

GROWTH OF ANODIC Al_2O_3 Films

by

Bruce Parmelee

5th Year Microelectronics Student
Rochester Institute of Technology

ABSTRACT

The anodic oxidation of Aluminum (Al), which is a low temperature process, was used to form an aluminum oxide (Al_2O_3) film for use as an insulating layer. The anodization was done in a diluted, approximately 10% by volume, sulfuric acid solution (H_2SO_4). Constant anodization voltages of 10, 15, and 20 volts, were maintained to obtain layers of Al_2O_3 ranging in thickness and refractive index from 3373-4077 Å and 1.46-1.95 respectively.

INTRODUCTION

Several methods for creating an Al_2O_3 films are plasma-oxidized Al_2O_3 , CVD- Al_2O_3 , and anodic Al_2O_3 .¹ The anodic oxidation of aluminum is easily accomplished by the use of an electrolytic cell. The electrolytic cell used contained a aluminum anode and lead cathode. For this process the cell was filled with an electrolytic solution of H_2SO_4 and a constant voltage maintained across the electrodes. Hydrogen is liberated at the cathode and the oxygen combines with Al at the anode to form a Al_2O_3 film.

Many parameters effect the growth and uniformity of the Al_2O_3 film. Control of the anodization process should aim at constant voltage.² The formation of a barrier layer will increase the series resistance of the cell and cause a decrease in the current density for a constant voltage.

Close temperature control is important since the current density and the rate at which the film is dissolved by the electrolyte alter markedly with temperature. As the temperature of the electrolyte rises its viscosity and resistance decrease leading to an increase in current density. This will alter the uniformity and growth rate of the film. Control to within plus or minus one degree celsius is imperative for consistent results.² To disperse the heat generated during the anodization process efficient agitation is needed.

The concentration of the acid in the electrolyte effects the dissolution of the aluminum anode as well as the porosity of the resulting Al_2O_3 film. The dissolution of aluminum increases with increasing acid strength. The films produced in a strong electrolyte tend to be more

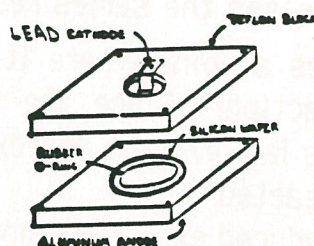
porous, softer, and more flexible than those produced in weaker electrolytes.

The time of the anodization process will effect the film thickness and content. Longer anodization times will produce thicker films for a given thickness of aluminum. If the reaction is allowed to go to completion substrate material will be incorporated into the film altering its properties.

EXPERIMENTAL

The anodization cell used consisted of an aluminum bottom plate and a teflon top plate between which the wafers could be placed as shown in Figure 1.³ A rubber O-ring was used between the wafer and the teflon top plate to contain the electrolyte.

Figure 1.) Anodization cell



The aluminum anode had an area of 5.5 cm^2 exposed to the electrolyte. The cathode, which was mounted to the teflon top plate, consisted of a 5.0 cm^2 piece of lead foil.

N-type $\langle 111 \rangle$ wafers, 3.5-6.6 ohms per cm, were coated with 2500\AA of Al using an evaporator. Then the aluminum was anodized in a 10%, by volume, sulfuric acid solution. Voltages of 10, 15, and 20 volts, were maintained across the electrodes. Current densities ranging from 36.8 to .3 mA/cm^2 were recorded. Agitation was supplied by a pipet in order to reduce any local heating effects. The temperature of the electrolyte was 23 degrees celsius and no change was observed during anodization. The electrode spacing was kept at 1 centimeter throughout the experiment.

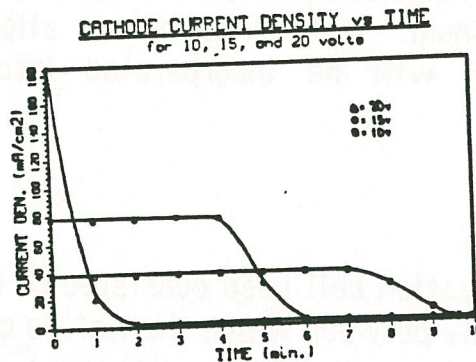
The thickness of the Al_2O_3 films were determined using a Gaertner L117 ellipsometer of wavelength 6328\AA . The C-V characteristics of the film were plotted after evaporating metal on top of the Al_2O_3 film to form a capacitor.

RESULTS/DISCUSSION

For each of the anodizing voltages the current density decreased as the anodization proceeded. The current density for each anodizing

voltage plotted as a function of anodization time can be seen in Figure 2.

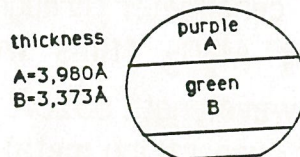
Figure 2.) Cathode current density as a function of time



The decrease in current density is due to the formation of the Al_2O_3 film which increases the series resistance of the electrolyte. The current density reaches a point where it remains constant signaling the completion of the reaction. Since the final resistance values for the solution, given by ohms law V/I , are approximately equal it appears that all of the aluminum was reacted.

The films produced exhibited nonuniform growth across the wafer represented by the formation of a rainbow of colors as shown in Figure 3. The film formation began at the point closest to the supply voltage and proceeded to traverse across the wafer. Since the measured thicknesses are seen to be approximately equal it appears that all of the aluminum was converted to an oxide. The largest variation in thickness from wafer to wafer was 1000\AA . The refractive index was also found to vary wafer to wafer and across the wafer. The variation in color across the wafer could be the result of varying refractive indices due to nonuniform growth and film content.

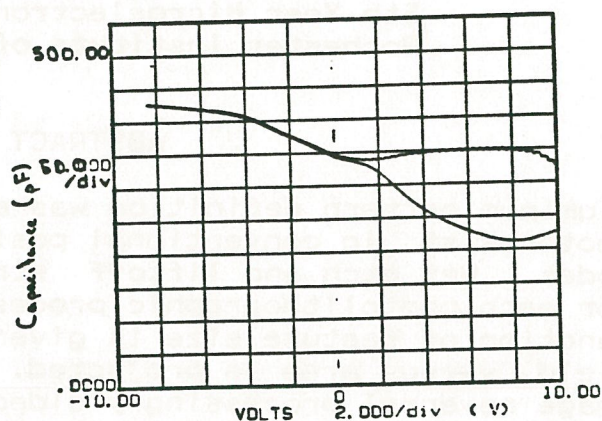
Figure 3.) Color variation across the wafer



The deposition and sintering of backside aluminum on the n-type wafers will form a p-n diode. Therefore the C-V plots will show nonidealities. A C-V plot is shown in Figure 4. The plot shows the generation of minority carriers due to room light on the uppermost curve. The depletion of the surface is depicted, in the lower curve, by the decrease

in capacitance. To improve the results of the C-V tests p-type wafers should be used to form a ohmic back contact. By producing several capacitors of different areas it would be possible to determine the thickness of the grown Al_2O_3 film.

Figure 4.) C-V plot



SUMMARY

The anodic oxidation of aluminum in a H_2SO_4 solution produced Al_2O_3 films of varying thickness. The growth rate was found to be higher for higher electrode potentials corresponding to a faster decrease in cathode current density as shown in Figure 2. The films produced exhibited nonuniform growth across the wafer which is not understood at this time. The C-V plot of figure 4 shows the depletion of the silicon surface is possible, and that Al_2O_3 could be used as an insulator.

REFERENCES

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- 3.) Grimsley, Thomas., RIT Senior Research Project, 1986