Plastic deformation and dissolution of ϵ -Cu particles by cold rolling in an over-aged particle dispersion strengthening Fe-2mass%Cu alloy

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Abstract

Deformation and dissolution behaviors are important characteristics of soft dispersion particles in steel to improve the deformability of high strength steel sheets by controlling the work hardening/softening and fracture processes associated with particle dispersion strengthening. In this study, the deformation and dissolution behaviors of ϵ -Cu precipitate particles in a ferrite matrix were investigated to understand how relatively soft dispersion particles influence mechanical responses of the steel compared to hard dispersion particles such as alloy carbides. 35nm diameter nearly spherical ϵ -Cu particles were initially elongated along the rolling direction by cold rolling, and then these were partly dissolved into the ferrite matrix as the equivalent strain increased. The dissolution of the Cu particles was suggested to be caused by a dynamic partitioning of Cu atoms from the precipitates into the matrix by dislocation shearing at the particle tip sharpened by severe cold working. Simultaneously, the contribution of the ϵ -Cu particles to the dispersion strengthening appeared to be decreased with increasing dislocation density in the matrix during cold rolling. The dislocation density and other defects in the ϵ -Cu particles also increased and the particle/matrix interfaces were serrated or became indistinct as deformation progressed. The morphology, internal defects, and interface structure changes of fine dispersion particles and their correlation with mechanical responses of the alloy are discussed in reference to an identically processed, hard particles dispersed Fe-V binary alloy.

Keywords

Steel; Strain hardening; Severe plastic deformation; Dissolution of precipitates; Mechanical property

1. Introduction

Historically, steels purposely added a trace amount of Cu, i.e., Cu-bearing steels, benefit from the large age hardenability of Cu particles precipitated in ferrite matrix [1-6]. Thus, the precipitation sequence and structural transformations of Cu particles under various circumstances and their influence on mechanical responses and age hardenability of Cu-bearing steels have been extensively investigated. Multiple commercial Cu-bearing steels demonstrate improved mechanical properties such as tensile strength [7,8], fatigue strength [9], heat resistance [10]. Since Cu forms fine particles in steels by aging treatments, the strength of the steels are expected to be increased by particle dispersion strengthening. However, the Cu particles are categorized as a soft particle relative to other precipitates and dispersion particles such as alloy carbides and nitrides, because its shear modulus is lower than that of iron matrix, and the yield strength of the particle would be also lower compared with typical hard alloy carbides and nitrides for precipitation and particle dispersion strengthening. Thus, the Cu particles would behave considerably differently from those hard dispersion particles, and the uniqueness of soft particle dispersion on mechanical properties of ferritic steels has not been fully discussed yet. In particular, there are only handful studies on the role of soft Cu particles on mechanical responses of cold-worked ferritic materials [11-13].

The Cu particle of interest, face centered cubic (fcc) structured ϵ -Cu, is formed generally in an over aged condition, and it is usually regarded as a soft particle. Therefore, the ϵ -Cu particle exhibits an attractive interaction with dislocations [3], and they are believed to be cut by dislocations during deformation depending on the particle size [3,11-12]. In other words, the Cu particles are continuously plastically deformed during cold working, resulting in changes in the particle morphology and their strengthening effects in highly strained regime.

While a number of studies have been carried out on the yield stress of aged Cu-bearing steels, only few experimental results have been reported on the work hardening behavior of such steels, especially the strength and fracture in a high strain regime. Todaka et al.[14] examined the hardness change of severely plastically deformed Fe-1.5 mass%Cu alloy before and after aging treatment, and found that the former exhibited work hardening while the latter showed work softening. A similar mechanical response was observed in a fatigue tested 1.5 mass%Cu bearing interstitial-free (IF) steel [11], where the maximum stress was gradually decreased during the cyclic loading under a

constant strain. The two examples mentioned above mean the strengthening effect of Cu particles is effective in the early stage of deformation but it would be markedly weakened or diminished after severe plastic deformation or cyclic stress loading. This phenomenon could suggest the number of strength-contributing particles decreases after a certain amount of cold working, i.e., the dissolution of the dispersed particles occurred as reported in cold worked precipitation hardened aluminum alloys [15,16] or in cold drawn pearlite wires [17]. To the best of our knowledge, there is no direct experimental evidence supporting the dissolution of Cu particles by heavy cold work in Cu bearing steels. The deformation and dissolution behaviors are important characteristics of soft particles that are essentially different from hard particles, and it would be possible to improve deformability of high strength steel sheets by utilizing these phenomena effectively for controlling the work hardening (or softening) and fracture processes.

In this study, the work hardenability and Cu particles' morphology and microstructure evolutions during heavy cold rolling were investigated by using an over-aged Fe-2 mass%Cu steel in which fcc ϵ -Cu precipitate particles are uniformly dispersed in the ferrite matrix. The morphology, internal defects, and interface structural changes of fine dispersion particles were observed with HRTEM to clarify the dissolution behavior of plastically-deformed Cu particles, and then an estimation for the amount of dissolved ϵ -Cu particles was carried out using X-ray diffraction technique by measuring the changes in lattice parameter of the matrix phase as a function of the equivalent strain. Finally, the correlation between Cu deformation and mechanical responses are discussed in reference to an identically processed Fe-V binary alloy containing hard dispersion particles.

2. Experimental procedure

An Fe-Cu alloy (Cu steel) was molten under an argon gas atmosphere, cast to 50 kg ingot, and then hot rolled to 12 mm thick plate at 1473 K. A referential material, Fe-V-C alloy (VC steel) was also prepared through the same process. The chemical compositions of these steels are listed in Table 1. In order to maintain the particle size to be similar in both steels, heat treatment process was individually optimized. The Cu steel was solution-treated at 1173 K for 0.6 ks in the austenite single phase region, and then water-quenched to obtain massive ferritic structure with supersaturated Cu.

The specimens were then annealed at 923 K for 180 ks to disperse fcc ϵ -Cu precipitate particles uniformly in the ferrite matrix. On the other hand, the VC steel was firstly solution treated at 1473 K for 1.8 ks followed by water quenching to obtain martensitic structure, and then tempered at 873 K for 1.8 ks to disperse VC carbide particles in the martensite matrix. After that, the VC steel was reheated to 1273 K in (austenite + VC) two-phase region, kept for 60 s at this temperature, and then furnace-cooled to take place ferritic transformation. The volume fraction of Cu particles in the Cu steel, estimated from the solubility of Cu in ferrite at 923 K, is 1.4 vol.%, while that of VC particles in the VC steel is calculated to be also 1.4 vol.% by assuming that the carbon is fully consumed for VC carbide formation. The specimens of the Cu and VC steels were then cold-rolled with the thickness reduction ranging between 5 and 90%.

The microstructure was observed by both optical and transmission electron microscopy (TEM) techniques. TEM and scanning TEM (STEM) observations were carried out using JEOL JEM-2010 operated at 200 kV, and FEI Titan 80-300 operated at 300 kV. The lattice parameters and dislocation densities were measured by Rigaku Co. Ltd., RINT2100 powder X-ray diffractometer, using the Cu-K α radiation generated at 40kV, 40mA. The dislocation density, ρ , was estimated from Eq. (1) [18], where b is the burgers vector (0.25 nm) and ϵ is the local strain obtained by Williamson-Hall method [19].

$$\rho = (14.4 \times \epsilon^2) / b^2 \quad (1)$$

Differential scanning calorimetry (DSC) analysis was conducted with SHIMAZU DSC-50 under Ar gas atmosphere with a heat-up rate of 10 K / min from room temperature to 993 K to detect the precipitation and growth of Cu particles. DSC curves are presented as a heat flow difference between the first run and the second run.

3. Results

3.1 Difference in work hardening behavior during cold rolling between hard and soft particle dispersion steels

Figure 1 shows TEM bright field images of Cu (ϵ -Cu) and VC particles in undeformed (0% cold-rolled) alloys, and the attached two histograms show their size distributions. The average sizes of these particles were nearly identical: 35 nm for ϵ -Cu and 37 nm for VC. The morphology of these

particles were also nearly identical, however the Cu particles tended to be slightly elongated along $\langle 111 \rangle_\alpha$ directions because they grew with holding the Kurdjumov-Sachs (KS) relationship with the ferrite matrix [20]. **Figure 2** shows the changes in the Vickers hardness (a) and the dislocation density (b) as a function of reduction of thickness during cold rolling of these steels. The experimentally measured values of a dispersion particle free pure iron (interstitial free (IF) steel) are also shown in this figure as a reference. The hardness comparison of three undeformed specimens indicate that both the Cu and VC steels were significantly strengthened by the particle dispersion strengthening compared with the dispersion particle free pure iron. With increasing the equivalent strain by cold rolling, the hardness and dislocation density were monotonously increased. The degree of particle dispersion strengthening, namely, the hardness difference between the particle dispersion steels and the pure iron, remained effective after cold rolling, or even became prominent with increasing the equivalent strain. The dislocation accumulation in relatively low strain regime ($\epsilon_t < 1.0$) took place most rapidly in the VC steel, followed by the Cu steel and then the pure iron (fig. 2(b)). On the other hand, the dislocation density of the Cu steel in relatively high strain regime ($\epsilon_t > 1.0$) became higher than that of the VC steel.

To evaluate the effect of particle dispersion strengthening itself in these two cold-rolled alloys, the measured hardness was re-plotted as a function of square root of the dislocation density as shown in **Fig. 3**. In this plot, all steels exhibited a liner relationship, i.e., the Bailey-Hirsch relationship [21]. This means that the dislocation strengthening due to dislocation accumulation is effective even in the particle dispersion strengthened steels. Since the pure iron does not contain dispersion particles, the dislocation strengthening is solely responsible to the hardness increments of this sample. Therefore, the hardness difference between the pure iron and the VC steel or the Cu steel at each equivalent strain value was attributed to the strengthening by dispersed particles. The hardness of the Cu steel was nearly identical to that of the VC steel in the early stage, and then gradually deviated with increasing dislocation density towards the lower hardness side. This suggests that the strengthening effect by the VC carbide particles was maintained even after heavily cold rolled, while that by the Cu particles diminished. The morphology of Cu particles changed from spherical to thin needle-like shape, and the volume fraction of the Cu particles was decreased by the cold rolling, as described in the following sections. This might make the resistance against deformation lower.

However, under the cold rolling reduction up to 90% employed in this study, we could not see the work softening phenomenon as reported in the previous studies by different severe plastic deformation methods [11,14].

3.2 Change in morphology of Cu particles by cold rolling

Figure 4 shows TEM images of 70% cold-rolled (the equivalent strain 1.39) Cu and VC steels observed from the transverse direction (TD) of sheet specimens. The hard VC particles remained spherical shape indicating no morphological change, while the Cu particles were markedly elongated and transformed to ellipsoid shape. **Figure 5** shows histograms providing the aspect ratio of Cu and VC particles measured from more than 300 particles randomly selected in the 70% cold-rolled specimens. The aspect ratio of the Cu particles was approximately 1 in undeformed sample. The aspect ratio of the Cu particles in the 70% cold-rolled sample showed a broad distribution, ranging from 1 up to 15. From this result, it is undoubtedly asserted that the Cu particles were anisotropically plastically deformed by cold rolling, although how much each Cu particle being deformed was significantly scattered. Suppose the Cu particles was plastically deformed by 70% that was equal to the amount of macroscopic deformation introduced by this particular cold rolling step, the aspect ratio of the Cu particle should be around 11, whereas the measured average aspect ratio was 6.9. Thus, the experimentally observed value was significantly smaller than the theoretically expected value even by assuming some smaller numbers represent the cross-section of the elongated particles due to the projection nature of regular TEM images [22]. This suggests that the Cu particles possess some resistance against plastic deformation, that is, they would behave as an obstacle against the dislocation motion in the ferrite matrix, thus could act as a strengthener. On the contrary, the VC particles never been deformed and always behave as rigid body, thus, the pinning force and the dislocation storing effect of them could be much stronger than that of the Cu particles.

3.3 Internal structure of plastically-deformed Cu particles

ϵ -Cu particles were obviously plastically deformed (**Fig. 4**) after 70% cold rolling (the equivalent strain 1.39), and deformation structure including structural defects and dislocations were developed in their interior and the surrounding area. In fact, the ϵ -Cu particles showed bright/dark

stripe contrast in their interior and relatively high density of tangled dislocations in their vicinity. The interior bright/dark stripe contrast suggests that the crystallographic orientation changes locally or very localized strain fields exist in the particles. Further, the atomic structure of a deformed and elongated ϵ -Cu particle observed in the 80% cold-rolled specimen (the equivalent strain 1.85) exhibited that lattice fringes were partially distorted due to the high density dislocations and twins or stacking faults (**Fig. 6**). The interface between elongated particle and matrix was not atomically flat as seen in the hard dispersion / matrix interfaces (marked by the arrows) but irregularly bent. Near the tip of this elongated ϵ -Cu particle (indicated by a dashed circle), the interface was no longer distinguishable, and the particle seemed being vanished or merged with the matrix. The EDS line profiles of Cu K α 1 and Fe K α 1 taken from near the ϵ -Cu particle tip, from the inside of the particle to the ferrite matrix (position of 0 to 30 nm) along the white arrow (**Fig. 7**), represented that the Cu concentration decreased with a hump in the ferrite matrix while the Fe concentration steadily increased. This indicates that the ϵ -Cu/ferrite boundary has become unclear due to the plastic deformation, and the transport or dissolution of Cu from precipitates into ferrite matrix has occurred.

3.4 Quantitative analysis of dissolved Cu by cold rolling

The lattice parameters of the cold-rolled Cu steels were measured by X-ray diffractometry to quantify the amount of dissolved Cu in the ferrite matrix. Since the radius of Cu atom in ferrite (α -Fe) solid solution (effective atomic radius) is larger than that of Fe atom by 18% [23], the lattice parameter of the ferrite matrix would be increased when Cu atoms dissolved into the ferrite matrix. Thus, the increase in Cu concentration in the ferrite matrix could be estimated by measuring the lattice parameter changes as a function of the degree of deformation. To perform quantitative evaluation, a calibration curve was made by measuring the lattice parameter of a series of quenched binary Fe-Cu alloys. **Figure 8** shows the experimentally measured lattice parameters of binary Fe-Cu alloys as a function of Cu concentration. The alloys were prepared by quenching from austenite single-phase region at 1373 K after homogenization by hot rolling and annealing at 1473 K. The lattice parameter, a , and the Cu concentration exhibits a linear relationship expressed by the following equation:

$$a = 0.286751 + 8.1 \times 10^{-5} [\text{mass\% Cu}] \quad (2)$$

Figure 9 shows changes in the lattice parameter of pure iron and the Cu steel (left axis of ordinate) as

a function of the reduction of thickness, plotted together with the concentration change of solute Cu in the Cu steel (right axis of ordinate). The experimental measured values of pure iron are also shown in this figure as a reference. With increasing the equivalent strain, the lattice parameter of the Cu steel was increased while that of the pure iron stayed the same. The lattice parameter of the 70% cold-rolled Cu steel reached 4.9×10^{-5} nm, which was equivalent to the increase in soluble Cu concentration by approximately 0.6 mass%. In other words, approximately one-third of initially precipitated out Cu particles (0.5 vol%) were dissolved back into the ferrite matrix after the 70% cold rolling under the assumption that the lattice parameter change was derived only from dissolution of Cu atoms. It is worth noting that the increase in the lattice parameter of the Cu steel never reached the value expected from the uniform solid solution, and the measured numbers only provide the mean value from the entire specimen. It is possible that the majority of dissolved Cu atoms might be localized in the vicinity of remaining ϵ -Cu particles as mentioned in the previous section.

In order to ensure the dissolution of Cu particles, DSC analysis was performed for the heavily cold rolled Cu steel. If the amount of solute Cu was truly increased by the Cu particle dissolution, reprecipitation should occur by heating the sample during the DSC measurement, and the exothermic peak corresponding to Cu precipitation would become an absolute evidence of the Cu dissolution by cold rolling. **Figure 10** represents the result of DSC analysis of the undeformed Cu steel and the 90% cold-rolled Cu steel (the equivalent strain 2.67). **At first, it was confirmed that the cold-rolled Cu steel does not undergo recrystallization during heating in the course of DSC analysis, and thus, all peaks detected in the DSC curves can be attributed to reactions related with Cu.** A large exothermic peak was observed at around 750 K in both specimens, but this peak was known to be exothermic reaction corresponding to the growth of Cu particles or the phase transformation from 9R to fcc structure [24,25], and not derived from the precipitation of the dissolved Cu atoms. On the other hand, an exothermic peak was seen at around 550 K in the 90% cold-rolled steel, whereas that peak was hardly seen in the underformed sample. This peak should be attributed to the cluster formation that has been observed between 533 and 733 K in a solution-treated Fe-1.4 mass% Cu alloy [24]. Although the peak position seemed to be shifted to lower temperature side likely due to the severe cold work, the exothermic peak dictated that excess solute Cu existed due to the ϵ -Cu particles dissolution by severe cold rolling, and then the solute Cu

formed clusters during the DSC measurement.

4. Discussion

4.1 Mechanism of dissolution of Cu particles with cold rolling

Hutchinson et al. [16] proposes a precipitation dissolution mechanism for GP zones in Al-Cu based alloys by cold work, which shows a dynamic partitioning of Cu atoms from the precipitates into solid solution by dislocation shearing during cold rolling (strain-induced dissolution). In this model, the constituent atoms are mechanically forced to be detached from thermally stable precipitates and mixed into the matrix. This model appears to be valid to explain the dissolution process observed in mechanical alloying of powder materials [26]. On the other hand, in the study of the drawing of a pearlitic steel by Sauvage et al. [27], cementite dissolution is supposed to be caused by the fragmentation of cementite lamellae leading to an increment of its solubility due to the interfacial energy contribution (Gibbs-Thomson effect). This means that the cementite becomes thermodynamically unstable by severe cold work, and thus, carbon atoms move from cementite to an energetically more stable site such as in solid solution (or segregation at dislocations) through the diffusion which can take place even at a low temperature with a slight temperature rising by cold work.

As for the Cu particles treated in this study, here, we suppose the changes in its stability by cold work would follow the Gibbs-Thomson effect. The solubility obtained from the Fe-Cu binary phase diagram, namely, the Cu concentration in the ferrite equilibrated with sufficiently large-sized ϵ -Cu is given by the following equation [28].

$$\log C_{Cu}^{\alpha} = 4.495 - \frac{4627}{T} \quad (3)$$

where C_{Cu}^{α} and T are the equilibrium Cu concentration (mass%) and temperature (K). Now we consider the local Cu solubility at the tip of Cu particles (curvature radius, r). Due to the free energy increase derived from the interfacial energy, it is expressed by the Gibbs-Thomson equation as follows:

$$X_r = X_{\infty} \exp\left(\frac{2\gamma V_m}{RT r}\right) \quad (4)$$

where X_r and X_∞ are the local Cu solubility at the tip and that of large-sized precipitate with a curvature radius of infinity (atomic fraction). γ and V_m are the ferrite/Cu interfacial energy (J/m^2) and the molar volume of the ϵ -Cu precipitate (m^3/mol). R is the gas constant. The interfacial energy is assumed to be 1 J/m^2 in consideration that the coherency is being lost in the case of the ϵ -Cu particles in an over-aged steel. As the other parameters, $V_m = 7.11 \times 10^{-6} \text{ m}^3/\text{mol}$ and $R = 8.314 \text{ J/K}\cdot\text{mol}$ are used for the calculation. **Figure 11** represents the relation between the curvature radius of the tip of deformed Cu particles and the solubility of Cu. In consideration of possible temperature increases by plastic working, calculation results at different temperatures are indicated. The calculation results demonstrate that the solubility of Cu hardly exceeds the initial concentration of 0.5 mass% (equilibrium concentration at the aging treatment temperature of 923 K) unless the temperature is significantly increased. Even when the specimen is heated-up to 473 K during cold rolling, the curvature radius must be reduced to an atomic scale as small as 0.35 nm for increasing solubility to more than the initial concentration to start dissolution. We conclude that it is unlikely that Cu particles as a whole get dissolved due to the Gibbs Thomson effect.

In consequence, the Cu particles dissolution by cold work can be described by the dislocation shearing mechanism, similar to the GP zone dissolution reported in Al alloys. However, it should be noted that GP zone would be easier to be cut and dissolved by dislocations because of its very thin structure composed of a few atomic layers. On the other hand, the ϵ -Cu particles focused in this study are much larger, and thus, dissolving the whole particle was not actually observed. Even after a number of dislocations passed through a ϵ -Cu particle, the thicker body would remain though it would be plastically deformed. Therefore, as shown in the HRTEM image (**Fig. 6**), it would be reasonable to understand that the dissolution of Cu particles could take place only at the tip sharpened by severe cold work.

4.2 Effect of deformation and dissolution of Cu particles on dislocation accumulation behavior and resultant strength in cold-rolled Fe-Cu alloy

In general, when a metallic material containing second phase particles is cold worked, geometrically necessary (GN) dislocations are introduced to accommodate the discontinuity of plastic deformation occurred between the matrix and the second phase. As a result, the increment of

dislocation density is amplified, which leads to a higher work hardening rate [29]. The dislocation accumulation is particularly effective when the second phase is hard particles with a high stiffness such as carbide and nitride. On the other hand, the dislocation accumulation is retarded when the second phase particles themselves are plastically deformed following the macroscopic deformation of the material. The ϵ -Cu particles fall in the latter case because the plastic discontinuity between matrix and second phase is reduced through the deformation of the particle. The dislocation accumulation behavior in the low strain regime observed in this study well exhibits such trend. This is similar to the case of tensile deformation confirmed in the previous study [13,30]. However, the phenomenon becomes complicate in the higher strain regime where the dislocation density is affected by not only dislocation accumulation but also the rate of dislocation annihilation. In the result of Fig. 2 (b), dislocation density becomes higher in the Cu steel rather than the VC steel in the high strain regime. This might suggest a possibility that the dislocation annihilation is being affected by the particle-dislocation attractive interaction under the condition that dislocations are highly stored. Moreover, the defect energy might be reduced by solute Cu segregation at dislocation cores, leading to a lower driving force for the annihilation of dislocations.

With increasing dislocation density by cold rolling, the hardness of VC steel is monotonously increased in accordance with the Bailey-Hirsch relationship with keeping the hardness difference from the cold rolled pure iron as shown in Fig. 3. Although it is not clear whether the dislocation strengthening can be simply added to the particle dispersion strengthening to estimate the total strength or not, Fig. 3 indicates the particle dispersion strengthening is fully maintained even after introducing high density dislocations in the hard VC dispersion steel. However, in the Cu steel, the contribution of the particle dispersion strengthening tends to be gradually diminished with increasing dislocation density. There may be two reasons for the decrement of the particle dispersion strengthening: one is that the volume fraction of the Cu particles was decreased with dissolution by cold working, and the other is that the pinning force was decreased through the elongating and thinning of the Cu particles. In other words, the particle cutting by dislocations became easier by the thinning of the particles. It is difficult to explain this phenomenon quantitatively, but it is obvious that the length of cutting path by dislocation is inversely proportional to the aspect ratio of the particle. In the case of the heavily cold-rolled specimen in which the aspect ratio of the Cu particles was increased

to more than 10, it is reasonable to estimate that the cutting stress is significantly lowered.

Although ϵ -Cu particles generally exhibit strong attractive interaction with dislocations resulting in the increments of yield stress and work hardening rate, they tend to lose the strengthening ability under heavily deformed condition. This characteristic of the soft particles turns out to be a preferable in terms of workability of steel, and it would also contribute to good local elongation and the necking deformability. Therefore, the application of deformable and dissoluble soft particles dispersion steels will grow to respond industrial demands on higher strength steel sheets with sufficient workability.

5. Conclusions

An over-aged Fe-2mass%Cu alloy was subjected to cold rolling in order to investigate the behaviors of deformation and dissolution of ϵ -Cu particles. The results obtained are summarized as follows:

- (1) The contribution of the dispersed Cu particles to the strength of cold-rolled Fe-2 mass% Cu alloy, which is estimated by subtracting the dislocation strengthening from the measured hardness, tends to be decreased with increasing the thickness reduction by cold rolling. It is possible that the plastic deformation and dissolution of Cu particles by cold rolling lowers the resistance against deformation.
- (2) The Cu particles are plastically deformed and their aspect ratio is markedly increased during cold rolling; however, the average aspect ratio values observed in cold-rolled specimen are still significantly smaller than the ideal value expected from the rolling reduction.
- (3) The heavily deformed Cu particles have a complex internal deformation structure and irregularly bended interface. In addition, the boundary at the tip part of the elongated particle looks like being vanished in or merged with the ferrite matrix, which strongly suggests the dissolution is occurring there.
- (4) Cu particles are partially dissolved into ferrite matrix by heavy cold rolling. When the thickness

reduction by cold rolling reached to 70%, approximately 0.5 vol.% of dispersed particles were estimated to be dissolved, i.e., Cu atoms went back in the ferrite matrix from the change in lattice parameter of ferrite phase, by assuming the lattice parameter change was derived only from the dissolution of Cu particles.

- (5) The dissolution of Cu particles is caused by dynamic partitioning of Cu atoms from the precipitates into solid solution by dislocation shearing at the particle tip sharpened by severe cold working. This appears to be the major difference of the soft dispersion particles from the hard counterparts. The dispersion strengthening is fully maintained even after introducing high density dislocations in the hard VC dispersion steel, and the VC particles remains stably.

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Captions

Table 1 Chemical compositions of alloys used in this study.

Fig. 1 TEM bright field images of Cu and VC particles in the undeformed alloys. Histograms show size distributions.

Fig. 2 Changes in the hardness (a) and the dislocation density (b) by cold rolling in the Cu and VC steels, and pure iron.

Fig. 3 The relation between the hardness and the dislocation density in the Cu and VC steels, and pure iron.

Fig. 4 TEM bright field images of Cu and VC particles in the 70% cold-rolled alloys.

Fig. 5 Distribution of the aspect ratio of Cu and VC particles in the undeformed and the 70% cold-rolled alloys.

Fig. 6 HRTEM image showing an elongated Cu particle in the 80% cold-rolled Cu steel. The arrows indicated irregularly bent interfaces and a dashed circle marks the tip of this elongated Cu particle.

Fig. 7. EDS line profiles of Cu K α 1 and Fe K α 1 taken from near the ϵ -Cu particle tip, from the inside of the particle to the ferrite matrix (position of 0 to 30 nm) along the white arrow.

Fig. 8 Experimentally measured lattice parameters of binary Fe-Cu alloys as a function of the Cu content.

Fig. 9 Changes in the lattice parameter of pure iron and the Cu steel (left axis of ordinate) as a function of the reduction of thickness, plotted together with the concentration change of solute Cu in the Cu steel (right axis of ordinate).

Fig. 10 DSC analysis of the undeformed Cu steel and the 90% cold-rolled Cu steel.

Fig. 11 The relation between the curvature radius of the tip of deformed Cu particles and the solubility of Cu at different temperatures.

Table 1 Chemical compositions of alloys used in this study.

	C	Si	Mn	P	S	V	Cu	N
Pure Iron	0.0056	<0.003	<0.003	<0.002	<0.0003	-	-	0.0011
Cu steel	0.0069	0.01	0.07	<0.004	0.001	<0.001	2.03	0.0029
VC steel	0.194	0.08	0.09	<0.004	0.001	0.94	0.01	0.005

(mass%)

Figure 1
TEM bright field images of Cu and VC particles in the undeformed alloys.
Histograms show size distributions.

Figure 2
Changes in hardness (a) and dislocation density (b)
with cold rolling.

Figure 3
Relation between hardness and dislocation density
in pure iron, Cu steel and VC steel.

Figure 4
TEM bright field images of Cu and VC particles in the 70% cold-rolled alloys.

Figure 5
Distribution of the aspect ratio of Cu and VC particles in the undeformed and the 70% cold-rolled alloys.

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HRTEM image showing an elongated Cu particle in the 80% cold-rolled Cu steel. The arrows indicated irregularly bent interfaces and a dashed circle marks the tip of this elongated Cu particle.

Figure 7

EDS line profiles of Cu K α_1 and Fe K α_1 taken from near the ϵ -Cu particle tip, from the inside of the particle to the ferrite matrix (position of 0 to 30 nm) along the white arrow.

Figure 8
Experimentally measured lattice parameters of binary Fe-Cu alloys as a function of the Cu content.

Figure 9
Changes in the lattice parameter of pure iron and the Cu steel (left axis of ordinate) as a function of the reduction of thickness, plotted together with the concentration change of solute Cu in the Cu steel (right axis of ordinate).

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DSC analysis of the undeformed Cu steel and the 90% cold-rolled Cu steel.

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The relation between the curvature radius of the tip of deformed Cu particles and the solubility of Cu at different temperatures.

Graphical abstract