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Characterization of bainitic/martensitic structures formed in isothermal treatments below the M_s temperature



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ABSTRACT

Advanced Multiphase High Strength Steels are generally obtained by applying isothermal treatments around the martensite start temperature (M_s). Previous investigations have shown that bainitic ferrite can form from austenite in isothermal treatments below M_s , where its formation kinetics is accelerated by the presence of the athermal martensite. That athermal martensite is tempered during the isothermal treatment, and fresh martensite may form during the final cooling to room temperature. The distinction between product phases present after the application of this type of heat treatments is difficult due to morphological similarities between these transformation products. The aim of this study is to characterize the structural and morphological features of the product phases obtained in isothermal treatments below the M_s -temperature in a low-carbon high-silicon steel. Multiphase microstructures, having controlled fractions of product phases, were developed by applying isothermal treatments above and below M_{s} , and were further studied by electron back scatter diffraction (EBSD) and scanning electron microscopy (SEM). The bainitic or martensitic nature of these product phases is discussed based on this characterization. Results showed that bainitic ferrite appears in the form of acicular units and irregularly shaped laths. Tempered martensite appears as laths with a sharp tip and as relatively large elongated laths with wavy boundaries containing protrusions.

1. Introduction

The development of new Advanced High Strength Steels (AHSS) requires the implementation of improved processing routes in which the resulting microstructures present a high degree of complexity. That is the case of multiphase steels obtained by applying isothermal holdings around the martensite start temperature (M_s) after austenitization, leading to microstructures generally formed by a mixture of martensite (fresh and/or tempered), bainite, and retained austenite. The presence of different product phases from austenite decomposition depends on whether the isothermal holding is applied at a temperature above M_s or below M_s .

After holding at a temperature above M_s and further quench, researchers agree that, in hypoeutectoid steels, the matrix is mainly formed by bainitic structures with retained austenite in the form of thin films and/or martensite-austenite (MA) islands [1–7]. Bainite is thus the isothermal product obtained from the decomposition of austenite. Carbides can also be present in bainite, depending on the composition of the steel and the isothermal holding temperature and time. Below M_s , however, the nature of the products formed during isothermal holding

is unclear due to the coexistence of product phases with morphological similarities such as tempered martensite and bainite.

Cooling from austenitization to a temperature between the martensite start (M_s) temperature and room temperature (RT) leads to the formation of a certain volume fraction of athermal martensite. This martensite has a strong accelerating effect on the subsequent transformation kinetics, mainly due to a higher density of potential nucleation sites [8–11]. Although bainite has been reported in hypoeutectoid steels as an isothermal decomposition product from austenite in isothermal treatments below M_s [4,10–14], there is experimental evidence showing the formation of other types of product phases in similar heat treatments. For example, several authors observed an isothermal product characterized by wide laths with characteristic wavy boundaries with ledges [16,17]. Kim et al. [7,15,16] stated that this isothermal product is neither purely martensitic nor purely bainitic, since it shows similarities with both types of product phases. Somani et al. [17] identified this product as isothermal martensite since it presents clear similarities to athermal martensite. According to the literature, isothermal martensite has mainly been reported in highcarbon steels and high-nickel alloys [18-20], but not in hypoeutectoid

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steels. Independently of the growth mechanism of these product phases, the dilemma is which microstructural features observed in isothermal treatments below M_s correspond to each phase formed in those treatments, such as bainitic ferrite, tempered martensite or fresh martensite.

The goals of this study are: to characterize the microstructural features obtained in isothermal treatments performed at temperatures above and below M_s ; relate those features to the product phases formed at each isothermal temperature; and determine, based on that characterization, the martensitic or bainitic nature of those product phases. For this purpose, isothermal heat treatments were applied at different temperatures around the $M_{\rm s}$ -temperature that were selected based on a previous study [11]. Various microstructures formed by combinations of different volume fractions of bainitic ferrite, tempered martensite, and fresh martensite were created. Volume fractions of those product phases were extracted from the dilatometry results presented in earlier work [11]. The identification of phases by dilatometry in combination with the characterization of microstructures formed at different temperatures by electron backscatter diffraction (EBSD) and scanning electron microscopy (SEM) provided a straight and systematic way of phase analysis.

2. Experimental procedure

The chemical composition of the investigated steel is 0.2C-3.51Mn-1.52Si-0.25Mo-0.04Al (wt. pct). The as-received material was hot rolled into a 4 mm-thick steel slab. Dilatometry specimens were extracted from hot rolled slabs, parallel to the rolling direction. These were cylindrical of dimensions 10 mm in length and 3.5 mm in diameter. Heat treatments were carried out in a Bähr 805A dilatometer. Specimens were placed between two quartz rods, heated by an induction coil, and cooled using nitrogen gas. A thermocouple was spot-welded in the middle of the specimens to control the temperature.

Different heat treatments were applied above and below the M_{s} -temperature, after a full austenitization at 900 °C for 240 s. The applied heat treatments are described as follows (Fig. 1):

- (i) A direct-quench treatment was performed to determine the experimental M_s -temperature and the kinetics of the martensite formation. The analysis of the dilatometric curve, presented elsewhere [11], determined an experimental M_s (1%) = 320 °C ± 5 °C, at which temperature the volume fraction of martensite formed is 1%.
- (ii) An isothermal treatment above the M_s -temperature was carried out at 340 °C to obtain a microstructure formed by a mixture of bainite, retained austenite and fresh martensite.
- (iii) Three isothermal treatments between M_s and room temperature (RT) were performed at 320 °C, 300 °C and 270 °C to obtain a



Fig. 1. Schematic representation of the heat treatments applied in the dilatometer to the selected steel.

multiphase microstructure formed by martensite obtained during cooling from austenitization to the isothermal temperature, bainitic ferrite, retained austenite, and fresh martensite obtained in the final cooling to room temperature.

The volume fractions of retained austenite at room temperature were determined by X-ray diffraction (XRD) experiments using a Bruker D8-Advance diffractometer equipped with a Bruker Vantec Position Sensitive Detector. CoK α radiation was used in the 2 θ scan from 40° to 130° with a step size of 0.035°. The fractions of austenite and ferrite were calculated by the integrated area method using the (111), (200), (220), and (311) austenite peaks, and the (110), (200), (211), and (220) ferrite peaks [21]. The heat-treated specimens were metallographically prepared by grinding and polishing. 2% Nital etching was applied to the specimens to reveal the microstructure. A pre-analysis of microstructures by Light Optical Microscopy (LOM) and Scanning Electron Microscopy (SEM) was done and presented elsewhere [11].

Electron Back Scatter Diffraction (EBSD) in combination with SEM was used to perform a detailed characterization of the transformation products obtained in the heat treatments previously described. In order to analyse the same area by EBSD and SEM, various micro-indentations (HV1) were made in the specimens to select representative areas. As a final preparation step, the specimens were mechanically polished with a colloidal silica solution in order to exhaustively clean the specimens and remove surface deformation. Areas selected in the specimens were first analysed by EBSD.

The EBSD patterns were acquired on a FEI Quanta 450 scanning electron microscope equipped with a Field Emission Gun (FEG-SEM) by means of the OIM Data Collection software. The analysis was performed under the following conditions: acceleration voltage of 20 kV, spot size 5, working distance of 16 mm, tilt angle of 70°, and step size of 50 nm in a hexagonal scan grid. The orientation data were post-processed with TSL OIM® Analyses 6.0 software. First, a grain confidence index (CI) standarization was applied to the raw data, selecting a tolerance angle of 5° and a minimum grain size of 6 pixels, and considering that grains are formed by multiple pixel rows. The second step of the postprocessing procedure included neighbour-orientation correlation with a tolerance angle of 5° and a minimum confidence index of 0.1. Finally, a down-filtering criterion of confidence index higher than 0.1 was applied to reduce the number of pixels poorly indexed, i.e., not attributed to either fcc or bcc phases.

After a comprehensive EBSD analysis, specimens were carefully polished with a colloidal silica solution for 5 to 10 min in order to remove the carbon deposition layer of the specimen surface. Afterwards, 2% Nital etching was applied to the specimens to reveal the different phases formed during the heat treatments. The EBSDanalysed areas were then studied with a JEOL JSM-6500F Scanning Electron Microscope (SEM) using a 15 kV electron beam and the Secondary Electron Imaging (SEI) detection mode.

3. Results and discussion

3.1. Volume fraction of phases

There was no evidence of the formation of ferrite, pearlite, or bainite during the initial cooling to the isothermal temperatures above and below M_s [11]. Only when the temperature decreased below the M_s temperature, a dilatation was observed due to the formation of a certain fraction of athermal martensite prior to the isothermal holding. Afterwards, dilatometric curves showed a significant dilatation during the isothermal holding, which implies the formation of an isothermal product phase identified as bainitic ferrite in ref. [11]. There was also a small deviation from linearity of the dilatometric curves during the final cooling to room temperature, showing the formation of fresh martensite during that cooling. The determination of the volume fractions of product phases obtained in the applied heat treatments is Download English Version:

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