Influence of magnetron sputtering modes of aluminum and aluminum nitride films on their surface, structure and composition

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Abstract. To ensure the optimal combination of the properties of the thin-film layers of piezoelectric structures and achieve the required characteristics of resonators and devices for selecting and generating of the signals based on them, the influence of technological modes of aluminum nitride films formation on the surface morphology, structure and elemental composition of films used in the construction of microelectronic bulk acoustic waves (BAW) resonator with Bragg reflector, the optimal modes are determined that satisfy the requirements for film layers for a piezoelectric transducer and Bragg reflector.

Keywords - magnetron sputtering, scanning electron microscopy, film surface morphology, elemental composition.

1. Introduction
In microelectronics, aluminum nitride is widely used in resonators, sensors, filters, and other devices utilizing surface acoustic waves due to the high speed of sound propagation, temperature stability, and high electromechanical coupling coefficient, as well as due to the unique combination of its following physical characteristics: high thermal conductivity, rather large band gap, high resistance to thermal shocks and stability when heated in inert media, low temperature coefficient of linear expansion, high values of relative permittivity and resistivity [1-3].

Further development of microelectronic devices and devices based on aluminum nitride films is associated with the use of methods and technologies for their formation that meet the requirements of widespread industrial use: simple technical implementation, reproducibility of parameters, the possibility of transferring the production technology to other installations. The study of technological processes that make it possible to obtain materials with desired properties is one of the main directions of such development. In turn, to solve this problem, it is necessary to control and manage the properties of the resulting films, to find their relationship with technological modes and deposition processes [4].

In connection with the above, the study of the formation processes of thin AlN films, determination of the most optimal modes of their formation, is of scientific and practical interest.

2. Formulation of the problem
A noticeable change in the technology of production of layered structures based on AlN and an increase in the efficiency of technological processes will become possible with the successful solution of a
The main problem that was solved in the present work was in performing the qualitative analysis of the surfaces of aluminum and aluminum nitride films, determining their elemental composition, finding the relationship between the properties of the films and the processes and technological parameters of magnetron deposition, and determining the modes of their formation.

3. Experimental technique

The formation of aluminum nitride films was carried out by DC magnetron sputtering in a nitrogen and argon atmosphere on a STEMS116-01 multicomponent coating installation. Aluminum films were deposited on polished sitall substrates while aluminum nitride films were deposited on aluminum substrates.

Table 1 shows the modes of formation of the investigated AlN films. Film 1.1. is an aluminum nitride film deposited on an aluminum substrate without preliminary etching; film 1.2. is a film of aluminum nitride obtained under the same technological conditions as the previous one, but the aluminum substrate was etched for 20 minutes before sputtering.

Table 2 shows the technological parameters of the formation of aluminum films.

The measurements of the films were carried out at the Analytical and Technological Research Center "High Technologies and Nanostructured Materials" (ATRC) of the NSU and the Institute of Semiconductor Physics SB RAS. Images of the film surfaces, as well as information on the composition and other properties of the films were analyzed by scanning electron microscopy (SEM) on a Hitachi SU8280 microscope at an accelerating electron beam voltage of 5 kV with a high lateral resolution.

4. Experimental results and discussion

SEM images were taken of the surface morphology of the samples (Figure 1-3).
In the SEM images presented, one can distinguish features of the relief with a scale of fewer than ten nanometers in the plane. The surface is highly developed where many protruding objects are broken-rounded in shape. Objects on the surface visually have a granular flower-like shape with a diameter about 400 nm and consist of ~10 petals ~200 nm long and ~80 nm thick. On the surface, there are “caps” of supposed vertical crystallites extended from the substrate.

As can be seen from Figure 2, the surface morphology of the films was not affected by the annealing of the substrate surface prior to the deposition of an aluminum nitride film on it.

After taking pictures of the surface morphology by the method of X-ray spectral microanalysis (SEM EDX) the elemental analysis of the surface of the samples was performed. Elemental mapping ("chemical mapping") of the films was carried out, which showed that aluminum was fairly uniformly distributed over the surface in all films, and aluminum was not concurrent with other elements in the surface composition. In addition to aluminum and nitrogen expected in the composition of the films, a significant amount of oxygen and carbon was observed. The most intense carbon signal was observed on upwardly protruding surface irregularities. Oxygen was found mainly by the edges of the surface irregularities. Moreover, the oxygen was distributed more evenly than carbon, due to the significant amount of surface irregularities.
The elemental composition of the films (in weight and atomic%) is given in Table 3.

Table 3. Elemental composition of films registered by SEM EDX.

| Sample No. |  | Al |  | N |  | C |  | O |  | Cu |
|------------|---|-----|---|---|---|---|---|---|---|---|
|            |  | Weight % | Quantity (atomic (molar)) % | Weight % | Quantity (atomic (molar)) % | Weight % | Quantity (atomic (molar)) % | Weight % | Quantity (atomic (molar)) % | Weight % | Quantity (atomic (molar)) % |
| 1.1        |  | 46.66 | 53.47 | 12.77 | 28.19 | 2.81 | 7.23 | 5.19 | 10.03 | 2.21 | 1.07 |
| 1.2        |  | 42.74 | 52.7 | 12.05 | 28.61 | 2.66 | 7.36 | 4.94 | 10.28 | 2.01 | 1.05 |
| 2          |  | 46.41 | 54.07 | 12.9 | 28.95 | 2.74 | 7.18 | 4.29 | 8.44 | 2.73 | 1.35 |
| 3.9        |  | 45.37 | 54.37 | 11.25 | 25.98 | 2.88 | 7.76 | 5.32 | 10.75 | 2.23 | 1.14 |
| 4          |  | 46.61 | 56.19 | 13.4 | 31.12 | 2.84 | 7.69 | 1.96 | 3.98 | 2.01 | 1.03 |
| 5          |  | 46.0 | 54.89 | 13.99 | 32.16 | 3.12 | 8.35 | 1.49 | 3.01 | 3.08 | 1.56 |
| 1- Al      |  | 74.95 | 83.52 | 0.12 | 0.25 | 3.15 | 7.89 | 3.55 | 6.68 | 2.72 | 1.28 |
| 2- Al      |  | 60.95 | 80.75 | 0.41 | 1.04 | 3.31 | 9.86 | 3.32 | 7.41 | 1.54 | 0.87 |
| 3- Al      |  | 53.8 | 67.09 | 1.75 | 4.2 | 2.77 | 7.75 | 8.98 | 18.89 | 3.91 | 2.07 |

In aluminum films, the fraction of atomic Al in the composition appeared to be lower than expected 100%, but about 83.5% and reduced aluminum content correlated with the higher substrate temperature. In aluminum nitride films grown, the fraction of aluminum atoms was ~54%, while the nitrogen atomic content was ~26%. Remarkably, the observed stoichiometry of Al and N does not correspond to the composition of AlN. Oxygen content in AlN films was ~10% and ~7% in Al films, where for a film sputtered at a substrate temperature of 2000°C its amount appeared almost 2.5 times higher than in films sputtered on substrates at a lower temperature. Oxygen was found at the edges of the irregularities. Moreover, it was distributed more evenly than carbon, due to large amount of surface these irregularities.

The presence of oxygen can be explained by the acidification of aluminum, which turned out to be more intense for irregularities on the surface. The protruding parts have a developed surface and therefore greater surface energy, which facilitates the chemical oxidation reaction in a gaseous environment of a strong oxidant, i.e. oxygen. The aluminum surface could likely oxidize after opening the chamber. Oxidation of aluminum is saturated in the first portions of seconds and is almost independent from the exposure time, therefore, the detection of native oxygen was anticipated. Carbon content was ~8% in both AlN and Al films. Carbon was also distributed quite evenly, but the areas with observable carbon spots on the film surface were found. Most of the carbon atoms were on the surface irregularities. This can be explained by the fact that some of the carbon could have settled on the surface from the growth chamber, and some was added during the elemental mapping technique as a methodological artifact. Copper content was ~1%, while most likely it has deposited on the surface of the substrate holder material during the film growth. It should be noted that the possible reasons for the presence of the aforementioned chemical elements may be associated not only with the formation of films but also partly with the measurement process. Indeed, the sample in the SEM chamber was fixed on a copper substrate, covered with a piece of adhesive copper foil from the side. Carbon contamination may have occurred while scanning the sample. Carbon could also be present in trace amounts on the walls of the SEM chamber.

5. Conclusions
The surface morphology and elemental composition of thin films of aluminum and aluminum nitride obtained by magnetron sputtering have been investigated by scanning electron microscopy.
It has been determined that aluminum films are obtained of higher quality at a substrate temperature below 2000 °C.

It was shown that the stoichiometry of Al was excessively exceeded in aluminum nitride films, which indicates that it is necessary to refine the growth technology in the magnetron chamber, including changing the technological growth parameters, and it is also necessary to eliminate all possible causes of film contamination arising in the process of film growth, possible gas leaks, including atmospheric air, vacuum level, and purity of precursors.

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