peak in the intermediate temperature range; the final drop at high temperatures. These observations imply a strong dynamic interaction, in the high temperature range, between dislocations and Li atoms, and are rather contrary to a prediction that no appreciable increase in strength is expected from a much smaller atom size effect in this alloy system. Strain rate change tests on polycrystals gave an apparent activation energy comparable to the energy of diffusion of Li in Al, and stress component $n=3-3.2$. Discussion will be made on the origin of the observed anomalous temperature dependence of the strength of Al-Li solid solution alloys.

KEYWORDS

Al-Li alloy, high temperature deformation, solution hardening, size effect, modulus effect, elastic interaction, drag stress.

INTRODUCTION

A world wide attention has been paid to the development of lithium-containing aluminum alloys as low density and high elastic modulus materials for aerospace application (for example, Champier and others, 1987). Strengthening mechanisms in these alloys have been discussed in terms of hardening by the precipitation of Li-containing and/or other phases. But few works have been reported on the strength of Al-Li solid solution. Negligible solution hardening may be expected from the small atom size difference between Al and Li, and a small change in the lattice parameter of Al by the addition of Li (Pearson, 1958). It has been reported so far that the addition of Li resulted in high rigidity (Noble, Harris and

Dinsdale, 1982) and Al-Li solid solution showed a serrated flow (Evans, 1987). These facts suggest rather peculiar nature of Al-Li solid solution, when compared with such alloy systems as Al-Mg, where high temperature mechanical properties have been extensively studied (Asada and coworkers, 1967).

In the present investigation, an experimental study has been made of the strength of Al-Li solid solution alloys, especially at high temperatures.

EXPERIMENTAL PROCEDURE

Both single crystals and polycrystals were used. Ingots of Al-Li binary alloys with different Li contents were made by induction melting. Single crystals were grown from rectangular ingots ($5 \times 7 \times 120 \text{ mm}^3$) by the modified Bridgman method in an argon atmosphere. The growth rate was $5 \times 10^{-3} \text{ mm s}^{-1}$. The grown single crystals were annealed at 823K for $1.08 \times 10^4 \text{ s}$ in glass capsules filled with argon, followed by quenching into iced water. Specimens for compressive test were cut out. Their dimensions are $3 \times 3 \times 6 \text{ mm}^3$. Polycrystalline specimens were cut out of the rolled plates and subjected to annealing with the same condition as for the single crystals. The Li content of the specimens was determined by atomic absorption spectrometry. Single crystals with 5.3at%Li and 4.9at%Li were used. Polycrystals with three different Li contents were also used, namely 2.0, 3.7 and 5.6at%Li. An Instron type machine was used for compressive test. The test temperature was in the range from 77 to 823K, and the strain rate was $1.4\text{--}6.6 \times 10^{-4} \text{ s}^{-1}$. Transmission electron microscopy was used to ensure that the specimens were completely free from precipitates.

RESULTS

Compressive Behavior of Single Crystals

In Fig. 1 the load-nominal strain curves are shown of the specimens with 5.3at%Li for different test temperatures. At lower temperatures high yield

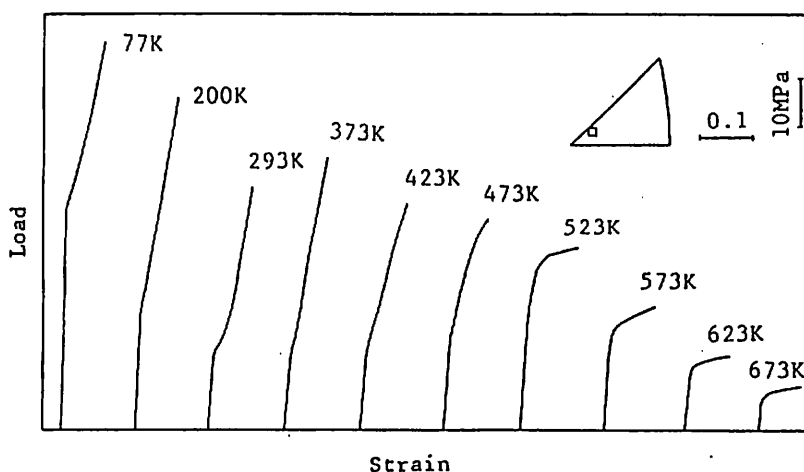


Fig. 1. The load-strain curves at different temperatures.

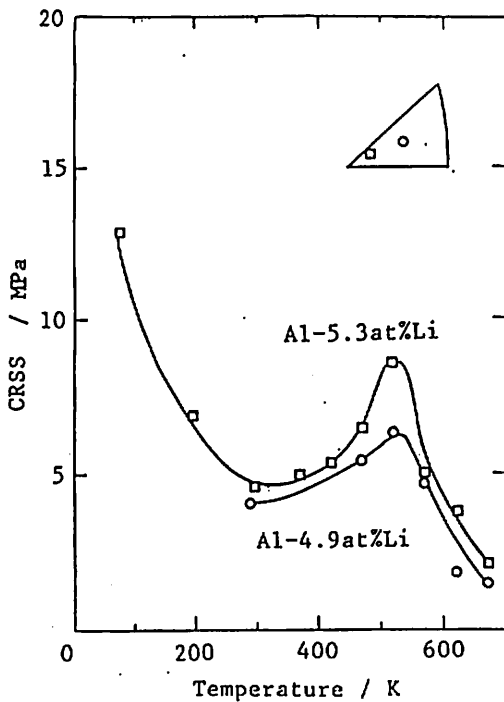


Fig. 2. CRSS vs Temperature.

strength and also high work hardening rate are observed. A drastic change occurs between 473K and 523K, where yield stress largely increases and work hardening rate becomes much decreased. A close examination of the curve for 523K reveals a stress drop after yielding. The CRSS in the primary $\{111\}\langle 110 \rangle$ system plotted as a function of temperature in Fig. 2.

The results for the crystals with 4.9at%Li are shown together. In the low temperature range, CRSS decreases with increasing temperature, but it begins to increase in the intermediate temperature range until it reaches a maximum. Finally CRSS drops in the high temperature range. The lower Li content gives generally lower values of CRSS. Strain rate change tests were carried out at 429K and 503K (Fig. 3). At 429K, in the intermediate range, no recognizable change in flow stress occurred by the change between 1.5×10^{-4} and $6 \times 10^{-4} \text{ s}^{-1}$. A serrated or jerky flow was observed. At 503K, which falls on the temperature slightly higher than the peak temperature, work hardening rate was negligibly small, with no serrated flow and sudden increase or decrease of the stress was observed corresponding to the sudden increase or decrease of strain rate.

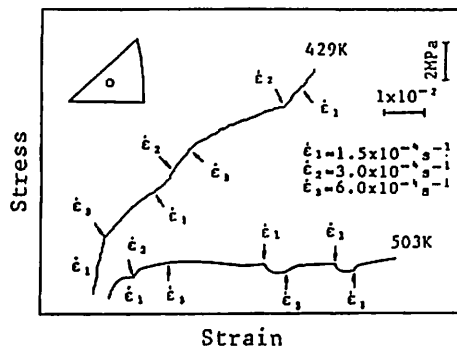


Fig. 3. Strain rate change.

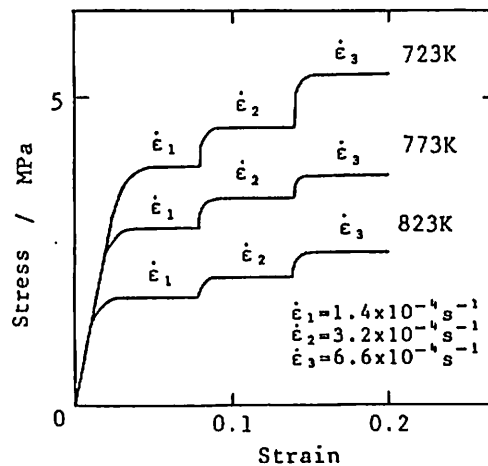


Fig. 4. Strain rate change.
(polycrystal)

Deformation of Polycrystals in the High Temperature Range

Polycrystalline specimens with three different Li contents were utilized for strain rate change tests at the high temperature range (723-823K), where

work hardening is negligible. These experiments were made in an attempt to estimate the values, n and Q , in the expression: $\dot{\epsilon} = A\sigma^n \exp(-Q/kT)$, where $\dot{\epsilon}$ is strain rate, σ is stress, Q is apparent activation energy, n is stress exponent, and A is a constant. Fig.4 shows the true stress-true strain relations for 3.7at% alloy. Negligible work hardening is observed at all test temperatures and strain rates employed. Similar results were obtained for the alloys with 2.0at%Li and 5.6at%Li. By the usual analysis of these data, stress exponent(n) and apparent activation energy(Q) were estimated to be 3-3.2 and 140-150kJ/mol, respectively. The variation of the values with Li content was not larger than the accuracy of the estimation.

DISCUSSION

Temperature Dependence of CRSS

The three step behavior of CRSS as a function of temperature can be interpreted in terms of dislocation-solute interaction. At lower temperatures, moving dislocations must overcome the obstacles of immobile solute atoms, which are randomly scattered and act as frictional resistance, or segregate around dislocations and act as locking obstacles. There seem to be a controversy over these two mechanisms of solution hardening(Suzuki, 1985). Stress for a dislocation to overcome these stationary obstacles is considerably high, but it decreases, as temperature increases, by the assistance of thermal activation. In the intermediate temperature range, where atomic diffusion becomes no more negligible, solute atoms tend to form an atmosphere around moving dislocations, forming viscous obstacles. This tendency increases with increasing temperature. Thus, CRSS in this range increases until it reaches a maximum. In the high temperature range, where diffusion is rapid and velocity of diffusing atoms becomes equal to that of moving dislocations, the stress to overcome the obstacles decreases rapidly as the solute atmosphere becomes diluted. The observed serrated flow in the intermediate range is attributed to the nonuniform motion of dislocations relative to the diffusing solutes. The estimated value of $n=3-3.2$ falls on the range for class I solid solution according to the classification by the previous workers(Shérby and Burke, 1966). The apparent activation energy $Q=140-150\text{kJ/mol}$ well agrees with the activation energy of self-diffusion of Al(Moreau, 1971) or of diffusion of Li in Al(costas, 1963). The above experimental results suggest the existence of a strong interaction between moving dislocations and solute atoms in Al-Li solid solution.

Solute-Dragging Stress

Solution hardening in substitutional solid solution is generally interpreted by the elastic interaction due to atom size difference. At high temperatures, where dislocations drag a solute atmosphere, the drag stress(τ) is expressed by(Cottrell and Jaswon, 1949; Yoshinaga, 1986):

$$\tau = (AN\alpha^2\mu^2cR^6b/DkT)v \quad (1)$$

where D is diffusivity of solute, N is the number of atoms in the unit cell, c is the average concentration of solute, μ is the shear modulus, α is atom size factor, R is the radius of solvent atom, b is Burgers vector, A is a constant, and v is the dislocation velocity. Since lithium changes little the lattice parameter of Al(Pearson, 1958) and the difference in atom size between Li and Al is small, a much decreased drag stress is expected from the above equation when compared with the case of Al-Mg. A

simple estimation of the drag stress per atomic per cent of Li is made for Al-Li system(τ_1) relative to that for Al-Mg system(τ_2). The ratio (τ_1/τ_2) at a certain temperature can be roughly estimated from the atom size factor(α), diffusivity(D) and dislocation velocity(v). Using the reported values for D(Costas, 1963; Moreau, 1971) and the estimated atom size factor on the basis of the lattice parameter change of Al(Pearson, 1958), one can obtain (τ_1/τ_2) \approx 0.05 if the equal dislocation density is assumed at the point of yield for both Al-Li and Al-Mg systems. This extremely small value estimated for the Al-Li system is not compatible with the experimental results, which show a considerable hardening. One possible reason for the discrepancy will be that dislocation velocity at the point of yield can be much higher in Al-Li system than in Al-Mg system. It is also possible that modulus effect, the alternative effect of elastic interaction, plays an important role. The bondings between Al and Li atoms are expected to be stronger than that between Li atoms, as one can imagine from the fact that apparent atom radius of Li is smaller in Al-Li solid solution than in pure Li. These discussions are consistent with the experimental fact that elastic modulus of Al becomes larger when Li is added (Noble, Harris and Dinsdale, 1982). Further study should be made to clear the details of the mechanism through which modulus effect leads to the observed peak of CRSS in the high temperature range.

CONCLUSION

1. Critical resolved shear stress of Al-Li solid solution single crystals varied with temperature in three steps: a large increase with decreasing temperature in the low temperature range, a gradual increase with increasing temperature to a peak in the intermediate range, and final drop in the high temperature range.
2. In the intermediate range, serrated flow was observed and the strain rate dependence of flow stress was negligibly small.
3. Flow stress decreased slightly after yielding at temperatures higher than the peak temperature.
4. Much larger solution hardening was observed at high temperatures than simply expected from the consideration of solute-dragging stress due to atom size effect only.

ACKNOWLEDGMENT

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FORMATION AND STABILITY OF OROWAN LOOPS IN Al-Li SINGLE CRYSTALS

Y.Niura* , K.Yusu**, S.Aibe**, M.Furukawa*** and M.Nemoto*

Modulus compensated yield strength of overaged Al-Li alloy single crystals, containing δ' -precipitates, is independent of temperature up to a critical temperature, beyond which it drops. Calculated Orowan stress using experimentally determined interparticle spacing well agrees with the measured strength. Softening at elevated temperatures is understood in terms of the increase in the effective interparticle spacings due to dislocation climbing by pipe diffusion. A high frequency of TEM observation of Orowan loops of the size equal to the particle size is consistent with the calculated longer life time of those equatorial loops for shrinkage.

INTRODUCTION

Al-Li alloys containing 2-3wt%Li harden by the precipitation of coherent ordered Al_3Li precipitates. Plastic deformation of the underaged alloys proceeds by precipitates shearing by super dislocations and the yield strength shows positive temperature dependence which is characteristic of $L1_2$ structure(1)(2). On the other hand, little work has been reported of mechanical behaviour of overaged Al-Li alloys. Orowan loops have been observed in Al-Li alloys, though experimental observation of Orowan loops has not been common, seldom in particle-hardened Al base alloys(3).

In the present investigation, deformation behaviour of overaged Al-Li binary alloy single crystals is studied, with main interest being in the estimation of Orowan stress and in the mechanism of softening at high temperatures, and also in thermal stability of Orowan loops.

* Faculty of Engineering, Department of Metallurgy, Kyushu University, Fukuoka, 812, Japan

** Graduate School, Kyushu University, Fukuoka, 812, Japan

*** Faculty of Education, Department of Technology, Fukuoka University of Education, Munakata, 811-41, Japan

EXPERIMENTAL PROCEDURE

Single crystals of a Al-Li binary alloy were grown by the modified Bridgman method, and rectangular crystals (2.5x2.5x5 mm³) were cut out. They were annealed to an overaging condition. Compressive deformation was made by an Instron type machine in the temperature range between 293 and 493K, with the initial normal strain rate 1.7×10^{-4} - 1.7×10^{-3} sec⁻¹. The details of experimental procedure are referred to the previous papers by the authors (1)(2). Table 1 summarizes the chemical composition, aging condition, and geometrical factor of the crystals. From the stress strain curves, critical resolved shear stress (CRSS) was determined. Transmission electron microscopic (TEM) observation was made of aging microstructure and deformation induced dislocation structures. Most observation was made on the thin films prepared parallel to the primary octahedral slip plane. An electron microscope, JEM-200CX, of the HVEM Laboratory of Kyushu University was utilized.

TABLE 1. Single crystal of Al-Li binary alloy.

Lithium content* (wt%)	aging condition	schmid factor S_{111}
1.94	443K 1.2x10 ⁶ sec	0.45

* determined by atomic absorption analysis.

RESULTS

Temperature dependence of CRSS (τ_c)

Figure 1 shows the shear modulus compensated CRSS (τ_c / μ) as a function of test temperature (T). The values for shear modulus of Al-Li single crystals reported by Müller et al. (4) have been utilized. At lower temperatures τ_c / μ does not vary much with temperature and stays constant until it starts to drop at critical temperature (T_c). The critical temperature increases from 400 to 440K with the increase in shear strain rate from 3.9×10^{-4} s⁻¹ to 3.9×10^{-3} s⁻¹.

TEM observation of Orowan loops and Al₃Li precipitates

Dark field electron microscopy revealed Orowan loops in the deformed crystals surrounding coherent Al₃Li precipitates. Loops are generally seen to tightly encircle the precipitates. In Photos 1 and 2 are shown pairs of 110 and 220 dark field electron micrographs showing such loops and Al₃Li particles

in the crystals deformed with a shear strain rate of $3.9 \times 10^{-3} \text{ sec}^{-1}$ to around 10% shear strain, at 393 and 493K, respectively. The temperatures correspond to below and above the critical temperature. The density of the observed loops decreases with the increase of deformation temperature. The close examination of a pair of 110 dark field and 220 dark field electron micrographs of the same area reveals that the size of each observed loop surrounding the particles is close to the diameter of the corresponding particles. Thickness of an area in a foil was determined by the contamination spot separation method(8), and a histogram of the Al₃Li particle size was made. The volume fraction (f) and mean square interparticle spacing (λ_s) are determined to be 0.107 and 106nm, respectively.

Orowan stress

The present result of compression test in Fig.1 shows that τ_c / μ is independent of T in the low temperature range, and TEM observation reveals dislocation loops surrounding the precipitates(Photo.1 and 2). These facts confirm that plastic deformation in the present overaged Al-Li crystal proceeds by Orowan bypassing. In the following, Orowan stress of the present crystal is estimated based on the common equation and comparison is made with the experimentally obtained strength.

The simplest equation for the Orowan stress, τ_{or} , is,

$$\tau_{or} = \alpha \mu b / \lambda \quad (1)$$

where α is a constant (<1), μ is the shear modulus, b is the magnitude of Burgers vector and λ is the interparticle spacing.

This equation is adequate for an order-of-magnitude estimation of CRSS. More refined versions can be obtained by considering such parameters as mean planer interparticle spacings, the dislocation dipole effect and the effect of the nature of dislocation on line tension(5). One of the most refined form is by Scattergood and Bacon(6), which appears as,

$$\tau_{or} = (0.8A \mu b / 4 \pi \lambda) \{ \ln(D/r_o) + B \} \quad (2)$$

where $A = 1$, $B=0.7$ for edge dislocations and $A=1/(1-\nu)$, $B=0.6$ for screw dislocations, ν is Poisson's ratio, $D = \lambda d / (\lambda + d)$: a harmonic mean of interparticle spacing and diameter of particles(d). Another factor that possibly has a critical effect is the distribution of particle size. Yeh et al.(7) made a statistical analysis of the effect and suggested that the interparticle spacing should be corrected, taking into consideration of the particle size distribution, as follows,

$$\lambda_1 = 1.25 \left[(2\pi/3f) (\bar{r}^3 / \bar{r}) \right]^{1/2} - (\pi/2) (\bar{r}^2 / \bar{r}) \quad (3)$$

where f is the volume fraction of the particle, $r=d/2$ is the particle radius, \bar{r}^3 , \bar{r}^2 , \bar{r} is the mean value of r^3 , r^2 , and r, respectively. From the histogram of the particle size the above mean values are obtained for the present crystal, and λ_1 is calculated to be 115nm, that is larger than the mean square interparticle spacing, $\lambda_s = 106 \text{ nm}$, by about 10%.

The result of calculation of Orowan stress using the equation (2) with the corrected and uncorrected interparticle spacings is shown in Table 2. The average value of τ_{or} / μ between screw and edge dislocations falls in $2.2-2.4 \times 10^{-3}$, being close to the measured value of $\tau_c / \mu = 2.5 \times 10^{-3}$ (Fig.1). A part of the discrepancy is attributed to the strength of the solid solution matrix, whose contribution can be in the order of 2×10^{-4} (9).

TABLE 2. Orowan stress calculated from equation(2).

Interparticle spacing (nm)	Calculated Orowan stress (τ_{or} / μ) $\times 10^3$		
	edge	screw	average
$\lambda_s = 106$	2.82	1.90	2.36
$\lambda_l = 115$	2.61	1.76	2.19

Softening at high temperatures

The observed drop in (τ_c / μ) at high temperatures and the strain rate dependence of critical temperature shown in Fig.1 indicate that certain thermally activated softening processes are taking place. At high temperatures, there is the possibility that dislocations bypass some particles by climbing over without forming Orowan loops, giving rise to larger effective interparticle spacings and thus the lower strength. The following equation is deduced, assuming that, under the critical condition, the rate of dislocation climb is equal to that of dislocation arrival to the particles(11)(12),

$$\dot{\gamma} = (M / T_c) \exp(-Q/k T_c) \quad (4)$$

where M is a constant including the shear modulus and the particle size, Q is the apparent activation energy for the diffusion process, k is the Boltzmann constant. A plot of $1/T_c$ against $\ln(\dot{\gamma} T_c)$ for a constant particle size in the present work is shown in Fig.2, which indicates a linear relationship. From the slope of the line, the apparent activation energy is estimated to be $\sim 0.8\text{eV}$, reaching about 60% of the activation energy for self-diffusion or for interdiffusion of Li in Al(10). These results implies that the softening observed at high temperatures takes place through dislocation climbing by pipe diffusion, or certain other low activation energy processes. Further work is necessary to investigate the details of the mechanisms, since the critical temperature T_c was only crudely measured in the present work.

DISCUSSION

Stability of Orowan loops

The electron micrographs of deformed crystals in Photos 1 and 2, show that single loops are surrounding the precipitates. These observation leads to an idea that the rate of formation, during deformation, of Orowan loops was generally smaller than the rate of removal by climbing within the present deformation condition. In the followings, simple estimation of the rates of formation and removal will be made.

The rate of formation of Orowan loops is simply estimated by calculating the time interval (t_a) between the arriving dislocations at particles,

$$t_a = L/v_d = (\rho^{1/2} b/\dot{\gamma}) \quad (5)$$

where L is the average distance between moving dislocations, being $\sim 10^{-4}$ cm if the density of the mobile dislocations is assumed to be $\sim 10^8$ cm $^{-2}$, $\dot{\gamma}$ is the applied shear strain rate equal to $\sim 10^{-4}$ s $^{-1}$, b is the magnitude of the Burgers vector. The calculated interval t_a falls in the order of one second.

It has been pointed out that Orowan loops around coherent precipitates such as δ' -Al $_3$ Li particles can climb out by pipe diffusion even in low temperature annealing(12)(13). Mori and Tokushige(13) discussed the climb rate of Orowan loops in low temperature recovery from a continuum approach. Assuming that the driving force for climbing comes from the energy decrease due to the shrinkage of the loop, they proposed an equation expressing the climb rate of a loop. According to their analysis, the time t_0 for an Orowan loop, originally staying at position $s = z/r$, to climb out is expressed by,

$$t_0 = (1/\alpha \mu \Omega \delta^2) (2kT/D_p) r^4 \times \{ -\ln s - (1/4)(1-s^2)(3-s^2) \} \quad (6)$$

where z is the distance from the center of spherical particle, α is a constant, Ω is the atomic volume, δ^2 is the cross section of dislocation pipe, r is the radius of the loop, D_p is the pipe diffusion coefficient, s is a dimensionless parameter. The equation is applied to estimate the life time of Orowan loops around δ' -Al $_3$ Li precipitates. Fig.3 shows the results of the calculation for a loop originally lying at the position s of a spherical particle of radius $r = 26.7$ nm (the average size of the δ' -precipitates in the present crystal). Here, $D_p = 1.7 \exp(-0.74\text{eV}/kT)$ cm 2 /sec was used. For simplicity, the activation energy (Q_p) for pipe diffusion is assumed to be one half of that for volume diffusion in Al(14), that is, $Q_p = Q_v/2 = 0.74\text{eV}$. The curves in Fig.3 is only for a short period of time, but they clearly show much decreased life time for the increased temperature and also for the decreased loop size (corresponding to the increased value of $s = z/r$).

The calculated life time of a loop originally lying at, for example, $s=0.01$ is 1h(293K), 50sec(343K), 2sec(393K) and 0.03 sec(493K). Although the equation (6) implies an infinite

life time for loops lying just on the equator of the particles ($s = 0$), this does not seem to be realistic. Instead, $s = 0.01$ will be the reasonable minimum, because it corresponds to the case where the loops come close to the equator within the interplanar distance of the present crystal. The argument suggests that Orowan loops of any size in the present crystal will climb out within several tens of seconds at temperatures above 343K. Under the dynamic deformation condition, where the loops interact with the late-coming dislocations, the life time will be even shorter.

The TEM observation of fewer loops for higher deformation temperature and of loops with the size comparable to the particle size (Photos 1 and 2, and also the Photos in the previous paper(2)) is qualitatively understood according to the results of the discussion above. But a question arises as to the observation of single loops surrounding tightly the particles even for the high deformation temperatures. At present, no rational explanation is made. It might be possible that the first loop formed is just on the equator of the particle, or that the loop cuts locally in the particle, because δ' -precipitate of $L1_2$ structure is shearable, having high coherency with the matrix(15) and comparable elastic modulus with the matrix(4). Once a segment of a loop cuts in and, if any climbing takes place, the whole loop will be stabilized, because diffusion in the ordered phase is expected to be slower.

SUMMARY

The present investigation on the Orowan stress and stability of Orowan loops in overaged Al-Li alloy single crystals leads to the following conclusion.

- (1) Overaged Al- 1.94wt%Li alloy single crystals deform by Orowan looping in the early stage of plastic deformation.
- (2) Compressive yield strength of the crystals at lower temperatures agrees with the calculated Orowan stress using the experimentally determined interparticle spacing.
- (3) Analysis of observed strain rate dependence of strength suggests that softening at elevated temperatures occurs due to dislocation climbing by dislocation pipe diffusion.
- (4) Simple estimation shows that, at high temperatures, the rate of the removal of Orowan loops by pipe diffusion is larger than that of dislocation arrival at the particles, which successfully explains the observed fewer loops in the crystals deformed at higher temperatures.

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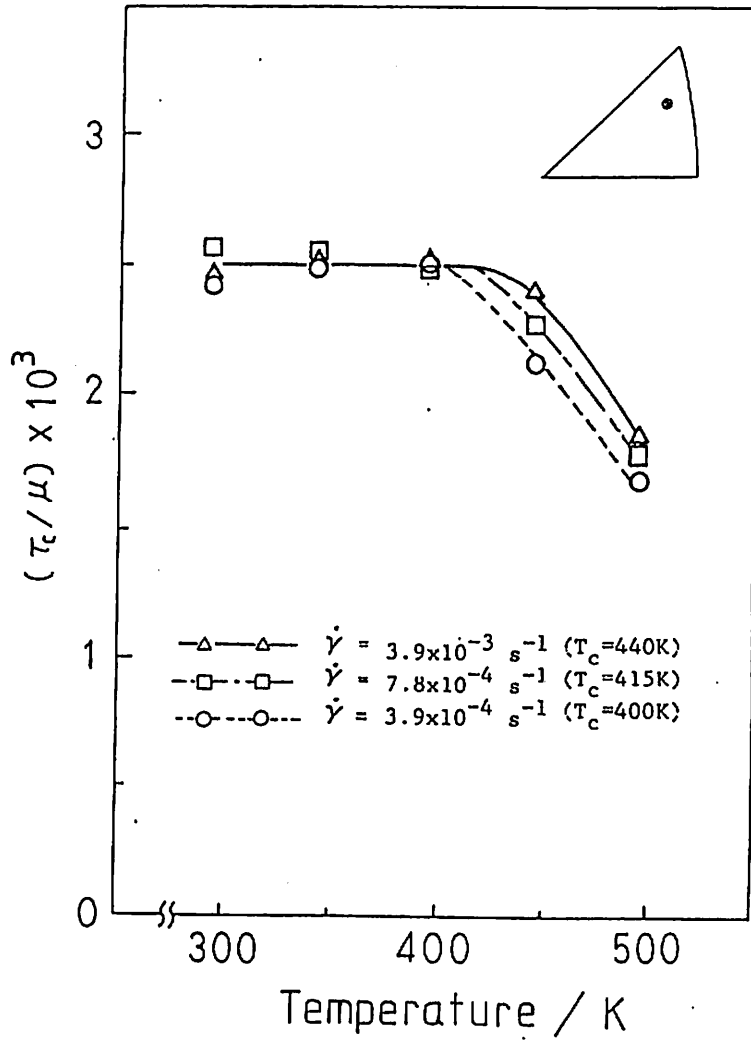


Fig. 1. Temperature dependence of modulus compensated critical resolved shear stress (τ_c).

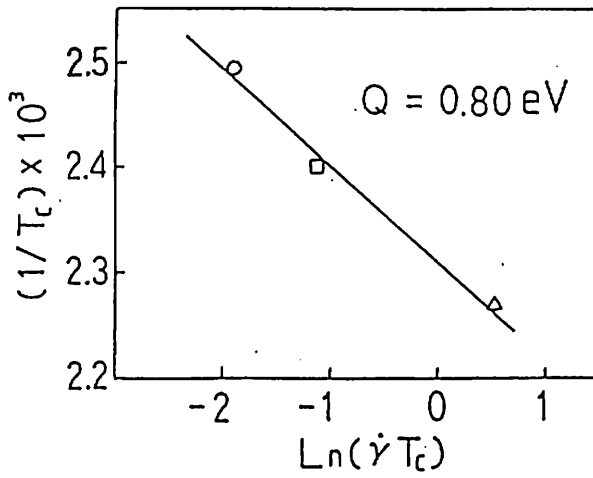


Fig. 2. A plot of $1/T_c$ vs $\text{Ln}(\dot{\gamma} T_c)$.

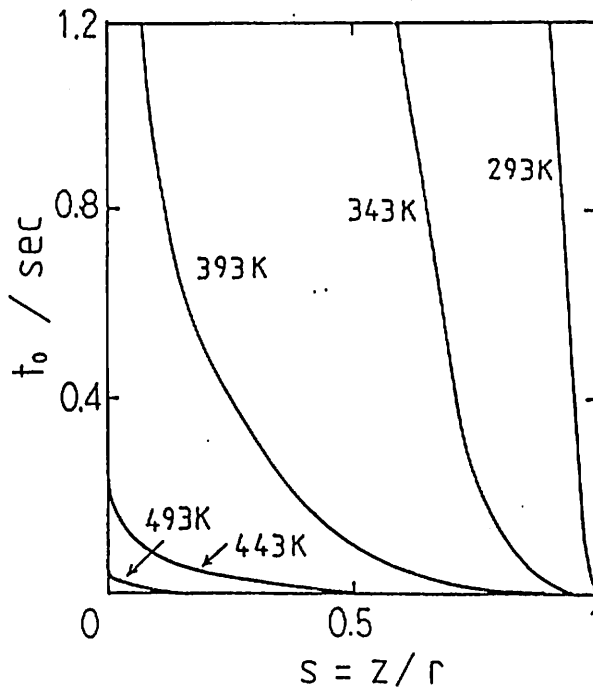


Fig. 3. Calculated time (t_0) for an Orowan loop, lying at the position s , to climb out by pipe diffusion at different temperatures.

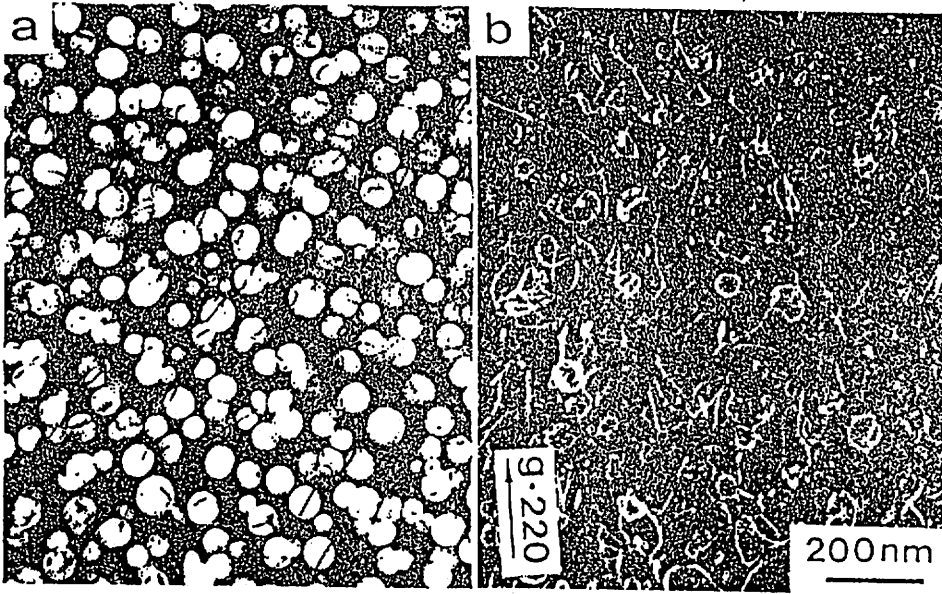


Photo. 1. Transmission electron micrographs of primary (111) slip plane section. (Deformed at 393K)
 (a):110 dark field micrograph of δ' -particles.
 (b):220 dark field micrograph of Orowan loops.

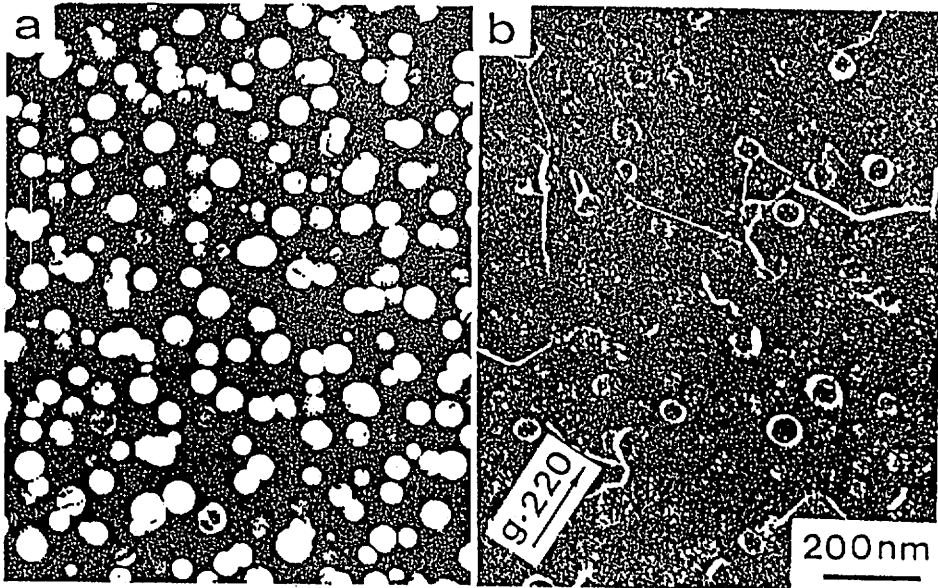


Photo. 2. Transmission electron micrographs of primary (111) slip plane section. (Deformed at 493K)
 (a):110 dark field micrograph of δ' -particles.
 (b):220 dark field micrograph of Orowan loops.