Technical Report

Development of Stainless Steel Foil with Superior Cyclic Fatigue Resistance

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Abstract

In order to clarify the factors affecting the repeated bending durability of SUS301 foil, which is used as a reinforcement plate for the rear side of flexible display panels such as foldable smartphones, the material was evaluated in terms of lattice strain, texture, and surface roughness. It was found that the following material design factors are important to suppress fatigue cracking in reinforcement plates: (1) suppression of fatigue cracking by increasing compressive strain, dislocation density, and γ/α' interface lattice strain; (2) formation of bendable microstructures by controlling texture; and (3) suppression of stress concentration by reducing surface roughness. We believe that this guideline will be a key factor in enhancing the overall performance and reliability of our products, increasing their competitiveness, and will be a breakthrough in next-generation technology.

1. Introduction

In recent years, foldable smartphones have been introduced to the market, and the market share of such smartphones in the $800 or more price zone increased to about 7% in the four years from 2020 to 2023. The share in the market continues to rise. There are vertically-foldable type smartphones and horizontally-foldable type smartphones, and both types provide a large screen when unfolded, and become compact when folded. Mechanical properties such as flexibility, repeated bending durability, and flatness are required for the screen which employs the organic EL display panel. However, the panel lacks rigidity, and a metal foil or a CFRP is glued to the rear side of the panel for reinforcement in use. In order to reduce the thickness of the case when it is folded, it is necessary to employ a slim type panel as well as minimizing the curvature of the bend.[1] However, the smaller the curvature becomes, the greater the stress on the bend becomes , and the number of repeated bending durability cycles deteriorates. As the target is to achieve 100000 to 200000 repeated bending durability cycles, studies are ongoing regarding the patentability assessment of a production method for a high-durability panel having reduced stress concentration realized by patterning the openings in a zigzag manner on the bend of a panel reinforcing plate.[2, 3] In order to clarify the factors influencing the repeated bending durability of the SUS301 stainless steel foil used as a reinforcing plate for the rear side of the panel, this report describes the results of the evaluation conducted in terms of lattice strain, texture, and surface roughness.

2. Experiment Method

2.1 Test piece material

SUS301 having the chemical composition as shown in **Table 1** was used as the test material. Cold-rolling was applied to material with a thickness of 30 μm (reduction ratio 50%) and 40 μm (reduction ratio 73%), the material was degreased to remove rolling oil, and the tension annealing (hereinafter referred to as TA) under the hydrogen atmosphere at 350 to 800°C was applied. The material was cut into a rectangle test piece 150 mm long and 40 mm wide. The long side ran parallel to the rolling direction (RD). In order to suppress the cracking at the edges of the ridge of the bend, the test

Table 1 Chemical composition of SUS301

(mass%)

C	Si	Mn	P	S	Ni	Cr	Fe
0.10	0.47	0.57	0.029	0.003	6.64	16.51	Bal.

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piece cut edges were polished with #1500 emery paper.

2.2 Repeated bending durability test

For the repeated bending durability test, the Tension-Free™ Folding Clamshell-type Bending Test Machine DR11MR of Yuasa System Co., Ltd. was used. The test piece was laid on the test machine so that the center line of the long side of the test piece was aligned with the direction of the bending ridge. A 180° bending angle was provided with a cycle of one Hz in the repeated bending test. Further, the radius of bending was adjusted and determined so that a strain of about 0.75% was produced on the neutral plane with respect to the sheet thickness direction at the ridge line position when the sheet was bent by 180°. Unless otherwise described in this paper, the repeated bending durability test was continued until the test piece was completely fractured.

2.3 X-ray diffraction

The lattice strain was measured by the X-ray focusing method. The sealed tube type Cu target was operated at a tube voltage of 40 kV and a tube current of 40 mA. In addition to the parent phase of the austenitic (γ) phase, in order to observe the martensitic (α') phase that has a low diffraction intensity, one-dimensional measurement was conducted within an angular range (2θ) of 40 to 140° at 1°/min with a step-size of 0.01°. In the line profile analysis depending on the split-type Voight function fitting, the lattice spacing and the full width at half maximum (FWHM) between $\{111\}_\gamma$ and $\{110\}_{\alpha'}$ were assessed, and were used as the lattice strain index. Furthermore, in order to investigate the crystallographic orientation, the texture was measured using X-ray diffraction. The Mo rotary target was operated at 40 kV and 200 mA. After defocusing correction by Ni powder, the pole figures and the inverse pole figures were analyzed with $\{111\}_\gamma$, $\{200\}_\gamma$, $\{220\}_\gamma$, and $\{311\}_\gamma$.

2.4 Surface roughness Rz measurement

The surface roughness Rz was measured pursuant to JIS B 0601: 2001 in the direction parallel to RD. The measurement condition was measured length: 1.25 mm, cutoff (λc); 0.25 mm, cutoff (λs); 0.0025 mm, scanning speed of tracer; 0.3 mm/s, load; 0.7 mN, and a cone type tracer having a radius of 2 μmR and a tip angle of 60° was used. The measurement was conducted at more than five different locations, and the average value was employed.

3. Results and Discussion

3.1 Influence of TA temperature on durability

The durability (the number of repeated bending durability cycles) is deeply related to the crack arrest, and there are mainly three factors.[4] The first is the deformation of the crack tip. The dislocation migration of remote stress and the deformation behavior of the crack tip influence the progress per cycle. The second is the crack closure phenomenon. A plastic region is formed at the crack tip when deformation develops while the peripheral region is restrained by the elastic deformation region; therefore, the crack tip extends in a plastic deformation mode, and is elastically compressed instead. This field of compression remains even after the load is relieved, and impedes crack opening. The residual compressive stress field extends even further as the minor cracks progress, and impedes the progress of cracks. The last factor is the hardness (resistance to plastic deformation) in the neighborhood of the crack tip. The higher the hardness at the crack tip is, the more the dislocation migration is inhibited, and the opening of cracks is impeded. In short, in order to improve the number of repeated bending durability cycles of a metal foil, the suppression of dislocation migration, the control of the compressive stress, and the work hardening (high dislocation density) are necessary.

Then, in order to quantify the microstructure parameters which influence the durability, by using the X-ray focusing method, $\{111\}_\gamma$ lattice spacing and $\{111\}_\gamma$ full width at half maximum (FWHM) were evaluated as the index of the face-centered cubic lattice γ phase compressive strain and as the index of the dislocation density, respectively.[5] As shown in **Fig. 1**, the lattice spacing increases with the increase in TA temperature (decrease in compressive strain). At about 550°C, as shown by the image of the transmission electron microscopy (TEM), on account of the high density precipitation of fine $M_{23}C_6$, the Cr concentration of the γ phase decreases, and the lattice spacing slightly decreases. Later, although the lattice spacing increases along with the relaxation of lattice strain, at 700°C, due to the disappearance of the deformation-induced α' phase with a body centered cubic lattice (reverse transformation to the γ phase), Cr is supplied to the γ phase, and $M_{23}C_6$ grows. Simultaneously, due to volume shrinkage, lattice spacing slightly decreases. Further, FWHM decreases greatly at 400°C and above, and at 550°C, a local maximum appears. This is presumed to be due to the introduction of dislocation by the tension to the γ phase softened by the precipitation of $M_{23}C_6$. As a result, as shown in **Fig. 2**, lattice spacing of $\{111\}_\gamma$ and FWHM of $\{111\}_\gamma$ are closely correlated with the number of repeated bending durability cycles. In short, by the decrease in lattice spacing (increase in compressive strain) and the increase in FWHM (increase in dislocation density), the number of repeated bending durability cycles increases.

Next, various studies were conducted on the bending habit angle (a characteristic of a metallic foil that will not return to the original shape even after being bent by a constant force, and then being relieved of the force), and the influence of the lattice coherency strain on the γ/α' interface was found. As the X-ray line profile inserted in **Fig. 3** shows, in addition to the γ phase, the α' phase is clearly observable, which exists by about 20% below 800°C in terms of volume percentage according to the Reference Intensity Ratio (RIR) method[6]. In addition, as Fig. 3 shows, the ratio of lattice spacing

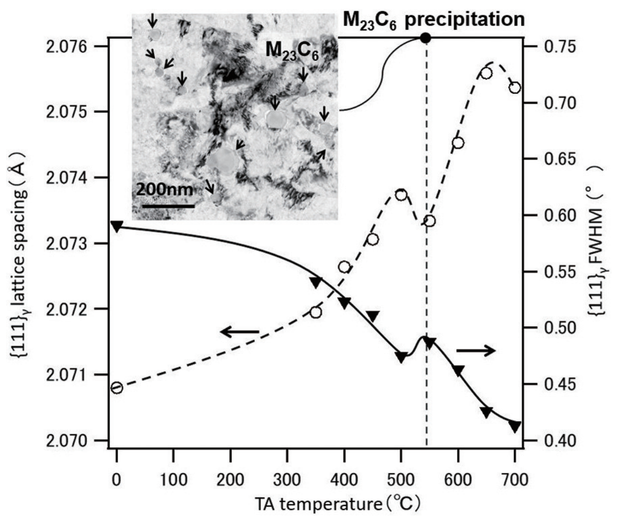

Fig. 1 Lattice spacing and FWHM as a function of TA temperature for 30 μm thick specimens (The $M_{23}C_6$ precipitates are shown by arrows in the TEM image.)

$\{111\}_{\gamma}/\{110\}_{\alpha'}$ (coherency strain) increases with the TA temperature, and reaches its highest at about 700°C. This behavior corresponds to the change in the bending habit angle measured in 20000 cycle repeated bending. Accordingly, as **Fig. 4** shows, a nonlinear relationship between the lattice spacing ratio of $\{111\}_{\gamma}/\{110\}_{\alpha'}$ and bending habit angle is obtained, and with respect to TA temperature, at 1.022 which corresponds to 550°C of TA temperature, an inflection point exists. This is presumed to be the influence of the decrease in lattice spacing of $\{111\}_{\gamma}$ due to the abovementioned increase in $M_{23}C_6$. The internal stress due to the γ/α' coherency strain is expected to suppress the dislocation migration and the stress concentration to the inclusion which triggers fracture. Furthermore, due to an increase in the dislocation cell size corresponding to the strain size by the deformation-induced α' phase reported,[7] it is presumed that the softening of the microstructure due to the formation of the dislocation cell during the repeated bending test influences the bending habit angle. As a result, at 700°C, γ/α' coherency strain becomes largest, and the bending habit angle becomes smallest. In other words, the increase in γ/α' coherency strain means the suppression of the plastic deformation, namely dislocation migration. This finding is considered to be one of the factors contributing to the enhancement of durability.

3.2 Influence of cutting angle on durability

As **Fig. 5** shows, the change in the cutting angle improves the number of repeated bending durability cycles. As X-ray (110) pole figures are compared, in the direction parallel to RD (0°), the crystallographic orientation is in the Brass texture {110}<112>, which is perpendicular to the slip plane of {111}, or the orientation inclined by approximately 20° or 90° from parallelism. At 30° where the number of repeated bending durability cycles reaches its highest, the crystallographic orientation is close to Goss texture {110}<100>. {111} exists symmetrically at approximately 55°. Since there are many {111} planes close to 45°of critical resolved shear stress known as the Schmid factor, slip is rate-limiting, and the number of repeated bending durability cycles is presumed to become the highest. In addition, at 90°, {111} is in the orientation perpendicular or nearly parallel, and it is difficult for slip to occur, and the cleavage fracture is rate-limiting; therefore, the number of repeated bending durability cycles is presumed to be lowered. Namely, the high value of the number of repeated bending durability cycles at the cutting

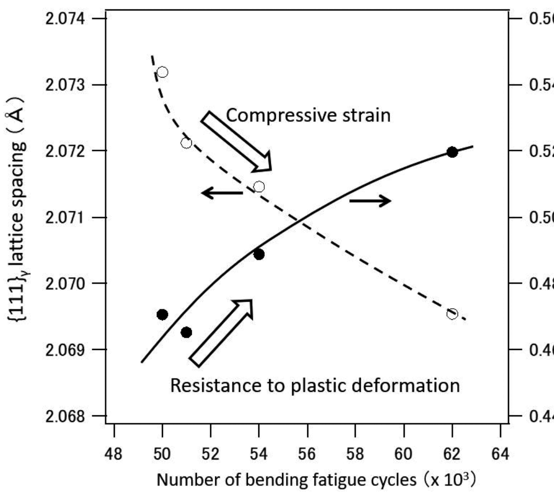

Fig. 2 Relationship between the number of bending fatigue cycles and the lattice spacing and FWHM of $\{111\}_{\gamma}$ for 30 μm thick specimens

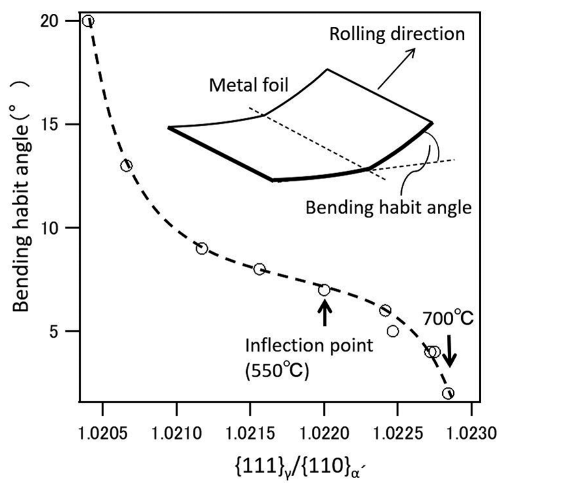

Fig. 4 Relationship between the bending habit angle and the fraction of $\{111\}_{\gamma}/\{110\}_{\alpha'}$ lattice spacing for 40 μm thick specimens

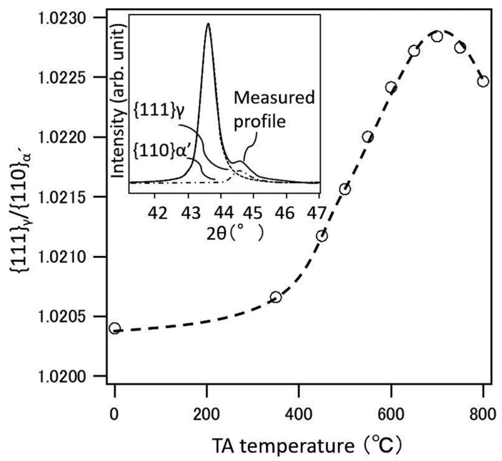

Fig. 3 Relationship between the TA temperature and the fraction of $\{111\}_{\gamma}/\{110\}_{\alpha'}$ lattice spacing for 40 μm thick specimens

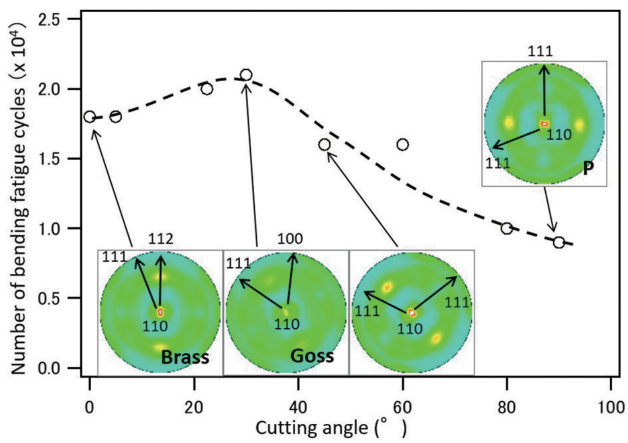

Fig. 5 Relationship between the cutting angle and the number of bending cycles with the X-ray (110) pole figure for 40 μm thick specimens

angle of approximately 30° from RD depends on the texture which renders easy slip.

As aforementioned, for the material design aimed at suppressing the fatigue cracking, the increase in compressive strain, increase in dislocation density, and the γ/α' interface lattice strain to suppress the dislocation migration were studied. In the meantime, the cutting angle means "easy to bend", or likewise, "difficult to break". In other words, in the case of the slip plain being perpendicular or parallel to the direction of stress, it is difficult for slip to occur, and cleavage fracture takes place (easily breakable). In the case of the slip plain inclined to the direction of stress, slip easily occurs (hardly breakable).

Next, in order to determine quantitatively the texture at the cutting angle showing the highest number of repeated bending durability cycles, we focused our attention on the integrity (volume fraction) of RD in inverse pole figures which varies greatly depending on the cutting angle. Examples of inverse pole figures of RD when the cutting angle is changed are shown in **Fig. 6**. (a) at angle 0° from the Brass texture, the integration of crystallographic orientation in the direction of <112> is observed, and the {110}<112> inverse pole figure is shown. (b) at the angle 35° from the Brass texture, the crystallographic orientation is integrated to <001>, and the inverse pole figure of Goss texture {110}<001> is shown. (d) at 90°, integration at <111> becomes large, and <111> becomes parallel to the bending direction. The equivalent <111> becomes close to perpendicular; therefore, the closer to 90°, the larger the cleavage fracture becomes. The highest number of repeated bending durability cycles lies at approximately 35°, and the integration of crystallographic orientation at <001> is observed. This is the region where <111> is inclined relatively in a large amount from the longitudinal direction. In short, the increase in the fraction of <001> increases the number of repeated bending durability cycles.

Thus, it has been clarified that, by changing the cutting angle, or by forming an "easy to bend" structure by controlling the texture, the number of repeated bending durability cycles can be improved.

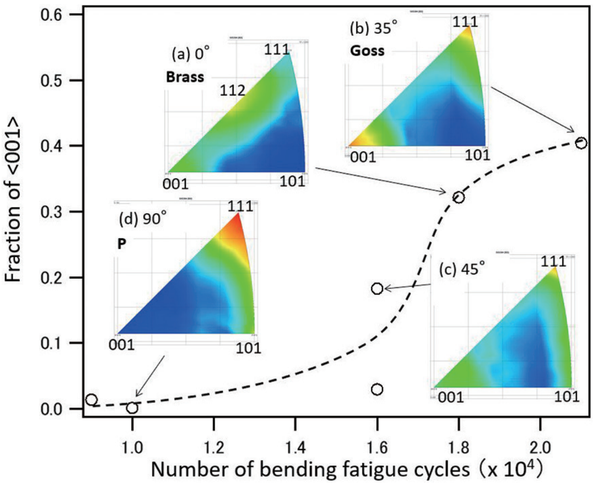

Fig. 6 Relationship between the number of repeated bending durability cycles and the fraction of <001> with inverse pole figures in the rolling direction for 40 μm thick specimens

3.3 Influence of surface roughness on durability

By changing the rolling roll surface roughness, 30 μm thick foils having different surface roughness were rolled. Later, by using the foil material TA treated at 650°C, the number of cracks at 60000th that of repeated bending endurance was assessed. The number of the cracks is the integrated value of N=12, and the cracks of 1 mm or longer excluding those developed from the material edge were counted. As shown in **Fig. 7**, the larger the surface roughness Rz, the larger the number of cracks, and the influence of the surface smoothness upon crack occurrence is large. In order to confirm the origin of the crack, the crack position was folded, and its cross-section was observed by SEM, and a fracture originating at an inclusion near the surface was observed. Both of the stress concentration on the surface irregularities and that on the inclusions near the surface[8] are considered to accelerate the generation of cracks in a combined manner. Therefore, in order to enhance the repeated bending durability, not only the smoothness of the surface but also the size, number, and the distribution of inclusions are also presumed as important factors.

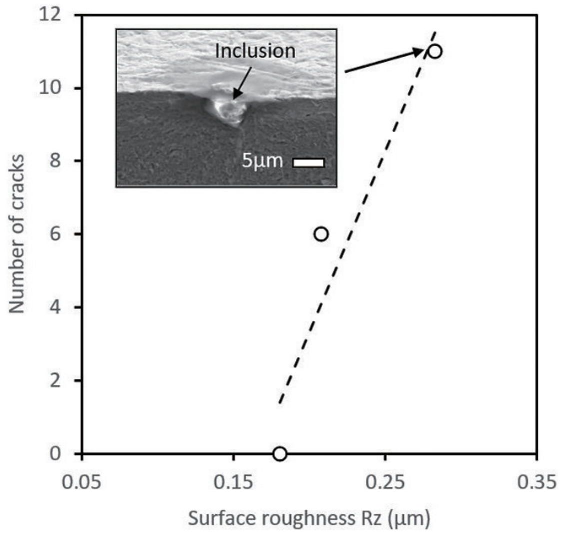

Fig. 7 Relationship between surface roughness Rz and the number of cracks for 30 μm thick specimens (Inset shows the fracture origin observed by SEM.)

4. Conclusion

The improvement of the repeated bending durability of a metal foil is an essential factor in technical innovation to extend the product life and reduce cost. To achieve this objective, the following were identified as important: suppression of fatigue cracking by increasing compressive strain, dislocation density and the γ/α' interface lattice strain, formation of the microstructure which renders easiness in bending by controlling the texture, and the suppression of stress concentration by reducing the surface roughness and the number of inclusions. These factors prevent the initial formation and the propagation of cracks, and suggest the guideline for the design of improving the repeated bending durability of a metal foil. Furthermore, this guideline strengthens the overall performance of the products and reliability, and is a key to enhancing competitiveness. Research and development for the improvement of the mechanical properties of a metal foil are continuously required, and we firmly believe that our R&D will be a breakthrough in the next generation technologies.

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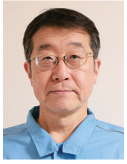

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