Annealing dependence of deposit morphology for Fe-Ga contained DLC film formed by FIB-CVD with ferrocene source gas

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Focused-ion-beam chemical vapor deposition (FIB-CVD) has a great potential to fabricate the arbitrary three dimensional (3D) nano- and micro-devices. The deposited material using phenanthrene ($C_{14}H_{10}$) source gas is diamond-like-carbon (DLC) and contained Ga due to Ga-ion-beam irradiation. It is noted that the DLC structure change and the Ga segregation with annealing treatment. And it is reported that the Young's modulus decrease and electronic conductivity increased with annealing treatment[1]. Therefore, it is very important to evaluate the annealing effect of structures fabricated by FIB-CVD. In this study, we formed Fe-Ga contained DLC film by FIB-CVD with the ferrocene ($C_{10}H_{10}Fe$) as a source gas and evaluated the annealing effect.

We evaluated the annealing effects of Fe-Ga contained DLC film by comparison with Ga contained DLC film formed by FIB-CVD with phenanthrene source gas. The Fe-Ga contained DLC film with 60 nm thickness and Ga contained DLC film with 120 nm thickness were deposited by Ga ion-beam with 30 keV at 20 nA. The films were annealed at a vacuum pressure of 1×10^{-3} Pa for 32 hours.

Figure 1 shows the scanning electron microscope (SEM) images and surface state obtained by scanning probe microscope (SPM) of Ga contained DLC films with and without annealing. The Ga droplets on the Ga contained DLC film with annealing at 400 ˚C were observed and evaporated by annealing at 600 ˚C.

The droplets, in the case of Fe-Ga contained DLC film, were observed at annealing of 400 ˚C and the holes on the Fe-Ga contained DLC films after annealing at 600 °C and 800 ˚C were observed, as shown in Fig. 2. Next, to evaluate the chemical state of Fe-Ga contained DLC films without and with annealing, we carried out X-ray photoelectron spectroscopy (XPS) and SEM energy-dispersive X-ray (SEM-EDX). Figures 3(a) and (b) show XPS measurement results of Ga and Fe-Ga contained DLC films with and without annealing, respectively. The Ga 2p peaks were observed for both of XPS spectra of Ga and Fe-Ga contained DLC films with 400 ˚C. This result indicates that the droplets on Fe-Ga contained DLC film with annealing at 400 ˚C are Ga droplets. To investigate the distribution of Fe on Fe-Ga doped DLC films after annealing at 600 ˚C and 800 °C, we carried out SEM-EDX. Figures 4(a) and (b) show the SEM images and EDX results of Fe-Ga contained DLC film with annealing at 600 °C and 800 °C. The deposit morphologies shown in SEM images correspond to approximately those of Fe distributions, as shown in Figs. 4(a) and (b).

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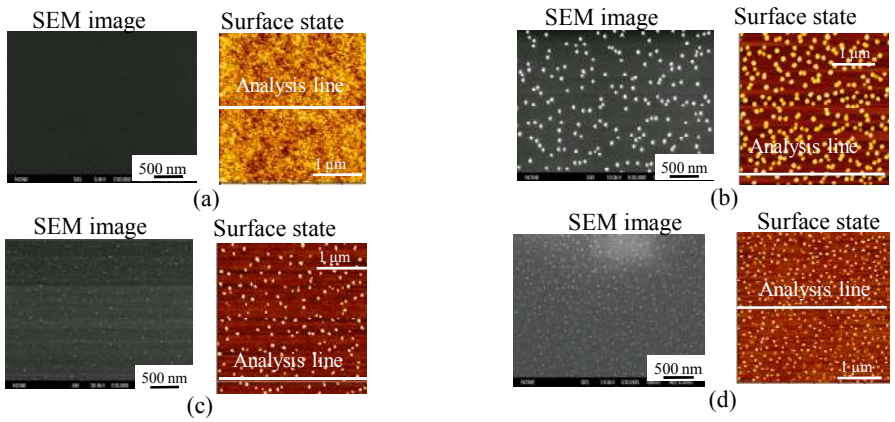

Fig. 1. SEM images and surface states obtained SPM of Ga contained DLC
(a) without and with annealing at (b) 400 ˚C, (c) 600 ˚C and (d) 800 ˚C.

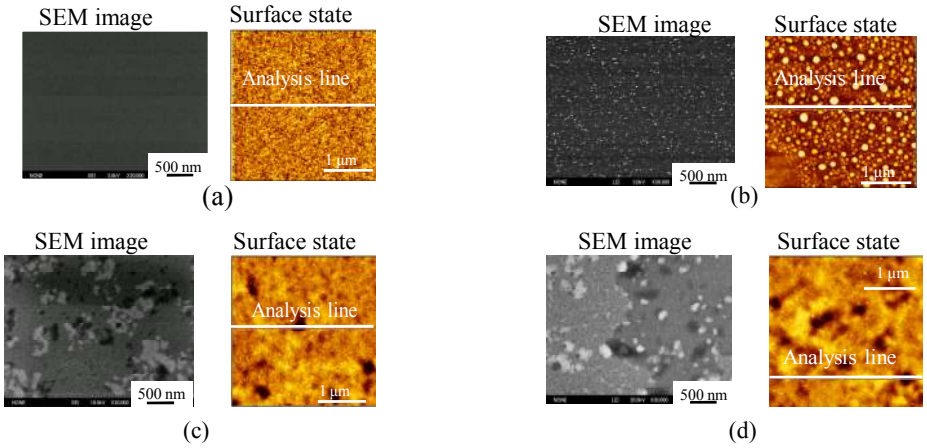

Fig. 2. SEM images and surface state obtained SPM of Fe-Ga contained DLC
(a) without and with annealing at (b) 400 ˚C, (c) 600 ˚C and (d) 800 ˚C.

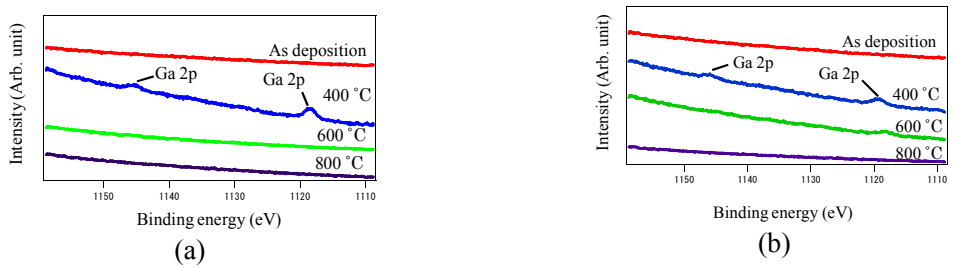

Fig. 3. XPS spectra of (a) Ga contained DLC and (b) Fe-Ga contained DLC without and with annealing.

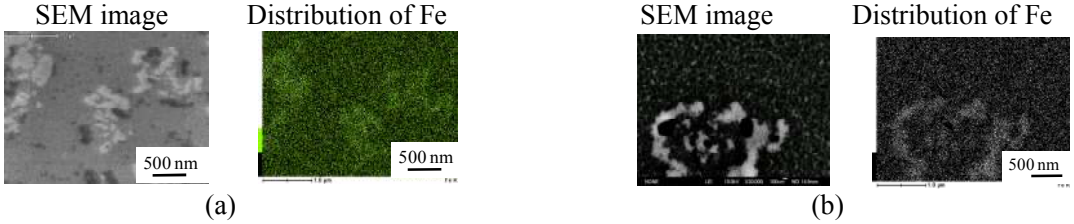

Fig. 4. SEM images and distributions of Fe obtained by EDX for Fe-Ga contained DLC with annealing at (a) 600 ˚C and (b) 800 ˚C.