



# Etch Damages of Ovonic Threshold Switch (OTS) Material by Halogen Gas Based-Inductively Coupled Plasmas

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The ovonic threshold switch (OTS) selector device is suitable for a phase-change random access memory (PRAM) requiring instantaneous high-output power due to the high-current density and high-speed operation. An amorphous chalcogenide-based compound composed of As-Te-Ge is a candidate for OTS materials and has excellent selector performances such as low leakage current, fast switching speed, scalability, and thermal stability. However, this material is known to suffer damage due to easy halogenation when exposed to halogen gas-based plasmas. In this study, the etch damages of OTS surface during the halogen gas based-inductively coupled plasma (ICP)-reactive ion etching (RIE) using CF<sub>4</sub> and Cl<sub>2</sub> were investigated. The OTS etched with Cl<sub>2</sub> showed a higher etch rate compared to that with CF<sub>4</sub>. However, the surface roughness was lower for the OST etched with Cl<sub>2</sub> than that etched with CF<sub>4</sub>. Also, the thickness of halogenated layer during the etching was also thinner for Cl<sub>2</sub>-etched OST than CF<sub>4</sub>-etched OST. Therefore, compared to CF<sub>4</sub>-etched OST, Cl<sub>2</sub>-etched OST was less damaged by the etching. In addition, it is found that, among the OST components of As, Te, and Ge, Ge was mostly halogenated while As and Te are not significantly halogenated during the etching. © 2019 The Electrochemical Society. [DOI: 10.1149/2.0051906jss]

Manuscript submitted January 2, 2019; revised manuscript received May 21, 2019. Published June 17, 2019.

Recently, phase-change random access memory (PRAM) is intensively investigated as one of the promising alternatives to the next-generation nonvolatile memory (NVM) due to the excellent performance such as high-speed, high-density, low-power consumption, scalability, etc.<sup>1-6</sup> The most investigated PRAM material is amorphous chalcogenide-based compound Ge<sub>2</sub>Sb<sub>2</sub>Te<sub>5</sub> (GST), and many studies on GST-based PRAM have been reported.<sup>7-11</sup> However, the leakage current is generated between each cell of the PRAM, and noise is generated due to the leakage current, so that the device is difficult to be implemented due to a failure of reading problem.<sup>12</sup> To solve these problems, a selector device has been investigated. Among the various selector devices, the most suitable type for PRAM is an ovonic threshold switch (OTS) selector device using an ovonic threshold switch effect.<sup>13-15</sup> One of the most promising materials for OTS is the amorphous chalcogenide-based compound (As-Ge-Te).<sup>16,17</sup>

In general, the amorphous chalcogenide-based compounds are known to be easily halogenated and are easily damaged by the exposure to halogen gas-based plasmas during the etching process.<sup>18</sup> Many studies on the etching of GST materials and their damage characteristics can be found in previous studies.<sup>19-24</sup> However, the etch characteristics of OTS materials including the damages by the etching using halogen gas-based plasmas have not been reported yet, and it is very important to understand the effect of the halogen etch gases for low-damage OTS etching.

In this study, the etch characteristics of the OTS material composed of amorphous chalcogenide-based compound (As-Te-Ge) were investigated for the inductively coupled plasma (ICP)-reactive ion etching (RIE) using halogen-based gases and the etch characteristics including damage characteristics between Cl<sub>2</sub> and CF<sub>4</sub> were compared. After the etching of the OTS using the halogen gas-based plasmas, the physical damage and chemical damage were investigated.

## Experimental

The sample used for the OTS etching is a multilayer thin film including 25 nm thick OTS material composed of amorphous chalcogenide-based compound (As-Te-Ge) and was stacked as carbon layer (1 nm)/OTS (25 nm)/carbon layer (5 nm) for oxidation prevention on SiO<sub>2</sub>/Si substrates by rf magnetron sputter deposition using an As-Te-Ge target. The etching experiments for the multilayer thin film

including OTS material were performed with an 8-inch diameter ICP-RIE system. The ICP-RIE system was operated with the ICP source power of 13.56 MHz 100 W while biasing the substrate at 50 W of 13.56 MHz. The etching was conducted at 3.0 mTorr of Cl<sub>2</sub> (100 sccm) or CF<sub>4</sub> (100 sccm). During the etching, the substrate was maintained at room temperature.

The etch depth was estimated by cross-sectional field emission scanning electron microscopy (FE-SEM, Hitachi S-4700). Also, after the etching, the possible change of material property was observed by Raman scattering spectroscopy (WITEC 2000, 532 nm wavelength) and the surface roughness was investigated by atomic force microscopy (AFM, Bruker Innova). The chemical binding characteristics of the OTS material exposed after the etching were investigated by X-ray photoelectron spectroscopy (XPS, ESCA2000, VG Microtech Inc.) using a Mg Kα twin-anode source. The XPS spectra were deconvoluted using a least square fitting technique by the Advantage fitting program supplied by VG Microetch.

## Results and Discussion

Figure 1a shows the SEM image of the 31 nm thick blanket C/OTS/C thin films stacked as carbon layer (1 nm)/OTS (25 nm)/carbon layer (5 nm) on SiO<sub>2</sub>/Si wafer and Figure 1b shows the etch depth of OTS (including 1 nm thick carbon layer on the top of OTS layer) as a function of etch time using 100 W of the 13.56 MHz ICP source power and 50 W of 13.56 MHz bias power with 3.0 mTorr of Cl<sub>2</sub> (100 sccm) and CF<sub>4</sub> (100 sccm). As shown in Figure 1b, the etch rate of OTS with Cl<sub>2</sub> was ~4 Å/s while that with CF<sub>4</sub> was ~0.5 Å/s. Table I shows the boiling points for halogen compounds of As, Te, and Ge.<sup>25,26</sup> As shown in the Table I, stable boiling points of As, Te, and Ge

Table I. Boiling points of the OTS-halogen compounds.

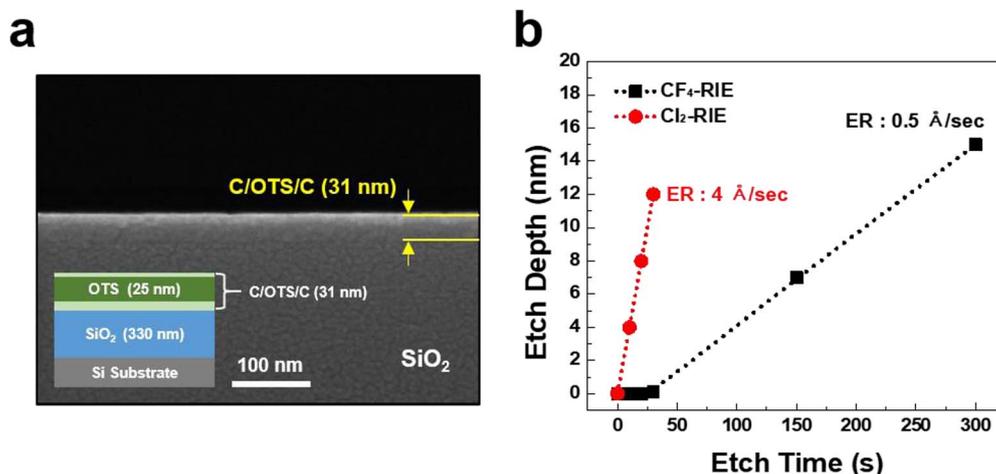
	As		Te		Ge	
	Bonding	b.p. (°C)	Bonding	b.p. (°C)	Bonding	b.p. (°C)
F	AsF <sub>3</sub>	60.4	TeF <sub>4</sub>	196	GeF <sub>2</sub>	130
	AsF <sub>5</sub>	-52.8	TeF <sub>6</sub>	-37.6	GeF <sub>4</sub>	-36.5
Cl	AsCl <sub>3</sub>	130.2	TeCl <sub>2</sub>	328	GeCl <sub>2</sub>	450
	AsCl <sub>5</sub>	-50	TeCl <sub>4</sub>	380	GeCl <sub>4</sub>	86.6

b.p.: boiling point (STP, standard temperature and pressure; 1 atm, 25°C)

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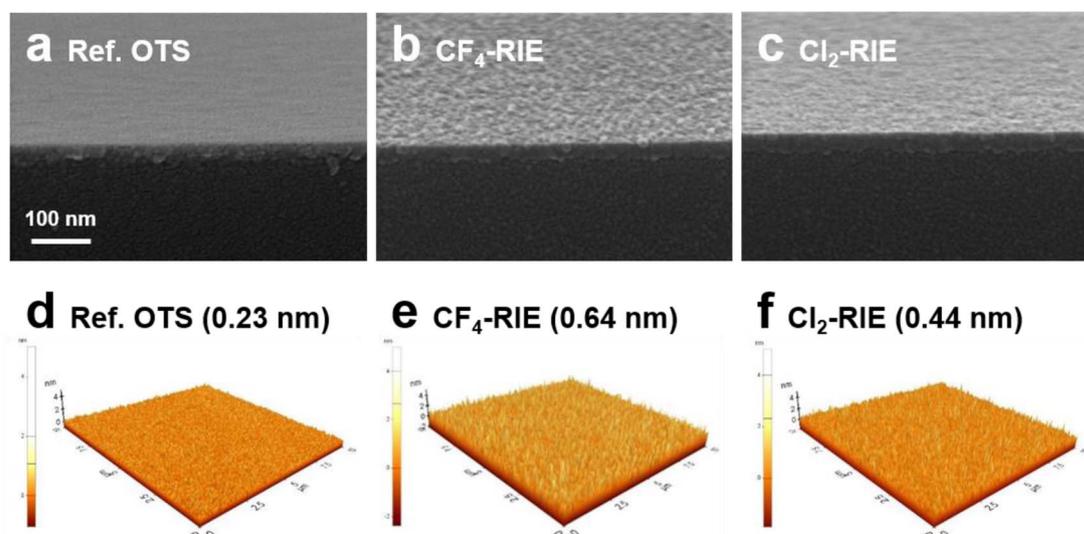
**Figure 1.** (a) SEM cross-sectional images of the multilayer thin film including OTS material stacked as C/OTS/C for oxidation prevention on SiO<sub>2</sub>/Si substrate. (b) Etch depth as a function of etch time and etch rate of OTS by ICP-RIE for Cl<sub>2</sub> and CF<sub>4</sub>.

fluorides are lower compared to the As, Te, and Ge chlorides, therefore, higher etch rates might be expected with F-based gases compared to Cl-based gases. However, as shown in Figure 1b, a lower etch rate of OTS was observed with CF<sub>4</sub> compared with Cl<sub>2</sub> possibly due to the formation of a C-F polymer on the OTS surface while no such polymer is formed with Cl<sub>2</sub>.<sup>23,27</sup> A very slow etch rate observed initially for the etching with CF<sub>4</sub> is also related to the slower etching of the 1 nm-thick carbon layer on OTS.

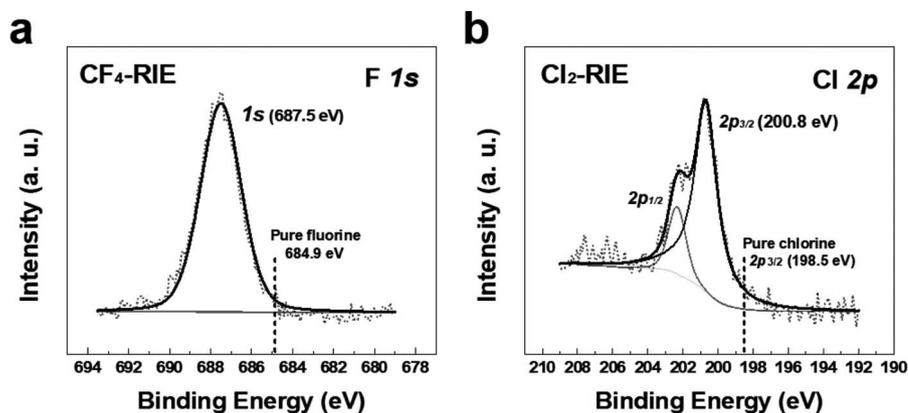
Figures 2a, 2b, and 2c show the tilted SEM images of the OTS surfaces for as-received and after the etching of ~20 nm thick OTS layer (including 1 nm thick carbon layer) using CF<sub>4</sub> and Cl<sub>2</sub>, respectively, with the same etching conditions in Figure 1. As shown in the SEM images, the OTS surface etched using CF<sub>4</sub> exhibited more rough surface compared that etched using Cl<sub>2</sub>. The surface roughness of the OTS surfaces was also investigated using AFM, and Figures 2d, 2e, and 2f are AFM data for as-received, CF<sub>4</sub>-etched, and Cl<sub>2</sub>-etched OTS surfaces, respectively, and, similar to the SEM images, the highest surface roughness of about 0.64 nm was observed for CF<sub>4</sub>-etched OTS surface compared to 0.23 nm for as-received OTS surface and 0.44 nm for the Cl<sub>2</sub>-etched OTS surface. The highest surface roughness for the CF<sub>4</sub>-etched OTS is believed to be related to a micro masking effect by the low etch rate of top carbon layer on OTS.

To investigate the possible chemical damage on the etched OTS surface, the OTS surfaces etched by CF<sub>4</sub> and Cl<sub>2</sub> shown in Figure 2 were investigated by XPS. Figures 3a and 3b show F 1s peak and Cl 2p peak of OTS surfaces etched using CF<sub>4</sub> and Cl<sub>2</sub>, respectively. As shown in Figure 3a, on the etched OTS surfaces, F 1s peak intensity was observed at 687.5 eV and Cl 2p<sub>3/2</sub> peak was observed at 200.8 eV while the pure F 1s peak and pure Cl 2p<sub>3/2</sub> peak are generally observed at 684.9 eV and 198.5 eV, respectively. Therefore, F 1s peak and Cl 2p<sub>3/2</sub> peak on the OTS surfaces were blue shifted ~2.6 eV and ~2.3 eV, respectively, possibly by forming halides with OTS. Using Raman spectroscopy, the OTS etched by CF<sub>4</sub> and Cl<sub>2</sub> including as-received OTS were also investigated and the results are shown in Figure 3. The OTS samples were etched about 10 nm and also were fully etched to expose SiO<sub>2</sub>/Si surface. As shown in Figure 4, the as-received OTS (reference) showed the Raman peaks at 169 cm<sup>-1</sup> and 194 cm<sup>-1</sup> but, after the etching using both CF<sub>4</sub> and Cl<sub>2</sub>, the peak at 169 cm<sup>-1</sup> was slightly red shifted to 166 cm<sup>-1</sup> possibly indicating the partial halogenation of OTS but not significantly (Also, when the C/OTS/C was fully etched, only the peak related to SiO<sub>2</sub>/Si substrate was observed as shown in Supplementary information Fig. S1).

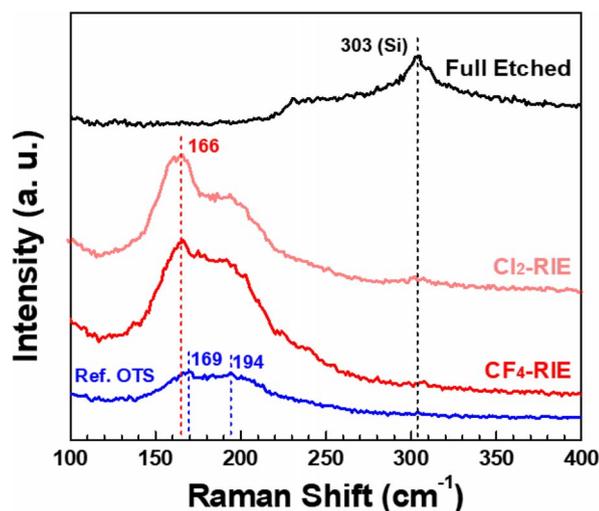
Using XPS depth profiling, the depths of halogenation between OTS etched using CF<sub>4</sub> and Cl<sub>2</sub> were compared. Figure 5 shows the



**Figure 2.** SEM images of the OTS material surface for (a) ref. OTS, (b) CF<sub>4</sub>-etched OTS, (c) Cl<sub>2</sub>-etched OTS, and AFM images of the OTS material surface for (d) ref. OTS, (e) CF<sub>4</sub>-etched OTS, (f) Cl<sub>2</sub>-etched OTS.

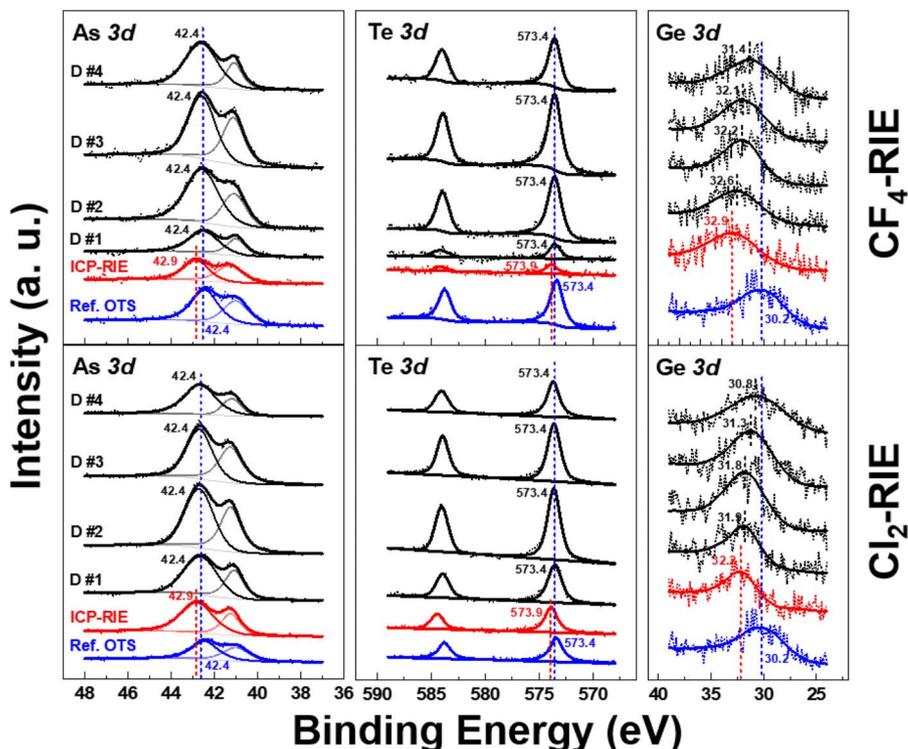


**Figure 3.** XPS narrow scan spectra of  $F 1s$  and  $Cl 2p$  peak intensities for the OTS material surface etched using  $CF_4$  and  $Cl_2$ .

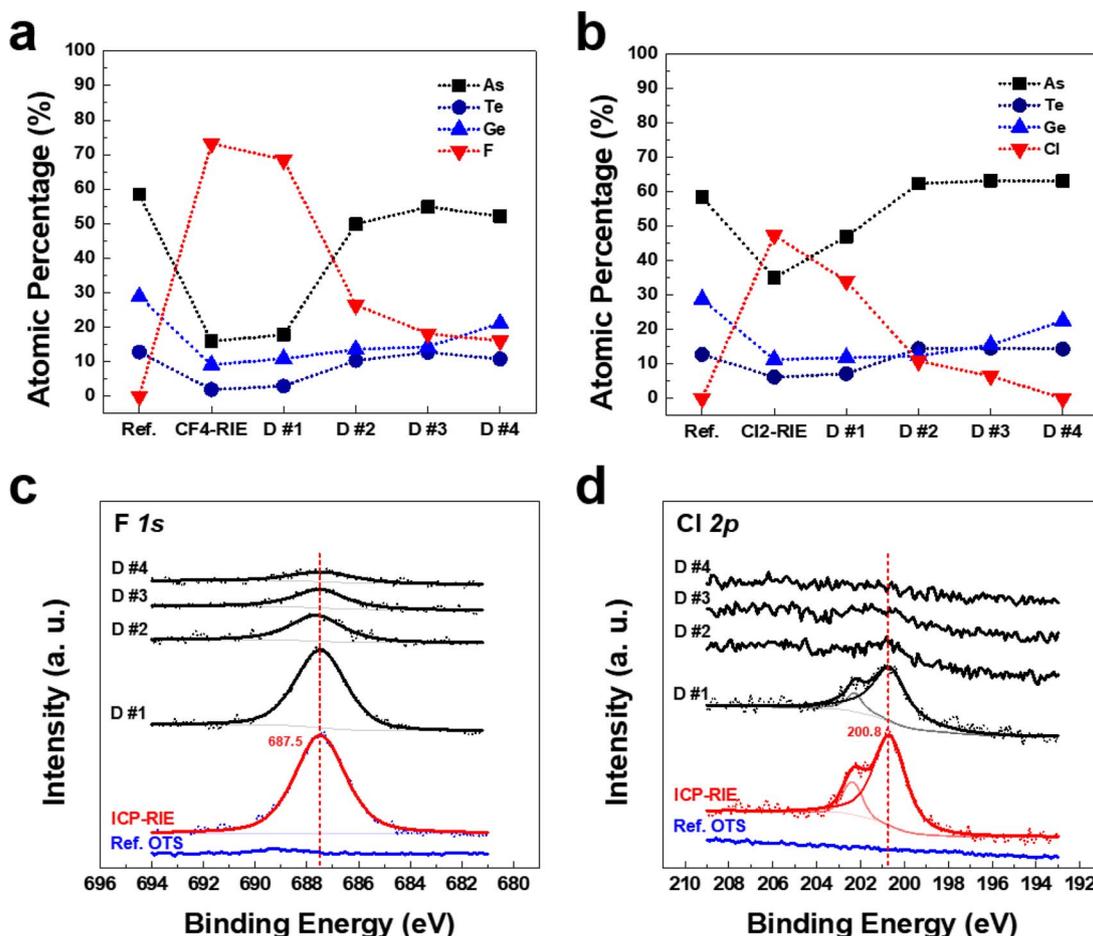


**Figure 4.** Raman spectra of as-received OTS and the OTS material surface etched using  $CF_4$  and  $Cl_2$ .

XPS narrow scan data of  $As 3d$ ,  $Te 3d$ , and  $Ge 3d$  measured as a function of depth profiling time with  $Ar^+$  ions (D #1 = 5 s, D #2 = 5 min, D #3 = 10 min, D #4 = 20 min). As shown in Figure 5, for the as-received OTS, the peaks of  $As 3d$ ,  $Te 3d$ , and  $Ge 3d$  were observed at 42.4, 573.4, and 30.2 eV, respectively. However, the peaks of  $As 3d$ ,  $Te 3d$ , and  $Ge 3d$  were shifted to higher energies of 42.9, 573.9, and 32.2 eV for  $CF_4$ -etched OTS and to 42.9, 573.9, and 32.2 eV for  $Cl_2$ -etched OTS indicating the halogenation of OTS during the etching using  $CF_4$  and  $Cl_2$ . As the etched OTS surface is depth profiled, the peak positions of  $As 3d$ ,  $Te 3d$ , and  $Ge 3d$  were moved toward the original peak positions of as-received OTS. Especially, for  $As 3d$  and  $Te 3d$ , from the depth profiling of 5 min (D #2), the peak positions were returned to original position indicating no deep halogenation of As and Te in OTS. However, in the case of Ge, the  $3d$  peak position was continuously moved toward the original position until the depth profiling for 20 min (D #4) for both  $CF_4$ -etched OTS and  $Cl_2$ -etched OTS, indicating significant halogenation of Ge by the etching. (During the experiment, we always observed more oxidation of Ge compared to As and Te in the OTS film when the OTS film was exposed to air. Even though more investigation is needed, we believe that more halogenation and/or oxidation of Ge compared to As and Te in the OTS film is related to more anodic property of Ge compared to As and



**Figure 5.** XPS narrow scan spectra of  $As 3d$ ,  $Te 3d$ , and  $Ge 3d$  in the OTS material surface etched using  $CF_4$  and  $Cl_2$  as a function of the  $Ar^+$  ion depth profiling time. For the ICP-RIEs, OTS was etched with 13.56 MHz 100 W of ICP power, 13.56 MHz 50 W of bias power, and 3.0 mTorr of  $Cl_2$  (100 sccm) or  $CF_4$  (100 sccm). After the etching the similar thickness using the conditions above, the depth profiling was performed on  $\sim 15$  nm thick OTS remaining after the etching.



**Figure 6.** Surface composition of the OTS material surface after etching using (a) CF<sub>4</sub> and (b) Cl<sub>2</sub> investigated using XPS as a function of the sputter time during the Ar<sup>+</sup> ion depth profiling. (c) and (d) are XPS narrow scan spectra of F 1s and Cl 2p in the OTS material surface etched using CF<sub>4</sub> and Cl<sub>2</sub>, respectively, as a function of the Ar<sup>+</sup> ion depth profiling time. The ICP-RIE conditions are the same as those in Figure 5.

Te). Therefore, it is found that, by the etching of OST using CF<sub>4</sub> and Cl<sub>2</sub>, mostly Ge is halogenated while As and Te are remained without significant halogenation.

The change of OTS surface composition was also investigated during the XPS depth profiling and the results are shown in Figures 6a and 6b for the OTS etched by CF<sub>4</sub> and Cl<sub>2</sub>, respectively. Before the etching, the as-received OTS showed the composition ratio of As: Te: Ge as 58.43: 12.75: 28.82 (shown in Table II) after the removal of 1 nm thick carbon layer on the OST surface (At the as-received OST surface before the removal of surface carbon layer, mostly carbon was observed due to carbon layer on the top of OST layer and As-rich OST surface was observed. As the surface carbon layer is removed, the ratio of As: Te: Ge was remained similar at ~58:~13:~29 during the depth profiling up to 20 min (D #4) as shown in supplementary information Fig. S2 and S3. During the depth profiling, in addition to OST materials, oxygen, silicon, and carbon were continuously observed due to the SiO<sub>2</sub>/Si substrate in addition to carbon layer under the OTS layer as shown in Fig. S2). After the etching of 10 nm thick

OTS (including 1 nm thick carbon layer), due to the halogenation of OST, the composition ratio has changed to F: As: Te: Ge = 73.2: 15.9: 1.9: 9.0% for CF<sub>4</sub>-etched OST and to Cl: As: Te: Ge = 47.4: 35.1: 6.2: 11.3% for Cl<sub>2</sub>-etched OST. The 73.2% of F for the CF<sub>4</sub>-etched OST is believed to be related to a thin C-F polymer layer formed on the etched OST surface in addition to the penetrated F into OST film. Therefore, more halogenation of OST surface was observed for CF<sub>4</sub>-etched OST compared to Cl<sub>2</sub>-etched OST surface as shown in Table II and Figures 6a and 6b. As the depth profiling time is increased, the atomic percentage of halogen component such as F and Cl was decreased and the percentage of OST components such as As, Te, and Ge were increased and recovered to original composition ratio of OST. Similar to Figure 5, atomic percentages of As and Te were recovered to original percentage from the depth profiling for 5 min (D #2) while the Ge atomic percentage was continuously increasing and halogen atomic percentage was decreasing until 20 min depth profiling (D #4). At the 20 min depth profiling, Cl percentage of Cl<sub>2</sub>-etched OST was decreased to ~ zero percentage while F percentage of CF<sub>4</sub>-etched OST was remaining at 16%. The XPS narrow scan data of halogen atoms of F 1s and Cl 2p for the etched OST during the depth profiling are shown in Figures 6c and 6d, respectively, and, after the 20 min depth profiling (D #4), no Cl 2p peak intensity could be observed while F 1s peak intensity was still observed. Therefore, it can be found that CF<sub>4</sub>-etched OST is more chemically damaged compared to Cl<sub>2</sub>-etched OST. (Other components such as carbon, silicon, and oxygen were also observed during the XPS depth profiling as shown in supplementary information Fig. S4. However, these carbon, silicon, and oxygen components were observed because they were located under the OST layer as carbon (10 nm)/SiO<sub>2</sub>/Si wafer).

**Table II.** Atomic percent of OTS and halogen components on the blanket OTS surface etched with CF<sub>4</sub> and Cl<sub>2</sub> (XPS data of ref. OTS was taken after XPS depth profiling with Ar<sup>+</sup> ions for 5 s).

	Ref. OTS	CF <sub>4</sub>	Cl <sub>2</sub>
Halogen	—	73.2	47.4
As	58.4	15.9	35.1
Te	12.8	1.9	6.2
Ge	28.8	9.0	11.3

## Conclusions

In this study, the multilayer thin film including OTS material has been etched using the halogen gases such as CF<sub>4</sub> and Cl<sub>2</sub> in an ICP-RIE system and the effect of halogen gases on the damages of the etched OTS surface was investigated. The OTS etched with Cl<sub>2</sub> showed a higher etch rate compared to that with CF<sub>4</sub> possibly due to the formation of a C-F layer on the OST surface for the etching with CF<sub>4</sub> even though the stable fluorides of OST components generally have higher vapor pressure compared to chlorides of OST components. SEM and AFM analysis showed that the physical damage such as the surface roughness was lower for Cl<sub>2</sub> etched OST than CF<sub>4</sub> etched OST. Also, the halogen atom was more deeply penetrated into OST for CF<sub>4</sub>-etched OST compared to Cl<sub>2</sub>-etched OST. Therefore, compared to CF<sub>4</sub>-etched OST, Cl<sub>2</sub>-etched OST was less damaged during the etching. Among the OST components of As, Te, and Ge, As and Te were halogenated only at the surface of OST while Ge were halogenated by Cl and F more deeply indicating that Ge is mostly chemically damaged by the etching using Cl<sub>2</sub> and CF<sub>4</sub>.

## Acknowledgments

This work is supported by the Samsung Electronics' university R&D program. [Development of post-etch passivation for prevent the oxidation in PRAM patterning].

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