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Adsorption and decomposition of H_2S on the Ge(100) surface

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Abstract

The adsorption and decomposition of H_2S on the Ge(100) surface is investigated. H_2S is a simple sulfur containing molecule that eventually decomposes to yield hydrogen gas and deposits sulfur on the germanium surface. The surface reactions of H_2S are investigated by ultraviolet photoelectron spectroscopy, Auger electron spectroscopy, and temperature programmed desorption. Room temperature exposure of H_2S to Ge(100) results in dissociative adsorption which can be followed easily by ultraviolet photoelectron spectroscopy. Warming the H_2S exposed surface results in some molecular desorption and further decomposition of the adsorbed species. At saturation, 0.25 ML of H_2S decomposes generating 0.5 ML of atomic hydrogen. Above the hydrogen desorption temperature some etching of the germanium surface is observed by sulfur. The etch product, GeS, is subsequently observed in temperature programmed desorption experiments. Exposure of H_2S to the Ge surface at elevated temperatures leads to higher sulfur coverages. A sulfur coverage approaching 0.5 ML can be deposited at the higher exposure temperatures. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

Passivation and protective methods for semiconductor surfaces are important for a variety of processing steps and to improve the lifetime of electronic components. There are several studies examining the preparation of passivation layers on germanium surfaces [1–8]. In these methods, a passivation layer is formed on the surface by an ex situ treatment, followed by removal of the passivating layer in situ. Two different passivation layers have been reported in the literature. These methods leave the Ge(100) surface terminated with S by $(NH_4)_2S$ treatment [6] or with O by the growth of GeO_2 layers [1–5,7,8]. Growth of GeO_2 is the more common method because growth of GeO_2 is better understood.

Preparation of clean and smooth GeO_2 surfaces has been hampered by the fact that there is a poor lattice match between GeO_2 and Ge. This mismatch leads to an inhomogeneous oxide layer. The oxide layer is also thin and water soluble which permits contamination to reach the Ge/GeO_2 interface. Once at the Ge/GeO_2 interface, the contamination is difficult to remove. The contamination at the semiconductor/oxide interface can then degrade electronic properties. Because of these difficulties, alternatives

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to GeO_2 as a passivation and protection layer are desired. Sulfur has recently been considered as an alternative protection layer. A number of ways to deposit sulfur in vacuum onto a germanium surface have been investigated and these methods are discussed next.

The adsorption of sulfur on Ge(100) has been studied previously by several investigators [9–13]. In these studies the saturation coverage of sulfur obtained for the Ge(100)-(2 × 1) surface was 0.5 ML. The sulfur source used in these studies was a solid state electrochemical cell that generated a S_2 molecular beam. The sulfur adsorption site was proposed as a bridge site in which the sulfur atoms bind to two Ge atoms. Etching of the surface by sulfur was also observed, which facilitated the formation of Ge_2S surface layers.

The adsorption and reaction of H_2S on germanium is less studied. The adsorption of H_2S was probed by Cohen et al. [14] as a way of depositing hydrogen at the Ge(100) surface. The examination of the desorption kinetics of hydrogen in the presence of sulfur was monitored. Kuhr and Ranke [15] investigated the adsorption of H_2S on a cylindrical Ge sample by photoemission. They were able to show that the adsorption of H_2S on the Ge(111) part of the sample was molecular, while adsorption of H_2S on Ge(100) was dissociative. The presence of surface SH and S were identified by photoemission. In each of the studies the saturation coverage of H_2S at room temperature was determined to be 0.25 ML.

In this study, we report the investigation of the adsorption and decomposition of H_2S on the Ge(100) surface. H_2S decomposes at the Ge(100) surface to yield hydrogen and deposits sulfur on the germanium surface. The surface reactions of H_2S are investigated by ultraviolet photoelectron spectroscopy, Auger electron spectroscopy, and temperature programmed desorption. Adsorption of H_2S at both room temperature and elevated temperatures is investigated.

2. Experimental

The experiments are conducted in a stainless steel ultra-high vacuum chamber. The chamber is equipped with a double-pass cylindrical mirror analyzer (CMA) for Auger electron and photoelectron spectroscopies, a differentially pumped ultraviolet discharge lamp, twin X-ray source, ion gun, and quadrupole mass spectrometer for both temperature programmed desorption and secondary ion mass spectrometry. The base pressure of the system is 6×10^{-11} Torr with a typical working pressure of 1×10^{-10} Torr.

Samples are cleaved into 10 × 25 × 0.4 mm rectangles from n-type Ge(100) wafers (Eagle-Picher, ±0.5° of the (100) plane, Sb doped, 0.04–0.4 Ω cm resistivity). Before placing the sample into the chamber, the Ge(100) substrate is degreased by (a) ultrasonically rinsing in trichloroethylene, acetone, and methanol for 3 min each, (b) immediately blowing dry with dry nitrogen, and (c) ultrasonically rinsing in deionized water for 15 min to lower the contact resistance [5]. The sample is held by molybdenum clamps for resistive heating and mounted to a manipulator. The sample temperature is monitored by a pair of chromel–alumel thermocouples attached to the back of the sample with Aremco 516 ceramic adhesive. Surface cleanliness is followed by Auger electron (AES) and ultraviolet photoelectron (UPS) spectroscopies. A clean surface is generated by the removal of the native oxide by repeated heating to 850 K in vacuum, followed by ion sputtering (3 keV Ar^+, 1 μA), and annealing at 850 K. The number of defects induced by sputtering can be reduced [16] by exposing the Ge sample to an apparent pressure of 5×10^{-8} Torr Ge_2H_6 (Voltaix, ultrahigh purity grade, minimum purity 99.999%) for about 5 min while the surface temperature is maintained at 570 K. The Ge_2H_6 gas flow is then turned off and the sample is annealed at 850 K before cooling. After several cycles of Ar^+ sputtering, annealing, Ge_2H_6 exposure, and annealing, a clean Ge(100) surface was observed by electron spectroscopy.

Dihydrogen sulfide (Genex, CP grade, minimum purity 99.5%) is further purified by several freeze-pump-thaw degassing cycles prior to introduction to the UHV chamber and the purity of the gas is checked by in situ mass spectrometry. The gas is admitted to the chamber through an effusive doser and directed onto the front face of the crystal for various periods of time. The actual pressure at the sample is higher than the chamber pressure as measured by the ion gauge and since these pressures should be proportional to one another, the chamber

pressure was used as a measure of the overall sample exposure. The exposures (uncorrected for ion gauge sensitivity to H_2S) are reported in Langmuirs (1 L = 10^{-6} Torr s).

Temperature programmed desorption (TPD) experiments are conducted with a linear temperature ramp of 5 K s^{-1} with the crystal in line-of-sight of the quadrupole mass spectrometer. The coverage at saturation for hydrogen on Ge(100) at room temperature has been determined as 1 ML (1 ML = one adsorbate per surface Ge atom and is equal to 6.23×10^{14} atoms cm^{-2}) [14,17–20]. The TPD area from a saturation coverage of H atoms is then used as a internal standard for H_2 thermal desorption.

Ultraviolet photoemission spectra are taken with He I radiation (21.2 eV) from a He gas discharge lamp and the CMA is operated at a fixed pass energy of 20 eV. All the reported binding energies are referenced to the Ge valence band edge, which is assigned as 0 eV binding energy.

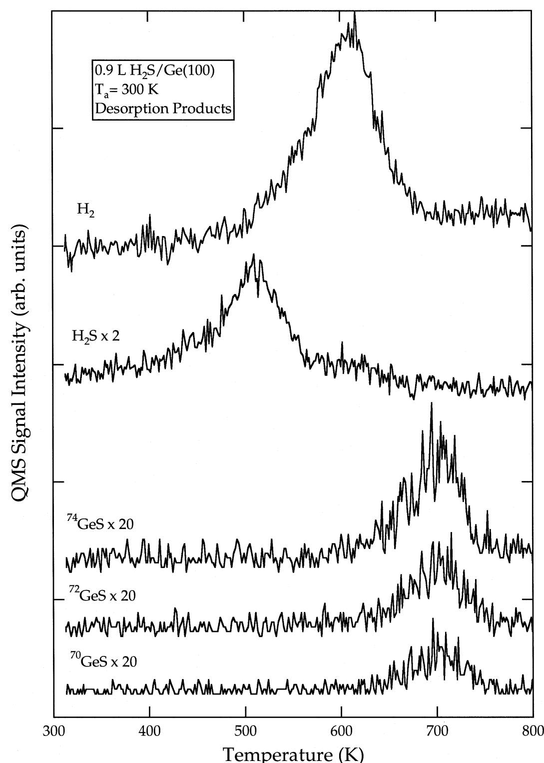


Fig. 1. TPD spectra following a 0.9 L exposure of H_2S to Ge(100) at a surface temperature of 300 K. The resulting coverage is 1.6×10^{14} H_2S molecules cm^{-2}.

3. Results

3.1. Temperature-programmed desorption results

The adsorption and decomposition of H_2S is followed by TPD. H_2, H_2S, and the etch product, GeS, are observed as desorption products. Figs. 1–3 summarize the results of the TPD experiments. No sulfur is observed on the Ge surface within the detection limits of AES after the temperature ramp is completed in these TPD experiments.

Fig. 1 shows the TPD results from a 0.9 L exposure of H_2S to the Ge(100) at a surface temperature of 300 K. This figure helps illustrate the overall features observed in TPD experiments. H_2 desorption is centered in a single peak near 610 K. This peak temperature is about 25 K higher in temperature than that observed for H_2 desorption from the recombination of H atoms when using atomic hydrogen as the adsorption precursor [21]. A smaller H_2S peak (multiplied by a factor of 2) is observed at 510 K and a smaller GeS desorption state occurs at 700 K (multiplied by a factor of 20). Following the most abundant Ge isotopes helps confirm that the product desorbing at 700 K contains germanium.

Fig. 2 summarizes the H_2 thermal desorption resulting from the decomposition of H_2S as a function of H_2S exposure. H_2 desorption contains a single peak centered at 610 K. The peak maximum does not change with increasing H_2S exposure, consistent with previous H_2 thermal desorption studies on Ge(100), and characteristic of first order desorption kinetics [18,21]. The position of the peak maximum also indicates that the hydrogen is a desorption-limited product. The intensity of the desorption state increases with increasing H_2S exposures that is the state is saturated for exposures greater than 0.9 L. At saturation, the H atom coverage resulting from the decomposition of H_2S is 3.1×10^{14} cm^{-2} (0.50 ML).

The thermal desorption results for the etch product GeS are contained in Fig. 3. The desorption state has a peak maximum temperature of 700 K that does not change with increasing H_2S exposures that is

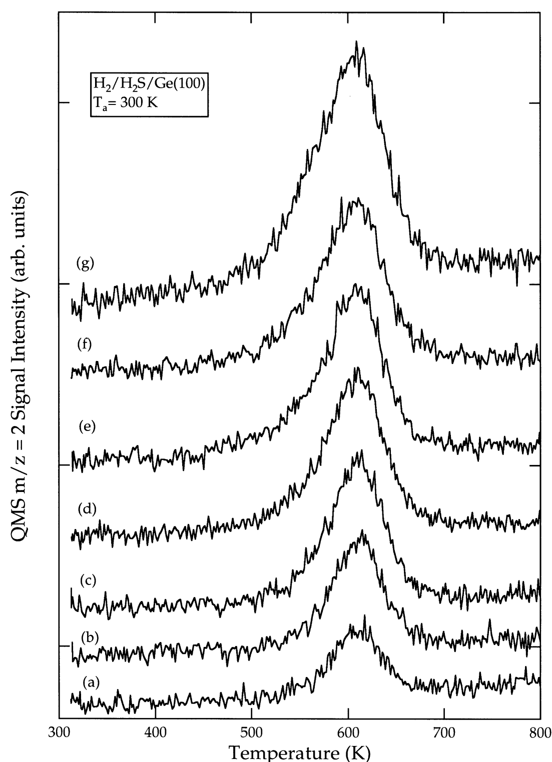


Fig. 2. TPD spectra of H_2 following different exposures of H_2S to Ge(100) at a surface temperature of 300 K. The H_2S exposures shown are (a) 0.03, (b) 0.06, (c) 0.09, (d) 0.15, (e) 0.23, (f) 0.30, and (g) 2 L. The resulting H atom coverages shown are (a) 8.7×10^{13}, (b) 1.7×10^{14}, (c) 2.3×10^{14}, (d) 2.7×10^{14}, (e) 2.9×10^{14}, (f) 3.0×10^{14}, and (g) 3.1×10^{14} cm^{-2}.

characteristic of first-order desorption kinetics. Auger spectra taken after the temperature ramp reveal that all sulfur from the decomposition of H_2S desorbs by 800 K.

3.2. Electron spectroscopy results

The adsorption of H_2S was also examined by ultraviolet photoelectron spectroscopy (UPS). Fig. 4 shows the He I UPS spectra for the clean Ge(100) surface and for several different exposures of H_2S at a surface temperature of 300 K. The He I photoelectron spectrum of the clean Ge(100) surface is included as Fig. 4a for reference. The UPS spectrum for a 0.03 L H_2S exposure is shown as Fig. 4b. Three features are observed at 3.1, 4.2, and 6.5 eV binding energy after this H_2S exposure. Increasing the H_2S exposure leads to further development of these features (Fig. 4c and 4d). The binding energy of the three peaks in Fig. 4d are 3.3, 4.6, and 6.8 eV. The lower portion of Fig. 4 (curves e–g) are difference spectra for the same three H_2S exposures. A difference spectrum is obtained by directly subtracting the spectrum for the clean Ge surface (Fig. 4a) from the spectrum of the H_2S covered surface. The UPS difference spectrum for the 0.03 L H_2S exposure is shown as Fig. 4e. The exposures for curves 4f and 4g are 0.09 and 0.3 L, respectively.

There are two bar graphs in Fig. 4; one is located above spectrum 4d and the other is located above spectrum 4g. These bar graphs are used to help assign the observed features upon H_2S exposure. The bar graph with the solid lines above spectrum 4d represent the energies obtained from the gas phase photoelectron spectrum of H_2S [22]. The line for the lowest energy peak from the gas phase spectrum

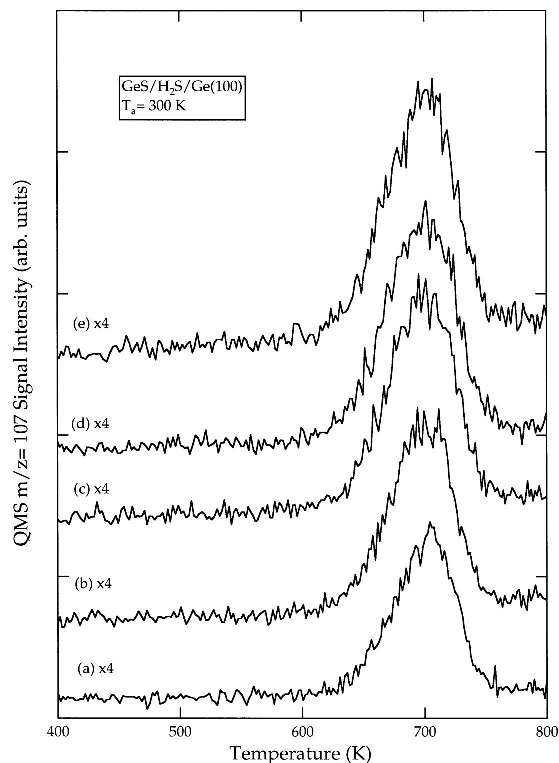


Fig. 3. TPD spectra of GeS following different exposures of H_2S to Ge(100) at a surface temperature of 300 K. The H_2S exposures shown are (a) 0.3, (b) 0.6, (c) 0.9, (d) 1.5, and (e) 2 L.

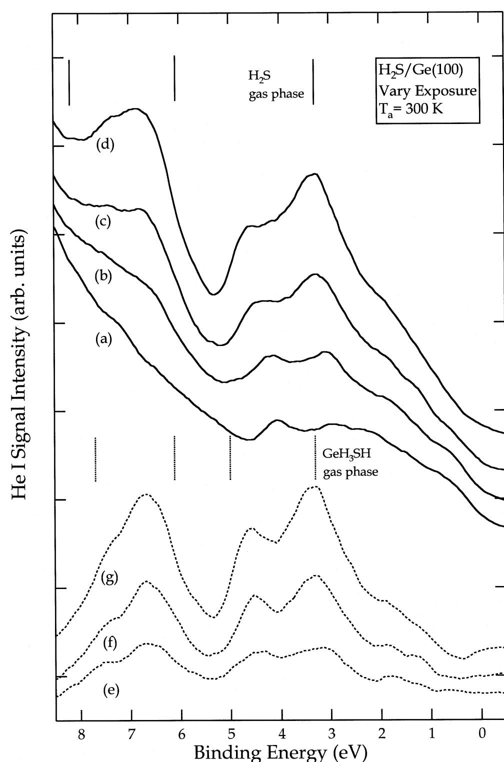


Fig. 4. He I UPS spectra of the Ge(100) exposed to H_2S at a surface temperature of 300 K. The clean Ge(100) surface is shown as spectrum (a). The H_2S exposures shown are (b) 0.03, (c) 0.09, and (d) 0.3 L. Difference spectra for H_2S exposures are shown as (e) 0.03, (f) 0.09, and (g) 0.3 L. The bar graphs represent the gas phase energies for the indicated molecules.

(highest occupied molecular orbital) is aligned with the peak at 3.3 eV binding energy in Fig. 4d. The bar graph with the dashed lines above spectrum 4g represents the energies obtained from the gas phase spectrum of GeH_3SH [23]. The line for the lowest energy peak from the gas phase spectrum of GeH_3SH (highest occupied molecular orbital) is aligned with the peak at 3.3 eV binding energy in Fig. 4g. Comparison of the gas phase H_2S energies with the measured binding energies for H_2S-exposed germanium surfaces clearly shows that H_2S dissociates upon adsorption to Ge(100) at a surface temperature of 300 K. The energies for GeH_3SH gas phase spectrum agree better with spectra 4d and 4g, however, the absolute energies differ somewhat. A better understanding of the nature of the adsorbed species can be gleaned by thermal annealing studies and these studies are presented next.

Fig. 5 summarizes the HeI UPS results for heating a H_2S-exposed Ge(100) to several different temperatures. Spectrum 5a is the UPS difference spectrum obtained after exposing the Ge(100) surface to 0.9 L of H_2S at 300 K. As shown in Fig. 4g, three main features are observed in the UPS spectrum. Annealing the surface prepared at 300 K to 470 K, cooling to 300 K, and recording the UPS spectrum results in Fig. 5b. Heating the surface to 470 K results in the loss of the peak near 7 eV binding energy and a decrease in intensity of the peak at 3.5 eV compared to the feature at 4.8 eV. In TPD experiments, molecular H_2S is observed as a desorption product at this annealing temperature. Based on the desorption of H_2S and comparison with the gas phase data of Cradock and Whiteford [23], we assign the peak near 7 eV as due to surface S-H bonds and annealing to 470 K results in the desorption of H_2S and the

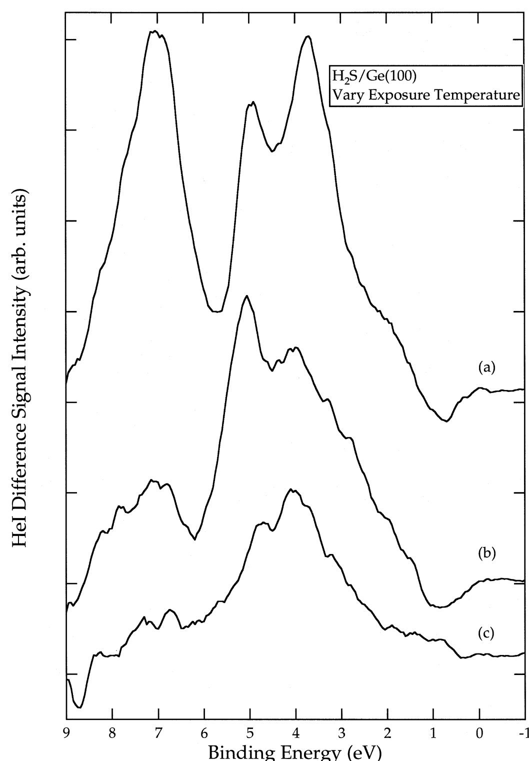


Fig. 5. He I UPS difference spectra for Ge(100) exposed to 0.9 L of H_2S at a surface temperature of (a) 300 K and then annealed to (b) 470 K, and (c) 600 K.

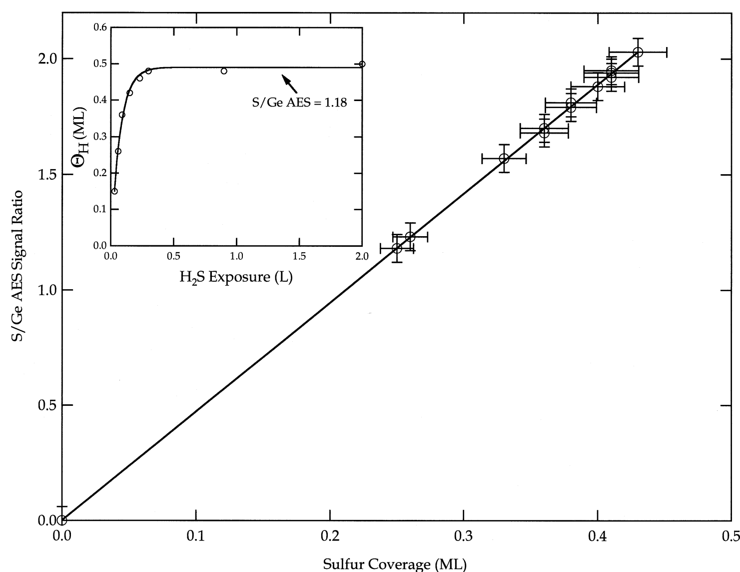


Fig. 6. The S/Ge AES signal ratio vs. sulfur coverage after exposure of H_2S to Ge(100) at 553 K. The inset to the figure shows the hydrogen atom coverage from H_2S decomposition as a function of exposure at a Ge(100) surface temperature of 300 K.

cleavage of S–H bonds. Annealing the H_2S-exposed surface further to 600 K results in spectrum 5c. Now both the peaks near 5 and 7 eV binding energy are gone. In TPD experiments, H_2 is observed as a desorption product at this temperature. We therefore assign the feature at 5 eV as due to surface Ge–H bonds. This assignment is also consistent with the previous hydrogen adsorption study of Landemark et al. [24]. The main feature in spectrum 5c, centered at 4 eV, is then assigned to sulfur on the Ge(100). The binding energy of this feature is consistent with previous work examining the adsorption of atomic sulfur on Ge(100) [12].

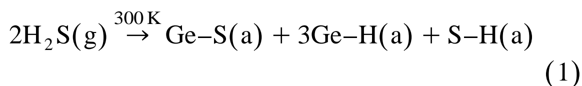
Exposing the Ge(100) to H_2S at higher surface temperatures results in a higher total sulfur coverage. These results are presented in Fig. 6 where the S/Ge Auger ratio as a function of sulfur coverage. H_2S was exposed to the Ge surface at a temperature of 553 K. The sulfur coverage in monolayers was calibrated by hydrogen thermal desorption. The inset to Fig. 6 shows the hydrogen coverage obtained by TPD vs. H_2S exposure at a surface temperature of 300 K. At saturation, 0.25 ML of S is deposited and the S/Ge AES ratio is 1.18. A linear relationship between the S/Ge AES ratio with sulfur coverage was observed for the 300 K H_2S exposures. The solid line in Fig. 6 indicates this relationship. The line is extrapolated beyond 0.25 ML. The S/Ge AES ratios obtained after exposure of H_2S at a surface temperature of 570 K are converted to a coverage in ML and are shown as open circles in Fig. 6. Exposing H_2S to Ge(100) at the elevated surface temperature results in a saturation coverage of almost 0.5 ML sulfur. UPS experiments of these higher sulfur coverages indicate that there is little co-adsorbed hydrogen.

4. Discussion

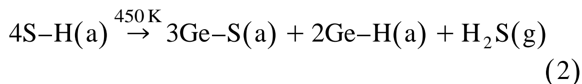
The results presented here demonstrate that S can be deposited onto the Ge(100) surface by the dissociative adsorption of H_2S. The maximum amount of S that can be deposited from a saturation exposure of H_2S at room temperature is 0.25 ML or 1.6×10^{14} S atoms cm^{-2}.

Before discussing details and before comparing this study with earlier work, we present below a

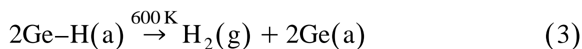
plausible mechanism for the decomposition processes.



$$2H_2S(g) \xrightarrow{300\,K} Ge\text{–}S(a) + 3Ge\text{–}H(a) + S\text{–}H(a) \quad (1)$$



$$4S\text{–}H(a) \xrightarrow{450\,K} 3Ge\text{–}S(a) + 2Ge\text{–}H(a) + H_2S(g) \quad (2)$$



$$2Ge\text{–}H(a) \xrightarrow{600\,K} H_2(g) + 2Ge(a) \quad (3)$$

$$Ge\text{–}S(a) \xrightarrow{700\,K} GeS(g) \quad (4)$$

Gas phase species are designated by (g) and (a) denotes an adsorbed surface species.

In the first step of the proposed mechanism, H_2S is exposed to the Ge(100) surface at 300 K which results in dissociative adsorption. This reaction step is followed in UPS experiments. As shown in Fig. 4b for low exposures of H_2S there are spectral features that cannot be explained as solely due to molecular adsorption. The peak at 3.1 eV binding energy in Fig. 4b (and Fig. 4e) can be attributed to the presence of sulfur. This assignment is in good agreement with previous work [12,15]. The two other reaction products, surface hydrides of germanium and sulfur, are identified in thermal annealing studies which are discussed further below. Annealing the H_2S exposed Ge(100) surface to 450 K results in the desorption of some molecular H_2S due to recombination of surface species. The desorption of H_2S is followed by TPD. Heating to 450 K also results in a significant decrease in the photoemission peak near 7 eV as shown in Fig. 5b. Cleavage of surface S–H bonds is consistent with the products shown in reaction (2). In step (3) of the above reaction sequence, surface germanium monohydrides decompose to produce molecular hydrogen as a gas phase product, as observed in the thermal desorption studies. Heating the surface to 600 K also removes the photoemission peak originally near 5 eV in Fig. 5a. The peak near 5 eV binding energy is assigned to surface germanium hydrides [24–26] and the removal of this peak by annealing to 600 K is consistent with H_2 desorption. The surface now contains only sulfur and there is a broad peak in photoemission experiments around 4 eV binding energy as shown in Fig. 5c. This broad peak has been previously observed in studies examining the adsorption of atomic sulfur on Ge(100) [12]. For saturation exposures of H_2S at 300 K, the resulting sulfur coverage is 0.25 ML. Heating the sulfur covered surface above 700 K (reaction (4)) results in the desorption of the sulfur overlayer as the etch product GeS.

Exposing the Ge(100) surface to H_2S at elevated temperatures alters the saturation coverage of sulfur. The surface temperature of 553 K was chosen because it is well below the desorption temperature of the GeS etch product and at a temperature where H_2 desorption is occurring at a significant rate. The saturation coverage of sulfur under these conditions approaches 0.5 ML. However, the results show that the actual coverage obtained is about 0.05 ML less. The lower measured coverage may be due to the presence of defect sites on the Ge surface. The number of defects could play an even larger role if H_2S requires a dimer site for adsorption and reaction and not a single surface Ge atom.

5. Conclusions

We have studied the surface chemistry of H_2S on the Ge(100) surface. This molecule dissociatively adsorbs at low coverages to yield adsorbed SH groups and surface hydrides. As the surface is heated, the surface SH groups decompose to yield adsorbed sulfur and germanium hydrides. At higher temperatures the surface hydrogen atoms recombine and desorb as molecular hydrogen. Sulfur is left at the surface after the desorption of hydrogen. The maximum amount of sulfur that can be deposited following a saturation exposure of H_2S at room temperature is 0.25 ML or 1.6×10^{14} s atoms cm^{-2}. Heating the surface to above 700 K results in the desorption of sulfur from the surface as the etch product GeS. Exposure of H_2S to Ge(100) at a surface temperature of 553 K results in a higher total sulfur coverage of about 0.5 ML. The higher coverage is a result of hydrogen desorption from the germanium surface during the exposure which generates additional surface sites for H_2S decomposition.

Acknowledgements

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