

# Interaction between Dislocation and Copper Particles in Fe–Cu Alloys

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The strengthening mechanism due to copper (Cu) particles was discussed in terms of the interaction between dislocation and Cu particles in aged Fe–Cu alloys. Since Cu particles are softer than the iron matrix, its interaction with dislocation is different from that with the Orowan mechanism. The moving dislocations can cut the soft Cu particles and pass through them when the bowing angle reaches some critical value ( $\theta_c$ ), and the precipitation strengthening due to Cu particles is expressed as a function of mean particle spacing ( $\lambda$ ) and the  $\theta_c$  ( $\pi/2$  gives Orowan stress). The  $\theta_c$  increased with increasing the size of Cu particles and reached  $\pi/2$  when the Cu particle size became 70 nm. This means that the precipitation strengthening due to Cu particles is dependent on not only  $\lambda$  but also the Cu particle size, and 70 nm is the minimum Cu particle size for obtaining the Orowan stress.

KEY WORDS: Fe–Cu alloy; precipitation strengthening; Cu particle; bowing angle of dislocation; mean particle spacing; particle size; cutting mechanism; Orowan mechanism.

## 1. Introduction

A dispersion of second-phase particles within the matrix is very effective for strengthening structural materials because the moving dislocations is pinned by the particles. This is known as the dispersion strengthening or the precipitation strengthening (when the second phase is precipitate). Carbide particles are often used as the second-phase particles in steels such as secondary hardening steels<sup>1)</sup> and heat-resistant steels<sup>2–4)</sup>. In general, the carbide particles are much harder than the iron matrix, thus, dislocations by-pass these particles, and the strength can be estimated as the conventional Orowan stress as a function of mean particle spacing<sup>5)</sup>.

Fe–Cu alloys are also well known as the steels which exhibit precipitation strengthening through the precipitation of fine Cu particles within the ferrite matrix. However, the interaction between Cu particles and dislocations is significantly different from the case of the Orowan mechanism. Since the Cu particles are softer than the iron matrix<sup>6)</sup>, the dislocations can cut and pass through the Cu particles. Russell *et al.*<sup>7)</sup> have proposed a theory of strengthening in Fe–Cu alloy based on differing Elastic modulus and reported that the theoretical strengthening agrees with experimental strength data. However there is no experimental evidence with respect to the interaction between Cu particles and dislocation in their study. So far, the exact mechanism of the interaction has not been clarified, although numerous other studies on the strengthening mechanism<sup>8–11)</sup> have been carried out for Cu-bearing steels. For the application of the Cu-bearing steels in practical uses, it is important to

determine such structural factors affecting the strength and control them so as to obtain the required properties.

In this study, precipitation strengthening due to Cu particles was related to the interaction between Cu particles and dislocations, focusing on the bowing angle of dislocation at Cu particles, and then the increment of strength due to precipitation of Cu particles was formulated in over-aged Fe–Cu binary alloys.

## 2. Experimental Procedure

Fe–(1~3)mass%Cu alloys (Cu steels) were used for investigating the mechanism of strengthening by precipitated Cu particles. Fe–0.05%Nb–0.02%C alloy (Nb steel) and commercial low alloy structural steel (SNCM420) were also used as examples in which Orowan mechanism can be applied for estimating strength. Chemical compositions of the steels are listed in **Table 1**. Ingots of the steels were produced with induction furnaces, and then hot-rolled about 20 mm thick plates after holding at 1 273 K for 3.6 ks. Specimens were subjected to solution treatment in Ar gas atmosphere under the conditions of 1 273 K–1.8 ks for the 1% Cu steel, 1 473 K or 1 573 K–1.8 ks for the others, fol-

**Table 1.** Chemical compositions of steels used. (mass%)

	Cu	C	N	Si	Mn	P	S	others	Fe
1Cu steel	0.99	0.007	0.0016	<0.01	0.006	0.001	0.0013	—	bal.
2Cu steel	1.98	0.007	0.0018	<0.01	0.008	0.001	0.0010	—	bal.
3Cu steel	2.98	0.007	0.0023	<0.01	0.007	0.001	0.0011	—	bal.
Nb steel	—	0.020	0.0016	0.02	0.19	0.004	0.010	Nb:0.048	bal.
SNCM420	—	0.21	—	0.24	0.57	—	—	Ni:1.59	bal.

lowed by water-quenching. The quenched specimens were aged at 773~973 K for 0.06~243.6 ks to make Cu or carbide precipitate within ferrite matrix.

Microstructures were observed with an optical microscope (OM) and transmission electron microscope (TEM). The bulk specimens for OM observation were chemically etched with 3% Nital etchant. Thin foil specimens for TEM observation were prepared by jet-polishing method using the solution of 10% perchloric acid and 90% acetic acid. Hardness was represented by the average of five measurements in Vickers hardness test (load 9.8 N). Mean particle spacing;  $\lambda$  was calculated with the Eq. (1).

$$\lambda = 1.25(\pi \bar{d}_p^3 / 6f \bar{d}_p) / (\pi \bar{d}_p^2 / 4 \bar{d}_p) \dots \dots \dots (1)$$

Where  $d_p$  is diameter of the precipitates and  $\bar{d}_p$ ,  $\bar{d}_p^2$  and  $\bar{d}_p^3$  denote the averages of  $d_p$ ,  $d_p^2$ ,  $d_p^3$ , respectively. The  $f$  is volume fraction of the precipitates estimated from equilibrium phase diagrams<sup>12)</sup>. Tensile tests were carried out with an Instron-type testing machine at the initial strain rate of  $1.67 \times 10^{-3} \text{ s}^{-1}$  for cylindrical test pieces of  $\phi 3 \times 10 \text{ mm}$  gauge dimension.

**3. General Theory of Precipitation Strengthening**

When a moving dislocation is pinned by dispersed precipitates as shown in **Figure 1**, shear stress ( $\Delta\tau$ ) required for bowing the dislocation with angle  $\theta$  is expressed by the Eq. (2).

$$\Delta\tau = (\alpha Gb / \lambda) \sin\theta \dots \dots \dots (2)$$

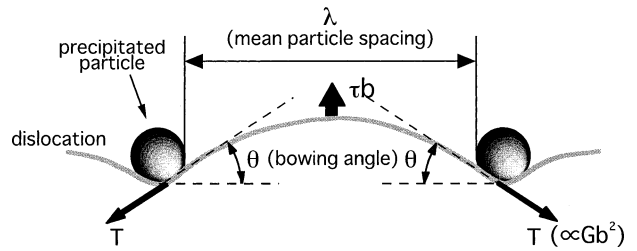
Where  $b$  is the magnitude of the Burgers vector,  $G$  is the shear modulus of matrix and  $\alpha$  is a coefficient depending on the material. Tensile stress ( $\Delta\sigma$ ) is given by multiplying Taylor factor to the shear stress and expressed using another constant ( $\beta$ ) as follows:

$$\Delta\sigma = (\beta Gb / \lambda) \sin\theta \dots \dots \dots (3)$$

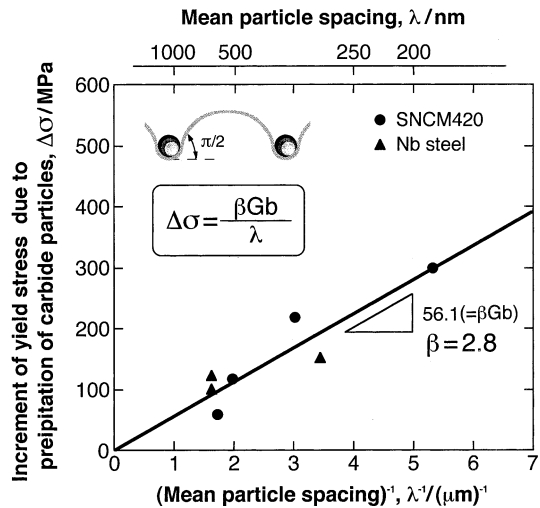
The constant;  $\beta$  can be determined by means of tensile testing in carbon steels with carbide particles because  $\theta$  is regarded to be  $\pi/2$ . **Figure 2** shows the relation between mean carbide particle spacing and increment of yield stress due to carbide particles in the Nb steel and the SNCM420. The increment of stress is proportional to the reciprocal of mean particle spacing. The slope of the line corresponds to the  $\beta Gb$  in the Eq. (3), and it was determined to be 2.8. Consequently, tensile stress of precipitation-strengthened steels is given by the following equation.

$$\Delta\sigma = (2.8 Gb / \lambda) \sin\theta \dots \dots \dots (4)$$

If the moving dislocation passes through dispersed particles before the  $\theta$  reaches  $\pi/2$  by cutting<sup>7)</sup> or climbing<sup>13)</sup> mechanism, the increment of stress due to precipitation strengthening ( $\Delta\sigma$ ) should be given by substituting the  $\theta$  at which the dislocation has just passed through the particles into the Eq. (4). We defined the  $\theta$  as the critical bowing angle;  $\theta_c$ . The  $\theta_c$  reflects the interaction between Cu particles and dislocation and has an influence on the behavior of precipitation strengthening. Since Cu particles are softer than the iron matrix, the  $\theta_c$  of Cu particles should be smaller than  $\pi/2$ . In this study, the strengthening mechanism due to Cu particles was discussed in terms of the  $\theta_c$  and its particle



**Fig. 1.** Schematic illustration showing a dislocation bowing out between two particles.



**Fig. 2.** Relation between increment of yield stress due to precipitation of carbide particles and mean particle spacing in Nb steel and SNCM420. Specimens were aged at 973 K after the solution treatment.

size dependence.

**4. Results and Discussions**

**4.1. Microstructure of Cu Steels before Aging Treatment**

**Figure 3** represents optical micrographs of Cu steels which were water-quenched after the solution treatment. All of the as-quenched specimens have ferritic structure with irregular grain boundaries which are characteristic of massive ferrite structure<sup>14)</sup>. The ferrite grain sizes of the 1% Cu, 2% Cu and 3% Cu steels are 80  $\mu\text{m}$ , 170  $\mu\text{m}$  and 50  $\mu\text{m}$ , respectively. In the 2% Cu and the 3% Cu steels, martensitic structure is partially formed as pointed by the arrows. However, strengthening by the martensite results in being negligible after the aging treatment because the dislocation density of martensite becomes quite low through recovery during the aging. Besides, the effect of grain refinement strengthening is also negligibly small compared with precipitation strengthening in the present results (grain size range: 50~170  $\mu\text{m}$ )<sup>15)</sup>. From these facts, it is certain that the increment of strength through aging treatment is only by the precipitation strengthening due to Cu particles.

**4.2. Change in Cu Particle Spacing during Aging Treatment**

**Figure 4** shows changes in hardness of the specimens during the aging at 873 K as a function of aging time.

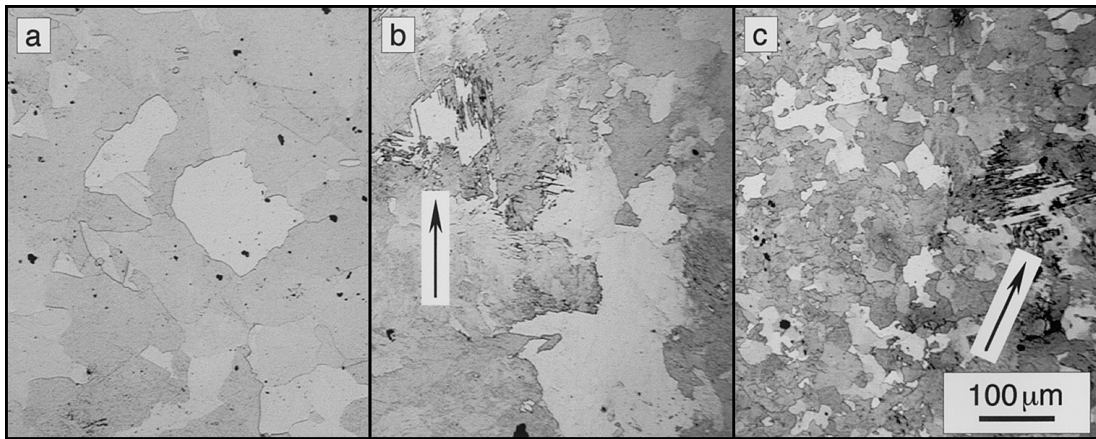


Fig. 3. Optical micrographs of 1% Cu steel (a), 2% Cu steel (b), 3% Cu steel (c). Specimens were water-quenched after the solution treatment.

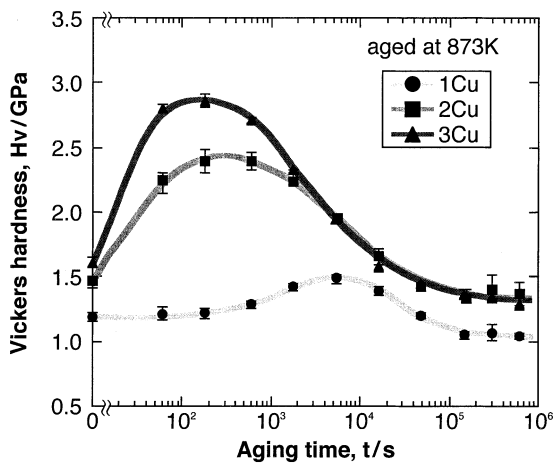


Fig. 4. Changes in Vickers hardness of (1~3)% Cu steels during aging at 873 K.

Typical age hardening and over-aging softening behavior are observed in all specimens. These behaviors are closely related to the behavior of Cu precipitation and growth. It is known that Cu precipitates as b.c.c. clusters which are coherent with the matrix in the early stages of aging<sup>16,17</sup>, and the hardness is significantly influenced by the coherent strain around the Cu particle<sup>11</sup>. On the other hand, in the over-aging stages, coherency at the interface between the Cu particle and the matrix is almost lost<sup>18,19</sup>, thus, precipitation strengthening in over-aged Cu-bearing steels is influenced only by  $\lambda$  and  $\theta_c$  and can be estimated with Eq. (4).

Figure 5 shows an example of TEM observations and a histogram showing size distribution of Cu particles in the 3% Cu steel aged in the over-aging stage at 873 K for 16.2 ks. Cu particles are finely dispersed within the ferrite matrix and the average size of particles is 24 nm. However, the particle size varies in the range from 10 to 50 nm as shown in Fig. 5(b). Strictly speaking, the size distribution should be taken into account for estimating the mean particle spacing. Nakashima's method<sup>20</sup> (Eq. (1)) enables a statistical evaluation of the mean particle spacing considering the size distribution. Figure 6 shows changes in the mean Cu particle size and spacing estimated with Eq. (1) as a function of aging time. The Cu particles gradually ripen with aging time and this leads to the enlargement of mean particle

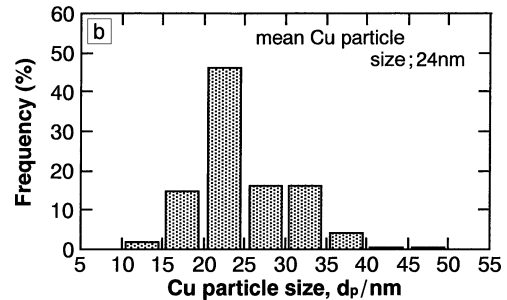
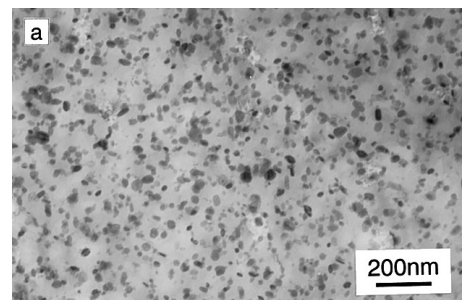


Fig. 5. Transmission electron micrograph (a) and a histogram showing size distribution of Cu particles (b) in 3% Cu steel aged in the over-aging stage at 873 K for 16.2 ks.

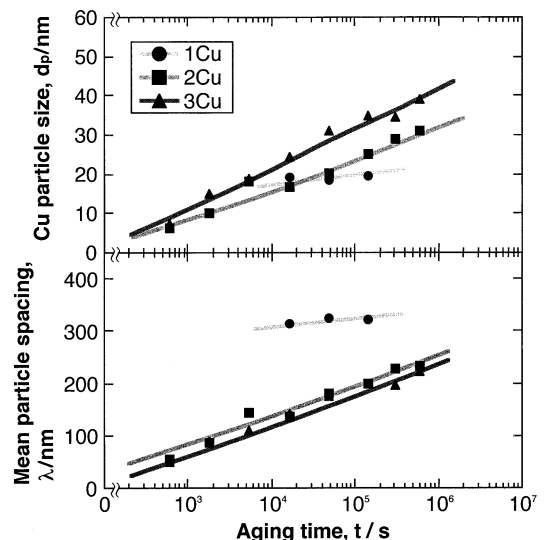


Fig. 6. Changes in the mean Cu particle size and spacing in (1~3)% Cu steels aged at 873 K.

spacing in all specimens. With decreasing Cu content, the mean particle spacing tends to increase. Especially, that of 1% Cu steel is markedly larger than the others because the volume fraction of Cu particles is quite small in the aged 1% Cu steel (0.3 vol.%). With this result, we can easily control the dispersion of Cu particles ( $d_p$  and  $\lambda$ ) through aging time and Cu content.

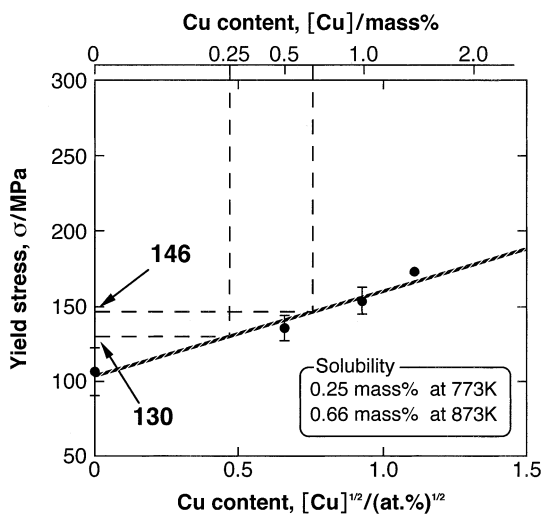
**4.3. Estimation of Increment of Yield Stress due to Precipitation of Cu**

It is generally accepted that yield stress of materials ( $\sigma_{0.2}$ ) consists of two components; thermal stress ( $\sigma^{th}$ ) and athermal stress ( $\sigma^{ath}$ ). The relation among them is expressed as follows:

$$\sigma_{0.2} = \sigma^{th} + \sigma^{ath} \dots\dots\dots(5)$$

In the case of aged Fe–Cu alloys, the thermal stress corresponds to the strength of ferrite matrix including solid solution strengthening due to Cu, and the athermal stress does to the precipitation strengthening due to Cu particles. In order to evaluate the strengthening due to Cu particles, the thermal stress, namely, strength of ferrite matrix must be subtracted from the experimentally measured yield stress. **Figure 7** shows relation between the yield stress and solute Cu content in the (0.5~1.5%) Cu steels which were water-quenched from the solution treatment temperature. These values represents the strength of ferrite matrix including solid solution strengthening due to Cu. Since solubility of Cu is varied with temperature, solid solution strengthening in ferrite is also varied depending on the aging temperature. For example, specimens aged at 873 K contains solute Cu of 0.66%<sup>12)</sup>, thus the strength of ferrite matrix can be estimated at 146 MPa. Consequently, the increment of yield stress due to Cu precipitation ( $\Delta\sigma$ ) in the 873 K aged alloy is given by the Eq. (6).

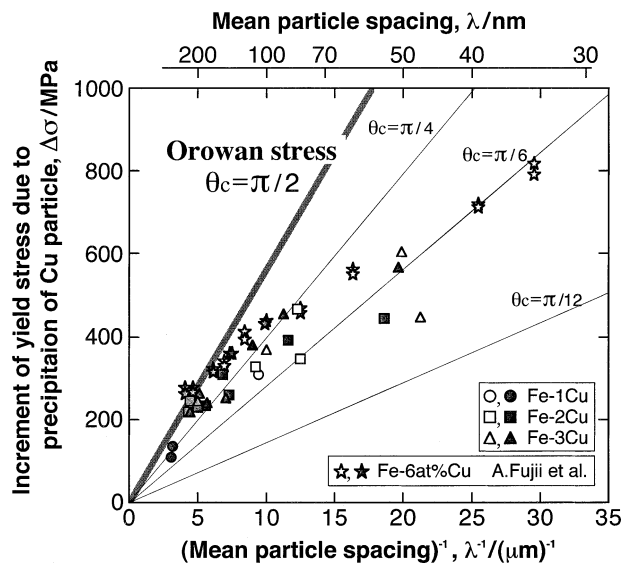
$$\begin{aligned} \Delta\sigma &= \sigma^{ath} = \sigma_{0.2} - \sigma^{th} \\ &= \sigma_{0.2} - 146 \text{ [MPa] (aged at 873 K) } \dots\dots\dots(6) \end{aligned}$$



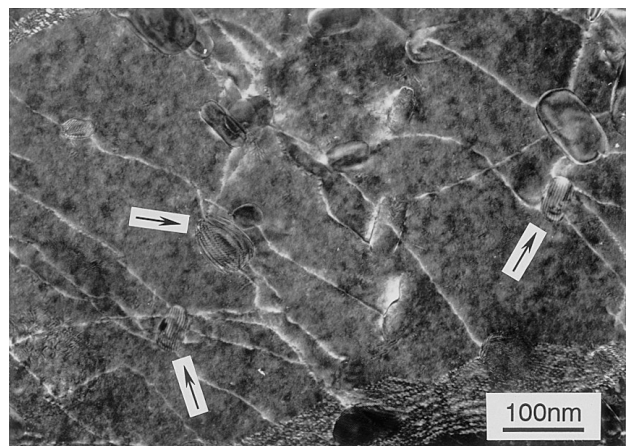
**Fig. 7.** Relation between yield stress and Cu content in the (0.5~1.5%) Cu steels. Specimens were water-quenched after the solution treatment.

**4.4. Strengthening Mechanism due to Cu Particles**

**Figure 8** shows relation between the mean particle spacing and the increment of yield stress due to precipitation of Cu particles which was estimated by the procedure shown in the prior chapter. The straight lines denote theoretical value of precipitation strengthening given by the Eq. (4) using various  $\theta_c$ . The measured yield stress increase with decreasing the mean particle spacing. However, the values of the measured stress are smaller than the Orowan stress ( $\theta_c = \pi/2$ ), besides, the gap between the stresses tends to be enlarged with decreasing mean particle spacing. **Figure 9** represents TEM micrograph taken by the weak-beam method in the 3% Cu steel which was tensile-deformed by 0.6%. There is no Orowan loop around the particles, although dislocations are tangling with Cu particles. This result means that the dislocations should have not by-passed but cut the Cu particles. **Figure 10** shows the relation between Cu particle size and the  $\theta_c$  which was estimated from the results of Fig. 8 and the Eq. (4). The  $\theta_c$  constantly in-



**Fig. 8.** Relation between mean particle spacing and increment of yield stress due to precipitation of Cu particles in Fe–Cu binary alloys. Specimens were aged at 773 K or 873 K after the solution treatment. Straight lines are theoretical precipitation strengthening using various critical bowing angle of dislocation ( $\theta_c$ ).



**Fig. 9.** TEM micrograph taken by the weak-beam method in the 3% Cu steel tensile-deformed by 0.6%.

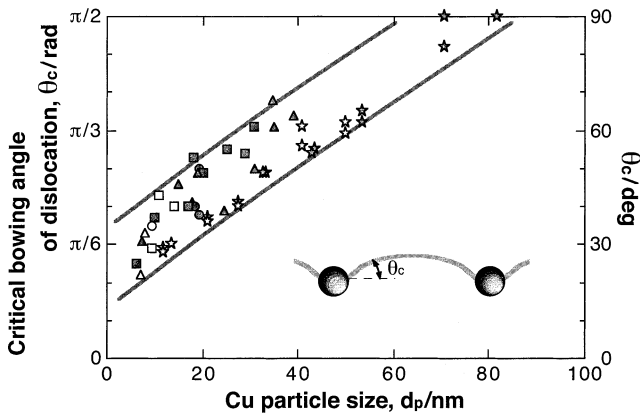


Fig. 10. Relation between Cu particle size and critical bowing angle of dislocation ( $\theta_c$ ) which was estimated from the results of Fig. 8.

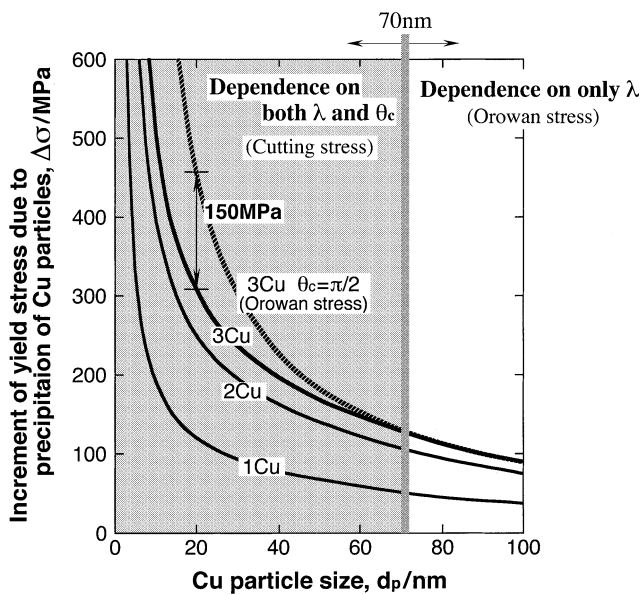


Fig. 11. Relation between Cu particle size and the theoretically calculated increment of yield stress due to precipitation of Cu particles in (1~3)% Cu steels aged at 873 K. Hatched curve is the Orowan stress for the 3% Cu steel.

increases with increasing the particle size, and reaches  $\pi/2$  when the Cu particle size becomes 70 nm. This means that the stress required for the cutting increases with growth of Cu particle and corresponds with the Orowan stress at the critical Cu particle size of 70 nm. When the Cu particle size is smaller than 70 nm, the  $\theta_c$  is approximately expressed by the Eq. (7) as a function of Cu particle size.

$$\theta_c = 24 + 0.92 \times d_p \quad [\text{degree}] \dots\dots\dots(7)$$

Inserting the  $\theta_c$  into Eq. (4) gives the increment of yield stress due to the precipitation of Cu particles and it is expressed as follows:

$$\Delta\sigma = (2.8Gb/\lambda) \sin(24 + 0.92 \times d_p) \dots\dots\dots(8)$$

Figure 11 shows the relation between Cu particle size and increment of yield stress which was calculated with Eq. (8). The Orowan stress is also shown for the 3% Cu steel by the hatched curve. The increment of yield stress due to the precipitation of Cu particles is lower than the Orowan stress when the Cu particle size is less than 70 nm. For example,

the gap between stresses is enlarged to as much as 150 MPa in the 3% Cu steel containing 20 nm Cu particles.

As mentioned above, it was found that the strengthening due to precipitation of Cu particles in the Fe-Cu alloys is dependent on Cu particle size as well as mean particle spacing. Therefore, in order to apply the Fe-Cu alloys for high strength steels, it is important to add sufficient amount of Cu and control not only spacing but also size of Cu particles.

**5. Conclusions**

The strengthening mechanism due to Cu particles was discussed in terms of the interaction between dislocation and Cu particles in aged Fe-Cu alloys, and the following findings were obtained.

(1) The size and spacing of Cu particles ( $d_p$  and  $\lambda$ ) can be controlled with aging time and Cu content in Fe-Cu binary alloys.

(2) When the Cu particle size is less than 70 nm, the dislocations moving during deformation cut Cu particles and pass through them before the bowing angle of dislocation reaches  $\pi/2$ . Therefore, the increment of yield stress due to precipitation of Cu particles ( $\Delta\sigma$ ) is dependent on the Cu particle size as well as the mean particle spacing. This is given by the following equation.

$$\Delta\sigma = (2.8Gb/\lambda) \sin(24 + 0.92 \times d_p) \quad (d_p < 70 \text{ nm})$$

(3) The bowing angle of the dislocation reaches  $\pi/2$  when the Cu particle size is more than 70 nm. In this case, the precipitation strengthening can be evaluated using the equation for Orowan stress expressed as follows.

$$\Delta\sigma = (2.8Gb/\lambda) \quad (d_p > 70 \text{ nm})$$

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