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극박 3%규소강에서 표면에너지 유기 선택적 결정성장 현상과 자성특성

(Surface-energy-induced Selective Growth and Magnetic Induction in 3%Si-Fe Strip)

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요 약

열간 압연판에 남아있던 {110}<001> 고스 집합조직으로부터 유기된 {111}<112> 냉간압연 집합조직은 기존 발표된 내용들과는 달리, 최종 재결정 집합조직인 {110}<001>고스 집합조직과는 상호 연관성이 없는 것으로 확인되었다. 최종 아닐링하는 동안 표면에 편석된 황의 농도에 따라 표면에너지 유기 선택적 결정성장 현상이 관찰되었다. 최종 압하율이 작아짐에 따라 초기 생성된 고스 결정립들은 다량의 황이 편석된 범위에서 {100} 또는 {111} 결정립들에 의해 완전히 잠식당하지 않고 살아 남아, 편석이 없는 후반 열처리 시간대에서 완전한 고스집합조직을 형성하는 선택적 결정성장의 기회를 갖게 됨에 따라, 최종적으로는 1.9 Tesla 이상의 우수한 자성 특성을 나타내는 극박 규소강 박대를 얻을수 있었다.

Abstract

The {111}<112> deformation texture, which originated from the {110}<001> texture near the surface of hot bands, is not prerequisite for the recrystallized {110}<001> Goss texture. During final annealing, surface-energy-induced selective growth of grains occurs at the strip surface of 3%Si-Fe alloys containing 6 ppm bulk content of sulfur. With decreasing final reduction, the probability that Goss grains survive under the highly segregated sulfur atmosphere and have a chance for later surface-energy-induced selective growth becomes higher, resulting in high magnetic induction.

1. INTRODUCTION

In 3%Si-Fe alloys, {110}<001> Goss texture is formed near the surface layer of hot bands when they are rolled in the α phase at elevated temperatures. The Goss texture, which is not stable with respect to plane strain deformation, rotates toward the {111}<112> orientation [1]. The recrystallized {110}<001> texture arises from the {111}<112> deformation texture. It was reported that the nucleation of {110}<001> Goss grains took place in the vicinity of the strip surface zone [2]. This phenomenon was attributed to the nucleation of Goss orientation in shear bands which occurred mainly in {111}<112> grains.

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On the other hand, the surface energy of the {110} in bcc crystal is less than the {100} and followed by the {111} and other less densely packed planes [3-5]. Adsorption of atoms from atmosphere onto solid-vapor surface varies the surface energy of planes in the free surface. Through an annealing experiment under hydrogen atmosphere with an additive of H2S [6], it has been proposed that the growth of {110} grains in 3%Si-Fe strips occurs under very clean surface condition and the growth of {100} grains under slightly sulfur-contaminated condition. If the surface is highly sulfur-contaminated, no surface-energy-induced selective growth occurs. Great attention has been paid to the study of segregation phenomenon, because interfacial segregation influences many mechanical and chemical properties of materials. Repulsion at surfaces or grain boundaries between Si and impurities (C, S) has been reported in 3%Si-Fe alloys [7]. Recently, it has experimentally been shown that the surface segregation of sulfur from bulk interior of 3%Si-Fe strips has a great effect on the surface energy and thus the surface-energy-induced selective growth kinetics of a grain [8,9]. The surface-energy-induced selective growth of a grain is determined by the concentration of surface-segregated sulfur during final annealing: from magnetically detrimental {111} grain to {100} and to {110} grain in the order of decreasing surface-segregated sulfur concentration.

In this study, effects of surface condition of hot bands or cold-rolled strips on evolution of initial recrystallization texture are reassessed in 3%Si-Fe alloys containing 6 ppm sulfur. Relationship between final reduction, interfacial segregation, and surface-energy-induced selective growth is investigated. Finally, effects of initial recrystallization texture on final texture and magnetic induction are also investigated.

2. EXPERIMENTAL PROCEDURE

3%Si-Fe strips, which contain only 6 ppm bulk content of sulfur without the addition of manganese or aluminum, were prepared through vacuum induction melting, hot- and two-stage cold-rolling processes. The surface of some hot bands or cold-rolled strips was chemically polished in order to investigate the effect of surface condition on initial texture evolution during annealing. The details in cold rolling process of the 6 ppm sulfur-contained alloy are given to Table 1.

Table 1. Surface conditions of hot bands and cold-rolled strips in the 6 ppm sulfur-contained alloy and the two-stage cold-rolling process.

Hot band surface (hot band thickness)	Cold-rolling reduction	Cold-rolled strip surface (final thickness)	Case
Un-removed (1[mm])	50%→60%	Removed (200[㎛]→100[㎛])	I
	75%→60%	Un-removed (100[㎛])	II
Removed (2[mm]→1[mm])	75%→60%	Un-removed (100[㎛])	III
Un-removed (2[mm])	93%	Un-removed (150[㎛])	IV

100[㎛] thick strips were also prepared through a triple-stage cold-rolling process. Final reduction of 60% was mostly given to the alloy. Between each cold rolling stage, an intermediate annealing was performed at 800℃ for 1.8 ks under a vacuum of 6 x 10^{-6} Torr. Most of final annealing was carried out at 1200℃ under the same condition. Texture was analyzed with ODF (orientation distribution function) and an etch-pit method. Surface segregation behavior on the strips was investigated with an ion-sputtering technique in an AES (Auger electron spectroscope) after final annealing and fast cooling. The primary beam energy was 2 keV. The differential peak heights were obtained every 15 seconds during ion-sputtering. The silicon, carbon and oxygen peaks around 92, 272 and 503 eV were chosen. In order to minimize the contamination effect from air, the peak height of S 150 eV were normalized with the peak height, 90, of Fe 703 eV that was obtained after ion sputtering for 4~8 min. Magnetic induction (B10, Tesla) was measured with a DC-fluxmeter under a magnetic field of 1000 A/m.

3. RESULTS AND DISCUSSION

Figure 1 shows changes in cold rolling texture with depth in Case I of Table 1. The deformation texture was changed from {110}<001> + {100}<011> + {111}<112> to strong {111}<112> with depth.

As shown in Fig. 2, all the initial texture after final vacuum annealing for 0.03 ks was, however,

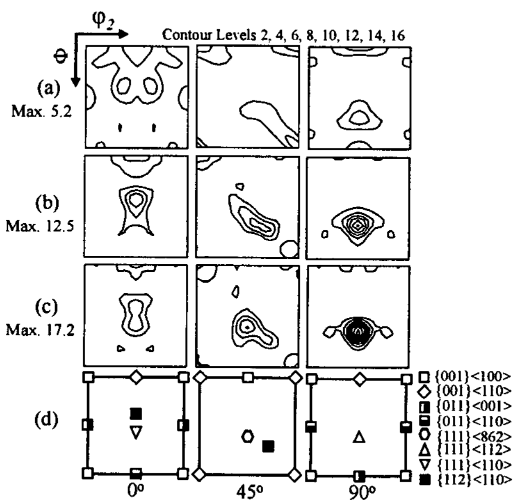

Fig. 1. Texture evolution in the 6 ppm sulfur-contained strips before final vacuum annealing at 1200℃ for 0.03 ks: (a) before chemical thinning (S=1.0), (b) 1/4 depth beneath the strip (S=0.5), (c) center of the strip (S=0.0) and (d) ideal orientations.

composed of strong {110}<001> Goss texture, irrespective of depth. Figure 3 shows ODF results of the strips (Cases II and III) after final vacuum annealing for 0.03 ks. Unlike other results [1], the strips showed strong recrystallized {110}<001> Goss texture, irrespective of the surface condition of hot bands. That is, the {111}<112> deformation texture, which originated from the {110}<001> texture near the surface of hot bands, is not prerequisite for the recrystallized {110}<001> Goss texture.

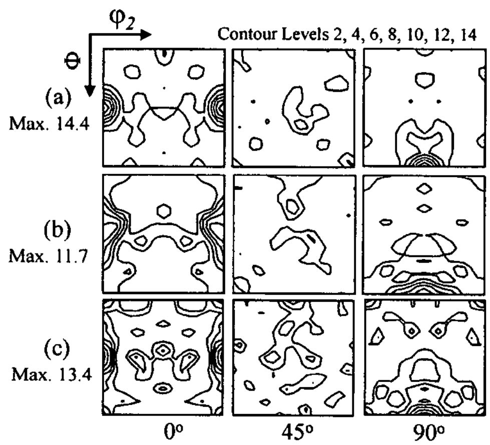

Fig. 2. Texture evolution in the 6 ppm sulfur-contained strips after final vacuum annealing at 1200℃ for 0.03 ks: (a) before chemical thinning (S=1.0), (b) 1/4 depth beneath the strip (S=0.5), (c) center of the strip (S=0.0).

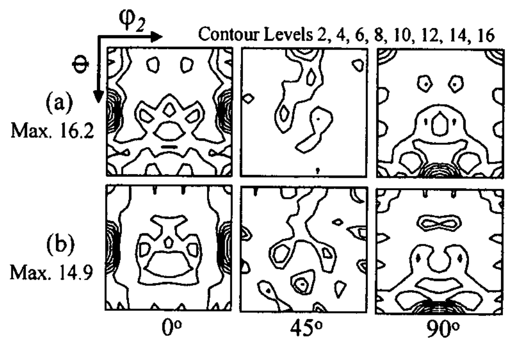

Fig. 3. The effect of surface condition of hot bands on texture of the 6 ppm sulfur-contained strips after final vacuum annealing 1200℃ for 0.03 ks: (a) un-removed surface and (b) removed surface.

On the other hand, the strip with final reduction of 93% (Case IV) showed strong {100}<011> deformation texture, as shown in Fig. 4.

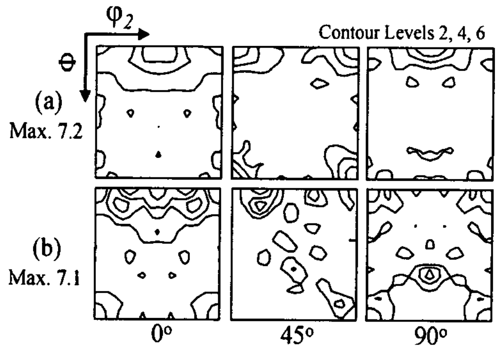

Fig. 4. ODFs in the 6 ppm sulfur-contained strip with final reduction of 93%: (a) before and (b) after final vacuum annealing at 1200℃ for 0.03 ks.

After final vacuum annealing for 0.03 ks, {111}<112> and weak {100}<001> recrystallization texture was only observed without Goss texture.

Figure 5 shows changes in surface-segregated concentration of sulfur and magnetic induction with final vacuum annealing time in the 6 ppm sulfur-contained strips (Case III). Through the surface-energy-induced selective growth of grains, the initial recrystallized {110}<001> Goss texture was replaced by α + γ fiber with increasing concentration of segregated sulfur. After the maximum of segregated sulfur, the recrystallization texture developed into strong {100}<011> and weak Goss texture with decreasing concentration of segregated sulfur. Within the segregated-sulfur-free range of annealing time, the weak Goss texture became again strong at the expense of other grains including {100}<011> ones. Complete {110}<001> Goss texture was obtained after final annealing for 7.2 ks. A trough in magnetic induction (B10,, Tesla) corresponded to the convex profile of sulfur arising from the evaporation of segregated sulfur. This is due to the formation of magnetically detrimental texture, i.e. {100}<uvw> and {111}<uvw>, under the condition of highly surface-segregated sulfur.

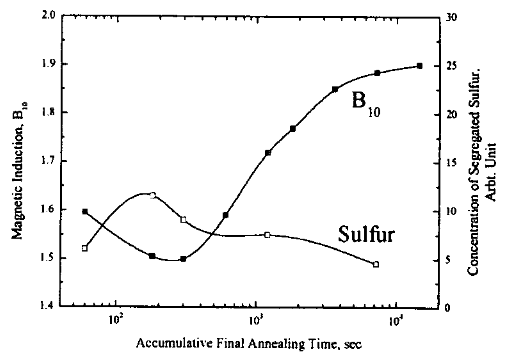

Fig. 5. Changes in surface-segregated concentration of sulfur and magnetic induction with final vacuum annealing time in the 6 ppm sulfur-contained strips (Case III).

The strip, in which Goss texture is initially observed, showed final magnetic induction higher than about 1.90 Tesla. A much lower magnetic induction of 1.65 Tesla was obtained in the other strip with final reduction of 93%, resulting from mostly {100}<011> grains without exception.

4. CONCLUSIONS

Initial texture evolution during final annealing and its effect on final texture have been investigated in 3%Si-Fe alloy strips containing 6 ppm bulk content of sulfur. The {111}<112> deformation texture, which originated from the {110}<001> texture near the surface of hot bands, is not prerequisite for the recrystallized {110}<001> Goss texture. Higher initial intensity of Goss texture corresponds to higher final magnetic induction. This is because, as the initial intensity of recrystallized {110}<001> Goss texture increases, the probability that Goss grains survive within the time range of highly segregated sulfur and have a chance for surface-energy-induced selective growth within the later sulfur-free time range becomes higher.

REFERENCES

[1] C. G. Dunn, "Cold-rolled and Primary Recrystallization Textures in Cold-rolled Single Crystalls of Silicon Iron", Acta Metall., 2, pp. 173, 1954.

[2] Y. Inokuti, C. Maeda, Y. Itoh and H. Shimanaka, ICOTOM 6, Tokyo, The Iron and Steel Institute of Japan, pp. 948, 1981.

[3] C. Herring, Structure and Properties of Solid Surfaces, R. Gomer and C. S. Smith, eds., Univ. of Chicage Press, Chicage, IL, pp. 5, 1953.

[4] H. J. Leamy, G. H. Gilmer and K. A. Jackson, Surface Physics of Metals, vol. I, J. M. Blakely, ed., Academic Press, New York, NY 121, 1975.

[5] J. M. MacLaren, J. G. Pendry, P. J. Rous, D. K. Saldin, G. A. Somorjai, M. A. Van Hoven and D. D. Vvedensky, Surface Crystallographic Information Service, eds., D. Reidel Publishing Co., Boston, MA, 1987.

[6] D. Köhler, "Promotion of Cubic Grain Growth in 3% Silicon Iron by Control of Annealing Atmosphere Composition", J. Appl. Phys., 31, pp. 408, 1960.

[7] P. Lejcek and S. Hofmann, "Segregation Enthalphies of Phosphorus, Carbon and Silicon at {012} Symmetrical Tilt Grain Boundaries in an Fe-3.5at.% Si Alloy", Acta Metall., 1991, 39, 2469.

[8] N. H. Heo, et al., "Effect of Surface Segregation of Sulfur on Recrystallization Kinetics in 3% Si-Fe Alloy Strip", J. Appl. Phys., 85, pp. 6025, 1999.

[9] N. H. Heo, K. H. Chai and J. G. Na, "Correlation between Interfacial Segregation and Surface-energy-induced Selective Growth in 3% Silicon-Iron Alloy", Acta mater., 48, pp. 2901, 2000.

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