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Cross-sectional Observations of Surface Structures on MgO{100} by Cs-corrected TEM

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Recently, a spherical-aberration (C_s) corrected 200kV TEM has brought a significant improvement in spatial resolution, which allows us to identify oxygen atom columns in crystalline materials [1,2]. Another important advantage of C_s-correction is that non-periodic structures such as surfaces/interfaces are observed precisely at almost just focus. In the present study, we have succeeded in observing structural changes of {100} surfaces of MgO cross-sectionally by a C_s-corrected TEM.

We used a 200kV TEM (JEOL: JEM-2100F) equipped with a C_s-corrector[3]. MgO crystalline thin films for TEM observations were prepared by electron-gum evaporation onto NaCl substrates in vacuum of 5 x 10^{-8} Pa. After removal of the substrates by water, the MgO films were mounted on micro-grids. Holes several nm diameter were created in the films due to local vaporization by a focused electron probe in the TEM (FIG.1). We observed clean {100} surfaces at side walls of the holes cross-sectionally in a high vacuum ($1x10^{-5}$Pa) (FIG.2). Mg and O atom columns appear as larger and smaller dark dots in the layers underneath the {100} surface layer. On the other hand, dark and bright contrasts in the surface layer elongate toward vacuum as indicated by black and white arrows in the figure. It has been known that there is no remarkable reconstruction on clean {100} surfaces of MgO except for slight rumpling and relaxation[4]. However, image simulations of such a surface structure do not reproduce the elongation of the contrasts in FIG.1(a) at any defocus values with any thicknesses (FIG.3(a)). It is considered that the elongation is related to surface adatoms/vacancies of Mg or O atoms created during the vaporization of the MgO films by the electron probe. We can estimate structural changes induced by the point defects on the surfaces roughly by Madelung energies. The results indicate that a di-vacancy which consists of two adjacent vacancies at the same sub-lattice induces displacement of adjacent atoms at another sub-lattice about 0.1 nm toward vacuum. Image simulations of the structure model in which some di-vacancies are introduced on a MgO{100} surface reproduce the elongation of the contrasts in FIG.2 (FIG.3(b)). It is considered that we succeeded for the first time in observing structural changes induced by surface lattice defects on MgO{100}.

It is difficult that we detect this kind of local structural changes by using diffraction or spectroscopic methods and that we analyze precisely them by using an ordinary TEM. Images with a non-zero C_s value do not allow us intuitive predicts of surface structures as shown in FIG.3(c) due to lack of spatial resolution and artificial contrasts around the surfaces by significant defocus values. It is considered that Cs-corrected TEMs make it possible to analyze surface structures with a high degree of accuracy.

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[5] The authors would like to thank Dr. M. Haider of CEOS GmbH, Prof. Y. Ikuhara of Tokyo University, Dr. T. Hirayama of Japan Fine Ceramics Center, Messrs.T. Tomita of JEOL Co.. The present study is partly supported by a Special Grant as "Active-Nano Characterization" from the Ministry of Education, Culture, Sports, Science and Technology, Japan.

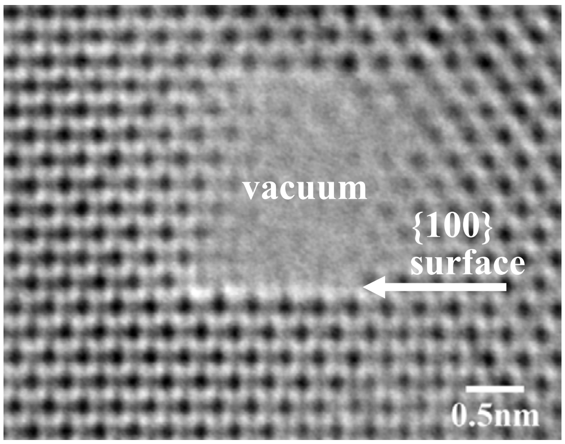

FIG.1. Hole created in a MgO{110} thin film by a focused electron probe in a TEM. Clean {100} surfaces are formed at edges at the hole.

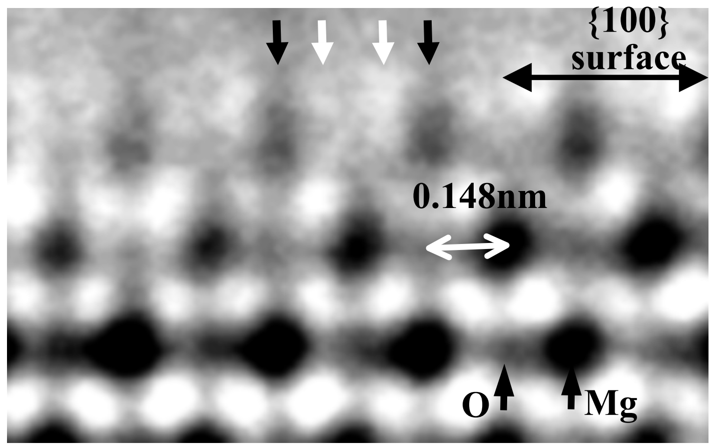

FIG. 2. Cross-sectional observation of the clean MgO{100} surface under Cs=0. Dark and bright contrasts in the surface layer elongate toward vacuum as indicated by black and white arrows.

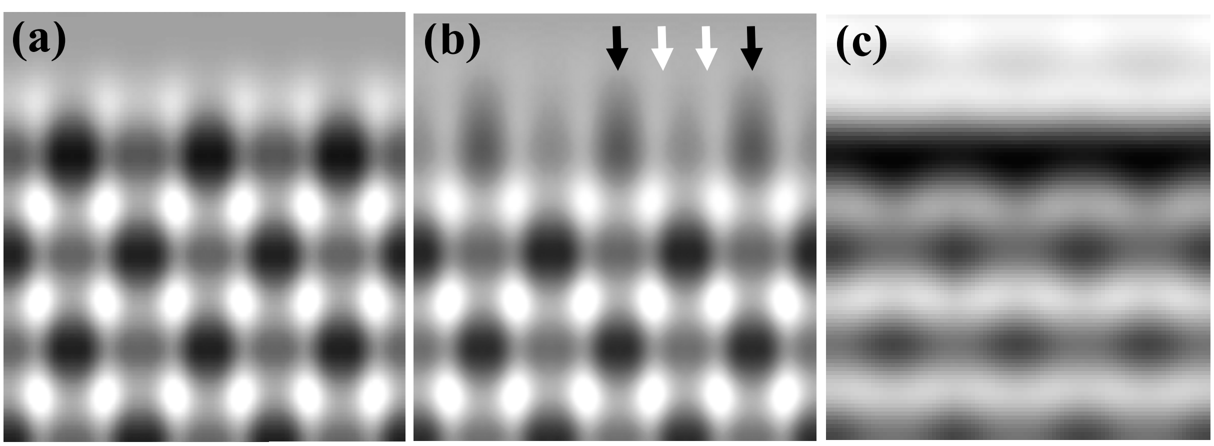

FIG. 3. Image simulations of MgO{100} surfaces. (a) Surface without reconstruction (Cs=0). (b) Reconstructed surface induced by surface vacancies. The elongation of the contrasts in FIG.2. is reproduced (Cs=0). (c) Surface without reconstruction by ordinary TEM (Cs=0.5mm).

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