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## Using AlN Coatings to Protect the Surface of AlGaAs/GaAs System Heterostructures from Interaction with Atmospheric Oxygen

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**Abstract**—Thin aluminum nitride (AlN) films have been synthesized by reactive ion–plasma sputtering (RIPS) and their properties have been studied in view of using this method for obtaining protective coatings on output facets of high-power semiconductor laser cavities based on  $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$  heterostructures. Investigations by energy-dispersive X-ray spectroscopy and ellipsometry techniques showed that, at a residual pressure of  $\sim 10^{-5}$  Torr, a layer of aluminum oxynitride is formed and the film–substrate heteroboundary may experience to oxidation. However, AlN films with thicknesses on the order of 100 nm grown in pure nitrogen at a residual pressure of  $\sim 10^{-7}$  Torr were evidently free of oxygen and could reliably prevent its penetration to the region of heteroboundary.

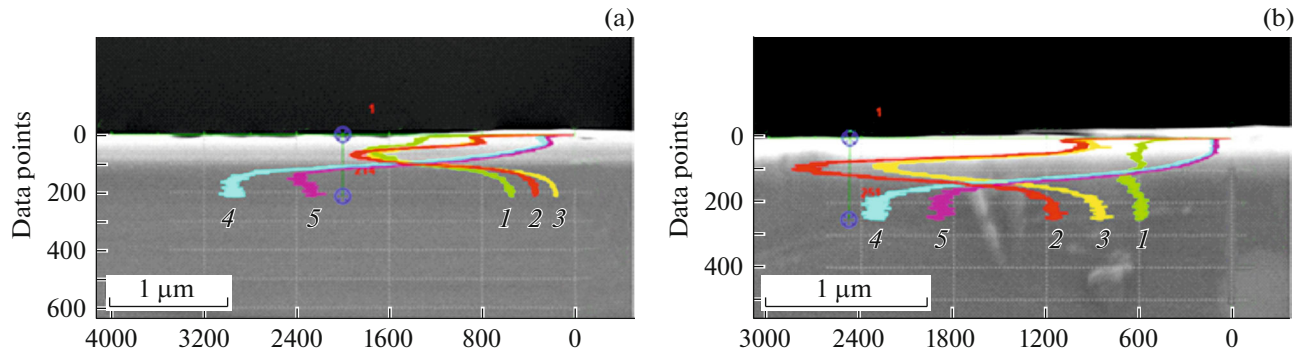
**Keywords:** semiconductor lasers, thin films, passivation, aluminum nitride, degradation of lasers.

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Semiconductor lasers are finding increasing application in various fields, but the continuous development of technologies is leading to increasing requirements to these devices. The reliability of operation and long working life are among the main requirements to modern semiconductor lasers. These properties are especially important for high-power lasers, which are subject to extremely high loads related to large densities of generated radiation power. In this respect, a special problem is caused by the catastrophic optical mirror damage (COMD) that leads to rapid drop in the laser output power and device breakdown [1–3]. One possible method to protect mirrors from degradation consists in passivation of the heterostructure output facets, e.g., by chemical treatment and/or deposition of special coatings that prevent interaction of the cavity surface with the atmosphere [4–7]. At the same time, effective laser operation usually requires the application of optical coatings, either reflective or antireflective, which ensure the effective lasing and output of generated radiation [8].

There are variants of applying several layers of various materials, one of which produces the passivating effect and the other of which provides for the neces-

sary reflection coefficient [1, 6]. It is also possible to produce cleavage of the laser heterostructure in vacuum with subsequent deposition of an antireflection coating without exposure to air [5, 7]. These measures can be effective, but they significantly complicate the process of laser manufacturing. Widely used materials for reflective or antireflective coatings include  $\text{Al}_2\text{O}_3$  and  $\text{Si}/\text{SiO}_2$ . Unfortunately, although the corresponding deposition technologies are simple and well developed, both these materials are insufficiently effective for the passivation of AlGaAs system heterostructures, since they contain oxygen. As is known [9], aluminum is subject to oxidation in the region of AlGaAs laser waveguide. Surface oxidation can lead to the appearance of defects in the waveguide region that result in the parasitic absorption of laser radiation, nonradiative recombination, and local heating of the laser waveguide region. The initial defects grow up as a result of heating, which leads to COMD and laser degradation [3]. Therefore, development of a coating capable of simultaneously performing the antireflective and protective functions for mirrors of high-power semiconductor lasers of the AlGaAs system is a topical and important task.



**Fig. 1.** SEM images of freshly cleaved cross sections of samples nos. (a) 1 and (b) 2 (see Table 1), showing scan lines and the corresponding relative concentration–depth profiles of elements: (1) O, (2) Al, (3) N, (4) Ga, and (5) As.

The present work was devoted to developing the synthesis of aluminum nitride (AlN) films by reactive ion-plasma sputtering (RIPS), studying their properties, and assessing the possibility of using these films as protective coatings for cavities of high-power AlGaAs/GaAs semiconductor lasers.

AlN films were obtained by reactive ion-plasma sputtering (RIPS) of pure aluminum target in special-purity  $N_2$  medium free of any other gases in a vacuum triode sputtering system. The films were deposited onto epi-ready (100)-oriented  $n$ -GaAs substrates. Prior to deposition, the working chamber was evacuated and the substrate surface was cleaned by ion etching in  $Ar^+$  plasma at a  $-20$ -V negative potential difference on the sample. The Al target was also etched in argon plasma for removal of the natural oxide film. The residual gas pressure during this preliminary cleaning and subsequent deposition stages was maintained on a level of  $(2-4) \times 10^{-3}$  Torr. The purity of working gases and Al target was no less than 99.999%.

A series of samples for investigation were prepared at residual gas pressures ranging from  $\sim 5 \times 10^{-5}$  to  $\sim 7 \times 10^{-7}$  Torr. After deposition, thickness  $d$  and refractive index  $n$  of the films were measured using LEF-3M-1 ellipsometer at a wavelength of 633 nm. The film thicknesses were additionally verified using scanning electron microscopy (SEM) techniques on Carl Zeiss Supra 25 instrument. Subsequently, the as-prepared samples were stored at room temperature under ambient air conditions for no less than half a year and then subjected to repeated ellipsometric investigation and qualitative estimation of chemical

composition with energy-dispersive X-ray (EDX) spectroscopy techniques. The EDX measurements were performed at an accelerating voltage of 5 keV with an EDAX Octane Elite Super detector on a JEOL 7900F SEM instrument. The samples were scanned across a freshly cleaved edge surface as shown in Figs. 1a and 1b. This approach allowed the concentration–depth profiles of elements in the film and substrate to be evaluated, thus gaining a notion of the film–substrate interface. Taking into account the accelerating voltage, the depth of analysis could be estimated as 500–550 nm from the film surface.

Table 1 presents characteristics of the two most representative samples. The results of EDX spectroscopy measurements show that the film–substrate interface and the AlN film proper in sample no. 1 (Fig. 1a) exhibit clearly pronounced gradient of the secondary electron emission intensity. This may indicate that an amorphous aluminum oxynitride sublayer was initially formed on the substrate and then gradually crystallized with increasing film thickness. This behavior, which was previously demonstrated in [10–12], may explain the low refractive index of sample no. 1 relative to the theoretical value ( $\sim 1.74$  versus 2.1–2.4). These partly amorphous films are also characterized by their higher (up to 1.5 times increased) growth rates as compared to those of nanocrystalline films. However, in the case of films grown at a residual gas pressure reduced to  $7 \times 10^{-7}$  Torr (sample no. 2, Fig. 1b), the SEM image shows a greater contrast between the film and substrate, indicating that the secondary electron emission intensity gradient in the film is much less pronounced and suggesting that the

**Table 1.** Characteristics of typical AlN films formed at different residual gas pressures

Sample no.	Residual gas pressure $P_0$ , Torr	Film thickness $d$ , nm	Index of refraction $n$	Film growth rate $v$ , Å/min
1	$5 \times 10^{-5}$	124.7	1.735	12
2	$7 \times 10^{-7}$	123.4	2.115	83

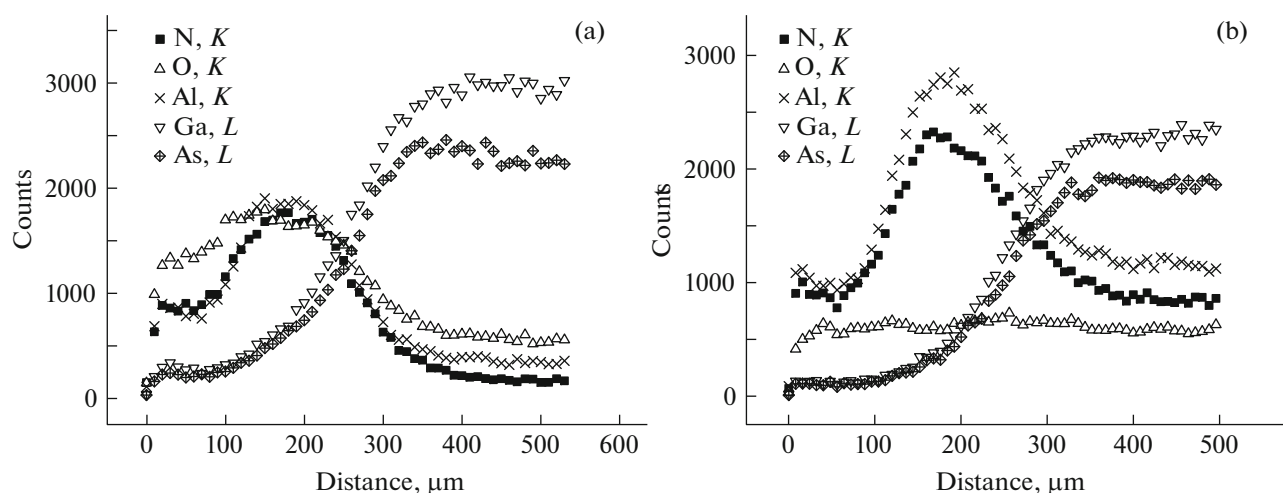


Fig. 2. Intensity–depth profiles of the EDX peaks of elements in samples nos. (a) 1 and (b) 2.

film has a nano- or microcrystalline structure. The index of refraction being close to theoretical also confirms this hypothesis.

Figures 2a and 2b show in-depth profiles of elements plotted as peak intensity versus distance from the surface in samples nos. 1 and 2. As can be seen, sample no. 1 formed at a higher residual gas pressure (Fig. 2a) contains a significant amount of oxygen in a coordinate region corresponding to the AlN film. In addition, the “tail” of the profile of oxygen partly extends to the region of substrate. In sample no. 2 (Fig. 2b), the oxygen peak intensity is almost constant and low in the entire scanned region (apparently representing background noise or a signal of oxygen adsorbed on the surface after cleavage the sample prior to measurements). Thus, oxygen in sample no. 2 is probably present neither in the AlN film nor in the GaAs substrate.

Therefore, it can be concluded that thin-film AlN coatings obtained by the method of RIPS in pure nitrogen atmosphere upon preliminary evacuation of the working chamber to a residual gas pressure of  $\sim 10^{-7}$  Torr retains the barrier properties with respect to atmospheric oxygen for a time period longer than half a year. Taking into account this circumstance, as well as the well-known mechanical strength, chemical stability, and extremely high thermal decomposition temperature of AlN, these films can be used as protective coatings for the facets of high-power semiconductor lasers. Moreover, in some cases, these films possessing a favorable index of refraction can also play the role of antireflection coatings. These possibilities suggest that they may be an alternative to using preliminary chemical passivation, multilayer coatings, or expensive technologies involving vacuum cleavage of laser heterostructures.

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#### CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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