TEXTURE AND SEGREGATION STUDY OF Fe—Si AND Fe—Si—Sb GRAIN — ORIENTED ALLOYS[1])

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The intergranular segregation of Sb and the texture of a 3% Si steel alloyed with Sb and treated by a model technology was studied. The segregation of Sb was investigated by the Auger electron spectroscopy. The grain and magnetic textures are studied by X-ray pole figures and the Mössbauer spectroscopy. The results show that antimony segregates to the grain boundaries and that the magnetic texture follows the crystallographic texture in these materials.

I. INTRODUCTION

The basic research of the magnetic properties and the study of the performance of silicon iron in practical applications is still topical in the fields of magnetism and physical metallurgy [1]. It has been shown in [2—4] that small additions of Sb result in the improvements of texture in the grain oriented silicon steel. The interest in antimony is also due to a strong correlation between the grain boundary segregation and embrittlement in Fe-base alloys. The aim of the present work was the investigation of segregation of Sb on grain boundaries and the relation between magnetic and crystallographic textures in the 3% Si commercial steel obtained by a model technology.

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II. EXPERIMENTAL METHODS AND RESULTS

The commercial transformer 3% silicon steel was prepared in an oxygen converter by a model technological process in the East Slovak Iron and Steel Works [5]. The alloying of this material by antimony was performed by addition of Fe_xSb_y intermetallic compounds below the limit of solubility. The ingots were hotrolled to 2.5 mm in thickness. The modelling treatment consists of a two step cold-rolling to a 0.35 mm thickness with intermediate annealing and the complex chemical-temperature treatment on a modelling train. The samples for the Auger spectroscopy study were isothermal annealed in Ar at 1050 °C for 10 h.

The chemical composition (in wt.%) of the final sheets was: C — 0.002, Mn — 0.06, Si — 3.12, P — 0.009, S — 0.013, Al — 0.013, Cr — 0.03, N — 0.009 and Sb — 0.048 or Sb — 0.040; the final sheets with industrial technology consist of 0.050 wt.% Sb (samples A, without antimony; samples B, with antimony).

It follows from metallographic analyses that the average grain diameter d_{av} measured on the sample surface is larger in samples containing antimony than in other investigated materials. Sb composition dependence of d_{av} for model alloys and industrial alloys is shown in Fig. 1.

The antimony is an embrittling impurity segregating at grain boundaries. We study its role in the steels using the Auger electron spectroscopy.

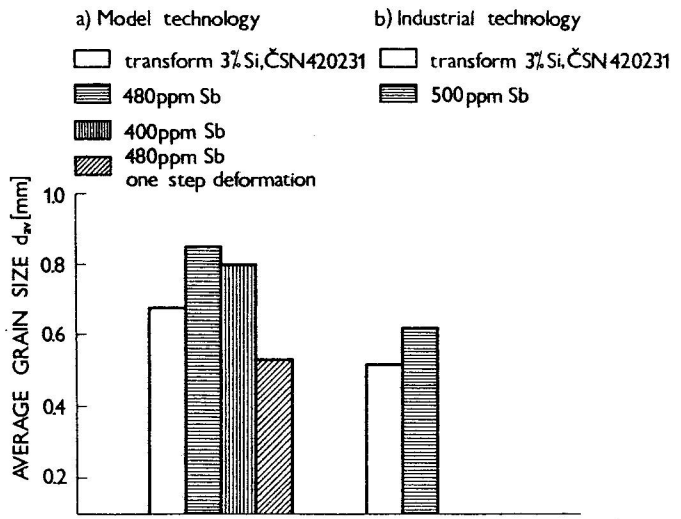

Fig. 1. Sb composition dependence of the average grain diameter d_{av} for model and industrial steels.

The local Auger microanalysis of our samples was made using the Auger spectroscope JAMP 10-S fy JEOL. The system was evacuated to a residual pressure of 10^{-7} Pa. The samples were fractured in situ and the Auger spectra were recorded mostly from the integranual fracture surface at room temperature.

The analysis of the Auger spectra recorded on different grain boundary facets shows that for samples B the concentration of antimony is much higher than the nominal Sb concentration in samples B. The measured Sb concentration on different facets varies from 0.4 at.% to 1.7 at.% and silicon is not observed. For samples A the observed Si concentration varies from 0.7 at.% to 11.6 at.% on intergranular facest. A typical Auger electron spectrum for an intergranular fracture surface of sample B is shown in Fig. 2.

These results support the assumption that Si—Sb interaction is repulsive. It agrees with the results of Iwasaki [6], were the grain boundary segregation of antimony was investigated by internal friction measurements. The observed repulsive interaction between silicon and antimony can be explained by Guttmann's theory of multisegregation.

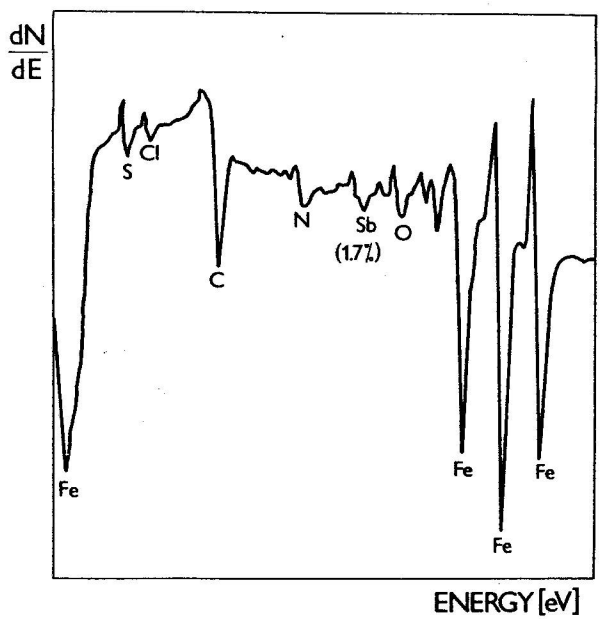

Fig. 2. Typical Auger electron spectrum for intergranular fracture surface of Fe—Si—Sb alloys (sample B).

The grain texture was investigated from the (110) pole figures measured using Shultz's method with MoK_α radiation and a Zr filter. The intensity ratios of reflections in comparison with powdered Fe are shown in Table 1.

Table 1

The results of X-ray pole figure analyses

Sample	The intensity rations of reflections from plane					
	(110)	(200)	(211)	(310)	(111)	(321)
A	5.13	0.25	0.27	—	0.18	0.17
B	6.00	—	—	—	—	—

Mössbauer spectra of the ^{57}Fe were recorded at room temperature in the backscattering geometry using a ^{57}Co in a Cr source. The analysis of the Mössbauer spectra yields the set of intensities of spectral lines, which allow to determine hyperfine field parameters and the ratio R of the intensities of the first and second lines in the Zeeman sextet depending on the angle between the directions of the detected photons and the magnetic field at the iron nuclei in the sample. The Sb atoms in the sample B do not influence the hyperfine field parameters. A component with a large quadrupole splitting would appear if the large Sb atoms were built in the regular FeSi lattice, but it was not observed in this case. We conclude that antimony occurs at grain boundaries. The magnetic texture is described through the ratio R which is equal to 1.24 for both samples A and B. The comparison of the R values agrees with the measurements of the crystallographic texture. In the samples A and B the magnetic moments are oriented nearly in parallel to the surface [7, 8].

III. CONCLUSIONS

The results of these investigations lead to the following observations:

1. The antimony segregates at the grain boundary in Sb-bearing steel (Sb — 0.048 wt.%) and the silicon-antimony interaction at the grain boundary is repulsive. The large grain size in Sb-bearing steel may be due to the increased mobility of differently oriented boundaries in connection with a different surface energy.
2. Sb promotes the formation of the magnetically favourable crystallographic

texture for the grain-oriented steel. The magnetic texture follows the crystallographic texture and the ratio R for our samples reaches the value of 1.24, which is in good agreement with the Fe—Si sheets of the best quality, e.g. and HI—B having $R = 1.26$.

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ТЕКСТУРНОЕ И СЕГРЕГАЦИОННОЕ ИЗУЧЕНИЕ Fe—Si И Fe—Si—Sb ЗЕРНИСТО-ОРИЕНТИРОВАННЫХ СПЛАВОВ

В работе на основе модельной технологии изучается межзеренная сегрегация Sb в текстуре сплава 3 % Si стали с Sb. Сегрегация Sb была исследована при помощи Оже электронной микроскопии. Магнитная текстура была рассмотрена при помощи образцов поля X-лучей и спектроскопии Мессбауэра.