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Current Voltage Characteristics through Grains and Grain Boundaries of High-k Dielectric Thin Films Measured by Tunneling Atomic Force Microscopy

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Abstract. Leakage current distributions of high-k dielectric thin films (8 nm HfSi_xO_y and 5 nm ZrO₂) were measured by tunneling atomic force microscopy (TUNA). In contrast to the thick HfSi_xO_y film, where grains and grain boundaries can be seen by TUNA topography maps, ZrO₂ films show no topography roughness. But large leakage current fluctuations can be seen for both dielectrics by TUNA current images. Higher leakage currents were found to flow through the grain boundaries of both analyzed high-k dielectric films. Furthermore, local current voltage (*I-V*) characteristics could successfully be measured precisely localized at grains and at grain boundaries, respectively, of the ZrO₂ film. The obtained local *I-V* curves showed significant differences between grains and grain boundaries, respectively. In the case of the ZrO₂ film, the leakage current through the grain boundaries was up to 8 times larger than that through the grains at a substrate voltage of -3.5 V.

Keywords: Tunneling AFM (TUNA), high-k, grain and grain boundary.

PACS: 77.55.D-

INTRODUCTION

The strong interest in high-k dielectric films is triggered by the applications of these materials as new gate oxides for metal insulator semiconductor (MIS) devices¹ and as capacitor materials for memory devices.² Required electrical properties of high-k dielectric films for such applications are low leakage currents and high dielectric constants. The electrical properties (i.e., leakage current and dielectric constant) of conventional dielectric films based on SiO₂ are usually characterized and evaluated by macroscopic measurements on MOS capacitors. But, in contrast to SiO₂, it is difficult to fully analyze and evaluate the electrical properties of high-k materials using conventional macroscopic measurements, since surface morphology and electrical properties of high-k materials are usually inhomogeneous on nanoscale. This is mainly due to their nanocrystalline structure after typical fabrication procedures. It is, therefore, important and necessary to investigate the surface morphology and the leakage current of high-k dielectric films with very high lateral resolution.

Knowledge obtained from those investigations would be extremely helpful to fabricate optimized high-k dielectric films with low leakage current.

The tunneling atomic force microscopy (TUNA) technique is well suited to this demand.³ The TUNA technique allows to simultaneously measure the local leakage current and the surface morphology of high-k dielectric films with extremely high current sensitivity (sensitivity limit is approximately 40 fA) and very high lateral resolution (< 20 nm), respectively. In our previous study, the surface morphology and leakage current distribution of high-k dielectric films (HfSi_xO_y and ZrO₂) has been simultaneously investigated at nanoscale by TUNA.⁴ It was found that the high leakage current paths through high-k dielectric films were located at the grain boundaries. Possible reasons for the larger leakage current through grain boundaries are different current conduction mechanisms for grains and grain boundaries or the minor thickness of grain boundaries compared to that of grains. So far, however, the mechanisms for the leakage current conduction of high-k dielectric films through grains and grain boundaries has not been investigated in detail and has not been fully understood.

The purpose of this paper is to measure local current-voltage (I - V) characteristics precisely at grains and grain boundaries for HfSi_xO_y and ZrO_2 high- k dielectric films by TUNA in order to clarify the current conduction difference between the grains and grain boundaries of high- k dielectric films.

EXPERIMENTAL PROCEDURES

Both, HfSi_xO_y and ZrO_2 high- k films (with thicknesses of about 8 and 5 nm, respectively) were deposited by atomic layer deposition on native oxide (thickness of 1.1 nm) on p-type silicon wafers. Subsequently, rapid thermal annealing (RTA) was performed for HfSi_xO_y at a temperature of about 1000 °C for 60 s to crystallize the films. For ZrO_2 films, RTA was performed in Ar atmosphere at 450 °C for 30 s.

Topography maps, leakage current maps, and local I - V curves were measured with a Bruker Dimension ICON tool equipped with NanoSope V controller (closed-loop system), an extended TUNA module (sensitivity 1 pA/V), and Pt/Ir coated silicon tips (the nominal tip radius of Pt/Ir coated probes is less than 25 nm). Within this work, substrate voltages from -10 V to +10 V can be applied to the sample. In all TUNA measurements, the substrate was negatively biased to avoid anodic oxidation of the sample surface in air. For negative substrate bias, the MIS structure formed by the conductive tip and the high- k dielectrics on p-Si substrate is in inversion, resulting in electrons being injected from the Si substrate. For all local I - V measurements in this study, the substrate bias was ramped from 0V to -10V. For all measurement (i.e., topography map, TUNA current map, and local I - V measurement) in this study the probe is in contact with the surface of the high- k layer in air. The TUNA experimental parameters for HfSi_xO_y and ZrO_2 are summarized in Table 1. Deflection setpoint is the parameter related to the contact force between the probe and sample during TUNA measurements (both, current maps and local I - V measurements). The contact force increases with increasing the positive value of the deflection set point.

Table 1. Parameters of TUNA current maps and local I - V measurements for HfSi_xO_y and ZrO_2 film.

	HfSi_xO_y	ZrO_2
Substrate voltage for TUNA current map (V)	-9.1	-2.7
Tip velocity for TUNA current map ($\mu\text{m/s}$)	1.50	1.50
Ramp rate for local I - V measurement (V/s)	0.50	0.25
Deflection setpoint (V)	-0.90	2.00

Additionally, topography map measurements and roughness analyses were carried out by tapping mode

atomic force microscopy (AFM). High-resolution transmission electron microscopy (HR-TEM) images were obtained by a FEI CM300 to analyze the film structure and morphology of the ZrO_2 films.

RESULTS AND DISCUSSION

Figure 1 shows a typical topography and corresponding current map of the 8.0 nm thick HfSi_xO_y film measured by TUNA. Crystalline grains can clearly be observed in the topography map. The height of the film at the grain boundaries is lower than that of the grains. The edges of the grains seem to be protuberant. Therefore, grains also exhibit local height variations. In the TUNA current map, the conductive structures (dark color), corresponding to the grain boundaries in the topography map, are clearly visible which indicate larger currents. In addition, two types of the grain boundaries are found to exist in this sample. One is a grain boundary with high leakage current (grain boundary A). Another is a grain boundary with low leakage current (grain boundary B)

Figure 1. (a) TUNA topography map and (b) corresponding TUNA current map of the HfSi_xO_y film. Scan size: $2 \times 0.5 \mu\text{m}^2$.

Figure 2 shows corresponding local I - V curves for the grains and grain boundaries of the HfSi_xO_y film. The local positions for the I - V curves through grains and grain boundaries were deduced from the TUNA topography map only. A difference between I - V curves through grains and grain boundaries can be observed.

The distribution of the local I - V curves through grains has a wide spread, while that through grain boundaries shows two tight spreads (i.e., spread A and B). The reason for the wide spread in the local I - V curves through grains is not apparent. Further measurements (e.g., HR-TEM analysis of the cross sectioned sample) are needed for clarification. The reasons for the two spreads in the local I - V curves through grain boundaries are also not fully understood yet but might be due to different current conduction mechanisms for two types of grain boundaries (i.e., grain boundary A and grain boundary B). Another reason might be a slight error in the measurement positions, since – in contrast to the measurements on ZrO_2 shown afterwards – the location of the measurement positions of the local I - V curves were not confirmed by subsequent TUNA current maps. In any case it should be emphasized that it can be observed that different types of grain boundaries as determined from the topography map show also very different current levels in the TUNA current map. Most important, leakage currents through grain boundaries are found to start at lower voltages for spread A. However, in the case of HfSi_xO_y films, the effect of the thickness variations on the I - V characteristics difference between grains and grain boundaries cannot be neglected. Namely, the reason for the larger leakage current at grain boundaries of spread A might be just due to a thinner thickness of the dielectric layer at the grain boundaries compared to that of the grains. It is, therefore, difficult to clarify the origin of the difference for the I - V characteristics between grains and grain boundaries for the given HfSi_xO_y films.

Figure 2. Local I - V curves through grains and grain boundaries of the HfSi_xO_y film measured by TUNA.

In contrast to the HfSi_xO_y thin film, it was found that no crystal grains could be observed for the given ZrO_2 film by tapping mode AFM (see Fig. 3). The surface of the ZrO_2 film was rather smooth. The average roughness and root mean square (RMS) roughness of the ZrO_2 surface was 0.12 nm and 0.18 nm, respectively. In order to confirm the morphology

of the ZrO_2 film, HR-TEM analysis of the cross sectioned sample was performed in a previous study⁴, as shown in Fig. 4. The HR-TEM image of the ZrO_2 clearly showed the nanocrystalline structure. It is also visible that the ZrO_2 surface and the underlying silicon oxide surface of the samples are very smooth, which confirm the results from the tapping mode AFM topography. Therefore, TUNA current maps will not be influenced by any artifacts arising potentially from topography (e.g., increased contact area at the grain boundaries or thickness variations).

Figure 3. Topography map of the ZrO_2 film measured by tapping mode AFM. Scan size: 500 x 500 nm².

Figure 4. HR-TEM image of ZrO_2 film annealed at 450 °C for 30 s (This image was reported in Ref. 4).

Figure 5 shows TUNA current maps of the ZrO_2 thin film before and after local I - V measurements at a grain and a grain boundary, respectively. Figure 5 (a) shows the initial TUNA current map of the ZrO_2 film before any local I - V measurement. A structure of areas with different current densities which will be attributed to grains and grain boundaries of the ZrO_2 film can clearly be observed in the TUNA current maps. The larger leakage current paths are located at the grain boundaries of the ZrO_2 film, although the surface of the ZrO_2 is rather smooth compared to the HfSi_xO_y film. Those findings in our previous study⁴ could be confirmed using the closed-loop system. This result indicates that the main reason for the larger leakage current at grain boundaries for this film is not due to a thickness variation of the layer.

Figure 5 (b) shows the TUNA current map after the first local I - V measurement at a grain boundary. The measured spot (spot 1) showed very high leakage current with an oscillating noise along the scan direction of the cantilever due to the breakdown of the

measured spot. Interestingly, another weak spot at a different grain boundary (spot 2) was observed in the second TUNA current map which already showed rather high leakage current in the initial TUNA current map (Fig. 1(a)). Probably the electrical stress applied during the initial mapping led to a dielectric breakdown of this spot.

Figure 5 (c) shows the TUNA current map after the second local I - V measurement through a grain. The measured spot at the center of the grain (spot 3) also showed very high leakage current similar to the measured spot at the grain boundary (spot 1). The measured points were exactly at the grain and grain boundary of the ZrO_2 films, respectively (compare the TUNA current maps before and after the local I - V measurements).

Figure 5. TUNA current maps of ZrO_2 films (a) before any local I - V measurement, (b) after the first local I - V measurement at a grain boundary, (c) after the second local I - V measurement at a grain. Scan size: $1.33 \times 0.5 \mu\text{m}^2$.

For our setup, the measurement positions for the local I - V curves could be very accurately set into the grains or the grain boundaries without any image drift by the closed-loop system. An additional remark which should be made here is that the resolution of the TUNA current map is not deteriorated after the local I - V measurements. This result indicates that the shape of

the tip apex for the conductive tip is practically unchanged by the local I - V measurements. Therefore, the I - V characteristics at grains and grain boundaries can be compared neglecting the tip shape degradation during the measurements. This measurement cycle (i.e., TUNA current map $\rightarrow$ local I - V at grain boundary $\rightarrow$ TUNA current map $\rightarrow$ local I - V at grain) was repeated 10 times using the identical experimental parameters (i.e., the same deflection set point, scan rate, substrate voltage, and voltage ramp rate for local I - V measurements) summarized in Table 1.

Figure 6 shows the local I - V curves for the ZrO_2 film at grains and grain boundaries, and the TUNA current map after the fifth cycle of the measurement, respectively. The local I - V curves show significant differences between curves at grains and grain boundaries, respectively. The leakage current through the grain boundaries exceeds the noise level and reaches the sensor limit for the TUNA module at lower substrate voltages, than the current through the grains. The leakage current through the grain boundaries is up to 8 times larger than that through the grains at a substrate voltage of -3.5 V . In addition, the TUNA current map in Fig. 6 (b) shows that the measured spots are accurately located at grains and grain boundaries. This result ensures the reproducibility and reliability of the local I - V curves. Namely, the local I - V curves could be exactly measured at grains and grain boundaries. Future work will focus on the detailed evaluation of the current conduction mechanisms through the grains and grain boundaries.

Figure 6. (a) Local I - V curves for the ZrO_2 film at grains and grain boundaries. (b) TUNA current map after several local I - V measurements at grains and grain boundaries. Scan size: $2 \times 0.5 \mu\text{m}^2$.

CONCLUSIONS

The potential of TUNA for the evaluation of high-k materials with respect to the leakage current could be demonstrated. The topography map and the leakage current distribution of the HfSi_xO_y thin film could be simultaneously measured by TUNA. The leakage current of HfSi_xO_y through grain boundaries was found to be larger than that through grains and two distinct types of grain boundaries in terms of local I - V characteristics could be distinguished. The major leakage current paths of the ZrO_2 film were found to be located at the grain boundaries from the TUNA current map, although the surface morphology of the ZrO_2 film was rather smooth. Together with results from HR-TEM analysis (i.e., very smooth interface of the underlying native oxide layer) this shows that the reason for the larger leakage current at grain boundaries is not due to a thickness difference between grains and grain boundaries. Furthermore, local I - V curves exactly located at either grains or grain boundaries could successfully be measured. The local I - V curves were found to show a significant difference between grains and grain boundaries. These results confirm that the grain boundaries of the ZrO_2 film are the dominant paths for the leakage current.

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