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Precipitates in the Casting Microstructure of Nb-containing Silicon Steel

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Abstract. The as-cast microstructure of niobium-containing slab was observed and characterized by metallographic microscope and scanning electron microscopy. The results show that the as-cast microstructure of the niobium-containing silicon steel slab is ferrite and a small amount of pearlite. There is no obvious difference in the microstructure of the niobium-containing silicon steel slab, and the as-cast (Cu, Mn) S, in addition to niobium carbon, nitride precipitation, AlN mainly strip and square, sulfide and niobium carbon, nitride was mainly Spherical or ellipsoidal. The number of precipitates in the slab is small but larger in size, and the size of precipitates is in the range of 100 nm to 500 nm.

Introduction

In the preparation process of oriented silicon steel, the final purpose is to obtain {110} <001> texture (goss texture) which causes the excellent magnetic properties and low iron loss [1-6]. The high component of Goss texture is the key factors to obtain the high magnetic and high stability of the finished product [7,8]. In the process of preparing the Goss texture by the secondary recrystallization method, the metastable second metastable phase in the matrix plays a key role, and these precipitates will effectively inhibit the growth of non-Goss orientation grains during the secondary recrystallization annealing process, And thus improve the composition of Goss grains, usually we will define these precipitates as inhibitors. These small dispersion inhibitors are through the slab heating and heating process, so that when the slag solidification of the second phase is completely dissolved in the matrix, and then in the hot rolling process and the process of re-dispersion of small particles to precipitate, in order to In the decarburization annealing and high temperature annealing play a role, so the as-cast organization will directly affect the subsequent production process. The feasibility of niobium carbonitride (Nb (C, N), NbC, NbN) as a low temperature oriented silicon steel inhibitor has been initially demonstrated [9]. Nb (C, N) has a low solution temperature in steel, enabling lower slab heating technology.

In this paper, the as-cast microstructures of niobium-containing slabs were observed and characterized by metallographic microscope and scanning electron microscopy. The distribution of microstructure of Nb-containing silicon steel and the types of microstructures were analyzed. The second phase morphology, distribution, size, and combined with energy spectrum analysis to determine the composition of precipitates in the as-cast structure.

Experimental Materials and Methods

The niobium-containing silicon steel slab was prepared from a 50 kg intermediate frequency vacuum induction melting furnace. The slab cooling method is water cooling. The slab size is: $150 \text{mm} \times 150 \text{mm} \times 260 \text{mm}$. Its composition is shown in Table 1.

Table 1 Chemical composition of slab (mass fraction, %)

| С | Mn | Si | S | Cu | Al | Nb | N | P |
|-------|------|-----|-------|-------|-------|-------|--------|-------|
| 0.042 | 0.08 | 3.2 | 0.016 | 0.085 | 0.018 | 0.045 | 0.0085 | 0.006 |

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The samples used for the observation of the low magnification were sampled on a slab with a band saw at a size of 75 mm x 50 mm x 10 mm. Samples were taken from different parts of the low magnification specimen using a wire cutter for observing the metallographic Organization, sample size is $15\text{mm} \times 10\text{mm} \times 10\text{mm}$. The samples were subjected to mechanical polishing from the low head to the high mesh on the sandpaper, and the sample was mechanically polished on the polishing machine. After the corrosion of the sample with 4% nitric acid, the low magnification structure of the sample was observed.

The metallographic structure was observed under the Zeiss Axiovert-200MAT optical microscope. The precipitates were observed on FEI's Quanta FEG 650 scanning electron microscope.

The billet microstructure specimen was processed into a $10\text{mm} \times 10\text{mm} \times 0.6\text{mm}$ sheet with a spark wire cutting machine. The sample was polished on a sandpaper to a thickness of about $20\mu\text{m}$. Diameter of $\Phi 3\text{mm}$ small disc. And then washed the small wafer at -30°C temperature double spray thinning, the voltage is 50V, electrolytic polishing solution by volume ratio of 1:19 perchloric acid and ethanol solution prepared by mixing. The obtained transmission sample was measured under JEM-2011 type transmission electron microscope.

Microstructure of Nb-containing Silicon Steel Slab

The low magnification of the oriented silicon steel slab is shown in Fig. 1. There is the order of the outer surface, the middle layer and the heart from left to right. It can be seen from Fig. 1 that the grains of the outer layer are smaller and the grains of the middle layer begin to grow larger and the grains of the core are smaller. However, there is no significant grain size difference in the billet structure, and there is no fine grain and columnar grains, and all of the billet is equiaxed.



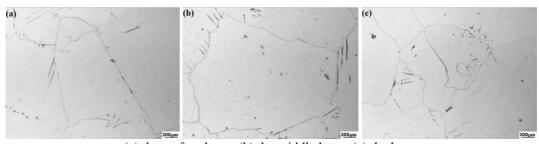
Fig.1 Low magnification of slab

This is because the cooling of this slab is water-cooled. In the early solidification, near the casting wall of liquid metal cooling speed, the nucleation rate is large, resulting in a smaller grain. With the solidification of the progress of the wall began to rise, the cooling rate began to slow down, nucleation speed is also reduced, the nucleus after the formation of dendrites to grow up, the formation of grain is also larger. After the formation of the intermediate layer, the temperature of the liquid metal in the central part of the slab dropped below the melting point to meet the requirement of nucleation and sub cooling. At this time, the remaining liquid was nucleated at the same time, and the number of nuclei was large, The In this solidification process of slab, the degree of undercooling is larger and the number of nucleation is more. After the nuclei are formed, they will grow freely in all directions, and they will meet each other and form an equiaxed crystal.

The microstructure of the microstructure of the niobium-containing silicon steel slab is shown in Fig.2. It can be seen from the figure that the matrix of the slab structure is ferrite, and there is a very small amount of black precipitates on the grain boundaries of the partial grains and grows inside the grains along the ferrite grains.

The experimental carbon content Wc= 0.042%, slab solidification process, will first form austenite, with the solidification of the austenite will be converted into ferrite, when the ferrite in the carbon saturation After the grain boundary will leave a small amount of precipitates.





(a) the surface layer; (b) the middle layer; (c) the heart Fig.2 OM images of slab of Nb-containing silicon steel

The precipitates at the grain boundary of the niobium-containing silicon steel slab can be seen in Fig.3. It can be clearly seen from Fig. 3 (f) that the black precipitate at the grain boundary in Fig. 2 is pearlite and there is no low temperature bainite or martensite in the tissue.

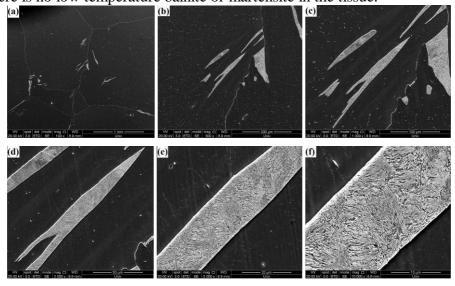


Fig.3 SEM images of slab of Nb-containing silicon steel

Precipitates in Slab of Nb-containing Silicon Steel

The precipitation phase of the niobium-containing silicon steel slab was observed by JEM-2011 transmission electron microscopy and analyzed by energy spectrum. The morphology and energy spectrum of the precipitates observed under transmission electron microscopy are shown in Fig.4.

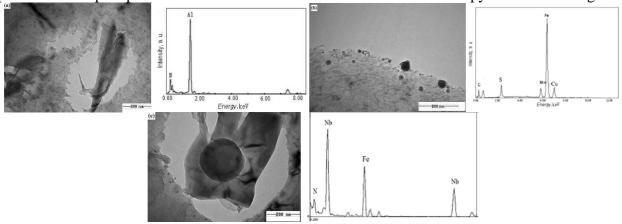


Fig.4 TEM images and EDS spectra of precipitates in sample

It can be seen from Fig. 4 that the second phase precipitated during the solidification of the niobium-containing silicon steel slab is Nb (N, C), AlN, (Cu, Mn) S, etc., and the precipitates are about 100 nm to 500 nm. The morphology of AlN is mainly stripe or square, and the morphology of (Cu, Mn) S and Nb (N, C) is spherical or ellipsoidal. Because these precipitates are precipitated directly in the solidification of the slab, the size is large and the number is small, so it can not play the role of the



inhibitor. It needs to be completely dissolved in the matrix in the subsequent slab heating process, And then in hot rolling or normalization to disperse fine particles precipitation.

Conclusions

The as-cast microstructure of the niobium-containing silicon steel slab is ferrite and a small amount of pearlite. There is no obvious difference in the microstructure of the niobium-containing silicon steel slab, and the as-cast (Cu, Mn) S, in addition to niobium carbon, nitride precipitation, AlN mainly strip and square, sulfide and niobium carbon, nitride was mainly Spherical or ellipsoidal. The number of precipitates in the slab is small but larger in size, and the size of precipitates is in the range of 100 nm to 500 nm.

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References

- [1] Yan M Q, Yang P, Jiang Q W, Fu Y J, Mao W M. Influence of rolling direction on thetexture evolution of fe-3%si alloy[J]. Acta Metallurgica Sinica, 2011(1):25-33.
- [2] Hu H. The formation of (110)[001] recrystallization texture in a cold-rolled and annealed (110) [001] crystal of silicon-iron[J]. Acta Metallurgica, 1960, 8(2):124-126.
- [3] Ushioda K, Hutchinson W B. Role of shear bands in annealing texture formation in 3%Si-Fe(111)[112] single crystals.[J]. Transactions of the Iron & Steel Institute of Japan, 1989, 29(10):862-867.
- [4] A. Böttcher, K. Lücke. Influence of subsurface layers on texture and microstructure development in RGO electrical steel[J]. Acta Metallurgica Et Materialia, 1993, 41(41):2503-2514.
- [5] Mishra S, Därmann C, Lücke K. New information on texture development in regular and high-permeability grain-oriented silicon steels[J]. Metallurgical Transactions A, 1986, 17(8):1301-1312.
- [6] Wang R P, Li S D, Fang Z M, etal. Microstructure and precipitate of low temperature hot rolled HGO silicon steel plate by normalizing [J]. Heat Treatment of Metals, 2009, 34(6):9-14.
- [7] GOSS N P. Electrical sheet and method and apparatus for its manufacture and test: U.S. Patent 1965559[P]. 1934-7-3.
- [8] TAGUCHI S, SAKAKURA A, MATSUMOTO F, et al. The development of grain-oriented silicon steel with high permeability[J]. Journal of Magnetism and Magnetic Materials, 1976, 2(1): 121-131.
- [9] Zhang Y, Fu Y L, Wang R W, et al. Feasibility of Nb(C,N)as Inhibitors in Oriented Silicon Steel[J]. China Metallurgy, 2008, 18(7):14-18.