In-Situ High Temperature X-ray Studies of Austempering Transformation in High Silicon Cast Steel

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The in-situ X-ray diffraction observations of the bainitic transformation of high silicon cast steel were performed using the high temperature X-ray diffraction technique. The volume fraction and carbon content of austenite depend on the transformation temperature. The experimental result has shown that the volume fraction of austenite ceases to a constant value which indicate that the transformation is almost finished after holding for about 1 000 s. Asymmetry diffraction peaks are obtained for samples at the early stage of transformation due to a heterogeneous distribution of carbon in different regions of austenite and thus exists two types of austenite: low-carbon austenite (γ_{LC}) and the high-carbon austenite (γ_{HC}). The volume fraction of bulk austenite with low carbon decreases greatly at the early stage of transformation and then tends towards zero. The lattice parameter of both low-carbon and high-carbon austenite increases with the holding time due to the carbon partition from the supersaturated ferrite to the austenite. The experimental results supports that the bainite growth is by a diffusionless mechanism when austempering temperature is in the lower bainite transformation temperature range.

KEY WORDS: high silicon cast steel; bainitic ferrite; austempering; in-situ X-ray diffraction.

1. Introduction

During the last two decades, many research efforts have been devoted to the study of high silicon steel.1–12) This kind of steel with chemical composition and fine duplex microstructure of bainitic ferrite and retained austenite (ausferrite) that similar to the matrix of ADI is a material of high interest, which demonstrates a combination of high mechanical properties such as strength, ductility, fatigue strength, fracture toughness, impact toughness and wear resistance. High silicon steels have great potential to be a low cost and high reliability structural material.4,9)

High-silicon low-alloy cast steel is used in the applications of bucket teeth for excavator and loader, lining for ball mill, hammer and rake chains screening machine for railroad successfully in China, but the role of the carbon during the bainitic ferrite transformation is still needed to study. The present study is conducted to investigate the phase transformation from austenite to bainitic ferrite of high silicon cast steel using high-temperature X-ray equipment, to study the volume fractions and carbon contents in austenite at different austempering temperatures during the course of transformation, and thus to give some advices to making the heat treatment process.

2. Experimental Procedures

The chemical composition of the tested steel is presented in Table 1.

The steel was produced in a 500 kg medium-frequency

Table 1. Chemical composition of the tested steel.

| Element | Fe  | C   | Si  | Mn  | Mo  | P   | S   | Cr  |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|
| Balance| 0.83| 2.15| 0.47| 0.36| 0.027| 0.027| 0.051|     |
coreless induction furnace with siliceous lining, with charge materials of silicon steel scrap, graphite, Fe–Si, Fe–Mn and Fe–Mo master alloys. The melt was superheated to 1600°C. After holding at the temperature for 5 min, the melt was cast into Y blocks by investment casting.

The samples were cut from the bottom of the Y blocks. After grinding the samples were finally polished using 0.25 μm diamond paste. A Philips X’pert-MPD X-ray diffractometer with a high-temperature diffractometer chamber HDK 2.4 was used to determine the volume fraction of austenite and carbon content in austenite during the bainitic transformation. The samples were positioned on a Mo sample holder in the furnace chamber, and were austenitized by resistance Mo-heating element at 900°C for 3 min. In order to avoid oxidation, the furnace was evacuated down to a pressure of 0.3 Pa before heating. The samples were cooled down to the austempering temperatures of 260 to 320°C for 3 h by using nitrogen. The in-situ XRD observations were done using a monochromatic Cu-Kα radiation at 40 kV and 45 mA. A PW3011/00 proportional rotating head anode diffractometer was used to scan the angular 2θ range of 41.5–45.5°, scanning time of every peak was 3 min 28 s (step 0.1°, time per step 5 s). The systematic errors in the measurements of the positions of austenite and bainitic ferrite peaks were corrected by a pure single-crystal silicon plate.

The profiles were analyzed on a computer to obtain the exact peak positions and the integrated intensity of the {111} planes of austenite and the {110} planes of bainitic ferrite by using the curve-fitting technique with a Gaussian function. The volume fraction of austenite (Xv) was determined by the direct comparison method(4) using the integrated intensities of the {110} planes of ferrite and the {111} planes of austenite. Three samples were examined and the average from these samples is reported here. The following equation(5) was used to determine the carbon content of austenite:

$$ a_v = 0.3578 + 0.0033 m_C + 0.000095 m_{\text{Mn}} - 0.00002 m_{\text{Ni}} + 0.00006 m_{\text{Cr}} + 0.0022 m_{\text{Mo}} + 0.00056 m_{\text{Al}} + 0.00015 m_{\text{Cu}} + 0.00031 m_{\text{Si}} + 0.000039 m_{\text{Nb}} + 0.00018 m_{\text{V}} \ldots \ldots (1)$$

where $a_v$ is the lattice parameter of austenite in nm and $m_C$ is the carbon content of austenite in wt%. The label $m_i$ corresponds to the weight percent of elements “i” in the austenite. The lattice parameter of retained austenite was estimated using the {111} plane of austenite. Three samples were examined for each heat-treatment condition, and the carbon content of austenite reported here are average values from these samples.

The samples after the austempering transformation were polished using 0.25 μm diamond paste. The microstructures of the austempered samples were examined on a Jeol JSM 6460 scanning electron microscope (SEM) using an accelerating voltage of 15 kV after etching with 3% nital solution.

3. Results and Discussion

3.1. Carbon Enrichment in Austenite during in-Situ Evolution of Transformation

The phase transformation from austenite to bainitic ferrite was monitored with the in-situ X-ray diffraction technique and the diffraction result from the austenite plane {111} and bainitic ferrite plane {110} during transformation is shown in Fig. 1.

It can be seen from Fig. 1 that the initial peaks of austenite are narrow and the intensity is high shows that the austenite remains homogeneous prior to bainitic transformation. The intensity of austenite {111} peaks decreases rapidly and the intensity of bainitic ferrite {110} peaks increases greatly at the early stage of transformation, and after that both of the intensity of austenite peaks and bainitic ferrite peaks remains nearly the same as the transformation develops.

The volume fraction of austenite of in-situ X-ray analyses of the bainitic transformation varying with the austempering time and temperature is shown in Fig. 2. The volume fraction of austenite depends strongly on transformation temperature. The bainitic ferrite transformation develops rapidly at the early stages of austempering transformation (<500 s) and then decreases gradually. After that the rate of bainitic ferrite transformation decreases with the austempering time and the transformation is almost finished after about 1000 s when the volume fraction of austenite attains a constant value, which shows that the reaction remains thermodynamically incomplete.

![Fig. 1. Austenite {111} and bainitic ferrite {110} peaks during austempering transformation at 320°C.](image)

![Fig. 2. Volume fraction of austenite during austempering transformation at various austempering temperatures.](image)
Austempering temperature and holding time also influence the lattice parameter of untransformed austenite and thus influence the carbon content of austenite. The carbon concentration in austenite of the bainitic transformation varying with the austempering time and temperature is shown in Fig. 3.

The carbon contents of austenite were calculated by using the lattice parameter of austenite $a_g$. In addition, to calculate the lattice parameters at austempering temperature the thermal expansion coefficient of austenite must be measured. The X-ray experiments were performed over the temperature range 820 to 1000°C and holding time for 5 min. These thermal expansion coefficient of austenite was measured to be $2.51 \times 10^{-5} \text{K}^{-1}$. The carbon content of austenite increases greatly with austempering time at the early stage of transformation and then attains a constant value at all transformation temperatures.

Figure 4 shows examples of curve fitting the X-ray diffraction line profiles of the austenite during the isothermal transformation, by using the Gaussian function. For the specimen austempered at 260°C for 240 s, asymmetrical diffraction peak is obtained. The full widths at half maximum (FWHMs) of the peaks in Fig. 4(a) are 0.15 and 0.35 degree, and the standard deviations are about 0.06 and 0.14 degree respectively. Thus the step in the measurements, 0.1 degree, is enough numbers to profile the peaks. This asymmetry of the diffraction peaks after the formation of bainitic ferrite cannot be explained with instrumental errors. It is attributed to a heterogeneous distribution of carbon in different regions of austenite, because it takes time for carbon to become homogenised after it is partitioned from supersaturated bainitic ferrite. The result shows exactly that two types of austenite with different carbon contents exist during the transformation: low-carbon austenite ($\gamma_{LC}$) which corresponds to the initial austenite and the high-carbon austenite ($\gamma_{HC}$) between sub-units and laths of bainitic ferrite. For the specimen held for 4020 s, the model function with two peaks fits adequately to the whole line profile.

Figure 5 shows the volume fraction and lattice parameter of low-carbon and high-carbon austenite during the isother-
mal transformation at 260°C. The volume fraction of bulk austenite with low carbon decreases greatly at the early stage of transformation (Fig. 5(a)), which indicates that the low-carbon austenite transforms first, and then tends towards zero. At the early stage of transformation the amount of low-carbon austenite isothermally transformed to bainitic ferrite increases, leading to the enrichment and stabilization of the retained austenite due to the rejection of carbon by the bainitic ferrite. Hence, much more high-carbon retained austenite can be formed in the vicinity of the bainitic ferrite laths. At longer times (>500 s) the precipitation and growth of individual plates bainitic ferrite continues on the parent austenite grain boundaries, resulting in the increase of the volume fraction of the ferrite and in gradual disappearance of the low-carbon austenite with increasing holding time. When the volume fraction of untransformed austenite keeps constant, that of the untransformed low-carbon austenite decreases to zero. The lattice parameter of both low-carbon and high-carbon austenite increases (Fig. 5(b)) with the holding time due to the carbon partition from the supersaturated ferrite to the austenite.

Figure 6 shows the microstructure of samples cooled to room temperature after austempering for 3 h at various holding temperatures. The microstructures of matrix consist of colonies of finer bainitic ferrite (dark etched) and carbon-enriched retained austenite. No carbides were precipitated in the structure because the silicon has delayed the austempering transformation of austenite. The austempered microstructure shows also that there is no significant decarburization on the surface of the samples. X-ray diffraction analysis shows the presence of a very thin layer of magnetite (Fe₂O₃) on the surface of the samples and has no obvious influence on the analysis of volume fraction and carbon content of austenite (Fig. 7).

3.2. Lattice Parameter of Bainitic Ferrite during in-Situ Evolution of Transformation

Figure 8 shows the variation of the lattice parameter of bainitic ferrite at the austempering temperatures based on XRD pattern peaks, which has been taking into account the influence of the thermal expansion coefficient of ferrite. The lattice parameter greatly decreases at the beginning of transformation of about 800 s, and then remains constant as the austempering holding time increases. It is due to the bainitic ferrite is supersaturated with carbon as the steel is cooled down from the austenitizing temperature, and then some carbon diffuses into austenite during the isothermal transformation, giving rise to a decrease in the lattice parameter of bainitic ferrite. It should be noted that the lattice parameter increases simultaneously with the beginning of decrease in the carbon-enriched untransformed austenite shown in Fig. 2.

3.3. Discussion on Bainitic Ferrite Transformation in High Silicon Cast Steel

Figures 2 and 3 have shown that the bainitic ferrite reaction remains thermodynamically incomplete. It is known that the transformation temperature that distinguishes upper and lower bainite transformation is approximately 350°C. Combining with other analysis results that the austenite re-
mains homogeneous prior to bainitic transformation, the lattice parameter of bainitic ferrite decreases greatly at the early stage of transformation (Fig. 8) and the volume fraction and carbon content of austenite remain constant after the early stage (Figs. 2 and 3), one can therefore support that the bainitic ferrite growth at the early stage of transformation is by the “displacive” mechanism, with carbon partitioning occurring subsequent to transformation when austempered at the lower bainite transformation temperature range.

4. Conclusions

The in-situ X-ray diffraction observations of the bainitic transformation of high silicon cast steel were performed using the high temperature X-ray diffraction technique. The volume fraction and carbon content of austenite depends on the transformation temperature. The volume fraction of austenite ceases to a constant value which indicate that the transformation is almost finished after holding for about 1000 s. The lattice parameter of bainitic ferrite decreases with increasing austempering temperature, which is related to the decrease of carbon concentration in bainitic ferrite.

Asymmetry diffraction peaks are obtained for samples at the early stage of transformation due to a heterogeneous distribution of carbon in different regions of austenite. The result shows that two types of austenite with different carbon contents exist during the transformation: low-carbon austenite ($\gamma_{LC}$) which corresponds to the initial austenite and the high-carbon austenite ($\gamma_{HC}$) between sub-units and laths of bainitic ferrite. The volume fraction of bulk austenite with low carbon decreases greatly at the early stage of transformation and then tends towards zero. It indicates that the low-carbon austenite transforms first which leads to the formation of the high-carbon untransformed austenite in the vicinity of the bainitic ferrite laths due to the rejection of carbon by the bainitic ferrite. The lattice parameter of both low-carbon and high-carbon austenite increases with the holding time due to the carbon partition from the supersaturated ferrite to the austenite.

The experimental results supports that the bainite growth is by a diffusionless mechanism at the early stage of transformation, with carbon partitioning occurring subsequent to transformation, when austempering temperature is in the lower bainite transformation temperature range.

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