Evaluation of Microplastic Flow Stress in Copper Alloys from Amplitude-Dependent Internal Friction^{*1}

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Stress-strain relations are evaluated from amplitude-dependent internal friction in polycrystalline solid solution copper alloys. The flow stress in microplastic strain range much below the yield point is examined for eight kinds of specimens, prepared by alloying commercial-grade pure copper with 0.3 at% solute atoms (aluminum, silicon, nickel, gallium, germanium, indium, tin and gold). Internal friction is measured at room temperature under atmospheric pressure using the free-decay method of flexural resonant vibration with both free ends around 600 Hz. The flow stress of Cu-Sn alloys required to cause the plastic strain of 1×10^{-9} is about 440 times larger than that of pure Cu, while that of Cu-Ni alloys only 6 times larger. Thus the flow stress evaluated from amplitude-dependent internal friction reflects sensitively the change of solute elements. The flow stress is examined in terms of the misfit parameters between solute and solvent atoms proposed by Fleischer, and it is shown that the flow stress in microplastic strain range is controlled by the elastic interactions between screw dislocations and solute atoms in solid solution copper alloys.

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1. Introduction

Since amplitude-dependent internal friction is caused by dislocation motion in microplastic strain range, we can observe the phenomena related to dislocation motion through the measurement of the internal friction. Particularly, plastic strain corresponding to dislocation motion is evaluated as a function of applied stress,¹⁾ and thus the measurement may be applied to the evaluation of strength of materials.²⁻⁶⁾ It is emphasized that the strength test by the internal friction measurement is non-destructive because internal friction is measured during elastic resonant vibration of a specimen. Furthermore, the detection of mechanical properties by the internal friction measurement is quite sensitive to the grain size,⁷⁾ the concentration⁸⁻¹⁰⁾ and the element of solute^{4,6)} in comparison with usual strength tests such as a tension test.

The dependence of solid solution hardening on the kind of solute elements has been studied systematically by Fleischer¹¹⁾ in copper alloys through the yield stress obtained by a tension test. He showed the substantial correlation between the rate of solid solution hardening and the elastic interaction force between screw dislocations and solute atoms. However such systematic studies on hardening in terms of solute elements are still limited in number. In general the yield stress is determined not only by the dislocation motion overcoming the resistance due to solute atoms but also by the multiplication rate of dislocations. Therefore it is reasonable to examine the correlation between the elastic interaction force and the solid solution hardening evaluated by the experiments where the multiplication of dislocations does not take place.

In the present study, we investigate stress-strain relations

from the data of amplitude-dependent internal friction and evaluate the flow stress in the strain range much below the yield point in polycrystalline solid solution copper alloys, where the flow stress is controlled only by the motion of dislocations but not by the multiplication of dislocations. The concentration of solute is fixed at 0.3% to examine precisely the effect of solute elements on flow stress. The flow stress evaluated from internal friction is examined using the misfit parameters between solute and solvent atoms proposed by Fleischer¹¹⁾ and the character of the dislocations which determine the flow stress is discussed.

2. Experiments

Solid solution copper alloys were prepared by melting commercial grade oxygen-free copper (purity; 99.99%) together with 0.3 at% solute atoms (aluminum, silicon, nickel, gallium, germanium, indium, tin and gold) in a smelting furnace substituted by argon gas. The concentration of solute evaluated by the X-ray fluorescence analysis included error within 10% of the nominal concentration. The specimens were cut into the rectangular shape with dimension of $1.0 \times 10 \times 80 \text{ mm}^3$. They were annealed in vacuum at 973 K for 86.4 ks, and then furnace cooled. The average grain size was about 150 μm in each specimen.

Internal friction was measured at room temperature under atmospheric pressure using the free-decay method of flexural resonant vibration with both free ends around 600 Hz. A small piece of iron foil ($8 \times 4 \times 0.02 \text{ mm}^3$) was attached to both ends of the specimen in order to excite effectively the vibration of a specimen by an electromagnetic force and to monitor sensitively the vibration by an induced current. After steady-state vibration for 60 s, driving signal was turned off and then a free-decay curve was recorded into a computer through an analogue-digital converter. Internal friction was determined from the logarithmic decrement of the free-decay curve. When internal friction depends on strain amplitude,

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the logarithmic decrement also varies during the decay of vibration. Thus, measuring continuously the logarithmic decrement at each moment during one run of decay, we obtained the continuous curve of amplitude-dependent internal friction as a function of strain amplitude.

It is notable that since strain is inhomogeneous in the vibrating specimen, the maximum value of strain amplitude $\varepsilon_{\max}$ is chosen as an experimental variable, which is determined from a direct measurement of the displacement amplitude in the middle of the specimen, and the logarithmic decrement δ_{av} means the internal friction averaged over the whole specimen.

3. Results

Figure 1 shows the measured internal friction δ_{av} against the maximum strain amplitude $\varepsilon_{\max}$ in polycrystalline solid solution copper alloys, together with that in polycrystalline pure copper (99.99%). The apparent amplitude dependence of internal friction was detected in most specimens but Cu–Sn alloys showed very slight dependence. The curves of the amplitude dependence were reproducible when successively measured under the same conditions. Such reproducibility suggests that no multiplication of dislocations occurs during measurement; the measurement is non-destructive. Scattering of the curves among different specimens of the same composition was within 20% in strain amplitude. As can be seen in Fig. 1, alloying causes the decrease in the internal friction and the suppression of the amplitude dependence. This would be referred to the decrease in mobility of dislocations due to solute atoms.

4. Discussion

4.1 Data analysis

Since strain amplitude differs at each part of a specimen, the quantity measured as δ_{av} is the volume average of the intrinsic internal friction δ arising from each part vibrating with the homogeneous strain amplitude ε_0 . Note that ε_0 is a func-

tion of position in a specimen. We should calculate the intrinsic internal friction $\delta(\varepsilon_0)$ from the measured internal friction $\delta_{\text{av}}(\varepsilon_{\max})$ in order to investigate stress-strain relations. Here, for convenience, we may express an experimental curve as

$$\delta_{\text{av}}(\varepsilon_{\max}) = A\varepsilon_{\max}^n + B, \quad (1)$$

where A , B and n are constants. This expression is applicable to a variety of metals and alloys, except for highly pure monocrystals, as discussed by Lebedev.¹²⁾ In the present paper, the constants A , B and n were determined by a method of least squares. The error of mean square between experimental and fitting curves is sufficiently small (8.40×10^{-6} – 7.15×10^{-5}), but somewhat large for Cu–Ni (3.75×10^{-4}). Then the intrinsic internal friction $\delta(\varepsilon_0)$ is given as follows¹³⁾

$$\delta(\varepsilon_0) = AK(n)\varepsilon_0^n + B, \quad (2)$$

where $K(n)$ is the characteristic function to the fundamental flexural vibration mode and is represented using the gamma-function Γ as

$$K(n) = \frac{\sqrt{\pi}}{2} \frac{n+3}{3} \Gamma\left(\frac{n+4}{2}\right) / \Gamma\left(\frac{n+3}{2}\right).$$

The intrinsic internal friction δ can be related formally to plastic strain ε_p due to dislocation motion. Based on the friction model of dislocation motion, which has been observed in some experiments^{14–16)} and also theoretically established at normal temperature,¹²⁾ the plastic strain is expressed as the following form;¹⁾

$$\varepsilon_p(\sigma) = \frac{\sigma}{E} \left[\frac{1}{4} \delta(\sigma/2) + \frac{1}{2} \int_0^{\sigma/2} \frac{\delta(\sigma_0)}{\sigma_0} d\sigma_0 \right], \quad (3)$$

where E is Young's modulus. Since the strain amplitude ε_0 can be replaced by σ_0/E when ε_p is much smaller than the total strain, the substitution of (2) to (3) gives a simple expression¹⁾

$$\varepsilon_p(\sigma) = A \frac{n+2}{2^{n+2}n} K(n) \left(\frac{\sigma}{E} \right)^{n+1}. \quad (4)$$

Although the strain due to elastic resonant vibration includes only a quite little amount of the plastic strain, the procedure as described above makes it possible to evaluate the plastic strain as a function of the stress from internal friction data.

Figure 2 shows the relation between ε_p and σ obtained from the data in Fig. 1, corresponding to the stress-strain curve in microplastic strain range, where the logarithm of stress is plotted on the abscissa. Although the value of ε_p is of the order of 10^{-9} which is too small to be detected in usual tension tests, microplastic flow indeed occurs even at stress one or two orders of magnitude lower than the macroscopic yield stress. The fraction of the plastic strain to the total strain is only 0.001% (Cu–Sn) and 0.1% (pure Cu) even at maximum. The curves for alloys lie in larger stress range of Fig. 2 than that for pure copper. This indicates a solid solution hardening. The stress at $\varepsilon_p = 1 \times 10^{-9}$ in Fig. 2, from now on referred to as the microplastic flow stress, is 0.0249 (pure Cu), 0.149 (Cu–Ni), 0.596 (Cu–Ga), 0.606 (Cu–Al), 0.814 (Cu–Si), 0.966 (Cu–Ge), 2.39 (Cu–Au), 3.82 (Cu–In) and 10.9 MPa (Cu–Sn). The microplastic flow stress of Cu–Sn alloys is about 440 times larger than that of pure Cu, while that of Cu–Ni alloys only 6 times larger. Thus the strength evaluated from amplitude-dependent internal friction reflects

Fig. 1 The measured internal friction δ_{av} expressed as a function of the maximum value of strain amplitude $\varepsilon_{\max}$ in various copper alloys with 0.3 at% solute atoms.

Fig. 2 The microplastic strain ε_p expressed as a function of stress σ in various copper alloys with 0.3 at% solute atoms. The curves are obtained from the data in Fig. 1.

very sensitively the change of solute elements.

4.2 Character of dislocations determining the microplastic flow stress

Examination into the dependence of solid solution hardening on solute elements may reveal elementary mechanism of plastic deformation, such as the character of dislocations determining flow stress. According to Fleischer,¹¹⁾ the elastic interaction force between a dislocation and a substitutional solute atom is proportional to a misfit parameter $|\varepsilon'_G - \alpha\varepsilon_a|$, where α is a constant, and ε_a and ε'_G are the misfit parameters of atomic size and shear modulus between solute and solvent atoms, that is,

$$\varepsilon_a = \frac{1}{a} \frac{da}{dc} \quad \text{and} \quad \varepsilon'_G = \frac{1}{G} \frac{dG}{dc} \left(1 + \frac{1}{2G} \left| \frac{dG}{dc} \right| \right)^{-1},$$

respectively, and a , G and c are the lattice parameter, the shear modulus and the concentration of solute in alloys. The value of α must be more than 16 for the edge dislocation interactions, while less than 16 for the screw dislocation interactions. Dislocations should overcome this interaction force due to solute atoms even in the measurement of amplitude-dependent internal friction, because there are many solute atoms around vibrating dislocations, taking account of the solution concentration of 0.3%. Thus, hardening of alloys by various solutes should be a monotonic increasing function of $|\varepsilon'_G - \alpha\varepsilon_a|$, the value of α indicating whether the flow stress is controlled by edge or screw dislocations.

In the examination in terms of the screw dislocation interactions, we adopted the value of 3 for α , following the case of a tension test.¹¹⁾ Figure 3 shows the dependence of the rate of solid solution hardening $d\sigma/dc$ on the misfit parameter $\varepsilon_s = |\varepsilon'_G - 3\varepsilon_a|$ for the alloys in Fig. 2 except for Cu–Au of which the misfit parameters are not obtained. As can be seen in Fig. 3, the rate of hardening $d\sigma/dc$ almost monotonically increases as the misfit parameter ε_s increases. On the other hand, we adopted the lower limit value of 16 for α representing the edge dislocation interactions. The relation of the rate

Fig. 3 The rate of solid solution hardening $d\sigma/dc$ in copper alloys against the misfit parameter $\varepsilon_s = |\varepsilon'_G - 3\varepsilon_a|$ between solute and solvent atoms corresponding to the elastic interaction between screw dislocations and solute atoms. The rate of solid solution hardening is evaluated from the stress required to cause the plastic strain of 1×10^{-9} in Fig. 2.

Fig. 4 The rate of solid solution hardening $d\sigma/dc$ in copper alloys against the misfit parameter $\varepsilon_e = |\varepsilon'_G - 16\varepsilon_a|$ between solute and solvent atoms corresponding to the elastic interaction between edge dislocations and solute atoms. The rate of solid solution hardening is evaluated from the stress required to cause the plastic strain of 1×10^{-9} in Fig. 2.

of hardening $d\sigma/dc$ to the misfit parameter $\varepsilon_e = |\varepsilon'_G - 16\varepsilon_a|$ is somewhat scattered, as shown in Fig. 4. We calculate a correlation coefficient r between $d\sigma/dc$ and $|\varepsilon'_G - \alpha\varepsilon_a|$ to determine the most suitable value for α . The coefficient r is 0.964 for Fig. 3 and 0.914 for Fig. 4. The maximum of r , 0.969, is attained at $\alpha = 1.27$, and for α greater than this the coefficient r decreases asymptotically to a constant of 0.820. This means that microplastic flow stress in solid solution copper alloys is determined by the stress required to move screw dislocations rather than edge dislocations.

4.3 Characteristics of internal friction measurements as a strength test

The stress-strain relations of copper alloys have been evaluated in the microplastic strain range through the measurement of amplitude-dependent internal friction. The microplastic flow stress read out from the stress-strain curves in Fig. 2 ranges over two orders of magnitude for the eight kinds of copper alloys in spite of the same concentration, in striking contrast with the macroplastic yield stress in tension tests which varies, in general, by at most several factors of magnitude in solid solution alloys. In addition, the rate of solid solution hardening $d\sigma/dc$ has a strong dependence on the misfit parameter ϵ_s ; the slope in Fig. 3 is 2.92, which is twice of that in tension tests, $3/2$.¹¹⁾ These differences imply that the multiplication process of dislocations is insensitive to solute atoms, because the multiplication takes place only in a macroplastic strain range.^{4,6)} Thus, the strength test by internal friction measurement is very sensitive to the composition of alloys. It is also valuable that this strength test is non-destructive. The prediction of yield stress is possible without destruction of specimens from the microplastic flow stress, if the correlation between microplastic flow stress and yield stress is sufficiently proved by further experiments in a variety of metals and alloys.

The examination into the microplastic flow stress in terms of the misfit parameters showed that microplastic flow stress in solid solution copper alloys is controlled by the elastic interaction force between screw dislocations and solute atoms. The yield stress evaluated by a tension test, in other words the macroplastic flow stress, is also determined by screw dislocations interactions in copper alloys, according to Fleischer.¹¹⁾ However, although the character of dislocations responsible for the flow stress is the same between the micro- and the macroplastic strain ranges, there is a substantial discrepancy in the interpretation of the flow stress. The monotonic increase of the microplastic flow stress to the screw dislocation interactions suggests that screw dislocations can move more easily than edge dislocations, because amplitude-dependent internal friction reflects the transition from elastic to plastic deformation and the dislocations hard to move do not contribute to the amplitude dependence. On the other hand, monotonic increase of the macroplastic flow stress means that screw dislocations are hard to move, because macroplastic yield takes place as a result of long-range motion of both edge and screw dislocations.

The cause of this discrepancy still remains question. However, a possible cause is attributed to the difference in con-

dition of dislocations under measurement. Since the specimen is well annealed before measurement, dislocations vibrate around the equilibrium configuration accompanied with solute atoms in internal friction measurement; edge dislocations would be still locked owing to the Cottrell effect. On the other hand, dislocations are released from the Cottrell locking and begin long-range motion below the yield point in a tension test. It is possible that the released edge dislocations move more easily than screw dislocations. Another cause of the discrepancy is probably due to the multiplication of dislocations. The yield stress contains the stress needed to multiply dislocations, while the microplastic flow stress does not. Since the multiplication is not taken into account in the determination of the misfit parameters ϵ_e and ϵ_s , the misfit parameters should be employed for the examination not into the yield stress but into the microplastic flow stress.

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