1. Introduction

Many industrial applications involving both high structural performance and good component formability require high ductility and strength. As these two properties are generally antagonistic for most materials, it is difficult to improve both properties simultaneously. Transformation-induced plasticity (TRIP)-assisted multiphase steel, which combines high strength with better formability than conventional steels of similar strength, has attracted much interest over the past 10–15 years due to its perceived capability to conciliate these two properties.\(^1\)\(^{-6}\)

TRIP-assisted multiphase steels generally consist of ferrite, carbide-free bainite, retained austenite and martensite.\(^2\)\(^,\)\(^3\) The high performance of the TRIP-assisted multiphase steel results from the mechanically-induced martensitic transformation (MIMT) of the retained austenite accompanying TRIP during deformation.\(^7\)\(^,\)\(^8\) As a consequence, both the strength and elongation of the TRIP-assisted multiphase steel increased during uniaxial tension due to the appearance of a harder phase.\(^6\)\(^,\)\(^8\)\(^,\)\(^9\) However, the transformed martensite has been reported to provoke the reverse effect on the enhancement of the drawing limit.\(^8\)\(^,\)\(^9\) This was described by the inference that the micro-cracks, which are formed either by the decohesion at the ferrite-martensite interface or the shear fracture between martensite islands, may decrease the drawing limit of the material.\(^8\)\(^,\)\(^9\) Therefore, from a fundamental point of view, further studies on the fracture of TRIP-assisted multiphase steel to investigate its fracture toughness, stress state dependence and cracking position are very important for its application.

In this study, the position of crack initiation in a severely drawn TRIP-assisted multiphase steel was investigated by using a focused ion beam (FIB), transmission electron microscopy (TEM) and energy dispersive spectroscopy (EDS). A novel FIB method, termed the In-Plane Lift-Out Technique (IPL), was used to obtain an overall plan-view of the TEM specimen containing the micro-crack. The equilibrium concentration ratio of Mn in austenite and ferrite was calculated for the alloy and compared with the EDS line profile of Mn concentration across the micro-crack in the TEM specimen. The interface between the ferrite and martensite was identified as the crack initiation site during drawing of this multi-phase steel.

KEY WORDS: TRIP steels; crack initiation; focused ion beam (FIB); transmission electron microscopy (TEM).
established techniques in TEM specimen preparation. In recent years, a more advanced “lift-out” technique has proven to offer unique advantages in cases where mechanical preparation is difficult or impossible. Especially several methods for preparing plan-view specimen using the focused ion beam (FIB) were introduced. In this study, the specimen was prepared in a FIB (FEI NOVA200) equipped with a Schottky field emission gun column, Ga ion beam column, Pt gas injection system, and Omniprobe internal micromanipulator. At an energy of 30 keV, a focused Ga ion beam was used for the machining under a beam current from 1 to 20 nA.

3. Results and Discussion

3.1. IPLOT to Obtain an Overall Plan-view of the TEM Specimen Containing the Micro-crack

Figure 1 shows the overall procedure of the IPLOT to prepare a plan-view TEM specimen containing a micro-crack. A 2.8 μm-long micro-crack running parallel to the drawing direction is evident in the material, as shown in Fig. 1(a). Pt was deposited on to the micro-crack by a Pt gas injection system to protect the micro-crack from damage caused by the focused Ga ion beam. Four landmarks around the deposition region were then presented to locate the position of the micro-crack, as shown in Fig. 1(b). A square pillar containing the specimen surface, in which the micro-crack exists, was machined by the focused Ga ion beam, as shown in Fig. 1(c). After one side of the square pillar was obliquely sliced to a thickness of about 3 to 8 μm, the remaining sample containing the feature of interest was lifted out by the Omniprobe internal micromanipulator, as shown in Figs. 1(e) and 1(f). This sample was then mounted onto a copper TEM grid using the Omniprobe internal micromanipulator, prior to final thinning, as shown in Figs. 1(g) and 1(h). The final ion milling was carried out under a very small beam current of 1 nA to minimize the damage by the focused Ga ion. A 65 nm-thick TEM sample was then finally obtained, as shown in Fig. 1(i).

3.2. Identification of Phases around Micro-crack at the Initial Stage of Cracking

Figure 2 shows a bright field TEM image of the TRIP-assisted multiphase steel tempered at 525°C for 32 h before drawing. The phases could be identified by the selected area diffraction pattern. (011) ferrite coincides with (111) austenite plane satisfying the Kurdjumov–Sachs orientation relationship. It can be confirmed that the morphology of retained austenite was close to that of the lath shape. The volume fraction of the retained austenite according to XRD was 42%. Figure 3 shows the effect of drawing strain variation on the crack density of the TRIP-assisted multiphase steel. Only cracks more than 0.5 μm long in the secondary electron image of 1000 magnification were counted. It is well known that the drawing limit of the material is decreased with increasing crack density during drawing. The results presented in Fig. 3 confirmed that crack density was
continuously increased with increasing drawing strain. The retained austenite also underwent MIMT with increasing drawing strain. Figure 4 shows that the volume fraction of retained austenite was decreased with increasing drawing strain and after the drawing strain of 2.5, most of the retained austenite transformed to martensite. Therefore, it was very difficult to identify the phases around the micro-crack in the severely drawn TRIP-assisted multiphase steel from only the SEM image due to the very thin lamellar structure and variety of phases in the drawn specimen.

In this study, the position of crack initiation in the drawn TRIP-assisted multiphase steel was investigated from the TEM observation of the sample surface containing the micro-crack prepared by IPLOT. Figure 5 shows the TEM image of the whole 2.8 μm-long micro-crack. EDS analysis revealed that the inside of the micro-crack was filled with Pt, which was attributed to the Pt deposition to protect the micro-crack from damage by the focused Ga⁺ ion. Positions A and B in Fig. 5 indicate the crack tip of the micro-crack. Figure 6 shows a TEM image of the crack tip. Since the micro-crack was filled with Pt, the neighboring microstructures are clearly distinguished, as shown in Fig. 6. The microstructure of the left side of the micro-crack was a thin lamellar type and the right side of the micro-crack had a wider morphology. The phases both of the left and right sides of the crack tip were confirmed as BCC crystal structure by their diffraction pattern. This indicates three possibilities for the cracking position: ferrite grain boundary, ferrite–martensite interface and martensite lath boundary.

To identify the phases around the micro-crack, the equilibrium concentration of Mn in austenite and ferrite was calculated for the alloy using Thermo-Calc. The thermodynamic calculation gave the result that the equilibrium concentration ratio of Mn in austenite and ferrite is 10 to 3. The austenite in the specimen was expected to mechanically transform to martensite during drawing while the Mn concentration in the martensite was remained constant. As shown in Fig. 6, the EDS line profile for Mn concentration across the micro-crack at the crack tip revealed the identity of the phases around the micro-crack. A Mn build-up was observed at the left side lath, indicating that the left side of the micro-crack was martensite that had been mechanically transformed during drawing and that the right side of the micro-crack was ferrite phase. This FIB-TEM analysis confirmed that the micro-crack was initiated at the interface between the ferrite matrix and the mechanically transformed martensite during the wire drawing of the TRIP-assisted multiphase steel. This can also be confirmed from Figs. 3 and 4. As shown in Figs. 3 and 4, the volume fraction of martensite as well as the crack density increases as the true strain increases. The increase of the crack density with the martensite volume fraction may be closely related to the increase of the crack initiation site of the interface between the ferrite matrix and the mechanically transformed.
martensite.\(^9\)

In TRIP-assisted multiphase steel, the increase in the volume fraction of deformation-induced, hard martensite increases the hardening rate, which thereby improves the material elongation. In addition to this beneficial influence, the mechanically transformed martensite has a negative effect on the elongation enhancement, possibly due to the decohesion at the interface of the ferrite matrix and mechanically transformed martensite with extended deformation.\(^4,8,10\)

4. Conclusion

The crack initiation in a drawn TRIP-assisted multiphase steel was investigated by using FIB, TEM and EDS. A novel FIB method, termed the In-Plane Lift-Out Technique (IPLAT), was applied to obtain an overall plan-view of the TEM specimen containing the micro-crack and to identify the phases around the micro-crack at the initial stage of cracking. The results obtained from this technique confirmed that the micro-cracks were initiated at the interface between the ferrite and the martensite that had been mechanically transformed during drawing of the TRIP-assisted multiphase steel.

Acknowledgement

This work was supported by the Korea Science and Engineering Foundation (KOSEF) grant funded by the Korea government (MOST) (R0A-2007-000-10014-0).

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