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Atomic-scale Analysis of Mechanical Response of $SrTiO_3$ by MEMS-based in Situ STEM Mechanical Testing

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Plastic deformation and fracture of crystalline materials are originated from the nucleation and propagation of lattice defects such as dislocations, twins and cracks. Since they are structural defects at the atomic level, it is essential to understand their atomic structure evolution upon loading. In situ transmission electron microscopy (TEM) mechanical testing is a powerful technique to directly observe mechanical response of lattice defects. However, in situ TEM holders for mechanical testing are not typically compatible with atomic-resolution TEMs because of limit on the size and lack of double-tilt capability. In recent years, micromachining techniques have been rapidly developed, and mechanical devices in micrometer scale, microelectromechanical systems (MEMS), are put to practical use. MEMS devices are produced from a Si-based substrate with flexible design and can be equipped with a precise loading mechanism within micrometer size. Therefore, MEMS devices can be promising experimental system for in situ TEM mechanical testing. Indeed, there are several reports on in situ mechanical testing using MEMS devices [1,2], although it is still difficult to perform the mechanical testing with atomic-resolution imaging. In this study, we designed a custom-made MEMS device compatible with a double-tilt TEM holder and performed atomic-resolution in situ mechanical testing. The strain filed formed due to stress concentration in a $SrTiO_3$ single crystal will be discussed.

A MEMS device for mechanical testing was fabricated by lithography technique. This MEMS device has two loading arms driven by electrostatic actuators, which is designed to be compatible with a double-tilt biasing TEM holder (Aduro300, Protochips) [2]. A $SrTiO_3$ single crystal was cut to a small chip and then mounted on the MEMS device by focused ion beam system (FIB: Helios G4 UX, Thermo Fisher Scientific). The sample was further milled and thinned to a shape for in situ TEM loading testing. The observation and loading axes were set to be [100] and [011], respectively. In situ loading testing was performed in order to apply step-by-step load to the sample in a scanning TEM (STEM: ARM-200F, JEOL) operated at 200kV. In each load step, an atomic-resolution still image was acquired by high-angle annular dark field (HAADF) imaging.

Fig. 1 shows a HAADF-STEM image of the sample at the applied load of 13.3μN. The dark region at the top center is a notch. Atomic columns are clearly seen over the image even upon loading, indicating that the present experimental system has enough stability for atomic-resolution imaging. The image panel is perfect square, while the atomic lattice appears to be slightly distorted, suggesting the sample is deformed elastically. To obtain a strain field of the sample, atomic column positions were analyzed. Assuming that each atomic column is imaged by a Gaussian function, fitting parameters were optimized by a Markov chain Monte Carlo method using the Metropolis–Hastings algorithm [3]. Fig. 2 shows a strain mapping of ε_{xx} (x: horizontal direction) corresponding to the HAADF-STEM image in Fig. 1. The blue circles indicate the optimized atomic column positions. Just below the notch, strong strain of ~10% is detected corresponding to stress concentration at the notch. Note that the strains obtained using a HAADF-STEM image under load-free condition were within 1-2%, which is considered to be the error of the present experiment and analysis.

In the presentation, the atomic-scale behavior of the sample upon loading until fracture will be demonstrated. Based on the observed atomic column positions, we will discuss the strain field around the notch and the crack propagation phenomenon.

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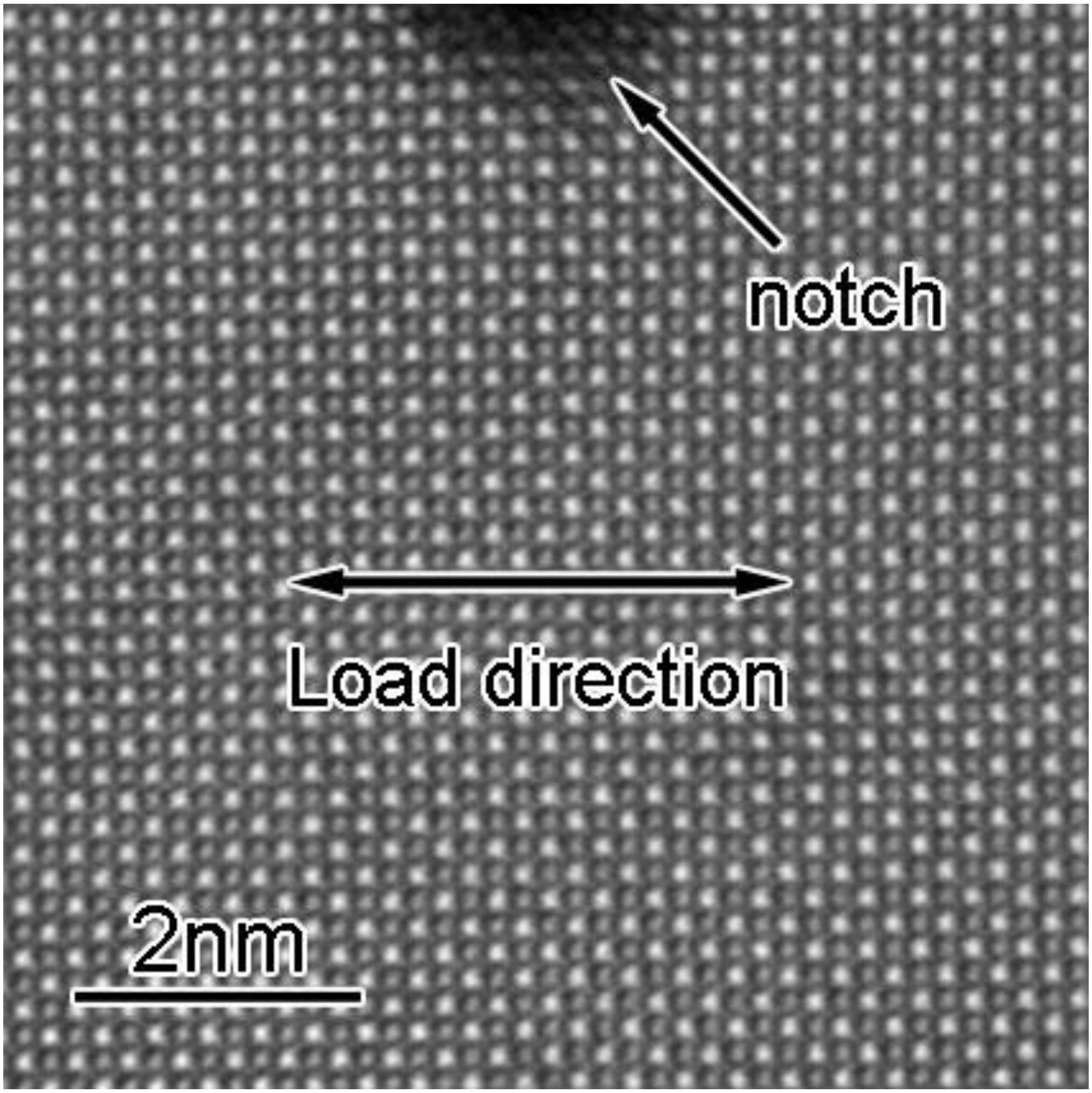

Figure 1. HAADF-STEM image of the sample at applied load of 13.3µN.

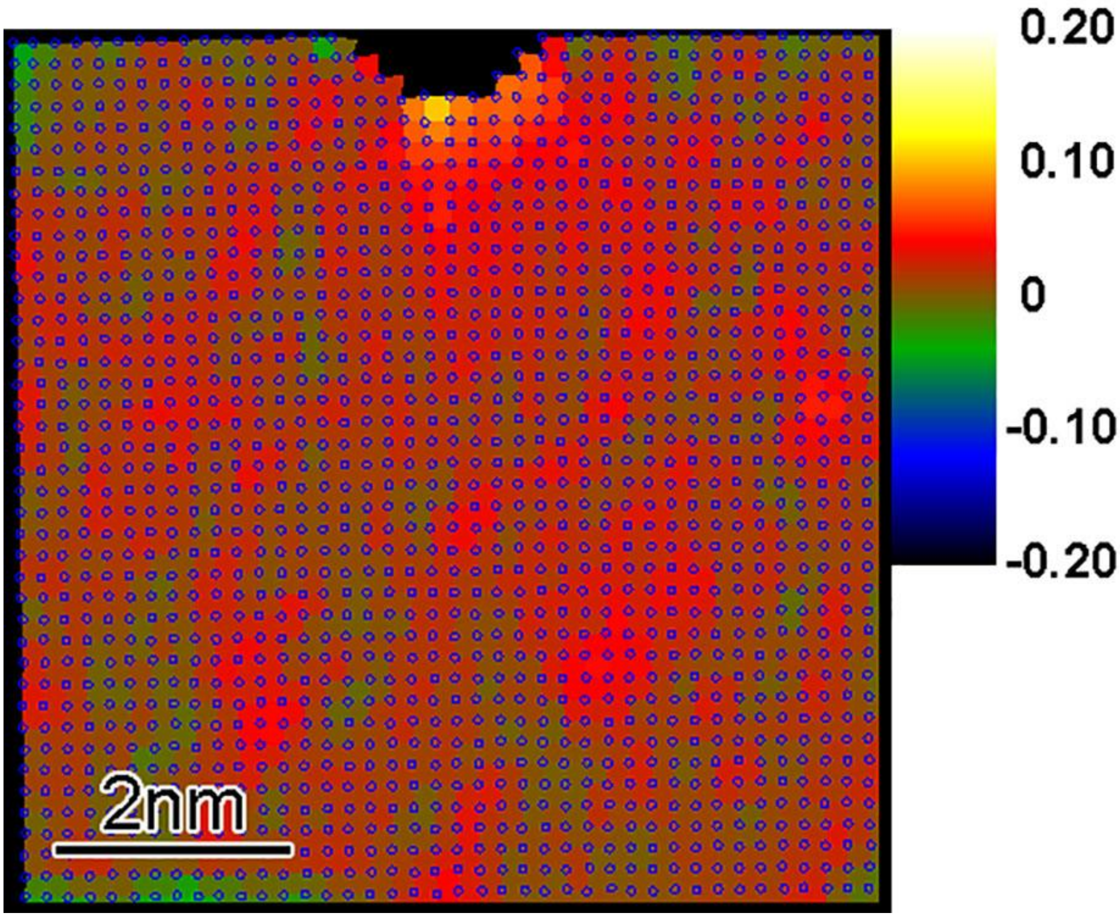

Figure 2. Strain mapping corresponding to Fig. 1.

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