

## Relative Density Characterization of Patterned Low-k material by VEELS

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### Abstract

Electron Energy Loss Spectroscopy (EELS) equipped with Transmission Electron Microscope (TEM) has been carried out to characterize nm-order structures of low-k interconnect dielectrics. Both compositional and structural change after plasma processes such as dry etch and ashing were performed by using conventional core EELS (core electron excitation region: C-EELS) for the compositional analysis and valence EELS (V-EELS) for relative thickness measurement, and results were discussed related to density of low-k dielectrics regions in a Cu/Low-k interconnect cross-section.

### Introduction

For a rapid development of low-k materials in copper metallization for ULSI devices, nm-order characterization methods of low-k materials after several processes such as dry etching, metal sputter deposition are required to optimize materials and processes quickly. We have already reported a dielectric constant measurement technique by VEELS [1]. Recently some difficulties were pointed out to measure the dielectric constant from VEELS [2] so that it is not an easy way to characterize damages in the low-k materials.

In this study, a 2-dimensional relative thickness measurement has been applied to obtain density or process damage information of low-k materials at higher spatial resolution by VEELS combined with conventional compositional analysis by core-EELS (CEELS).

### Experimental

A porous SiOC (p-SiOC) of  $k=2.4$  patterned structure on a SiO<sub>2</sub> layer with Cu interconnect (Ta/TaN barrier) was used for characterization. A TEM cross sectional specimen was prepared by a FIB method. The TEM, STEM, EELS analysis were performed with JEOL JEM-2100F at 200keV, equipped with GATAN Imaging Filter (Tridiem). Typical energy resolution was 0.8eV FMHM. EELS were recorded with the illumination semi-angle  $\alpha$  of about 5.0 m-rad and the collection

semi-angle  $\beta$  of about 40 m-rad at probe diameter of 1 nm.

Specimen thickness  $t$  was determined from the low-loss region of the EELS by the log-ratio method [3],

$$t/\lambda = \ln(I_t/I_0) \quad (1)$$

where  $I_t$  and  $I_0$  are integrals of the entire spectrum and of the zero loss peak, respectively.  $\lambda$  is inelastic mean free path. To estimate absolute specimen thickness, it is necessary to know  $\lambda$  however that of the low-k p-SiOC material is unknown. So we used  $\lambda = 129\text{nm}$  of SiO<sub>2</sub> which was under layer of patterned samples. We assumed that the thickness of each TEM specimen was constant by FIB thinning. We used STEM spectrum imaging mode for acquisition of a 2-dimensional VEELS map of the patterned area and then derived  $t/\lambda$  profile. Quantitative analysis of the SiOC region were also carried out at the same region by CEELS.

### Results and Discussion

#### 1. Composition Analysis

Figure 1 shows a STEM image of the patterned structure, and Figure 2 shows composition profiles analyzed along an arrow in Fig.1. Carbon composition gradually decreased close to the TaN side wall in the area of 40nm. Such composition analysis by CEELS is often used for low-k films [4] and is used to estimate the degree of low-k damages. However we could know only the change of composition such as carbon depletion and this approach is not so sensitive for structural changes which might affect electrical and mechanical properties.

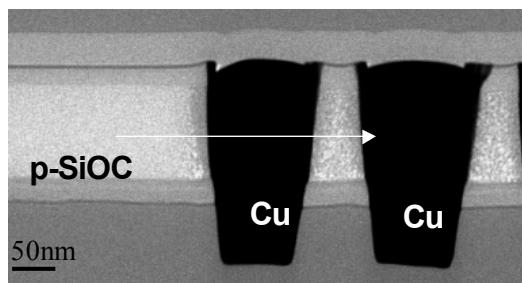


Fig. 1. Bright field STEM image of the patterned p-SiOC film.

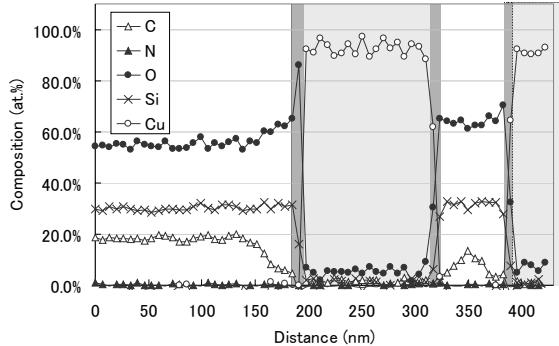


Fig. 2. Compositional line profile of the patterned p-SiOC film. Line is corresponding to the arrow in Fig.1.

## 2. Relative density characterization

Figure 3 shows a relative thickness ( $t/\lambda$ ) profile derived from VEELS at the same line in Fig.1. In composition profiles (Fig.2), the slope of carbon decrease was simple in the range of 40nm however the relative thickness profile was rather complicated, i.e. thickness was firstly decreased at 40-20nm from side wall then increased from 20nm towards the side wall. The inelastic mean free path varies by chemical composition but it does not change too much if the composition changes slightly [2]. Suppose that the inelastic mean free path of p-low-k at each position is constant, the changes of  $t/\lambda$  value might indicate that the density or porosity of the p-SiOC has been changed at the adjacent area of the side wall. Carbon depletion from the p-SiOC during patterning processes such as dry etch might have caused shrinkage of the p-SiOC to increase of density at 0-20nm from the side wall (region A in Fig.3). This shrinkage has secondarily set off the fracture at outside of the dense region (20-40nm from the side wall; region B in Fig.3).

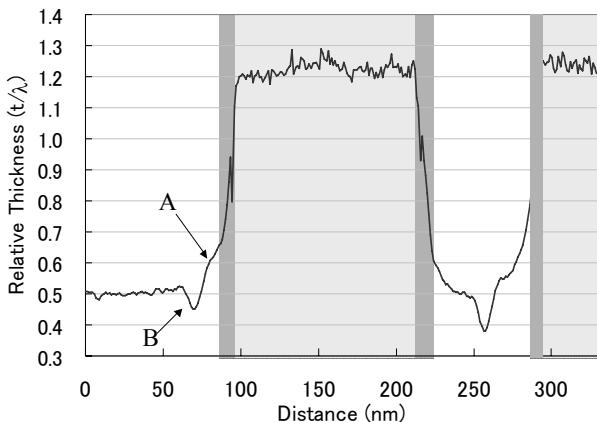


Fig. 3. Relative thickness line profile of the patterned p-SiOC film. Line is corresponding to the arrow in Fig.1.

Figure 4 shows a 2-dimension relative thickness map of the sample. It is clearly seen that the distribution of dense and light regions of the p-SiOC at the adjacent of the TaN/Ta/Cu lines. Notably in a dielectric region between interconnect lines (a right p-SiOC area with a SiO<sub>2</sub> cap layer in Fig.4), a triangular low density region and a line shape high density region is seen at a bottom and a top area, respectively. These changes in density of the p-SiOC might affect the electrical/mechanical reliabilities.

These information of density cannot be obtained by the EELS conventional composition analysis, but uniquely obtained from this proposed method.

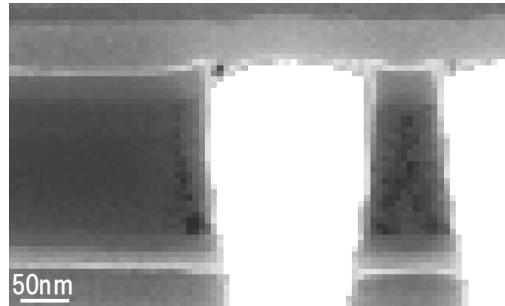


Fig. 4. 2-dimensional relative thickness map of the patterned p-SiOC region.

## Conclusion

Using the relative thickness derived from VEELS technique, 2-D distributions of relative density have been characterized. At the adjacent of the side wall, bottom and top layers, dense and light regions have been observed by carbon depletion damage which might be generated during Cu/Low-k processing. It has been demonstrated that the combination of conventional composition analysis and relative thickness measurement is very useful for characterization of density or damages in the patterned Cu / low-k structure for advanced ULSI processing, and it might shorten process development cycles.

## References

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