Measuring Interface Strains in Depth Using X-Ray Bragg-Surface Diffraction

An X-ray diffraction method, using three-beam Bragg-surface diffraction, is developed to measure strains at the interface of MBE Au/GaAs(001), where grazing-incidence diffraction cannot apply due to the difference in refractive index between Au and GaAs. Changes in diffraction images of the surface reflection (1-13) of GaAs(006)/(1-13) three-beam Bragg-surface diffraction and the (-1-13) of GaAs(006)/(-1-13) at different azimuth and Bragg angles give the depth penetration of 2Å resolution. The variations of lattice constant, -49%, -27% and 2% along the surface normal[001] and in-plane directions [-1-10] and [1-10] are determined within the depths of 18Å, 72Å, and 72Å, respectively.

The strain induced at interfaces may deteriorate material properties due to crystal lattice distortion, thus degrading the performance and lifetime of the devices fabricated. Methods for characterizing interface strain are therefore most desired. For the grazing incidence diffraction frequently employed for surfaces/interfaces structural studies, X-rays scattered from the interface may be overshadowed by that diffracted from the substrate due to the large X-ray penetration depth. Moreover, when the refractive index of a thin film is smaller than that of the substrate, external total reflection does not occur for X-rays travelling through the interface of the thin-film and substrate. Without the totally reflected beam, the information about the interface structure may not be easily extracted from the intensity measurement of the transmitted diffracted beam. To overcome this difficulty, we proposed a method to determine three dimensional interface strain non-destructively with an atomic resolution for the molecular beam epitaxial (MBE) Au/GaAs(001) sample system where the refractive index of gold is smaller than that of GaAs for X-rays. No total external reflection occurs unless diffraction takes place from the backside of the sample. The proposed method adopts the so-called Bragg-surface multiple diffraction (BSD), where a surface diffracted beam carries interface structural information.

X-ray Bragg-Surface diffraction measurements were conducted at the wiggler beamline BL17B, NSRRC. A Si(111) double-crystal monochromator and a collimating mirror provided a highly monochromatic ($\Delta E/E \sim 10^{-4}$) and collimated beam. The X-ray energy used was 11.0577 keV and the incident beam size was 0.5mm x 0.5mm. The sample mounted on an 8-circle diffractometer was aligned first for the symmetric Bragg reflection GaAs(006), the primary reflection

Beamline

17B2 X-ray Diffraction

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G, by adjusting the Bragg angle θ = 36.492˚. The crystal was then rotated around the normal of the G-reflecting planes (the reciprocal lattice vector $\overrightarrow{G}$), the azimuthal φ scan, to bring the additional set of atomic planes L(=(1-13)), the secondary reflection, also satisfying Bragg's law. Two diffracted beams along the wavevectors $\vec{K}_G$ and $\vec{K}_L$ were generated by the G and L reflections for an incident (000) beam $\vec{K}_O$ simultaneously (Fig.1a). The latter (1-13) is a surface diffraction, because the reciprocal lattice point L lies on the equatorial plane of the Ewald sphere (Fig.1b) parallel to the crystal surface. All the reciprocal lattice points, O(000), G(006), and L(1-13) lie simultaneously on the surface of the Ewald sphere of the radius equal to 1/λ (Fig.1b) The surface diffracted (1-13) beam was monitored by a scintillation counter and an image plate (IP) placed 30 cm away. The diffraction intensity profiles of the (006) and (1-13) versus θ and φ (not shown), respectively, were measured by the counter. Images of the surface diffracted waves at different φ were recorded for the incident X-ray beam hit the center of the sample. In principle, for a fixed θ, the surface diffraction images taken at varying φ should provide the information about lattice-constant variation parallel to the interface. For fixed φ, the surface diffracted images taken at various θ yield the information about lattice-constant variation normal to the interface. Figure 2a shows the surface (1-13) diffraction images obtained for increasing φ in a step of 0.02˚ at θ = 36.492˚, where the intensity of (006) is maximum. The lower tiny spot is the image of the Bragg-surface diffracted (1-13) beam from the deep GaAs lattice, and the upper rather diffuse spot is due to the specular reflection from an isostrained layer near the interface. For a fixed θ angle the vertical separation between the two images decreases when the angle φ increases. This is schematically shown in Fig.2b, where C is the position of maximum diffuse intensities for a φ angle. Point A represents the tiny diffraction spot from the deep substrate. The vertical angular separation ξ between the diffuse maximum C and spot A increases, when the φ angle decreases. Meanwhile spot A also shifts horizontally to D (Fig.2c). We also detected that at a fixed φ the sharp substrate spot A moves upward to E for increasing θ angles. The vertical movement of the diffuse spot for varying φ angles can be considered as scattering of the surface diffracted wave from an iso-strained layer at different depths normal to the interface, as was described for scattering from quantum dots. Namely, the φ angle is associated with the depth at which an iso-strained layer located.

The vertical and horizontal shifts of spot A (Fig.2c) are closely related to the lattice-constant variation of GaAs perpendicular and parallel to the interface, respectively. These variations can be determined by considering the geometric condition for a general three-wave (O, G, L) diffraction (Fig.1b).

Using the positions of diffraction spots, especially the shifts A→D and A→E (see Fig.2c), at various φ and θ, two-dimensional lattice-constant variations and strains along [1-10] near the interface can be determined. Also a φ angle gives the information about the depth *z* of an iso-strained layer which satisfies the surface L diffraction condition. Moreover, when φ is rotated 90˚ from φ = 37.654˚, the three-beam (000)(006)(-1-13) Bragg-surface diffraction occurs. We can use that diffraction and repeat the same procedure. The lattice-constant variation along [-1-10] can be determined. Thus we have three-dimensional information about the interface strains, i.e., $\Delta S_{\perp} = \frac{\Delta l_{\perp}}{l}$, $\Delta S_{//} = \frac{\Delta l_{//}}{l}$, and the strain normal to the interface. $l_{\perp}$ and $l_{//}$ are the vertical and horizontal components of the reciprocal lattice vector $\vec{l}$ of the secondary reflection.

For the Au film, we employed the conventional grazing-incidence X-ray diffraction (GIXD) and Bragg diffraction to measure lattice-constant variations. Au (-220) and Au(004) in-plane reflections and Au(-2-20) Bragg reflection were used for estimating the lattice variations in the two in-plane directions and the normal to interface, respectively. From the observed diffraction spots at different azimuth angle φ and Bragg angle θ, the variations of lattice constant at different heights from the interface, like that in quantum dots, were determined according to Bragg's law. The results are combined into Fig.3.

Figures 3a, 3b and 3c show the

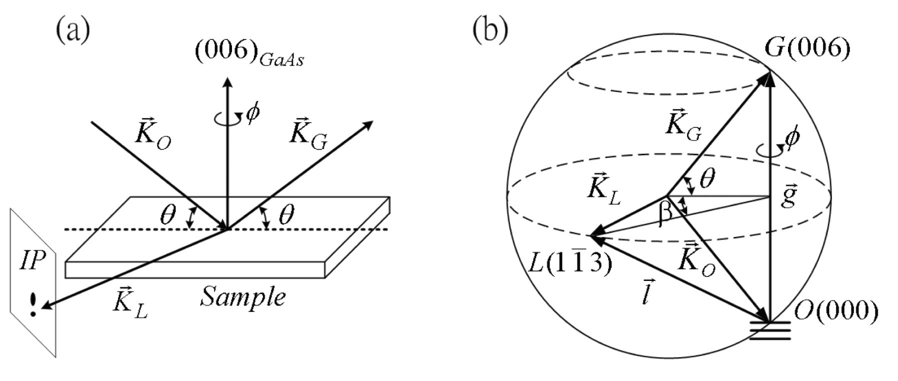

Fig. 1: Bragg-Surface diffraction geometry for GaAs(006)/(1-13): (a) in real space, (b) in reciprocal space.

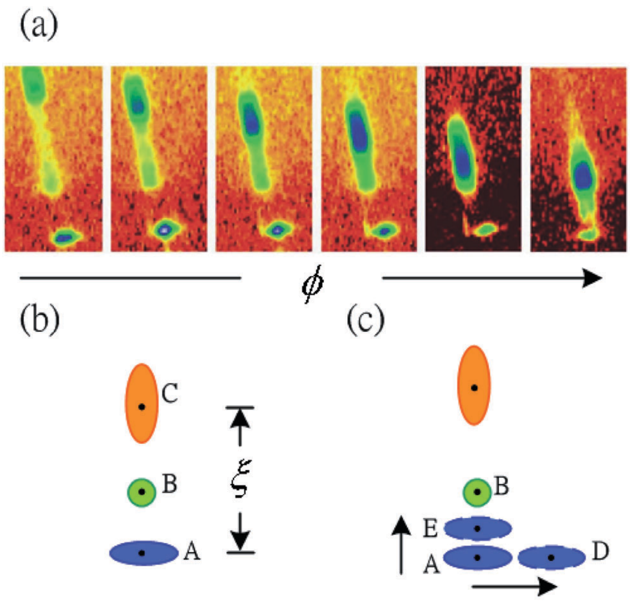

Fig. 2: (a) Diffraction images of GaAs(1-13) vs. f (f=37.554˚ for the first image, 0.02˚ per step in f); (b) Schematic of the diffuse surface scattering images from the interface and the GaAs diffraction spot A. B is the interfacial diffraction spot due to internal total reflection and C is the maximum of the diffuse images at a given f angle; (c) The movements of spot A, A® D and A® E, for varying f and q, respectively.

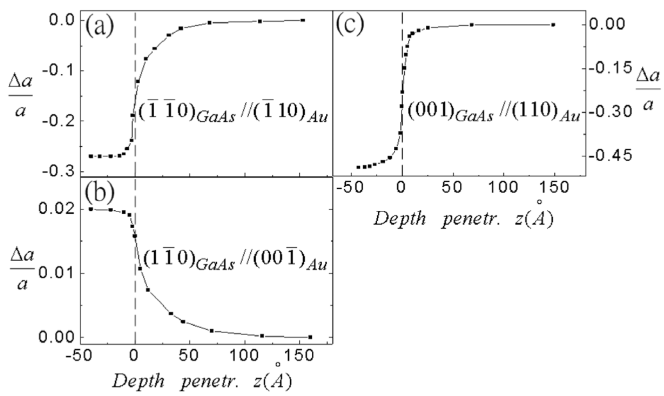

Fig. 3: Lattice-constant variations and strains parallel and perpendicular to the interface with respect to the X-ray penetration depth. z £ 0: the Au thin film; z ³ 0: the GaAs substrate. The resolution in depth is 2.0 Å.

strains along the directions GaAs[-1-10] and GaAs[1-10] parallel and GaAs[001] normal to the interface for both the Au film and GaAs substrate as functions of the depth z. z ≤ 0 is the Au thin film and z ≥ 0， for the GaAs substrate. Similar to the structure of a quantum dot, the strained substrate crystal near the interface can be considered as an ensemble of iso-strained layers with different strain in a different layer. The closer the layer to the interface is, the larger the strain. Fig.3a and Fig.3b are obtained from the measurements of three-beam Bragg-surface diffractions, (000) (006)(-1-13) and (000)(006)(1-13), together with Au (-2-20) and (2-20) GIXD, respectively. The lattice distortions occur in the range from 65Å in the substrate below the interface to 7Å in the film above the interface. The distortion about -27% with respect to GaAs along GaAs [-1-10] is of one order of magnitude larger than that (2%) along GaAs [1-10]. Figure 3c is obtained from GaAs (006) and Au (-2-20) Bragg reflections. The lattice-constant variation about -49% with respect to GaAs normal to the interface is rather abrupt, which occurs about 10 Å in the substrate below and 8 Å in the film above the interface (Fig.3c).

In conclusion, we have demonstrated an X-ray diffraction method, which is capable of determining strain field of interfaces in epilayer/crystal sample systems. This method can be equally applied to other nanoscale single-crystal sample systems, like thin films, multilayers, quantum dots, etc., of any combinations in refractive indices, without invoking total reflection.

Experimental Station

Eight-circle diffractometer

Publications

W.-C. Sun, H.-C. Chang, P.-K. Wu, Y.-J. Chen, C.-H. Chu, M.-T. Tang, Yu. P. Stetsko, M. Hong, and S.-L. Chang, Appl. Phys. Lett. **89**, 091915 (2006).

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