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Effect of Sub-Structure on Flow Stress of Polycrystalline Aluminum*

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The influence of polygonized sub-structure on flow stress of pure aluminum (99.9%) was investigated.

As flow stress increases parabolically with the inverse square root of sub-grain size, Petch's relation does not hold true between flow stress and sub-grain size. It is clarified that not only sub-grain size but also misorientation across the sub-boundaries has great influence on flow stress.

In addition, it is observed during the experiment that flow stress σ_f increases with the square root of excess dislocation density D_b of the sub-boundary, yielding the relation

$$\sigma_f = \sigma_0 + \alpha' G b D_b^{1/2}.$$

The effect of sub-structure on flow stress appears to be better explainable in terms of the work-hardening theory which is inclusive of the interaction of dislocations rather than Petch's pile-up theory.

1. Introduction

Metals generally used in industry are polycrystalline aggregates. The difference between a polycrystalline metal and a single crystal lies in the existence of grain boundary. This grain boundary is acknowledged as having a great effect on yield strength and strain hardening of metals. Many investigators have devoted themselves to investigating the effect of grain size on flow stress of polycrystalline metals.

Petch⁽¹⁾ has demonstrated experimentally that a simple relation exists between flow stress σ_f and the average grain diameter d in the form,

$$\sigma_f = \sigma_0 + K d^{-1/2} \dots \dots \dots (1)$$

In interpreting Eq. (1), Petch proposed a microscopic model utilizing the stress concentration produced by pile-up of dislocations at grain boundary. According to him, when the stress concentration at a grain boundary reaches a critical value, it will trigger the motion of dislocations in next grain, and yielding takes place.

Actual polycrystalline metals, however, do not have the simple structure such as was assumed by Petch. Individual grains in polycrystalline metals contain sub-structure formed during the process of manufacture and cold working.

There is abundant evidence that sub-structure has a considerable effect on yield strength of metals. Most investigations regarding the effect of sub-structure on the mechanical properties have been performed on single crystals or lightly worked polycrystals.

J. C. Ball⁽²⁾ introduced sub-structure into polycrystalline aluminum (99.994%) by 35% tensile strain at different temperatures ranging from -183 to 375°C , and found that flow stress increased proportionally to the inverse square root of the sub-grain size. A similar relationship was established for iron^{(3) (4)}.

On the other hand, F. Hultgren⁽⁵⁾ studied the influence of polygonized sub-structure on flow stress of polycrystalline aluminum (99.98%). An analysis of these results demonstrated that Petch's relation did not hold true between flow stress and sub-grain size, but rather that flow stress increased in proportion to the square root of dislocation density.

In the present work, the influence of polygonized sub-structure size in commercially pure aluminum (99.9%) on flow stress was investigated. Discussion as to whether or not Petch's relation holds true between the sub-grain size and flow stress has been made.

2. Experimental procedure

The specimen used in this investigation is commercially pure aluminum, the chemical composition of which is given in Table 1. The

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geometry of the specimen is shown in Fig. 1. The specimens were annealed at 600°C for different lengths of time to produce a variety of grain sizes listed in Table 2, and then at 300°C for 3 hours. The specimens were given pre-strain in tension from 5 to 20% at room temperature and annealed at 300°C for 5 hours to produce various polygonized sub-structures. All specimens were rapidly cooled in air from the annealing temperature to retain the sub-grain wall structure which had developed at the annealing temperature. An apparently polygonized sub-structure had developed in all specimens. It has been stated by Kelly⁽⁶⁾ that such heat treatment affects neither the sub-structure size nor misorientation angle. Before measurements of sub-structure size, all specimens were electrolytically polished to a depth of approx. 150 microns, as the surface layers were not characteristic of the interior of the metals⁽⁷⁾.

Measurements of sub-grain size and misorientation angle were made, by utilizing the X-ray micro-beam technique developed by Hirsch and Kellar⁽⁸⁾. In the present experiment, back-reflection photographs were taken on all specimens, using the beam of CuK_α in 100 μ diameter without filter. The operating power was 400 μ A at 50kVP, and the focus size 0.015 $\times$ 0.09mm². The (422) ring was examined.

The sub-grain size t was determined by means of counting spots in arc on the micro-beam photographs, according to Taira and Hayashi⁽⁹⁾,

$$t = v^{1/3} = T_0 (\pi/4)^{1/3} m_i^{-2/3} \cong T_0 m_i^{-2/3} \dots (2)$$

where v denotes volume of the sub-grain, T_0

Table 1 Chemical composition of specimens (%)

Al	Cu	Si	Fe
99.9	0.003	0.03	0.06

Table 2 Recrystallized temperature and mean grain size of specimens

Specimen	Recrystallized temperature °C	Time hr	Mean grain size μ
A	600	0.5	60
B	600	1	80
C	600	2	100

Fig. 1 Specimen geometry

original grain size, m_i number of spots in arc.

Total misorientation angle β was estimated from the length of the arc on the micro-beam photograph, as given by Hirsch⁽⁸⁾ (10):

$$\sin(\beta/2) = \cos\theta \sin(\gamma/2) \dots (3)$$

where γ is the angle subtended by the arc at the centre of the rings on the micro-beam photograph and θ is Bragg's angle. The average of the values calculated from each arc length appearing on several photographs was adopted as the values of t or β for a specimen.

Excess dislocation density D_b of the sub-boundary can be calculated numerically from the value of sub-grain size t and misorientation angle α by Eq. (4)⁽¹¹⁾,

$$D_b = \alpha/bt \dots (4)$$

where b is Burgers vector. The magnitude of α cannot be directly determined from the photograph. In the present work, sub-grains were presumably oriented about the mean position in the form of Gaussian distribution. On this assumption, α is given by Eq. (5)⁽¹¹⁾,

$$\alpha = \beta/3 \dots (5)$$

Then excess dislocation density D_b is given by Eq. (6)⁽¹¹⁾,

$$D_b = \beta/3bt \dots (6)$$

Mechanical testing was performed on an Instron machine at room temperature. Flow stress was calculated from the load-elongation curve obtained from the tensile test. Flow stress was defined as the nominal stress which produced a given strain, the 0.2% flow stress, for example, being the stress which produced 0.2% plastic deformation. Tensile speed was 8×10^{-2} mm/sec for all specimens.

3. Experimental results and discussion

3.1 Sub-grain size and total misorientation

The sub-grain size t is plotted in Fig. 2 as a function of pre-strain ϵ for various original grain sizes. It can be seen that at little pre-strains there is a rapid fall in sub-grain size, but that after a pre-straining of approx. 10% the change in sub-grain size slows down. For a given pre-strain, sub-grain size has a tendency to decrease with a decrease in the original grain size of the specimen; at approx. 5% pre-strain, there is an approx. 8 micron difference in sub-grain size among the specimens of various grain sizes, but this decreases with increasing pre-strain, to 1~2 microns at approx. 20% pre-strain.

A. Kelly⁽⁶⁾ observed the sub-grain size change of aluminum, using the X-ray micro-beam technique, and found that the sub-grain size reached

a limiting value of approx. 2.8 microns independently of the original grain size. In Kelly's experiment, however, the specimens had not been annealed after deformation, therefore spots in the arcs were inconspicuous, and so determination of sub-grain size was made difficult. In the present experiment, as specimens were annealed after pre-straining, the arcs on the micro-beam photograph still remained sharp after the pre-straining of approx. 20% as shown in Fig. 3; hence the number of spots in arc could be determined with good accuracy. This could account for the original grain size dependency of sub-grain size as was observed

Fig. 2 Relation between sub-grain size and pre-strain

(a) Specimen A, $\epsilon = 20.0\%$
 (b) Specimen B, $\epsilon = 19.6\%$
 (c) Specimen C, $\epsilon = 19.0\%$
 Fig. 3 Micro-beam photographs

in the present study. In the deformation range so far examined, the sub-grain size t can be related to pre-strain ϵ by the following equation,

$$t = a/\epsilon + b \quad (7)$$

where a and b are constants dependent on the original grain size T_0 . The values of a and b are given in Fig. 2. Equation (7) encompasses the same form as that outlined by Kelly⁽⁶⁾.

Figure 4 shows the relationship between the total misorientation β and the pre-strain ϵ . This total misorientation increases with an increase of pre-strain. At approx. 5% pre-strain, the value of β is almost independent of the original grain size. Over 10% pre-strain, β for the fine-grained specimen has a tendency to become a little larger than for the coarse-grained one, although some of the coarse-grained specimens do have the same value of β as the fine-grained ones. As previously observed by Kelly⁽⁶⁾ as well as the present authors⁽⁷⁾ the total misorientation in tensile straining was independent of the original grain size. As the average value of β for each pre-strain in this investigation is approximately the same as that in previous works, the total misorientation can be considered as almost independent of the original grain size.

3.2 Excess dislocation density of sub-boundary

Figure 5 shows the relationship between the average excess dislocation density D_b and the pre-strain ϵ . Excess dislocation density of sub-boundaries was calculated according to the formula (6), assuming that the angular misorientation of the sub-grain conformed to the Gaussian law. The present experimental results indicate that dis-

Fig. 4 Relation between total misorientation and pre-strain

location density increases proportionally to the square of tensile pre-strain. So far as the experiment is concerned, the relation can be approximately expressed by the equation,

$$D_0 = a'\epsilon^2 - b' \quad (8)$$

where a' and b' are constants dependent on the original grain size T_0 . The values of a' and b' for the pre-strains from 5 to 20% are given in Fig. 5. For a given pre-strain, the finer the original grain size of the specimen, the higher the dislocation density and the rate of increase to the pre-strain.

3.3 Relation between sub-grain size and flow stress

Figures 6 (a), (b), (c) show the relationship between the inverse square root of sub-grain size t and flow stress σ_f in re-straining of the specimens having polygonized sub-structure. If Petch's relation holds true between the flow stress and sub-grain size in the same manner as in original grain size, the relation should be represented by straight lines determined by the least mean square, as shown by the dotted lines in the figures. The present experimental results indicate, however, that the relationship between the inverse square root of sub-grain size and 0.2, 1, and 4% flow stresses can be represented not by straight lines as predicted by Ball⁽²⁾, but by smooth curves with varying slopes shown by solid lines in the figures. It does appear, however, that a linear relationship can be established between the 7% flow stress

Fig. 5 Relation between excess dislocation density and pre-strain

and the inverse square root of the sub-grain size.

Ball⁽³⁾ adopted Petch's equation in accounting for the work hardening, by assuming that the sub-grain boundaries formed during deformation became barriers to dislocation movement, and obtained

(a) Relation between flow stress and inverse square root of sub-grain size of specimen A

(b) Relation between flow stress and inverse square root of sub-grain size of specimen B

(c) Relation between flow stress and inverse square root of sub-grain size of specimen C

Fig. 6

$\sigma_f = \sigma_0 + 2\{2Gb\tau_B/\pi(1-\nu)\}^{1/2}t^{-1/2}$ (9)
 where σ_0 denotes frictional stress, G shear modulus, τ_B sub-boundary strength and ν Poisson's ratio. Putting in Eq. (9)

$$K_s = 2\{2Gb\tau_B/\pi(1-\nu)\}^{1/2},$$

we arrive at

$$\sigma_f = \sigma_0 + K_s t^{-1/2} \text{ (10)}$$

Equation (10) is the same form as Petch's which represents the relation between the flow stress and original grain size. Equation (9) suggests that the flow stress depends only on the sub-grain size, and that strength of sub-boundary τ_B is constant, and independent of sub-grain size. According to the present investigation, however, the relation between the 0.2, 1, and 4% flow stresses and the inverse square root of sub-grain size is represented by a parabolic one. Hence, the slope K_s in Eq. (10) is not constant but varies with sub-grain size. It is feasible that the variation of K_s with the sub-grain size is due to a change of sub-boundary strength caused by the change in the character of the sub-boundary for variation of the pre-strain.

On the other hand, the effect of the misorientation angle on the flow stress has been studied by J. Washburn⁽¹²⁾, who found that the influence was greater at a low angle boundary than a high angle one.

J. C. M. Li⁽¹³⁾ has introduced an equation equal to Petch's by proposing a new mechanism in which dislocations can be generated from the sub-boundary without requiring a pile-up. He assumed that after the dislocations moved away from the sub-boundary, they were left in the sub-grain approximately parallel to each other, forming a Taylor-type forest, and he expressed the stress required for moving dislocations in such a forest as a function of sub-grain size t and misorientation angle α ;

$$\sigma_f = \sigma_0 + Gb/2\pi(1-\nu)\{(8\alpha/\pi b)\}^{1/2}t^{-1/2} \text{ (11)}$$

Equation (11) can be written as below;

$$\sigma_f = \sigma_0 + K_s' t^{-1/2} \text{ (12)}$$

where

$$K_s' = 1.6G\sqrt{b\alpha}/2\pi(1-\nu).$$

Equation (12) has the same form as the relation (10) obtained by Ball. Li's model predicts K_s' depends on α . In Eq. (12), if the relation between flow stress and the inverse square root of sub-grain size be linear, the slope K_s' and hence the angle of misorientation between sub-grains should be constant, independently of sub-grain size. The present results indicate however that the angle of misorientation between sub-grains varies according to the degree of pre-strain, as demon-

strated in Fig. 4. The slope K_s' in Eq. (12) increases in proportion to the square root of the misorientation between sub-grains, resulting in a parabolic relation of the flow stress to sub-grain size.

From the aforementioned facts, it is evident that not only sub-grain size but misorientation greatly influences flow stress.

3.4 Relation between the flow stress and dislocation density

Figure 7 (a) shows the relationship between the 0.2% flow stress and the square root of excess dislocation density. The flow stress σ_f as seen in the figure, increases proportionally to the square root of the dislocation density D_b . This relation can be expressed by a linear equation:

$$\sigma_f = \sigma_0 + CD_b^{1/2} \text{ (13)}$$

which is in good accord with that introduced from Wiedersich's work-hardening theory⁽¹⁴⁾. The result in Fig. 7 (a) indicates that C is almost independent of the grain size. Conrad et al.⁽¹⁵⁾ have already pointed out that σ_0 is constant when the grain sizes are less than the thickness of the specimen, but it begins to decrease as the grain sizes become greater than the specimen thickness. Equation (13) applies also for the 1, 4, and 7% flow stresses, as shown in Fig. 7 (b), (c), (d).

Table 3 shows the values of σ_0 and C derived from the intercepts and slopes of the straight lines in Fig. 7 (a), (b), (c), (d). As plastic strain increases, the value of σ_0 increases, whereas C decreases. The value of C for the 0.2% flow stress is in fairly good accord with that for the 1% flow stress.

In Fig. 7 (a), the deviation of the 0.2% flow stress from the straight line has no definite inclination to the original grain size. The deviation of the 1, 4, and 7% flow stresses depends, however, on the original grain size, as shown in Fig. 7 (b), (c), (d). For example, in Fig. 7 (d), the flow stresses of specimen A exist above the straight line and those of specimen C under it, whereas only those of specimen B are on the straight line. The deviation depends on the strain where the flow stress is determined. When the amount of

Table 3 Values of σ_0 and C in equation $\sigma_f = \sigma_0 + CD_b^{1/2}$

Strain %	C cmkg/mm ²	σ_0 kg/mm ²
0.2	0.30×10^{-4}	3.54
1	0.31	4.37
4	0.26	5.43
7	0.18	6.08

re-strain is as little as 0.2%, dislocation multiplication is so minute that flow stress will be controlled by the initial dislocation density, regardless of the original grain size. As multiplication of dislocations proceeds with increasing re-strain, however, the original grain size as well as pre-strain does play a role in flow stress. In this case, the effect of dislocation density on the flow stress will be controlled by summation of initial and multiplied dislocations. Further research is necessary to establish the proposition. The objective of the present paper is to discuss the relationship between the 0.2% flow stress and dislocation density only.

The result in Fig. 7 (a) indicates that the effect of excess dislocation density on the flow stress may be expressed as

$$\sigma_f = \sigma_0 + \alpha' G b D_b^{1/2} \quad (14)$$

where σ_0 is frictional stress, and α' is a constant. Taking $G = 2.7 \times 10^3 \text{ kg/mm}^2$, $b = 2.86 \times 10^{-7} \text{ mm}$, we obtain $\alpha' = 0.39$ from the slope in Fig. 7 (a).

On the other hand, substituting $D_b = \alpha/bt$ and

$\nu = 0.35$ in Eq. (11) which was derived by Li, we obtain,

$$\sigma_f = \sigma_0 + 0.39 G b D_b^{1/2} \quad (15)$$

The coefficient of the second term on the right side of Eq. (15) is in quite good accord with α' in the present result.

Certain mechanisms for work-hardening of metals which have hitherto been proposed shall now be examined.

Taylor⁽¹⁶⁾ suggested that the dislocations which accumulate in lattice are responsible for work hardening. He assumed that the stress required to push a dislocation through an array of dislocations must be as great as the internal stress caused by accumulated dislocations.

Seeger⁽¹⁷⁾, in order to explain the linear hardening in fcc metals, suggested a super lattice of pile-ups or glide zones surrounded by sessile dislocations. According to this author, the sources in the centre of the glide zones are blocked by the back stress of the piled-up groups.

Mott⁽¹⁸⁾ and Hirsch⁽¹⁹⁾, with a view to ex-

(a) Relation between 0.2% flow stress and square root of excess dislocation density

(c) Relation between 4% flow stress and square root of excess dislocation density

(b) Relation between 1% flow stress and square root of excess dislocation density

(d) Relation between 7% flow stress and square root of excess dislocation density

Fig. 7

plaining the linear hardening of fcc metals, suggested that dragging of sessile jogs in screw dislocations determines flow stress, and that the number of jogs created is related to the density of forest dislocations.

These models essentially bring about the same relation between flow stress and dislocation density that can be written in a form similar to Eq. (14) obtained from the present experiment. Thus, the values of α' in Eq. (14) calculated by these three models are of the same order, that is, between 0.3 and 0.5.

It is most difficult to determine which model is most adaptable for describing the present results when utilizing the present data only.

4. Conclusions

Commercially pure aluminum containing polygonized substructure of various sizes was prepared by pre-straining followed by annealing, and the influence of polygonized sub-structure on the flow stress in re-straining was investigated. The experimental results may be summarized as follows;

(1) Sub-grain size t decreases with an increase of pre-strain ϵ . The relation between them can be approximately expressed by

$$t = a/\epsilon + b.$$

(2) Total misorientation β within a original grain increases with an increase of pre-strain ϵ independently of the original grain size.

(3) Excess dislocation density of sub-boundary D_b increases proportionally to the square of pre-strain ϵ , and at a definite pre-strain, it is higher in fine-grained specimens than coarse-grained. The relation can be written in the form,

$$D_b = a'\epsilon^2 - b'.$$

(4) Flow stress increases parabolically with the inverse square root of sub-grain size; and so Petch's relation does not hold true. Not only sub-grain size but also misorientation across the sub-boundary has a great influence on the flow stress.

(5) Flow stress σ_f increases proportionally

to the square root of excess dislocation density D_b ;

$$\sigma_f = \sigma_0 + \alpha' G b D_b^{1/2},$$

where the value of α' for 0.2% flow stress is 0.39. This value is in good agreement with that obtained from the equation by Li⁽¹³⁾ who introduced a mechanism by which dislocations could be generated from sub-boundary.

(6) In conclusion, the effect of sub-structure on flow stress appears to be better explained in terms of the work-hardening theory than Petch's.

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