Formation of Stable Dislocation Network in Austenite and Its Effect on Nucleation of BWING in Bainitic Steels

by Tatsuaki Sakamoto**, Takaumi Hiramoto***, Kiyomichi Nakai****, Sengo Kobayashi ***,
Hiroaki Ohfuji***** and Tetsuo Iriun*****

Whether dislocation network could be introduced into austenite (γ) even during austenitization or not was analyzed by optical microscopy. Austenitization at 1450°C for 300s dissolves MnS completely into γ. It is clarified that isothermal holding at 900°C for 1200s after austenitization forms MnS along small-angle boundary within γ grain. It suggests that dislocation network forming the small-angle boundary is introduced and stable even in the high temperature region of γ. Serial sectioning observations of bainite laths with optical microscope reveals that BWING (bainite lath within γ grain) could nucleate from the dislocation network. Dislocation network forms by reaction of dislocations on each slip system. Each dislocation in a network would have different Burgers vector. Therefore, climbing rate of each dislocation in a network due to absorbing or generating vacancies might be different, resulting in bowing-out of dislocation belonging to a network and an increase in self-energy of a dislocation network. It could be concluded that dislocation network is stable even in γ and the network acts as nucleation site for BWING. Based on the conclusion, nucleation of BWING is enhanced through introduction of dense dislocation networks into γ.

Key Words: Bainite, Dislocation Network, Pretreatment, MnS, Serial Sectioning Observation

1. Introduction

Bainite laths within austenite grain (abbreviated to “BWING”) improve strength and toughness in steels1,2). BWING nucleates at inclusions such as oxides, sulfides and so on3,4). However inclusions are responsible for deterioration of ductility because inclusions act as initiation sites of crack propagation. Our group is investigating small-angle dislocation network (SADN) in austenite (γ) as an alternative nucleation site for BWING1,5). In order to enhance the introduction of SADN into γ, pretreatment before austenitization, such as isothermal holding at 500°C and cold rolling, was carried out in our previous work5,7). However existence of dislocation in γ is still not clear. In this paper, existence of dislocation in γ was examined in steels which were subjected to pretreatment before austenitization in order to introduce dislocation into γ.

2. Experimental procedure

Chemical compositions of steels used in this investigation are listed in Table 1. A high-carbon and sulfur-rich specimen is designated as HCSR, and a low-carbon and sulfur-poor one as LCSP. Heat treatments shown in Figs. 1(a) and (b) were performed for HCSR and LCSP, respectively. For HCSR, austenitization was firstly performed at 1400 or 1450°C for 300s. Then isothermal holding was carried out at 900°C for 1200s for MnS formation, and finally at 500°C for 10s for bainite formation, followed by iced brine quenching (IBQ). This heat treatment was done in order to examine the existence of SADN in γ by decorating dislocations with MnS. For LCSP, pretreatment was firstly performed at 750°C for 180s, followed by austenitization at 1400°C for 300s. Then isothermal holding was carried out at 900°C for 1200s for MnS formation, and finally at 500°C for 10s for bainite formation, followed by IBQ. Pretreatment was performed in order to enhance the introduction of dislocations into γ during austenitization5,7). Microstructure was observed with an optical microscope after etching specimens with nital. In order to investigate where BWING nucleates, serial sectioning observation was carried out by repeating optical microscopic observation and specimen thickness reduction alternately. The specimen thickness was reduced by mechanical polishing with alumina powder. The decreased thickness was calculated from a

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**Department of Materials Science and Biotechnology, Ehime University, 3 Bunkyo-cho, Matsuyama 7908577, Japan
***Graduate Student of Ehime University
****Professor Emeritus of Ehime University
*****Geodynamics Research Center, Ehime University

Table 1 Chemical compositions of steels investigated (mass%).

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCSR</td>
<td>0.13</td>
<td>0.29</td>
<td>1.44</td>
<td>0.001</td>
<td>0.062</td>
</tr>
<tr>
<td>LCSP</td>
<td>0.076</td>
<td>0.25</td>
<td>1.45</td>
<td>0.001</td>
<td>0.008</td>
</tr>
</tbody>
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diagonal length of a Vickers indentation left on specimen surface and a vertex angle between opposite edges of an indenter as shown in Fig. 2.

Fig. 1 Schematic illustration of heat treatment in (a) HCSR and (b) LCSP.

Fig. 2 Calculation of the thickness reduction using a Vickers indentation. (a) a plan-view and (b) a cross-sectional view of Vickers indentation.

3. Results

Figure 3 shows optical micrographs of HCSR. The initial structure of HCSR consists of ferrite (α), pearlite and aligned precipitates of MnS encircled by dashed lines (Fig. 3(a)). Ferrite is a white grain, and pearlite is a black area inside α. Pearlite and MnS are distinguishable due to the difference of morphology between them. MnS has a granular shape but pearlite does not. The aligned precipitates of MnS are probably produced during the rolling which was carried out before the specimen was given to the present study. Some of the aligned precipitates of MnS still remain after austenitization at 1400°C (Fig. 3(b)), whereas they disappear after austenitization at 1450°C. It is noted that austenitization at 1450°C dissolves MnS completely into γ (Fig. 3(c)). Isothermal holding at 900°C after austenitization at 1450°C re-precipitates MnS in a network configuration (Fig. 3(d)). It is seen that MnS shown in Fig. 3(d) does not form on γ grain boundary because BWINGs form across a line of MnS precipitates. In other words, if the line of MnS corresponded to γ grain boundary in Fig. 3(d), prior γ grain boundary could readily be recognized with a sharp contrast because bainite laths form without crossing the γ grain boundary. However, such a sharp contrast is not observed in Fig. 3(d).
Therefore, MnS probably forms on small-angle boundary, that is, SADN. It is noticed that SADN could be introduced into γ and stable even in the high temperature region of γ as discussed later.

Figure 4 shows the results of the serial sectioning observation in order to investigate where BWING nucleates in LCSP. Figures 4(b) and (c) were taken from the specimens whose thicknesses were decreased by 1.48 and 3.79 μm from (a). As can be seen from Fig. 3, SADN can exist in γ during austenitization, and MnS precipitates on SADN during the heat treatment of MnS formation. In LCSP, the introduction of SADN could be enhanced by pretreatment 5-7). Therefore, MnS probably precipitates at SADN in Fig. 4. Although an exact configuration of SADN in Fig. 4 is difficult to be seen due to lower content of sulfur than HCSR, two neighboring particles of MnS might form on the same SADN. It can be considered that SADN exists along the dashed line in Fig. 4. In Fig. 4(a), a BWING shown by a black arrowhead is observed in contact with SADN. A white arrowhead indicates the closest edge of the BWING to SADN. Serial sectioning micrographs from Fig. 4(a) to (c) show that the BWING separates from the SADN as the specimen thickness decreases. It is indicated that the BWING, which is long along <111>α and thin along the direction perpendicular to {110}α including the <111>α 8), nucleates at SADN and grows obliquely, i.e., grows both from the SADN and toward the depth. Therefore, this serial sectioning observation indicates that BWING could nucleate at SADN.

4. Discussion

It has been often reported that dislocations existing at room temperature disappear at high temperature where γ is stable in steels. However, Fig. 3(d) suggests the existence of small-angle boundary in γ although the temperature is very high. In general, small-angle boundary consists of dislocation array or dislocation network. At high temperature, dislocations which do not form a network might disappear more easily than dislocation network owing to their climb at high temperature. In contrast, dislocation network could exist stably because of the followings. Dislocation network forms by reaction of dislocations on each slip system. Not all dislocations connected at a node necessarily have same Burgers vectors 9). Therefore, climbing rate of each dislocation at a node due to absorbing or generating vacancies might be different from one another, resulting in bowing-out of each component dislocation of a network, namely, in increase in self-energy of a dislocation network. That is the possible reason for the relatively stable existence of SADN in the high temperature region of γ. It could be suggested that the formation of SADN is enhanced by pretreatment 5-7). Therefore, it is feasible in Fig. 4 that SADN exists in γ at high temperature.

In Fig. 4, MnS probably precipitates at SADN, although a network configuration is not clearly seen due to a low amount of precipitation of MnS. Some MnS precipitates do not exist in a line, and do not seem to constitute SADN. This is probably because these MnS precipitates nucleate at other dislocations which have slipped to SADN and reacted or tangled with SADN 9). It is also considered that the configuration of SADN might be irregular, and that the mesh of SADN might not be a regular hexagonal shape 9,10).

In Figs. 4(a-c), MnS indicated with arrow (A) can be seen. The diameters of these MnS are approximately 2μm.
Meanwhile, the reduction of thickness in this serial sectioning process is 3.79 μm. It is seen that MnS is in a rod-like shape. These MnS might nucleate at dislocation and form along it. Other MnS indicated with arrows (B) and (C) can be seen in Fig. 4. Unlike MnS of (A), MnS of (B) disappears in Fig. 4(c), and MnS of (C) appears from Fig. 4(b). Such appearance and disappearance of MnS could indicate that there are nodes of dislocation network, although the mesh of dislocation network may be irregular hexagon mentioned above. There is a possibility that dislocation network exists across the specimen surface.

SADN has strain field around itself. The strain field enhances the nucleation of BWING at SADN as shown in Fig. 4. It is noted that SADN acts as nucleation site for BWING like inclusions such as oxides or sulfides. After a BWING forms at SADN, another BWING would nucleate at the already existing BWING in the way of sympathetic nucleation\(^{11,12}\). However, the sympathetic nucleation alone might not cover the rest of microstructure with BWING. Although such a problem remains to be solved, it is worthwhile to stress in the present study that SADN probably exists at elevated temperature where γ is stable, and that SADN has a strong influence on the nucleation of BWING.

### 4. Conclusions

The existence of SADN in γ was investigated by the decoration with MnS on dislocations. MnS dissolves into γ during austenitization at 1450°C, and re-precipitates from γ during the isothermal holding at 900°C, resulting in the arrangement of MnS on a network, SADN.

Serial sectioning observation by optical microscopy reveals that BWING nucleates at SADN. SADN is likely to be stable even in γ. It could be concluded that SADN introduced into γ acts as a nucleation site for BWING, resulting in the enhancement of formation of BWING.

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### Reference