Extended Abstract of PSA-19

O-14

Quantitative Analysis on Diffused Trace Elements by Atom Probe Tomography

Bong Ho Lee,* Taehoon Cheon, Youngmoo Eun, Ki Hee Kim, Jin Bae Bang, Byung Mok Sung and Sang-il Kim
Center for Core Research Facilities, DGIST, Daegu 42988, Republic of Korea
*corresponding author's e-mail: bhlee@dgist.ac.kr

(Received: May 30, 2019; Accepted: June 12, 2019)

Elemental analyses for detecting lowly concentrated elements at small volume such as interfaces and surface are very difficult due to the limitations of detection and analytical spot size. Through the analysis by atom probe tomography, we were able to confirm the distribution and concentration of the trace elements diffused into the passive layer of stainless steel and the thin film on SiC complementarily with x-ray photoelectron spectrometer, secondary ion mass spectrometer, and transmission electron microscopy.

1. Introduction

On the nature of the nanomaterials, the region of interest to be analyzed is mostly a very small volume and the concentration of elements is very low. Therefore, conventional analysis techniques have a low signal to noise ratio and thus have limitations in detection or visualization of trace elements. Recently, the development of analytical techniques makes it possible to acquire unknown information at the small volume such as surface and interfaces.

Meanwhile, the performance of the analytical technique depends on the analytical spot size and detection limit. Advanced structural analysis technology generally has an ability to identify atoms or molecules such as in scanning probe microscopy (SPM) and transmission electron microscopy (TEM). However, in the case of component analysis, there are still many limitations in terms of the size and sensitivity of analysis ability for detecting trace elements in small volumes. Even in secondary ion mass spectrometer (SIMS), which has an analytical sensitivity as high as ppb (parts per billion), there is a still limitation in controlling the analysis beam size down to 100 nm or less. As a result, the development of nano-analysis is advancing toward improving the two conditions (analytical spot size and detection limit), and in this respect atom probe tomography (APT) must be one of the most advanced technologies[1].

APT applies high voltage field and concentrates pico-second laser pulses on the specimens to cause field evaporation of the constituent atoms at the tip of the specimen. The time of flight (ToF) from the surface of a specimen to the position sensitive detector (PSD) and the collision position on PSD are measured, simultaneously to define the nature and the position of the accelerated ions. Especially, since the analytical beam size is the individual atom itself, the spatial resolution is about 0.2 nm - 0.5 nm and the efficiency of atom detection can be up to 60%. Consequently, the analysis sensitivity is ppm level even at the nano-volume.

In this study, we report the results of quantitative analyses on the diffused trace elements for two cases. One is for the surface analysis on several nanometer-sized passive layers on a stainless steel which contains some trace elements diffused from the matrix even at the room temperature, and the other is for the analysis of the diffused constituent elements to the oxide or silicide on the top of substrate after heat treatment in the system of silicon carbide (SiC) semiconductors.

2. Results and Discussion

2.1 Passive Film Analysis

In stainless steels, anti-corrosion or oxidation-

resistance also can be provided by thin passive films, which are spontaneously formed on the surface and primarily composed of chromium oxide. In the TEM analysis with energy dispersive x-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS), the passive films were composed of chromium oxide with iron oxide and some carbon and silicon supposed to be segregated at the surface layer. XPS analysis revealed that chromium oxide was Cr_2O_3 and carbon was graphitic carbon, but could not detect the segregated Si and other trace element. Segregated Cr, Si, C and a trace element were detected in SIMS analysis due to its good sensitivity. Through APT analysis, not only all of the constituent elements of the passive layer could be confirmed in three dimensions,

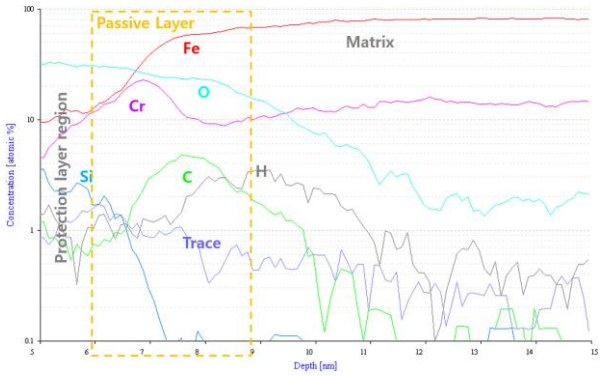

Fig. 1 APT analysis of concentration profile for a passive layer.

but the order of the layers in a few nm could be confirmed. This is the first result that a passive layer has been identified through APT analysis for a very thin film of several nm in my knowledge[2].

2.2 SiC Analysis

SiC has been widely studied since it has high applicability to power electronic devices because SiC has excellent wide band gap, high temperature conductivity, and high threshold voltage compared to Si. In addition, the SiC process for semiconductors has compatibility with the current silicon industry since the fabrication process for circuits is similar to the Si.

In SiC for using a device substrate, dopant and impurity control is important to the electrical characteristics. It is also necessary to understand the migration of trace element as well as constituent element during the heat treatment process[3].

In this study, we visualized and quantified the elemental diffusion of dopants and impurities as well as the constituent elements during oxidation and silicidation. When the silicide was formed, carbon separated from SiC diffused into the defect trapping sites, such as defects, interfaces and surface, to form graphitic layers.

3. Conclusions

Since APT analysis has high sensitivity (ppm level) and spatial resolution (sub nm) as an elemental analysis tool, it is suitable to take three-dimensional elemental information even at the small volume region and for low density trace elements. We investigated the compositional segregation on the passive film of a stainless steel and the diffusion of elements in SiC semiconductors, followed by confirmation of the quantitative concentration of the elements. APT is a good complementary method that can be employed for the surface and interface analysis to improve the reliability of results through cross-validation with various analytical methods.

4. References

[1] David N. Seidman, *Annu. Rev. Mater. Res.* **37**, 127 (2007).
[2] A.H. Heuer et al., *Acta Materialia* **60**, 716 (2012).
[3] M. H. Maneshian et al., *Applied Physics Letters*, **97**, 024103 (2010).