Research Article

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Effect of Nb addition on the solidification structure of Fe-Mn-C-Al twin-induced plasticity steel

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Abstract: The solidification microstructure of an Fe-Mn-C-Al high-Mn steel with added Nb was studied by optical microscopy and scanning electron microscopy. The results showed that the size of the columnar and equiaxed grains in the solidified structure of the high-Mn steel without added Nb was large. When Nb was added, the size of the equiaxed and columnar grains in the solidification structure of the high-Mn steel decreased. The twin-induced plasticity in the high-Mn steel with added Nb clearly refined the grain size of the solidified structure and simultaneously increased the equiaxed crystallization rate. The highmelting-point second-phase particles increased the heterogeneous nucleation rate, which was beneficial for grain refinement. A large amount of Nb precipitates from the solidification process increased the equiaxed crystallization rate of the solidified microstructure. The increased crystallization rate affected the growth direction of the columnar and equiaxed grains during solidification and indirectly decreased the probability of crack formation, which improved the quality of the twin-induced plastic steel.

Keywords: high-manganese steel, solidification microstructure, grain size refinement, isometric crystal, inclusion

1 Introduction

With the growth of the automobile industry, the demand for automobile steel is increasing. The manufacturing requirements of diversified shape, high performance, light weight, and low cost are moving the development focus of iron and steel materials toward high strength and high plasticity. An improvement in the strength and plasticity can reduce the weight of a car body to achieve energy savings and reduced emissions without reducing safety [1–3]. The twinninginduced plasticity (TWIP) steel [4,5] is an automobile steel with high strength, high plasticity, and high energy absorption capacity and that does not exhibit lowtemperature brittle transition. Therefore, it is considered an ideal next-generation automobile steel [6-8].

At present, the large-scale industrial production and application of the TWIP steel still faces difficulties [9-13]. The traditional melting process of the high-Mn steel is not modified during melting. Thus, in the as-cast microstructure of the ordinary high-Mn steel, carbides are distributed in a network along the grain boundaries, where the reticulated carbides increase in thickness with increasing the C content [14–16]. Li and Xu [17] concluded that these reticulated and brittle carbides caused the plasticity and toughness of a material to decrease. As a crack source, their sizes and distribution greatly reduce the plasticity and toughness of the high-Mn steel. In addition, the nonmetallic inclusions also distribute into a network, whereas molten steel solidifies, which not only increases the brittleness of the material but also tends to break the casting during the solidification process. Yong [18] proposed that the carbides could be dissolved without modifying the agent when toughening the high-Mn steel and that a single austenite structure could be obtained after the austenite temperature is maintained for an extended period. Experiments have shown that grain growth is inevitable with increasing holding time. The original distributions of carbides leave microcracks due to the dissolution of carbides, reducing the toughness of materials and making the high-Mn steel

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prone to failure during service. For this reason, modifiers have been added to the high-Mn steel during smelting to improve the morphology and distribution of carbides and the performance of the resulting steel.

A metamorphic treatment is a melt process that occurs when a certain trace element or a nonmetal is added to the alloy liquid to control the properties of the alloy [19–22]. The realloying of the high-Mn steel by adding elements such as Nb, Cr, Mo, W, V, and Ti can further solidify and strengthen the austenite, and a small amount of undissolved carbides can also play a role in precipitation strengthening. However, in the experiments where a modifier was added, researchers proposed that only Nb, Cr, Mo, W, V, or Ti could be added to austenite to further strengthen the solid solution; thus, the solidification structure of the high-Mn steel with the addition of Nb was not systematically studied.

Wang et al. [23] and Takamiya and Furukimi [24] found that niobium carbide inclusions (i.e., NbC and Nb₂C) were formed in the high-Mn steel when Nb was added. Takamiya and Furukimi [24] explored the austenite grain growth mechanism in the high-Mn steel by adding Nb and found that, with a decrease in temperature, the precipitated austenite continuously grew. At the interface front during austenite growth, the C, Mn, and Nb solutes were enriched [25]. It was concluded that C, Mn, and Nb in the high-Mn steel segregated readily. Upon liquid quenching, with a decrease in temperature, the enrichment of C, Mn, and Nb at the front of the austenite growth interface was inevitable. Secondary austenite grains were easily prevented from necking; the austenite grains were therefore clearly refined. However, few studies on the effect of Nb inclusions on grain refinement and the source of cracks in TWIP steel solidified with different contents of Nb have been reported. Therefore, the effect of different Nb contents on the solidification structure of the Fe-Mn-C-Al TWIP steel warrants further investigation because such studies would provide valuable information for the continued study of the solidification structure of TWIP steel [26-28].

In the past experiments and literatures, although niobium element has explored the austenite grain mechanism of TWIP steel and found some change mechanisms of austenite growth, no one has accurately studied and calculated the growth changes of the columnar crystal and the equiaxed crystal on the solidified structure surface of TWIP steel after adding niobium element. One of the points in this article is that niobium element and the columnar and equiaxed grains of TWIP steel are systematically measured and analyzed. In addition, the theoretical calculation of mismatch degree in the crystallography of TWIP steel is carried out for the first time in the calculation process. The orientation relationship between niobium and austenite grains and the difficult and easy process of precipitation are calculated. Theoretically, the precipitation process of niobium inclusions is explained. Therefore, by control-ling the content of niobium, the solidification process of TWIP steel of the Fe–Mn–C–Al system is systematically analyzed and summarized. The research shows that the addition of niobium to the high-Mn TWIP steel can play a role in grain refinement and crack reduction and

provide reference for producing the high-quality TWIP

2 Experimental materials and methods

steel.

Four heats of the Fe–Mn–C–Al series TWIP steel (A, B, C, and D) were produced. The melting process was as follows: With industrial pure Fe (99.94%), electrolytic Mn, metallic Al, and Nb as the main raw materials, the melting material was proportionally allocated to 10 kg. The melting material was placed in a medium-frequency induction heating furnace and heated to 1,650°C. Al and Nb were then added. After melting completely, the ingot was finally cast in the ingot shown in Figure 1(a) at a constant temperature for 5 min. Al and Nb were not added to steel A; only Al (not Nb) was added to steel B. Al and Nb were both added to steels C and D, but the Nb content was varied. The casting and cooling conditions for the four steels were the same. The main components of the steels are shown in Table 1.



Figure 1: Sampling diagram.

Table 1: Chemical composition of the Fe-Mn-C-Al high-Mn steel

Number	C	Mn	Al	Nb	N	Fe
A	0.71	15.83	0	0	0.0084	Balance
В	0.65	17.65	1.50	0	0.0076	Balance
С	0.69	17.16	1.51	0.05	0.0091	Balance
D	0.61	16.02	1.51	0.08	0.0052	Balance

The cast ingot was symmetrically cut into two halves along the central axis and planed longitudinally using the method shown in Figure 1(a). The shape of the ingot is shown in the same figure. It was then machined and polished to a mirror finish. The ingot was corroded by a mixture of hydrochloric acid, nitric acid, and anhydrous ethanol with a volume ratio of 1:4:5. The macroscopic morphology of the solidified structure was observed after corrosion.

Different positions of the ingot were cut into small cubic metallographic samples with dimensions of $10 \times 10 \times 10$ mm by the method shown in Figure 1(b). Then the morphology of inclusions was observed and analyzed using a GX51 DP26 optical microscope and a SIGMA X-Max 20 scanning electron microscope. The chemical composition of the steel used in this work is shown in Table 1.

3 Experimental results and analysis

3.1 Analysis of solidification structure and appearance

The four TWIP steels from the smelting used herein were cut along their longitudinal axis to obtain two symmetrical steel samples. Some of the steel samples (shown in Figure 1(a)) were polished and etched to obtain the samples shown in the original solidification structure (Figure 2). Figure 2 shows that the solidification structure of the high-Mn steel with added Nb mainly comprised equiaxed and columnar grains and that the content of the fine grains on its surface was very small. This phenomenon is similar to the results reported by Shun et al. [29], Wang et al. [30]. The main reason for the development of columnar grains is that the heat in the molten steel is transferred unilaterally from the center to the edge during steelmaking and casting; columnar grains then grow in the direction opposite the heat transfer until the central part of the final ingot solidifies completely. In addition, the number of cores of uniform nucleation and nonuniform nucleation in the liquid phase of the crystallization front influence the ratio of columnar grain formation. A decrease in the number of nucleation cores results in more favorable growth of columnar grains.

Figure 2 shows that the columnar grains and the equiaxed grains in the TWIP steel that did not contain Nb were large: the average size of the A and B steel grains in Figure 2 was 3.21 mm. The size of the columnar grains and equiaxed grains of the TWIP steel after Nb addition was substantially smaller: the average size of all interface grains was measured to be 1.73 mm. When the Nb content was increased (Figure 2(d)), the grain size of the TWIP steel was further reduced and the average size was only 1.25 mm. These results show that Nb strongly influenced the grain refinement.

3.2 Analysis of columnar and equiaxed grain size

To further analyze the effect of the Nb addition on the solidification structure, the length of the columnar grains, the dendrite width, and the grain size of the central equiaxed grains of the longitudinal section of the four Fe–Mn–C–Al TWIP steels were determined. A detailed analysis was carried out; the results are shown in Figure 3.

Figure 3 shows that, for the same Fe–Mn–C–Al TWIP ingot, the size of the different types of grains in the solidification structure varied greatly. The equiaxed grain size of the profile was 2.86 mm, and the average length of the columnar grains of the B, C, and D steels of the Fe–Mn–C–Al system was greater than 3.5 mm. However, the size of the equiaxed grains of TWIP steels C and D in the Fe–Mn–C–Al system was less than 1.1 mm.

A comparison of the grain sizes in the same region (columnar grain region or equiaxed grain region) of the four steels revealed that the length of the columnar grains did not substantially change and that the columnar grain width and the equiaxed grain size were small.

The length of columnar grains accounted for 16.23% of the columnar grains, whereas the widths of the columnar and equiaxed grains accounted for 62.63% and 61.41%, respectively. Analysis of the four Fe–Mn–C–Al



Figure 2: Solidification structure of the longitudinal section of the high-Mn steel: (a) Fe–Mn–C solidification structure; (b) Fe–Mn–C–Al solidification structure; (c) Fe–Mn–C–Al–0.05Nb solidified structure; and (d) Fe–Mn–C–Al–0.08Nb solidified structure.



Figure 3: The grain size and average column length (width) of four Fe-Mn-C-Al twin crystal-induced plastics.

TWIP steels shows that the addition of Nb had no obvious effect on the length of columnar grains, whereas the addition of Nb greatly reduced the width of the columnar grains and the length of the equiaxed grains; this behavior may be related to the pinning effect of Nb at the grain boundaries and the inhibition of the nucleation, thus resulting in certain grain refinement.

3.2.1 Proportional analysis of isometric grains

The equiaxed grain rate is the proportion of equiaxed grains in the solidification structure and is an important index to measure the solidification structure characteristics of ingots. Under the same conditions, the higher the medium-axis grain rate of steel, the better the processing performance of the ingot and the lower the probability of cracking [31]. The equiaxed crystallinity of the longitudinal section of steels A, B, C, and D is shown in Figure 4.

Because the longitudinal profile is observed in the vertical direction of the equiaxed grains, Figure 4 shows that the longitudinal isometric grain rate reached approximately 32%. For Fe-Mn-C-Al TWIP steels A, B, C, and D, the longitudinal isometric grain rate gradually increased. The equiaxed grain ratios of steels C and D were clearly higher than those of steels A and B, whereas the equiaxed grain ratios of steels A and B were approximately the same. The equiaxed grain ratio of the Fe-Mn-C-Al-based TWIP steel exhibited no obvious change after Al was added; however, the equiaxed grain ratio was obviously improved after Nb was added. This result shows that the addition of Nb can effectively improve the equiaxed grain ratio of the Fe-Mn-C-Al TWIP steel. Figure 4 and the corresponding experimental data also show that Fe-Mn-C-Al twins were treated by adding Nb. The equiaxed crystallinity of the longitudinal section of the induced plastic steel increased by approximately 8%.

3.2.2 Growth of columnar grains

Because the size of grains formed at the beginning of grain growth is different, the size of columnar grains formed later is different. With the migration of the solidification



Figure 4: The equiaxed crystal ratio of the Fe-Mn-C-Al system twinned plasticity steel.

interface, columnar grains continued to extend forward, as shown in Figure 2. Because the solid–liquid interface protruded toward the solution, the grains grew along the normal direction perpendicular to the solid–liquid interface and the columnar grains increased in thickness during the growth process. Therefore, the columnar grains that grew in parallel in Figure 2 experienced competition for lateral space in the forward growth direction; in general, the growth and expansion of the coarse columnar grains prevailed, suppressing the coarsening of the fine columnar grains in the forward growth direction.

Figure 2 shows that the growth angles of steels A and B without Nb were 60° and 65°, respectively, and that those of steels C and D with Nb were 52° and 46°, respectively. The growth angle of columnar grains can directly affect the

probability of crack generation. During ingot processing, the stress of the material exceeds the strength of the material itself to cause cracks. The partial stress can be generally decomposed into the normal stress σ and the shear stress τ in the plane, and the magnitude of the two is directly related to their frictional force; importantly, the interface between the two columnar grains affects the stress. The friction angle (generation angle) is also a factor.

According to the Moore's theory, if a bulk material is broken along a certain plane under the action of a twodirection stress, there is a certain combination of normal stress σ and shear stress τ in this plane. The normal stress σ and the shear stress τ in the plane can be obtained by force balance:

$$\sigma = \sigma_1 \cos \theta + \sigma_3 \sin \theta \tag{1}$$

$$\tau = (\sigma_1 - \sigma_3)\cos\theta\sin\theta \qquad (2)$$

where σ_1 is the maximum principal stress, σ_3 is the minimum principal stress, and θ is the angle between the two columnar interfaces.

When $\theta = 45^{\circ}$, the values of the normal stress σ and the shear stress τ are minimal, which is the optimal solution of equation (2), and the material can withstand the maximum processing stress under the optimal solution. As shown in Figure 2, the growth angles of steels A and B without Nb were 60° and 65°, respectively. The growth angle of steel C with 0.05% Nb was 52°, and that of steel D with 0.08% Nb was 46°. The calculated theoretical values agree with the experimental results. The growth angle of the solidified structure is similar to the optimal theoretical value of 45° after Nb was added to the steel, which is beneficial for mitigating the damage caused by cracks, preventing further cracks and improving the quality of steel.



Figure 5: Variation of NbC element precipitation in the high-Mn steel as a function of temperature.



1200

T(K)

1400

1600

1800

Figure 6: Inclusion in the Fe-Mn-C-Al TWIP steel.

1000

800

3.3 Analysis of the grain refinement mechanism

Scanning electron microscopy (SEM) and the energy spectrum analysis show that most of the inclusions in the Fe–Mn–C–Al TWIP steel after the addition of Nb were composites containing Nb, N, Al, and other elements. Most of them were regular quadrilaterals with a size of approximately $2-3 \mu m$; their specific shape is shown in Figure 6.

3.3.1 Thermodynamics of Nb precipitation

Because the high-Mn steel contains microalloyed Nb, the carbide precipitation process of microalloyed elements influences the evolution of the solidification structure in the casting process [32]. Therefore, the dissolution and precipitation of carbides must be considered during the heating process of the sample. The solubility product formula of niobium carbide in austenite is as follows [18]:

$$\log [\text{Nb}][\text{C}] = 2.96 - \frac{7,150}{T}$$
(3)

The total solution temperature (T_{AS}) of the binary second-phase MX can be calculated by the following equation [18]:

$$T_{\rm AS} = \frac{B}{A - \log(M \cdot X)} \tag{4}$$

The chemical composition of the steel is substituted to calculate the initial precipitation temperature of the niobium carbide in the steel. The calculation process is as follows, and the calculation results are shown in Table 2:

High-manganese steel with different	Full solution			
niobium content	temperature <i>T</i> _{AS} (°C)			
Fe-Mn-C-Al-0.05Nb	1,425			
Fe-Mn-C-Al-0.08Nb	1,485			

$$T_{AS} = \frac{7,510}{2.96 - \log(0.69 \times 0.05)} = 1,698 \text{ K} = 1,425^{\circ}\text{C}$$
$$T_{AS} = \frac{7,510}{2.96 - \log(0.61 \times 0.08)} = 1,758 \text{ K} = 1,485^{\circ}\text{C}$$

In the experiment, the goal of heating the sample is to dissolve the alloying Nb. According to the aforementioned calculation, if the Nb element in the steel is completely in solid solution, the sample should be heated to 1,425°C. The calculation results show that the precipitation temperatures of Nb carbide in the two kinds of sample steels were 1,425°C and 1,485°C. At a high temperature, the precipitation at the original grain boundaries inhibited the grain growth. During the solidification and cooling of the high-Mn steel, the alloy element precipitates pinned the original austenite grains and the pinning effect mainly depended on the precipitation temperature and precipitation amount.

The precipitation form of the binary second-phase MX in steel is a combination of M atoms and X atoms. The formula for calculating the equilibrium solid solution of the binary second-phase MX in steel is as follows [18]:

$$\frac{\text{Nb}-[\text{Nb}]}{C-[C]} = \frac{A_{\text{Nb}}}{A_{\text{C}}}$$
(5)

where Nb is the content of Nb added, [Nb] is the content of Nb in the precipitate, *C* is the content of C added, [C] is the content of C in the precipitate, $A_{\rm Nb} = 92.9064$, and $A_{\rm C} = 12.011$. From equations (3) and (5), the amount of MX in equilibrium solid solution in the matrix at any temperature [M], [X], and the amount M–[M] in the precipitation phase MX, X–[X], can be obtained; the results are shown in Table 3.

From the calculation results in Figure 5, it can be seen that in the high-Mn steel with a Nb content of 0.05%, the Nb and C were completely dissolved at a high temperature of 1,700 K; in the high-Mn steel with 0.08% Nb, the complete solution temperature is 1,760 K. As evident in Table 3, the precipitation of Nb carbide increases with increasing the Nb content in steel at the same temperature. Therefore, the addition of Nb in the high-Mn steel not only increases the precipitation

Temperature (K)	Solid solution [Nb] %	Precipitation Nb%	Solid solution [C]%	Precipitation C%	
(a) High-Mn steel wit	h 0.05% Nb content				
1,700	0.050	0	0.6901	0	
1,600	0.0269	0.0231	0.6870	0.0030	
1,500	0.0131	0.0369	0.6852	0.0048	
1,400	0.0058	0.0442	0.6843	0.0057	
1,300	0.0022	0.0478	0.6838	0.0062	
1,200	0.0007	0.0493	0.6836	0.0064	
1,100	0.0002	0.0495	0.6836	0.0064	
1,000	0	0.05	0.6835	0.0065	
900	0	0.05	0.6835	0.0065	
800	0	0.05	0.6835	0.0065	
(b) High-Mn steel wit	h 0.08% Nb content				
1,760	0.0800	0	0.6101	0	
1,700	0.0574	0.0226	0.6070	0.0029	
1,600	0.0306	0.0494	0.6036	0.0063	
1,500	0.0149	0.0651	0.6015	0.0084	
1,400	0.0066	0.0734	0.6005	0.0094	
1,300	0.0025	0.0775	0.5999	0.0100	
1,200	0.0008	0.0792	0.5997	0.0102	
1,100	0.0002	0.0798	0.5996	0.0103	
1,000	0	0.0800	0.5996	0.0103	
900	0	0.0800	0.5996	0.0103	
800	0	0.0800	0.5996	0.0103	

Table 3: Calculation of NbC precipitation at different temperatures

temperature of niobium carbide but also increases the precipitation of niobium carbide inclusions at the same temperature, both of which further influence the precipitation. The pinning action of the austenite grain boundaries can thereby enhance the refinement of the crystalline grains (Table 4).

3.3.2 Theoretical analysis of the carbides induced by the carbides

The nonhomogeneous nucleation theory by Turnbull and Vonnegut [33] holds that the high-melting-point phase in the liquid phase can provide nonhomogeneous nucleation interface for metals. When the mismatch between the high-melting-point phase in steel and some crystal planes of the matrix is low, the nonhomogeneous nucleation interface energy is low; thus, the solidified grains are easily nucleated and refined. The mismatch formula proposed by Bramfitt [34] can predict whether nucleation can occur in the matrix. When the mismatch of the two phases is greater than 12%, nucleation is impossible. When the mismatch of two phases is less than 12% and greater than 6%, nucleation can be achieved but it cannot be used as a nucleation core. When the mismatch is less than 6%, the nucleation

Table 4: Calculation results of lattice mismatch of inclusions

Parallel crystal face	[<i>uvw</i>] _s	[<i>uvw</i>] _n	d[uvw] _s	d[uvw] _n	θ , deg	Mismatch (%)
(100)NbC//(100)γ-Fe	[011] _{NbC}	[010] _{v-Fe}	3.708	3.73	0	4.27
	[010] _{NbC}	[011] _{v-Fe}	5.244	5.28	0	
	[001] _{NbC}	[001] _{γ-Fe}	3.708	3.73	0	
(110)NbC//(110)γ-Fe	[100] _{NbC}	[Ī10] _{γ-Fe}	3.708	5.28	0	14.3
	[112] _{NbC}	[1 12] _{γ-Fe}	6.423	9.134	0	
	[001] _{NbC}	[001] _{γ-Fe}	5.244	7.460	0	
(111)NbC//(111)γ-Fe	[110] _{NbC}	[Ī10] _{γ-Fe}	3.708	5.280	0	23.6
	[211] _{NbC}	[2 11] _{γ-Fe}	6.423	9.134	0	
	[101] _{NbC}	$[\bar{1}01]_{\gamma-Fe}$	3.708	5.280	0	

effect is good. In this study, the mismatch between the face-centered cubic system of the Fe–Mn–C–Al TWIP steel matrix and the face-centered cubic system of NbC was calculated as follows:

$$\delta_{(hkl)_n}^{(hkl)_s} = \sum_{i=1}^{3} \left[\frac{|d_{[uvw]_s}^i \cos \theta - d_{[uvw]_n}^i|}{d_{[uvw]_n}^i} \right] \times \frac{1}{3} \times 100\%$$
(6)

The research shows that the mismatch of the (100) plane in the Fe-Mn-C-Al TWIP steel matrix and the (100) plane in NbC is 4.27%. The mismatch of the (110) plane in the Fe-Mn-C-Al TWIP steel matrix and the (110) plane in NbC is 14.3%. Finally, the mismatch of the (111) plane in the Fe-Mn-C-Al TWIP steel matrix and the (111) plane of NbC is 23.6%, which means that nucleation on the (110) plane and the (111) plane is impossible, whereas nucleation on the (100) plane is easy. The nucleation direction then becomes the priority growth direction. NbC has a higher melting point than the matrix and a lower mismatch with it; thus, NbC can serve as an effective heterogeneous nucleation core that promotes nucleation to refine the solidified structure. Yao et al. [35] concluded that increasing the inhomogeneous nucleation rate can reduce the grain size when the grain growth rate is constant; that is, increasing the inhomogeneous nucleation rate can reduce the grain size when other conditions remain unchanged. The casting temperature and solidification conditions of steels A, B, C, and D were the same as those in the present study; i.e., the growth rate of the grains was the same, but after Nb was added, NbC inclusions formed in the steel. That is, a high-melting-point phase was formed in the melt, which increased the number of nucleation cores, increased the nucleation rate, and refined the solidification structure of the alloy.

4 Conclusion

- (1) The solidification structure of Fe–Mn–C–Al TWIP steels without Nb addition has large grains whose columnar and equiaxed lengths are approximately 3.5 and 2.4 mm, respectively. After Nb addition, the grain size of the solidified structure was refined. The width of the columnar grains was refined from 1.84 mm to less than 1.06 mm, and the size of the equiaxed grains was reduced from 2.85 mm to less than 0.96 mm, which is approximately 66.32%.
- (2) The growth direction of columnar and equiaxed grains is affected by Nb addition. When Nb is not added, the growth angle of the high-Mn steel is approximately 60°.

When Nb is added, the growth angle of the high-Mn steel decreases to approximately 45°, which is conducive to reducing the generation of cracks and improving the quality of the steel.

(3) The high-melting-point NbC and other second-phase particles formed by adding Nb into Fe–Mn–C–Al TWIP steels are beneficial to grain refinement, and a large number of Nb precipitates is beneficial to improving the equiaxed grain rate.

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