MICROSTRUCTURAL EVOLUTION OF A LOW-CARBON STEEL DURING THE AUSTENITE DECOMPOSITION BELOW M_S

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Abstract

The microstructural evolution of a low-carbon steel has been studied during the austenite decomposition, subsequent to initial martensite formation. Isothermal holding treatments after rapid cooling to various temperatures between martensite start (M_s) and martensite finish (M_f) temperatures were carried out in a dilatometer with the intention to form controlled volume fractions of initial martensite and austenite. The purpose of the above heat treatments was to create conditions in which the phase transformations during isothermal treatment between the M_s and M_f temperatures can take place. Different microstructures consisting of bainite, martensite, retained austenite and carbides were obtained depending on the isothermal holding temperature and time. The transformation kinetics was monitored by means of dilatometry and the microstructure was characterized by Scanning Electron Microscope (SEM), Electron Backscatter Diffraction (EBSD) and Transmission Electron Microscope (TEM). Hardness measurements of the resulting microstructures were analysed. The results revealed that the microstructures formed below M_s are mainly formed by different fractions of tempered martensite and isothermal bainite with carbide precipitation.

Keywords: Austenite decomposition, phase transformations, isothermal transformation below M_s, low-carbon steel

1. INTRODUCTION

The athermal martensitic transformation, which occurs during quenching, has been widely investigated and well-documented [1,2]. However, the isothermal transformations below M_s and the resulting microstructures are not completely understood. Issues like isothermal formation of martensite and/or bainite in this temperature range are still under discussion, especially in low-carbon steels [3-5]. A recent study [4] investigated various aspects of decomposition of austenite in a low-carbon steel during one- and two-step of quenching and partitioning (Q&P) processing by dilatometry. Dilatation curves showed that besides carbon partitioning, isothermal martensite transforms during only a short partitioning step. On the other hand, given more time during partitioning, austenite transforms to lower bainite. In steel production, such cases can be observed if the steel is coiled at temperatures below M_s, but above the M_f temperature where the austenite is not completely transformed. The understanding of the variation in resulting microstructures is of fundamental importance since it determines the mechanical properties of the final product. As such, the present study intends to clarify the microstructural evolution of a low-carbon steel during isothermal decomposition of austenite at temperatures below M_s. To reach this purpose, different microstructures were produced by controlled heat treatment in a dilatometer and subsequently analyzed by SEM, EBSD and TEM, whereas the mechanical properties were characterized by hardness measurements.

2. EXPERIMENTAL PROCEDURE

The chemical composition of the low-carbon steel studied is 0.16C-1.6Mn-0.8Cr-0.4Si-0.3Mo (wt. %). The heat treatments were carried out in a Bähr DIL 805A/D dilatometer, with flat samples of size 10×5×1 mm^3. Different sets of experiments were performed (cf. **Fig. 1**), all starting from full austenitization at 900 °C for 30 seconds and continued as follows: (*i*) Direct quenching to room temperature (RT) to investigate the martensite transformation: The dilatometry curve obtained indicated that $M_{s\,(5\%)}$ = 420 °C and $M_{f\,(95\%)}$ = 260 °C; (*ii*) Fast cooling to bainite temperature range (T_{iso} = 450 °C), isothermal holding at this temperature for 1 hour and subsequent quenching to RT to evaluate the bainite formation; (*iii*) Fast cooling to temperatures below M_s and M_f (T_{iso} = 350 °C and 100 °C, respectively), isothermal holding at corresponding temperatures for 1 hour and subsequent quenching to RT to evaluate the isothermal transformation below M_s when a certain fraction of initial martensite (IM) is present. Samples were polished and etched with 4 % Nital reagent to reveal different microstructural components and subsequently analyzed by SEM. Samples were prepared for EBSD analysis by electro-polishing with Struers A2 electrolyte after final polishing step with 1 μm diamond paste. Foils were prepared for TEM by first thinning to 80 μm by mechanical polishing. Discs of 3 mm diameter were cut from the thinned sheet and subsequently, electropolished in a twin-jet method using a solution of 5 vol. % perchloric acid and acetic acid at room temperature. Vickers hardness was measured with a load of 3 kgf (HV_3).

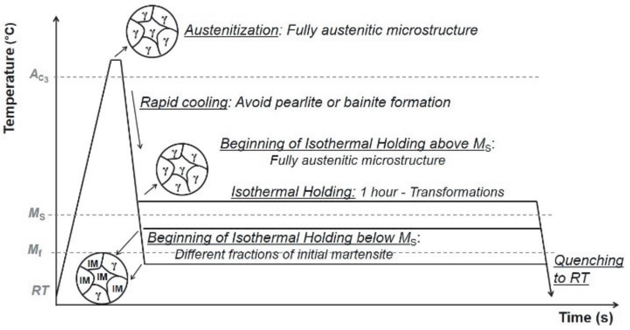

Fig. 1 Schematic illustration of the heat treatment performed in dilatometer. γ = austenite, IM = initial martensite

3. RESULTS AND DISCUSSIONS

3.1 Dilatometry

The dilatometry curves corresponding to the cooling, isothermal holding in the temperature range of 450-100 °C and quenching to RT are plotted as a function of temperature and time in **Fig. 2(a)-(b)**. The curve corresponding to T_{iso} = 450 °C shows the decomposition of austenite to bainite, as expected during the isothermal holding. The transformation of bainite is manifested in the dilatometry curve by an increase of the sample length, shown in **Fig. 2(a)-(b)**. Interrupting the bainitic transformation after 1 hour (by quenching to RT) did not lead to a pronounced transformation observed in dilatometry curve (cf. **Fig. 2(a)**).The curves corresponding to T_{iso} = 350 °C and T_{iso} = 100 °C (below M_S and M_f, respectively) show, apart from the isothermal transformations, the typical martensitic transformation during cooling before reaching the corresponding isothermal temperatures, as shown in **Fig. 2(a)**. The volume fractions of initial martensite formed at those isothermal temperatures are approximately 0.73 ± 0.03 and 0.98 ± 0.01, respectively. An initial length increase occurs during the subsequent isothermal holding at T_{iso} = 350 °C, where the transformation progresses rapidly in the first minute of isothermal holding (cf. **Fig. 2(b)**). The bainitic transformation is assumed to be the main contributor to this length increase. Initial martensite is also tempered during the isothermal hold (which can be confirmed by the microstructure) but, this tempering is considered to have a negligible effect on the change in length, according to a previous study in this steel [6]. The curve corresponding to the lowest temperature (T_{iso}

= 100 °C), shows a very small transformation effect during the isothermal holding, after the martensite formation (cf. **Fig. 2(a)-(b)**).

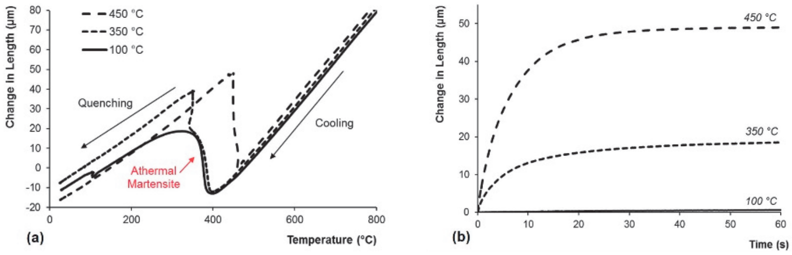

Fig. 2 Dilatometry curves of the 1 h isothermal holding experiments in the range of T_{iso} = 450 °C to 100 °C: (a) as a function of temperature, (b) as a function of time

3.2 Scanning Electron Microscopy

The formation of isothermal bainite at T_{iso} = 450 °C is accompanied by carbide precipitation, (cf. **Fig. 3(a)**). Besides bainite, another transformation product has been observed, which is highlighted within the dashed line in **Fig. 3(a)**. This transformation product was analyzed in further detail by EBSD (**Fig. 4**) and it was identified as fresh martensite (FM). When the isothermal holding is interrupted, a certain volume fraction of untransformed austenite remains. Most of this untransformed austenite transforms to FM during quenching to RT. Isothermal holding at T_{iso} = 350 °C (**Fig. 3(b)**) gives rise to a microstructure with a relatively large fraction of fine carbides. This can, in part, be explained by the tempering of IM and part by the formation of isothermal bainite. Isothermal holding at T_{iso} = 100 °C results in lath martensite **Fig. 3(c)** which bears striking similarities to the one obtained from direct quenching to RT (cf. **Fig. 3(d)**). Carbides are distributed within the quenched martensite. This carbide precipitation is related to the auto-tempering process due to the relatively high M_s temperature.

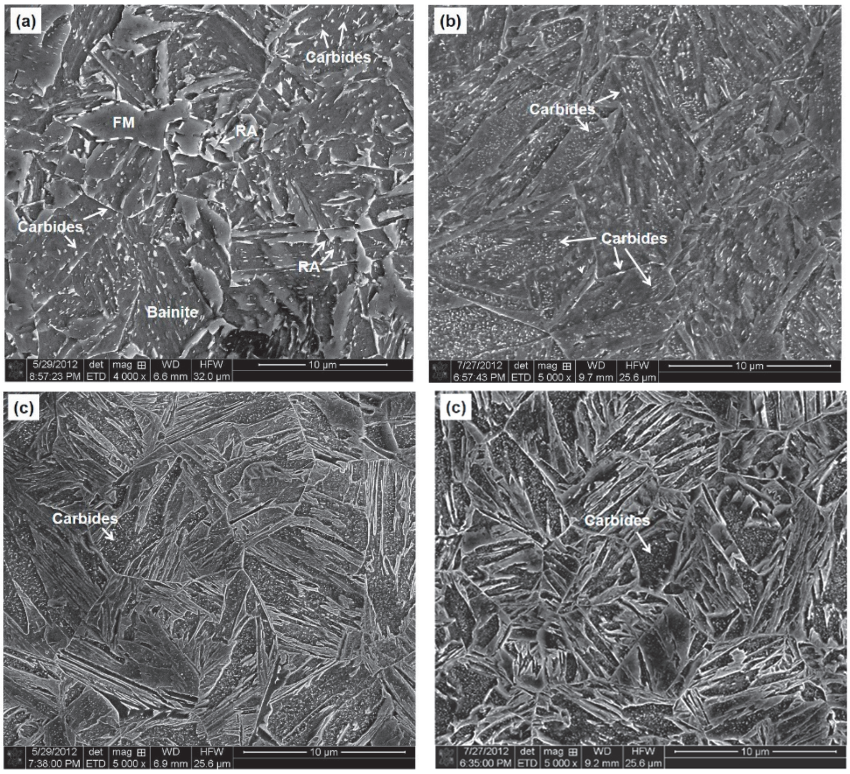

Fig. 3 SEM micrographs of samples at: (a) T_{iso} = 450 °C, (b) T_{iso} = 350 °C, (c) T_{iso} = 100 °C and (d) quenched to RT

3.3 Electron Backscattered Diffraction

In order to confirm some of the microstructural features mentioned, EBSD map scans are shown in **Fig. 4(a)-(c)** and **Fig. 5(a)-(d)**. As observed earlier, bainite is incompletely transformed during the isothermal holding at T_{iso} = 450 °C, and FM and RA were found in the final microstructure. FM is considered to be untempered martensite, with a blocky phase structure and smooth surface [7]. This type of martensite is more difficult to etch than other carbide-containing phases. An Image Quality (IQ) map of the area containing FM and RA, the same region at higher magnification and the corresponding phase map, are shown in **Fig. 4(a)-(c)**, respectively. In the phase map (**Fig. 4(d)**), the retained austenite (FCC) is highlighted in green and the different BCC transformation products (martensite and bainitic ferrite) are shown in red. Volume fraction calculations of FM (plus RA), based on the IQ map and SEM micrographs, yielded a value of 0.15 ± 0.03. The resulting microstructures corresponding to T_{iso} = 350°C and 100 °C do not show evidences for formation of FM or RA, as observed in the phase maps (**Fig. 5(b)-(d)**), which is an indication that the transformations had been completed during the isothermal holding.

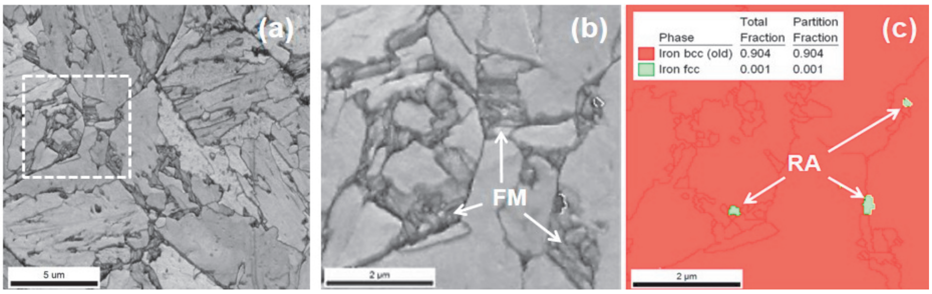

Fig. 4 EBSD maps showing presence of retained austenite and FM for T_{iso} = 450 °C - 1 h: (a) Gray scale IQ map, (b) Higher magnification of selected region in (a), (c) Phase map with grain boundaries with misorientations between 15-180°

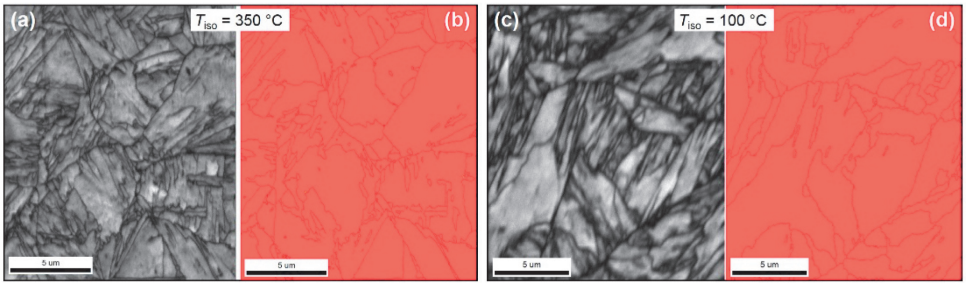

Fig. 5 EBSD IQ and Phase maps: (a)-(b) T_{iso} = 350 °C, (c)-(d) T_{iso} = 100 °C

Combining the results from EBSD scans and the dilatation changes during cooling and isothermal holding, the volume fractions of transformation products were estimated and listed in **Table 1**.

Table 1 Volume fractions of transformation products formed during the heat treatment.

T_{iso} (°C)	*IM*	*Bainite*	*FM + RA*
100	0.98 ± 0.01	0.02 ± 0.01	-
350	0.70 ± 0.04	0.30 ± 0.04	-
450	-	0.85 ± 0.03	0.15 ± 0.03

3.4 Transmission Electron Microscopy

Bright field (BF) TEM micrographs are shown in **Fig. 6(a)-(d)**. They clearly reveal the morphology of rode-like carbides in all samples [8]. In the isothermal bainite formed at T_{iso} = 450 °C, the carbides are aligned within the ferrite platelets (cf. **Fig. 6(a)**). The same kind of carbides was found at T_{iso} = 350 °C, confirming the hypothesis for isothermal bainite formation, as shown in **Fig. 6(b)**. Additionally and as expected, carbide precipitations with multiple morphological variants originated from the tempered martensite [8] were also observed at this isothermal holding temperature (cf. **Fig. 6(c)**). The sample at T_{iso} = 100 °C shows only the multi-variant carbide precipitation from tempered martensite. The selected area diffraction (SAD) pattern of the carbides and the possible solutions for corresponding indexation are illustrated on the upper right corner of TEM micrographs. The SAD patterns exhibit diffraction spots from orthorhombic cementite (Fe_3C). According to the steel composition, which has low silicon content, Fe_3C formation is favoured over that of other carbides. Additionally, it is assumed that the isothermal temperatures are too low for significant Cr- of Mo-diffusion.

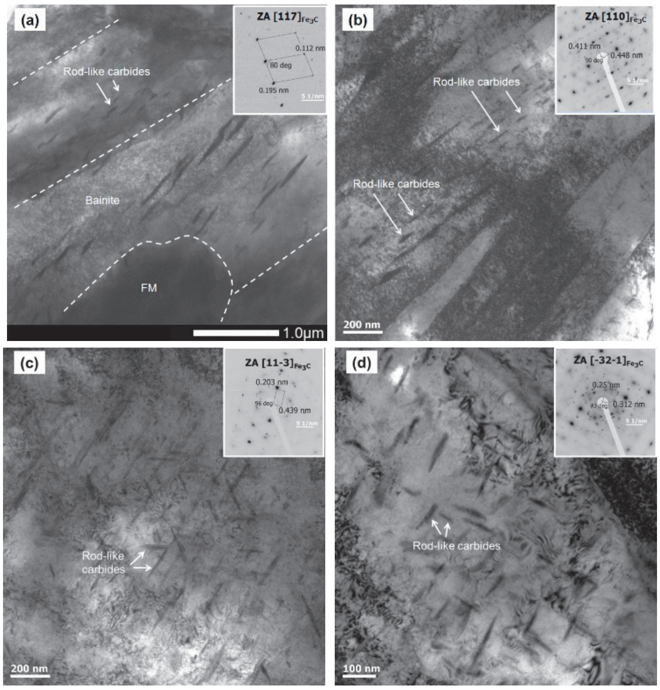

Fig. 6 TEM BF micrographs showing the carbide precipitation (a) T_{iso} = 450 °C, (b)-(c) T_{iso} = 350 °C and (d) T_{iso} = 100 °C

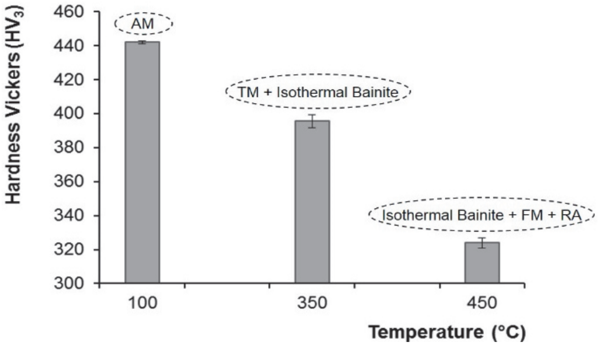

Fig. 7 Hardness after the entire heat treatment for the isothermal temperatures. (AM = auto-tempered martensite, TM = tempered martensite, FM = fresh martensite, RA = retained austenite)

3.5 Hardness Vickers (HV)

Hardness values vary with the microstructure of the material and decrease with increasing of the isothermal holding temperatures. This progressive softening is primarily caused by: *(i)* a decrease in the fractions of initial martensite and *(ii)* an increase of the isothermal transformation products (cf. **Fig. 5**).

CONCLUSIONS

The microstructural evolution of a low-carbon steel during the austenite decomposition has been studied and the main results are summarized as follows:

(i) Incomplete bainite transformation takes place in isothermal conditions at temperatures above M_s.

(ii) Resulting microstructure from isothermal temperature below M_s consists mainly of bainite and tempered martensite.

(iii) Cementite precipitation was formed at all studied temperatures.

(iv) Microstructure consisting of high fraction of initial auto-tempered martensite is harder than the microstructure consisting of mixtures of transformation products formed during isothermal holding.

ACKNOWLEDGEMENTS

The authors would like to thank ArcelorMittal Global R&D Gent for the financial and experimental support to the research project.

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