Abstract

Transformation Induced Plasticity (TRIP) steels are mostly used due to high mechanical properties in automotive industry. Especially the mechanical properties of these steels are related to the volume fraction of retained austenite. Therefore, accurate detection of the retained austenite amount is important. In this study, Electron Backscatter Diffraction (EBSD) and color etching methods were performed to measure retained austenite (RA) and phase characterization. Also sample preparation is another important issue for EBSD analyses. Therefore, electro polishing and mechanical polishing were carried out and results were compared with each other. The effect of sample preparation method on the volume fraction of the retained austenite was analyzed.

1. Introduction

The usage of TRIP in automotive industry increases remarkably because it has the great combination of the high strength and ductility. These steels microstructure consists of ferrite, bainite and retained austenite phases due to heat treatment applications. The steel grade has a significant amount of austenite phases (5-30%). TRIP steels exhibit excellent formability properties due to the metastable retained austenite transforms to the martensite (\(γ \rightarrow α′\)) with plastic deformation. Therefore, sample preparation is important process in order to determine sensitively volume fraction of RA [1-3].

There are many studies dealing with the volume fraction of RA measurement and comparison of different measurement methods in literature. However there is limited information sample preparation. Studies generally focus on only electro polishing or mechanical polishing as sample preparation but there is not any study both polishing methods are used [1-2, 4-6].

In this study, different sample preparation methods such as argon ion milling, electro polishing and mechanical grinding & polishing were carried out in order to determine volume fraction of RA sensitively. In this scope EBSD method was performed on each sample surface and results of measurements were compared with each other. Light optical microscope (LOM) and scanning electron microscope (SEM) were used for microstructure characterization.

2. Experimental Procedure

2.1. Material and Sample Preparation

The material was used in this study consisted of cold rolled TRIP steel with a thickness of 1.25mm.

Two different methods were performed for sample preparation. Mechanical polishing is a conventional method and samples were cut in cross-section, hot mounted, grinded and polished respectively. For this analysis, samples were additionally polished with colloidal silica with 0.05 \(\mu\)m particles. Second method is electro polishing and mechanical polishing without colloidal silica is followed by electro polishing. Struers A2 electrolyte and 10 kV voltage were applied with different times (10, 20, 40 and 60 sec.) as an electro polishing conditions. Electro polish was evaluated as function of time. After the sample preparation process, all samples were characterized by using the EBSD (Oxford Nordlys Nano EBSD detector).

2.2. Microstructural Characterization

In order to determine the volume fraction of RA and discriminate phases (bainite, ferrite, martensite and retained austenite), the polished surface was etched with LePera and characterized by light optical microscope (Nikon Eclipse MA200). SEM (Jeol JSM 7100F) was also used for detailed microstructural characterization.

All sample surfaces were analyzed with EBSD method. Working distance, acceleration voltage and step size were selected as 17mm, 20kV and 0.1\(\mu\)m respectively for EBSD conditions. All EBSD measurements were carried out in the transverse direction (TD) plane.
3. Results and Discussion

Image of the microstructure with etching LePera is given in Fig. 3. LePera etching allows to distinguish ferrite from martensite and retained austenite due to color etching. However retained austenite and martensite phases are not separately identifiable because retained austenite and martensite appears white. There are a need for additional methods such as EBSD, XRD, magnetic measurements [7]. Fig. 2 shows SEM image of microstructure with etching 2 % Nital.

Figure 1. Light optical microscope image of microstructure with etching LePera (white: martensite/retained austenite; dark brown: bainite; blue: ferrite).

Figure 2. SEM image of microstructure with etching 2 % Nital (M/RA: martensite/retained austenite; F: ferrite; B: bainite).

Whereas ferrite, martensite and bainite have body centered cubic (bcc) structure, retained austenite has face centered cubic (fcc) structure. Therefore EBSD can easily distinguish between fcc and bcc due to different crystallographic structure. In this way the austenite is determined sensitively. The image of Fig. 3 was obtained from image quality and color mapping. The color of retained austenite in this image is pink and ferrite and bainite phases are blue. EBSD results are also given in Table 1. Results of mechanical polishing and electro polishing are close. However the measurement results obtained from electro polishing, except of 60 seconds, are lower than mechanical polish. Because electro polishing in operating conditions could not eliminate effect of the remaining damage layer from last polishing step. As shown in Table 1, amount of zero solution slightly increases from 6.23% to 8.10% with in time due to the rise in relief.

The volume fraction of RA increased with decreasing load (from 30N to 15N) in conventional mechanical polishing because transformation from austenite to martensite is inhibited with the decline of the applied load.

Table 1. EBSD results determined by means of different sample preparation.

<table>
<thead>
<tr>
<th>Methods</th>
<th>RA volume fraction (%)</th>
<th>Zero solution fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Mechanical polish</td>
<td>9</td>
<td>6</td>
</tr>
<tr>
<td>2 Mechanical polish with low loading in last step</td>
<td>14.96</td>
<td>8.50</td>
</tr>
<tr>
<td>3 Electro polish (10V 10sec.)</td>
<td>12.00</td>
<td>6.23</td>
</tr>
<tr>
<td>4 Electro polish (10V 20sec.)</td>
<td>12.15</td>
<td>6.48</td>
</tr>
<tr>
<td>5 Electro polish (10V 40sec.)</td>
<td>13.07</td>
<td>6.77</td>
</tr>
<tr>
<td>6 Electro polish (10V 60sec.)</td>
<td>15.00</td>
<td>8.10</td>
</tr>
</tbody>
</table>

Figure 3. Image Quality and color mapping obtained from EBSD measurements.
4. Conclusion

TRIP steel was characterized by using LOM, SEM and EBSD. Especially EBSD examinations carry out a few nano meters under the sample surface. Therefore sample preparation process is important since it affects directly the measurement results. The main results can be summarized as follows:

- Last polishing step with colloidal silica in mechanical sample preparation is more effective than low time electro polishing applications.
- RA volume fraction increases by decreasing load in mechanical polishing due to transformation to martensite.
- As electro polishing time increases, effect of etching increase. Therefore the highest RA amount was obtained at 6th experimental study.

References


