Effect of Surface Orientation on Surface Composition in a Polycrystalline Fe-Cr-Ni Alloy

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Abstract

Auger electron spectroscopy (AES) and electron channeling patter (ECP) have been used for studying the effect of surface orientation on the surface composition in an Fe-Cr-Ni alloy sheet. The alloy sheet was annealed in a mixture of hydrogen and nitrogen gases at a high temperature, and subsequently exposed to air at room temperature. The AES mappings show that the surface composition is different from grain to grain, and the degree of oxidation of the surface depends on the surface orientation. The results of AES and ECP for each grain indicate that nitrogen enrichment on the surface is correlated with chromium enrichment, and that the enrichment is higher in low index planes such as (011) and (111), where oxide layers is relatively thin.

1.Introduction

When the surfaces of a varieties of grains are analyzed by a surface analytical method, the surface composition has been often found to be different from grain to grain in polycrystalline alloys. For instance, relative intensities of x-ray photoelectrons of constituent elements have been different from grain to grain in a chemically etched Fe-Cr and Fe-Si alloys [1]. This suggests that surface reaction or phenomena depend on the surface orientation in polycrystalline materials. Therefore, distribution of the surface composition in each grain should be considered, when chemical characters of the surface of polycrystalline materials are discussed. Since such phenomena have been investigated in only a few systems so far, further microscopic experiments should be conducted to establish the effect of the surface orientation on the surface reaction.

The purpose of the present work is to present new results on dependence of the surface orientation on surface enrichment of alloying elements in an Fe-Cr-Ni. The enrichment takes place when the alloy is annealed at a high temperature. Auger electron spectroscopy (AES) and the electron channeling pattern (ECP) method were employed for investigating the relationship between the surface enrichment and surface orientation.

2.Experimental Procedure

An ingot of high purity Fe-18%Cr-8%Ni alloy was prepared by induction melting under vacuum. It was rolled to a sheet of 0.5 mm thick, and mechanically polished. A sample sheet of 10 mm square was cut for measurement. The sheet was annealed at 1400 K in a mixture of 20% hydrogen and 80% nitrogen gasses. The average grain size was about 40 micrometer. It was mounted on a sample holder for AES measurement.

AES measurements were performed for evaluating the surface composition of a lot of grains in the sample [2]. After the AES measurements, measurements of electron channeling pattern (ECP) [1] were carried out to determine the surface orientation of the grains measured by AES.

3.Results and Discussion

3.1 Auger maps

In order to visualize enrichment of main constituent elements on the surface, their AES images were taken. Figures 1 (a)-(d) show a secondary electron image and Auger maps of O KLL, Cr LMM, and N KLL in a part of this sample, respectively. Oxygen detected may arise from a native oxide layer formed on the surface by air exposure, since it thickness is comparable to a previous result [3]. This result clearly indicates that thickness of oxide layers on the surface of each grain is different form grain to grain. The enrichment behavior of chromium on the surface is similar to that of nitrogen, as shown in Figs.(c) and (d). Such mutual enrichment of chromium and nitrogen has been also observed in surface segregation in an Fe-Cr-N alloy [2]. From comparison of these AES maps, it would be of interest to note that the signal intensities of oxygen is opposite to those of chromium and nitrogen in each grain. These results may correspond to the fact that enrichment of chromium and nitrogen reduces thickness of oxide layers on Fe-Cr-Ni alloys [3].

3.2 Surface orientation dependence of Auger electron spectra

Figures 2 (a) and (b) exemplify Auger spectra from two grain surfaces, showing large and small peaks of chromium, respectively. These two spectra also indicate that chromium enrichment on the surface is correlated with that of nitrogen. Carbon peaks are caused by contamination from air. Significant differences are observed in the surface composition of chromium and nitrogen. Here, let us consider a ratio of nitrogen to oxygen peak heights as a parameter in order to compare the surface compositions of many grains.

The Auger peak height ratios, N/O, which are represented as marks, are plotted in a stereo-triangle, as shown in Fig.3. The AES spectra given in Fig.2 (a) and (b) were obtained grains with orientations denoted as (a) and (b) in the stereo-triangle, respectively. Their orientations are close to (111) plane and a intermediate plane in the triangle, respectively. High enrichment of nitrogen is also observed in a plane close to (011). Geometry of atomic steps, which are formed by deviation from stable low index planes, may depend on the surface orientation for atomic layered structure. Therefore, the atomic geometry is not simply related to enrichment of nitrogen, but it may not say too much that nitrogen and chromium are enriched on the relatively dense crystallographic plane.

3.Concluding Remarks

AES and ECP have been used for investigating dependence of surface orientation on the surface composition in an annealed Fe-18%Cr-8%Ni alloy .

(a) The AES images show that the surface composition is different from grain to grain, and the degree of oxidation of the surface depends on the surface orientation.

(b) The results of AES and ECP for many grains indicate that nitrogen enrichment on the surface is correlated with chromium enrichment, and that the enrichment is higher in low index planes such as (011) and (111), where oxide layers is relatively thin.

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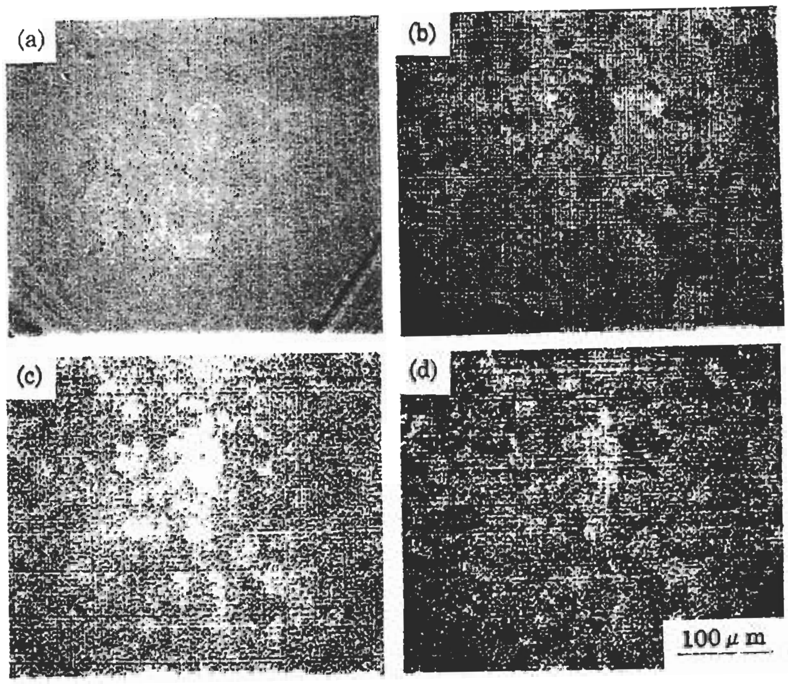

Fig.1 (a) A secondary electron image, and Auger maps of (b) O KLL, (c) Cr LMM, and (d) N KLL of an Fe-Cr-Ni sample.

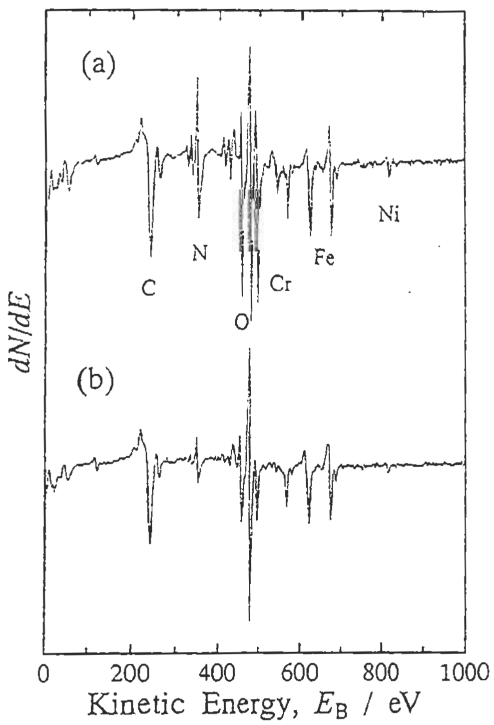

Fig.2 Auger spectra from two grain surfaces with (a) a large chromium peak and (b) a small chromium peak.

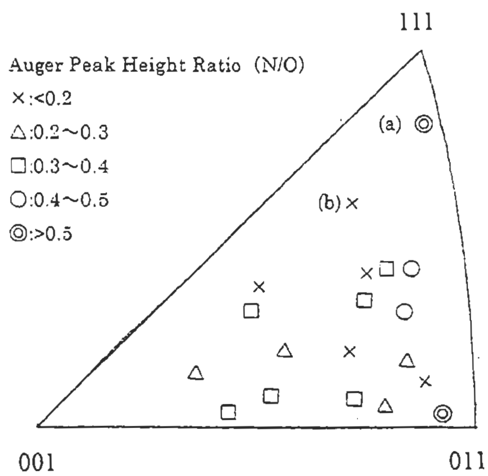

Fig.3 Plot of the Auger peak height ratio, N/O, in a stereo-triangle. The ratio range is given in the figure.