Optics & Laser Technology 69 (2015) 92–103

Contents lists available at ScienceDirect

Optics & Laser Technology

journal homepage: www.elsevier.com/locate/optlastec

Deposition pressure effect on chemical, morphological and optical properties of binary Al-nitrides

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ARTICLE INFO

Article history:
Received 17 August 2014
Received in revised form
3 December 2014
Accepted 8 December 2014
Available online 13 January 2015

Keywords:
Pulsed laser deposition
Aluminum nitride
Morphology and optical properties

ABSTRACT

Aluminum nitride films (AlN) were produced by Nd:YAG pulsed laser (PLD), with repetition rate of 10 Hz. The laser interaction on Al target under nitrogen gas atmosphere generates plasma which is produced at room temperature with variation in the pressure work from 0.39 Pa to 1.5 Pa thus producing different AlN films. In this sense the dependency of optical properties with the pressure of deposition was studied. The plasma generated at different pressures was characterized by optical emission spectroscopy (OES). Additionally ionic and atomic species from the emission spectra obtained were observed. The plume electronic temperature has been determined by assuming a local thermodynamic equilibrium of the emitting species. Finally the electronic temperature was calculated with Boltzmann plot from relative intensities of spectral lines. The morphology and composition of the films were studied using atomic force microscopy (AFM), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy analysis (XPS) and Raman Spectroscopy. The optical reflectance spectra and color coordinates of the films were obtained by optical spectral reflectometry technique in the range from 400 nm to 900 nm. A clear dependence in morphological properties and optical properties, as a function of the applied deposition pressure, was found in this work which offers a novel application in optoelectronic industry.

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1. Introduction

Aluminum nitride (AlN) thin films are applied widespread because they have some excellent properties such as chemical stability, high thermal conductivity, low electric conductivity and wide band gap (6.2 eV). Moreover, it presents a thermal expansion coefficient similar to that of GaAs, and a higher acoustic velocity, making it excellent for optical devices in the ultraviolet spectral region, acoustic optic devices, and surface acoustic wave (SAW) devices. Polycrystalline films exhibit piezoelectric properties and can be used for the transduction of both bulk and surface acoustic waves. Pulsed laser deposition (PLD) growth of AlN films is rather critical because of its tendency to present micro-cracking. This tendency is more evident with increasing the thickness of the film and when using silicon substrates, particularly in the (100) orientation, while using silicon substrates has been shown to improve the films' growth. Pulsed laser deposition (PLD) using nanosecond pulses is considered to be one of the most promising techniques for the synthesis and deposition of thin films [1–4]. This method has advantages such as high reproducibility, control of the film growth rate and stoichiometry and low impurity concentration in the composition of deposited films. On the other hand aluminum nitride (AlN) exhibits attractive properties such as thermal and chemical stability, high thermal conductivity, high dielectric permittivity, breakdown field, high-speed piezoacoustic wave and mechanical hardness [1].

Many authors in the literature have discussed the effect of growth conditions of AlN thin films deposited by PLD related to the crystallinity, morphology and optical response [5–8]. Clearly, the growth characteristics influence the final properties of the materials in a thin layer, but there is a deficiency in the discussion of the effect produced by the variation of the pressure tank to the variation in color purity layered AlN obtained by PLD.

The study of pulsed laser ablation plumes has increased the attention recently due to its importance in laser deposition. The plasma state is often called the fourth state of matter and transient phenomenon in nature with characteristic parameters dependent

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http://dx.doi.org/10.1016/j.optlastec.2014.12.009
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on the rapidly evolving component species. These parameters are highly dependent on the irradiation conditions, laser intensity, pulse duration, wavelength, composition and atmosphere. Taking into account that the relationship between plasma and morphological quality in the films is very important, in this sense the AlN films are used as substrates for SAW sensors where the surface quality is a decisive factor in the sensors performance [9,10].

So, the goal of this work is to study the effect of the applied deposition pressure on the chemical, morphological properties and optical properties of binary AlN films deposited by PLD on Si (100) for use in optical and electronic applications. Here, using nitrogen as working gas, results on AlN films deposited from Al targets, their characterization by X-ray photoelectron spectroscopy (XPS), Raman Spectroscopy and scanning electron microscopy (SEM) as well as investigations associated to changes in optical response such as reflectance and color purity as function of pressure deposition values were reported.

2. Experimental

In this research the experiments were carried out in usual PLD configuration consisting of a laser system into the multiport stainless steel vacuum chamber equipped with a gas inlet, a rotating target and a heated substrate holder. The Nd:YAG laser that provides pulses at the wavelength of 1064 nm with 9 ns pulse duration and a repetition rate of 10 Hz was used. The laser beam was focused with an $f=23$ cm glass lens on the target at the angle of 45°, with respect to the normal. The target was rotated to 2.2 rpm to avoid fast drilling. The distance between the target and the substrate was 6.5 cm. The vacuum chamber was evacuated down to 10^{-6} Pa before deposition by using a turbo-molecular pump backed with a rotary pump. The AlN thin films were deposited in nitrogen atmosphere as working gas, in an atmosphere of nitrogen reactive, the nitrogen gas pressure varied between 0.39 Pa and 1.5 Pa and aluminum target (99.99%). The films were deposited with a laser fluence of 7 J/cm^2 for 15 min on silicon (100) substrates. So, the plasma characterization was performed by optical emission spectroscopy (OES) by using a spectrometer model Jobin Yvon Triax 550 of 0.55 m, $f=6.4$ equipped with two gratings of 1200 l/mm and 150 l/mm, coupled to a CCD camera model 3000 air-cooled multi-channel and 512×512 pixels. The crystal structure of the coating was determined by using a D8 Advance Bruker X-ray diffractometer with Cu-Ka ($\lambda=1.5405$ Å) radiation. For the surface study a scanning electron microscope Philips XL 30 was used . The AlN layers thickness around 150 nm was determined by the design of a step between the substrate and the film. A profiler was used to perform continuous scanning surface that takes into account the film and the substrate area. A Dektak 8000 profilometer device with a tip diameter of 12 ± 0.04 μm, scan length range (X) of 50 ± 0.1 μm–200 ± 0.1 mm, scan height range (Y) from 100 ± 1 nm to 1000 ± 0.1 μm, measurement range 50 A–2.520 kA, vertical resolution (max.) of 1 A, sample thickness (max.) of 63.5 mm, horizontal resolution of 0.0033 um, stylus force from 1 to 100 mg and sample stage theta rotation of 360° was used.

Chemical composition analysis of the coatings was done with a Philips XL 30 FEG scanning electron microscope, an X-ray detector and secondary electrons detector of Lithium Beryllium inside the chamber with the purpose of amplifying the signal in the EDS analysis. Moreover, the XPS also was used on AlN samples to determine the chemical composition and the bonding of aluminum and nitrogen atoms using ESCA-PHI 5500 monochromatic Al-Kα radiation and a passing energy of 0.1 eV. The surface sensitivity of this technique is so high that any contamination can produce deviations from the real chemical composition; therefore, the XPS analysis is typically performed under ultra-high vacuum conditions with a sputter cleaning source to remove any undesired contaminants. Morphological characteristics of the coatings like grain size and roughness were obtained using an atomic force microscope (AFM) from Asylum Research MFP-3Dr and calculated by a scanning probe image processor (SPIP) which is the standard program for processing and presenting AFM data, therefore, this software has become the de-facto standard for image processing in nanoscale. The Al–N bond was verified by infrared spectroscopy and Raman and Fourier transform infrared spectroscopy (FTIR) characteristics of Al–N vibrational modes were found. Optical reflectance spectra and color coordinates of the samples were obtained by spectral reflectometry in the range of 400–900 nm by means of an Ocean Optics 2000 spectrophotometer. The coated samples received the white light from a halogen lamp illuminator through a bundle of six optical fibers, and the light reflected on the samples was collected by a single optical fiber and analyzed in the spectrophotometer. The fiber was fixed in perpendicular direction to the sample surface. An aluminum deposited by rapid thermal evaporation in high vacuum was used as the reference sample, and the experimental spectra were normalized to 100% reflectance of the reference sample. The morphology on AlN surface films was analyzed by SEM (Leika 360 Cambridge Instruments).

3. Results and discussion

3.1. Optical emission for the AlN plume

For the plasma generated by AlN materials a large number of emission lines attributed to emission bands of aluminum nitride was identified. In Fig. 1a, the most intense lines are emission of aluminum species, apparently the main species emitted in the ablation of aluminum species, being once ionized aluminum (Al II). The strongest lines in the spectrum of XII in plasma are at 631.337 nm, for electron configuration 1s^{3}p–1s^{3}d. Atomic spectral lines are also indicating the presence of Al and atomic N$_2$ (Fig. 1b and c). The oxygen presence was observed in optical emission for the AlN with 0.53 Pa and 0.66 Pa, which is a product of contamination in the vacuum chamber. All atomic emission lines were identified through the database of the National Institute of Standards and Technology-NIST. Also emission lines of nitrogen species (neutral and multiply ionized) with most intense peaks at 618.909 nm (N I), 644.902 nm (N III), 740.359 nm (N III) were observed. The emission peak of atomic nitrogen was dominant compared to the emission peaks of atomic aluminum.

The oxygen presence is also attributed to low flow of nitrogen gas during the degassing processes. The oxygen species observed are O II (762.882 nm), O III (751.325 nm) and O V (676.585 nm and 743.153 nm). Shown in 509.985 nm an emission band of AlN (0.0) [11] is observed. A second emission band, weaker, is analyzed at 523.060 nm for AlN (1.0) [8]. In this work the oxygen bands only are evidenced for a working pressure of 0.53 Pa. Moreover, in this work participle density in Debye sphere $\mathrm{Nd}=2.46\times10^{-1}$ m was found.

3.2. Local thermodynamic equilibrium for AlN films

In the local thermodynamic equilibrium for AlN it is possible to take into account that the plume is in local thermodynamic equilibrium (LTE) [12], therefore, the emission line intensity (I) in a specific wavelength (λ_m) may be expressed by

$$\mathrm{Ln}\left(\frac{I_{mn}\lambda_{mn}}{A_{mn}g_{mn}}\right)=\mathrm{Ln}\left(\frac{N}{Z}\right)-\left(\frac{E_{mn}}{\kappa T_e}\right)\tag{1}$$

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