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SEGREGATION OF Sb IN Fe-Si-Sb ALLOYS[1])

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Progressive technological methods of production of grain oriented transformer materials employ surface active elements B, Sb, Se as inhibitors. The grain boundary and free surface segregation of Sb in model Fe-3 % Si alloys was studied by the microphractographic method and by the X-ray photoelectron spectroscopy (XPS). The results exhibit intercrystalline cleavage fracture of Fe-Si-Sb alloys in cotrast to transcrystalline fracture of Fe-Si alloys. The enrichment of Sb on the free surface of Fe-Si-Sb alloys is determined by XPS measurements.

СЕГРЕГАЦИЯ СУРЬМЫ В СПЛАВАХ Fe-Si-Sb

В новых технологических методах приготовления текстурированных материалов используются в качестве ингибиторов некоторые активные химические элементы (например B, Sb, Se). В работе при помощи фрактографического анализа и метода рентгеновской фотоэлектронной спектроскопии (РФС) изучается сегрегация сурьмы на границах зерен и свободной поверхности для модельных сплавов Fe-3% Si. Полученные результаты у сплавов Fe-Si-Sb показали межкристаллитный характер разрушения в противоположность с транскристаллитным разрушением сплавов Fe-Si. При помощи РФС анализа было обнаружено количественное обогащение свободной поверхности элементом Sb.

I. INTRODUCTION

The segregation of interstitial and substitutional "sp" impurities to free surface and grain boundary interfaces of Fe-based alloys has been the subject of numerous studies [1, 2]. Especially, with respect to antimony, this interest is due to a strong correlation between grain boundary segregation and embrittlement and because Sb is used as a new grain growth inhibitor. It is known that addition of a small amount of antimony is effective for developing the preferred (110) [001] texture and the (100) [Ovw] texture in grain oriented silicon steel [3, 4, 5]. In addition, there has been a growing interest in studying free surface impurity segregation in order to get information concerning grain boundary composition, since it is assumed that both types of segregation, namely those to free surfaces and those to grain boundaries, are related.

II. EXPERIMENTS

The paper presented describes the study of segregation of Sb in Fe-3% Si alloys by X-ray photoelectron spectroscopy (XPS) and microfractografic methods.

Alloying of these alloys by Sb was performed by the addition of an Fe_xSb_y intermetallic compound below the limit of solubility [6]. The composition of the materials used in the present investigation is shown in Table 1. These model alloys were prepared by the method of vacuum induction melting [5, 6]. Bars from the ingot were hot rolled from 1100 °C to a thickness of about 2 mm. After heat treatment at 700 °C for 30 min., the samples were annealed in an Ar-atmosphere at 1000 and 1100 °C for different times up to 840 min.

Table 1
Composition (wt %) of Fe-Si alloys

Sample	Alloy	Si	Sb	N ($\times 10^{-4}$)	C ($\times 10^{-4}$)	S ($\times 10^{-4}$)
1	Fe-Si	3.02	—	4	5	2
2	Fe-Si-Sb	3.01	0.007	2	5	2
3	Fe-Si-Sb	3.00	0.120	4	5	2
4	Fe-Si-Sb	3.02	0.140	2	5	2

III. RESULTS

Photoelectron spectra of the samples of number 1 and 3 (see Table 1) were measured at pressure $2.7 . 10^{-7}$ Pa in the V 6 ESCA 3, Mk II spectrometer with an Al K_α radiation source. The energy resolution is of 0.2 eV. The samples were measured as casting and after cleaning by Ar-ion sputtering (with a kinetic energy of 2 keV and a current density of 20 $\mu A/cm^2$) for 5 min. Identification of Sb is difficult, because its strongest line $3d_{5/2}$ interferes with line O_{1s}. Typical XPS spectra are shown in Fig. 1, where spectrum 1 is from sample 3, spectrum 2 from sample 1 and spectrum 3 is the difference between spectrum 1 and 2.

Ratio concentration 0.07 of Sb to Fe (calculated to the Fe_{2p} line) for sample 3 decreases to the value 0.02 afer Ar^+-ion cleaning. The results of these XPS measurements exhibit the enrichment of Sb on the free surface of Fe-Si-Sb alloys.

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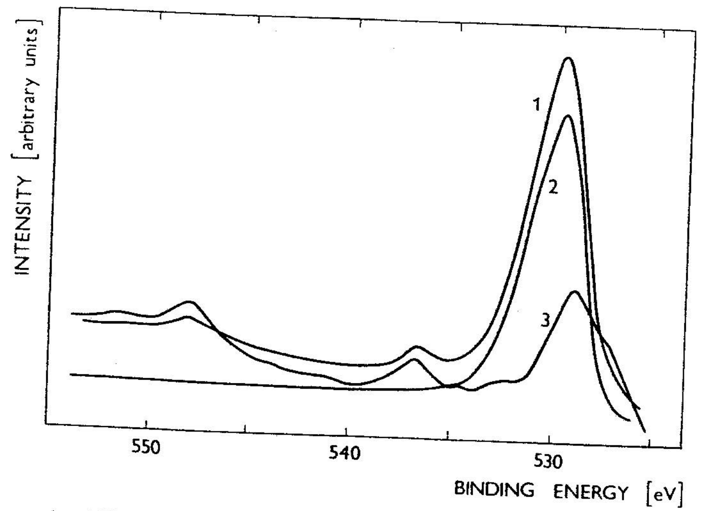

Fig. 1. The core-level XPS spectra: 1 — spectrum from Fe-Si-Sb alloy (sample 3), 2 — spectrum from Fe-si alloy (sample 1), 3 — the difference between spectrum 1 and 2.

Microfractografic studies of the fracture surface of all the samples were performed by scanning electron microscopy JSM-U3. In Fe-Si alloys only an transcrystalline fracture was identified (Fig. 2), while in Fi-Si-Sb alloys mostly an intercrystalline cleavage fracture was identified (Fig. 3). From our microfractog-

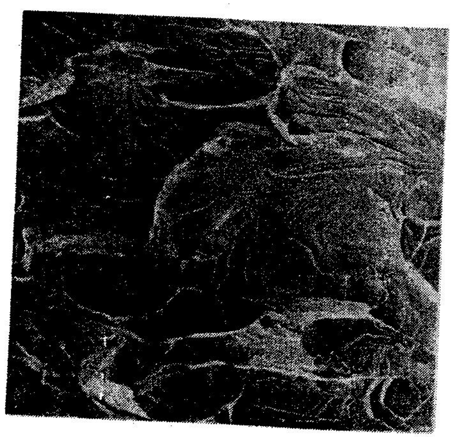

Fig. 2. Morphology of the transcrystalline cleavage fracture of Fe-3% Si alloy (100×).

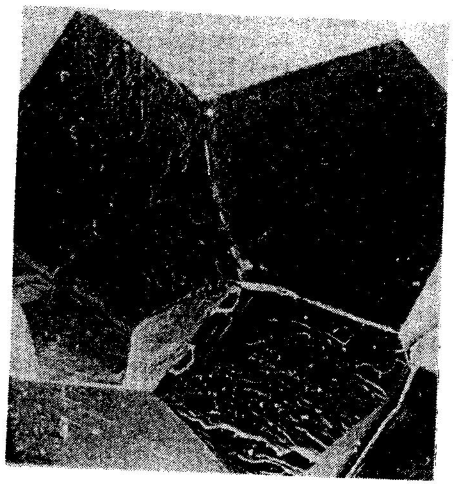

Fig. 3. Morphology of the intercrystalline cleavage fracture of Fe-3% Si-O, 14 % Sb alloy (100×).

rafic analyses it can be concluded that Sb segregates on grain boundaries in Fe-Si-Sb alloys.

Assumed and experimentally exhibited enrichment of Sb on grain boundaries and free surface is probably related with its inhibition effect in the recrystallization process of the studied Fe-Si-Sb alloys [5, 7, 8].

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