Transactions of the Materials Research Society of Japan Vol.28 19-22 (2003)

Characterization Techniques for Next Generation ULSI Materials using Synchrotron Radiation — Current Status and Future Prospects —

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Si-ULSI has reached the sub-100 nm regime. In the past decades, performance of Si-ULSI devices has been improved by continuing miniaturization or downscaling. In the sub-100 nm regime, however, simple downscaling faces severe physical limitations, resulting that new materials and processes are requested to continue the evolution of ULSI devices. In this circumstance, synchrotron radiation research is expected to become important for characterization and/or analysis of the next generation ULSI materials and processes Current status and future prospects of characterization techniques for ULSI materials research using synchrotron radiation are reviewed.
Key words: X-ray absorption fine structure, X-ray photoemission spectroscopy, X-ray reflectivity, X-ray microscopy, X-ray microdiffraction

1. INTRODUCTION

In the past decades, performance of Si-ULSI devices has been improved by continuing miniaturization or downscaling. In the sub-100 nm regime, however, simple scaling faces severe physical limitations, resulting that new materials and processes are requested to continue the evolution of ULSI devices. For example, thickness reduction of SiO_2 gate dielectric films will create the problem of increased electricity leakage, which will increase semiconductor power consumption. In addition, ULSI data-processing signal delay caused by interconnects is becoming a major issue, raising the demand for the development of new materials.

Figure 1 shows typical parts where introduction of new materials are currently under consideration. High dielectric constant (high-κ) materials, polycrystalline SiGe electrodes, Cu interconnects and low dielectric constant (low-κ) materials are considered as potential replacements for SiO_2 gate dielectrics, poly-Si gate electrodes, Al interconnects and SiO_2 interlayer dielectrics, respectively. In this circumstance, synchrotron radiation (SR) research is expected to become important because various characterization techniques are required for developments of new materials and processes used in the next generation ULSI devices.

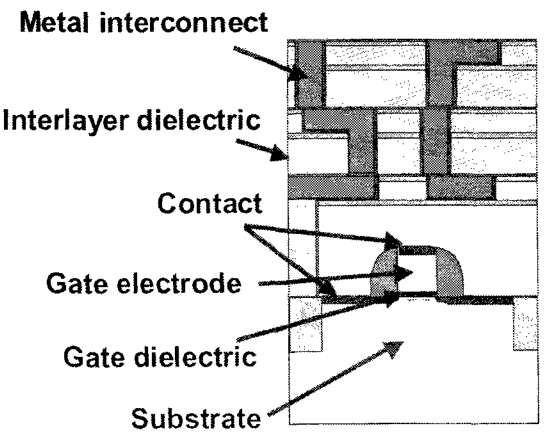

Fig. 1. Typical parts where introduction of new materials are currently under consideration.

In the following sections, we assess the present and future requirements for characterization and/or analysis techniques using SR to develop the new materials and processes such as high-κ gate dielectrics, Cu interconnects and low-κ interlayer dielectrics.

2. NEW MATERIALS FOR NEXT GENERATION ULSI DEVICES AND CHARACTERIZATION TECHNIQUES USING SR

2.1 High-κ materials

SiO_2 systems are now nearing their size limits with physical thicknesses currently down to 1.5 nm. This leads to impermissible large leakage currents (Fig. 2). As an alternative to SiO_2 systems, much work has been done on high-κ materials as a means to provide a substantially thicker dielectric for reduced leakage and improved gate capacitance. A systematic consideration of the required properties of gate dielectrics indicates that the key guidelines for selecting an alternative gate dielectric are (a) permittivity, band gap, and band alignment to silicon, (b) thermodynamic stability, (c) film morphology, (d) interface quality, (e) compatibility with the current or expected materials to be used in processing for CMOS devices, (f) process compatibility, and (g) reliability [1]. Many dielectrics appear favorable in some of these areas, but very few materials are promising with respect to all of these. The most commonly studied high-κ gate dielectric candidates have

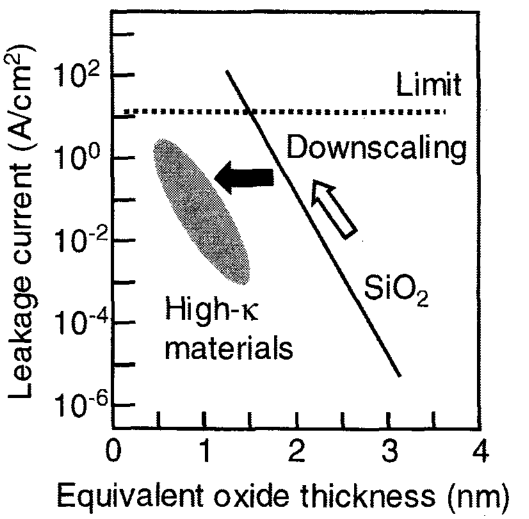

Fig. 2. Physical limits of SiO_2 systems.

been oxides or silicates of Zr and Hf [2-5]. These films have however many subjects which must be conquered about film and interface quality, thermodynamic stability and so on.

To investigate the film quality and the thermodynamic stability of amorphous films, X-ray absorption fine structure (XAFS) measurements are very effective because the bond length and the coordination number can be determined. However, very few works have been reported yet. This is because XAFS measurements of very thin films (less than 5 nm-thick) needed very long data taking time. Recently, XAFS measurements by total electron yield (TEY) method have come into use for thin film measurements [6]. Since this method is much efficient as compared with usual X-ray fluorescence (XRF) method with using single solid-state detector, measurement time can be shortened. This is a great advantage for characterization of high-κ materials. Thus this method should be promoted.

X-ray photoemission spectroscopy (XPS) is also a useful characterization tool for high-κ gate dielectrics. Various laboratory XPS equipments have become commercially available and have been widely used for chemical state analysis of thin films. However, the probing depth is limited at most 4 nm because these equipments usually use Al*Kα* (1486.6 eV) or Mg*Kα* (1253.6 eV) lines for excitation. Even excitation by strong soft X-rays from SR cannot change the probing depth. Extension of the probing depth up to 10 nm will be greatly effective to analyze high-κ gate dielectrics because the depth distribution of the components can be analyzed precisely with take-off angle dependence measurements. Although a straightforward way to extend the probing depth is to raise the photon energy for excitation, rapid decrease in subshell photo-ionization cross section at hard X-ray regions had prevented us to use hard X-ray excitation until quite recently. However, high resolution XPS with hard X-ray (5950 eV) excitation was succeeded at BL29XU of SPring-8 [7]. This is because extremely high flux from X-ray undulators of the third generation synchrotron light sources well compensates the decrease in the cross section. Using this technique, take-off angle dependence of Si 1s spectra for HfO_2 (4 nm)/SiO_2 (1 nm)/Si(100) structures were precisely measured. As a result the substrate Si 1s peak at very low take-off angle of 8° could be clearly detected, demonstrating that this system can non-destructively probe the embedded interlayers and/or interfaces located deeper than 35 nm from the surface. Thus, this technique is also very interesting for characterization of high-κ dielectric systems.

Also, X-ray reflectivity (XRR) and grazing incidence X-ray diffraction (GIXD) measurements might be useful techniques for characterization of high-k materials even though few works have been reported using SR.

2.2 Cu interconnects

In modern ULSI devices, more than 100 million transistors have to be integrated. As the number of devices increases and the relative feature size becomes smaller, the overall performance is increasingly determined by interconnect design, technology and materials. Propagation delay and increased power consumption due to capacitive coupling between adjacent metal lines poses a serious problem to improve ULSI performance. To resolve the issue, the interconnect resistance and the relative dielectric constant of interlayer dielectric film have to be reduced. Thus, Al-based interconnects are being replaced by Cu interconnects due to its higher conductivity and improved electromigration (EM) resistance. However, EM phenomena remain as significant reliability issues for Cu interconnects as the dimensions of the interconnect lines continue to shrink. The interconnect structures in modern ULSI devices are currently 200 nm wide and encapsulated with special metallic or dielectric barriers to prevent Cu diffusion into the silicon. An understanding of the parameters affecting EM is very important for device design. Thus EM in Cu interconnects must be studied in a realistic device structures.

For this purpose, transmission X-ray microscopy (TXM) might become an important new tool. A

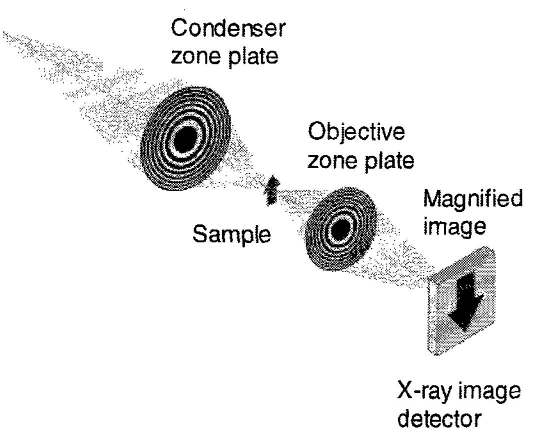

Fig. 3. Schematic diagram of transmission X-ray microscope. The X-rays are collected by the condenser zone plate and projected onto a sample. The image of this region is magnified by the objective zone plate and projected onto the X-ray image detector.

schematic diagram of general transmission X-ray microscope is shown in Fig. 3. Monochromatized SR are collected by the condenser zone plate and projected onto a sample. The image of this region is magnified by the objective zone plate and projected onto the X-ray image detector. Recently, sub-100 nm spatial resolution has been achieved at wide X-ray range up to about 10 keV. Schneider *et al.* have demonstrated that it is possible to visualize the void formation process by absorption contrast during an in-situ EM experiment using the X-ray microscope with 40 nm spatial resolution at 1.8 keV photon energy installed at the Advanced Light Source (ALS) [8,9]. This will be surely an important tool to study the EM phenomena in Cu interconnects. At the European Synchrotron Radiation Facility (ESRF), Zelnike-type phase contrast X-ray microscopy at 4 keV photon energy with 60 nm spatial resolution was applied to imaging of Cu interconnects and clear phase contrast micrographs of Cu interconnect structures were obtained [10]. For future perspective of these X-ray microscopes, developments of high-resolution tomography are expected.

To study Cu interconnects, X-ray microdiffraction is also a powerful technique because strain distribution and grain orientation, which are essential factors for EM phenomena, can be measured.

Zhang *et al.* first applied X-ray microdiffraction measurements to study Cu interconnects [11]. They used monochromatic X-ray microbeams with a size of 1×5 μm^2 focused by a phase zone plate for the incident beam of the microdiffraction system installed at the Advanced Photon Source (APS). They showed large compressive strain was found around the hillock area after EM test.

Recently, Tamura *et al.* developed a powerful new microdiffraction technique to obtain the orientation and strain map of polycrystalline thin films with sub-micrometer spatial resolution [12]. This was achieved by using white X-ray microbeams with a size of 0.8×0.8 μm^2 produced by a pair of orthogonal elliptical mirrors in a Kirkpatrick-Baez (KB) configuration [13]. Combination of white microbeams, large area X-ray charge coupled device camera and on-line two-dimensional diffraction pattern analysis software enabled to determine rapidly the orientation and strain map of polycrystalline thin films even if multiple grains were illuminated by the microbeams. This technique is like electron back-scatter diffraction (EBSD), but is superior to EBSD in strain sensitivity, accuracy of grain-orientation measurements and depth probing capability. Thus, this technique is a very powerful tool to study Cu interconnects.

In the future, X-ray microscope and microdiffraction should be combined to provide simultaneous spatially resolved data about the mass flow and the corresponding grain orientation during EM experiments.

2.3 Low-κ materials

Development of new low-κ interlayer dielectric materials is another important subject to resolve the propagation delay and power dissipation of the interconnect structure. Recently, developments of very low-κ films ($\kappa \leq 2$) have been tried to introduce a lot of nanometer-sized pores into dielectric films. In such porous low-k materials, however, the pore structure seriously affects other crucial properties. It is therefore important to characterize the structure of pores and their size distributions. XRR and small angle X-ray scattering (SAXS) are promising for the characterization of the structural properties of porous thin films.

Lee *et al.* demonstrated that the film thickness, average film density, density depth profile, wall density, and porosity of low-κ films could be determined quantitatively and sensitively by XRR [14]. SAXS is a powerful technique for determining density fluctuations in the scale of sub-nm.

Omote *et al.* applied SAXS in reflection geometry using offset $\theta/2\theta$ scans for characterizing pore-size distribution in porous low-κ dielectric films and succeeded to determine the pore-size distribution in the range from sub-nm to several nm [15]. This work was performed using a laboratory X-ray source. If using SR, the SAXS technique using a transmission geometry might be applied for measuring thin film, because high photon flux enable us to detect very weak scattering intensity. This might make detectable range of pore-size distribution much wider.

3. SUMMARY

Current status and future prospects of characterization and/or analysis techniques using SR for developments of new materials and processes used in the next generation ULSI devices were reviewed. Various techniques, such as XAFS, XPS, TXM, X-ray microdiffraction, XRR, SAXS and so on, are expected to become powerful tools. I would like to encourage researchers, who are facing difficulties due to the limits imposed by conventional experimental techniques, to use SR facilities to solve the difficulties.

Acknowledgments

The author wishes to express his appreciation to Dr. Y. Uehara of Mitsubishi Electric Corporation and Dr. K. Kobayashi of Japan Synchrotron Radiation Research Institute for providing their experimental results.

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(Received July 22, 2003; Accepted August 21, 2003)