Application of Scanning Electron Microscope to Dislocation Imaging in Steel

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Dislocation imaging using the scanning electron microscope with super hybrid lens will be demonstrated to investigate the dislocation cell walls and single dislocations inside cells introduced by shear deformation in conventional steel. The resolution of the dislocation by electron channeling contrast imaging method is similar to that obtained by conventional TEM observation, and a new approach for the study of dislocations which is possible to detect the heterogeneity of the deformation microstructure will be expected on the view point of the advantage of SEM-BSE techniques. There are two different imaging configurations for doing ECCI, one is a fore-scatter geometry, and the other is a back-scatter one. In the present study, the latter case is utilized, which has advantages for little restriction to the sample size and shape and several applications to the stage design in SEM. Since there are some discussions in the contrast mechanism, the improvement of the backscattering electron detector will bring us several ideas for the application of microscopy to the dislocation study with a combination of the conventional TEM technique.

Introduction

With the increase of the use of high-strength steel, the formability control of steel materials with complex phases becomes important, resulting in the requirement for the understanding of the local defect structure, such as dislocations and several boundaries based on both experimental and computational aspects. The characterization of dislocations and other defects has been mainly studied using transmission electron microscopy (TEM) utilizing diffraction imaging contrast and this has brought us a great success in understanding local dislocation structure and further several interaction models among dislocations and other defects such as solute atoms, impurities, clusters, and precipitation. On the other hand, most structure materials, such as steels, consist of polycrystals, and their mechanical properties must be considered using a more macroscopic view, taking in the picture of the whole material, including the effect of grain boundary, the crystallography of grains, and other heterogeneities of microstructure. The TEM thin-foil analysis of dislocations has some limitations regarding these multi-scale formation and deformation aspects, due to the thin-foil effect and some difficulty in the preparation of TEM thin foils themselves, including the heterogeneous large area.

Recently, in order to examine the dislocation structure over a larger area of the materials in question, the electron channeling contrast imaging (ECCI) technique using scanning electron microscopy (SEM) has been discussed in various papers. The theory underlying channeling contrast, of course, goes back to the beginning of TEM contrast interpretation in the 1960's, and it is considered that the defect regions in dislocations should give rise to a modulation of the back-scattered electron (BSE) intensity. In the 1990's, several examples in attempt to obtain dislocation imaging using SEM with and without a field emission gun, along with the imaging and contrast simulation of the clusters for misfit dislocations at the interface, were demonstrated, for example, in strained Si-Ga layers on silicon [1]. Taking into account the effects of surface stress relaxation, back-scattered electron intensity within electron channeling contrast images of dislocations has been also calculated using a two-beam dynamical diffraction model and the simple treatment of multiple scattering [2]. The imaging of screw dislocations near the surface of Si, Ni, and Ga thin films using the ECCI method have been also studied in the 1990's, in which the $\mathbf{g} \cdot \mathbf{b}=0$ criterion for the dislocation image has been discussed [3]. Based on these results, it was established that the intensity of back-scattered electrons is strongly dependent on the orientation of the incident beam with respect to the crystal lattice. Thus, it was pointed out that the information regarding the crystal orientation and its diffraction conditions

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observed was necessary to understand the dislocation structure using the ECCI method, and the electron channeling patterns (ECP's) and electron backscatter diffraction (EBSD) method were extensively investigated at the same time [4].

In the 1990's, on the other hand, the dislocation images obtained using the ECCI method did not provide sufficient contrast regarding the resolution, which was necessary in order to investigate the dislocation structure for the deformed steel materials. As a result, the dislocation cell structure composing of a set of dislocations introduced under fatigued experiments has been mainly investigated regarding the application of the ECCI method [5]. The study of dislocation structure using the ECCI method has not been achieved for use as a conventional method when compared to TEM for the routine imaging and analysis of dislocations. However, with the progress of instrumentation in the SEM fields in around the 2000's, the imaging of individual dislocations has been gradually achieved over a wide range of conditions using the ECCI method utilizing a standard commercially available SEM, using a straightforward experimental configuration, in which the sample is tilted with high angles such as 60 or 70 degrees [6,7].

In high tilting sample geometry using fore-scatter electrons, it is easy to compare crystal orientation data, such as EBSD, because of the same sample stage geometry, however, it is not so convenient for *in situ* observation, such as in a deformation experiment, as well as for large bulk samples after being deformed, in which experiments have been expected in the research field for the deformed microstructure of structure materials. There are two ways progress on the ECCI techniques with fore-scatter and back-scatter geometry measurements. In comparison with data obtained by TEM and SEM, the contrast change of edge dislocations in FeAl alloy has been investigated under conditions of $\mathbf{g} \cdot \mathbf{b}=0$ and $\mathbf{g} \cdot \mathbf{b} \times \mathbf{u}=0$ criteria in 2001 [8]. However, the image formation process in ECCI was not so simple, and the overall image quality for ECCI was not as high as that for TEM.

The strong and clear contrast for single dislocations by the ECCI method must be obtained. In 2006, dislocation images observed for a crack tips and edges under low tilting conditions have been reported in deformed NiAl single crystal using a CamScan 44 FE SEM [9]. In contrast to TEM, electron channeling imaging of dislocations is optimized with s=0, and the image contrast falls off rapidly with both positive and negative deviations from the perfect Bragg's condition. The dislocation contrast using ECCI is very sensitive for the Bragg's condition, resulting in the misunderstanding for distribution of dislocations, in comparison with the thin foil observation under TEM. In order to consider the simple channeling condition, the fore-scatter geometry for the ECCI method is now superior that the back-scatter one. The careful study using single crystals of 4H-SiC [10] and GaN [11] have been carried out using the fore-scatter geometry detector, and the experimental diffraction parameters and contrast features are determined for the threading screw dislocations. It is concluded from the study of 4H-SiC that the combined influences of the diffraction vector, Burgers vector, and deviation parameter on channeling contrast are found to be markedly similar to the corresponding parameters controlling TEM diffraction contrast [10].

With the progress on the basic approach for the mechanism of dislocation imaging, the applying of SEM instead of TEM will be expected to observe dislocations, because of a lot of advantages on the flexibility for the observation condition. In order to overcome the problems of the actual application of the ECCI method in the field of conventional materials, it is valuable to improve the detector of the back-scattered electron signals and other instrumental conditions. In the present paper, it is demonstrated that single dislocations in deformed and non-deformed steels have been observed with strong contrast and in a wide range of areas using a new detector for back-scattered electrons made by JEOL Ltd.

ECCI Experimental Configuration

The observation of dislocations has been carried out using a JEOL JSM-7001F with super hybrid lens and a Schottky thermal field emission gun operated at 0.1-25keV. A new detector such as an Si detector for back-scattered electrons has been mounted on the pole piece of the objective lens, as illustrated in **Figure 1**. Since the intensity of the back-scattered electrons from the surface in this configuration is smaller than that in the high tilting condition (60-70°), the large collection angle is preferable to obtain a good contrast of ECCI. The new detector is possible to set a condition of short working distance (WD) by 2mm, the observation conditions are estimated under several conditions, as indicated on the table in Figure 1. The maximum detection solid angle (Ω) is obtained by 2.7str. under the working distance of 3mm. The present experiment for the ECCI technique was carried out using short working distances from 3 to 4mm.

When increasing at a large tilting angle for the sample, it is possible to expand the working distance than 5mm, similar to the conventional SEM. The EBSD for the orientation measurement and the EDS for the composition analysis were carried out using both detectors equipped on the SEM. The other advantage for the design of super hybrid lens is to achieve the minimum external magnetic and electrostatic field on the sample position under the observation, resulting in the easy alignment of the stigmatism and focusing even for strong magnetic materials such as steel.

The steel used in this study consists of conventional low carbon steel with and without a crystal orientation texture. In comparison with the dislocation microstructure, both observation using TEM and SEM were carried out for the electro polished samples with 3mm in diameter, using the SEM stage for a thin foil TEM sample. The surface of a bulk steel plate was also electro polished to observe the dislocation microstructure by the ECCI method, after mechanical polishing. It is not necessary to take account too much for surface conditions of steel, that is, a slight surface oxide film is not affected to the contrast of ECCI, which is a large advantage against the low voltage observation less than 1keV of the thin film surface microstructure on the top of sample.

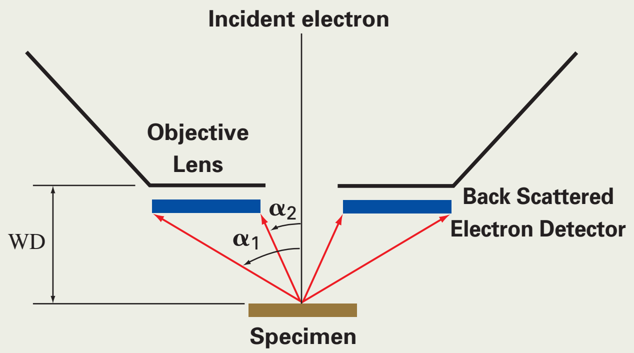
Dislocation Cell Walls and Single Dislocations Using the ECCI Method

In metals, dislocations have been introduced during deformation as a result of activated process of slip system in relation to the crystal structure. The most of steels used for the structure materials such as automobiles, ships, and buildings etc. are composed of the ferrite phase with a body centered cubic crystal structure, in which screw dislocations are mainly introduced during the deformation at room temperature. The burgers vector of a screw dislocation is <111> direction and the slip planes are parallel to {110} plane in BCC structure.

The most excellent elongation behavior in the thin plates is of γ-fiber plates with the {111} texture in the BCC ferrite phase, which are mainly applied as the panel plates of the automobile body. When the simple shear deformation test has been carried out for the model bulk plate with {111} texture, it is known that a characteristic directional dislocation cell walls parallel to {110} planes have been formed with an increasing of the shear deformation [12]. **Figure 2** is a SEM-BSE micrograph showing dislocation cell walls introduced after 60% simple shear deformation to the ferrite steel with {111} texture orientation. The magnification observed was 2,000 times, and the accelerated voltage was 25kV and WD was equal to 4mm. The dislocation cell walls are clearly seen on the whole grains, and the morphology of them is one directional lamellar structure, which is correspondence to the observation result in the thin foil TEM analysis. On the other hand, the perpendicular cell walls appear close to the triple junction, as seen in the center part of the photograph. It is considered that the other slip system on {110} planes was activated under the deformation. This is an example showing the heterogeneity of the deformation, and in comparison with the grain orientation relationship and each dislocation cell walls in the large area using ECCI method, it will be possible to get the information about the heterogeneity of the microstructure developed under deformation.

Since the electron diffraction pattern is not obtained in the conventional SEM, the crystal orientation information using the EBSD measurement was investigated. **Figure 3** is a series of SEM-BSE micrographs (a,b) and the corresponding EBSD orientation maps (c,d). The black right upper part of Fig.3 (a) is a square vacant region used as a marking, which was formed by focused ion beam fabrication. The normal direction of the center grain marked by yellow square is confirmed to be <111> direction, judging from the ND direction mapping in Fig.3(c). For above the photograph (a) and (b), the direction was determined to be parallel to <10 -1> direction by the EBSD mapping in Fig.3(d), Based on the analysis, the crystal orientation information in the center grain is determined as indicated in Fig.3 (b). As clearly seen in Fig.3 (b), the formation of two directional traces of dislocation cell walls with (01-1) and (-110) plane are confirmed, which are reasonable planes predicted from the activated principle slip systems in BCC ferrite steel.

Using the present SEM-BSE system, morphology of single dislocations between dislocation cell walls is clearly distinguished using the ECCI method with more high mag-



Acc (kV)	WD (mm)	α_1 (°)	α_2 (°)	$\alpha_1-\alpha_2$ (°)	Ω (str)
10 ~ 25	2	80	64	16	1.7
10 ~ 25	2.5	72	45	27	2.5
10 ~ 25	3	64	30	34	2.7
10 ~ 25	4	51	20	31	2.0

Fig. 1 Schematic diagram showing geometry of specimen and back scattered electron detector in FE-SEM with super hybrid lens. In the table, accelerating voltages (Acc), working distance (WD), take-off angles, and detection solid angles are also listed.

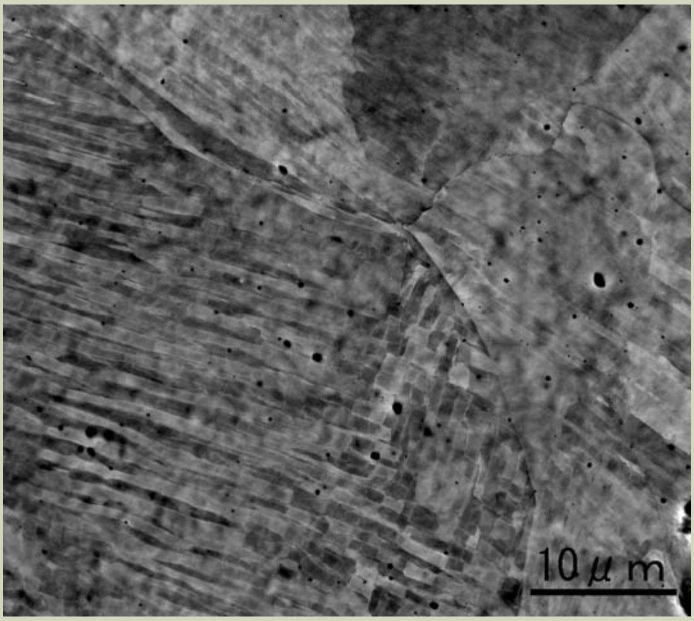


Fig. 2 SEM-BSE micrograph showing dislocation cell walls introduced to each crystal grain after 60% simple shear deformation in steel. A center part of the micrograph is a triple junction of grain boundaries.

nification in the same sample, as seen in **Fig.4**. The magnification of the photographs; (a) is 30,000 times and (b) is 50,000 times under conditions that WD is 4 mm and the accelating voltage is 25keV. In Fig.4 (a), the center area between the dislocation cell walls is satisfied with a strong channeling condition, resulting in a black contrast of the matrix. The incident electrons proceed to the deeper region in the crystal and the characteristic contrast of a single dislocation will be white. With enlarging the figure, the single dislocation and its tangled morphology are remarkably observed in Fig.4 (b). The top of a screw dislocation on the sample surface is represented by a pair contrast of white and black, and the dislocation line expanding to the bulk is a white line with a gradient contrast. It was confirmed by TEM observation using the same sample that the morphology and density of dislocations and dislocation cell walls are almost similar between the data of SEM and TEM.

The ECCI contrast of the single dislocation is very sensitive to the orientation change of the sample. A series of SEM-BSE photographs with stage tilting conditions of 0,1,2,3

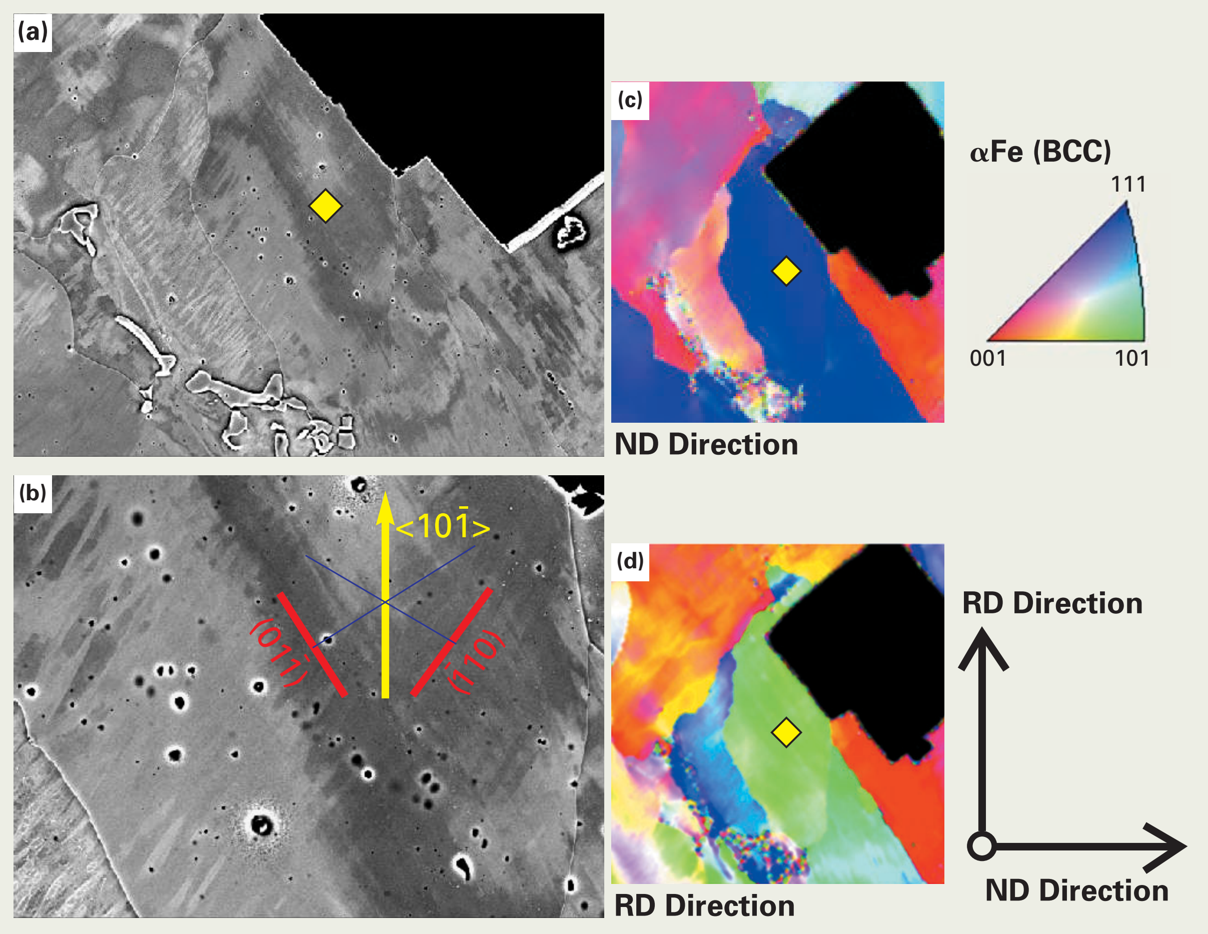


Fig. 3 Orientation analyses for dislocation cell walls using EBSD measurement for same sample investigated by SEM-ECCI method. Micrographs (a)(b) are BSE images, and (c)(d) are orientation mappings images taken from ND direction and RD direction, respectively. The small yellow squares are markings indicating the same grain.

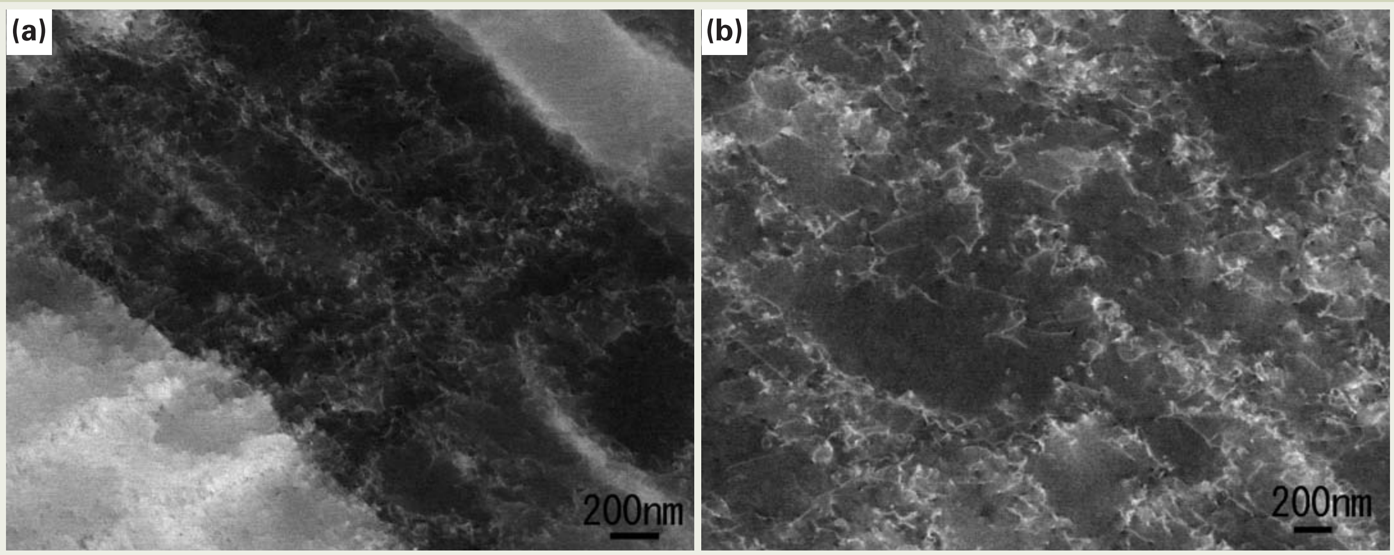


Fig. 4 SEM-BSE micrographs showing single dislocations distributed among dislocation cell walls, in which micrograph (b) is enlarged image of (a).

degrees, respectively in **Fig.5**(a)(b)(c) and (d). The tilting axis is parallel to the horizon line on the photograph. Since the incident beam is parallel to <111> direction on the grain observed, all dislocation lines must be imaged. The dislocations are screw one with the burgers vector of **b**=[111], that is not satisfied with the condition of **g** • **b**=0 criteria. It is found that the dislocation contrast changes within one degree, where the Bragg's angle is about one degree under the accelerating voltage of 25keV. The detail of the contrast mechanism will be discussed in other paper.

Application of the ECCI Method for Other Defects in Steels

The ECCI method is useful to observe other defects, which are mainly investigated by conventional TEM. **Figure 6** is a SEM-BSE micrograph showing complex steel microstructure including ferrite and martensite. The area A is a martensite grain transformed from austenite, enlarged by the inserted micrograph (a). It is found by the SEM-BSE observation that the inner defect of the martensite composes of twined structure, which is identified to be a high carbon martensite. During the steel making process, the carbon enriches from a ferrite to an austenite phase in the complex phase region, resulting in the different internal defect microstructure of the martensite, which is transformed from the austenite phase during the cooling. If the carbon concentration is low in the previous austenite phase, the internal defects of martensite becomes to be random dislocations, and on the other hand, it is known that the internal defect of the martensite phase changes from the dislocations to the internal twined microstructure with increasing of carbon content in the austenite phase. The SEM observation is an easy way to examine the internal microstructure of the martensite phase, which has been mainly carried out by the TEM observation. The present demonstration shows that such an examination will be possible using the recent ECCI method instead of the TEM one. When the sample is slightly tilted, the other dislocation image in the neighboring ferrite grain marked B appear with a slight change of the crystal orientation condition. With careful tilting experiment, it may estimate the difference of dislocation density depending on the amount of local deformation condition around the martensite grain. In addition, the advantage of the ECCI method is easy to investigate a large area with a heterogeneous microstructure such as the complex phases of ferrite and martensite with different internal defects.

It must be noticed here the sample preparation technique. The SEM-BSE image is not sensitive to the surface oxide film, which is another advantage of ECCI method, because the oxidation of the metal is not avoided. In the purpose to observe the dislocation imaging, only surface residual stress after mechanical polishing is taken care for the sample preparation. After the bulk sample is mechanically polished and ground by the alumina polishing solution, the surface of the sample is performed by only the electrolytic polishing.

As a similar observation example, dislocation cell structures and mechanical twins of 30nm thickness have been observed by the ECCI method using a SEM-EBSD based setup system in twinning induced plasticity (TWIP) steels [13]. The advantage to use the EBSD system instead of ECP's to orientate the crystal into optimal diffraction conditions is proposed in the article. The recent advance of the BSE detector and corresponding improvement for the SEM system provide us the sufficient quality of dislocation images and other defects.

As another example showing a new application of the ECCI method instead of the TEM one, the grain boundary morphology and naturally introduced dislocations are indicated in **Fig.7**. The sample is a conventional ferrite steel with a bulk size, whose grain size is several tens of microns with random orientation, as seen in SEM-BSE micrograph of Fig.7(a). Under the observation with a high magnification as 50,000 times, there are some dislocations in a grain and the grain boundary in Fig.7(b). The sufficient contrast and resolution for the dislocations enable us to investigate

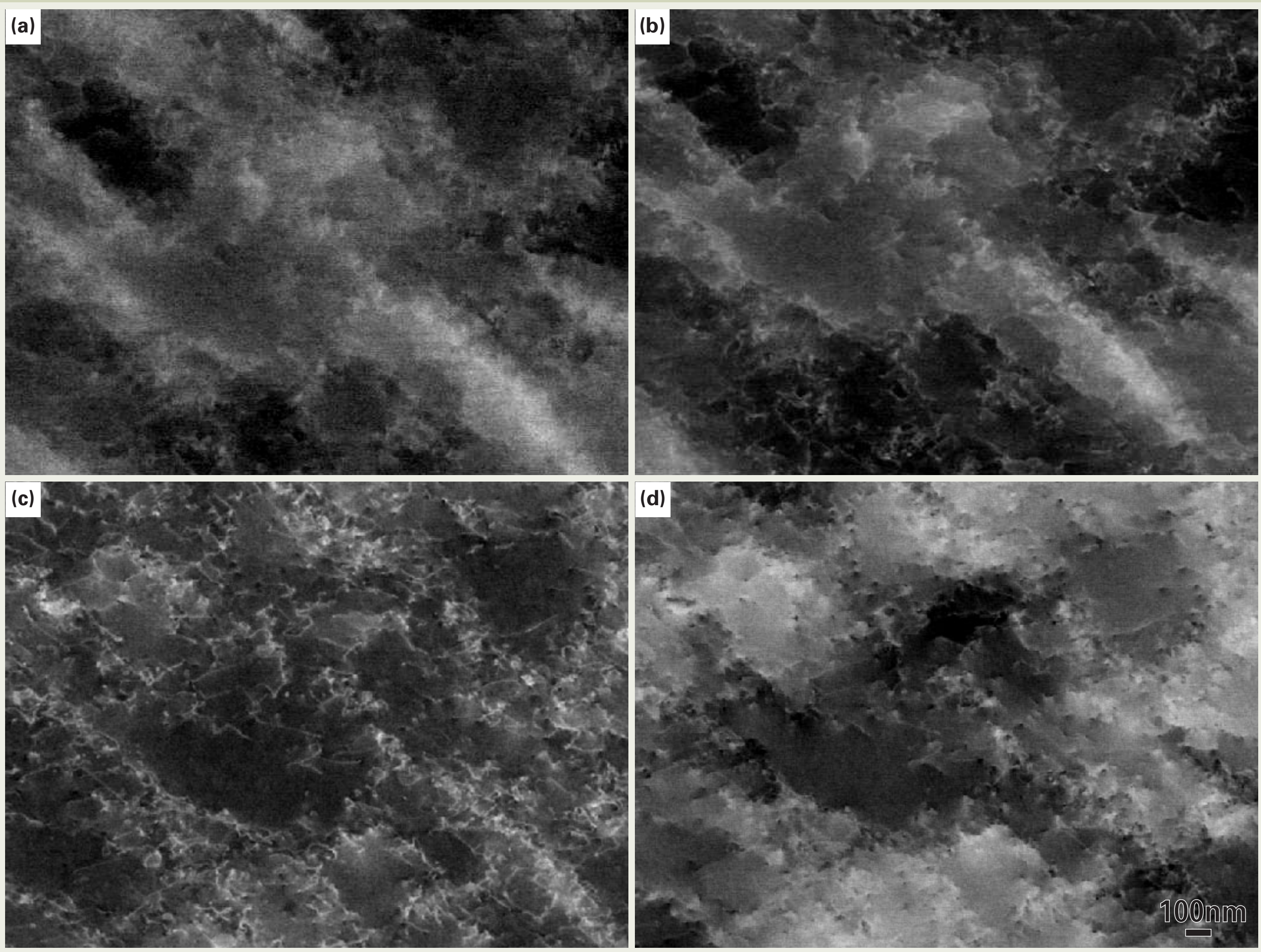


Fig. 5 Change of dislocation image contrast taken by ECCI method with different tilting conditions, (a) 0°, (b) 1°, (c) 2°, (d) 3°, respectively, based on the flat position of the SEM sample stage.

the generation and annihilation of a dislocation under the deformation process.

Summary

A single dislocation image using ECCI method has been clearly obtained using the conventional FE-SEM equipped with the super hybrid lens, which will be also useful for the magnetic material such as steel. Since the resolution of dislocation images is similar to that obtained by TEM, the advantage that wide area can be observed will be a powerful tool to understand the heterogeneity of microstructure under deformation in steel. In the present paper, it is demonstrated that the dislocation cell walls and single dislocation images are observed in the back-scatter geometry of ECCI method, where the sample is mounted on the flat position of the stage in SEM and incident electrons are irradiated perpendicular to the specimen surface. In this geometry, the sample shape and size are not restricted in comparison with the fore-scatter geometry, and several *in situ* observations will be utilized. However, the interpretation of the contrast mechanism for the ECCI method will be more difficult, because several kinds of back-scattering electrons in a material must be considered to understand the case of back-scatter geometry. The detail for the contrast origin and several kinds of imaging data for dislocations will be published in others.

At the present, the limitation of the observation for dislocations still remains because the contrast is strongly affected by the sample tilting condition. The two ways of ECCI methods, which are fore-scatter and back-scatter geometry, will be investigated to make clear the origin of the dislocation contrast in detail and also the diffraction controlling technique will be investigated for the application. Since it is demonstrated to obtain the sufficient contrast of dislocation image using the conventional SEM, the improvement of the sample stage tilting system in SEM will be expected.

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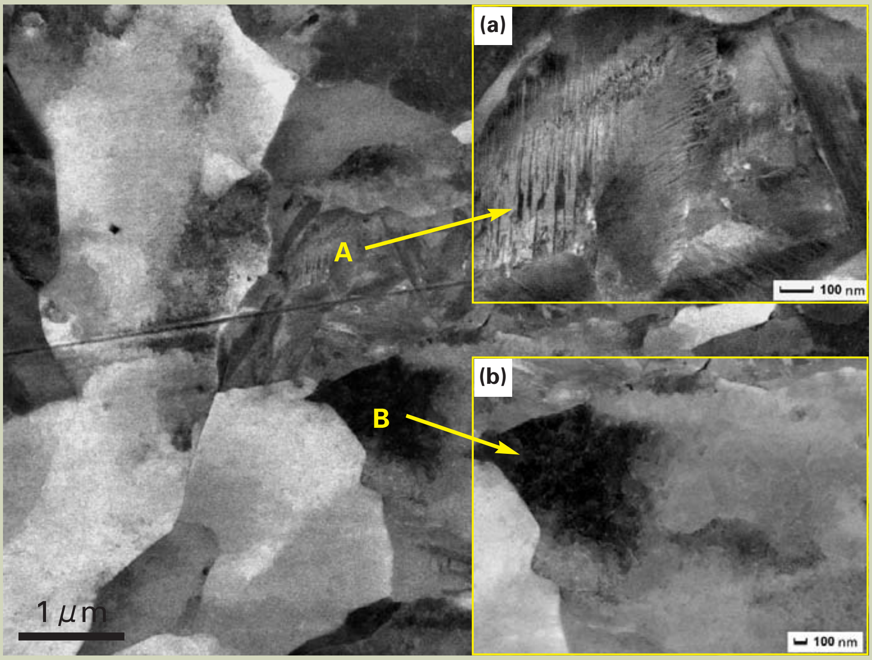

Fig. 6 SEM-BSE micrographs showing morphology of complex microstructure with martensite and ferrite phases in steel. Mark A indicates the martensite grain with internal defects of twined microstructure as identified by the enlarged micrograph (a), and mark B indicates the locally deformed region in the ferrite grain next to the martensite, the tangled dislocations are seen in the enlarged micrograph (b).

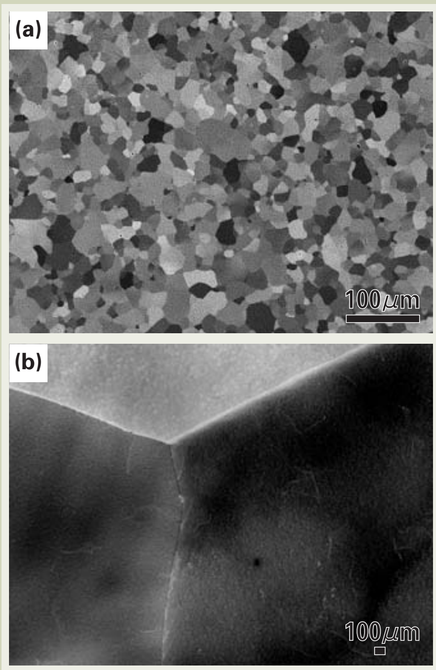

Fig. 7 SEM-BSE micrograph showing conventional polycrystalline steel with random orientation grains (a) and enlarged BSE micrograph taken by magnification of 50,000 times showing naturally introduced dislocations in a grain near to triple grain boundary (b).