ELSEVIER

CrossMark

Available online at www.sciencedirect.com

ScienceDirect

Procedia Engineering 81 (2014) 149 – 154

Procedia Engineering

www.elsevier.com/locate/procedia

11th International Conference on Technology of Plasticity, ICTP 2014, 19-24 October 2014, Nagoya Congress Center, Nagoya, Japan

High efficiency warm-cold rolling technology of Fe-6.5wt%Si alloy sheets

Jianxin Xie*, Hongjiang Pan, Huadong Fu, Zhihao Zhang

Institute for Advanced Materials and Technology, University of Science and Technology Beijing, Beijing 100083, China

---

Abstract

Compared with the common electrical steel, the high silicon electrical steel (Fe-6.5 wt%Si alloy) exhibits excellent soft magnetic properties and a wide application prospect in high frequency electromagnetic fields. However, the inadequate ductility and limited formability are the two well-known bottlenecks that severely limit the widespread engineering application of Fe-6.5 wt%Si alloy. In this study, composite slabs with columnar-grained Fe-6.5 wt%Si alloy at the inner part and pure iron at double sides were prepared by the Bridgman zone melting technology under the melting temperature of 1485±5 °C and withdrawing velocity of 1mm/min. The composite slab could be directly warm-cold rolled to prepare sheet with the thickness of 0.2 mm and slight edge cracks. A new technical prototype for producing Fe-6.5 wt%Si alloy sheets was proposed with high efficiency and compact process.

Selection and peer-review under responsibility of the Department of Materials Science and Engineering, Nagoya University

Keywords: Fe-6.5wt%Si alloy; Cold-rolled sheets; Recrystallization annealing; Texture

---

1. Introduction

Compared with the common electrical steel, 6.5wt% high silicon electrical steel (Fe-6.5wt%Si alloy) exhibits excellent soft magnetic properties, which has a wide application prospect in high frequency fields (Tanaka et al., 1990; Arai et al., 1994; Phway et al., 2008). However, the ductility of the Fe-6.5wt%Si alloy at room temperature

---

* Corresponding author: Tel.: +86 10 62332254; fax: +86 10 62332253.
E-mail address: jxxie@mater.ustb.edu.cn

Selection and peer-review under responsibility of the Department of Materials Science and Engineering, Nagoya University
doi:10.1016/j.proeng.2014.09.142

is nearly zero due to the high Si content, which makes it difficult to fabricate Fe-6.5wt%Si alloy sheets using conventional casting-rolling process (Bolfarini et al., 2008).

For avoiding the intrinsic embrittlement of the alloy, several special methods, such as physical vapor deposition method (Tian et al., 2010), rapidly quenched method (Varga et al., 2001), direct powder rolling method (Yuan et al., 2008), spray forming (Okada et al., 1996), etc. have been developed to produce the Fe-6.5 wt%Si alloys sheets. However, these methods are still in the stage of laboratory research due to their high production cost. Chemical vapor deposition (CVD) method (Yamaji et al., 1994), the unique commercial method to produce Fe-6.5 wt%Si alloy sheets was invented by NKK Corporation (JFE Corporation now). However, the process of the CVD method is complicated, including preparing the common electrical steels, deposition and siliconization.

Considering the simple process, low energy consumption and low cost, special rolling process has become one of the main development directions of manufacturing the Fe-6.5 wt%Si alloy. Liang et al. (2010, 2012) fabricated the Fe-6.5wt%Si alloy sheets with the thickness of 0.05-0.3 mm successfully through hot rolling combined with warm and cold rolling as well as multi-passes intermediate annealing. However, the serious edge cracks and very low rolling yield need to be further improved. Liu et al (2013, 2014) put forward a new idea of preparing Fe-6.5 wt%Si alloy sheets by strip casting, hot rolling and warm rolling. However, the surface quality and the stamping performance of the alloy sheets need to be further enhanced.

The authors' previous studies (Xie et al., 2012; Fu et al., 2013) indicated that the plastic formability of the Fe-6.5 wt%Si alloy can be improved by controlling the microstructure and grain orientation by directional solidification, precipitates and the order degree control by appropriate heat treatment. Based on the previous works mentioned above, a new technology for producing Fe-6.5 wt%Si alloy sheets was proposed for highly efficient and compact fabrication of the Fe-6.5 wt%Si alloy sheets. However, many edge cracks were observed frequently after severe rolling deformation by this method.

Therefore, a new directional solidification technology of composite material was developed (Fu et al., 2013). The composite slab with intensive <100> orientation columnar-grained Fe-6.5 wt%Si alloy at the inner part and pure iron at double sides was prepared to improve the warm-cold rolling performance and control the edge cracks. After the warm-cold rolling of the composite slab, the Fe-6.5 wt%Si alloy sheets could be produced by scraping edge or homogenization heat treatment. The result of this study is expected to provide a new process for highly efficient and compact fabrication of the Fe-6.5 wt%Si alloy sheets.

2. Materials and methods

Taking pure iron (99.9 wt%), pure silicon (99.9 wt%) and Fe-B alloy (B: 17.5 wt%, Fe: 82.45wt%) as the raw materials, an Fe-6.5 wt%Si alloy with 0.02 wt%B was firstly melted in a vacuum induction furnace and then mold casted. The rods for directional solidification were cut from the as-cast ingot by electric discharge machining with the size of 48 mm×6 mm×100 mm. After being polished, cleaned and dried, the rods were used to directional solidification.

The composite slab was fabricated by directional solidification through an improved Bridgman zone melting equipment. The casting mould was made up by two pieces of quartz plates (thickness: 1.5 mm, width: 56 mm, length: 120 mm), two pieces of pure iron (thickness: 2 mm, width: 6 mm, length: 120 mm) and some refractory. The size of the casting mould lumen was 50×6×120 mm. The size of the composite slab after directional solidification would be 6 mm in thickness, 54 mm in width and 120 mm in length. Argon was blown to the casting mould to reduce the oxidation during directional solidification. The schematic diagrams of casting mould and directional solidification are shown in Fig. 1.

The specimens produced by directional solidification were polished and etched with a solution of 7% HNO_3 and 93% CH_3COOH for 2 minutes at room temperature. The composition and Vickers hardness at the interface of the composite slab were detected by energy disperse spectroscopy of ZEISSEVO18 scanning electron microscope and

HXD-1000T microhardness tester, respectively. The micro-texture and the magnetic properties of the cold-rolled sheets after recrystallization annealing were measured by D5000 X-ray diffractometer and single sheet magnetic properties tester.

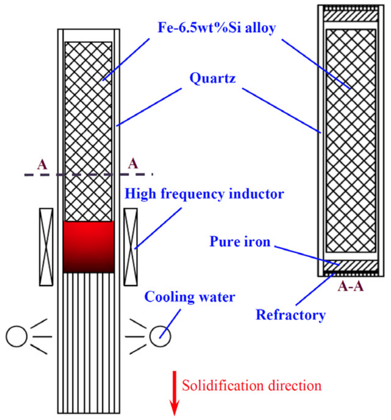

Fig. 1. Schematic diagrams of directional solidification and casting mold (thickness of pure iron of 2 mm).

3. Results and discussion

3.1. Effect of solidification parameters on microstructure of the composite slab

The effect of melt zone temperature and withdrawing velocity on microstructure of the pure-iron-cladded columnar-grained Fe-6.5wt%Si alloy composite slab was mainly investigated. According to previous studies, the melting zone temperatures and withdrawing velocities of the experiment were 1455±5, 1485±5 and 1515±5 °C and 1, 2 and 3 mm/min, respectively.

3.1.1. Effect of melting zone temperature on microstructure of the composite slab

The effect of melting zone temperature on the interface between pure iron and Fe-6.5 wt%Si alloy was investigated at the withdrawing velocity of 1 mm/min. When the temperature is 1455±5 °C, an obvious gap is found between the pure iron at the side of the slab and Fe-6.5 wt%Si at the inner part. With the increasing of melting zone temperature to 1485±5 °C, the microstructure and composition distribution curve is shown in Fig. 2. No obvious gap is found between pure iron at the side and Fe-6.5 wt%Si alloy. Remarkable composition transition is found at the interface, which indicates a good metallurgical-bonded composite interface is formed. The distributions of Si and Fe element are shown as a gradient slope shape at the interface and the composition transition distance is 1.8 mm away from the edge with a width of 0. 3 mm.

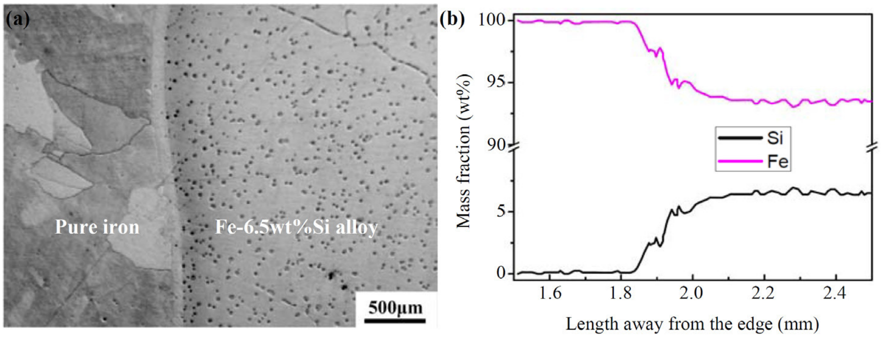

Fig. 2. (a) Microstructure and (b) composition distribution curve of interface between pure iron and Fe-6.5 wt%Si alloy with melting zone temperature of 1485±5 °C.

When the melting zone temperature reaches 1515±5 °C, the pure iron at the side is melted and the compositions of the edge and inner part are homogenous. Therefore, the slab fails to preventing and reducing edge cracks by the good ductility and workability of pure iron at the side.

3.1.2. Effect of withdrawing velocity on microstructure of the composite slab

Under the favorable interface bonding between pure iron and Fe-6.5 wt%Si alloy with the melting zone temperature of 1485±5 °C, the effect of withdrawing velocity (range of 1, 2 and 3 mm/min) on microstructure and grain orientation of the Fe-6.5wt%Si alloy at inner part were investigated in this study.

Fig.3 shows the macrostructure of the composite slab at different withdrawing velocities. When the withdrawing velocity is 1 mm/min, the columnar grains in the slab grow along the withdrawing direction, the grain boundaries are parallel to each other and the grain size is uniform. With the withdrawing velocity of 2mm/min, the axial direction of columnar grains in the slab deviates from the withdraw direction, and the grains become thinner than the former ones. With increasing withdrawing velocity to 3 mm/min, the growing direction of columnar grains in the slab has 30° deviation from the withdrawing direction and the macrostructure of the grains shows a shape like “^”.

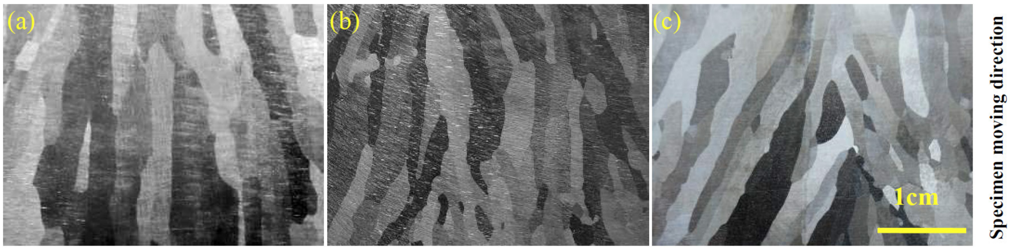

Fig. 3. Macrostructure of Fe-6.5 wt%Si alloy at inner part of slab at different withdrawing velocities for (a) 1 mm/min, (b) 2 mm/min and (c) 3 mm/min.

Thus it can be seen that the withdrawing velocity has a significant impact on macrostructure of the Fe-6.5 wt%Si alloy. With increasing withdrawing velocity, the deviation angle between the grain growing direction and withdrawing direction becomes larger because of the change of heat transfer condition during solidification. During the solidification of composite slab, the melting Fe-6.5 wt%Si alloy contacts with pure iron at two sides. Due to the high thermal conductivity of pure iron, the heat transfer increases remarkably at two sides with an increase in the withdrawing velocity. The solid-liquid interface of the composite slab curves from the shape of straight line to “U”, which leads to the increase of deviation angle between the grain growing direction and the withdrawing direction.

To sum up, the appropriate process of the directional solidification is the melting temperature of 1485±5 °C and withdrawing velocity of 1 mm/min.

3.2. Warm rolling and cold rolling of the composite slab

In order to show the effect of cladded pure iron on the edge cracks of the composite slab during warm/cold rolling, the directional solidification slab with the size of 54×6×100 mm was cut into two parts with single side pure iron cladded composite slab, and rolled at intermediate temperature. The warm and cold rolling were performed on the four-high rolling mill with the speed of 5-10 m/min. The specific process of the rolling is shown in Table 1.

Table 1. Rolling process of the single side pure iron cladded Fe-6.5wt%Si alloy composite slab.

Rolling temperature	Variation of thickness (mm)	Reduction of per pass (%)	Total reduction (%)	Situation of edge
550 °C	6.0→0.47	11.4~23.6	92.2	Without edge crack
Room temperature	0.47→0.35	14.9, 12.5	94.2	Without crack on side of pure iron, side of Fe-6.5wt%Si alloy crack slightly
Room temperature	0.35→0.30	14.3	95.0	Without crack on side of pure iron, the side of Fe-6.5wt%Si alloy crack seriously

The macrostructure of the single side pure iron cladded composite slab after warm-cold rolling is shown in Fig. 4. The edge cracks on the two sides of the slab are significant different. The side cladded by pure iron does not form edge cracks, while the naked side forms serious cracks with the depth of more than 1cm. This implies that the Fe-6.5 wt%Si alloy slab cladded by pure iron can effectively solve the edge crack problem of the Fe-6.5 wt%Si alloy during warm-cold rolling, which has a great significance in engineering.

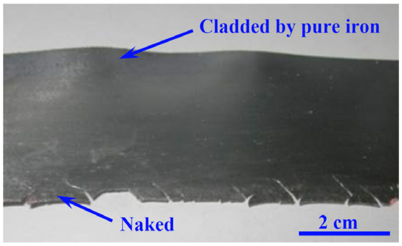

Fig. 4. Macrostructure of the single side pure iron cladded Fe-6.5wt%Si alloy composite slab after cold rolling (thickness: 0.3 mm).

Referring to the results of the single side pure iron cladded specimen, the double sides pure iron cladded Fe-6.5wt%Si alloy slab was still performed on the four-stand rolling mill with the rolling speed of 5~10m/min. Meanwhile, the reduction of per pass was controlled in the range of 10%~30%. The specific process of the warm-cold rolling is shown in Table 2.

After warm rolling to 0.5 mm by 12 passes and pickling the oxide skin, the slab was cold-rolled into the thickness of 0.2 mm with a total reduction of above 96%. As shown in Fig. 5, the cold-rolled sheet has a good shape with slight edge cracks, and can be curved.

Table 2. The rolling process of the double sides pure Fe cladded Fe-6.5wt%Si alloy composite slab.

Rolling temperature	Variation of thickness (mm)	Reduction of per pass (%)	Total reduction (%)	Situation of edge
550°C	6.0→0.48	12.0~28.4	92.0	Without edge crack
Room temperature	0.48→0.20	13.0~20.8	96.7	Edge crack slightly

Fig. 5. Cold-rolled sheet of the Fe-6.5 wt%Si alloy composite slab (thickness: 0.2 mm).

4 Conclusions

(1) The columnar-grained Fe-6.5 wt%Si alloy composite slabs cladded by pure iron at double sides were prepared by the zone melting technology through controlling the melting zone temperature and the withdrawing velocity. In the study, the appropriate process of the directional solidification is the melting temperature of 1485±5 °C and withdrawing velocity of 1 mm/min.

(2) The columnar-grained Fe-6.5 wt%Si alloy composite slab, cladded by pure iron with good plasticity on sides, can effectively solve the edge crack problem of the Fe-6.5wt%Si alloy during warm and cold rolling, which can improve the efficiency and rolling yield of the sheets significantly.

(3) A new technical prototype for producing Fe-6.5 wt%Si alloy sheets was proposed with high efficiency and compact process.

References

Arai, I., Ishiyama K., 1994. Recent developments of new soft magnetic materials. Journal of Magnetism and Magnetic Materials, 133, 233-237.

Bolfarini, C., Silva, A., Jorge, M., et al., 2008, Magnetic properties of spray-formed Fe-6.5%Si and Fe-6.5%Si-1.0%Al after rolling and heat treatment. Journal of Magnetism and Magnetic Materials, 320, e653-e656.

Boer, D., Wieting, J., 1997. Formation of a Near {001}<110> Recrystallization Texture in Electrical Steels. Scripta Materialia, 37, 753-760.

Fang, X., Liang, Y., Ye, F., et al., 2012. Cold rolled Fe-6.5wt.% Si alloy foils with high magnetic induction. Journal of Applied Physics, 111, 94913.

Fu, H., Zhang, Z., Pan, H., et al., 2013. Warm/cold rolling processes for producing Fe-6.5wt% Si electrical steel with columnar grains. International Journal of Minerals Metallurgy and Materials, 20, 535-540.

Fu, H., Zhang, Z., Xie, J., et al., date of application: 5-24-2013, A preparation method of composite slab with pure iron-clad columnar-grained high silicon electrical steel. Chinese patent: 201310198770.7.

Haiji, H., Okada, K., Hiratani, T., et al., 1996. Magnetic properties and workability of 6.5% Si steel sheet. Journal of Magnetism and Magnetic Materials, 160, 109-114.

Li, H., Liu, H., Liu, Z., et al., 2014. Characterization of microstructure, texture and magnetic properties in twin-roll casting high silicon non-oriented electrical steel. Materials Characterization, 88, 1-6.

Liang, Y., Lin, J., Ye, F., et al., 2010. Microstructure and mechanical properties of rapidly quenched Fe-6.5wt.% Si alloy. Journal of Alloys and Compounds, 504, 476-479.

Liu, H., Liu, Z., Sun, Y., et al., 2013. Development of λ-fiber recrystallization texture and magnetic property in Fe–6.5wt% Si thin sheet produced by sheet casting and warm rolling method. Materials Letters, 91, 150-153.

Okada, K., Yamaji, T., Kasai, K., 1996. Basic investigation of CVD method for manufacturing 6.5%Si steel sheet. ISIJ International, 36, 706-713.

Phway, P., Moses, J., 2008. Magnetostriction trend of non-oriented 6.5% Si-Fe. Journal of Magnetism and Magnetic Materials, 320, e611-e613.

Stojakovic, D., Doherty, D., Kalidindi, R., et al., 2008. Thermomechanical Processing for Recovery of Desired <001> Fiber Texture in Electric Motor Steels. Metallurgical and Materials Transactions A, 39, 1738-1746.

Tanaka, Y., Takada, Y., Abe, M., et al., 1990. Magnetic properties of 6.5% Si-Fe sheet and its applications. Journal of Magnetism and Magnetic Materials, 83, 375-376.

Tian, G., Bi, X., 2010. Study on the Si penetration into Fe sheets using PVD method and its application in the fabrication of Fe-6.5wt.% Si alloys. Surface and Coatings Technology, 204, 1295-1298.

Varga, K., Mazaleyrat, F., Kovac, J., et al., 2001. Magnetic properties of rapidly quenched Fe100-xSix (15<x<34) alloys. Materials Science and Engineering A, 304-306, 946-949.

Walter, L., Koch, F., 1963. Substructures and recrystallization of deformed (100)[001]-oriented crystals of high-purity silicon-iron. Acta Metallurgica, 11, 923-938.

Xie, J., Fu, H., Zhang, Z., et al., 2012. Deformation twinning feature and its effects on significant enhancement of tensile ductility in columnar-grained Fe–6.5wt.%Si alloy at intermediate temperatures. Intermetallics, 23, 20-26.

Yamaji, T., Abe, M., Takada, Y., et al., 1994. Magnetic properties and workability of 6.5% silicon steel sheet manufactured in continuous CVD siliconizing line. Journal of Magnetism and Magnetic Materials, 133, 187-189.

Yang, W., Mao, W., Sun, Z., 1998. Deformation and recrystallization texture in alloys based on Fe-16Al and Fe-28Al. The Chinese Journal of Nonferrous Metals, 8, 390-394.

Yuan, W., Li, J., Shen, Q., et al., 2008. A study on magnetic properties of high Si steel obtained through powder rolling processing. Journal of Magnetism and Magnetic Materials, 320, 76-80.