RHEED-based Biaxial Texture Measurement Verification with Synchrotron X-Ray Rocking Curves

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Introduction

Biaxially textured MgO is technologically interesting since it provides a suitable path for silicon integration of single crystal-like films for many important perovskite materials. This is accomplished by using ion beam-assisted deposition (IBAD) to create biaxially textured films (polycrystalline films with a preferred in-plane and out-of-plane grain orientation) on amorphous substrates [1]. Heteroepitaxy of biaxially textured piezoelectric films on IBAD MgO buffer layers would enable monolithic integration of new types of actuators for microelectromechanical (MEM) systems on silicon integrated circuits.

Previous efforts to optimize the biaxial texture of IBAD MgO have been impeded by the *ex situ* nature of conventional biaxial texture analysis techniques, either transmission electron microscopy (TEM) or x-ray diffraction (XRD). Because the biaxial texture develops within 11 nm of growth, XRD cannot resolve crystallographic texture unless the x-ray source has synchrotron brightness, making it difficult to investigate biaxial texture development mechanisms. To circumvent these obstacles, we have developed a reflection high-energy electron diffraction (RHEED)-based method for quantitative *in situ* biaxial texture analysis of MgO [2]. Because RHEED is sensitive to films as thin as 30 Å thick, we expect to have the capability of analyzing the biaxial texture development during grain growth, film coalescence, and film growth.

Methods and Materials

Validation of the RHEED-based biaxial texture measurements was accomplished by comparing them with measurements of biaxial texture made at the APS. A series of 4- to 8-nm IBAD MgO films were grown on Si_3N_4 by using e-beam evaporation of MgO with a simultaneous 750-eV Ar^+ ion bombardment at 45° incidence. The biaxial texture (the in-plane and out-of-plane orientation distributions) was controlled by varying the ion/MgO flux ratio. The narrowest biaxial textures were observed at an ion/MgO flux ratio of 0.47, so by either increasing or decreasing the flux ratio, broader orientation distributions were produced. Before going to APS, the in-plane and out-of-plane orientation distributions were measured by using RHEED to ensure that the series of samples had a broad range of distributions. Rocking curves around the (200) and (002) peaks of the MgO were taken at APS to measure the in-plane and out-of-plane orientation distributions, respectively.

Results and Discussion

A direct comparison between the RHEED and x-ray measurements of the in-plane and out-of-plane orientation distributions is shown in Figs. 1 and 2, respectively. Both the in-plane and out-of-plane distribution measurement comparisons are well-represented by a linear fit, indicating that the RHEED method successfully determines the biaxial texture. However, there is an offset between the RHEED and x-ray measurements of about 3° and 1° for the in-plane and out-of-plane orientation distribution measurements, respectively. By using RHEED, we have been able to measure the biaxial texture distribution as a function of film thickness. These measurements reveal that the first layers of IBAD MgO have broader orientation distributions than the surface, as shown in Fig. 3. Because electrons interact strongly with the MgO scattering lattice, the electrons measure only the biaxial texture of the surface, while x-rays can easily penetrate the entire 7-nm film thickness and therefore get contributions to the biaxial texture measurements from

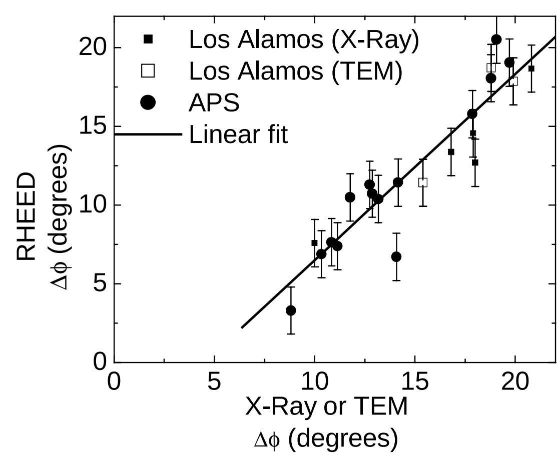

FIG. 1. In-plane orientation distribution ($\Delta\Phi$) measured by RHEED analysis vs. TEM or x-ray diffraction measurements. X-ray rocking curves were collected by using either a rotating anode source at Los Alamos or synchrotron radiation from the APS.

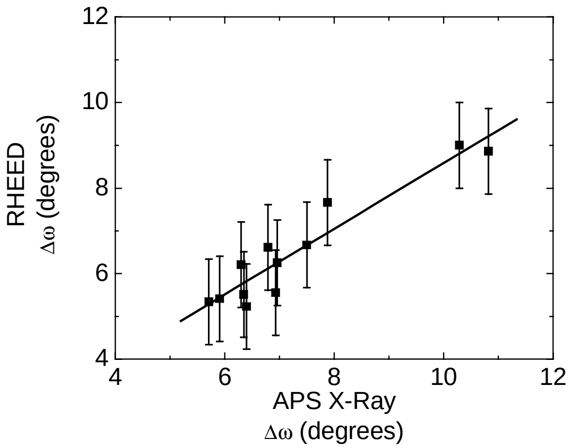

FIG. 2. Out-of-plane orientation distribution (Δω) measured by using RHEED and synchrotron x-ray out-of-plane rocking curves. The line is a linear fit to the data.

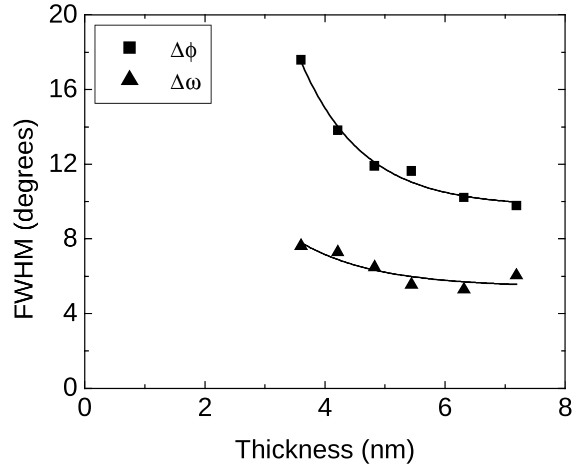

FIG. 3. In-plane (Δϕ) and out-of-plane (Δω) orientation distribution for IBAD MgO growth as a function of film thickness measured by using RHEED. The lines are a fit to the data.

from the entire film thickness. By integrating the orientation distribution measurements as a function of film thickness (Fig. 3), we find that the difference between the average orientation distribution and the surface orientation distribution is about 2.5^o and 1^o for in-plane and out-of-plane measurements, respectively. This result is consistent with the offset observed between the RHEED and x-ray biaxial texture measurements. The systematic offsets between RHEED analysis and x-ray measurements of biaxial texture, coupled with evidence that biaxial texture improves with increasing film thickness, indicate that RHEED is a superior technique for probing surface biaxial texture.

Acknowledgments

Use of the APS was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38. We would also like to thank UNI-CAT and P. Zsack for technical support. Additional funding was provided by the DARPA VIP III program and ARO MURI Grant No. DAAD 19-01-1-0517.

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