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THICKNESS OF LARGE AREA CERAMIC FILMS FORMED BY AEROSOL DEPOSITION

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Large area ceramic films are needed in many industries like electronics and energies. For example, large area insulating films are needed in big displays. However large area films of quality are difficult to make. Aerosol deposition, based on room temperature impact consolidation (RTIC), is a very promising technology to form quality ceramic films directly onto substrates at room temperature. Therefore large area lead zirconate titanate (PZT) films were made on 4 inch silicon wafers by scanning nozzles in two directions. When the films become large in area the evenness of the thickness tends to decrease. So the evenness of the film thickness was evaluated through the measurements of film thickness with step height of masked edges and with ellipsometry. The deviation of the thickness was smaller in the direction of nozzle movement than in the direction perpendicular to it. The reason is that the density and/or amount of aerosol blown out of the nozzle fluctuate less in the short time range, but fluctuate more in the long time range. Therefore uniform aerosol generation is important to form films that have even thicknesses. The nozzle that blows off a broadening beam was good for making large area films. Key words: Aerosol deposition, Ceramic films, Thickness, Large area films

1. INTRODUCTION

Large area ceramic films are needed in many industries like electronics and energies. For instance, anti-corrosion films are needed for semi-conductor manufacturing systems, and functional ceramic films are needed in big flat displays. Also in semiconductor manufacturing systems the wafer sizes are becoming bigger, which of course requires larger area films to be fabricated on the wafers.

Technologies based on room temperature impact consolidation (RTIC) are very promising for ceramic film forming. In RTIC ceramic particles are collided and form films that are mostly equal in quality to conventionally sintered ceramics on the substrates.

When gas pressure is used to drive the ceramic particles, the technology is called aerosol deposition. It is good at forming films of several μ m thick. This thickness is too thick for the technologies that build up atoms or molecules one by one like CVD. The thickness is too thin for conventional sintering of thin plates. Namely aerosol deposition can cover the gap in between.

However researches on aerosol deposition have been mainly directed on small area films.[1] Therefore aerosol deposition of large area ceramic films was tried.

When the films become larger in area the evenness of the thickness and other material characteristics tend to be worse. So the thickness distributions of aerosol deposited films were experimentally studied. The material of the films was lead zirconate titanate (PZT). It was deposited on 4 inch silicon wafers. An important factor that determines the thickness distribution was the density and/or amount of aerosol blown out of the nozzle. Aerosol deposition experiment was carried out with the big machine developed by us and shown in Fig. 1. It can move nozzles or samples at a distance of 500 mm to X direction and 400mm to Y direction.

The substrates were a 4 inch diameter silicon wafers with thermally oxidized surfaces. The powder used was PZT-LQ made by Sakai Chemical Co. The carrier gas was helium. The flow rate of helium was ranged from 3 to 15 l/min. Two types of nozzle were used. One is a nozzle that makes an aerosol beam broaden toward the end and another is a nozzle that ejects a straight beam of aerosol. The aerosol was blown down from the moving nozzles and the substrates were kept still under the nozzles. The nozzle scanned the substrate and the surrounding area as shown in Fig. 2. The scanning started from the lower end to upward



Fig. 1 Big aerosol deposition machine

2. EXPERIMENTAL

direction (Y) in the figure by repeating the component cycles. Once the whole area was covered, the Y direction was reversed with a shift of half of the Y movement. When the nozzle comes down to lower than the starting point, it was directed to the starting point, and this concluded one cycle of the scanning. This cycle was repeated several times. The velocity of X movement ranged from 5 to 20 mm/s.

Ten square adhesive tapes in 5 pairs were placed on the substrate as shown in Fig. 2 before the deposition to measure the thickness of the film and to show the possibility of mask patterning. Since aerosol deposition is carried out at room temperature, ordinary adhesive tapes work very well. After the deposition the tapes were removed and the film thicknesses between the pairs of masks were measured by measuring the step heights with the surface roughness meter with a diamond stylus (Mitsutoyo, Surftest SV-3100S8). Surface roughnesses of the deposited films were also measured. The film thicknesses of some of the samples were measured also with an ellipsometer (J. A. Woollam, M-200).

3. RESULTS AND DISCUSSION

PZT films were successfully deposited on silicon wafers. The color of the films was yellowish green as the result of color blending of oxidized silicon surface and the PZT film.

The 10 black squares in the left part of Fig. 3 are the original wafer surfaces that had been covered by the masks. To indicate the location of thickness measurement we use the term upper as the location nearest to the orientation flat of the wafer. The rest are center, lower, left and right as you see them in the left part of Fig. 3.

The three black portions at the left, right and lower rims of the wafer are the original wafer surfaces that had been covered by adhesive tapes to hold the wafer during



Fig. 2 Scanning path of nozzle

the aerosol deposition.

In the right part of Fig. 3 the sectional profiles of each paired masked points are shown. The thicknesses of the films between the paired masked points were measured from these figures as the step height. The surfaces between the paired masks seem relatively flat.

The thicknesses of the film shown in Fig.3 were measured as upper $2.36\mu m$, center $2.14\mu m$, lower $2.31\mu m$, left $2.20\mu m$ and right $2.20\mu m$. The thicknesses of left, center and right which are lined in the horizontal direction are less diversified than the thicknesses of upper, center and lower which are lined in the vertical direction.

This tendency was observed in most of the films in this study as shown in Fig. 4. The figure shows the values of (maximum thickness - minimum thickness) of vertically lined three thicknesses and horizontally lined three thicknesses of each sample. The value of (maximum thickness - minimum thickness) shows the magnitude of variation of thicknesses. In most samples the variation of thicknesses are larger in the vertical



Fig. 3 An example of film thickness measurement

direction than in the horizontal direction. The average of (maximum thickness - minimum thickness) was 0.41μ m in the vertical direction and was 0.09μ m in the horizontal direction.

The thickness distribution of one sample that was measured with ellipsometer is shown in Fig. 5. The number of measured points was 137. The white parts in the distribution figure are the measured points that included the masked points, namely the substrate.

Fig. 5 also shows that the thickness variation along the horizontal (X) direction is smaller than that of the vertical (Y) direction.

This phenomenon is considered to have relation with the scanning method of the nozzle. As shown in Fig. 2 the deposition is carried out only when the nozzle moves toward the horizontal direction. The nozzle moves to the vertical direction only when the nozzle is out of the wafer and thus has no effect on the deposition. What the thickness distribution tells is that during the short time when the nozzle scans the horizontal line once or twice little thickness variation does exist. However when the nozzle moves from lower to upper positions or vice versa by repeating the component cycles many times, the thickness variation becomes larger.

In the parameters that have effects on the film formation, those that belong to the substrate surface are thought to be mostly constant all over the substrate. Therefore the parameters that belong to the aerosol jet, namely the density of aerosol or the amount of blown aerosol jet seems to change during the long deposition period, but they do not change during the short deposition period. So, to increase the uniformity of thickness it is important to increase the uniformity of aerosol density or aerosol amount blown from the nozzle.

The surface roughness profiles of the sample in Fig. 3 are shown in Fig. 6. The roughness profiles were cut off at wavelength of 8 mm and 0.025 mm to conform to the Y(cm)

Fig. 5 Thickness distribution of Aerosol deposited PZT film measured with an ellipsometer



Vertical direction Horizontal direction Fig. 4 Difference of the variation of the film thicknesses between the vertical and horizontal directions

Thickness (nm) 2600 2500 3 2400 2-2300 499480 2200 Ũ 2100 -1 2000 - 2-1900 -73 1800 --- 4 1700 -5 3 5 -5 -3 2 X(cm)

surface roughness standards. The sampling length is 8 mm and each roughness profile includes 10 samples. The profiles are different between the vertical and horizontal directions. Longer wavelength deviations are seen in vertical direction profile. This again tells the difference in the thickness of film in the long deposition period and relatively uniform thickness during the short period that corresponds to some horizontal scans. However the arithmetical mean roughness Ra is the same in both directions and was 0.18 μ m. In other samples Ra in the vertical direction.

The difference of appearance of the surfaces deposited by different nozzles is shown in Fig. 7. The film made by the straight beam nozzle has clearer horizontal stripes than the film made by the broadening beam nozzle.

This is attributed to the fact that the edges of the straight aerosol beams are clearer than those of broadening beams that have the gradually fading edges.

Therefore the broadening beam nozzle is suited to form large area films by overlapped scanning.

4. CONCLUSION

Lead zirconate titanate (PZT) films were deposited on 4 inch silicon wafers by aerosol deposition with nozzle scanning. The thicknesses of the deposited films were measured. The deviation of the thickness was smaller in the direction of nozzle movement than in the direction perpendicular to it. The reason for this is the density and/or amount of aerosol blown out of the nozzle fluctuate less in the short time range, but fluctuate more in the long time range. Therefore uniform aerosol generation is important to form films that have even thicknesses. The surface roughness was mostly the same along the nozzle movement direction and perpendicular to it. The nozzle that blows out a broadening aerosol beam is better for forming large area films than the straight beam nozzle.

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Straight beam nozzle



Horizontal profile

420

84.0 [mm] (x1.0)

Fig. 6 Surface roughness profiles of Aerosol deposited film in Fig. 3

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Broadening beam nozzle

Fig. 7 Surface difference of the films made by different nozzles