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Application of Controlled Cu Nano-Precipitation for Improvement in Fatigue Properties of Steels

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Abstract

In order to improve fatigue properties of steels, it is essential to inhibit crack initiation and crack propagation. Crack propagation mainly depends on the micro-order structure, for example, the grain boundary and the texture. To the contrary, crack initiation depends on the nano-order structure, for example, precipitation, slip and dislocation motion. By the way, it is reported that Cu in steel exhibit high resistance to fatigue crack initiation, but the mechanisms causing Cu to have this effect have not yet been clarified in detail. Thus, in order to clarify the mechanisms that make Cu in steel improve fatigue properties of steel, dislocation structure and precipitates of Cu added steels under different precipitation conditions have been observed and analyzed with electron microscopy. Consequently, it is clarified that reduction of the cross-slip frequency by Cu solid solution or Cu cluster in steel retards fatigue crack initiation.

1. Introduction

There are myriad examples of failure of machines for transportation, for example vehicles, or ships, and of structures such as bridges caused either directly or indirectly by structural fatigue. Of course, there are applications of shapes and structures that reduce stress concentration, or designs that embody a certain degree of safety therein. All are efforts to increase the reliability and life of these structures. However, there are also many contradictions in the form of environmental protection, or running costs. For that reason, there are great expectations for the development of materials that are conducive to the improvement of endurance of such structures. For example, materials having superior fatigue characteristics are applied to vehicle parts for which sheet thickness limits are predetermined for fatigue durability, and the mass of those parts are reduced by making them thinner to improve fuel efficiency and to contribute to protecting the environmental.

In order to improve fatigue properties of steels, it is essential to inhibit crack initiation and crack propagation. Crack propagation mainly depends on the micro-order structure, for example, the grain boundary and the texture. To the contrary, crack initiation depends on the nano-order structure, for example, precipitation, slip and dislocation motion. The steel plates used in large structures such said ships or bridges are comparatively thick. For that reason, ceasing the propagation of fissures or reducing the effect of the speed of propagation can be very well expected to ensure the endurance and durability of such large structures, but in the case of thin sheets such as those used on the parts in vehicles, it is very important to inhibit the initiation of cracks in some way.

Note that at room temperature Cu does substantially not disperse in a solid solution in steel. However, at around 800°C, it does disperse at about 2% in the solid solution. Therefore, after thoroughly diffusing in solid solution, the piece is cooled. Then, by heat treating the over saturated solid solution, it is possible to vary the state of its

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existence in a solid solution, clusters, bcc coherent precipitation and fcc incoherent precipitation[1,2]. Not only is Cu a base element having a notably ability to strengthen precipitates, it also has been reported[3-5] to be effective in inhibiting the initiation of cracks due to fatigue. However, there are still many unanswered questions relating to the underlying reasons thereto. Thus, to clarify the mechanisms that improve fatigue characteristics through the application of Cu, the status of the Cu in the steel was varied. Then, observations and analyses of the dislocation structure and precipitation were applied using a transmission electron microscope for the test pieces provided for testing of fatigue.

2. Test Methods

2.1 Test pieces

To eliminate the effects of base elements in an advancing type solid solution, some test pieces were not added with Cu, and some were varied at 1.5%, using the components of ultra low carbon steel with the Ti additive as a base. **Table 1** shows the chemical compositions of the test pieces. Test pieces were melted in a 300 kg vacuum furnace and cast into 100 kg ingots respectively, and those ingots thus formed were rolled after heat treatment up to 1,200°C as shown in **Fig. 1**. This created 40 mm hot bands. These were then refinished into 5 mm sheets by hot rolling with the rolling temperature set above the Ar_3 point. **Fig. 2** shows the microstructure at 1/4 thickness of hot rolled steel sheets. While the Cu additive steel had a tendency for slightly mixed grains, the average grain diameter was substantially equivalent to the steel without the Cu additive.

Then, steel sheets thus obtained were heat treated for 60 minutes at 450°C to 750°C in order to change the state of Cu. **Table 2** shows a representative example of heat treatment and the state of Cu.

2.2 Tensile testing

Tensile strength testing was performed based upon the JIS Z 2241 metal materials tensile strength testing method, using a JIS #5 test piece. However, the tensile direction was set to be parallel to the rolling direction.

2.3 Plane bending fatigue testing

A fatigue test piece of the shape shown in the **Fig. 3** was cut out so that the rolling direction and the length direction of the test piece

Table 1 Chemical compositions of investigated steels

Steel	C	Si	Mn	P	S	Al	Ti	N	Cu
A	0.0018	0.01	0.20	0.004	0.004	0.04	0.05	0.0016	<0.002
B	0.0020	0.01	0.20	0.004	0.004	0.04	0.05	0.0016	1.51

Values in mass%

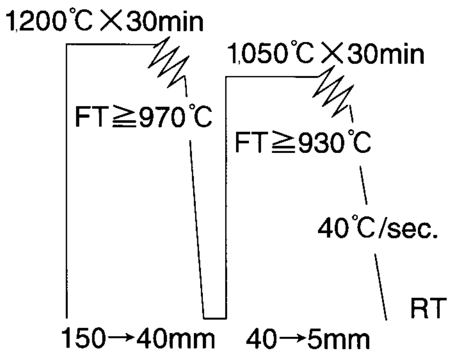
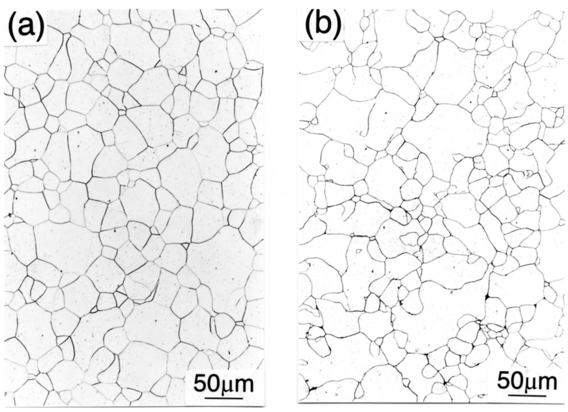


Fig. 1 Process of hot rolling



(a) Steel A (Cu free) (b) Steel B (Cu added)

Fig. 2 Optical micrographs of microstructure of investigated steels

Table 2 Heat treatment and expected Cu state of specimens

Specimen	Heat treatment	Cu state
A	As rolled	Cu free
B1	As rolled	Solid solution
B2	550°C × 60min	Fine precipitation

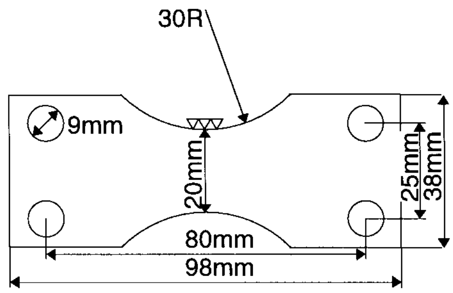


Fig. 3 Dimension of the specimen for stress controlled fatigue test (plane bending test)

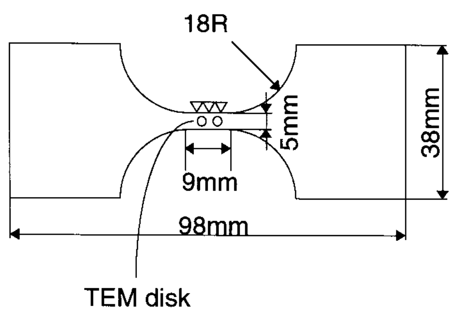


Fig. 4 Dimension of the specimen for strain controlled fatigue test (axial tension-compression test)

were parallel. Then, to eliminate the effects of the surface, the front and back faces of the test piece were ground. The plane bending fatigue test was performed using an electro-hydraulic servo-motor fatigue testing machine. Stress ratio: –1; completely reversed; sin wave: 5 to 15 Hz.

2.4 Strain control fatigue testing

The fatigue test piece of the shape shown in **Fig. 4** was cut so that the rolling direction and the length direction of the test pieces were parallel. The front and back faces of the test pieces were chemically polished to enable verification of changes in the status of the surface during the fatigue test. The strain control fatigue test was performed while controlling using signals from a differential transformer type strain meter. The test was performed with a light load; strain ratio: –1; complete compression triangular wave strain speed: 4×10^{-3}/s.

2.5 Surface roughness observations

In order to observe the behavior for the initiation at the starting point of fatigue cracks, the surface of the test piece under investigation for strain control fatigue was observed repeatedly at 1×10^3 using a scanning type laser microscope.

2.6 Observations using a transmission electron microscope

After conducting predetermined strain control fatigue test, the specimen for observation using the transmission electron microscope was taken from a quarter of the thickness of the position shown in Fig. 4. Acceleration voltage on the transmission electron microscope (TEM) was 200 to 400 kV. At the same time, quantitative analysis was performed for Cu on the test piece using EDS.

3. Experimentation Results

3.1 The results of Cu additive affecting fatigue strength

Fig. 5 shows the effect of heat treatment temperatures on tensile strength in the steels added with Cu and not added with Cu. While strength was substantially not dependent on temperatures in heat treatment for steels not added with Cu, steels added with Cu notably increased in strength at temperatures around 550°C to 650°C.

Fig. 6 shows the relationship of heat treatment temperatures on steel with and without the Cu additive, and strength over time, repeated 2×10^6 cycles (called fatigue strength below). In the same way as tensile strength, the fatigue strength of steel without the Cu additive is substantially unaffected by the temperatures in the heat treatments. However, fatigue strength does increase in steel added with Cu at temperatures between 550°C to 650°C. However, fatigue strength increases less remarkably than tensile strength.

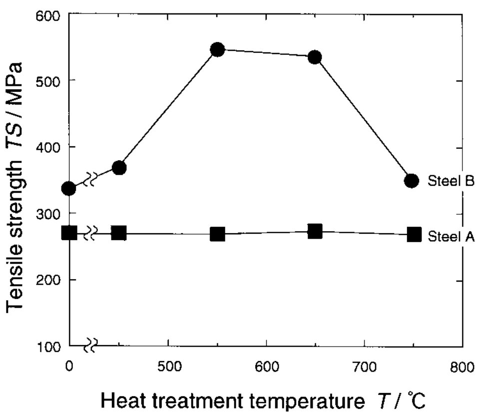


Fig. 5 Effect of heat treatment temperature on tensile strengths of the two investigated steels

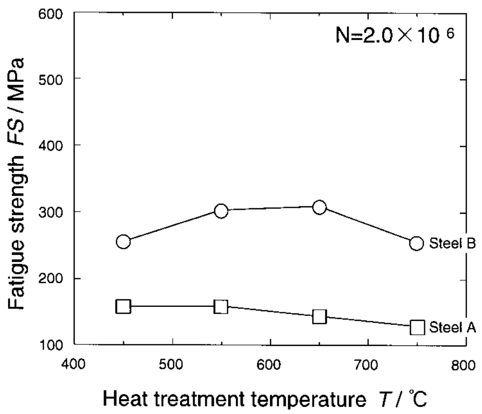


Fig.6 Effect of heat treatment temperature on fatigue strengths at 2.0×10^6 of the two investigated steels

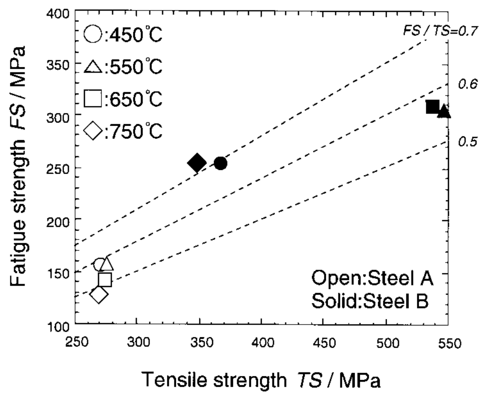


Fig. 7 Relationship between tensile strength and fatigue strength of investigated specimens

Fig. 7 shows the relationship between tensile strength and fatigue strength. The dotted lines in the figure represent the ratio of fatigue strength to tensile strength (called fatigue strength ratio below). Normally, the fatigue strength ratio is generally between 0.4 to 0.6. However, in the Cu added steel, when treated at temperatures between 450°C and 750°C, the fatigue strength ratio was extremely high at approximately 0.7. This suggests that the solid solution, the clustered Cu or the incoherent coarse Cu precipitate in the steel, little contributed to tensile strength, contributed somehow to the increase in fatigue strength.

3.2 The affect of Cu additive affecting cyclic deformation behavior

Almost all the life span rate for fatigue life spans thoroughly smooth test pieces that have no defects that will cause stress concentration is a lifespan where cracks are formed. In other words, life is extended and fatigue strength rate increases as the initiation of cracks is suppressed.

On the other hand, because the phenomenon of cracks occurring is the due to locally cyclic plastic deformation, fatigue strength can be attained if the resistance to that cyclic plastic deformation is high. Cyclic deformation behavior whether there was the presence of Cu additive and when changed by heat treatment with the presence of Cu additive was studied.

Fig. 8 shows the results of fatigue tests where strain amplitude

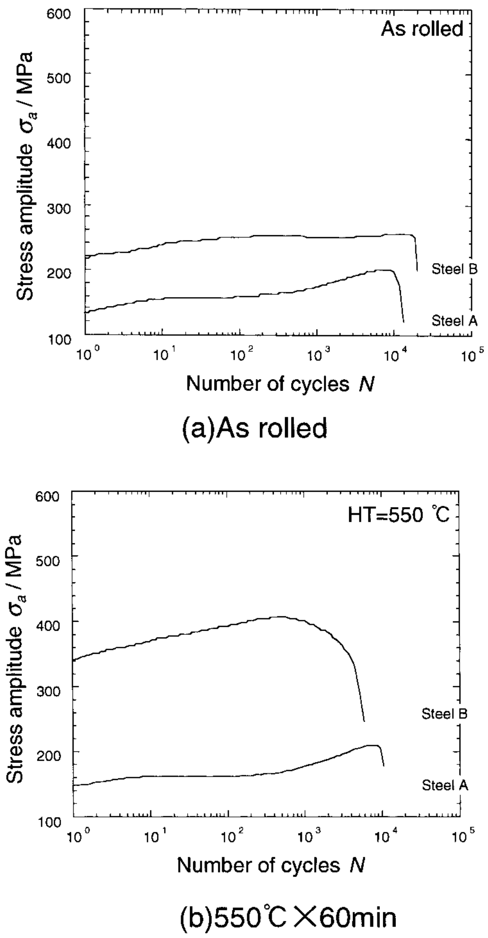


Fig. 8 Cyclic stress response curves at total strain amplitude 0.3%

was controlled to a constant. The pieces with larger stress amplitude under the same strain had higher resistance to plastic deformation. While there were substantially no differences in the shape of the stress response curves in the steel without Cu additive and not heat treated, and the steel that was heat treated at 550°C, there was large difference in the shape of the stress response curves depending on

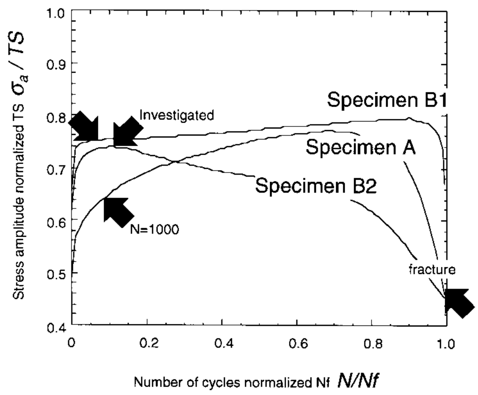


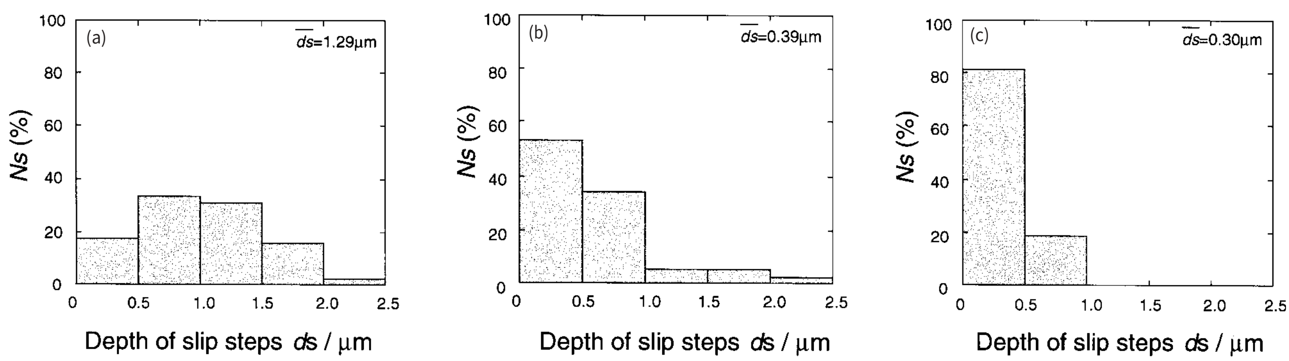
Fig.9 Cyclic stress response behaviors for the three investigated specimens

whether there was heat treatment applied to Cu additive steel.

Fig. 9 shows standardized stress response behavior. The horizontal axis represents the number of cycles normalized by ruptured life; the vertical axis represents stress amplitude normalized by tensile strength. At the very initial outset of repetitions, the Cu additive increased stress amplitude. However, the material having the Cu additive and that was not heat treated (hereinafter called the Cu solid solution material) showed stable stress amplitude until rupturing in all repetitions. However the heat treated at 550°C material having the Cu additive (hereinafter referred to as the Cu precipitate material) showed a continued drop in the stress amplitude until rupture after reaching a peak at the initial number of repetitions indicating behavior for cyclic softening.

3.3 The affect of Cu additive affecting cracking behavior

The occurrence of cracking from stress is directly related to the formation of intrusion and extrusion which are the starting point of the cracking on the material surface and is associated with the state prior to cracking. The fatigue test was stopped after the 1×10^3 cycles which is the very beginning of the rupture repetition, and the depth of the unevenness on the test piece surface formed with cyclic strained and the numerical distribution were measured. **Fig. 10** shows the results. The rate of the shallower unevenness was greater on the Cu additive material in comparison to Cu non-additive material regardless of the heat treatment conditions. This means that the formation



(a) Specimen A (Cu free) **(b) Specimen B1 (Cu solid solution)** **(c) Specimen B2 (Cu precipitation)**

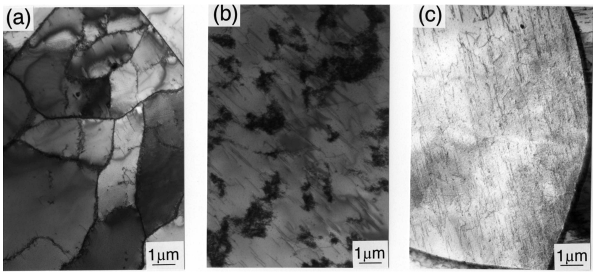
Fig. 10 Distribution histograms of depth for slip steps on the surface of specimens

of unevenness on the surface was suppressed, which is where for cracking initiates. Stress concentration was reduced because the unevenness was a shallow. Thus, the suppression of the initiation of cracking led to the improvement in fatigue strength.

3.4 The affect of Cu additive on dislocation structures

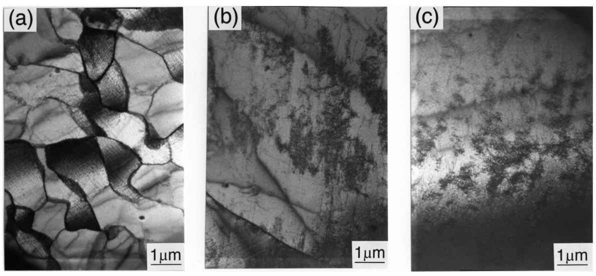
Fig. 11 shows TEM photographs of Cu non-additive material, Cu solid solution material, and Cu precipitation material that 1×10^3 cycles. The number of cycles of 1×10^3 is equivalent to approximately 1/10 of the rupture live. It is prior to the peak of cyclic hardness for the Cu precipitation material. While the dislocation structure suggests a typical cell structure on the Cu non-additive material, and the Cu solid solution has a vein structure, and the Cu precipitate material suggests a planar array structure.

Fig. 12 shows TEM photographs after rupture of each material. While the Cu non additive material had a cell structure, and the Cu solid solution material had a vein structure and there was no change, the Cu precipitate material changed from a planar array structure to a vein structure. The changes of the dislocation structure of the Cu precipitate materials, correspond to the stress response behavior for cyclic hardening shown in Fig. 9 to softening.



(a) Specimen A (Cu free) **(b) Specimen B1 (Cu solid solution)** **(c) Specimen B2 (Cu precipitation)**

Fig. 11 TEM micrographs of specimens at 1/10Nf cycles



(a) Specimen A (Cu free) **(b) Specimen B1 (Cu solid solution)** **(c) Specimen B2 (Cu precipitation)**

Fig. 12 TEM micrographs of ruptured specimens

4. Considerations

The results of experiments and observations are summarized in **Table 3**. While the fatigue strength ratio (FS/TS) of the Cu non-additive material is a general value, the Cu solid solution material was extremely high. This is thought to be related to that Cu solid solution shows highly stable stress amplitude up to rupturing in the number of cycles in the strain control fatigue test. In other words, the Cu additive controls dislocation movement, and as a result of the changes in the dislocation structure, the mode of the unevenness caused by intrusion and extrusion on the surface changed which delayed the generation of the cracking[6].

Load stress and Stack Fault Energy (or SFE) have been clarified[7-9] as factors that apply characteristics of dislocation structures that are formed under cyclic loads in fcc metals, beginning with AI alloys. Furthermore, cross-slip frequency has been proposed[10-12] as a substitute for this in bcc metals for which SFE cannot be, such as iron and steel materials. Dislocation structures formed under cyclic loads on materials where cross-slip is easy, such as pure steel show a cell structure[13]. Because it is difficult to leave the primary slip plane for the dislocation on materials having difficult cross-slip, such as that on Fe-Si alloys, a planar-array dislocation array is shown under low load stress. Under high stress, a vein structure is reported[14].

On the other hand, the frequency of cross-slip is an important factor[15] for dislocation structures at the extreme surface layer. Persistent Slip Bands (PSBs) that are deeply related to the forming of unevenness of the surface require cross-slip in their forming so there is no observation of this on materials that are difficult to cross-slip. The PSBs region formed at the surface layer corresponds to the cell formation region. The slip plane at the repeat deformations is limited by the boundary of that formed region. Still further, the edge of PSBs correspond to the unevenness which is the starting point for fatigue cracking on the surface. Non-dense and deep unevenness is caused by the formation of PSBs[16]. On the other hand, the concentration of the uneven surfaces is shallow on the material having difficult cross-slip. As can be seen in **Fig. 13**, this alleviates the concentration of stress on the surface which leads to a delay in the initiation of fatigue cracks[17].

The fatigue strength ratio of the Cu precipitate material was approximately equivalent to that of the non additive material regardless of whether Cu was added. Therefore, there was no affect in the adding of Cu. This shows that stress amplitude was at its maximum value in the number of cycles of approximately 1/10 the rupture life for the Cu precipitate material in the strained control fatigue test. It is thought that after this shows cyclic softening behavior in the section. In order to clarify the causes of cyclic softness in the Cu precipitate material, TEM observations and EDS's measurements were taken to observe Cu precipitate material after a number of cycles of 1×10^3.

Fig. 14 shows a TEM photograph and representative EDS spectra measurement. Cu peaks were obtained in all dislocations observed.

Table 3 Fatigue properties including substructure and surface condition

Specimen	FS/TS	Cyclic response	Substructure		Slip steps
			N=1,000	Fracture	
A (Cu free)	0.58	Gradually hardening	Cell	Cell	Deep
B1 (solid solution)	0.69	Steady hardening	Vein	Vein	Shallow
B2 (precipitation)	0.55	Softening from peak	Planar-array	Vein	Shallow

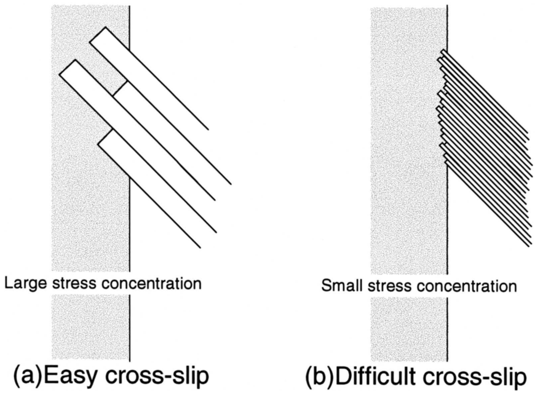
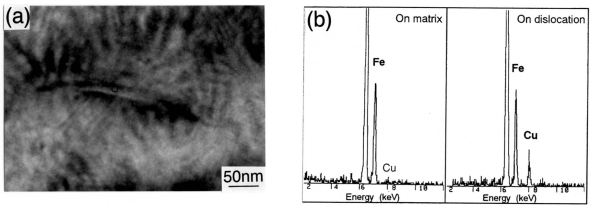


Fig. 13 **Schematic illustrations of slip steps growing into intrusions and extrusions on the surface**



(a) TEM micrograph in a dislocation. Arrowhead indicates an analyzed point for the EDS.

(b) EDS spectra on matrix and in a dislocation

Fig. 14 Results of the TEM observation and the EDS analysis

Table 4 Quantitative EDS analyses in Cu segregation at 1,000 cycles

Specimen	On matrix	In dislocation
B1 (solid solution)	1.57	1.57
B2 (precipitation)	1.52	2.85

Values in mass%

See **Table 4** for details on Cu quantitative measurements. In the Cu solid solution material, the Cu amount did not change on the dislocation or in other regions. However, there was Cu segregation on the dislocation in the Cu precipitate material. In other words, a result of the fine Cu precipitate matter cutting at the repeat dislocation action is a loss in the precipitate strength because of the Cu precipitate matter becoming thermodynamically unstable and becoming a solid solution again[18-20]. This is thought to indicate the softening behavior during cyclic load. Therefore, for the degree that tensile strength increases for the Cu precipitate material, fatigue strength does not arise.

5. Example Application

The discussion above considers controlling the state of existence of Cu to an appropriate degree and dramatically increasing fatigue characteristics in steel materials. Furthermore, the mechanism of that increase was clarified. The following will describe an example of applying in the effects of Cu to high strength steel sheets requiring in

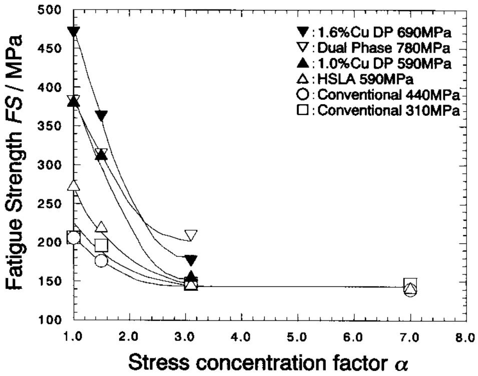


Fig. 15 Fatigue strength of Cu added dual phase steel under stress concentration

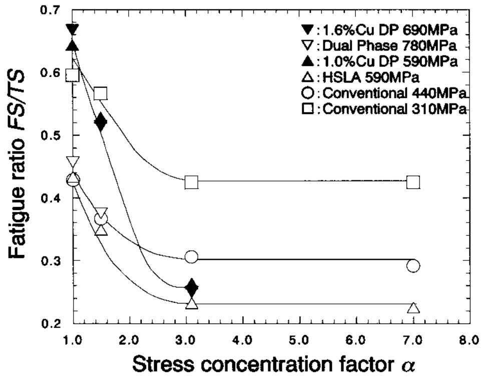


Fig. 16 Fatigue ratio of Cu added dual phase steel under stress concentration

improved fatigue characteristics.

In order to apply Cu effectively to the improvement of fatigue characteristics, it is necessary to control the status of this existence as a solid solution or clusters. In order to keep Cu from precipitating after coiling in the hot rolling process, it is preferred to keep the temperature of coiling below 400°C which is lower than the Cu precipitate temperature. On the other hand, the hot rolling DP steel sheets[21] which currently are used as a high fatigue strength steel sheets are manufactured with a coil winding temperature below 400°C in order to attain the compound structure of ferrite and martensite. The test was made in-situ to increase fatigue strength by adding Cu to hot rolled DP steel sheets on the optimum status of the existence of Cu.

Fig. 15 shows fatigue strength under concentrated stress. In the fatigue test, a flat, bent test specimen notch was deformed. When the stress concentration coefficient α exceeds 3.0, high strength steel sheets having a high sensitivity attain the fatigue strength only of a level equivalent to soft steel sheets. However, Cu added DP steel sheets attained higher strength than steel sheets having tensile strength equivalent for the stressed concentration coefficient α at a max. of 3.0.

Fig. 16 shows the results of that of Fig. 15 organized under a fatigue strength ratio. The fatigue ratio of Cu added steel sheets is superior to that of conventional steel sheets with a stress concentration coefficient α that is less than 3.0.

6. Conclusion

In order to clarify the mechanism that improves fatigue strength through Cu in steel, fatigue tests and a TEM replied to observed and analyzed Dislocation structures and precipitates. The following were clarified.

1. Cu in a solid solution or in clusters has the effect of improving fatigue characteristics. Cu suppresses cross-slip at various dislocation structures under cyclic loads from cell to vein structures. It suppresses the initiation of fatigue cracks making the unevenness of the surface a shallow.
2. Fine Cu precipitate matter does not improve a peak strength to the degree of tensile strength. Fine Cu precipitate matter reforms a solid solution through dislocation cutting at the very initial stages of life. Therefore, precipitate strength is lost.
3. Cu additive-hot rolled DP steel sheets apply to the improvement of fatigue strength and its effects are verified. In a range less than 3.0 for the stress concentration coefficient, high fatigue strength was attained compared to conventional steels having the same strength level.

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