Direct Characterization of Interaction Processes between Dislocations and Grain Boundaries by *in situ* TEM Nanoindentation

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Interaction between dislocations and grain boundaries is thought to greatly affects the dislocation behavior, and thus governs the mechanical properties of polycrystalline materials[1][2]. Many efforts have been devoted to understand the mechanism of dislocation-grain boundary interaction, and many microscopic models of the interaction processes have been proposed. However, it is still difficult to directly characterize the elementary processes of dislocation-grain boundary interaction through experiments because the interaction proceeds dynamically and microscopically. In recent years, *in situ* transmission electron microscopy (TEM) enables direct observation of dynamic and microscopic deformation processes. By using *in situ* TEM nanoindentation techniques, we can apply the compressive stress to an arbitrary area in a TEM specimen, and directly observe deformation processes at nanometer scale. Moreover, using the bicrystals as a TEM specimen, we will be able to observe the interaction of dislocations with well-defined grain boundaries. In the present study, we demonstrate the direct observation of dislocation-grain boundary interaction in strontium titanate ($SrTiO_3$) by using the *in situ* TEM nanoindenation techniques.

In this study, we started with characterization of dynamic dislocation behavior within a grain during the nanoindentation. Here, we used $SrTiO_3$ single crystal as a model of in-grain region. Using the knowledge obtained from nanoindentation experiments of single crystals, we performed direct observations of dislocation-grain boundary interaction by using bicrystal specimens. As a model grain boundary, we selected two kinds of grain boundary, {100} low-angle tilt and twist grain boundary ($\theta \sim 1°$). On the low-angle tilt grain boundary plane, the periodic edge dislocations are introduced, while the screw dislocation network is introduced on the twist grain boundary. Thus, we will be able to observe the interaction of lattice dislocations with each type of grain boundary dislocation individually.

In situ TEM nanoindentation experiments were performed by JEM-2010 (JEOL Ltd.) operated at 200 kV, which is equipped with the double-tilt TEM NanoIndenter holder (Nanofactory Instruments AB.). This holder consists of a fixed indenter tip made of diamond and movable specimen which can be precisely controlled by piezo actuator. During the nanoindentation experiments, the sequential TEM images were recorded as a dark-field movie to highlight the dislocation by a video camera with the frame rate of 30 fps.

FIG. 1(a) shows the captured image from the movie of nanoindentation to a $SrTiO_3$ single crystal. We inserted the indenter tip along the [001] direction and observed the deformation processes from the [010] direction. As we inserted the indenter tip, dislocations were introduced into the crystals from the specimen edge. *Ex* *situ* dislocation analyses using g·b invisibility criterion revealed that the slip systems of introduced dislocations belong to the <110>{110} family, which is consistent with the slip system of $SrTiO_3$ at room temperatures in the past reports[3]. From the *in situ* observation and *ex situ* dislocation analyses, dislocation behavior during the nanoindentation can be modeled as shown in FIG. 1(b). The dislocations are emitted from the edge of specimen and propagate in a semicircle shape. Then, a part of dislocation line intersects with the specimen surface, resulting in the separation of the single dislocation segment into two segments. Each of the segments propagates to ±[100] direction as a near-screw dislocation[4].

Next, we carried out the nanoindentation experiments to the bicrystal specimens including low-angle tilt/twist grain boundary. Taking account of the above experiments, we prepared bicrystal specimens so that the grain boundary could stand screw dislocations' way, and we attempted observing that the screw dislocations interact with each grain boundary. In the case of low-angle tilt grain boundary, screw dislocations crossed the grain boundary by intersecting with grain boundary edge dislocations, resulting in kink formation on the screw dislocations and a jogs on the grain boundary edge dislocation. However, in the case of low-angle twist grain boundary, lattice screw dislocations piled up on the grain boundary plane and hardly passed through the grain boundary plane. This can be explained as follow. In order to cross the twist grain boundary, the lattice screw dislocations will have to drag the jogs on the screw dislocation which is formed as a result of intersection with the grain boundary screw dislocations. But, the jog dragging will need the formation of vacancies or interstitial atoms, hindering the dislocation motion at room temperatures. Thus, screw dislocations piled up on the low-angle twist grain boundary plane.

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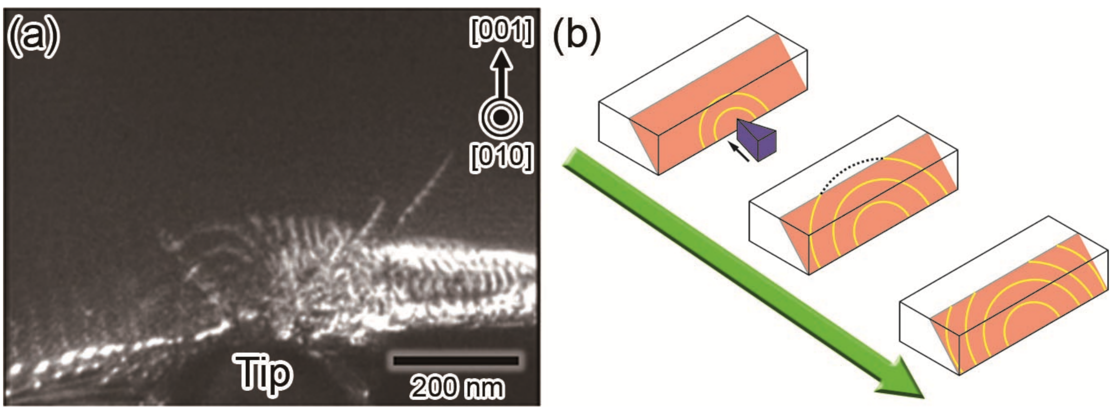

FIG. 1. (a)Dark-field TEM image during the TEM nanoindentation to $SrTiO_3$ single crystal and (b)Schematic illustrations of introduction mechanisms of lattice dislocations