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Characterization of clusters and ultrafine precipitates in Nb-containing C–Mn–Si steels

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Abstract

Atom probe tomography was used to characterize the composition, sizes and distribution of Nb-, Fe-, Mo- and C-containing clusters and fine particles in thermomechanically processed CMnSi transformation induced plasticity steels, alloyed with Nb, Mo and/or Al. Nb-containing clusters and fine particles were detected in all the phases. The presence of C-rich clusters and significant increase of cluster density indicated that further decomposition of supersaturated retained austenite took place during isothermal hold at 450–500 °C.

Keywords: TRIP steels; Thermomechanical processing; Three-dimensional atom probe; Clustering; Transmission electron microscopy

1. Introduction

The strength of TRansformation Induced Plasticity (TRIP) steels typically ranges from 500 to 800 MPa. The conventional composition of these steels is 0.15–0.2 wt.%C–1.5 wt.%Mn–1.5 wt.% Si (0.68–0.91 at.%C–1.54 at.%Mn–3.01 at.%Si) with possible substitution of Si by Al and/or addition of Mo and P [1–4]. Further increases in strength may be achieved by microalloying with Nb, Ti or V. These additions lead to a refinement of the microstructure and the precipitation of carbides or carbonitrides.

Recently, with advances in atom probe tomography, attention has been drawn to the formation of solute clusters in steels and light alloys [4–12]. Clusters play an important role also in nucleation processes of second-phase precipitates [11,12] and in determining the mechanical properties of materials. It has been suggested that these clusters present a network of elastically soft and relatively diffuse obstacles for the movement of dislocations [9]. As a result, an increase in strength is achieved while maintaining good toughness [9].

This paper addresses the formation of nanosized particles and clusters in Nb-containing TRIP steels with additions of Mo and/or Al after laboratory simulated thermomechanical processing.

2. Experimental

The compositions of C–Mn–Si TRIP steels with additions of Nb, Al and Mo are shown in Table 1. All the steels were subjected to laboratory simulated thermomechanical processing described in detail elsewhere [13–15]. After the final rolling pass at 875 °C, the steels were slowly cooled through the ferrite formation region, followed by accelerated cooling from T_{AC} (Table 1) to avoid the formation of pearlite. After an isothermal hold at 450–500 °C (Table 1) during which bainite was formed, the samples were quenched to room temperature. The microstructures of thermomechanically processed steels were investigated with a Philips CM20 analytical transmission electron microscope (TEM), operated at 200 kV, at Monash University

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Table 1
Steel compositions and processing parameters

Steel	Element, at.%								Parameters		
	C	Si	Mn	Mo	Al	Cu	Nb	P	T_{AC} °C	T_{IH}, °C	t_{IH},s
Nb–Mo	0.96	2.88	1.49	**0.11**	0.02	0.017	**0.021**	0.044	735	450	1200
Nb–Al	1.00	2.31	1.52	0.002	**1.15**	0.026	**0.022**	0.048	840	500	1800
Nb–Mo–Al	0.95	2.29	1.51	**0.17**	**1.15**	0.026	**0.021**	0.048	760	470	1200

$T_{AC,}$ accelerated cooling start temperature; T_{IH}, isothermal hold temperature; t_{IH}, holding time.

and a local electrode atom probe at Oak Ridge National Laboratory [16,17]. Atom probe tomography (APT) characterizations were performed with a specimen temperature of 60 K, a pulse repetition rate of 200 kHz and a 20% pulse fraction.

The extent of solute enriched regions (radius of gyration) and the local solute concentrations in these regions were estimated with the maximum separation envelope method with a grid spacing of 0.1 nm [18]. A maximum separation distance between the atoms of interest of $d_{max} = 1$ nm was used for the clusters in polygonal ferrite in the Nb–Al and Nb–Mo–Al steels, whereas $d_{max} = 0.5$ nm was used for all other clusters. A minimum of 20 atoms was used for the small clusters to eliminate random fluctuations. Isoconcentration surfaces were also used to visualize the clusters and precipitates.

3. Results and discussion

3.1. TEM characterization

The microstructure of these steels comprises ~50% polygonal ferrite (PF), ~30% of bainite, (including upper bainite (UB) and carbide-free morphologies of granular bainite (GB) and acicular ferrite (AF)) and some retained austenite (RA) and martensite (M). A detailed description of the microstructure of these steels is given elsewhere [13–15]. The presence of fine particles was observed by TEM in PF and bainite in all the Nb-alloyed steels (Fig. 1a and b). These particles were identified as NbC from diffraction patterns. No fine precipitates were observed in the martensite or retained austenite. However, the high dislocation density makes detection difficult. An earlier study [12] that used carbon replicas on samples quenched immediately after the 875 °C deformation stage (Fig. 1c) revealed an uniform distribution of fine (Fe,Nb)C (Fig. 1d) precipitates throughout the microstructure, indicating that they formed in austenite. The average precipitate radius was determined to be 3.7 ± 1.2 nm with an average interparticle distance of 54 ± 20 nm.

3.2. APT characterization

APT revealed the presence of numerous predominantly Fe–Nb–Mo–C, Nb–C and Nb–Mo–C clusters in all phases of the alloyed steels. The majority of these clusters were uniformly distributed within the matrix. In addition, some fine precipitates of presumably Fe_3C with traces of Nb, Mo and Mn were also detected. Based on experimental observations we use here the term precipitates where clearly defined crystallographic arrangements of atoms (i.e., clearly visible planes) were observed. The term cluster is used for smaller size segregation of solute atoms, than precipitates. In addition, during several runs of samples in the local electrode atom probe no distinct non-iron planes were observed in the regions of nanoclusters while Fe crystallographic planes were detected at the same time in these areas. This indicates an absence of clearly defined crystallographic structure in clusters. The identification of the matrix phase was based on its composition as described in detail in [15]. Volumes of the matrix that contained between 0.02 and 0.06, 0.1 and 0.25 and in excess of 2 at.% C were deemed to be PF, BF and M/RA, respectively. In addition, the PF was enriched in Si, whereas a lower than nominal Si content was measured in the BF. As the observed clusters and precipitates were similar in PF and BF, and all the RA transformed to martensite at the analysis temperature of 60 K, these PF/BF and M/RA matrix regions are simply referred to as ferrite (F) and martensite, respectively.

Examples of clusters and particles are shown in the atom maps and 3 at.% (C + Nb) isoconcentration surface in the Nb–Mo steel in Fig. 2, in the martensite in the Nb–Al steel (Fig. 3a) and ferrite in the Nb–Mo–Al steel in Fig. 3b. The majority of the clusters were roughly spherical or ellipsoidal, with some clusters and iron carbides having a plate-like morphology, as shown in Fig. 3.

The distribution of cluster sizes and compositions as determined by the maximum separation method observed in the different matrix phases (F and M) in the Nb–Mo, Nb–Al and Nb–Mo–Al steels are shown in Figs. 4–6, respectively. The average radii of gyration of the clusters in the martensite were 0.8 ± 0.3, 0.8 ± 0.2 and 0.8 ± 0.3 nm in the Nb–Mo, Nb–Al and Nb–Mo–Al steels, respectively. The average Guinier radius of these clusters was 1.1 ± 0.3 nm. The compositions of almost all the clusters, as estimated by the maximum separation method, were similar. Low levels (1–3 at.%) of niobium and molybdenum were found in these clusters in all three steels and in the molybdenum-containing steels, respectively. No aluminum was detected in these clusters. However, the estimated levels of metallic solute were significantly lower and the carbon level was higher than the stoichiometric NbC, Mo_2C or MoC carbides. This extremely small size and