Abstract:

JFE Steel has developed observation techniques utilizing scanning electron microscopy (SEM) and their application to steel products. An appropriate selection of SEM conditions and detectors achieves selective imaging of surface information such as the chemical state, topography, mean atomic number and crystallography. Elemental analyses under low primary electron energy improve spatial resolution and also enable characterization of chemical states. A combination of these techniques improves understanding of the relationship between the microstructures and important properties of steel products and can serve as a lodestar for the design and development of novel materials.

1. Introduction

Steel materials are used in a variety of applications, as exemplified by automobiles, electrical appliances and ships, among others. In addition to mechanical prval pal ulin ! , l famlqq lp nemplg ve y q/bma ql n l t,

paintability, coatability, external appearance, sliding properties during forming, etc. In developing steel products with superior properties, it is important to understand the steel microstructure and surface morphology, which contribute to these properties, in a wide range of scales from a few nanometers (nm) to a few 100 micrometers (μm).

instantaneously. After introducing ULV-SEM in 2001, the company grappled with the development of top surface observation techniques for steel materials[4–14]. “Special Issue on Analytical Sciences and Microstructural Characterization” (Japanese edition of JFE Technical Report) in 2006 introduced ULV-SEM technology and reported its advantages[6]. The present report introduces the results of study of selective visualization techniques for secondary electron (SE) and backscattered electron (BSE) information. In addition, JFE’s efforts in connection with elemental analysis techniques utilizing low accelerating voltages (LV) are also presented. It should be noted that the experiments in the examples presented herein were performed with a Schottky type SEM made by LEO Elektronenmikroskopie GmbH (now Carl Zeiss Microscopy GmbH) or Carl Zeiss Microscopy GmbH equipped with hybrid objective lenses combining electrostatic and magnetic field lenses.

2. Selective Visualization of SE Information

Secondary electron (SE) emission occurs when primary electrons that have penetrated a specimen diffuse while losing energy. That energy is low, only a few 10 electron volts (eV) or less, and the amount of emission from the specimen surface is easily affected by the surface state of the specimen such as surface topography, differences in material, electrical charge, and so on. JFE Steel established a technology for selective visualization of surface information by selecting the range of SE energy which is to be detected preferentially under a low primary electron energy (E_P) condition by appropriate selection of the type and arrangement of detectors.

Photo

with an energy filter[10]. SEM images of the same field of view of the cross section of a carbon steel specimen, which had been heat-treated to form an oxide layer on the surface, were taken at different filter voltages, and subtraction images were obtained by performing arithmetic image processing on these SEM images. These subtraction images selectively incorporate SE of a certain energy range. **Photo 2**[16] shows the SEM subtraction images obtained by the procedure outlined above. Photo 2 (ubpo which lost energy. Channeling contrast is mainly caused by the fact that the LLE produces anisotropy corresponding to the crystal orientation[17]. On the other hand, Z contrast is due to the dependence of backscattering coefficient on the mean atomic number of the substance, and both the LLE and the inelastic BSE contribute to Z contrast[17]. By applying surface-sensitive observation conditions by setting a low θ and low E_P, it is possible to increase the detection ratio of the LLE component, which occurs at the surface from approximately a few nm to a few 10 nm, and thereby emphasize channeling contrast. Conversely, Z contrast can be emphasized by setting high θ and high E_P conditions to reduce the LLE component detection ratio.

Thus, it is also possible to emphasize surface information corresponding to the purpose of observation in BSE images by selecting appropriate observation conditions and detectors based on the contrast formation mechanism.

3.2 Visualization of Steel Microstructure

For control of the microstructure of multi-phase steels, it is important to evaluate the phase fraction, such as ferrite (F) and martensite (M) phases, the crystal size and shape, etc. In observation of the microstructure of steel materials, the method of SEM observation of irregularities after polishing the sample, followed by etching with acid or other chemicals or ion irradiation, was widely used. However, with progress in the refinement of the steel microstructure and the adoption of multiphase materials in recent years, it has become difficult to identify phases and observe microstructures by the conventional method. Observation of these fine, complex microstructures is also possible by techniques for selective detection and visualization of BSE[13)]