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Abstract

Chemical segregation of alloying elements during solidification of steel grades leads to development of a banded microstructure, causing a degree of anisotropy that can be detrimental to the mechanical behavior under service conditions. It is well-known that the presence of strongly orientated martensite bands in carbide-free bainitic microstructures, associated to inhomogeneous Mn redistribution during solidification, leads to a remarkable deterioration in toughness in advanced high strength bainitic steels. In this study, while bands were clearly visible on light optical micrographs of continuously cooled carbide-free bainitic steels, scanning electron microscopy examination revealed only a gradual transition between matrix and bands, both with a granular bainitic structure. Electron backscatter diffraction was used to quantify the bainitic packet size and volume fraction of martensite/austenite constituent between and within the bands, after a process of optimization of the analysis settings in order to minimize the inherent difficulties linked to sub-micrometric and minority phase indexation. The quantitative microstructural results showed negligible morphological differences between bainitic structure bands and matrix, only solute segregation of Cr and Mo was detected by energy-dispersive X-ray spectroscopy within bands, which must be responsible for a stronger resistance against metallographic etching in those regions.

Introduction

Reducing the weight of car components has led to more filigree parts and the use of stronger materials. Conventional solutions with quench and tempered steels not always fulfil the wishes concerning technical reliability, economics and environmental friendliness. Continuously cooled TRIP-assisted bainitic steels are sometimes clearly better balancing these aspects which has led to a successful introduction into car components during the last decade. Avoiding the use of expensive alloying elements like Ni and Mo, steelmakers have focussed on Mn and Cr alloying concepts.

However, low carbon steels with elevated amounts of Mn and Cr often present chemical segregation during solidification. Substitutional alloying elements such as Mn, Cr and Si are expelled from the first δ -ferrite, leading to some regions enriched and others depleted in those elements. This solute segregation may result, after a deformation process such as hot rolling, in the formation of a microstructural banding; that is, alternate bands of different phases or microconstituents. This is the case of the well-known ferrite-pearlite or the bainite-martensite structures, where the Mn has been proved to be the alloying element playing the major role in microstructural banding [1,2,3].

The appearance of microstructural banding is not a desirable phenomenon, since it introduces a degree of uncontrolled anisotropy in the material, responsible of dispersion in its mechanical response [4,5]. The commercial air-cooled bainitic steel grade of this study was produced with elevated amounts of Mn and Cr to increase the hardenability of the steel and with Si to retard carbide precipitation during bainite formation. This particular steel grade is also alloyed with sulfur to guarantee an optimum machining behavior. Band formation therefore is expected to occur after conventionally hot rolling. In case of martensitic bands a decrease in toughness, due to premature crack nucleation linked to the heterogeneous hardness distributions should be suspected [6]. The microstructure of the steel under investigation consists of ~70% granular bainitic ferrite, ~15% retained austenite and ~15% martensite. In metallographic investigations a complex microstructural banding was observed, which did not consist of martensitic bands embedded in a bainitic matrix, but in continuous granular bainitic bands only revealed for having a different resistance to chemical etching. Microstructure has been examined using Energy-Dispersive X-ray Spectroscopy (EDS) and Electron Backscatter Diffraction (EBSD) in order to investigate possible chemical and morphological differences within and between the bands.

Material and experimental procedure

An air-cooled bainitic steel grade [7] with following chemical composition: 0.23C-0.97Si-1.55Mn-0.15S-1.55Cr-0.14Mo (wt. %) was investigated. The material was conventionally hot rolled to bars with a diameter of 40 mm, followed by air cooling, at an approximate cooling rate of 2 °C/s. The resulting microstructure of this air-cooled bainitic steel grade mainly consists of granular bainite morphology, which may be revealed by Light Optical Microscopy (LOM) and Scanning Electron Microscopy (SEM) as a semi-equiaxed ferrite structure and discrete islands of martensitic/austenitic grains (M/A constituent). The bainitic ferrite grains are actually divided into bainite subunits only visible through Transmission Electron Microscopy [8].

To study the microstructure, longitudinal samples were cut, ground and polished following standard procedures. A 2 % Nital etching solution was applied to make bainitic microstructures visible. Detailed examination was carried out using a JEOL JSM 6500F field emission gun scanning electron microscope (SEM-FEG) operating at 7 kV. Volume fraction of M/A constituent in matrix was measured by means of quantitative metallography, following the standard E 562-11 [9], using SEM micrographs.

EDS analysis with an Oxford Inca detector coupled to a FEG-SEM HITACHI S-4800 operating at 15 kV was performed to observe the chemical segregation in bands and matrix in a qualitative way, scanning in smart-map mode over a polished sample and obtaining the chemical profiles. After EDS scans, samples were etched to allow the localization of bands.

EBSD analysis, for the identification of phases and crystalline orientations, was performed over longitudinal samples previously polished using colloidal silica suspension. A FEG-SEM JEOL JSM 6500F coupled to a Channel 5 detector (HKL Technology) and a Thermoionic Electron Gun SEM coupled to a Crystal detector linked to software INCA (Oxford Instruments), both SEM operating at 20 kV were used.

A previous EBSD phase map at high magnification had been examined, subjected to a post-processing stage and compared to a SEM micrograph of the same area, after etching, in order to optimize the indexing process and to allow the identification of the different phases present. As a result, the following settings were proved to be optimum: high accuracy scanning, 7 minimum indexed bands and two phases to be indexed (Fe- α and Fe- γ). A subsequent filter had been applied on the high magnification map in order to remove non-reliable EBSD data (randomly oriented as-austenite-indexed pixels). The filter consisted of a Pattern Quality Threshold (PQT) and a Maximum Residual Angle (MRA), and different values were chosen for austenite and ferrite: Ferrite PQT, 25; austenite PQT, 95; and ferrite MRA, 3.1; austenite MRA, 2.2. The comparison

between this filtered high resolution phase map and the SEM micrograph of the same area, after etching, allowed to conclude that M/A features in SEM micrograph are mainly unindexed, whereas ferrite is almost completely and correctly indexed. The bad quality of unindexed patterns could not be attributed to a particular austenite crystal orientation, therefore big unindexed regions should be identified with quite deformed austenite but also martensite.

In addition, two EBSD analyses at low magnification were performed to examine the austenite distribution (step-size of 0.24 μm and following the post-process as explained above) and the bainite packet size (step-size of 0.45 μm and indexing just ferrite) within bands and in matrix. Both samples were then etched to allow the localization of bands within the scanned areas, using SEM.

With quantitative X-ray diffraction analysis the overall fraction of retained austenite was determined. For this purpose, cross-section samples were polished using 1 μ diamond paste and then subjected to several cycles of etching and polishing to obtain an undeformed surface. They were then step-scanned in a Bruker-Axs D8 X-ray diffractometer using unfiltered Co $K\alpha$ radiation. The scanning speed (2θ) was less than 0.3 degree/min. The machine was operated at 40 kV and 30 mA. The volume fraction of retained austenite was calculated from the integrated intensities of (111), (200), (220) and (311) austenite peaks, and those of (110), (200) and (211) for ferrite, using this number of peaks avoids possible bias due to crystallographic texture [10].

Results and discussion

Standard chemical etching and LOM examination of longitudinal samples allow to observe the presence of severe bands in the bainitic microstructure, Fig. 1 (a). By changing hot rolling conditions the size and amount of bands could be influenced within some limits. However due to their microscopic extensions there was not found any effect on classical macroscopic mechanical response, from tensile tests or impact toughness.

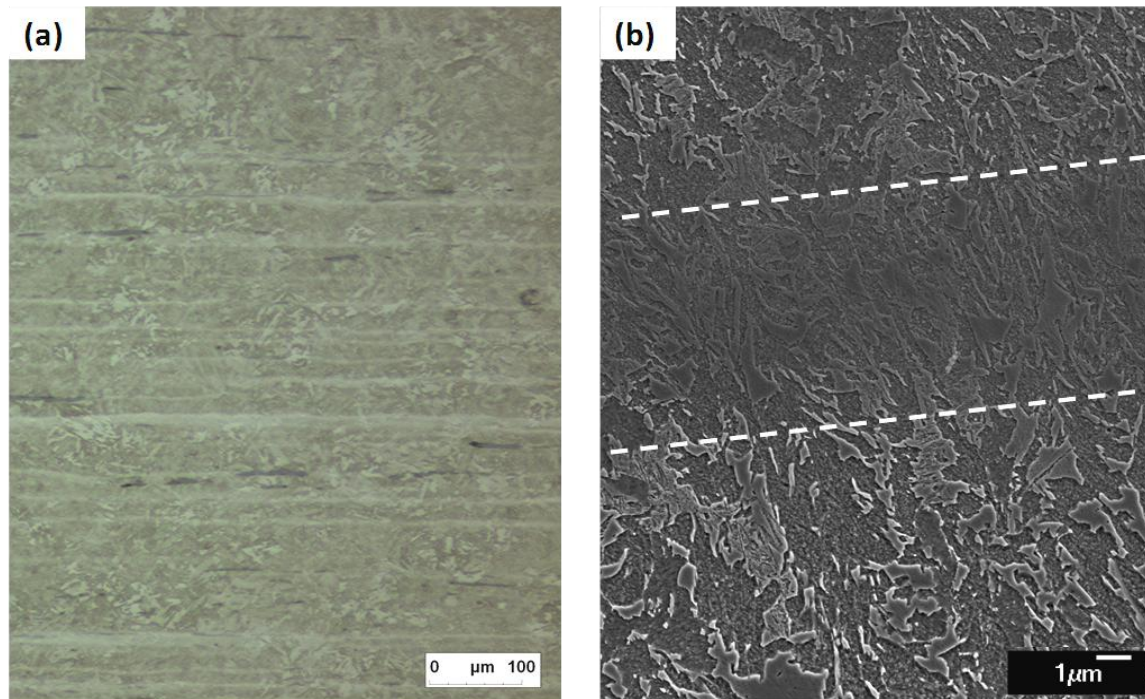


Fig. 1. (a) LOM and (b) secondary-electron SEM micrographs of a longitudinal sample after standard etching, revealing the presence of bands inside a bainitic matrix. Dashed lines in the SEM micrograph delimit band region.

At higher magnifications using SEM (Fig. 1(b)) the morphology of the microstructure within the bands could be identified as granular bainite. Both, matrix and bands, consist of the same bainitic structure with no evident differences in their respective morphologies. Nonetheless the resistance to etching obviously is different. This could be due to elemental segregation which is likely to affect phase transformation too. Indeed, by qualitative EDS profiles diverse concentrations in some substitutional alloying elements within bands and matrix were measured, Fig. 2. Bands are shown to be enriched in Cr, Si, Mo and depleted in Mn.

Although morphology is the same in bands and matrix the percentage of phases (bainite, retained austenite, martensite) could be different. From Fig. 1(b) a higher contribution of M/A constituent within the bands could be supposed. Bearing in mind that for this steel grade the TRIP effect (Transformation Induced Plasticity) is of major importance, the distribution of retained austenite might affect steel properties.

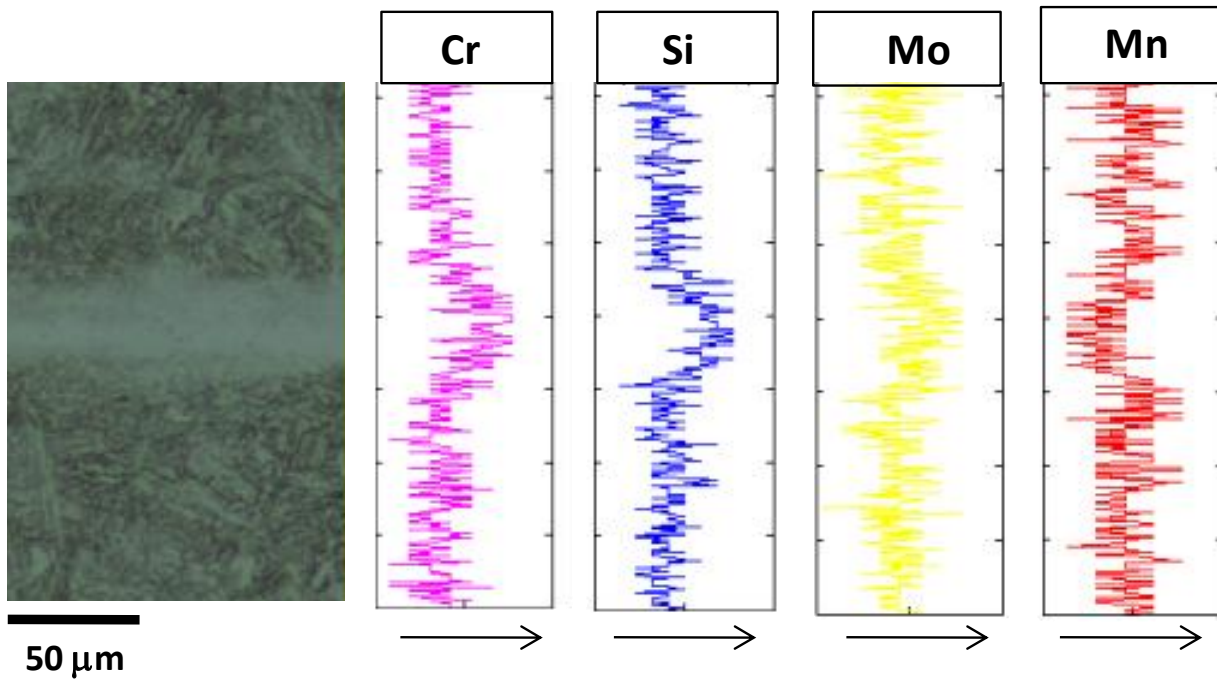


Fig. 2. EDS qualitative profiles after scanning in smart-map mode and the corresponding LOM micrograph of the same area confirming the presence of a chemical band.

In order to investigate possible differences between bands and matrix with respect to the amount of retained austenite, a low magnification EBSD analysis, Fig. 3(a), was applied over an area including both matrix and band. The corresponding secondary-electron SEM image is shown in Fig. 3(b). Assuming that unindexed pixels in the phase map stand for M/A constituent, and not taking into account the inclusion present, quantification yields to a gently higher M/A volume fraction in band, 50% in band vs. 40% in matrix. This EBSD result is in good agreement with those from quantitative metallography, 33% M/A volume fraction in matrix. On the other hand, the comparison with X-Ray diffraction results confirm the poor indexing rate of austenite, detected to be 20% austenite volume fraction by X-Ray diffraction and only 3% indexed by EBSD. However, in spite of this bad EBSD performance, the higher number of as-austenite-indexed pixels in matrix might suggest a slightly more deformed austenite and a higher martensite volume fraction within the band.

Finally, the bainitic packet size distribution in bands and in matrix was determined using the map in Fig. 4, having indexed just ferrite. The bainitic packet means, in this case, the effective grain controlling toughness, that is, the grain delimited by high angle boundaries ($>15^\circ$) [11,12]. In this sense, neither size nor crystallographic orientations differences on the bainite packets are detected.

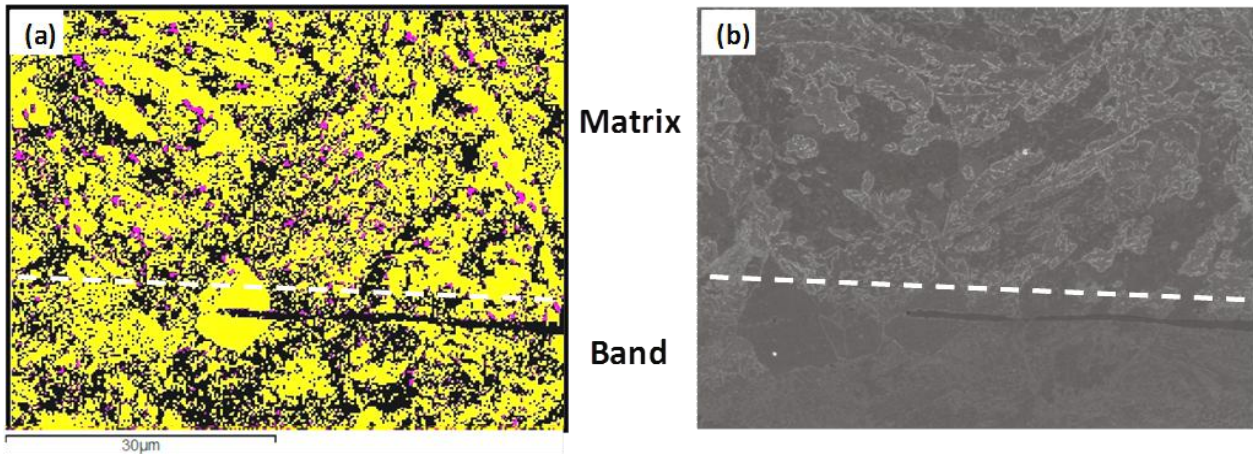


Fig. 3. (a) Phase map of a low magnification EBSD map and (b) the SEM micrograph of the same area, after etching, revealing the presence of a band. In the phase map, yellow stands for ferrite; pink, for austenite and black, for unindexed pixels. Please refer to the online version of the paper for a color view of the picture.

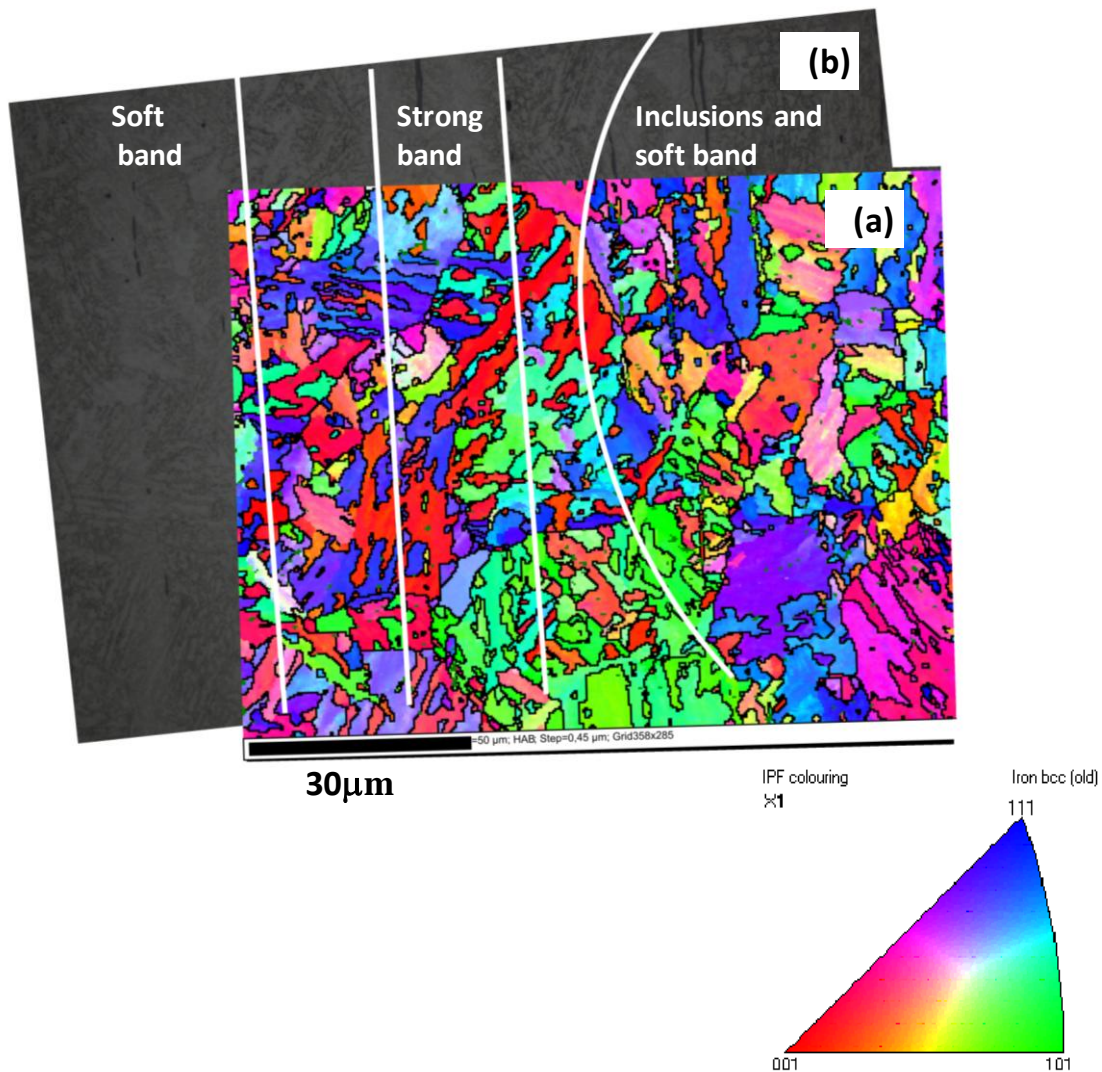


Fig. 4. EBSD orientation map of ferrite (a) over the corresponding LOM micrograph after etching (b), revealing the presence of bands.

Summary

The microstructure of TRIP-assisted carbide-free bainitic steel 0.23C-0.97Si-1.55Mn-0.15S-1.55Cr-0.14Mo (wt. %) after hot rolling was analyzed:

- After etching severe banding was observed
- The bainitic morphology did not change from bands to matrix
- Crystallographic packet sizes and orientations were not distinguishable between bands and matrix
- A variation of Cr, Si, Mo and Mn concentration was found to exist, which is probably the reason for a stronger resistance against metallographic etching in bands. Although the gradient in substitutional alloying elements has not been determined quantitatively, the Mn depletion in bands may be counteracting the effect of Cr and Mo on transformation kinetics, resulting in a similar microstructure in bands and matrix.
- Due to larger internal strains the amount of retained austenite could not be quantified precisely by EBSD. There is an indication that bands exhibit an elevated amount of martensite and a more deformed retained austenite

Although the studied bands could be easily seen after etching, their effect on steel properties is thought to be minor.

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