1. Introduction

It has been clarified that the structure consisting of bainite lath nucleated within grain (BWING), not at grain boundary, is greatly resistant against brittleness.\(^1,2\) The nucleation of BWING, therefore, is preferable for the improvement of mechanical properties. The introduction of inclusions such as MnS, Ti-oxides, complex compounds and so on into austenite is utilized for giving nucleation site for BWING.\(^3\) The nucleation of BWING would be induced to relax the stress field around inclusion or due to concentration gradient of \(\alpha\)-formation element around inclusion. However, inclusion itself is brittle and at the interface between inclusion and matrix, high density of dislocations are apt to be accumulated under deformation, inducing cleavage at the interface. As density of inclusion increases, steel would be strengthened, but brittle. Therefore, the introduction of dislocation network, that is, Small-Angle Dislocation Network (SADN) into austenite is tried in the present work. SADN would be stable in the high temperature region such as in austenite, because pinned dislocation existed in SADN would prevent the bow-out of dislocations through absorbing point defects. The formation of SADN in austenite, which act as nucleation site for BWING, was clarified experimentally. The bainite lath neighbor to the nucleated one might be formed under relaxing the stress field around the nucleated one and/or inclusion. It was analyzed that the average size of Aggregates of bainite Laths having nearly Parallel Slip systems between neighboring bainite laths (ALPS) corresponds to that of dimples on fracture surface. The process for ductile fracture is offered based on the crystallographic analysis using transmission electron microscopy.

KEY WORDS: heat treatment; BWING; strength; toughness; ALPS.

2. Experimental Procedures

Steels listed in Table 1 are used for the observation of microstructure with optical, scanning electron (SEM) and transmission electron (TEM) microscopies. The steel containing higher concentration of S is designated as SR, and the lower one of S as SP, which were prepared for clarifying the effect of S on the introduction of SADN into austenite. Process of heat treatment is illustrated in Fig. 1. Solution treatment in austenite \((\gamma)\) region at the high temperatures of 1 400 and 1 450°C were done for the suppression of nucleation of bainite lath at \(\gamma\) grain boundary. The solution treatment at 1 400°C keeps as-rolled MnS, which existed in supplied steel. The increase in the temperature for austenitization to 1 450°C is for making MnS decompose into \(\gamma\) almost completely. Holdings at 900 and 500°C are for the precipitation of MnS and BWING in \(\gamma\), respectively. Thin foil for TEM is prepared by means of a twinjet electropol-

| Table 1. Chemical compositions of steels (mass\%). |
|-------------------------------|---|---|---|---|---|---|---|
| Steel | C  | Si | Mn | P   | S     | N    | O   |
| SP    | 0.14 | 0.29 | 1.44 | 0.001 | 0.001 | 0.0004 | 0.002 |
| SR    | 0.13 | 0.29 | 1.44 | 0.001 | 0.001 | 0.0003 | 0.002 |
ishing at 50 V and 278 K using 10% perchloric acid/acetic acid electrolyte. The observation of microstructure and analysis for crystallographic information (mainly with nano-beam diffraction technique) were carried out using JEM-2100 and JEM-3010 operated at 200 and 300 kV, respectively. Tensile test was performed under a strain rate of $1 \times 10^{-4}$/s at room temperature.5)

3. Results and Discussion

Figure 2 shows optical micrographs taken from steels (a) SP and (b) SR. In Fig. 2(a) intergranular bainite is greatly nucleated at γ grain boundary in contrast to dominant formation of BWING in Fig. 2(b). In the microstructure of BWING, rows of MnS particles (bar-shaped MnS in 3 dimension) are often observed. Optical micrographs were taken to examine nucleation site for BWING after polishing surface of steel SR every 0.3 μm thick. In Fig. 3, bainite laths and a row of MnS particles (encircled) are shown. The row of MnS particles could be frequently observed in steel SR. In Fig. 4, a bainite lath is shown to contact with a MnS particle by polishing the surface of steel SR, that is, the bainite lath nucleates at the MnS. On the other hand, bainite laths go away from the dotted line, which is drawn on a raw of MnS particles, by polishing the surface of steel SR as shown in Fig. 5. The results suggest that bainite lath might be easy to nucleate at both MnS particles and the plane containing rows of MnS particles.

Figure 6 shows optical micrographs taken from steel SR (a) austenitized at 1 450°C for 300 s followed by quenching into iced brine, and (b) as-received, which consists of pearlite (black region), ferrite (white region) and elongated MnS particles with cold rolling (indicated white arrows). The MnS particles in as-received steel is obviously almost dissolved during the austenitization at 1 450°C. MnS particles precipitate easily around 950°C having the Kurdjumov–Sachs orientation relationship\(^6\) with bainite. This result suggests that MnS may form through solid-to-solid reaction in austenite without melting as being indicated in the pseudobinary system, FeS–MnS.

Steel SR austenitized at 1 450°C was held at 900°C for 1 200 s, and after that, held at 500°C for 10 s. The optical micrograph taken from the specimen heat-treated mentioned above is shown in Fig. 7. During the holding at 900°C for 1 200 s, MnS particles seem to precipitate along

---

Fig. 1. Schematic illustration of heat treatment process.

Fig. 2. Optical micrographs showing (a) dominant intergranular bainite in steel SP and (b) BWING in steel SR.

Fig. 3. Optical micrograph showing BWING and a row of MnS particles (encircled) formed along dislocations. It was analyzed that MnS acts as nucleation site for BWING.

Fig. 4. Optical micrographs taken from the same area enclosed with dotted square in Fig. 3. The surface (a) was polished 0.3 μm thick (b). Bainite lath indicated by an arrow in (a) approaches and contacts with a MnS particle showing black-dot contrast.

Fig. 5. Optical micrographs taken from the same area. The surface (a) was polished 0.3 μm thick (b). A bainite lath indicated by an arrow in (a) might contact with a plane containing a row of MnS particles.
The steel SR austenitized at 1450°C for 300 s followed by quenching into iced brine, and (b) as-received, which consists of pearlite (black region), ferrite (white region) and elongated MnS particles with cold rolling (indicated with arrows).

Fig. 7. The steel SR austenitized at 1450°C for 300 s followed by 900°C×1200 s and 500°C×10 s. MnS particles show black dot contrast and incline to arrange making cellular shape. Some bainite laths are continuous across the rows of MnS particles.

Fig. 8. Optical micrograph taken from region containing a row of MnS particles (dotted line).
Fig. 9. (a) Pole figures of (111), and (011), (indicated by dots) taken from the left side of the dotted line in (b). The dots encircled in stereo-projections cause from the variants of bainite laths encircled in (b). The dotted line in (b) corresponds to that in Fig. 8.

Fig. 10. Stereographic projection of crystallographic directions in a single crystal of austenite. This result is obtained from the analyses of EBSPs taken from BWINGs in the left side of the dotted line in Fig. 9(b).

Fig. 11. Stereographic projection of the directions, /H20855100/H20856g, /H20855110/H20856g and /H20855111/H20856g in the regions across the dotted line in Fig. 9(b). The directions in the left side region of the dotted line is indicated with rigid marks and those in the right side region is indicated with open marks.

Fig. 12. Stress vs. strain curves taken from steel SR solution-treated at 1450°C (rigid curve) and 1400°C (dotted curve) for 300 s followed by 900°C×1 200 s and 500°C×10 s.

Fig. 13. Scanning electron micrograph showing the fracture surface of the SR obtained after the tensile test (stress vs. strain curve (A) in Fig. 12). Ductile fracture occurs, however, bar-shaped MnS (some of them indicated by arrows), which might be formed along dislocations with edge component in small angle dislocation network, acts as nucleation site for cleavage.

Fig. 16. TEM micrograph showing BWINGs around (Ti, Mn) based complex oxide. The number on interface between neighboring bainite laths is the number of nearly parallel slip systems between them. Small numbers below 7, including 0 are rather dominant around the oxide. Dotted line reflects small angle dislocation boundary.
reactions, resulting in the formation of faceted fracture surface (average size of 1 face being a few tens μm). At the bottom of the slender dimples, many MnS rods being indicated by arrows (having the Kurdjumov–Sachs orientation relationship with bainite) lie. It is reasonable that the MnS rod nucleates and grows along dislocation with edge component in SADN through the elastic interaction between the dislocation and both Mn and S. The dislocations in SADN would aggregate through both elastic interaction among them and reaction between dislocations, leading to periodical arrangement of dislocations as well as the MnS rods. The dislocation network tends to be flat to decrease its free energy. This result leads to the conclusion that SADN could be formed in austenite, corresponding to the faceted fracture surface. It is obvious that the introduction of SADN into austenite is enhanced by an addition of S. Sulfur might decrease mobility of dislocation through being dragged by dislocation. The decrease of the mobility of dislocation would increase the possibility for encountering dislocations, enhancing reaction between dislocations as well as inducing the formations of sessile dislocation and dislocation network. Dislocations would be introduced into austenite when reverse transformation, ferrite (α)+cementite (θ)→γ, occurs. This transformation would form thin γ at the interphase between α and θ, and thin layer of γ would be easily deformed by the stress generated through transformation because of the γ being with low density of dislocations. The fracture surface of the steel (B) is shown in Fig. 14. Ductile fracture occurs and dimples are clearly observed. However, MnS particles are scarcely observed. The average size of dimples is larger than that of a bainite lath. It is obvious that size of dimple correspond to that of aggregate of bainite laths.

It should be noted that nearly parallel slip systems, \((111)_\alpha\) and \((112)_\gamma/(110)_\gamma\), in some variants of bainite laths operate in a dimple region. The fraction of nearly parallel slip systems (both slip plane and slip direction) all of combination of slip systems in neighboring variants (2 variants) is about 2.5%, on the other hand, that in poly-crystal with random crystallographic distribution of grains is about 0.1% (analytical solution). Namely, the fraction of nearly parallel slip systems in BWING is higher than that in polycrystal. The number of nearly parallel slip systems (within 15° with respect to both slip plane and slip direction) in neighboring bainite laths were also analyzed. The result of calculation for the dependence of the number of combination of variants with nearly parallel slip systems on the number of nearly parallel slip systems between neighboring bainite lath is shown in Fig. 15. The average number of parallel slip systems in neighboring bainite lath only with nearly parallel slip systems is about 7, which is larger than that (around 2) between random oriented grains in the case of, so called, poly-crystals. It should be emphasized that the size of the dimple on fracture surface corresponds to the Aggregate of bainite Laths with nearly Parallel Slip systems between neighboring bainite laths (hereafter, referred to as ALPS) according to the results obtained by TEM. However, the possibility of existence of ALPS determined by TEM are so large, being beyond one predicted by the calculated result, 2.5%. The unexpected larger possibility of ALPS existence obtained by TEM would be due to the preferred formation of the neighboring bainite lath, which relaxes easily the transformation stress around former bainite lath. In the cases of heterogeneous nucleation of BWING around inclusion or SADN, size of ALPS might depend greatly on the stress field around them. In the case of the size of inclusion below 1 μm in average, ALPS consists of 5 variants in average in steel SR austenitized at 1 400°C. As shown in Fig. 16, the boundary of ALPS with the number of nearly parallel slip systems between neighboring variants being 0 exists rather dominantly around inclusion. The size, shape and crystal structure of inclusion might control the size and boundary (the number of nearly parallel slip systems between neighboring variants being 0 or smaller than 7) configuration of ALPS. Size of ALPS also could be controlled by the number density of nucleation site of BWING. In the case of Fig. 16, nucleation site would be supplied by the large stress field around inclusion, some bainite laths nucleating independently. The each bainite lath nucleated once around inclusion would induce the neighboring bainite lath with the slip system nearly parallel to that in the nucleated bainite lath, namely, corresponding to the formation of ALPS. It would be possible that the boundary of ALPS generates at the inclusion. Ductile fracture might occur along the ALPS boundary.

![Fig. 14. Scanning electron micrograph taken from the fracture surface in steel SR austenitized at 1 400°C. This fracture surface was obtained after tensile test (stress vs. strain curve (B) in Fig. 12).](image)

![Fig. 15. The number of combination of variants with parallel slip systems as a function of the number of parallel slip systems between neighboring bainite laths.](image)
Also in the case of the nucleation of bainite lath at SADN, size of ALPS might depend on the number density of nucleated bainite lath. However, in the above case, accumulation of high density of dislocations at interface of inclusion, which induces cleavage, never occurs. The slip systems in neighboring laths within an ALPS are “nearly parallel” with each other, inducing accumulation of relatively low density of dislocations per a unit area of interface between bainite laths as well as work-hardening under deformation. The stress concentration would be relaxed by the deformation of the interface between neighboring bainite laths as shown in Fig. 3 of the Ref. 7). The area of the interface between neighboring bainite laths increases as deforming the bainite laths, lowering the dislocation density at the interface between bainite laths. It could be emphasized that the formation of ALPS leads to both strengthening (work hardening) and ductility.

Formation of BWING at SADN was enhanced by the cold rolling before austenitization in steel SP. Hardened $\alpha$ by the cold rolling would increase transformation stress into $\gamma$ when reverse transformation ($\alpha + \theta \rightarrow \gamma$) occurs, resulting in the introduction of dislocations as well as SADN into $\gamma$. The stress vs. strain ($s$–$s$) curve (c) in Fig. 17 shows that the BWING in steel SP without inclusion induces higher yield strength and elongation, compared with the $s$–$s$ curve (a) in steel SP, containing intergranular bainite. Smaller elongation in the $s$–$s$ curve (d) in Fig. 17 compared with that in the $s$–$s$ curve (b) in Fig. 17 is due to the cleavage of inclusion due to the cold rolling before austenitization. Relatively large work hardening in the $s$–$s$ curve (b) would be due to fine BWING nucleated at high density of MnS particles. Larger elongation in the $s$–$s$ curve (b) would be greatly promoted if no MnS particles exist in steel SR. The larger ALPS consisting of finer bainite laths without inclusion should be prepared in order to improve both strength and ductility.

4. Conclusions

(1) BWING nucleates at both inclusion such as MnS and SADN, and forms ALPS.

(2) ALPS consists of bainite laths with nearly parallel slip systems between neighboring ones. ALPS develops from one bainite lath as nucleation site, forming neighboring bainite lath, relaxing the stress field around former bainite lath. It could be suggested that when bainite lath forms contacting to existed one, the slip system in the bainite lath might be oriented nearly parallel to that in the existed one to relax the stress field around the existed bainite lath.

(3) The size of ALPS might be controlled by the density of nucleated bainite laths independently, becoming larger under the density being lower.

(4) The ductile fracture is controlled by the size of ALPS. The size of dimple on fracture surface nearly corresponds to the size of ALPS, ductile fracture (induced by dominant conservative motions of dislocations locally) proceeding along the boundary of ALPS (interface without parallelism of slip systems in neighboring bainite laths).

(5) The strengths of the steels would come from the fine bainite lath and fine ALPS. On the other hand, ductility might be developed by enlarging the size of ALPS.

(6) Toughness (lower ductile–brittle transition temperature) might be improved by forming “larger ALPS”.

(7) As for nucleation site of BWING, SADN is preferable compared with inclusion, because SADN would supply less nucleation site for cleavage.

Acknowledgements

The authors would like to express their sincere thanks to The Iron & Steel Institute of Japan for a part of support of this work by The ISIJ Research Promotion Grant. Thanks are also due to Mr. Norihiro KANNO of the Nippon Steel & Sumikin Stainless Steel Corporation for his cooperation, and the INCS, Ehime University for the use of the transmission electron microscope.

REFERENCES

1) Y. Kotani, Y. Kagawa, H. Ueda, K. Nakai, S. Kobayashi, T. Sakamoto, M. Hamada and Y. Komizo: CAMP-ISIJ, 22 (2009), 535.
2) M. Hamada, S. Okaguchi, Y. Komizo, A. Yamamoto, N. Takahashi, T. Ikeda and I. Takeuchi: Proc. of 4th Int. Conf. on Pipeline Technology, Scientific Surveys, Beaconsfield, UK, (2004), 1077.
3) J.-H. Shim, Y.-J. Oh, J.-Y. Suh, Y. W. Cho, J.-D. Shim, J-S Byun and D. N. Lee: Acta Mater., 49 (2001), 2115.
4) R. Asakura, K. Nakai, S. Kobayashi, T. Sakamoto, N. Isomura, K. Manabe, M. Hamada and Y. Komizo: CAMP-ISIJ, 21 (2008), 525.
5) K. Nakai, R. Asakura, T. Sakamoto, S. Kobayashi, T. Yamada, H. Terasaki, M. Hamada and Y. Komizo: J. Jpn. Soc. Heat Treat., 49 (2009), 620.
6) K. Nakai, N. Kanno, S. Kobayashi, M. Hamada and Y. Komizo: The 16th Int. Cong. on Microscopy, Publication Committee of IMC16, Japan, (2006), 1681.
7) K. Nakai, T. Megumi, S. Kobayashi, M. Hamada and Y. Komizo: Tetsu-to-Hagané, 91 (2005), 882.