



AlN:Cr thin films synthesized by pulsed laser deposition: Studies by X-ray diffraction and spectroscopic ellipsometry

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ABSTRACT

The structure and optical properties of AlN thin films doped with Cr atoms were studied by X-ray diffractometry, Fourier transform infrared spectroscopy and spectroscopic ellipsometry analyses. The films were synthesized by pulsed laser deposition from an AlN:Cr (10% Cr) target onto Si(1 0 0) wafers in vacuum at residual pressure of 10^{-3} Pa or in nitrogen at a dynamic pressure of 0.1 Pa. The study of the XRD patterns revealed that both phases co-existed in the synthesized films and that the amorphous one was prevalent. Two different amorphous matrices, i.e. two types of chemical bond arrangements, were found in films deposited at 0.1 Pa N_2 . By difference, deposition in vacuum resulted in the coexistence of hexagonal and cubic crystallites embedded into an amorphous matrix. The introduction of Cr atoms into the AlN lattice causes a broadening of the IR spectrum along with the shift toward higher wavenumbers of the characteristic Al–N bands at 2351 cm^{-1} and 665 cm^{-1} , respectively. This was related to the generation of a compressive stress inside films. In comparison to the optical constants of pure AlN films, the synthesized AlN:Cr films exhibited a smaller refractive index and showed a weak absorption throughout the 300–800 nm spectral region, characteristic to amorphous AlN structure.

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1. Introduction

Many spintronic application perspectives are envisaged for diluted magnetic semiconductors, where cationic substitutional doping of the semiconducting oxides and nitrides with magnetic impurities leads to intrinsic ferromagnetic material with relatively high Curie temperature [1]. Room-temperature magnetism was reported in several studies of Cr- and Mn-doped AlN semiconducting films [2–8]. Different techniques were applied for film synthesis, among which reactive magnetron sputtering and co-sputtering of Cr and Al targets [2–4], molecular beam epitaxy [5,6], pulsed laser deposition (PLD) [7] and ion implantation [8]. Mn and Cr dopants have cubic structure and antiferromagnetic spin–spin coupling. CrN is also antiferromagnetic, while Cr oxides are ferromagnetic. Cr has one electron in the conduction band, while Mn has two electrons. Both atoms are heavier than Al, and have larger ion radius, causing the lattice broadening. The solubility of Cr in AlN was found to be much higher than that of Mn [6], so that larger Cr concentration in Cr-doped AlN has been achieved [2]. The

magnetic and transport properties of Cr-doped AlN depend on the structure, concentration of defects and carriers.

We report herewith new structural study of AlN films doped with Cr atoms, synthesized by PLD, by applying X-ray diffractometry (XRD), Fourier transform infrared spectroscopy (FTIR) and spectroscopic ellipsometry (SE) analyzes. The effect of the ambient gas was considered as depositions were performed either in vacuum or nitrogen gas ambient.

2. Materials and methods

Thin AlN films doped with Cr were synthesized on single crystalline Si(1 0 0) substrates by pulsed laser deposition. The PLD targets were prepared by homogeneously mixing AlN and Cr (10% at.) powders, followed by pressing and sintering. The films were grown using a PLD installation consisting of a KrF* ($\lambda = 248\text{ nm}$) excimer laser source generating pulses of $\tau_{\text{FWHM}} \leq 25\text{ ns}$ at a frequency repetition rate of 10 Hz and a deposition chamber (Fig. 1). During ablation the target was rotated and translated along two orthogonal axes to avoid piercing and to improve the morphology of deposited films. The incident laser beam was incident at 45° , while ablation plasma was evolving normally to target surface. The separation distance target–substrate was set to 5 cm. After loading the substrate and target, the deposition

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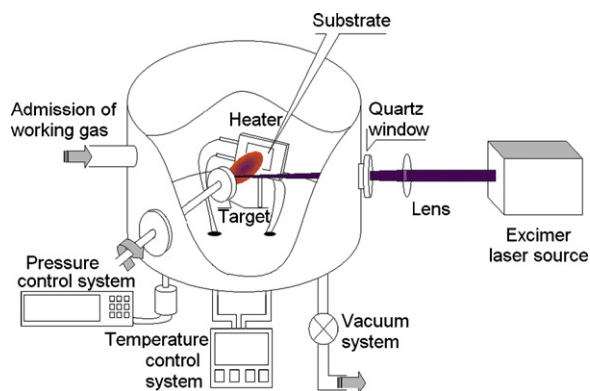


Fig. 1. PLD experimental setup used in our experiments.

chamber was pumped down to a residual pressure of 5.0×10^{-4} Pa. Before deposition, the substrate was heated up to 800°C for 1 h, aiming at decomposition of native SiO_2 on the Si surface. The substrate temperature was kept at 800°C during deposition, providing conditions for crystalline growth of AlN. The incident laser fluence on target was 10 J/cm^2 . For the deposition of one film 15,000 subsequent laser pulses were applied. The corresponding thickness of obtained structures was $\sim 300\text{ nm}$ as measured by profilometry. The synthesis of AlN:Cr films was undertaken either in vacuum (residual gas pressure of 10^{-3} Pa) or in nitrogen gas ambient at a low pressure of 0.1 Pa.

Information on the crystalline phases in deposited films was acquired by large-angle ($2\theta = 0\text{--}90^\circ$) XRD measurements using Philips X'Pert equipment. FTIR reflectance spectra were measured on Shimadzu IR Prestige-21 spectrometer within the range $4000\text{--}400\text{ cm}^{-1}$. Ellipsometric measurements were carried out with a Rudolph 436 ellipsometer within the spectral range of $300\text{--}800\text{ nm}$ at an incidence angle of 50° . The obtained optical dispersion spectra were analyzed by Bruggemann effective medium approximation (BEMA) revealing the film composition.

3. Results and discussion

3.1. XRD investigations

The analysis of the XRD patterns presented in Fig. 2, evidenced that different phases co-existed in synthesized films and that the amorphous phase was dominant. In case of structures deposited in 10^{-3} Pa, the Bragg peak positions in XRD spectrum (Fig. 2a) reveal that a certain degree of crystallization is reached inside prevalent amorphous matrix. The crystallites are in both hexagonal and cubic phases. The co-existence of two crystalline phases is in good accordance with our previous studies of un-doped AlN films [9]. However, by contrast with pure AlN which is polycrystalline with

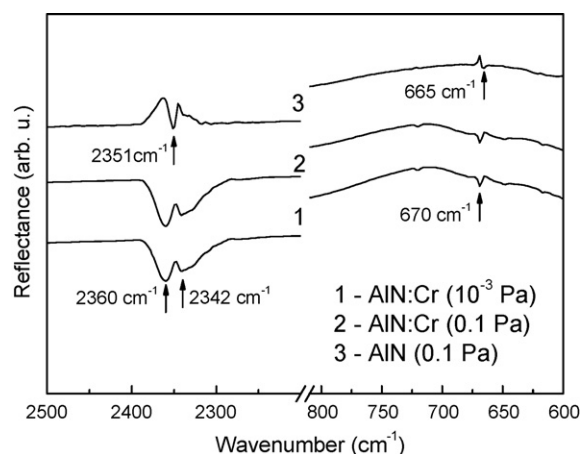


Fig. 3. IR reflectance spectra of PLD Cr-doped AlN films, deposited in vacuum at a residual gas pressure of 10^{-3} Pa (Curve 1) and in low-pressure N_2 of 0.1 Pa (Curve 2). For comparison, the IR spectrum of the un-doped AlN film deposited at 0.1 Pa (Curve 3) is shown.

dominant cubic phase crystallites, in case of Cr-doped films the dominant crystalline phase is hexagonal. According to the ASTM data [10], the diffraction peak around 33.04° is attributed to the crystallographic plane (1 0 0) of hexagonal phase with lattice constants $a = 3.111\text{ \AA}$ and $c = 4.979\text{ \AA}$ (JCPDS 25-1133), while the diffraction peaks around 38.19° and 44.37° correspond to crystallographic planes (1 0 0) and (0 0 2) of cubic phase with a lattice constant $a = 4.045\text{ \AA}$ (JCPDS 46-1200).

Deposition in nitrogen ambient at low gas pressure (0.1 Pa) yields amorphous films; no sharp peaks related to crystalline phases are visible in the XRD spectrum (Fig. 2b). The observed two broad and diffuse humps centered at 33° and 37° , respectively, suggest the formation of different amorphous environments, probably caused by various chemical bond arrangements.

3.2. FTIR data

Fig. 3 presents the IR reflectance spectra of the Cr-doped AlN films (Curves 1 and 2), compared to the pure AlN films synthesized by PLD under identical conditions (Curve 3). The characteristic features at 2351 cm^{-1} for pure AlN film could be related to vibrational excitations of polycrystalline AlN [11]. The feature occurring at $670\text{--}665\text{ cm}^{-1}$ is associated with the $E_1(\text{TO})$ phonons excitation in AlN films [12,13], while the very weak feature appearing around 619 cm^{-1} could be related to $A_1(\text{TO})$ vibrational mode of Al–N bonds [14]. If we assume that the zero-stress energy position is at 657 cm^{-1} [12] and an 8 cm^{-1} shift corresponds to 1 GPa stress (as reported in [15]), we estimate a compressive residual stress in pure AlN films reaching $\sim 1\text{ GPa}$.

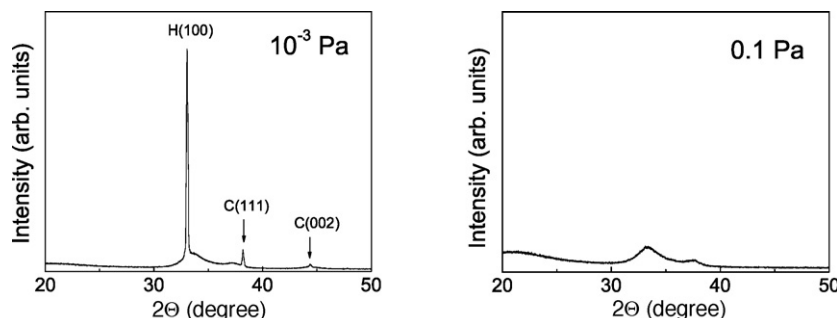


Fig. 2. Typical XRD patterns of AlN:Cr films deposited in residual gas pressure of 10^{-3} Pa (a) and in low-pressure N_2 of 0.1 Pa (b).

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