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Low-Temperature MoS_2 Film Formation Using Sputtering and H_2S Annealing

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ABSTRACT Low-carrier density and high-crystallinity molybdenum disulfide (MoS_2) films were fabricated by low-temperature and clean process based on a UHV RF sputtering system. This paper focuses on improving crystallinity and reducing the number of sulfur defects of sputtered-MoS_2 film. We have fabricated MoS_2 films at lower than 400°C using the sputtering and H_2S post-deposition annealing processes. Consequently, MoS_2 films with high crystallinity and appropriate S/Mo ratio were obtained. Eventually, a low carrier density of 3.5×10^{17} cm^{-3} and the Hall-effect mobility of 12 $cm^2V^{-1}s^{-1}$ were achieved.

INDEX TERMS Transition metal di-chalcogenide, TMDC, molybdenum disulfide (MoS_2), UHV RF sputtering, H_2S annealing.

I. INTRODUCTION

Molybdenum di-sulfide (MoS_2) has attracted considerable attention because of its remarkable electrical and mechanical properties. Examples of such properties include a bandgap of 1.2–1.8 eV [1], [2] and high mobility up to 400 $cm^2V^{-1}s^{-1}$ even in atomically thin film [3]–[5] in which the mobility seriously decreases in the case of bulk materials such as silicon and InGaAs [6]–[8].

Transfer-type film formation processes such as Scotch-tape and liquid exfoliation are widely adopted [3]–[5], [9]. The exfoliation has an advantage in which it easily enables the formation of a thin MoS_2 film and achieves high mobility in the MoS_2 film up to 400 $cm^2V^{-1}s^{-1}$. However, the exfoliated-MoS_2 film has a high carrier density of 10^{20} cm^{-3} caused by pollutions such as carbon and alkali metals, and also a difficulty in forming the large-area MoS_2 film, which is necessary for industrial applications.

To overcome these problems, bottom-up processes such as chemical vapor deposition (CVD), atomic layer deposition (ALD), pulse laser deposition (PLD) and sputtering are required. The CVD, in which MoO_3, sulfur powder, and hydrogen sulfide (H_2S) are commonly used as precursors, is widely used because it allows us the synthesis of high-quality MoS_2 film on a sapphire substrate [10]–[16]. Although large-grain and high-quality MoS_2 film can be obtained, the deposition temperature is commonly high at 600°C or more which is not appropriate for application such as 3D-monolithic ICs which require low thermal budget [17], [18]. Thus, reducing the formation temperature is important for maintaining high crystallinity of the channel material.

The ALD has been investigated because of the possibilities such as low-temperature process, large-scale deposition and high controllability of film thickness. It has been reported that MoS_2 films were able to be formed by using $Mo(CO)_6$ [19]–[21], $MoCl_5$ [22]–[25] or $Mo(thd)_3$ [26] with H_2S gas. However, these processes are simultaneously associated with some disadvantages such as the high temperature of post-deposition annealing, carbon contamination, and low-crystallinity of MoS_2 film.

On the other hand, a sputtering process is considered to allow us a low-temperature, carbon-free, and large-scale film formation. Recently, the reactive sputtering with a molybdenum (Mo) target and sulfur (S) powder was used to form MoS_2 thin film as a semiconductor, although it is a high-temperature process at up to 700°C [27]. Thus, an alternative sputtering method, which can be used to form MoS_2 films at low temperature, is required. Instead of the Mo target, MoS_2 target is selected to reduce temperature on large area with less impurities [28]–[36]. However, it was found that the carrier density of sputtered-MoS_2 film is comparatively high as 10^{18} cm^{-3} approximately due to sulfur defects which have been discussed as n-type dopants in the case of the MoS_2 film because sulfur defects generate states with an energy level of 0.2 eV near the conduction band minimum in monolayer MoS_2 film [37]. The film prepared by sulfur compensation process using sulfur powder annealing has high crystallinity and low carrier density of 10^{16} cm^{-3}, approximately [32]. However, the annealing temperature up to 700°C is comparatively higher than we had expected. In order to reduce annealing temperature, we used H_2S gas because of its high reactivity.

In this study, we conducted low-temperature H_2S annealing on sputtered-MoS_2 film to achieve high crystallinity MoS_2 film resulting in the low carrier density.

II. EXPERIMENTAL METHODS

A SiO_2/Si substrate of 2 cm × 2 cm was cleaned by a wet process using piranha solution. Then, a MoS_2 film of 5-nm thickness was deposited on the substrate by RF sputtering system with four-inch-diameter sample stage. The conditions were: RF power of 50 W; distance between the target and substrate of 150 mm; substrate temperature at 400°C; argon (Ar) flow of 7.0 sccm; and partial pressure under 0.55 Pa. Then, the MoS_2 film was annealed in Ar gas with 1% H_2S gas instead of forming gas (F.G.: 3% H_2 in N_2) under 10k-300 Pa at up to 400°C using hot-wall annealing system as an *ex-situ* process. Raman spectroscopy with a laser wavelength of 488 nm, X-ray photoelectron spectroscopy (XPS) using an Al Kα X-ray source and scanning transmission electron microscopy (STEM) were performed. As electrical characteristics, the Hall effect was measured in centimeter-level MoS_2 film with silver past by using ResiTest8400 of TOYO Corporation.

III. RESULTS AND DISCUSSION

Figs. 1 and 2 show the Raman spectra for as-sputtered and annealed MoS_2 films depending on the gas/pressure and annealing temperature, respectively. In these figures, H_2S annealing reduces the intensity of MoO_3 peak over 300°C as compared with as-sputtered and F.G.-annealed MoS_2 films. Although it has been reported that sulfur vacancies cause tensile strain in MoS_2 film formed by exfoliation process [39], E^1_{2g} peaks with H_2S annealing process shift to higher wavenumber direction because of the tensile stress release as our speculation. Fig. 3 summarizes the values of full width half maximum (FWHM) for Raman spectra in the E^1_{2g} and A_{1g} mode peaks. The FWHM values decrease

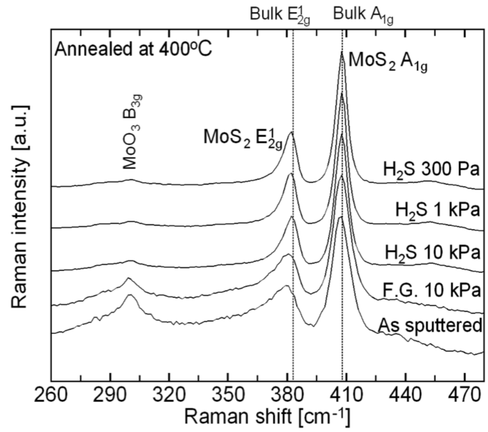

FIGURE 1. Raman spectra for as-sputtered and annealed MoS_2 films depending on annealing gas and pressure.

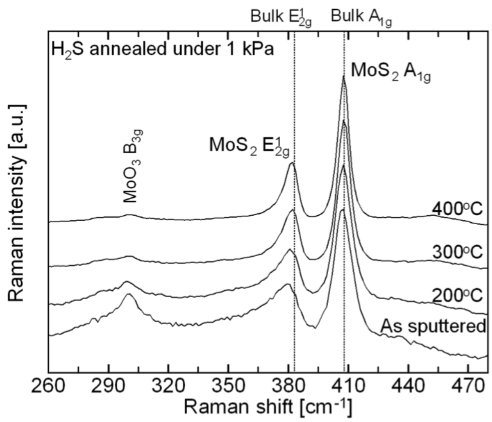

FIGURE 2. Raman spectra for as-sputtered and H_2S-annealed MoS_2 films depending on annealing temperature.

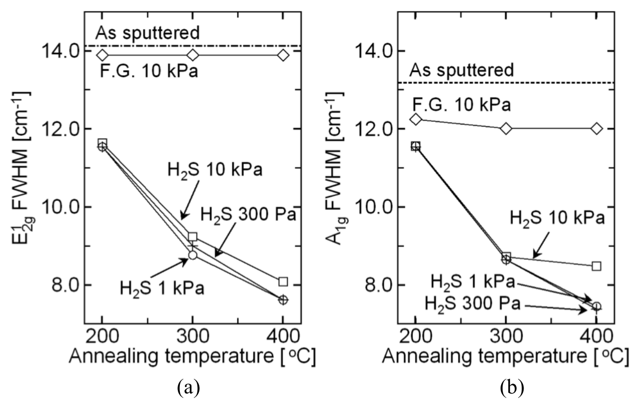

FIGURE 3. FWHM values for sputtered-MoS_2 Raman spectra in (a) E^1_{2g} and (b) A_{1g} mode peaks.

with an increase in the annealing temperature. Reducing the annealing pressure is effective to reduce the FWHM values except 10 kPa. As a result, the FWHM values of E^1_{2g} and A_{1g} remarkably decrease from 14.1 to 7.6 cm^{-1} and from 13.2 to 7.4 cm^{-1} when optimal H_2S annealing is used. It

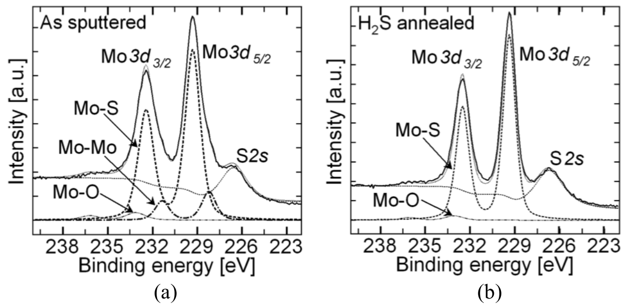

FIGURE 4. **XPS spectra of molybdenum *3d* for (a) as-sputtered and (b) H_2S-annealed MoS_2 films at 400°C under 1 kPa.**

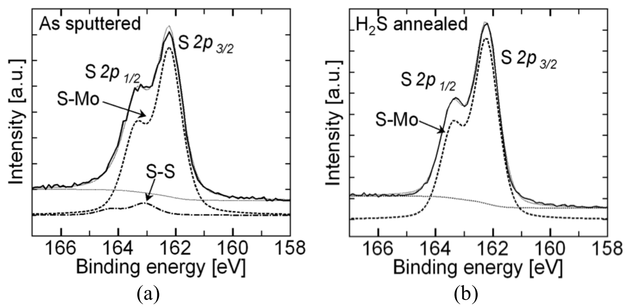

FIGURE 5. **XPS spectra of sulfur *2p* for (a) as-sputtered and (b) H_2S-annealed MoS_2 films at 400°C under 1 kPa.**

is speculated that all of these results are obtained because of the successful compensation of sulfur into sulfur defects using the H_2S annealing in sputtered-MoS_2 film.

Figs. 4(a) and (b) show the XPS spectra of Mo *3d* for the as-sputtered and H_2S-annealed MoS_2 films. To analyze the Mo *3d* components, the peaks were fitted with a pseudo-Voigt function. Although an as-sputtered MoS_2 film has the Mo–S, Mo–Mo, and Mo–O components [29]–[31], a Mo–Mo component is not observed in the H_2S-annealed MoS_2 film indicating successful sulfurization of residual molybdenum by H_2S annealing. However, the MoS_2 film still has a slight Mo–O component even after H_2S annealing. On the other hand, Figs. 5(a) and (b) show the XPS spectra of S *2p*. The S–S component is observed within the MoS_2 film after the sputtering process. In contrast, the S–S peak is not observed after H_2S annealing. This agrees with a previous report in which residual sulfur in the as-sputtered MoS_2 film reacted with sulfur defects when F.G. annealing is carried out at 400°C [36]. These results in this paragraph agree with the results of Raman spectra as shown in above.

The S/Mo ratio was also investigated to estimate the state of chemical bonds before and after the H_2S annealing process. We calculated the area of each peak of the Mo–S, Mo–Mo, and S–Mo components. The results showed that the S/Mo ratio improved from 1.47 to 1.90 by H_2S annealing.

A cross-sectional high-angle-annular dark field (HAADF) STEM image in Fig. 6 shows overall features of the H_2S-annealed MoS_2 film at 400°C under 300 Pa. It is confirmed that a two-dimensionally layered MoS_2 film is successfully

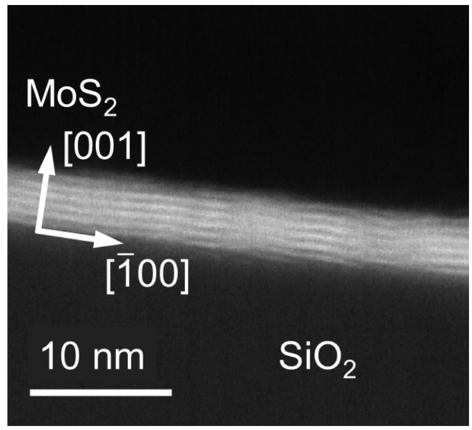

FIGURE 6. **Cross-sectional STEM image for MoS_2 film annealed in H_2S gas at 400°C under 300 Pa.**

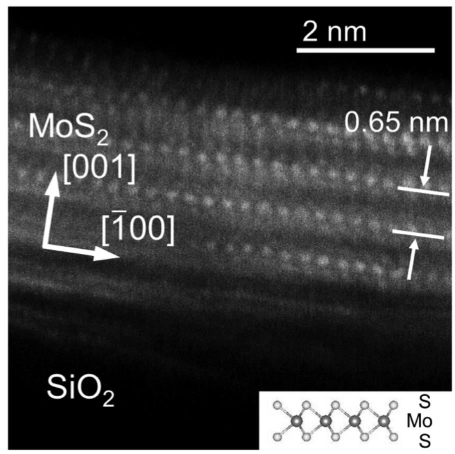

FIGURE 7. **High-resolution image from [010] direction of H_2S-annealed MoS_2 film at 400°C under 300 Pa and schematic image of MoS_2.**

formed with uniform thickness. Here, the significant thickness change between before and after H_2S anneal process was not observed yet, however it is needed to confirm carefully. Moreover, a grain size is approximately 10 nm, which is smaller than what we had expected. Fig. 7 shows a high-resolution HAADF-STEM image of the same sample. It is successfully observed a triangle shape of the Mo-S atomic arrangement within a 5-layer MoS_2 film having an interlayer distance of 0.65 nm. Therefore we considered that an appropriate MoS_2 film was obtained using the H_2S annealing.

To determine the electrical characteristics, the carrier densities of the MoS_2 film are firstly shown in Fig. 8. The carrier density in n-type decreases with an increase in the annealing temperature. It is considered that this is because of the compensation of sulfur defects. A carrier density of 9.3×10^{15} cm^{-3} is achieved even at 400°C, which is significantly lower compared to reported value with 10^{20} cm^{-3} for an exfoliated-MoS_2 film [40], [41]. As shown in Fig. 9, the Hall-effect mobility of electrons is enhanced by H_2S annealing at up to 300°C, even though

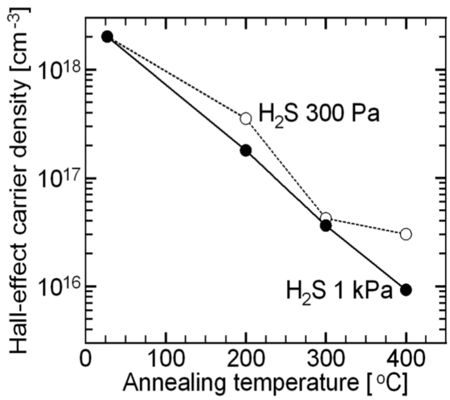

FIGURE 8. Carrier density by Hall-effect measurement depending on annealing temperature.

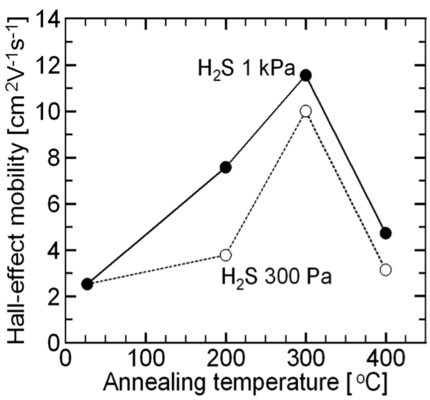

FIGURE 9. Hall-effect mobility depending on annealing temperature.

carrier density continuously decreases up to 400°C. Here, H_2S gas is decomposed over 400°C; this might be one of the reasons that mobility decreases at 400°C. Eventually, the H_2S annealing remarkably achieves the Hall-effect mobility of up to 12 $cm^2V^{-1}s^{-1}$ and the carrier density of 3.5×10^{17} cm^{-3} at 300°C. This value of carrier density is still low enough to realize a normally-off accumulation-mode MOSFET with the MoS_2 channel.

IV. CONCLUSION

High-quality MoS_2 films were formed by sputtering at 360°C and by performing H_2S annealing at 300°C. The sulfur defects of the sputtered MoS_2 film were effectively compensated by H_2S annealing, and MoS_2 films with good crystallinity in Raman spectra and a higher S/Mo ratio of 1.90 were obtained. Eventually from the Hall-effect measurement, carrier density reduction down to 3.5×10^{17} cm^{-3} and mobility improvement up to 12 $cm^2V^{-1}s^{-1}$ were simultaneously achieved. These results show that sputtering and H_2S annealing is applicable to obtain high-quality MoS_2 film for 3D-monolithic IC applications.

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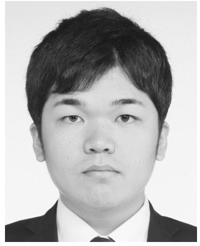

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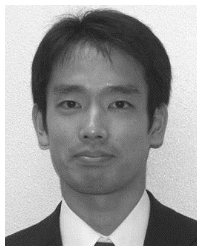

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