Change in intragranular misorientation during stress relaxation behavior in Cu-Ni-Si alloy subjected to continuous cyclic bending

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Abstract. Stress relaxation resistance is often required in application of electrical parts for copper and its alloys. The stress relaxation behavior should be influenced by stored strain through manufacturing process. In the present study, a Cu-Ni-Si alloy solution-treated sheet was subjected to continuous cyclic bending (CCB), which was proposed as a useful straining technique to impose higher strain on the surface and lower strain in the center layer of metal sheets. Samples were analyzed by scanning electron microscope/ electron back scatter diffraction (SEM/EBSD) technique to investigate stored strain and stress relaxation process. CCB raised up the strength of Cu-Ni-Si alloy sheets at room temperature. The stress relaxation ratios of the CCBent sheets increased with an increment of test exposure time at 423K. The stress relaxation process was accompanied with change in intragranular misorientation of the samples. Change in intragranular misorientation through the stress relaxation test was understood as a result of release of residual and applied stresses with consumption of stored strain.

1. Introduction
Smaller size, higher performance and multi-functions are required for electrical components of transportation vehicles, made of copper alloys, whose properties should be improved. In particular, for current-carrying members, stress relaxation characteristics are most important from assurance of reliability at the contacts. As lots of commercial copper alloys are precipitation-strengthened ones, the effect of precipitation on their stress relaxation resistance should be paid attention. Effect of continuous cyclic bending (sometimes abbreviated as CCB, below), which can affect particle size of precipitation on the stress relaxation resistance of the commercial alloys, should be extremely interesting. The author's group reported previously that the stress relaxation process of a Cu-Ni-Si alloy bent repeatedly can be monitored by a change in intragranular misorientation, kernel average misorientation (KAM) [4].

In the present study, the stress relaxation process is divided into four stages: (I) unloaded before stress relaxation test (SRT), (II) loaded before SRT, (III) loaded after SRT and (IV) unloaded after SRT. The relationship between the stress relaxation and change in KAM of a Cu-Ni-Si alloy subjected to continuous cyclic bending was examined. Grain orientation spread (GOS) [6] was investigated by electron back scattering diffraction (EBSD) [5,6,7].
2. Experimental procedure

2.1. Material and specimen
A Corson alloy (Cu-Ni-Si alloy) sheet solution-treated was received as a starting material. Chemical composition of the sample is listed in Table 1. Dimensions of workpiece for CCB were 200mm long in rolling direction (RD), 25mm wide in transverse direction (TD) and 1.2mm thick in normal direction (ND). From CCBent workpiece, stress relaxation specimen is machined as 24mm long in RD, 7mm wide in TD and 1.2mm thick in ND.

| Table 1. Chemical composition of Corson alloy used |
|------------------|-----|-----|-----|-----|
| Cu-Ni-Si          | 2.44| 0.49| 0.03| 0.03| Bal. |

2.2. Continuous cyclic bending (CCB)
CCB is one of severe straining process and can impose strain to a specimen by continuously bending it by passing the specimen between five rolls. Table 2 shows the conditions for continuous and repeated bending. Three kinds of specimens, As-received, aged and 40-pass-CCBent/aged specimens were prepared in this experiment.

| Table 2. Continuous bending conditions |
|------------------|------------------|------------------|------------------|------------------|
| Roll drive speed (rpm) | Roll radius (mm) | Number of processes (pass) | Offset amount (mm) | Processing temperature |
| 40                | 16               | 0, 40             | 0.5               | room temperature   |

2.3. Aging process
Using a muffle furnace, heat at 723K (heating time 10 min) and then remove from the muffle furnace to cool naturally.

2.4. Polishing procedure
In order to perform SEM/EBSD analysis, mechanical polishing with emery papers (#800/ #1200/ #2000) and buffing (alumina oxide abrasive, 3µm/ 1µm/ 0.3µm/ 0.05µm, 20 minutes each degree) were applied, and followed by electropolishing. The conditions for electropolishing are listed in table 3.

| Table 3. Electrolytic grinding conditions |
|------------------|------------------|
| Electrolytic solution | HNO₃ : CH₃OH = 3 : 7 |
| Temperature       | 238K             |
| Electric current  | 0.2A             |
| Time              | 90s              |
| Electrode plate   | Stainless steel electrode |
2.5. Stress relaxation testing
Using a muffle furnace, stress relaxation test was performed at 423 K. The heating time was 10 min, the test exposure time was ranged from 1 to 100 h, and the test was carried out in an Ar atmosphere. A laser displacement meter was used to measure the deflection displacement. During the stress relaxation process the permanent strain occurs when the loaded deflection is released. The stress relaxation ratio is expressed as the ratio of the permanent deflection displacement produced to the initial deflection displacement loaded. The stress relaxation ratio is calculated by the following equation:

\[
\text{stress relaxation ratio(\%)} = \frac{\delta_t}{\delta_0} \times 100
\]

\(\delta_t\): Permanent deflection displacement of the specimen caused by stress unloading after the test (mm)

\(\delta_0\): Initial deflection displacement of the test specimen at a given stress load (mm)

The stress relaxation process is divided into four stages: (I) unloaded before stress relaxation test (SRT), (II) loaded before SRT, (III) loaded after SRT and (IV) unloaded after SRT, as illustrated in figure 1.

![Figure 1. Description of the stress relaxation test stage](image)

2.6 EBSD Analysis
The analysis was performed on solution-treated, aged and 40 Pass/aged samples before and after stress relaxation. The analysis area was 180 µm x ND 1200 µm from the center and front and back of the RD plane, respectively. The step size of the electron beam was set to 1 µm and minimum misorientation angle to recognize grain boundaries was set to 5°.

3. Experimental results and discussion

3.1. Stress relaxation test
The relationship between the stress relaxation ratio and test exposure time at 423 K in the stress relaxation tests (SRT) for as-received, aged and CCBent/aged materials is shown in figure 2. SRT of CCBent/aged materials were performed with the high-hardness back side as the top surface and subjected to initial deflection displacement. As a result, aging and CCB on the as-received material reduce the stress relaxation ratio. In addition, at a test exposure time of 100h showed that the aged specimen had better stress relaxation characteristics than the aging and CCBent specimen. This is attribute to the effect of CCB did not work effectively precipitation site formation in the aging process. It is also likely that the effect of CCB was not observed in the 40P/aged specimen because a sufficient amount of strain was not added.
Figure 2. Stress relaxation behavior for non-worked, 723K aged and CCBent/aged specimens, tested at 423K as a function of test exposure time.

3.2. EBSD Analysis

3.2.1 Variation of KAM value near the surface in the process of stress relaxation. Figure 3 shows the changes in KAM values near both surfaces (distance from bottom surface, 40-80 μm and 1120-1160 μm) for (A) As-received, (B) 40p/aged specimens. The Top side, KAM value decreased by about 0.1 from stage II and decreased to stage IV-1h. After that, it can be seen that KAM value increased by about 0.8 in stage III-1h and decreased by about 0.3 in stage IV-1h. On the top side of 40Pass/aged material, KAM value decreased by about 0.1 from stage I to stage II, and increased by about 0.5 in stage III-1h and stage IV-1h. Thereafter, KAM values decreased by about 0.5 in Stage III and Stage IV, and increased by about 0.2 in Stage III and Stage IV. On the bottom side, there was no significant difference, but KAM value increased about 0.3 from stage II to stage III-1h, and KAM value decreased about 0.1 in stage IV-1h and stage III-10h. Thereafter, KAM values increased by about 0.1 at Stage 3-100h and stage IV-100h. The difference between the Top and Bottom of 40P/aged specimen could be the effect of external stresses due to residual stresses and stress loading and the effect of releasing stored strains due to SRT.

Figure 3. Changes in mean KAM value near the surface in each stage of stress relaxation process for (a) As-received and (b) 40P/Aged specimens.
3.2.2. Comparison of KAM value distribution near the surface. Two figures comparing KAM change distributions between the bottom of as-received (or solution treated) and aged specimens are shown in figure 4. The vertical axis is the change in stage II to stage III-1h and the horizontal axis is the change in stage III-1h to stage III-10h. For the as-received specimen (figure 4(a)), there was almost no change between no load and 1h of stress relaxation, and the KAM value increased by about one between stage III stress relaxation 1h and stage III stress relaxation 10h, indicating that there are many points in the positive direction of the Y-axis. This means that the KAM value does not increase with stress loading, but the KAM value increases with stress relaxation, suggesting that a dynamic recovery process may have taken place. For the aged specimen (figure 4(b)), the KAM value increased by about 0.5 between stage II and stage III stress relaxation 1h. The KAM value decreased by about 0.5 between stage III stress relaxation 1h and stage III stress relaxation 10h. Therefore, there is a negative correlation for this sample. This means that the KAM value increases with stress loading and decreases with stress relaxation, which may be due to sliding dislocation deformation.

![Figure 4](image)

Figure 4. Variation of average KAM values of grains near the surface at each stage of the stress relaxation process (a) As-received specimen and (b) Aged specimen.

3.2.3. Comparison of GOS and KAM values. Figure 5 shows the results of investigating the average KAM and GOS values of the grains of each specimen. The KAM value was higher than the GOS value for each specimen. The difference between the two values for the aged specimens compared to the other specimens may be due in part to the recovery after the aging process. The 40p/aged specimens were subjected to CCB treatment, which introduced strain, but the aging treatment recovered the strain and the amount of strain was considered to have decreased. It is expected that further CCB will lead to accumulation of strain and the GOS value will exceed the KAM value. It is expected that further CCB processing will result in strain accumulation and the GOS value will exceed the KAM value. Next, since some crystal grains show higher GOS values than KAM values, the orientation difference within the grains was investigated using line profiles. Grains with an orientation difference of up to 3.5° showed a KAM value of 0.538 and a GOS value of 1.71. For grains with an orientation difference of up to 0.5°, the KAM value was 0.257 and the GOS value was 0.185. Thus, depending on the conditions, the change in GOS value was observed when the change in KAM value was small.
4. Conclusions
In the present study, the stress relaxation process was divided into four stages: (I) no loading, (II) stress loading, (III) stress relaxation, and (IV) unloading. The relationship between the stress relaxation and the intragranular misorientation difference of Cu-Ni-Si alloys subjected to continuous cyclic bending was investigated. The obtained results are summarized as follows.
(1) The stress relaxation properties of Cu-Ni-Si alloys are improved by CCB and aging.
(2) The kernel average misorientation (KAM) of as-received, aged and 40P/aged specimens changed due to the effects of external stresses, residual stresses and stress loads and the release of accumulated strains by stress relaxation tests.
(3) The mechanisms of stress relaxation were different for as-received (or solution-treated) and aged and CCB/aged specimens.
(4) A change in the GOS value was observed when the change in the KAM value was obviously small in some conditions of the stress relaxation process.

5. References
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