High Quality Oxide Films Deposited at Room Temperature by Ion Beam Sputtering

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Abstract

We have deposited dense and pinhole-free thin films of SiO_2, Al_2O_3 and ITO at room temperature via ion beam sputtering. The SiO_2 films were found to be of similar quality as thermal oxide with a resistivity greater than 10^{13} Ω·m and breakdown field in excess of 700 MV/m. The Al_2O_3 films were part of a Pt- Al_2O_3-Pt vertical tunnel junction and were kept extremely thin, from 2 nm to 4 nm. The current-voltage characteristics of these junctions indicated a breakdown field in excess of 2000 MV/m, roughly twice that achieved by ALD films. This breakdown voltage was found to be independent of junction area, strongly suggesting the absence of pinholes in the film. The ITO films were 50 nm to 100 nm thick. As deposited, they are fully transparent with an electrical resistivity of $5x10^{-6}$ Ω·m.

Introduction

The highest quality oxides such as SiO_2, Al_2O_3 and Indium Tin Oxide (ITO) require high temperature processing either during the growth of the film or annealing post-growth. Thermal SiO_2 is grown at ≈1000 °C [(1)], ALD Al_2O_3 is deposited at ≈300 °C [(2)], and magnetron-sputtered ITO must be annealed above 350 °C in order to turn the film conductive [(3)]. These elevated temperature requirements are not compatible with polymer substrates used for flexible electronics. Our objective was to explore ion beam deposition as an alternative room temperature technique and characterize the film properties.

Film deposition technique

The films of SiO_2, Al_2O_3 and ITO were deposited on silicon wafers at room temperature via ion beam sputtering as depicted in figure 1. The deposition system consists of a 3-grid 14 cm RF ion gun directed at 200 mm targets of SiO_2, Al and ITO. All three processes require a small flow of O_2 to achieve stoichiometry. Typical conditions were: argon flow rate 3.3×10^{-7} m^3/s (20 sccm), beam voltage 600 V, beam current 220 mA and acceleration voltage 150 V. The substrate wafers rotated at 10 rpm and were kept at 20 °C. The base vacuum prior to deposition was 2.6×10^{-6} Pa (1.5×10^{-8} Torr).

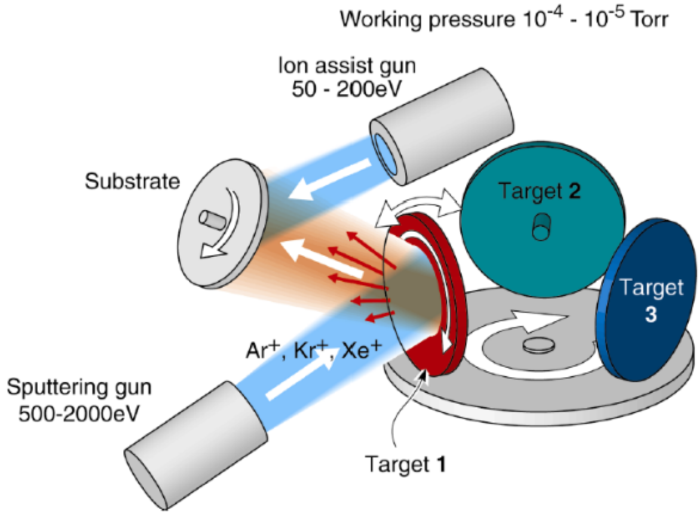

Figure 1. Schematics of the ion beam deposition system

Measurement techniques

The measurements techniques consisted of current-voltage, ellipsometry, chemical etching and X-ray diffraction (XRD). We used a mercury probe directly on the oxide wafers, and a standard probe station on the devices, to measure resistivity, breakdown voltage and detect pinholes. Ellipsometry yielded the index of refraction and extinction coefficient. Chemical etching revealed general oxide quality and checked for the presence of pinholes. XRD was used to assess the degree of crystallinity of the deposited films.

Results

SiO_2 – 100 nm films

Figure 2 shows the index of refraction and extinction coefficients. The index is somewhat higher (by ~0.04) than that of thermal oxide and the extinction coefficient (absorption) nearly zero.

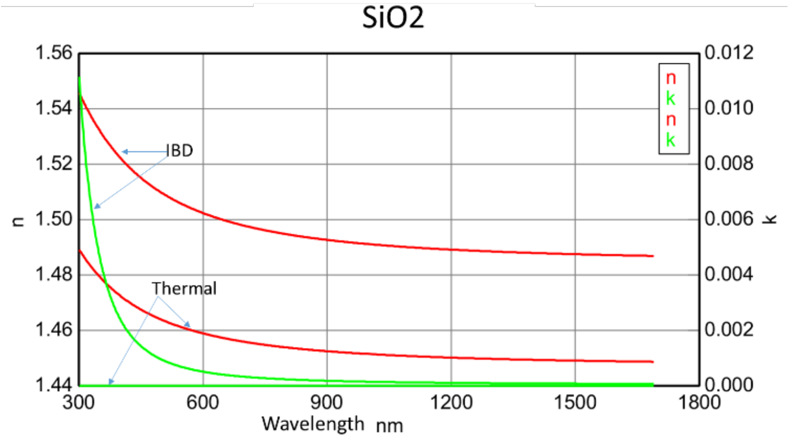

Figure 2. Index of refraction and extinction coefficient of IBD oxide and thermal oxide

We measured the density of IBD oxide: 2,410 kg/m^3, which lies between that of thermal oxide (2,220 kg/m^3) and that of quartz (2,650 kg/m^3). The etch rate in buffered oxide etch (6:1 BOE) was 1.6 nm/sec, identical to that of thermal oxide, and therefore strongly suggesting the absence of pinholes in the film. XRD revealed that the films were totally amorphous.

The resistivity versus electric field was measured via mercury probe between zero and 400 MV/m. The IBD oxide (10^{13}-10^{14} ohm.m) proved somewhat inferior to thermal oxide (10^{14}-10^{15} ohm.m).

Figure 3 shows the breakdown field distribution of IBD oxide and thermal oxide, as measured at 65 points on each wafer via mercury probe. The IBD oxide exhibits a very high breakdown field, from 700 to 1,400 MV/m, higher than thermal oxide.

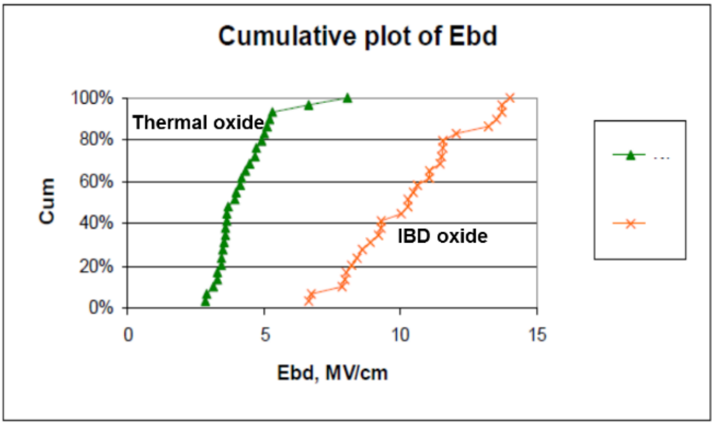

Figure 3. Breakdown field distribution of IBD and thermal oxides

Al_2O_3 – 50 nm films

We measured the index and extinction coefficient in the wavelength range of 280 nm to 1650 nm, and found them identical to those of ALD (Atomic Layer Deposition) oxide. The index is 1.67 at 600 nm while the extinction coefficient is less than 0.01.

The electrical resistivity was measured by a mercury probe, and is in excess of $3 x 10^{12}$ ohm.m up to fields of 150 MV/m. This value is superior to that of thermal oxide (obtained by oxidation of AlN, ~10^{10} ohmcm). The breakdown voltage was also measured via a mercury probe and found to yield a breakdown field in the range 200-370 MV/m, slightly lower than that of thermal oxide (400-500 MV/m.)

XRD revealed that the films were totally amorphous.

ITO – 100 nm films

The index and extinction coefficient were found nearly similar to those of standard magnetron-sputtered ITO. The electrical resistivity, *as deposited*, was $5 x 10^{-6}$ ohm.m, similar to that of magnetron-sputtered ITO but *obtained after annealing at 350 °C.* The films were transparent.

In contrast to all other standard deposition techniques, the as-deposited films were crystalline, as shown in figure 4.

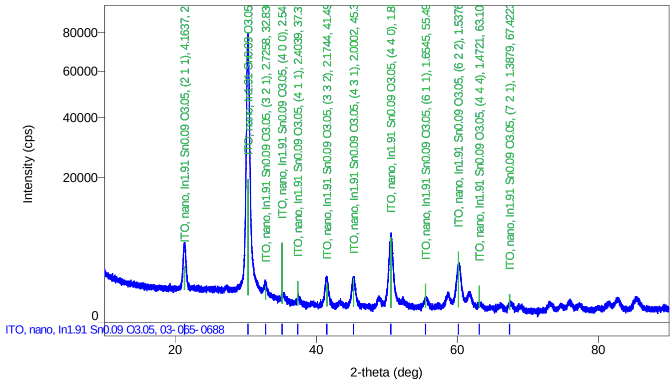

Figure 4. Grazing incidence XRD of ITO film (deposited on glass)

Al_2O_3 – 2-4 nm films

These oxide films were part of a tunnel junction Pt/Al_2O_3/Pt used in a DNA sequencing device. A TEM cross-section of the tunnel junction is shown in figure 5. Of great interest is the sharpness of the interfaces between layers, where interdiffusion can be minimized: first, by using a slow deposition rate for the oxide, and second, by using the Biased Target Deposition technique (BTD) for the titanium and platinum. In BTD, the bias on the metal target can be adjusted to yield a very low adatom energy for the first few monolayers (200-300 eV), thereby preventing interdiffusion, and increasing the bias (to ~800 eV) for the rest of the layer to achieve uniform coverage.

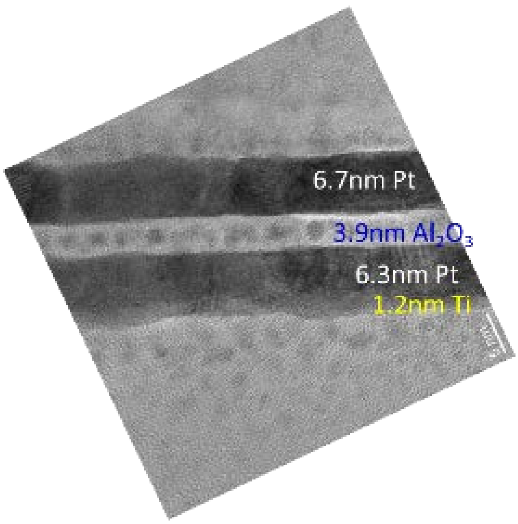

Figure 5. Transmission electron micrograph of tunnel junction cross-section

The oxide thickness was varied from 1 to 8 nm while the junction width was varied from 0.5 to 10 µm. We fabricated junctions with ALD and IBD oxides. We found that the IBD oxide has a superior breakdown field (2,000-3,000 MV/m) to that of ALD oxide (1,300 MV/m). Furthermore, the breakdown field was found to be independent of the junction area, strongly suggesting the absence of pinholes. The data comparing ALD and IBD oxides is shown in figure 6. It is to be noted that, because of the extreme thinness of the junctions, such high breakdown fields arise from quantum tunnelling, and not from material degradation due to defects.

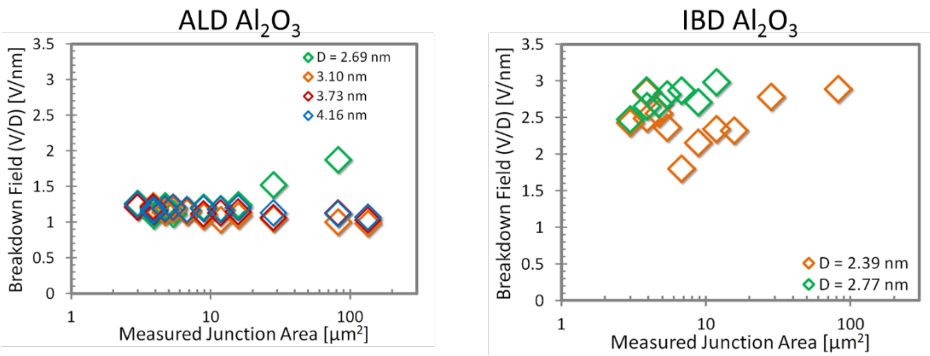

Figure 6. Comparison of breakdown fields for ALD and IBD Al_2O_3

Conclusions

Ion beam deposition is capable of depositing very high-quality oxides at room temperature. The technique allows sub-nanometer control over the film thickness. The biased target version of IBD is able to produce sharp interfaces with minimal interdiffusion.

References

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