# **Composition analysis of spin-on-glass films**

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### **1. Introduction**

A spin-on-glass (SOG) is often applied to semiconductor manufacturing as an intermediate layer for multilayer pattern transfer process to form smaller critical dimension. It has already known that well-controlled SOG surface is needed to obtain precise photoresist patterns. For example, pattern lifting after development has been reported onto hydrophilic SOG surface. The surface was dramatically changed by loading only a small amount of methylsilsesquioxane [MSQ =  $(CH_3)SiO_{1.5}$ ] to the original SOG formulation, and photoresist patterns were finally obtained on the modified surface [1]. The surface change was explained by concentrated hydrophobic methyl group of MSQ at the SOG film surface.

In order to investigate the depth profile of MSQ more precisely, near edge x-ray absorption fine structure (NEXAFS) measurements were performed on a bunch of SOG films having MSQ in varying amounts.

## 2. Experimental

The SOG films were coated after baking a precursor sol [1]. The precursor sol was a mixture of tetramethoxysilane and methyltrioxysilane to afford a co-polymerized  $SiO_2/MSQ$  (= 80/20) sol. MSQ homo-polymer was loaded in varying amounts to the co-polymerized sol. Three samples were formulated which contain the MSQ homo-polymer in 2.5, 5.0, and 10.0 %, referred to as MSQ-2.5%, MSQ-5.0%, and MSQ-10.0%, respectively. For reference, extra two films were prepared from the original co-polymer and MSQ homo-polymer, which are referred to as MSQ-0% and MSQ-100%, respectively.

NEXAFS measurements were performed at the BL-8 of SR Center at Ritsumeikan

University, equipped with a grazing incidence monochromator with а varied-line-spacing plane grating [2]. Silicon L-edge NEXAFS spectra of the samples were measured in both partial and total electron yield by a micro-channel plate detector with retarding grids. In the partial electron yield (PEY), the retarding voltage was set to -50 V, while that was 0 V in the total electron yield (TEY). PEY spectra have information of the surface while TEY spectra have information deeper from the surface.

## 3. Results and Discussion

Figure 1 shows Si L-edge NEXAFS spectra of the samples in PEY. The spectral line-shapes of MSQ-0% and MSQ-100% were very different. Therefore, we could analyze the MSQ composition from the spectral line-shape. The spectral line-shapes of all the SOG films were similar to that of the MSQ-100%, indicating that the surfaces of the SOG films are covered with MSQ.

Figure 2 shows Si L-edge NEXAFS spectra of the samples in TEY. The spectral line-shape of MSQ-10.0% was similar to that of MSQ-100%, while those of MSQ-2.5% and MSQ-5.0% seem to be mixtures of MSQ-0% and MSQ-100%. These spectra were well reproduced by the linear combinations of MSQ-0% and MSQ-100% and the ratio of the MSQ-100% component increased with a loaded amount of MSQ. These results indicated that the surface MSQ layer-thickness increases with a loaded amount of MSQ.

#### 4. Conclusions

We have investigated the depth profile of MSQ in the SOG films by NEXAFS. We have found that the surfaces of the SOG films are covered with MSQ and the surface MSQ layer-thickness can be controlled by a loaded amount of MSQ homo-polymer.



#### References

[1] H. Sugita et al., J. Appl. Polym. Sci. 88, 636 (2003).

[2] H. Namaba et al., J. Synchrotron Rad. 5, 557 (1998).



spectra of the SOG films in PEY.



spectra of the SOG films in TEY.