Real-time observations of deformation in ceramics by *in situ* TEM nanoindentation

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Introduction

Ceramic materials have been widely used for structural applications because of their superior mechanical properties. Although ceramic materials are commonly known as brittle materials, some ceramics can plastically deform even at room temperatures. Plastic deformations in crystalline materials typically proceed by two types of microscopic processes: dislocation slip and deformation twinning. To fully understand the mechanism of plastic deformations in ceramics, it is necessary to reveal dynamic behavior of dislocations and deformation twins.

Recent progress in *in situ* transmission electron microscopy (TEM) enables us to directly observe plastic deformation and fracture processes in ceramics[1],[2]. *In situ* TEM nanoindentation techniques, in particular, can apply compressive stress to an arbitrary area in the TEM specimen, and we can dynamically observe the deformation behavior at microscopic scale. In the present study, we observed the deformation processes in strontium titanate ($SrTiO_3$) and lithium niobate ($LiNbO_3$) by the *in situ* TEM nanoindenatation, and the dynamic information of the dislocation slip and the deformation twinning was obtained.

Experimental method

Commercially available $SrTiO_3$ and $LiNbO_3$ single crystal substrates supplied by SHINKOSHA CO., LTD. were used as starting materials. From each substrate, we fabricated thin foils on half-moon grids for TEM nanoindentation by using Ar^+ ion milling method. *In situ* TEM nanoindentation experiments were carried out by JEM-2010 (JEOL Ltd.) operated at 200 kV, equipped with the double-tilt TEM-NanoIndenter holder (Nanofactory Instruments AB.) as schematically drawn in Fig. 1(a). This holder can perform nanoindentation experiments inside a TEM by controlling the specimen motion using a piezo actuator, and double-tilt capability enables the TEM observations under suitable crystal orientation. The indenter tip made of diamond was used in these experiments. Fig. 1(b) shows the dark-field image showing the configuration of the specimen and the indenter tip. In the TEM nanoindentation experiments of $SrTiO_3$, we inserted the indenter tip along the [001] direction and observed the sample near from the [010] direction. In the case of $LiNbO_3$, we inserted the tip along the [0001] direction and observed near from the $[2\bar{1}\bar{1}0]$ direction. The sequential TEM images during the nanoindentation experiments were recorded as movies with the frame rate of 30 fps.

Results and Discussion

Fig. 2(a) and (b) show the dark-field TEM images captured from the movies of the nanoindentation experiments in $SrTiO_3$ and $LiNbO_3$, respectively. In the case of $SrTiO_3$, some dislocations were introduced from the edge of the specimen where the indenter tip actually contacted. Image contrast analyses after the nanoindentation revealed that all the introduced dislocations belonged to the same family of the $\langle 110 \rangle \{1\bar{1}0\}$ slip system, which is known to be the primary slip system of in $SrTiO_3$ at room temperatures[3]. In contrast to the $SrTiO_3$ case, no obvious contrast indicating the dislocations was observed in $LiNbO_3$ during the TEM nanoindentation. Instead, deformation twins nucleated from the edge of the specimen. Static analyses identified that the deformation twins were identified as $\{01\bar{1}2\}$ rhombohedral twin. This result also agrees with the previous macroscopic studies[4].

In the both cases, the dynamic and microscopic deformation processes were directly revealed. *In situ* TEM nanoindentation techniques thus will become powerful method to fundamentally understand the deformation mechanism in ceramic materials.

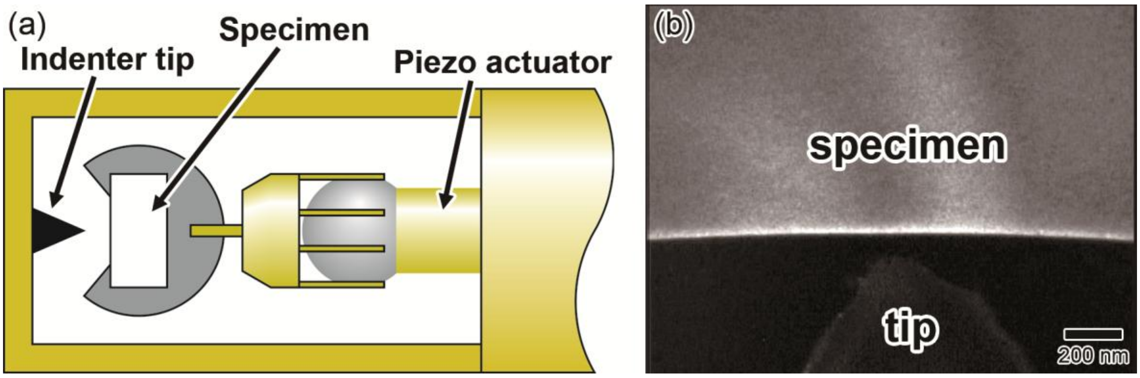

Fig. 1 (a) Schematic illustration of the TEM nanoindentation holder used in this study. (b) Dark-field TEM image showing configuration of the specimen and the indenter tip.

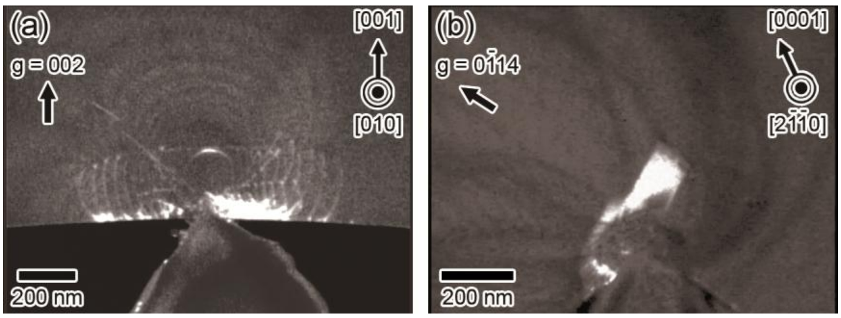

Fig. 2 Dark-field TEM images captured from the movies recorded during the nanoindentation into (a) $SrTiO_3$ and (b) $LiNbO_3$.

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