Reactive DC sputter deposition and charactersation of AlN thin films

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Abstract. Thin films of Aluminium Nitride (AlN) have been deposited on Si wafers using RF reactive sputter deposition in a nitrogen ambient. AlN is a wide bandgap semiconductor suitable for deep ultraviolet optoelectronics. The films have been characterized using Atomic Force Microscopy (AFM), Rutherford Backscattering Spectrometry (RBS), Scanning Electron Microscopy (SEM) and the oxygen content has been profiled using resonant RBS. The films were found to be smooth and uniform and adhere well to the Si substrate.

1. **Introduction**

Aluminium nitride is a semiconducting material with a bandgap of 6.2 eV making it suitable for deep ultraviolet optoelectronics. It has a high thermal conductivity which ranges from 70 to 285 $W.m^{-1}.K^{-1}$ depending on whether it is polycrystalline or a single crystal [1]. Many techniques have been used to deposit AlN on various substrates. The most popular method of depositing AlN films is by reactive RF sputtering in a nitrogen atmosphere [3,4,5,6]. Other techniques that have been used to deposit AlN are chemical vapour deposition [7], laser chemical vapour deposition [8], molecular beam epitaxy and pulsed laser ablation [3].

In this work we wanted to deposit AlN using reactive sputtering, study its surface using AFM and SEM and also use both RBS and resonant RBS to study both composition and possible incorporation of oxygen in these crystals as we could not find any reference to the use resonant RBS to study oxidation of AlN.

2. Experimental procedure

Silicon wafers were cut into squares of edge 12 mm, chemically cleaned in an ultrasonic bath and then rinsed in de-ionized water. The chemicals used to clean the substrate wafers were methanol, acetone, trichloroethylene, acetone and finally methanol, before using distilled water. The wafers were then dried and loaded into a sputtering system. The sputtering machine used is an AJA Orion 5 Sputtering System.

Once good vacuum was achieved the chamber of the sputtering system was pre-heated at 600 ^{o}C for one hour to drive out any moisture and also to degas (remove oxygen from its walls). At the start of the experiment, before introducing any gas, the chamber had a base pressure of $2x10^{-7}$ Torr. The process gas used to sputter the aluminium was argon introduced at a flow rate of 6 sccm. The reactive gas used to obtain AlN was nitrogen introduced at a flow rate of 6 sccm. Both gases were high purity gases from AFROX (~99.98% pure). The aluminium target used was a disc of 2 inch diameter, 0.250 inch in thickness and had a purity of 99.999%. The target to substrate distance was 34 mm. The substrate was rotated in order to obtain a uniform film. The deposition was done for a period of two hours while maintaining a pressure of $3x10^{-3}$ Torr inside the chamber. After deposition the samples were left in the chamber for one hour to cool. Dry nitrogen was used to break vacuum, thus eliminating condensation inside the chamber as well as oxygen contamination.

3. Results and discussion

3.1. AFM results

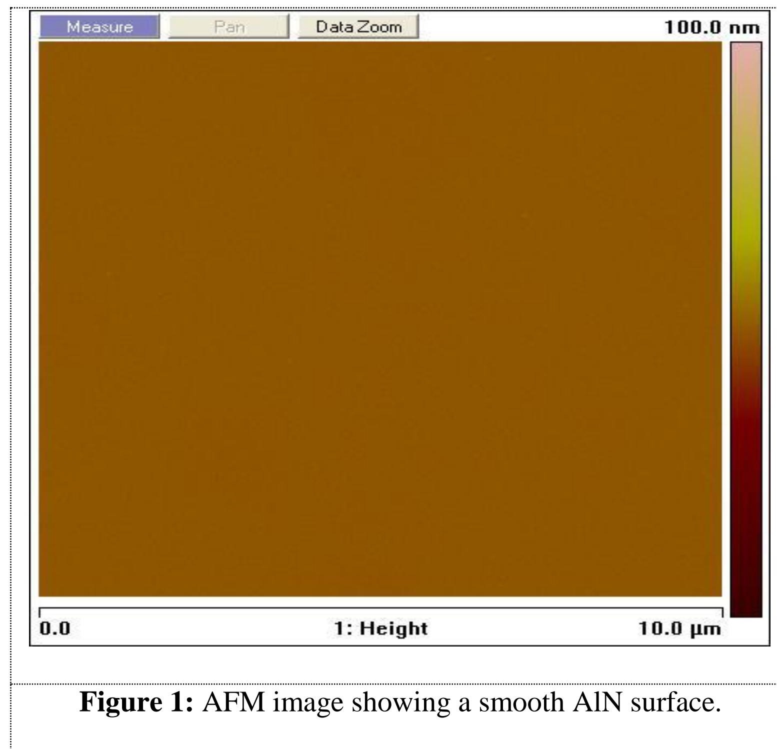

Figure 1: AFM image showing a smooth AlN surface.

3.2. Normal RBS

The samples obtained had a uniform colour, looked smooth and showed no signs of peeling (see figure 1). Rutherford Backscattering Spectrometry (RBS) was done using charged helium atoms accelerated to an energy of 2 MeV. The RUMP software code was used to determine the Al:N stoichiometric ratio of deposited layers and to find layer thicknesses of the film.

Figure 2 shows RBS spectra of AlN on silicon. Normally it is not easy to analyse a sample using RBS if the mass of the two elements in the sample are close to each other. In this case Al is close to Si of the substrate. However Al (an element slightly lighter than Si) has been deposited on top of Si. The overlap between the spectra of the two creates an observed peak seen at the higher energy end of the spectrum (see

figure 2). It is this bump which helps us to do the analysis. The surface positions of the elements Si and Al are marked by means of arrows on the graph. These are positions at which alpha particles scattered on the surface will be recorded. Surface positions of both N and O have been shown also. RBS results show that the thickness of the AlN film deposited using RF magnetron sputtering at an energy of 60 W for two hours is about 1 400 Å. It seen from the other spectrum that the thickness of the sample deposited at RF magnetron power of 130 W is very close to that of the other sample. RF sputter deposition is less efficient when compared to DC magnetron deposition, in general. The gas flow rates of nitrogen and argon during the deposition of both samples were equal at 6 sccm.

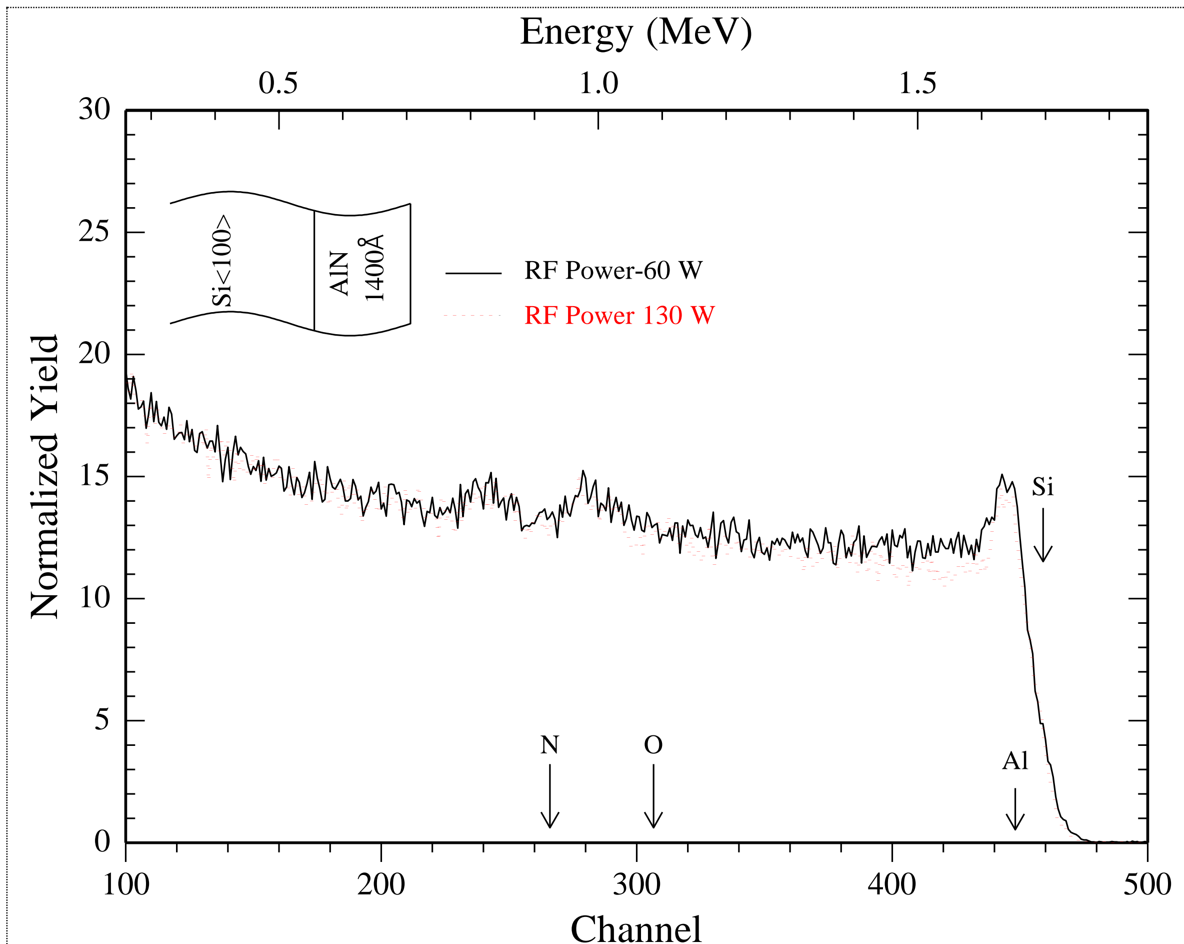

Figure 2: RBS spectra obtained from samples deposited at an RF magnetron power of 60 W and 130 W respectively for a period of 2 hrs. The argon and nitrogen flow rates were 4 sccm and 10 sccm respectively for both samples. The film thickness was found to be 1 400 Å. It is also obvious from both spectra that the samples have oxygen. The chamber pressure was maintained at $3x10^{-3}$ Torr.

Both depositions were done at room temperature; figure 2 shows the presence of oxygen on both samples. It shows that the amount of oxygen is almost the same in both samples.

We found using RUMP simulation that the ratio of Al:N is close to unity (i.e. we do have AlN, possibly oxidized).

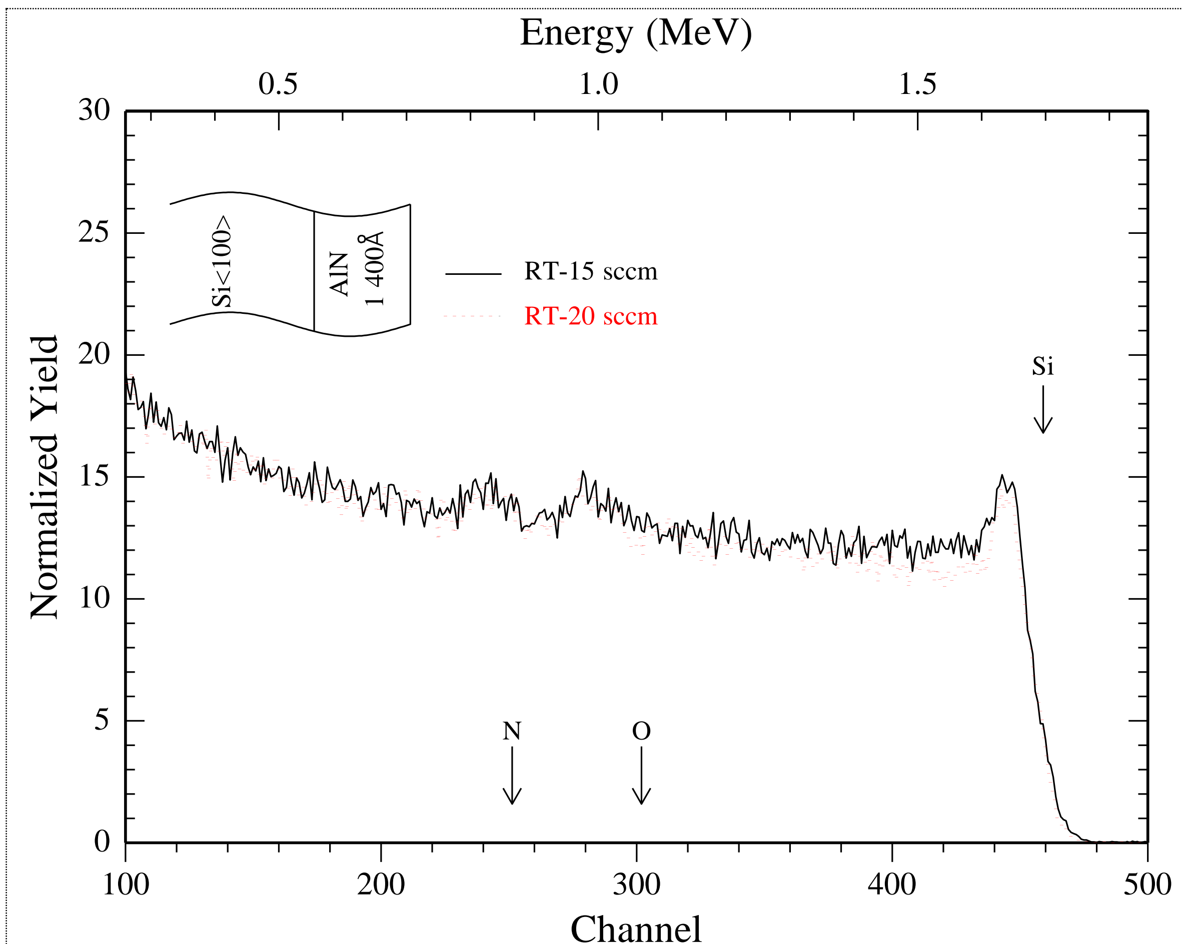

Figure 3: RBS spectra of samples deposited using RF magnetron power of 130 W for a period of 2 hrs. In an attempt to reduce oxygen contamination the flow rate of nitrogen was raised to 15 and 20 sccm for the two samples. It is difficult to tell whether the amount of oxygen has been reduced from these results. Sample thicknesses were found to be the same as before.

In an attempt to reduce oxygen contamination or its inclusion within the film, we increased the nitrogen flow rates to 15 sccm and 20 sccm for the two samples spectra shown in figure 3. The flow rate of argon was kept at 6 sccm and the chamber pressure was maintained at $3x10^{-3}$ Torr. The deposition was done for a period of two hours. It is noticed from figure 3 that the thickness of both samples remain at 1 400 Å. This is to be expected since sputtering results are fairly reproducible. The same amount of material for a given element will be deposited if the same power and time duration is used. On analyzing the data using RUMP, we could not find any difference in oxygen amounts for the two samples deposited using different nitrogen flow rates.

We decided to use resonant oxygen scattering to enhance the oxygen peak.

3.3 Resonant RBS

If the energy of the oncoming alpha particles exceeds the Coulomb barrier the alphas begin to interact with the nucleus of the target atom resulting in non-Rutherford backscattering. In this case the scattering cross section may increase resulting in an enhanced scattering yield. We used the process $^{16}O(^{4}He;\,^{4}He)^{16}O$ inside a van De Graaf accelerator with He^{2+} ions of kinetic energy 3.05 MeV at a scattering angle of 165° and a sample tilt of 10° (to avoid channeling effects).

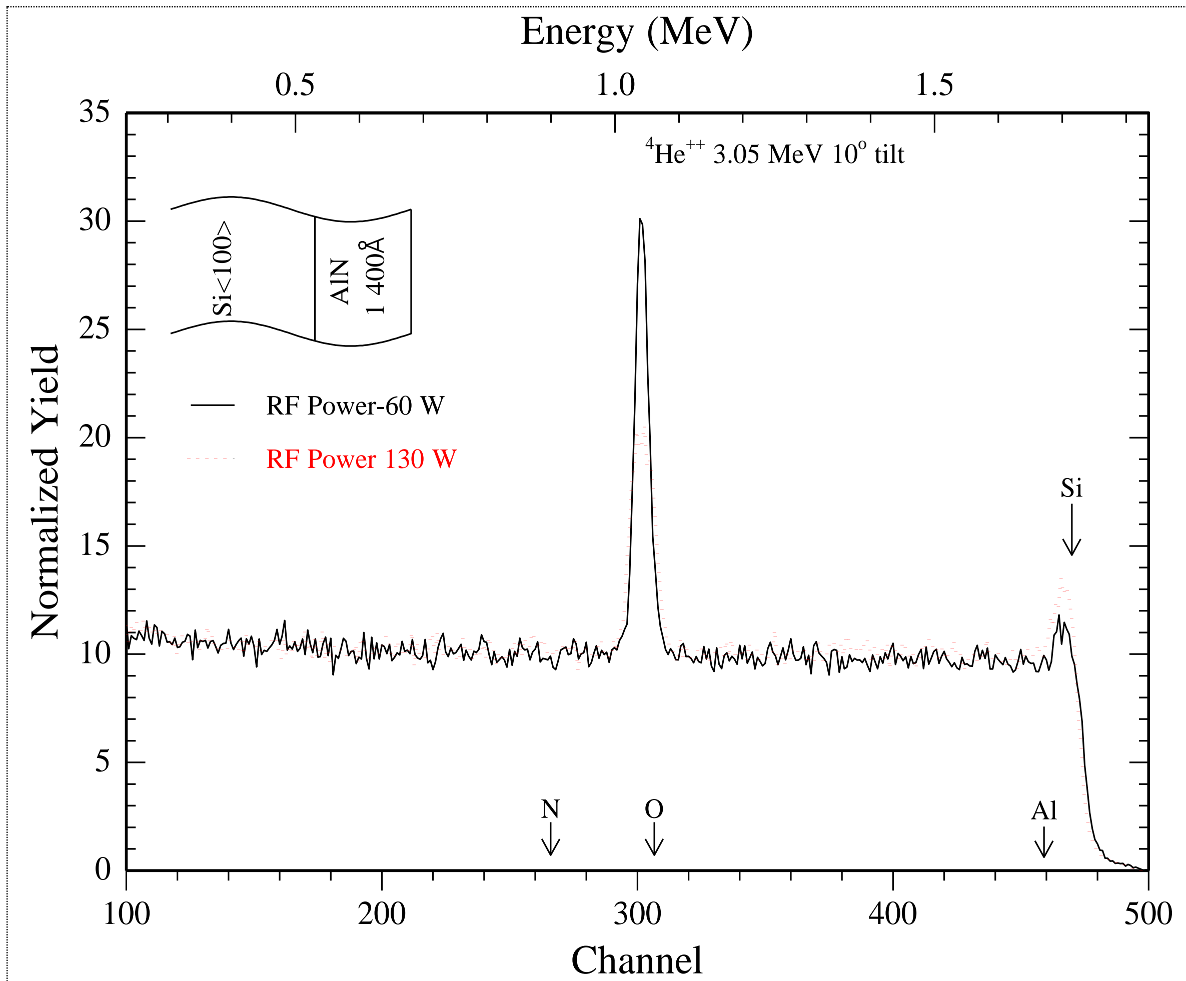

Figure 4: Backscattering with 3.05 MeV alphas. The spectra is that of samples used to obtain figure 3 (at 2 MeV). We note now the enhanced oxygen peak. Notice that there is more oxygen in the sample deposited using magnetron power of 60 W compared to that of 130 W.

The spectra shown in figure 4 was obtained by backscattering 3.05 MeV alpha particles on two samples, one obtained at magnetron power of 60 W, while the other at 130 W (they are the same samples investigated at 2 MeV in figure 3). Since the alphas scatter at resonance on oxygen at an energy of 3.05 MeV, the oxygen peak in the spectra is enhanced. We therefore notice that there is much more oxygen in the sample obtained using 60 W when compared to that in which 130 W was used. The peaks belonging to N are however even lower (compare these in both figure 3 and figure 4). It does not mean there is less nitrogen but simply because the surface barrier detectors used are less efficient at higher energies. Note also that nitrogen is not at resonance at this energy (3.05 MeV). Comparing the area under the RBS curves for the oxygen peak we find that there is 23.6% more oxygen in the sample deposited at 60 W when compared to that deposited at 130 W.

4. Summary and conclusion.

We have sputter deposited films of AlN using RF sputtering where the reactive gas used is nitrogen. We have found that RF sputtering is a suitable method to make films of AlN. The sputter rates are however low and thicker films can only be made over longer time periods. AFM analysis has shown that the resulting films are smooth, show no signs of peeling and are adherent on the Si substrate. We have used RBS to determine sample thicknesses. It also allowed us to find out the ratio of Al:N, which was found to be close to unity (meaning AlN films deposition achieved). We however serendipitously found signs of oxidation. We found resonant RBS to be a suitable tool to analyse samples that had oxidized. In trying to find the source of oxidation, it was discovered that higher sputter rates (higher RF power) lead to lower oxygen contamination. We also found that higher flow rates are better and lead to lower oxidation. The source of the oxidation is possibly the chamber itself.

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