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Spatial Fluctuation of Electrical properties in Hf-Silicate Film Observed with Scanning Capacitance Microscopy

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1. Introduction

Recently, several intensive studies have evaluated the microscopic electrical properties of high-k gate dielectrics. Not only has insulating reliability been investigated by using electrical stressing [1], but also some studies have revealed the compositional change of high-k silicate films due to thermal annealing [2]. Such local fluctuation of composition might lead to deteriorating reproducibility of device performance. Therefore, quantifying the spatial fluctuation of dielectric properties in high-k films is quite important.

In previous work, we measured the local dielectric properties in Hf-based layered films with scanning capacitance microscopy (SCM) [3]. In this work, we used SCM to examine Hf-silicate films with simpler structure to find the origin of observed spatial variations in the SCM images.

2. Experiment

Hf-silicate dielectric films (with an atomic ratio of Hf/Si of 0.6/0.4) were deposited on *p*-Si substrate by using atomic-layer-deposition. After deposition, we annealed them in nitrogen ambient for 30s at various temperatures, as summarized in Table 1.

The detailed setups for our SCM are described in an earlier report [4]. Instead of the conventional optical deflection method, we employed a quartz resonator and etched tungsten wire for a self-sensing probe. Our SCM simultaneously yields three kinds of images of the topography, conventional dC/dV, and distance differential capacitance (dC/dZ). Figure 1 shows the SCM measurement geometry and the schematic structure of the sample used in this study. An AC modulation bias (V_{ac}) and DC offset bias (V_{dc}) was supplied to the sample, while the SCM probe was grounded through the capacitance sensor. All of the experiments were performed in high vacuum (~10^{-4} Pa) environment at room temperature.

3. Results and Discussion

Figures 2(a), (b), and (c) are a set of simultaneously obtained images of sample No.4 under bias conditions V_{ac}=0.4V_{P-P} at a frequency of 500 kHz and V_{dc}=0V, and Fig. 2(d) shows the dC/dZ image of the same area taken under V_{dc}=+5V (accumulated condition). The topography image of Fig. 2(a) shows the surface microroughness. The surface roughness of every sample in this study was almost the same and lower than 0.15 nm in RMS value. The dC/dV image of Fig. 2(b) seems to have slight contrast. On the other hand, the dC/dZ image of Fig. 2(c) shows topographic information overlapping with corrugation-like contrast showing little correlation with the dC/dV image. Further, under the accumulated condition, no specific contrast appears and the dC/dZ image simply reflects topography (Fig. 2(d)). From these results and scanning capacitance spectroscopy measurements (not shown) we concluded that the corrugation-like contrast in the dC/dZ image is due to inhomogeneous depletion depth just beneath the SCM probe tip.

Figures 3(a)~(e) show the changes of observed contrast between dC/dZ and corresponding topography image with annealing temperature. These images were taken under the same bias condition (V_{dc}=0V). When the sample was annealed at 1100°C (Fig. 3(a)), we observed a distinctive corrugation-like contrast in the dC/dZ image. However, the dC/dZ image of sample No.3 (Fig. 3(b)) and No.2 (Fig. 3(c)) show fainter contrast in comparison with Fig. 3(a). Further, no specific feature without topography structure appears in the dC/dZ image of as-deposited film, as shown in Figs. 3(d) and (e).

Local depletion depth is governed by (i) spatial distribution of charges in the dielectric film, (ii) equivalent thickness, and (iii) dopant concentration. The macroscopic C-V curves of each sample with Al top electrode (ϕ130μm) are shown in Fig. 4. Negative shift of C-V curves are clearly observed with increasing annealing temperature for samples No. 1, No. 2, and No .3. Therefore, the dC/dZ image of Fig. 3 (d) can be explained as resulting from positive charges homogeneously distributed in as-deposited film, and the slight contrast of Fig. 3(c) is caused by the inhomogeneous distribution of charges. On the other hand, the C-V curve of the sample No. 4 exhibits a little shift in comparison with that of No. 3. Therefore, the origin of the corrugation-like contrast in the dC/dZ image of Fig. 3(a), which might affect the reproducibility of device performance, cannot be attributed to the change of macroscopic C-V curve.

Figures 5 (a) and (b) show the high-angle annular dark-field (HAADF) imaging of samples No. 4 and No. 3. The image in Fig. 5(a) clearly depicts the typical microstructure formed by spinodal decomposition. The gray-colored region denotes the Hf-rich phase, and the typical dimensions of sample No. 4 (~20 nm) are obviously larger than those of No. 3 (5~10 nm). Thus, these images indicate the conspicuous development of phase separation with annealing at 1100°C.

From the results of shown in Fig. 3 and Fig. 5, we conclude that the spatial fluctuation of composition induced by the phase separation causes the corrugation-like contrast in the dC/dZ image of sample No. 4. However, the lateral scale of observed contrast from SCM observations (~200 nm) is much larger than that of the typical dimension of compositional fluctuation by HAADF imaging.

A possible explanation is as follows. As mentioned above, spatial fluctuation of dielectric constant in Hf-silicate film is not directly sensed by dC/dZ imaging, but sensed through the local depletion depth. Consequently the resolution of dC/dZ image, or "depletion depth image," is limited by the dopant concentration of the Si substrate (~10^{15}/cm^3). Therefore, the corrugation-like contrast in the dC/dZ image of sample No. 4 resulted from the convolution of two components; actual dimension of composition fluctuations in Hf-silicate film and dopant concentration of the Si substrate.

4. Conclusion

We have investigated the origin of the corrugation-like contrast of dC/dZ imaging observed in the Hf-silicate film annealed at 1100°C. We have concluded that the composition fluctuation in Hf-silicate film, which might be detrimental for the reproducibility of device performance, can be visualized through the spatial variation of depletion depth in dC/dZ image.

Acknowledgements

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Table 1 Sample Specifications

sample	Structure	Annealing condition
No. 1	HfSiO (5 nm) / SiO_2 (0.6 nm)	As-depo
No. 2	HfSiO (5nm) / SiO_2 (0.6 nm)	900℃/30s
No. 3	HfSiO (5 nm) / SiO_2 (0.6 nm)	1000℃/30s
No. 4	HfSiO (5 nm) / SiO_2 (0.6 nm)	1100℃/30s

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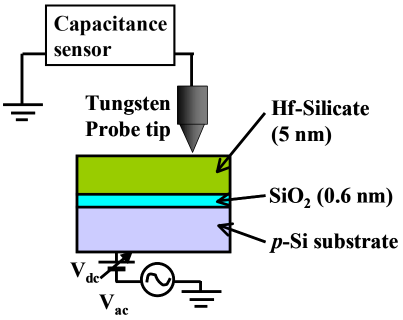

Fig. 1. Schematic diagram of stacked gate dielectric structure and measurement geometry

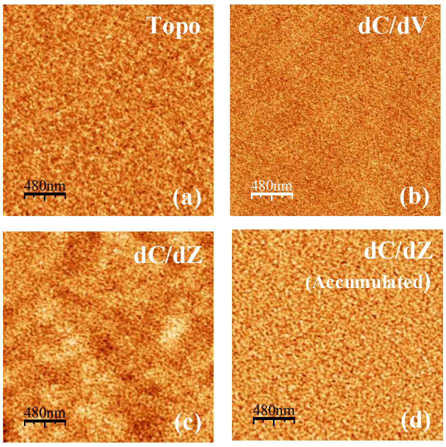

Fig. 2. A set of SCM images of sample No.4 simultaneously obtained (a) Topography, (b) dC/dV image, (c) dC/dZ image, and (d) dC/dZ image under accumulated condition. Scanned areas are 2.4 µm x 2.4 µm squares in all images.

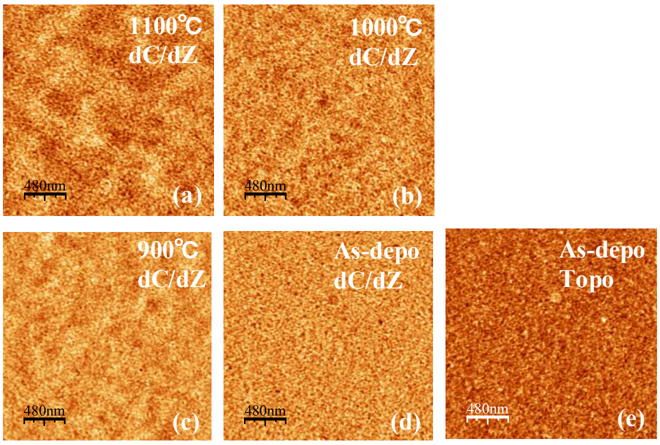

Fig. 3. (a) dC/dZ image of No. 4, (b) dC/dZ image of No. 3, (c) dC/dZ image of No. 2, (d) dC/dZ image of No. 1, and (e) corresponding topography image of No. 1. Scanned areas are 2.4 µm x 2.4 µm squares in all images.

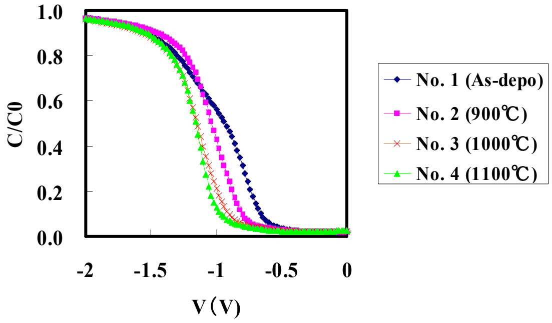

Fig. 4. Macroscopic C-V characteristics of samples No.1~4 with Al top electrode.

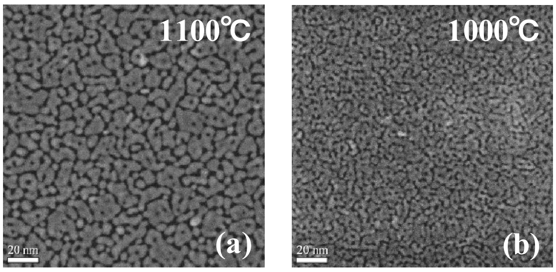

Fig. 5. 160 nm x 160 nm HAADF image of (a) No.4 (annealed at 1100C) and (b) No. 3 (annealed at 1000C).