Original Article

Effect of microstructure quenched around $A_{c3}$ point on the damage behavior in 0.087C–1.35Mn steel

Chengyu Guo\textsuperscript{a}, Leilei Hao\textsuperscript{a}, Shengci Li\textsuperscript{b}, Yonglin Kang\textsuperscript{a,∗}, Yuguo An\textsuperscript{c,∗}

\textsuperscript{a} School of Materials Science and Engineering, University of Science and Technology Beijing, China
\textsuperscript{b} School of Materials Science and Engineering, Jiangxi University of Science and Technology, China
\textsuperscript{c} Research and Development, Tata Steel, 1970 CA Ijmuiden, The Netherlands

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\textbf{ABSTRACT}

The present study was aimed at microstructure quenched around $A_{c3}$ point and its effect on the damage behavior. Thermo-Calc was used to ensure the $A_{3}$ while the $A_{c3}$ was identified by means of heat treatments on DIL805A thermal dilatometer, and the quenching temperatures were determined according to the above parameters. The scanning electron microscopy (SEM) and electron back scattering diffraction (EBSD) were carried out to observe the microstructure. In-situ tensile tests with SEM were performed and the development of microcracks were recorded. The results illustrated that the microstructure depends on the quenching temperature: a dual phase microstructure of martensite islands with ferrite around them is observed when the material is quenched below $A_{3}$, approximately 842 °C. Ferrite near martensite island and interface between two martensite islands with different misorientation are generally the weak spots in microstructure. When the material is quenched from austenite region around or above $A_{c3}$, approximately 880 °C, martensite with lath submicrostructure is obtained. The location with large misorientation between two packets is usually the weak spot in microstructure and leads to increased possibility of microcracks.

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1. Introduction

Advanced high strength steel (AHSS) is widely used in automotive industry to meet the requirement of weight reduction and to improve safety for automobile body. Application of dual phase (DP) steel is one of the most efficient ways to reduce fuel consumption and protect environment, while maintaining vehicle safety with light weight [1–3].

The microstructure of DP steel is ferrite matrix and martensite, which has a good combination of strength and ductility with continuous yielding and high initial work hardening rates. The dual phase microstructure is obtained by quenching from austenite to martensite at a temperature in two phase region. The DP steels consisting of martensite phases dispersed in ferrite matrix shows an improvement on strength with an increasing martensite volume fraction by controlling the heat treatment process. The volume fraction of martens-
site in the microstructure leads to different strength levels in several grades of DP steels. Microstructure of martensite in DP steel is a typical kind of martensite in island, which leads to generation of microcrack around the island easily as the hard phase [4–8].

Another typical microstructure of martensite is lath martensite, which is one of the essential phase in high strength and ultra-high strength steels. Several investigators have shown that lath martensite has a hierarchical structure composing of prior austenite grains, packets, blocks and laths, as shown in Fig. 1 [9–11]. The strength and toughness of martensitic steels are strongly related to its effective grain sizes in lath martensite structure. Some researchers considered that packet size is the effective grain size [12–14], while some other studies indicate that block size governs the effective grain size [15–17]. Grain refinement was effective in improving the resistance to cleavage fracture by introducing barriers to crack propagation. Besides, packet boundaries and block boundaries hold similar ability to impede the propagation of crack [18]. So it is important to understand the effect of martensite microstructure on the initiation and development of microcracks.

Micro-mechanical testing techniques have been used frequently with the development of MEMS technology. Micro-indentation testing were used to investigate the effect of the block and sub-block boundaries on the strengthening of the lath martensite structure, while complicated stress distribution of the bending test may not be appropriate for analyzing the activated slip systems. The deformation behavior of lath martensite structures with several types of boundaries in carbon steel was investigated by micro-tension testing technique [19]. In-situ tension can be used to observe the generation and propagation of microcracks in different kinds of martensite.

The present study was aimed at microstructure quenched around Ac3 point and its effect on the damage behavior. In this study, the A3 temperature was calculated using Thermo-Calc® software, while the Ac3 was identified by means of heat treatments on DIL805A thermal dilatometer. Materials were quenched around Ac3 to make samples for further study. The scanning electron microscopy (SEM) and electron back scattering diffraction (EBSD) were carried out to observe the microstructure. In-situ tensile tests with SEM were performed and the development of microcracks were recorded.

2. Materials and experiments

2.1. Materials

A low carbon DP600 steel was used as the original material with a thickness of 2.16 mm, whose specified chemical composition is (in wt.%): 0.087 C, 1.35 Mn, 0.38 Si, 0.014 P, 0.0065 S, 0.012 Nb, 0.0022 N and Fe balance. The original materials were quenched with different temperatures around Ac3 to obtain different microstructures.

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**Fig. 1 – Schematic illustration showing hierarchical structure of lath martensite.**

**Fig. 2 – (a) Equilibrium phase diagram for given steel composition from Thermo-Calc, (b) local enlarged drawing of the red box in (a) showing the A3.**
2.2. Quenching temperature predictions and quenching experiments

Commercial software Thermo-Calc was used to generate equilibrium phase diagrams for predicting upper critical temperature (A$_3$). Steel composition is the main input parameter, and the equilibrium phase diagram of given steel is shown in Fig. 2. The upper critical temperature (A$_3$) was predicted as 842°.

For the non-equilibrium, the upper critical temperature in heating (A$_{c3}$) is higher than the upper critical temperature in equilibrium (A$_3$) because of superheat. In order to ensure the A$_{c3}$ for given composition, heat treatments of quenching at temperature higher than A$_3$ by DIL805A thermal dilatometer were carried out. The samples in size of $4 \text{ mm} \times 10 \text{ mm}$ were heated to 840°, 870°, 900° and 930° at 10°/s, held for 3 min, and cooled at 50°/s to the room temperature. The results in temperature-dilation curves are shown in Fig. 3. The determined A$_{c3}$ temperature is around 880° using the tangent method on the measured temperature-dilation curves.

Based on the A$_{c3}$ temperature, the DP600, 100 mm × 100 mm in size, were heated to 810°, 840°, 870° and 900° for 20 min in resistance furnace, followed by water quench to room temperature. The quenched samples were coded as WQ810, WQ840, WQ870 and WQ900, respectively. They were used further for microstructure and substructure characterization, tensile testing and in-situ observation. Scanning electron microscope (SEM) micrographs were obtained from specimens after grinding, polishing and erosion with 4 vol% nital for 15 s to show the microstructure of martensite. Electron backscatter diffraction (EBSD) was used to achieve morphology information of submicrostructure by electro-polishing with 5 vol% perchloric acid.

2.3. Tensile test

Uniaxial tensile tests were performed by MTS 810 at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$, using specimens with a gauge length of 25 mm, and a gage width of 6 mm, as shown in Fig. 4.

2.4. In-situ tensile test and fracture observation

In-situ tension tests with SEM observation were performed by MT-M0560 at a speed of 0.1 mm/min with micro-tension device. The samples after quenching were cut into dog-bone shape with gauge dimensions as shown in Fig. 5. The surface of samples were polished, followed by etching with 4 vol% nital for 15 s to show the microstructure. A length of 0.5 mm notch was designed in the middle of gauge for the convenience of observing microcrack growth with the observation area in the bottom of the notch.

3. Results and discussion

3.1. Microstructure of the DP600 steel

Fig. 6 shows that the microstructure of the DP600 are ferrite and martensite. Microstructure of the martensite is martensite rim in the edge of austenite islands, which is a typical kind of martensite called martensite-austenite island. The diffusion rate of manganese in ferrite is three times higher than in austenite, making the formation of manganese-rich rim. As a result, the hardenability of the edge portion is higher than the
central portion for austenite islands, producing a martensite rim in the edge after quenched.

3.2. Microstructure of the martensite quenched around \( A_{c3} \)

The SEM micrographs of samples quenched at different temperature showed the microstructure of martensite quenched around \( A_{c3} \), as shown in Fig. 7. Different microstructure of martensite can be observed at different quenching temperature. The microstructure of WQ810 and WQ840 is ferrite and martensite in island, which is similar to the martensite-austenite island in DP600. Furthermore, there are some martensite islands in ferrite grain when the material was quenched at 840 °C, which is seldom observed in samples quenched at 810 °C. The microstructure of WQ870 and WQ900 is full martensite with lath submicrostructure, which is a typical martensite in low carbon steel after fast quench treatment.

The IPF color maps of WQ840, WQ870 and WQ900 samples showed the orientations of martensite and ferrite with their distribution, as shown in Fig. 8. The white lines in Fig. 8(a) and (b) or the red lines in Fig. 8(c) presented the low angle grain boundaries (LAGBs) with misorientation lying between 3° and 15°. The black lines in the three IPF maps describe the high angle grain boundaries (HAGBs) with misorientation larger than 15°. The WQ840 sample in Fig. 8(a) showed a typical martensite in island distributed on grain boundaries or within ferrite grains. For WQ870 and WQ900 with martensite lath, the prior austenite grain boundary, packet boundary and block boundary were basically all high angle boundary as shown Fig. 8(b) and (c). The packets contained a group of blocks with the same or similar extension directions, but the orientations could be totally different. The morphology of lath was not regular either.

The WQ810 and WQ840 were held for 20 min in two phase region, and led to a small fraction of fine austenite. The existence of huge amount of ferrite reduces the martensitic transformation shear and limits the generation of martensite lath. The small size of austenite and the limited generation of martensite lath lead to the microstructure of martensite in island. For WQ870 and WQ900, almost complete austenizatation during heating stage followed with rapid cooling bring martensitic transformation shear and the microstructure of lath martensite.

3.3. Tensile properties

Fig. 9 shows the engineering stress-strain curves for the four samples quenched from 810 °C to 900 °C. The mechanical
properties measured in the tensile test results are summarized in Table 1. It is clear that higher temperature quenching led to higher strength but lower ductility with continuous yielding phenomenon. The change in strength and ductility is remarkable when quenching was conducted below 870° in dual-phase region. Increase in quenching temperature from 870° to 900° resulted in less change the mechanical properties, which is clearly related to the similar microstructure of lath martensite generated at both temperatures.

3.4. In-situ tensile test

The SEM micrographs of the sections near the notch of in-situ tension samples of WQ810 and WQ900 at zero loading are shown in Fig. 10(a) and (b), respectively. With increasing tension, initiation and propagation of microcracks can be observed. At the same time, the strain distribution can be calculated by DIC algorithm, which will be described in detail below.

3.4.1. Strain distribution in in-situ tension

The SEM micrographs of the sections near the notch of in-situ tension samples of WQ810 at an in-situ tension of 1330 N is shown in Fig. 11(a), and that of WQ900 at an in-situ tension of 1200 N is shown in Fig. 11(b). Clear plastic deformation can be observed when the SEM micrographs in Fig. 11 are compared with those in Fig. 10 at zero loading. The strain distribution at different loading can be calculated by the DIC algorithm.
The DIC algorithm is based on the comparison of the initial and final locations of the characteristic points, which can be the martensite island or lath, the precipitates and even some impurities. With the specimen loading, the gauge part got longer and the characteristic points would move and rotate. The essence of the DIC algorithm is to calculate the displacement of characteristic points and relevant strains. A color bar is generated to show the strain distribution clearly. The strain concentration in Fig. 12 caused by the cracks shown in Fig. 11 extends along the direction with an angle of 45° to the loading direction, which is the shear direction. The strain distribution is related to the propagation of microcracks, which will be discussed next.

The SEM microstructure of the sample at 1700 N is shown in Fig. 13(a), together with the strain distribution at the same location at an early stage at a load of 1330 N as shown in Fig. 13(b). In comparison with the SEM microstructure at 1330 N in Fig. 11(a), large deformation and several new microcracks became visible at 1700 N. The new microcracks formed in the red box in Fig. 13(a) are at the positions of originally high strains illustrated in black box in Fig. 13(b). Though the strain distribution around the cracks is difficult to calculate due to large displacement, it is clear that localized shear strain play a very important role in crack propagation.

3.4.2 Microcracks generation and propagation in WQ810
The weak spot in microstructure for microcracks formation has been observed in detail as seen in Fig. 14 for WQ810. It can be seen that some microcracks generated in ferrite at locations near the boundary with martensite. In other cases, a few microcracks generated at locations between two martensites with different orientation. It is clear that the ferrite near martensite and the interface between two martensites with different orientation are the weakness in microstructure.

Fig. 15 shows the microcracks propagation and microstructure around microcracks in a few continuous stages along the red line for WQ810 as illustrated from (a) to (c). The microstructure around the red lines with less deformation are simpler to observe than the microstructure around microcracks. Fig. 15(d) shows complete morphology of the long microcrack, which is divided into 3 parts. Part 1 is the initial microcrack with irregular microstructure around it as shown in Fig. 15(a). Part 2 is the beginning stage of microcrack propagation as in Fig. 15(b). The crack propagates mainly in the direction with an angle of 45° to the loading direction, which is the shear direction during tension. Part 3 is the stable stage of microcrack propagation with the direction roughly perpendicular to the loading direction, which illustrates that the influence factor of the propagation path in Part 3 is the microstructure mainly with less effect from shear stress.

Fig. 16(a) shows the microcrack in Part 4 in Fig. 15(d). Fig. 16(b) shows the original microstructure of the same region with red arrows indicating the microcrack path. The microcrack can be devided into two parts, part 5 and part 6, with two deflection points on the microcrack. Microcrack in part 5 mainly propagates along the grain boundaries with sev-
Fig. 12 – Distribution of strain in various stress for different samples solved by DIC algorithm (a) WQ810 at 1330 N, (b) WQ900 at 1200 N.

Fig. 13 – (a) The SEM microstructure at 1700 N for WQ810, (b) the strain distribution at the same position at 1330 N.

Fig. 14 – Microcracks generation and morphology of the microcracks in WQ810 at 1890 N.
Fig. 15 – Microcracks propagation and microstructure around the microcracks in WQ810, (a), (b), (c), (d) microcracks generating at 1890 N, 1900 N, 1910 N and 1920 N.

Fig. 16 – Microcracks in Part 4 (a) and the original microstructure around the microcrack with path in red lines (b).

3.4.3. Microcracks initiation and propagation in WQ900
Fig. 17(a) and (b) shows two typical microcrack morphology at 1260 N for the WQ900 material. Typical lath martensite is shown and the orientations of martensite packets are marked with red arrows as shown in Fig. 17(a) and (b). Fig. 17(a) indicates that the misorientation between two packets on both sides of the microcrack is greater than 45° (55°), while the misorientation is close to 90° (80°) in Fig. 17(b). The large misorientation leads to the weakness and increases the possibility of microcrack initiation, which is more likely to happen at prior austenite grain boundaries or packet boundaries.

Fig. 18 illustrates the microcrack propagation and morphology in WQ900. Fig. 18(a)–(c) shows the propagation of a microcrack with red lines. Fig. 18(d) shows complete morphology of the microcrack, which is divided into 3 parts. Part 1 is the microcrack with irregular microstructure around it. Parts 2 and 3 are the stable stage of microcrack propagation. Part 2 propagates with the direction roughly perpendicular to the loading direction, while part 3 propagates mainly in the direction with an angle of 45° to the loading direction. The effect of microstructure on crack propagation is discussed below using the crack path in part 2.
Fig. 17 – Microcracks initiation and morphology of the microcracks in WQ900 at 1260 N.

Fig. 18 – Microcracks propagation and morphology of the microcracks in WQ900, (a), (b), (c), (d) microcracks generated at 1250 N, 1260 N, 1270 N and 1280 N.

Fig. 19(a) shows the microcrack in part 2 with rule section in red lines, which is roughly perpendicular to the loading direction with less shear stress. The microstructure in the same area before microcrack formed is shown in Fig. 19(b) with red lines drawn for the microcrack path to be formed. There is large deformation occurred on the left side of the microcrack as shown in Fig. 19(c) in the original microstructure of the same position. The regular path of the microcrack is marked with red lines in Fig. 19(d), while the orientations of martensite packets are signed with red arrows. There are two typical sections on the microcrack in Fig. 19(d), coded as A and B. For section A, the microcrack propagate along the grain boundaries. In section B, the misorientation between two packets on both sides of the microcrack is greater than 45° (55°). It can be concluded that the grain boundaries are likely to be the path of the microcracks propagation. The large misorientation of martensite packets leads to dislocation pile-up, and is likely to be the path of microcrack propagation.

4. Conclusions

The present study investigated the microstructure quenched around $A_{C3}$ point and its effect on the damage behavior. The main conclusions are summarized as follows:

1. The microstructure of martensite quenched around $A_{C3}$ point and their effects on macroscopic mechanical properties are different. The microstructure of martensite quenched below $A2$ is martensite in island with ferrite.
around it. A variation in the martensite fraction leads to large variation in strength and ductility of the dual phase steels. A complete austenization above \( A_{c3} \) and rapid cooling leads to lath martensite. The austenization temperature does not change the microstructure of martensite much, neither the mechanical properties.

2 Observation of microcrack initiation and propagation under in-situ tension indicates that the damage mechanism for dual phase and full martensite is different. For dual phase steel microcracks usually occur in ferrite at sections near the boundary with martensite or interface between two martensites with different misorientation, which is the weakness in microstructure. For full martensite with lath structure, microcracks formed often at sections with large misorientation between two packets on both sides of the microcrack. The large misorientation, which is likely to be prior austenite grain boundaries or packet boundaries, leads to the weakness and are preferred location for microcrack initiation and propagation.

**Conflicts of interest**

No conflict of interest exits in the submission of this manuscript, and manuscript is approved by all authors for publication. I would like to declare on behalf of my co-authors that the work described was original research that has not been published previously, and not under consideration for publication elsewhere, in whole or in part. All the authors listed have approved the manuscript that is enclosed.

**REFERENCES**


