Grain Boundary Engineering for Control of Fatigue Fracture in 316L Austenitic Stainless Steel

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Roles of grain boundaries in fatigue crack nucleation and propagation in 316L austenitic stainless steel were investigated to obtain a clue to the grain boundary engineering for control of high-cycle fatigue fracture. The fatigue crack nucleation preferentially occurred at grain boundaries at the low-stress amplitude conditions less than about 160 MPa. In particular, the 82% of cracked grain boundaries were random boundaries. The fatigue crack nucleation at the random boundaries occurred irrespective of the geometrical configuration of grain boundary plane to the stress axis and the persistent slip bands (PSBs) in the neighboring grains. Although the fatigue cracks nucleated even at the annealing twin boundaries, namely the {111}/Σ3 coincidence site lattice (CSL) boundaries the crack nucleation occurred only when the surface trace of the Σ3 CSL boundaries was parallel to the PSBs in the neighboring grains. Moreover, in-situ observations of the fatigue crack propagation revealed that the grain boundaries played important roles as crack path, crack deflection sites and barrier of crack propagation, depending their character. In particular, although the Σ3 CSL boundaries became crack propagation path, the crack propagation rate locally decreased when the crack propagated along the Σ3 CSL boundaries. On the other hand, the crack propagation rate considerably increased when the crack propagated along random boundaries. The usefulness of grain boundary engineering for control of high-cycle fatigue fracture was demonstrated. The higher fraction of CSL boundaries achieved higher fatigue strength and longer fatigue life in 316L stainless steel.

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1. Introduction

The 316L austenitic stainless steel has been extensively applied as structural components for the chemical and the power plants because of its superior corrosion resistance and heat resistance. A long-term use of the structural components is important for lowering the burden on the environment. The control of fatigue fracture and grain boundary degradation phenomena like intergranular corrosion is necessary for improvement of the lifetime of the structural components. In the face centered cubic (fcc) materials with low stacking fault energy like austenitic stainless steels, the grain boundary engineering (GBE) to inhibit grain boundary degradation has been achieved by control of grain boundary character distribution (GBCD) through the introduction of quite high fraction of annealing twin boundaries, namely {111}/Σ3 coincidence site lattice (CSL) boundaries. Shimada et al.,¹¹ Tsurekawa et al.,¹² and Michiuchi et al.¹³ revealed that the intergranular corrosion in austenitic stainless steels can be significantly suppressed by increasing fraction of low-Σ CSL boundaries, particularly {111}/Σ3 boundaries. Lo et al. overviewed the recent development of stainless steel from various viewpoints including GBE.¹⁴ Recently, one of the present authors revealed that the intergranular corrosion in 316L austenitic stainless steel was more precisely controlled by the GBE based on the fractal analysis of grain boundary connectivity.¹⁵

Heinz and Neumann¹⁶ have demonstrated that the fatigue cracks preferentially nucleate at {111}/Σ3 boundaries in austenitic stainless steels, even though the Σ3 boundaries possess the highest intergranular fracture strength under static deformation. They suggested that the fatigue cracking at {111}/Σ3 boundaries results from the stress concentration owing to the significant interaction between the {111}/Σ3 boundary and localized crystal slip during cyclic deformation. In contrast, it has been reported that incoherent twin boundaries showed intrinsically high resistance to fatigue fracture.¹⁷

It has been suggested that the {111}/Σ3 boundaries in fcc materials often became the propagation path of fatigue cracks. Kaneko et al.¹⁸ reported on the fatigue crack propagation at the vicinity of coherent twin boundaries in copper bicrystals. According to their findings, the ratio of intergranular cracking to transgranular cracking increased in the bicrystals having the deviation ranging from 3° to 5° from the exact misorientation angle for the {111}/Σ3 boundaries. However, the fatigue cracks predominantly propagated in the grain interior when the bicrystals had the deviation angle less than 3° or more than 9°.¹⁹

Recently, in-situ observations of fatigue crack propagation in SUS304 steel specimens revealed that the crack propagation rate along {111}/Σ3 boundaries was lower than that along random boundaries. From the viewpoint of the fatigue crack propagation, therefore, {111}/Σ3 boundaries does not necessarily degrade the fatigue property. However, further investigations about roles of grain boundaries in crack propagation have been required.

Unfortunately, there have been little studies on the effect of GBCD on the fatigue life in polycrystalline materials. Lehockey et al.¹¹ have reported that the fatigue life of Ni-
The 316L stainless steel sheets were cold-rolled up to 3 in air at 1273 K for 3.6 ks, and subsequent water quenching. with 3 mm thick were subjected to solution heat treatment. 

2. Experimental Procedure

2.1 Specimen preparation

The commercial cold-rolled 316L stainless steel sheets with 3 mm thick were subjected to solution heat treatment in air at 1273 K for 3.6 ks, and subsequent water quenching. These 316L stainless steel sheets were cold-rolled up to 3%, 4%, 5%, and 20% in reduction ratio and subsequently annealed in air at 1273 K for different times of 7.2 ks (2 h) or 259.2 ks (72 h), respectively, then finally water quenched.

The specimens for high-cycle fatigue tests and fatigue crack propagation tests were cut from these thermomechanically processed sheets using a spark machine. Figure 1 shows the shape and dimensions of these two type specimens. The dimensions of the specimen for high cycle fatigue test were 10 mm length, 4 mm width and 2.8 mm thick. The dimensions of the compact type (CT) specimens (ASTM standard E-64724)) for fatigue crack propagation test were 30 mm wide and 2.8 mm thick. The specimen surface was mechanically polished using emery papers of 320–1500 grade and diamond powder slurry of 0.1 µm in particle size. Thereafter the specimen surface was electrolytically polished in an electrolytic solution of 23 vol% perchloric acid and 77 vol% acetic acid at a current density of 4.0 mA/mm² at 277 K for 20 s.

2.2 Evaluation of grain boundary microstructure

The average grain size and the GBCD was quantitatively evaluated using an orientation imaging microscopy (OIM) analysis based on the field emission gun-scanning electron microscopy (FEG-SEM)/electron backscatter diffraction (EBSD) measurements. The electron beam was scanned at 1.5–4.0 µm step size at an accelerated voltage of 20 kV and an emission current of 10 µA on a specimen surface. The grain boundaries with Σ ≤ 29 including the low-angle boundaries were determined as low-Σ CSL boundaries according to Brandon’s criterion, Δθ = 15/Σ1/2.25) Although the 5 degrees of freedom of the grain boundary is not fully determined by the Σ-value, it has been demonstrated in the previous studies that the low-Σ CSL boundaries exhibit a higher resistance to fatigue crack nucleation in fcc polycrystalline materials, such as pure aluminum.26) The GBCD was evaluated by the number fraction for different types of grain boundaries in individual 316L stainless steel specimens.

2.3 High-cycle fatigue tests and in-situ observations of fatigue crack propagation

The high-cycle fatigue tests of 316L stainless steel specimens were carried out using a servo-hydraulic machine (Shimadzu, Servopulser) in air at room temperature. Sinusoidal stress on different levels was applied at a stress ratio of 0.1 and at a frequency of 10 Hz.

Fatigue crack propagation tests were also carried out using a servo-hydraulic machine (Shimadzu, Servopulser). The specimens were subjected to cyclic loading in air at room temperature. Sinusoidal loading was applied at the load ratio of 0.1 at the frequency of 5 Hz. The stress intensity factors range was 35 MPa m²/2.

In-situ observations of fatigue crack propagation were performed to reveal the effect of grain boundaries on the crack propagation path and the local crack propagation rate. The crack propagation evaluation system consists of a CCD camera mounting a high magnification long lens, a computer with an A/D converter, a video capture card and capture software.19) The analog signals of load and displacement from the servo controller was input into the computer by the A/D converter. The image of fatigue crack was captured with every predetermined constant value of sinusoidal loading which synchronized with the signal of load in the A/D converter. The average propagation rate of the fatigue crack for the predetermined intervals was analyzed by motion analysis based on the EBSD measurements. Figure 1 shows the shape and dimensions of the specimens for (a) the high-cycle fatigue tests and (b) the fracture crack propagation tests.
analysis software (DETECT, DIPP-Motion PRO 2D). The propagation path and the propagation rate of fatigue cracks were evaluated in connection with the grain boundary microstructures obtained by EBSD measurements.

3. Results and Discussions

3.1 Effect of grain boundary character on intergranular fatigue cracking

Figure 2 shows the grain boundary microstructure of the 316L stainless steel specimen produced by cold rolling at 20% in reduction ratio and subsequent annealing at 1273 K for 7.2 ks, and then water quenching. The specimen had an average grain size of 10 µm and the fraction of the low-CSL boundaries of 51%. In particular, the specimen had a reasonably high fraction of Σ3 CSL boundaries of 30%. This specimen was designated by the base material (BM) specimen.

The relationship between the stress amplitude and the number of cycles to fracture, namely S-N curve for the BM specimen are shown in Fig. 3(a). The fatigue limit when the number of cycles to fracture reached $10^7$ cycles was estimated at about 140 MPa. Figure 3(b) shows the SEM micrographs of fatigue cracks observed on the surface of fractured specimen subjected to cyclic deformation at the stress amplitude of 164 MPa. The fatigue cracks preferentially nucleated at grain boundaries rather than the persistent slip bands (PSBs) at this condition. Most of cracked grain boundaries were random or Σ3 CSL boundaries. Although the random boundaries fractured regardless of their geometrical distribution to the stress direction, the Σ3 CSL boundaries fractured only when the boundary trace on the specimen surface was parallel to the PSBs, as shown in the center of Fig. 3(b)(ii).

Figure 4 shows the fraction of intergranular fatigue cracking for different types of grain boundaries. The 82% of cracked grain boundaries were random boundaries. The nucleation of fatigue cracks at random boundaries was hardly affected by the geometrical configuration of grain boundary...
plane to the stress axis and the PSBs. On the other hand, the fatigue crack was never observed at low-angle boundaries. These findings are in good agreement with the case of polycrystalline aluminum specimen. Moreover, the 14% of cracked grain boundaries were Σ3 CSL boundaries, but all of Σ3 CSL boundaries did not necessarily fracture. The fatigue crack nucleated at the Σ3 CSL boundaries were only observed at several ⟨111⟩/Σ3 CSL boundaries; namely at the coherent twin boundaries. Conversely, the Σ3 CSL boundaries whose surface traces were not parallel to the PSBs showed higher fatigue fracture resistance, as shown in the previous studies on intergranular fatigue crack nucleation in fcc materials. Therefore, it is concluded that the fatigue crack nucleation can be controlled by introducing Σ3 CSL boundaries even in the 316L austenitic stainless steel with fcc structure, although the resistance of fatigue cracking is lower at ⟨111⟩/Σ3 (coherent twin) boundaries than at other Σ3 grain boundaries.

3.2 Roles of grain boundary microstructure in fatigue crack propagation

The propagation process of high cycle fatigue cracks in polycrystalline materials must be predominantly affected by the grain boundary microstructure, such as GBCD and grain boundary geometrical distribution. Unfortunately, a little information is available about the effect of the grain boundary character on fatigue crack propagation. Vinogradov et al. studied the effect of the grain boundary character on fatigue crack propagation using copper bicrystal specimens having Σ9 and random boundaries with the same geometry to the tensile orientation axis. They demonstrated that the fracture mode changes from transgranular fracture into intergranular or mixed fracture when the crack tip reached the random boundary even though the cracks were never propagated along Σ9 CSL boundary. These results suggested that Σ9 CSL boundaries had a high resistance to fatigue fracture. In-situ observations of fatigue crack propagation connected with grain boundary microstructure can give the fundamental knowledge of GBE for the control of fatigue crack propagation. For this propose, the CT specimens with coarse-grained structure were produced by strain-anneal grain growth. The CT specimens were subjected to cold rolling at 3% in reduction ratio and to subsequent annealing at 1273 K for 216.0 ks (60 h). This processing was repeated by 3 times. Figure 5 shows the SEM image of the coarse-grained CT specimen. The specimen had the average grain size of 165 µm but included considerable coarse grains more than 800 µm in grain diameter.

Figure 6 shows the example of in-situ observations of fatigue crack propagation in the coarse-grained CT specimen. In this figure, the micrographs around the fatigue crack tip were captured every 1000 cycles of sinusoidal loading.

Figures 7(a) and (b) show the grain boundary map in the vicinity of fatigue crack and the local change in the crack propagation rate corresponding to the respective crack positions shown in the grain boundary map, respectively. The fatigue crack propagation test of the specimen was carried out at ΔK of 35 MPa m^{1/2}. The value of ΔK was controlled to be in the range from 35 MPa m^{1/2} to 37
MPa m$^{1/2}$. These values of $\Delta K$ were ranged in the power-law behavior. In the grain boundary map shown in Fig. 7(a), the fatigue crack path was indicated by the region drawn by a set of black analyses points either that had low-confidence index (CI) values or that could not be indexed by OIM software. In Fig. 7(b), the vertical and the horizontal axis shows the local crack propagation rate and the crack length from the tip of the pre-crack, respectively. The dashed lines vertically drawn in between Fig. 7(a) and Fig. 7(b) indicate the position where the fatigue crack passed across the grain boundaries. The bands indicate the position where the crack propagated along grain boundaries. The width of the bands displays the length of each region where intergranular fracture occurred. The colors of these dashed lines and bands indicate the type of grain boundaries where fatigue crack crossed or propagated, respectively. For example, green and black indicated the $\Sigma 3$ and random boundaries, respectively.

The red allows in Fig. 7(a) showed the positions where crack deflection occurred. The crack deflection tended to occur when the crack crosses the grain boundaries owing to change in slip plane between two adjacent grains. The fatigue crack was also largely deflected by the crack propagation along grain boundaries. Gao et al.\textsuperscript{22}) studied that the relationship between the deflection angle of small surface crack across the grain boundaries and the misorientation angle of grain boundaries in the nickel-based superalloy during high cycle fatigue. According their findings, the grain boundaries with the larger misorientation angle tend to give a rise to a larger crack deflection angle.

When the crack deflection occurred at grain boundaries, the crack propagation rate locally lowered. Although the crack deflection occurred in the grain interior, the change in crack propagation rate hardly occurred. The local crack propagation rate was strongly affected by the type of grain boundaries. The crack propagation rate considerably decreased when the fatigue crack passed across the grain boundary, particularly $\Sigma 3$ CSL boundaries, in comparison with that in the grain interior. Surprisingly, the local crack propagation rate of fatigue crack was considerably lowered when the crack propagated along $\Sigma 3$ boundaries, although the crack propagation was locally accelerated by the propagation along random boundaries.

Therefore, these results suggested that the fatigue crack propagation in 316L stainless steel is controlled by introduction of finer grain structure and higher fraction of $\Sigma 3$ CSL boundaries.

**3.3 Control of grain boundary microstructure to inhibit fatigue fracture**

In this section, the thermomechanical processing route to obtain an optimal grain boundary microstructure for the control of fatigue fracture will be investigated. In this work,
the microstructure having the fine grained structure and the high fraction of low-$\Sigma$ CSL boundaries was desired for the control of fatigue fracture, as predicted in the previous section.

Figure 8 shows the grain boundary maps of 316L stainless steel specimens produced by different conditions of thermomechanical processing. In this figure, the specimen produced by 20% cold rolling and subsequent annealing at 1273 K for 7.2 ks is the same as the BM specimen shown in Fig. 2.

The average grain size in the specimens decreased with increasing reduction ratio of cold rolling. However, prolonging of the annealing time up to 259.2 ks (72 h) at 1273 K did not give rise to significant grain growth. The highest fraction of low-$\Sigma$ CSL boundaries of 77% was obtained in the specimen which produced by 3% cold rolling and subsequent annealing at 1273 K for 7.2 ks.

Figure 9 shows the relationship between the fraction of low-$\Sigma$ CSL boundaries ($F_\Sigma$) and the reduction ratio of cold rolling before annealing in 316L stainless steel specimens. The data of $F_\Sigma$ for each specimen was obtained from more than 700 grain boundaries in number. Although the data for the specimens annealed at 1273 K for 259.2 ks was scattered, the fraction of low-$\Sigma$ CSL boundaries increased with decreasing reduction ratio of cold rolling. The fraction of low-$\Sigma$ CSL boundaries in the specimens did not necessarily increased by the longer annealing for 259.2 ks. Although the twin-induced GBE reported requires the processing of low-strain deformation and subsequent long time annealing at high temperature, the findings in this study likely suggest that the enough high fraction of $\Sigma 3$ CSL boundaries is
achieved by low-strain deformation and subsequent short-time annealing at high temperature.

In the twin-induced GBE processing, to obtain the fine-grained structure with a high fraction of CSL boundaries is one of the important issues to be settled. The present study demonstrated that both the fine grain structure less than 30 µm in average grain size and high fraction of low-3 CSL boundaries more than 70% can be achieved.

3.4 Grain boundary engineering for improvement in fatigue property in 316L stainless steel

In this section, we will investigate the usefulness of GBE based on the control of fatigue crack nucleation and propagation for improvement of fatigue life in the austenitic stainless steel. In this purpose, the specimen with the highest fraction of low-3 CSL boundaries shown in Fig. 8 and 9 was designated as the GBE specimen. The high-cycle fatigue property of GBE specimen was compared with that of the BM specimen with low-3 CSL boundaries of 51%.

The GBE specimen possessed a yield stress (σy) of 271 MPa and an ultimate tensile strength (UTS) of 579 MPa. The values of σy and UTS in GBE specimen were slightly lower than those in the BM specimen (σy = 280 MPa and UTS = 589 MPa).

Figure 10(a) shows the S-N curves for the GBE and the BM specimens. The fatigue limits of the GBE and the BM specimens were estimated to be 168 MPa and 135 MPa, respectively. Although the GBE specimen had the larger average grain size than the BM specimen, the GBE specimen showed the higher fatigue limit.

It is well known that the fatigue limit increased with increasing tensile strength of the materials, and that the value of fatigue limit normalized by ultimate tensile strength (UTS), namely fatigue ratio, commonly becomes approximately 1/3UTS. In the present work, as the values of the UTS of the GBE and the BM specimens were difference, the relationship between the stress amplitude normalized by the UTS and the number of cycles to fracture was shown in Fig. 10(b). The fatigue life at the high stress amplitude level of about 0.4 UTS was almost the same between the GBE and the BM specimens. At the low stress amplitude level below 0.3 UTS, however, the fatigue life of the GBE specimen was considerably improved as compared with the BM specimen.

It was concluded that the GBE based on introduction of high fraction of Σ3 CSL boundaries was useful for improving fatigue property in 316L austenitic stainless steel under the low stress amplitude conditions.

4. Conclusions

The roles of grain boundaries in the fatigue crack nucleation and propagation in 316L austenitic stainless steel was investigated to obtain a fundamental knowledge of the GBE process for the improvement in fatigue property. The effectiveness of GBE for the improvement in high-cycle fatigue property was examined using the 316L steel specimens with different GBCDs. The main results obtained are as follows.

(1) High-cycle fatigue cracks preferentially nucleated at random boundaries. The cracks never nucleated at low-angle boundaries. Although the fatigue cracks also nucleated at Σ3 boundaries, they showed higher resistance to fatigue cracking than random boundaries.

(2) The deflection of fatigue crack path occurred when the cracks propagate across the grain boundaries, particularly random boundaries. The local crack propagation rate was lower when a crack passed across the Σ3 CSL boundaries than when it passed across the random boundaries and in the grain interior.

(3) The fatigue crack propagation rate was considerably lowered when the crack propagated along Σ3 boundaries, whereas the crack propagation was accelerated when the crack propagated along random boundaries.

(4) The GBE through the introduction of a high fraction of Σ3 CSL boundaries significantly improved fatigue property by preventing intergranular fatigue crack nucleation and propagation, resulting in the increase in the fatigue limit.

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