Research of surface morphology of aluminum nitride films obtained by magnetron sputtering method

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Abstract. In this paper, we studied the surface morphology of aluminum nitride films obtained by magnetron sputtering intended for use in electronic devices. The main problem with this method of film formation is the large surface roughness of the film; therefore, the task to be solved is to improve the surface quality of aluminum nitride films by smoothing the surface roughness.

1. Introduction

A promising material in creating electronic devices: filters and microwave delay lines, temperature, pressure sensors, SAW signal processing devices, etc. are films with good piezoelectric and semiconductor properties, as well as high thermal conductivity and chemical inertness. Aluminum films of aluminum nitride are the best among films with a high degree of crystallinity and an ordered structure of the crystalline phase, possessing all of the above properties [1]. Recent studies have shown that for many of the above devices it is more efficient to use layered structures with ordered layers instead of homogeneous materials. Multilayer heat sink substrates consisting of AIN dielectric layers deposited on a metal base are designed for a wide temperature range. In addition, aluminum nitride has the highest speed of propagation of surface acoustic waves among piezoelectric materials, which makes it possible to create filters and microwave delay lines based on layered sound ducts. AIN is also considered one of the most promising materials for creating signal processing devices for SAWs. It has low insertion loss, high electromechanical coupling coefficient (0.8%), and high surfactant velocity ($\approx 6 \cdot 10^3 \text{m/s}$) [1]. Therefore, special attention is paid to the growth of AIN films and the creation of layered structures based on them.

The growth of such films with the ordered structure of each layer on nonorienting amorphous or polycrystalline substrates is possible by ion-plasma and ion sputtering methods. Magnetron sputtering is characterized by nonequilibrium crystallization conditions, when the preferred growth direction is determined by the atomic structure of the surface on which the films grow.

However, aluminum films obtained by thermal, electron beam evaporation or ion sputtering have a significant drawback, they have a developed surface. It was shown that tubercles (knolls) or whiskers can form on the surface of an aluminum film if the temperature coefficients of linear expansion ($\alpha$) of the two materials differ by more than five times [2]. The presence on the surface of tubercles (knolls), whiskers and large grains of aluminum determines the large roughness of the films surface [3].

Thus, in the manufacture of electronic devices based on aluminum nitride films, the problem of producing films with a smooth surface arises. The values of protrusions and depressions (roughness)
on the surface of the films should be minimal. Therefore, it is important to be able to calculate the statistical and physico-chemical characteristics of the surface, to determine the roughness of the formed coatings, its dependence on the deposition parameters. This work presents the results of atomic force microscopy (AFM) and scanning electron microscopy (SEM) studies of the surface of aluminum nitride films obtained by magnetron sputtering, and analyzes the dependence of the surface roughness of the samples on the substrate temperature.

2. Formulation of the problem

The main problem in the formation of aluminum films is the large roughness of their surface, which is especially appeared during magnetron sputtering. The height of the surface roughness of the obtained films can reach 40 ... 50 nm. A bulk acoustic wave is scattered on such surface roughness, which leads to a decrease in the Q factor of resonators based on these films [3].

In the industrial implementation of layered structures based on AlN films, establishing the relationship between the technology for growing AlN films and changing their structure and properties is an important task. In order to form and stability compounds synthesized under strongly nonequilibrium conditions, it is necessary to understand the processes of phase formation and creation of materials with desired properties, the synthesis of which by other methods is impossible. It is necessary to study the texture, phase and elemental composition, surface morphology of the grown films, mechanical stresses in the films, their adhesion. For this purpose, atomic force, scanning electron microscopy, and X-ray diffraction analysis methods are most suitable.

The task in which this work was carried out is to study the possibility of improving the surface quality of aluminum nitride films by smoothing the surface roughness.

3. Theory

Aluminum nitride is a material with covalent bonds, having a hexagonal crystal structure, which is an analog of the structure of zinc sulfide, known as wurtzite, it is a semiconductor with a band gap of 6 eV. The material is resistant to very high temperatures in inert atmospheres. In air, surface oxidation occurs above 700 °C, and surface oxidized layers 5–10 nm thick are formed at room temperature. This layer protects the material from oxidation up to 1370 °C [4].

It is known that aluminum films have a granular structure in the form of columns and voids and are well described by the model of structural zones. According to this model, the structure of the film surface is determined by the temperature of the substrate (Ts). If the value Ts <0.3 × Tm (Tm is the melting temperature of the film material), then a fine-grained structure of the film with a smooth surface is formed, which is typical for the first structural zone. With an increase in the substrate temperature, the mobility of aluminum atoms increases, and the process of formation of the film structure proceeds in the region of the 2nd and 3rd structural zones, where the coarse-crystalline structure of the film is formed and, as a result, the surface roughness of the aluminum film increases. Surface roughness is a combination of surface irregularities with relatively small steps. To separate the surface roughness from other irregularities with relatively large steps (shape deviation and waviness), it is considered within a limited area, the length of which is called the base length [5].

Quantitatively, the surface roughness of the film is conveniently estimated by the standard deviation (Sq) of the protrusions and depressions. A large value of the standard deviation indicates a large spread in the values of the protrusions and depressions on the surface of the aluminum film in comparison with the average value; a small value (Sq), respectively, shows that the values of the protrusions and depressions on the surface of the film are grouped around the average value. It is known that with an increase in the thickness of an aluminum film, the surface roughness increases [5].

These disadvantages are due to the fact that with an increase in the deposition rate of the film, the kinetic energy of the atomized aluminum atoms also increases and their mobility on the substrate increases. This leads to an increase in grain sizes and, as a consequence, to an increase in the surface roughness of the aluminum film [6].
Research methods

Atomic force microscopy (AFM) is a direct method for studying the morphology and local properties of a surface, which makes it possible to obtain a surface topography and calculate its statistical and physicochemical characteristics, which allows non-destructive testing of optical parts in the production of optical elements. The AFM also makes it possible to identify areas with a small difference in elevation with developed macrorelief of the surface, taking into account the peculiarities of image formation in the phase contrast mode.

The surface topography was studied by the AFM method, and the dependences of the surface roughness of the aluminum nitride films obtained by magnetron sputtering on the substrate temperature were analyzed.

Scanning electron microscopy (SEM) is designed to obtain an image of the surface of an object with a high (up to 0.4 nm) spatial resolution, as well as information on the composition, structure and some other properties of near-surface layers.

4. Experimental results

The most promising method for producing AlN films by the structure and properties close to single-crystal is reactive HF magnetron sputtering. This method allows under non-equilibrium conditions to grow AlN layers with an ordered structure on substrates of crystalline amorphous materials at low temperatures (370-570 K) in a mixture of argon and nitrogen at low temperatures. The pressure of the gas mixture during spraying was 0.07 Pa.

Table 1 for various samples presents the variable parameters of the magnetron sputtering process and the controlled parameters of aluminum nitride films (film thickness).

| Parameter | № Sample | Parameters of the magnetron sputtering process | Characteristics of aluminum nitride films |
|-----------|----------|-----------------------------------------------|----------------------------------------|
|           |          | Magnetron power, W | Mixture consumption Ar/N₂, sccm | Substrate temperature, °C | Film thickness, microns |
| Substrate temperature | 99      | 650 | 4/5 | 300 | 0.620 |
|                      | 102     | 650 | 4/5 | 360 | 0.546 |
|                      | 103     | 650 | 4/5 | 380 | 0.546 |
|                      | 104     | 650 | 4/5 | 400 | 0.490 |
| Mixture consumption Ar/N₂ | 107     | 650 | 3,5/5 | 350 | 0.546 |
|                      | 109     | 650 | 2,5/5 | 350 | 0.500 |
|                      | 110     | 650 | 2/5 | 350 | 0.460 |
|                      | 111     | 650 | 4,5/5 | 350 | 0.546 |
| Magnetron power | 116     | 600 | 4/5 | 300 | 0.513 |
|                      | 117     | 700 | 4/5 | 300 | 0.546 |
|                      | 118     | 400 | 4/5 | 300 | 0.468 |
The morphology of the surface of aluminum nitride films was studied by scanning electron microscopy and atomic force microscopy using a Jeol JCM-5700 scanning electron microscope and a Bruker Multimode 8 atomic force microscope, respectively.

The influence of the temperature of formation of thin films of aluminum nitride on the surface roughness was studied.

During AFM measurements, different surface areas were recorded, which made it possible to average the microrelief parameters at different spatial scales (20x20 μm, 5x5 μm and 2x2 μm). Using the obtained images, the value of Sq was calculated - the root mean square roughness (volume), for Ra - the average roughness along the profile (along the line) and for Rq - the mean square roughness along the profile (along the line).

Scanning results for different samples are shown in figures 1, 2. These images reflect the surface topography over an area of 5 x 5 μm. This is the so-called two-dimensional representation of the scan data, in which the heights corresponding to the individual scan points are displayed using the color palette in such a way that darker tones correspond to the troughs, and as the height of the relief increases, the color becomes lighter. The color scale of heights is shown to the right of the scan.

![Figure 1. AFM image of the surface of sample No. 99, 5x5 μm, obtained at a temperature of 300 °C.](image1)

![Figure 2. AFM image of the surface of sample No. 102, 5x5 μm obtained at a temperature of 360°C.](image2)

Using a two-dimensional image of the surface relief of the films, it is possible to estimate the grain size, their uniform distribution over the surface, and the uniformity of their shape and size (figure 3).

To measure the roughness parameters of the studied areas, three-dimensional images of the surface topography of aluminum nitride films were taken (figure 4).

A 2D topography of the surface of the samples was carried out for areas of size 20, 5, and 2 μm. Resolution 512x512 pixels (figure 5). Three-dimensional topography (AFM image) of the surface of the same regions was obtained on real scales of the Z scale: 20, 5, and 2 μm (figure 6).

To study the surface morphology and compare the roughness values, we measured the topography of the samples using AFM images of the surfaces of aluminum nitride films captured using an NT-MDT Solver Next microscope with an image size of 10x10 μm and a resolution of 512x512 pixels. The samples were flat surfaces. In AFM images, particles of various diameters and small furrows are distinguishable, while furrows have a depth of the entire order of nanometers (figure 7).
Figure 3. 2D – image of the surface of a $2 \times 2 \mu m$ sample obtained at a temperature of 400 $^\circ$C.

Figure 4. 3D – image of the surface of a $2 \times 2 \mu m$ sample obtained at a temperature of 400 $^\circ$C.

Figure 5. Real-time 2D topography (all three x, y, z axes) for sections 20x20 microns, 5x5 microns and 2x2 microns.

Figure 6. AFM images of the sample surface at real scales (all three axes x, y, z) for sections 20x20 $\mu m$, 5x5 $\mu m$ and 2x2 $\mu m$.

Figure 7. AFM – image of the surface of the sample.

To obtain an image of the surface of the object, as well as information on the composition, structure and some other properties of the surface layers, a Hitachi scanning electron microscope was used. Pictures were taken of the morphology of the surfaces of the samples studied on the AFM (figure 8, 9).
Images turned out less contrast. To increase the conductivity and runoff of the charge introduced by the electron beam when shooting, a gold coating was applied to the surface of the samples. The coating was columnar, possibly due to the low wettability of the AlN surface with gold.
Figure 9. SEM images of the surface of sample No. 102.

SEM images of the samples were taken with good detail, which allows you to compare surfaces on SEM and AFM. In the last image, particles are visible that were also observed on the AFM (a three-dimensional model is shown above).

Thanks to the combined use of topography and detection of the oscillation phase, the images contain direct information about the depth of the relief, which is important for the study of roughness. Three roughness values for three measured sections were calculated in the SPM data processing program “Image Analysis 3.2.4 10128” in the Nova-Px 3.2.4 rev 10128 software package (NT-MDT, Russia) and are presented in table 2. Three values were calculated roughness for three measured sites. The values in nanometers are presented for $S_q$ - root mean square surface roughness (volumetric), for $R_a$ - mean surface roughness in profile (line) and for $R_q$ - root mean square roughness in profile (line).

Table 2. Values of surface roughness of the studied samples.

| Sample | $S_q$ | $R_a$ | $R_q$ |
|--------|-------|-------|-------|
| 99     | 11.6  | 8.4   | 10.3  |
|        | 10.2  | 6.3   | 8.0   |
| 102    | 16.1  | 14.8  | 16.7  |
| 103    | 2.6   | 1.7   | 2.2   |
| 104    | 3.2   | 3.6   | 2.8   |
| 116    | 7.7   | 4.8   | 6.0   |
5. Discussion of results
Using the calculated values of Sq - the root mean square roughness along the surface (volume) and Rq - the root mean square roughness along the profile (along the line), the dependence of the roughness of the samples on the substrate temperature was constructed. The dependence is shown in figure 10.

![Figure 10](image)

Figure 10. The dependence of the roughness of the samples on the temperature of the substrate.

Starting from a temperature of 360 °C, the roughness decreases and at substrate temperatures of 380 - 400 °C it becomes stably low for this method of producing films.

6. Summary and conclusion
Using the methods of scanning electron microscopy and atomic force microscopy, the surface morphology of thin films of aluminum nitride obtained by magnetron sputtering has been studied.

For measurements, samples were chosen at different film formation temperatures.

The values of Sq are the mean square roughness along the surface (volumetric), Ra is the average roughness along the profile (along the line) and Rq is the mean square roughness along the profile (along the line) for different regions of the film surfaces.

The influence of the temperature of formation of thin films of aluminum nitride on the surface roughness of thin-film structures was studied. In films grown at substrate temperatures from 300 to 360 °C, surface roughness, both volume and profile, increases from 5 to 20 nm, an island mesostructure forms on the surface of the films, with a further increase in temperature to 380 °C. With the roughness becomes less than 5 nm, there is some leveling of the relief. With a further increase in the substrate temperature, the roughness remains stably low. This can be explained by the fact that at a given temperature the surface migration of the deposited atoms is easier and the surface becomes more perfect.

References
[1] Odintzov V, Suschentzov N, Kudryavtzev T et al. 1996 (Yoschkar-Ola) pp 49-52
[2] Noge S, Ueno H, Hohkawa K, Yoshikawa S 1995 IEEE Ultrasonic Symposium pp 379-382
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