Solidification Microsegregation and Hot Ductility of Fe-Mn-C-Al-xNb TWIP Steels

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The influence of Nb addition on casting microstructures and high-temperature mechanical properties of Fe-Mn-C-Al-xNb TWIP steels was analyzed by phase-field modeling and experiments. Phase-field simulations showed that Mn, Nb, and C are enriched in inter-dendritic regions while Al is enriched in dendritic regions during solidification process of the investigated TWIP steels. Both phase-field simulations and microstructural characterization show that NbC precipitates are preferentially present near inter-dendritic boundaries. Nb addition slightly reduces hot ductility of the investigated steel at 1173 K (900 °C) while the Nb-added TWIP steels show better hot-ductility than the reference steel for deformation temperatures above 1373 K (1100 °C). NbC precipitates and inter-dendritic distances appear to be the most important variables that affect hot-ductility behavior of the investigated TWIP steels.

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I. INTRODUCTION

HIGH Mn twinning-induced plasticity (TWIP) steel is a type of structural steel, characterized by both high strength and superior formability.^[1–4] Deformation twinning is known to be the main strain hardening enhancing mechanism in TWIP steels *via* the dynamic Hall–Petch effect.^[5–7] TWIP steels generally show a large strength-ductility balance, *i.e.*, the product of ultimate tensile strength (UTS) and total elongation (TE), in excess of 50,000 MPa pct.

During straightening operation of continuous casting processes, casting defects such as transverse cracks are easily formed in steel.^[8,9] Hot ductility from high-temperature tensile tests can assess susceptibility to cracking during continuous casting.^[8] Hot ductility of TWIP steels has been an important focus of research due to high sensitivity of surface cracking in TWIP steels.^[10–22]

In TWIP steels, reduced Mn concentration resulted in an increase in hot ductility.^[10,17] Bleck *et al.*^[10] suggested that the wide solidification range of high Mn steels promoted micro-shrinkage and inter-dendritic segregation. Lan *et al.*^[16] reported that reduced matrix homogeneity by Mn micro-segregation and micro-porosity led to a decrease in hot ductility of a Fe-22Mn-0.6C (wt pct) TWIP steel. Kang *et al.*^[12,18–22] investigated the effect of various alloying elements such as Al, B, Ti, Nb, V, P, N, and S on hot ductility of TWIP steels.

Micro-alloying has been considered as a way to increase strength of TWIP steels.^[23–26] Previous works reported increases of strength of TWIP steels by Nb addition.^[24,26] Nb addition in TWIP steels also noticeably retards grain recrystallization as compared to micro-alloying with Ti or V.^[25] Mejia *et al.*^[15] reported beneficial influence of Nb addition on the hot ductility of a Fe-21Mn-1.3Al-1.5Si-0.5C (wt pct) TWIP steel, particularly in a temperature range of 1073 K to 1173 K (800 °C to 900 °C). However, the role of Nb addition in hot ductility of TWIP steels has not been clearly described.

Development of Nb-added TWIP steels requires understanding of the impact of Nb addition on castability, solidification microstructures, and high-temperature tensile properties of TWIP steels. The present work therefore aims at understanding the effect of Nb on solidification microstructures and high-temperature mechanical properties of Fe-17Mn-0.6C-1.5Al-xNb TWIP steels with a systematic variation of Nb content. To correctly describe the solidification process of micro-alloyed steels, it is essential to understand the partitioning of solutes between dendritic regions and

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interdendritic regions and the formation behavior of precipitates. Phase-field models are useful for the simulation of microstructure evolution during solidification.^[23–26] Therefore, the present work combines thermodynamics-based phase-field modeling, thermo-mechanical simulation tests and in-depth microstructural characterization and reports on the effect of Nb on solidification microstructures and high-temperature mechanical properties.

II. PHASE-FIELD SIMULATION

Phase-field computations, using the Micress[®] phase-field modeling software coupled to the Thermo-Calc[®] equilibrium computational thermodynamics software, were conducted to provide information about the evolution of solidification microstructures, elemental distribution and precipitation of compounds in equilibrium and off-equilibrium conditions. The software Micress[®] is based on the phase-field concept for various multiphase systems including steels.^[27] The cast microstructures were obtained within an initial cell where spatial and temporal phase field, $\phi_i(\vec{x}, t)$, corresponded to i phase ($\phi_i = 1$) and diffuse boundaries ($0 < \phi_i < 1$). The following governing equation was solved at each time step using the coupled thermodynamic database:

$$\dot{\phi}(\vec{x}, t) = \mu \left[\sigma \left(\nabla^2 \phi - \frac{(1-\phi)(1-2\phi)\phi}{\eta^2} \right) + \frac{1}{\eta} \Delta G \phi (1-\phi) \right],$$

where μ is the interfacial mobility; σ is the interfacial energy; η is the interfacial thickness, and ΔG is the thermodynamic driving force.^[28–31]

Dendritic casting microstructures and precipitation behavior of the Fe-17Mn-0.6C-1.5Al-xNb steels were simulated by phase-field modeling. The initial cells were $400 \mu\text{m} \times 600 \mu\text{m}$ in size, and they were fully liquid phases containing a zero-radius austenitic seed at the center of the cells. The simulations were started at 1688 K (1415 °C) using a cooling rate of 1.3 K s^{-1} , a typical cooling rate in continuous casting processes. Phase and interface mobility data used for the phase-field computations were obtained *via* coupling of the Micress[®] software with the Thermo-Calc[®] TCFE 7 database. The cells used for calculations of NbC precipitation behavior were $10 \mu\text{m} \times 5 \mu\text{m}$ in size. The starting cells had two flat liquid/austenite interfaces.

III. EXPERIMENTAL

Four TWIP steels containing 0, 0.025, 0.05, and 0.1 wt pct Nb were prepared by vacuum induction melting. Hereafter, the TWIP steel containing 0, 0.025, 0.05, and 0.1 wt pct Nb is referred to as the reference, 25Nb, 50Nb, and 100Nb steel, respectively. The chemical composition of the TWIP steels is listed in Table I. The experimental ingots had a dimension of $15 \text{ cm} \times 15 \text{ cm} \times 6 \text{ cm}$.

As-cast microstructures were investigated by scanning electron microscopy (SEM), electron probe micro-analysis (EPMA). Samples for SEM and EPMA analysis were prepared by mechanical polishing, followed by electrolytic polishing. Electrolytic polishing was conducted in a mixture of 5 pct perchloric acid and 95 pct ethanol using a voltage range of 20 to 40 V. SEM observations were conducted with a JEOL JEM-7100F

Table I. Composition of the TWIP Steels Used in the Present Study

Steel	C Wt Pct	Mn Wt Pct	Al Wt Pct	Nb Wt Pct
Reference	0.59	16.3	1.29	0.000
25Nb	0.61	16.7	1.45	0.025
50Nb	0.61	16.9	1.52	0.048
100Nb	0.59	16.3	1.48	0.088

Bold values indicate the change of Nb chemical composition.

Fig. 1—Pseudo-binary equilibrium phase diagram of the (a) Nb-free reference steel, (b) 25Nb steel, (c) 50Nb steel, and (d) 100Nb steel.

Fig. 2—Phase-field simulated (a) phase maps showing distribution of austenite and liquid phase during cooling and (b) phase fraction of austenite and liquid during cooling for the Nb-free reference steel.

SEM operating at 15 kV. Elemental analysis of precipitates was conducted by energy dispersive X-ray spectroscopy (EDS) in SEM. Elemental distribution in the as-cast materials was examined in a JEOL JXA-8100 EPMA operated at 15 kV using a step size of 0.4 μm .

High-temperature tensile tests were conducted in a laboratory continuous casting and thermo-mechanical simulator. Cylindrical tensile specimens were machined from the cast materials along one of the long edges (15 cm) of the ingots, with 10 mm in diameter, 150 mm in length and with a gauge section of 6 mm in diameter and 7 mm in length. The specimens were heated to 1173 K, 1373 K, and 1473 K (900 °C, 1100 °C, and 1200 °C) with a heating rate of 10 K s^{-1} , followed by isothermal holding for 200 seconds. Subsequently, the specimens were strained at a strain rate of $5.7 \times 10^{-3} \text{ s}^{-1}$. The microstructures in gauge areas of the tensile specimens after high-temperature tensile tests were investigated by EPMA and transmission electron microscopy (TEM). TEM samples were prepared as 3-mm-diameter disks which were mechanically polished to a thickness less than 100 μm and thinned by an electrolytic double jet technique at room temperature. A mixture of 5 pct perchloric acid and 95 pct ethanol was used as electrolyte. TEM observations were conducted in a JEOL JEM-2100F TEM operating at 200 kV.

IV. RESULTS

A. Phase-Field Simulation of Casting Microstructures of Fe-Mn-C-Al-xNb TWIP Steels

Figure 1 shows equilibrium phase diagrams of the investigated TWIP steels, calculated by Thermo-Calc. Equilibrium solidification-start and solidification-finish temperature of the reference Fe17Mn0.6C1.5Al steel is 1698 K and 1600 K (1425 °C and 1327 °C), respectively. Nb addition shows a negligible effect on these temperatures. At the constant C content of 0.6 wt pct, equilibrium NbC precipitation start temperature increases considerably by Nb addition, *i.e.*, from 1431 K (1158 °C) for the 25Nb steel to 1598 K (1325 °C) for the 100Nb steel.

Figure 2 shows Micress-simulated solidification behavior of the reference steel below the equilibrium solidification-start temperature. Figure 2(a) shows phase distribution of austenite and liquid phase during cooling. The austenite seed develops into austenitic dendrites and the fraction of austenite increases during cooling. At 1485 K (1212 °C), the percentage of austenite is about 90 pct. The phase map at 1093 K (820 °C) in Figure 2(a) shows that complete solidification is still not achieved at this temperature. Equilibrium solidification-finish temperature is 1600 K (1327 °C) for the

Fig. 3—Phase-field simulated elemental distribution maps of (a) C, (b) Mn, and (c) Al for the Nb-free reference steel.

reference steel (Figure 1(a)). In contrast, the Micress simulation, which considers solidification kinetics, predicts that 99 pct of liquid phase is solidified at 1333 K (1060 °C) for a cooling rate of 1.3 K s^{-1} (Figure 2(b)). Phase-field simulation predicts a much wider solidification temperature range as compared to equilibrium Thermo-Calc calculations. Below 1615 K (1342 °C), isolated islands of liquid phase are formed in the inter-dendritic regions. These may become potential sites for the formation of micro-porosities, which reduces hot ductility of steel.^[10]

Figure 3 shows the Micress-simulated evolution of elemental distribution of C, Mn, and Al in the reference steel during cooling. Figures 3(a) and (b) show that C and Mn are enriched in the liquid inter-dendritic regions. In contrast, Figure 3(c) shows that Al is enriched in the austenite dendritic regions.

Figure 4(a) shows the Micress-simulated phase map at 1576 K (1303 °C) for the reference steel, which consists of liquid and austenite. Figures 4(b) through (d) show temperature-dependent concentration plots of Mn, C, and Al along the black line drawn across the dendrite/liquid boundaries in Figure 4(a). Figure 4(b) shows that width of the dendrite increases with

decreasing temperature due to growth of the dendrite. At 1679 K (1406 °C), the dendrite has Mn concentration of approximately 14 wt pct while the inter-dendritic liquid has Mn concentration of about 19 wt pct. With decreasing temperature, average Mn concentration in both regions increases and degree of Mn partitioning also increases. For example, at 1485 K (1212 °C), the dendrite has Mn concentration of approximately 16 wt pct while the inter-dendritic region has Mn concentration of approximately 23 wt pct. At 1093 K (820 °C), however, average Mn concentration of the inter-dendritic regions is lower than that at 1485 K (1212 °C). The Mn concentration of the dendrite is higher than that at 1485 K (1212 °C). The decreased Mn concentration in the inter-dendritic regions at 1093 K (820 °C) is due to merge of the dendrite with the neighboring dendrite. At 1093 K (820 °C), the Mn concentration in the dendritic region ranges from 16 to 17 wt pct while that in the inter-dendritic regions ranges from 18 to 19 wt pct. In addition, difference in Mn concentration between the dendritic region and inter-dendritic regions at 1093 K (820 °C) is reduced as compared to the situation at 1485 K (1212 °C).

Fig. 4—Phase-field simulated (a) phase map at 1576 K (1303 °C) for the reference steel, which consists of liquid and austenite. Temperature-dependent concentration plots of (b) Mn, (c) C, and (d) Al along the black line drawn across the dendrite/liquid boundaries in (a).

Figure 4(c) shows that C is enriched in the inter-dendritic liquid phase. The C concentration in the dendrite remains almost unchanged with decreasing temperature. The dendrite has C concentration of approximately 0.3 wt pct. With decreasing temperature, average C concentration of the liquid inter-dendritic regions increases and then decreases. The C concentration in the inter-dendritic regions is higher than 2 wt pct at 1485 K (1212 °C), and ranges from 1.3 to 2 wt pct at 1093 K (820 °C).

In contrast to distribution of Mn and C, Al is enriched in the solidified dendrite as shown in Figure 4(d). The average Al concentration in the dendrite gradually increases with decreasing temperature from approximately 1.7 wt pct at 1679 K (1406 °C) to approximately 1.9 wt pct at 1093 K (820 °C). The Al concentration in the inter-dendritic regions is approximately 1.4 wt pct at 1679 K (1406 °C), and it ranges from 0.4 to 0.9 wt pct at 1093 K (820 °C).

Figure 5 shows the Micress-simulated solidification behavior of the 100Nb steel from the equilibrium solidification-start temperature to the NbC formation temperature. Nb is enriched in inter-dendritic liquid phase (Figure 5(a)). Figure 5(b) shows a

Micress-simulated Nb distribution map at 1641 K (1368 °C). Figure 5(c) shows temperature-dependent concentration plots of Nb along the black line drawn across the dendrite/liquid boundaries in Figure 5(b). The Nb concentration in the dendrite remains almost unchanged with decreasing temperature. The dendrite has Nb concentration of approximately 0.03 wt pct. Average Nb concentration of the inter-dendritic regions increases with decreasing temperature. The Nb concentration in the inter-dendritic regions is approximately 0.15 wt pct at 1677 K (1404 °C) and 0.23 wt pct at 1641 K (1368 °C). Considering that both C and Nb are enriched in the inter-dendritic region, NbC precipitation is expected to occur in this region.

NbC precipitation behavior for the Nb-added TWIP steels was simulated using a starting cell with two flat liquid/austenite interfaces. Figure 6(a) shows the simulated phase maps and distribution of NbC precipitates at 1379 K (1106 °C). The phase maps show thin inter-dendritic zones composed of the solid phase and the remained liquid phase. The distribution maps of NbC precipitates show preferential presence of the precipitates near inter-dendritic boundaries. Figure 6(b) shows quantitative information on NbC

Fig. 5—Phase-field simulated (a) Nb distribution maps for the 100Nb steel, (b) Nb distribution map at 1641 K (1368 °C), and (c) temperature-dependent Nb concentration along the black line drawn across the dendrite/liquid boundaries in (b).

precipitation, obtained by Micress-simulation. Average radius of NbC precipitates ranges from 15 to 35 nm, and it increases with increasing Nb concentration. Volume percentage of NbC precipitates also increases with increasing Nb concentration. It is approximately 0.07 pct for the 100Nb steel. The NbC precipitation start temperature increases with increasing Nb concentration. The precipitation start temperature obtained by the Micress simulation is about 100 K lower than the equilibrium precipitation start temperature predicted by the Thermo-Calc calculations shown in Figure 1.

B. Characterization of Casting Microstructures of Fe-Mn-C-Al-xNb TWIP Steels

Figure 7 presents EPMA elemental distribution maps of Mn, C, and Al for the as-cast reference steel. Figure 7(b) shows inhomogeneous Mn distribution. While the dendrites have low Mn concentration, the inter-dendritic regions are Mn-enriched. This is in agreement with the Micress simulation results (Figure 3). The C distribution map of Figure 7(c) shows the presence of coarse cementite precipitates indicated by black circles. Smaller C-rich particles are also present in the microstructure. These are most likely fine cementite precipitates. Figure 7(d) shows particles indicated by

red circles, and the particles are identified as AlN precipitates.

Figure 8 compares the concentration of Mn, C, and Al along the black-dotted line in Figure 7 with the Micress-simulated compositional profiles at 1093 K (820 °C). The concentration is plotted with respect to distance of the black-dotted line in Figure 7 for EPMA analysis and with respect to the black line in Figure 4(a) for Micress simulation. The Mn concentration from EPMA analysis shows similar distribution with the Micress-simulated Mn concentration, showing higher Mn concentration in the inter-dendritic region. In contrast, Figures 7(b) and (c) show that the experimentally determined distribution of C and Al does not show clear micro-segregation in either the inter-dendritic or the dendritic region. A scale of local compositional variations of C and Al is smaller than the micro-segregation distance between dendrites. The experimentally determined distribution of C and Al is not consistent with the Micress-simulation results. Absence of micro-segregation of C and Al in EPMA analysis is most likely due to formation of cementite and AlN precipitates.

Figure 9 presents EPMA elemental distribution maps of Mn, C, and Nb for the 100Nb steel. Figure 9(b) shows that the microstructure of 100Nb steel shows

Fig. 6—(a) Phase-field simulated phase maps and distribution of NbC precipitates for the Nb-added steels at 1379 K (1106 °C). (b) Nb concentration dependence of the precipitation start temperature, the volume percentage, and the average radius of the NbC precipitates.

inhomogeneous distribution of Mn. The C (Figure 9(c)) and Nb (Figure 9(d)) distribution maps show presence of NbC precipitates, preferentially located at interdendritic boundaries. The preferential presence of NbC

precipitates at interdendritic boundaries is consistent with the Micress-simulation results in Figure 6.

Figure 10 shows SEM micrographs and EDS spectra of the identified precipitates in the reference steel and the 100Nb steel. AlN precipitates and MgO inclusions are present in all the investigated steels. MgO inclusions are often associated with AlN precipitates as shown in Figure 10(b). Figure 10(d) shows the SEM micrograph and the corresponding EDS spectrum for a NbC precipitate in the 100Nb steel. The EPMA analysis for the 100Nb steel in Figure 9 shows that NbC precipitates are more frequently observed compared to AlN precipitates.

C. High-Temperature Mechanical Properties and Deformation Microstructures of Fe-Mn-C-Al-xNb TWIP Steels

High-temperature tensile tests were conducted at 1173 K, 1373 K, and 1474 K (900 °C, 1100 °C, and 1200 °C) for all the investigated TWIP steels. Figure 11(a) shows details of test procedures. Figures 11(b) through (d) provide stress–strain curves of the investigated TWIP steels. Peak stresses decrease with increasing deformation temperature for all the investigated steels. The stress–strain curves at 1173 K (900 °C) in Figure 11(b) show that strength of the Nb-added steels is higher than that of the reference steel. The 100Nb steel shows the highest peak stress while the reference steel shows the lowest peak stress. Fracture strain of the reference steel is slightly larger than that of the Nb-added steels. The peak stresses at 1173 K (900 °C) range from 80 to 110 MPa. The stress–strain curves at 1373 K (1100 °C) in Figure 11(c) shows that the 50Nb steel has the highest peak stress, and the 100Nb steel has the largest fracture strain. The peak stresses at 1373 K (1100 °C) range from 40 to 60 MPa. Figure 11(d) shows the stress–strain curves at 1473 K (1200 °C). The peak stresses for all the investigated TWIP steels are similar at 1473 K (1200 °C). The Nb-added steels show larger fracture strain than the reference steel. The peak stresses at 1473 K (1200 °C) are approximately 30 MPa. Mejía *et al.*^[14] investigated effect of Nb and Mo on the high-temperature mechanical properties of an as-cast Fe-21Mn-1.3Al-1.5Si-0.5C (wt pct) TWIP steel produced by ingot casting in a sand mold. They reported that the peak stress of the Nb-added TWIP steel at 1173 K (900 °C) was higher than that of their reference Nb-free TWIP steel. They also reported similar peak stresses for both the reference steel and the Nb-added steel at 1373 K (1100 °C). Lan *et al.*^[16] investigated high-temperature mechanical properties of an as-cast Fe-22Mn-0.7C (wt pct) TWIP steel in the range of 973 K to 1573 K (700 °C to 1300 °C). They reported that the peak stress decreases with increasing deformation temperature. These results^[14,16] are in agreement with those of the present work.

Figure 12(a) presents stress–strain curves of the reference steel at different deformation temperatures. The strength decreases with increasing deformation temperature. The area under the stress–strain curve, *i.e.*, the energy absorption capacity, is 3805 MPa pct for 1173 K

Fig. 7—(a) SEM backscattered electron (BSE) image and EPMA elemental distribution maps showing (b) Mn, (c) C and (d) Al concentration in the as-cast reference steel.

(900 °C), 1514 MPa pct for 1373 K (1100 °C) and 1922 MPa pct for 1473 K (1200 °C). Figure 12(b) shows the peak stress and the reduction of area (RA) with respect to deformation temperatures. The peak stress decreases continuously from 90 MPa at 1173 K (900 °C) to 28 MPa at 1473 K (1200 °C) while the RA increases from 48 pct at 1173 K (900 °C) to 55 pct at 1373 K (1100 °C). The RA then slightly decreases to 54 pct at 1473 K (1200 °C). Lan *et al.*^[16] reported three brittle zones for an as-cast Fe-22Mn-0.7C (wt pct) TWIP steel. The first brittle zone was observed at temperatures above 1473 K (1200 °C). In this zone, RA of the TWIP steel decreased with increasing deformation temperature. The second brittle zone was observed from 1473 K to 1173 K (1200 °C to 900 °C) while the third second brittle zone was observed from 1223 K to 1073 K (950 °C to 800 °C). The third brittle zone showed lower hot ductility than the second brittle zone. The RA values of the Nb-free reference steel in the present work are higher than those reported by Lan *et al.*^[16] This could be due to the fact that the reference steel in the present work shows lower Mn concentration of 17 wt pct compared to the material investigated by Lan *et al.*,^[16] since reduced Mn concentration results in an increase in hot ductility.^[10,17]

Figure 13 shows EPMA elemental distribution maps of Fe, Mn, and C for the reference steel after deformation at different temperatures. The inhomogeneous distribution of Mn observed in the as-cast microstructure (Figure 7(a)) is still present after high-temperature tensile deformation, *i.e.*, dendritic structures show low

Mn concentration while inter-dendritic regions show high Mn concentration. The AlN precipitates indicated by red circles are mostly present at interdendritic boundaries in the tested samples for all the deformation temperatures, and they are rarely present in the dendritic regions. Cavities and cracks are present in the tested samples for all the deformation temperatures. These defects indicated by black circles are primarily present in the Mn-rich inter-dendritic regions.

Figure 14(a) presents stress–strain curves of the 100Nb steel at different deformation temperatures. The strength decreases with increasing deformation temperature. The area under the stress–strain curve, *i.e.*, the energy absorption capacity, is 4815 MPa pct for 1173 K (900 °C), 2553 MPa pct for 1373 K (1100 °C) and 1745 MPa pct for 1473 K (1200 °C). Figure 14(b) shows temperature dependence of the peak stress and the RA. The peak stress decreases continuously from 110 MPa at 1173 K (900 °C) to 33 MPa at 1473 K (1200 °C). The RA increases considerably from 24 pct at 1173 K (900 °C) to 79 pct at 1473 K (1200 °C). The increase of RA from 1173 K (900 °C) to 1473 K (1200 °C) was also reported in a Fe-18Mn-0.6C-0.02Nb (wt pct) TWIP steel.^[11]

Figure 15 shows EPMA elemental distribution maps of Fe, Mn, Al, and C for the 100Nb steel after deformation at different temperatures. The materials show inhomogeneous distribution of Mn, similarly with the case of the reference steel. The AlN precipitates indicated by red circles are present in the steel after deformation at 1173 K, 1373 K, and 1473 K (900 °C,

Fig. 8—Comparison of concentration of (a) Mn, (b) C, and (c) Al along the black-dotted line in Fig. 7 with the Micress-simulated compositional profiles at 1093 K (820 °C). The concentration is plotted with respect to the distance of the black-dotted line in Fig. 7 for EPMA analysis and the black line in Fig. 4(a) for Micress simulation.

1100 °C, and 1200 °C). NbC precipitates indicated by violet circles are mostly present at interdendritic boundaries. Cavities and cracks are present in the tested samples for all the deformation temperatures. These defects, indicated by black circles in Figure 15, are mostly present in the Mn-rich inter-dendritic regions.

Figure 16 summarizes deformation temperature dependence of RA for the investigated TWIP steels. Hot ductility decreases with increasing Nb concentration

for the tests at 1173 K (900 °C). In contrast, hot ductility of the Nb-added steels is higher than that of the Nb-free reference steel for the tests at 1373 K (1100 °C) and 1473 K (1200 °C). The observed different Nb effects on hot ductility of the investigated steels, depending on deformation temperatures, will be discussed in Section V.

V. DISCUSSION

Partitioning of alloying elements between the liquid phase and growing solid dendrites during solidification results in micro-segregation in solidified microstructures.^[32] Phase-field simulations showed that Mn, Nb, and C are enriched in inter-dendritic regions while Al is enriched in dendritic regions during solidification of the investigated TWIP steels. Enrichment of C and Mn in the inter-dendritic liquid phase (Figures 3(a), (b)) is consistent with previous works.^[32,33] Al is known to have a small tendency to segregate to the solid phase, *i.e.*, either in δ -ferrite or austenite, during solidification of carbon steels.^[33,34] Ennis *et al.*^[35] however reported enrichment of Al in dendritic structures in an as-cast steel. Their observations are consistent with the Micress simulation in the present work (Figure 3(c)). Enrichment of Nb in the inter-dendritic liquid phase (Figure 5(a)) is also consistent with previous works that reported high tendency for Nb to segregate to the inter-dendritic liquid phase.^[33,34] Enrichment of both C and Nb in the inter-dendritic liquid phase leads to preferential precipitation of NbC at inter-dendritic boundaries (Figures 6 and 9).

Variables that control hot-ductility of steel include strain rate, grain size, precipitation, inclusion concentration^[8] and matrix homogeneity.^[16] Increasing strain rates improves hot ductility by reducing amount of grain boundary sliding. Refinement of a grain size improves hot ductility by suppressing crack propagation along grain boundaries.^[8] Fine precipitates, in particular at grain boundaries, reduce hot ductility since the particles located close enough one another facilitate linking of cracks.^[8] Reduced matrix homogeneity characterized by pronounced micro-segregation and presence of micro-porosities also reduces hot ductility.^[16]

Previous works on hot ductility of TWIP steels reported an increase of hot ductility with increasing strain rates.^[12,13] Increasing strain rates considerably to improve hot ductility of steel is generally not possible in conventional continuous casting.^[8] Lan *et al.*^[16] reported that a small grain size and weak micro-segregation improved hot ductility of a TWIP steel. The Mn segregation in inter-dendritic regions of high Mn steels led to low hot ductility due to the presence of zones that have low melting temperatures in the inter-dendritic areas.^[10]

Nb addition slightly reduces hot ductility of the investigated steel at 1173 K (900 °C) (Figure 16). This is most likely due to fine NbC precipitates present at inter-dendritic boundaries, which could result in weakening of interfaces. The presence of fine NbC precipitates could also contribute to the higher peak stress of

Fig. 9—(a) SEM backscattered electron (BSE) image and EPMA elemental distribution maps showing (b) Mn, (c) C, and (d) Nb concentration in the as-cast 100Nb steel.

Fig. 10—Microstructure and corresponding composition of the precipitates: (a) AIN and (b) MgO surrounded by AIN in the Nb-free reference steel. (c) AIN and (d) NbC precipitates in the 100Nb steel.

Fig. 11—(a) Details of procedures of high-temperature tensile tests. (b) Stress-strain curves of the TWIP steels tested at (b) 1173 K (900 °C), (c) 1373 K (1100 °C), and (d) 1473 K (1200 °C).

the Nb-added steels (Figure 11(b)). Mejia *et al.*^[15] mentioned that Nb addition resulted in a loss of hot ductility due to NbC precipitation at grain boundaries. According to them, grain boundary precipitation prevented dynamic recrystallization and led to grain boundary sliding. Since the quantity of NbC precipitates increases with increasing Nb concentration, NbC precipitation could result in low hot ductility at lower deformation temperatures. Deformation microstructures of the reference and 100Nb steel after deformation at 1173 K (900 °C) were observed by TEM (Figures 17(a) through (c)). Deformation microstructure of the reference steel shows homogeneous distribution of dislocations and the presence of low-angle boundaries indicated by white arrows (Figure 17(a)). Figures 17(b) and (c) show deformation microstructures of the 100Nb steel. Figure 17(b) shows homogeneous distribution of fine NbC precipitates while Figure 17(c) shows heterogeneous distribution of fine NbC precipitates. Some precipitates indicated by white arrows show a row-like characteristic, which are most likely interface precipitates. The average diameter of NbC precipitates in the 100Nb steel deformed at 1173 K (900 °C) is approximately 15 nm. The interaction between dislocations and NbC precipitates is evidenced by pinning of some dislocations by the precipitates. Compared to the reference steel, distribution of dislocations is more heterogeneous in the 100Nb steel. Therefore, presence

Fig. 12—(a) Stress-strain curves of the Nb-free reference steel tested at 1173 K (900 °C), 1373 K (1100 °C), and 1473 K (1200 °C). (b) The peak stress and the reduction of area of the Nb-free reference steel with respect to deformation temperatures.

Fig. 13—EPMA elemental distribution maps of Fe, Mn, and C for the Nb-free reference steel tested at (a) 1173 K (900 °C), (b) 1373 K (1100 °C), and (c) 1473 K (1200 °C).

of fine NbC precipitates and heterogeneous distribution of dislocations could affect decreased hot ductility of the Nb-added TWIP steels.

The Nb-added TWIP steels show better hot-ductility than the reference steel for deformation temperatures above 1373 K (1100 °C). As shown in Figures 13 and 15, typical inter-dendritic distances are smaller in the Nb-added steel as compared to the Nb-free reference steel. Nb addition is known to inhibit grain growth, and a fine grain size is beneficial to hot-ductility of high Mn steels.^[16] In addition to the effect of dragging or pinning forces, Nb micro-alloying could increase thermal conductivity and decrease effective solidification time, which resulted in a decrease in secondary dendritic arm spacing.^[36] These combined effects can lead to refined solidification microstructures by Nb micro-alloying. Cracks are frequently observed in the Mn-enriched inter-dendritic regions (Figures 13 and 15). These cracks may propagate easily in the coarse inter-dendritic regions of the Nb-free reference steel. NbC precipitates coarsen at high temperatures, and they could be less detrimental than fine precipitates. The presence of coarsened NbC precipitates could also result in similar peak stresses of the reference steel and the Nb-added steels (Figure 11(d)). Figure 17(d) shows a deformation

Fig. 14—(a) Stress-strain curves of the 100Nb steel tested at 1173 K (900 °C), 1373 K (1100 °C), and 1473 K (1200 °C). (b) The peak stress and the reduction of area of the 100Nb steel with respect to deformation temperatures.

Fig. 15—EPMA elemental distribution maps of Fe, Mn, Al, and C in the 100Nb steel tested at (a) 1173 K (900 °C), (b) 1373 K (1100 °C), and (c) 1473 K (1200 °C).

microstructure of the 100Nb steel deformed at 1473 K (1200 °C). Dislocation density of the 100Nb steel deformed at 1473 K (1200 °C) is much lower than that at 1173 K (900 °C). Some dislocations are pinned by the NbC precipitates. The NbC precipitates in the material deformed at 1473 K (1200 °C) are coarser than that at 1173 K (900 °C). The average diameter of NbC precipitates in the 100Nb steel deformed at 1473 K (1200 °C) is approximately 40 nm. Both effects, *i.e.*, fine inter-dendritic areas and coarsened NbC precipitates, therefore

explain the increased hot ductility in the Nb-added TWIP steels tested at high temperatures.

Dynamic recrystallization (DRX) is an important phenomenon affecting hot deformation behavior of high Mn steels.^[14] In the present case, however, effect of DRX on hot-ductility of the investigated steels is very limited. EBSD analyses of deformation microstructures at 1473 K (1200 °C) showed that fraction of recrystallized grains is less than 0.005 for both the reference steel and the 100Nb steel. The absence of DRX is closely

related to composition of TWIP steels. Liu *et al.*^[17] reported that TWIP steels with high Mn content inhibited DRX and promoted grain boundary sliding during high-temperature tensile tests, which decreased hot ductility of the TWIP steels. They also reported that

Fig. 16—Deformation temperature dependence of the reduction of area for the investigated TWIP steels.

no DRX grains were observed in the Al-added TWIP steels. This is consistent with the present work that the investigated Al-added TWIP steels with high Mn content does not show significant DRX during high-temperature tensile tests.

VI. CONCLUSIONS

In this study, we investigated effect of Nb addition on casting microstructures and high-temperature mechanical properties of Fe-Mn-C-Al-xNb TWIP steels using both phase-field simulations and experiments. We draw the following conclusions:

1. Phase-field simulations showed that Mn, Nb, and C are enriched in inter-dendritic regions while Al is enriched in dendritic regions during solidification of the investigated TWIP steels. Both phase-field simulations and experimental characterizations show that NbC precipitates are preferentially present near inter-dendritic boundaries.

Fig. 17—TEM deformation microstructures of the (a) reference, (b, c) 100Nb steel after deformation at 1173 K (900 °C), and (d) 100Nb steel after deformation at 1473 K (1200 °C).

2. Nb addition slightly reduces hot ductility of the investigated steel at 1173 K (900 °C). The presence of fine NbC precipitates and heterogeneous distribution of dislocations most likely result in the slight reduction in hot ductility.
3. The Nb-added TWIP steels show better hot-ductility than the reference steel for deformation temperatures above 1373 K (1100 °C). Typical inter-dendritic distances are smaller in the Nb-added steel as compared to the Nb-free reference steel. Easy propagation of cracks in the coarse inter-dendritic regions of the Nb-free reference steel could result in worse hot-ductility of the reference steel. In addition, NbC precipitates coarsen at high temperatures, and they could be less detrimental to hot ductility than fine precipitates.

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