



Evaluation of critical stress intensity for crack initiation and rising R-curve behavior in wurtzitic AlN film grown on (001)Si substrate

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ABSTRACT

Raman spectroscopy and scanning electron microscopy, combined with the Vickers indentation method, were applied to analyze the fracture behavior of a thin (i.e., 1 μm in thickness) aluminum nitride (AlN) film deposited on a (001)Si substrate. A series of indentations were introduced in the AlN/Si system with applying gradually increasing loads, and the stress intensity factor, K_{R} , stored at the tip of cracks propagated from the indentation corners was determined according to the shift of selected Raman bands from wurtzitic AlN in response to the crack-tip residual stress field. A steeply rising crack resistance curve was found in the AlN film, starting from an intrinsic toughness, $K_{\text{I0}} = 0.6 \text{ MPa m}^{1/2}$, for crack initiation up to $K_{\text{R}} \approx 5 \text{ MPa m}^{1/2}$ (at a crack length of $\sim 120 \mu\text{m}$). Such rising R-curve behavior was attributed to the presence of a compressive residual stress field stored in the AlN film. The results obtained by Raman spectroscopy were consistently supported by direct crack opening displacement measurements in a scanning electron microscope.

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1. Introduction

Owing to its superior mechanical, thermal and optical properties, aluminum nitride (AlN) has great potential for applications in high-power high frequency electronics, UV/blue-light emitting devices and photo-detectors, as well as film bulk acoustic resonators [1–3]. Because of an intrinsic difficulty in growing bulk AlN single-crystals, all these applications are based on the growth of high quality epitaxial AlN films on suitable substrates, such as sapphire, silicon carbide or silicon [4–6]. Films eventually become subjected to impacts, abrasive and compressive loading during processing and/or packaging, which may induce film delamination and buckling and, thus, significantly degrade the lifetime and the performances of the devices in which they are embedded. Therefore, from the viewpoint of device fabrication, a better understanding of the mechanical stress state in the film is required, in addition to the commonly monitored electrical performance. Recently, the hardness and the Young's modulus of both AlN films and bulk materials have been evaluated by means of Vickers and Berkovich nanoindentations [7,8].

In this study, we attempted to analyze the fracture toughness of an AlN film deposited on a (001)Si substrate by combined Raman spectroscopy and crack opening displacement (COD) methods applied to Vickers indentation cracks. A series of indentations were introduced

in the AlN/Si system and a steeply rising crack resistance curve was found in the AlN film. The obtained results by Raman spectroscopy were further supported by direct COD measurements.

2. Experimental details

The investigated AlN film was grown to a thickness of $\sim 1 \mu\text{m}$ on a Si(001) wafer by reactive magnetron sputtering at a deposition temperature of 400°C [3]. A series of indentation prints were produced on the film surface by a diamond-tip Vickers indenter (AVK-C1, Akashi Co., Tokyo, Japan) with applying different loads, $P = 3, 5, 10, 20$, and 50 N , respectively. When $P < 20 \text{ N}$, half-penny-shaped near-surface cracks were generated at the corners of the indentation print, which extended beyond the full thickness of the film through the Si substrate, and preferentially propagated along the $\langle 110 \rangle$ direction of the Si substrate. However, at $P = 50 \text{ N}$, film delamination was found at the corners of the indentation print.

Raman spectra were collected at room temperature upon irradiation with an Ar^+ laser (488 nm), by means of a triple monochromator spectrometer (T-64000, ISA Jobin-Yvon/Horiba Group, Kyoto, Japan). Experiments were conducted in a confocal probe configuration with a pinhole aperture of $100 \mu\text{m}$. Spectral lines were analyzed with the aid of a commercially available software package (Labspec 4.02, Horiba/Jobin-Yvon, Kyoto - Japan). Fitting was performed according to Gaussian–Lorentzian spectral modes after subtracting a linear baseline. The mathematical procedures followed for the theoretical calculations were carried out with the aid of commercially available

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computational software (Mathematica 5.2; Wolfram Research Inc., Champaign - IL).

3. Results and discussion

Polarized Raman spectroscopy was first applied to examine the film orientation and to study the crack-tip residual stress fields generated with different applied loads. Fig. 1 shows the polarized Raman spectra of the investigated AlN film, as collected on the top surface and on the cross section of the sample, respectively. In inset to the figure, our choice of Cartesian system in space is also given: the x -axis and the z -axis are parallel to the $[1\bar{1}0]$ and $[001]$ crystallographic directions of the Si substrate, respectively. The Raman spectrum of wurtzitic AlN has already been reported and the details of the assignment of its bands are available in the published literature [9,10]. As can be seen from Fig. 1, three optical modes, i.e. 609 cm^{-1} A_1 (TO) mode, 895 cm^{-1} A_1 (LO) mode, and 655 cm^{-1} E_2 (high) mode, are observable in $z(xx)\bar{z}$ polarization configuration, while only the 246 cm^{-1} E_2 (low) mode and the 655 cm^{-1} E_2 (high) mode can be observed in $z(xy)\bar{z}$ polarization configuration. This change in the appearance of Raman line in the spectrum is a direct consequence of the Raman selection rules for the wurtzitic structure. Moreover, upon rotating the specimen, the intensities of the observed Raman bands were found to be invariant. Therefore, it was confirmed that the top surface of the grown AlN epilayer represents its (0001) plane. In fact, deposition of AlN films on (001)Si was reported to correspond to highly $\langle 0001 \rangle$ oriented textured polycrystalline wurtzitic AlN [11]. A cross section of the film (corresponding to the $(1\bar{1}0)$ plane of Si) was also studied. Observation of the A_1 (TO) mode in $x(zz)\bar{x}$ configuration and of the E_1 mode in $x(yz)\bar{x}$ configuration suggests that the normal direction to the investigated cross section of the film corresponded in average to the crystallographic axis $[11\bar{2}0]$ of the hexagonal structure (i.e., $x/[11\bar{2}0]$). The deposition relationship is thus given by $[0001]\text{AlN}/[001]\text{Si}$, $[10\bar{1}0]\text{AlN}/[110]\text{Si}$, and $[11\bar{2}0]\text{AlN}/[1\bar{1}0]\text{Si}$, which was supported by the results obtained by x-ray diffraction patterns and selected area electron diffraction patterns [6,12]. Consequently, the generated crack directions are along AlN $[11\bar{2}0]$ or AlN $[10\bar{1}0]$. A spectral line scanning along the crack propagation direction was performed, starting from an arbitrary position behind the observed crack tip. Fig. 2 shows the variation of

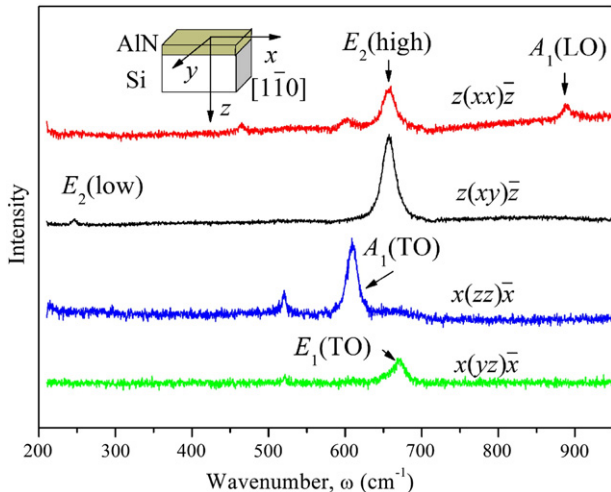


Fig. 1. Typical polarized Raman spectra of the investigated AlN film.

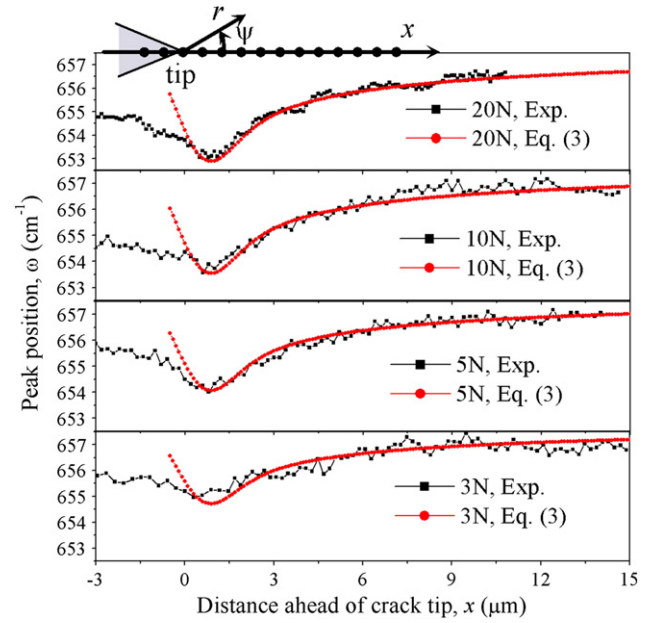


Fig. 2. Variation of the spectral position of the E_2 (high) mode of AlN with the distance from the crack tip.

the spectral position, ω , of the E_2 (high) mode of AlN as a function of distance, x , from the crack tip along a direction straight ahead of the crack propagation path. Spectral profiles collected on cracks propagated under different loads showed a similar morphology but different overall amounts of spectral shift, as discussed in detail hereafter.

For brittle materials, the surface stress field developed ahead of the tip of a planar crack propagating along the direction x can be given as [13]:

$$\begin{aligned}\sigma_{xx} &= \frac{K_{IC}}{\sqrt{2\pi r}} \cos \frac{\psi}{2} \left[1 - \sin \frac{\psi}{2} \sin \frac{3\psi}{2} \right] \\ \sigma_{yy} &= \frac{K_{IC}}{\sqrt{2\pi r}} \cos \frac{\psi}{2} \left[1 + \sin \frac{\psi}{2} \sin \frac{3\psi}{2} \right] + \text{higher-order terms} \\ \tau_{xy} &= \frac{K_{IC}}{\sqrt{2\pi r}} \sin \frac{\psi}{2} \cos \frac{3\psi}{2}\end{aligned}\quad (1)$$

where, K_{IC} is the crack-tip stress intensity factor and r and ψ are the polar coordinates with radial and angular origins at the crack tip and in correspondence of the crack propagation direction, respectively (in the case of $\psi = 0$, $r \equiv x$, cf. draft in Fig. 2). Since the surface dimension of the sample is sufficiently large as compared to crack length, the crack tip near the surface of the specimen is embedded in a plane-stress state ($\sigma_{zz} = \sigma_{33} = 0$) and, straight ahead of the crack tip ($\psi = 0$), an equi-biaxial stress state exists ($\sigma_{xx} = \sigma_{yy} = \sigma_{11} = \sigma_{22} = \frac{K_{IC}}{\sqrt{2\pi r}}$, $\tau_{xy} = 0$) along the direction of crack propagation. The subscripts 1, 2 and 3 locate here the crystallographic axes, $[11\bar{2}0]$, $[10\bar{1}0]$, and $[0001]$ of the wurtzitic structure, respectively.

In a strained wurtzitic AlN crystal, the frequencies of the zone-center optical phonons are shifted with respect to a reference strain-free value, ω_0 , and the shifts in response to principal strain components can be given in a linear approximation, as follows [14]:

$$\Delta\omega = \omega - \omega_0 = a_\lambda(\varepsilon_{11} + \varepsilon_{22}) + b_\lambda\varepsilon_{33} = \tilde{a}_\lambda(\sigma_{11} + \sigma_{22}) + \tilde{b}_\lambda\sigma_{33}\quad (2)$$

where the coefficients a_λ , b_λ and $\tilde{a}_\lambda$, $\tilde{b}_\lambda$ are the so-called phonon deformation potentials for the selected vibrational band of the wurtzitic AlN structure. The values of a_λ ($\tilde{a}_\lambda$) and b_λ ($\tilde{b}_\lambda$) for the Raman modes have been calibrated [15–18] and were given as: $a_\lambda = -1134\text{ cm}^{-1}$

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