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Formation of columnar-grained structures in directionally solidified Fe-6.5wt.%Si alloy

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ABSTRACT

Fe-6.5wt.%Si high silicon steel has superior magnetic properties such as high permeability and low core loss. However, such high Si content results in room-temperature embrittlement and poor workability due to the formation of the ordered intermetallic phases in the Fe-6.5wt.%Si alloy. The thin sheet production of this alloy with high silicon contents by mechanical deformation is very difficult due to its extreme brittleness. Zone Melting Liquid Metal Cooling (ZMLMC) directional solidification technique was employed to produce the Fe-6.5wt.%Si alloy with columnar-grained structures.

1. Introduction

High silicon steels (Si content > 4.5wt.%) have excellent soft magnetic properties compared with conventional Si steels (Si content ≤ 3.5wt.%). When the Si content reaches 6.5wt.%, the alloy has the best magnetic properties due to its high electrical resistivity, high permeability, low magnetocrystalline anisotropy and near-zero magnetostriction, which results in low core loss and low device noises [1–3]. It has a good perspective to apply this alloy in magnetic devices at high frequencies when it is fabricated in sheets.

When the Si content increases the material becomes brittle and loses its ductility due to the formation of intermetallic phases. It is very hard to produce Fe-6.5wt.%Si alloy sheet by conventional cold rolling method. To avoid the rolling problem, several methods have been developed to produce the alloy sheet, such as hot dipping and diffusion [4], chemical vapor deposition (CVD) [5,6], rapid quenching [7], etc.

Watanabe [8] found that the ductility of fully annealed Fe-6.5wt.%Si polycrystalline ribbons is improved because large amount of special grain boundaries with strong resistance to intergranular fracture are formed. Kim found that small amount of boron could decrease the grain size of the ingot and reinforce the grain boundary cohesion [9]. In this paper, Zone Melting Liquid Metal Cooling (ZMLMC) directional solidification technique was employed to produce the Fe-6.5wt.%Si alloy with columnar-grained structures and special grain boundaries. Small amount of Boron was doped to enhance the grain boundary cohesion.

2. Experimental

Bridgman ZMLMC directional solidification equipment was employed in this work (Fig. 1). It mainly consists of vacuum induction melting system, argon protection system, cooling system and pulling system. Vacuum is 5×10^{-3} Pa. Ga–In–Sn alloy was used as cooling liquid. Master alloy ingot was arc melted from pure elements and chemical composition is listed in Table 1.

φ7 mm × 100 mm rod was prepared by spark cutting from the ingot, and ultra pure alumina tube was used for holding the rod in the ZMLMC. The rod is induction heated for 5 min at the given temperature, before the sample began to be pulled downward at a constant withdrawal speed. The withdrawal speed varied from 5 to 120 μm/s, and the power of the melting system varied from 11 to 13 kW. Microstructure evolution of the directionally solidified alloy was observed using optical microscope after chemical etching with 8% nitric acid aqueous solution.

3. Results and discussion

Directional solidification is a crystal growth technique in which temperature gradient (G) and crystal growth velocity (V) can be

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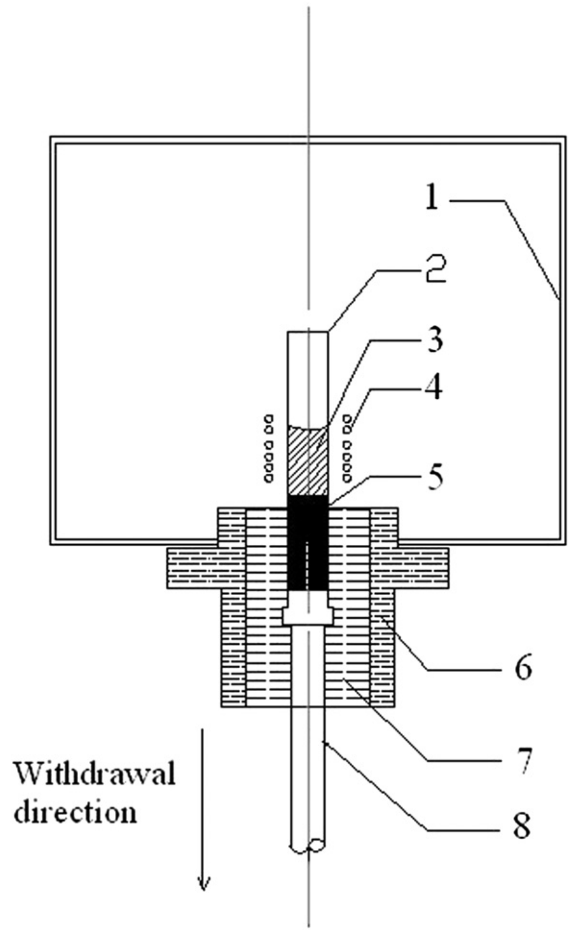

Fig. 1. Schematic of the Bridgman ZMLMC directional solidification equipment: 1. Furnace wall, 2. High purity Alumina tube, 3. Melted metal 4. Induction coils, 5. Solidified metal, 6. Water-cooled chamber, 7. Ga—In—Sn liquid coolant, 8. Pulling device.

Table 1
Chemical composition of high silicon steel (wt.%).

C	Si	Mn	S	P	B	Al	Fe
0.02	6.52	0.14	0.0036	0.013	0.057	0.02%	Bal

controlled independently. In ZMLMC, *G* is controlled by the power of the heating device, and *V* is controlled by the pulling system. The effect of *G* and *V* on the microstructure characteristics was investigated respectively.

Fig. 2 shows the microstructure evolution of the directionally solidified Fe-6.5wt.%Si alloy with *G* varying from 933 to 980 K/cm and constant $V = 8$ μm/s. One can see that the grain size is more than 2 mm in width at $G = 933$ K/cm, and it becomes 300 μm while the gradient is increased to 980 K/cm.

The effect of *V* on the microstructure of the directionally solidified Fe-6.5wt.%Si alloy is shown in Fig. 3. The growth velocity varied from 10 to 60 μm/s with constant $G = 980$ K/cm. One can find that the crystals are all solidified directionally into columnar-grained structure. When the growth velocity increases from 10 to 60 μm/s, the grains become thinner. The width of the grain decreased from about 600 μm to about 180 μm.

When the growth velocity becomes 60 μm/s, the width of the grain varied from 300 to 100 μm with *G* changing from 933 to 1010 K/cm (Fig. 4).

When solidification occurs in alloy melt, dendritic structure is often formed. Solidification interface morphologies, especially the primary dendrite arm spacing, secondary dendrite arm spacing, dendrite tip radius, and mushy zone depth, are close related with the solidification conditions. Most experimental studies have shown that the microstructural parameters decrease as solidification parameters (*G*, *V*) increase [10]. The product $G \times V$ stands for the cooling rate of the solidification. Hence high value of $G \times V$ is critical to maintain a fine structure.

Table 2 summarizes various crystal dimensions obtained in directionally solidified Fe-6.5wt%Si alloy. It can be seen that the cooling rate *GV* plays an important role to form columnar grain.

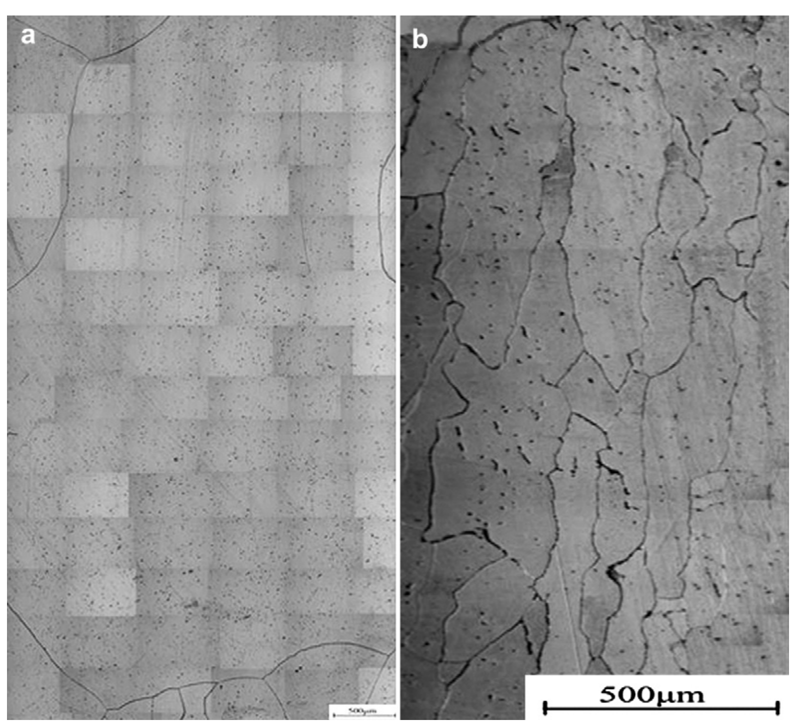

Fig. 2. Microstructure evolution of directionally solidified Fe-6.5wt.%Si alloy at constant growth rate of 8 μm/s but different temperature gradient: (a) 933 K/cm, (b) 980 K/cm.