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Citation: *Appl. Phys. Lett.* **102**, 111608 (2013); doi: 10.1063/1.4798248

View online: <http://dx.doi.org/10.1063/1.4798248>

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## Optical *in situ* monitoring of hydrogen desorption from Ge(100) surfaces

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(Received 12 February 2013; accepted 12 March 2013; published online 22 March 2013)

Molecular hydrogen strongly interacts with vicinal Ge(100) surfaces during preparation in a metal organic vapor phase epitaxy reactor. According to X-ray photoemission spectroscopy and Fourier-transform infrared spectroscopy results, we identify two characteristic reflection anisotropy (RA) spectra for H-free and monohydride-terminated vicinal Ge(100) surfaces. RAS allows *in situ* monitoring of the surface termination and enables spectroscopic hydrogen kinetic desorption studies on the Ge(100) surface. Comparison of evaluated values for the activation energy and the pre-exponential factor of H desorption evaluated at different photon energies reflects that H unevenly affects the shape of the RA spectrum. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4798248>]

In the last years, there has been a renewed interest in the characterization of the structural and chemical properties of germanium (100) substrates for applications in microelectronics,<sup>1</sup> nanotechnology,<sup>2</sup> and photovoltaics.<sup>3–6</sup> A suitable surface preparation (in terms of surface cleanliness, termination, and reconstruction) is crucial for subsequent material and device properties.<sup>7,8</sup> In technologically relevant process ambients, the carrier gas (such as H<sub>2</sub>) might affect surface reconstruction,<sup>6,9</sup> step formation,<sup>10</sup> and epitaxial growth.<sup>11</sup> However, the presence of a carrier gas limits surface characterization with standard surface science techniques, which typically require UHV conditions.

From UHV-based studies, it is known that on the clean Ge(100) surface, atoms form dimers to reduce the number of free dangling bonds. Dimer buckling is present at ambient temperature and areas of (2 × 1), c(4 × 2), and p(2 × 2) surface reconstructions may coexist.<sup>12</sup> Hydrogen can passivate residual Ge dangling bonds leading to monohydride or dihydride termination, depending on process temperature as well as quantity of atomic H supply.<sup>13,14</sup> Monohydride terminated Ge surfaces (with a H coverage of 1 monolayer (ML)) show a pure (2 × 1) surface reconstruction. Typically, H desorption kinetics has been studied by temperature-programmed desorption (TPD),<sup>15</sup> scanning tunnelling microscopy (STM),<sup>16</sup> and laser-induced desorption (LID)<sup>17</sup> analysis. As a result, hydrogen desorbs from the Ge(100) surface following first order kinetics<sup>15–17</sup> with activation energies ( $E_d$ ) in the range of 1.65 to 1.82 eV and pre-exponential factors ( $\nu_d$ ) in the range of  $2.7 \times 10^{13}$  to  $2.0 \times 10^{15} \text{ s}^{-1}$ , see Refs. 16 and 17.

In H<sub>2</sub> ambient in a low-pressure metal organic vapor phase epitaxy (MOVPE) reactor, annealing of the Ge(100) surface at high temperatures leads to a clean and monohydride-terminated surface.<sup>6</sup> However, the interaction between H<sub>2</sub> and the Ge(100) surface is not completely understood during MOVPE preparation, where only optical tools can be applied for *in situ* monitoring. For materials with cubic crystal structure and isotropic bulk linear optical response, reflection anisotropy spectroscopy (RAS) is a

widely used surface sensitive *in situ* probe.<sup>18</sup> RAS measures the normalized reflectance difference between orthogonal polarization directions in near normal incidence configuration, i.e.,

$$\frac{\Delta r}{r} = 2 \frac{r_{[0\bar{1}1]} - r_{[011]}}{r_{[0\bar{1}1]} + r_{[011]}}$$

RA spectra are difficult to understand due to various contributions to the signal. The origins of the RAS signals can be identified either by *ab initio* calculations,<sup>19,20</sup> modeling,<sup>21</sup> or by correlation of RAS signals to results from other surface-sensitive techniques.<sup>22</sup> Therefore, RAS signals provide control and understanding of processes on the sample surface during preparation in any process gas ambient. The potential of RAS for monitoring the temporal evolution of adsorbates was first shown for metals.<sup>23</sup> Regarding semiconductor surfaces, RAS enabled studies of H adsorption on Si(100)<sup>24,25</sup> and the observation of H interaction with Si(100) depending on process conditions in a MOVPE reactor.<sup>26</sup>

According to Ref. 6, the monohydride-terminated Ge(100) surface shows a characteristic RAS signal. Here, we provide experimental evidence for the sensitivity of RAS to hydrogen termination by monitoring the RAS signal during thermal annealing of a monohydride-terminated Ge(100) surface in N<sub>2</sub> ambient. We correlate the RA spectrum obtained to UHV-based surface-sensitive tools such as Fourier-transform infrared (FTIR) spectroscopy, STM, and X-ray photoelectron spectroscopy (XPS) accessed by a dedicated MOVPE-to-UHV transfer system.<sup>22</sup> *In situ* RAS enables direct observations of changes on the Ge(100) surface related to hydrogen termination in any process environment, whereas LID, STM, and TPD are only applicable in UHV environment. Comparison of estimated values—derived from our RAS signals—for the activation energy and the pre-exponential factor of H thermal desorption at several photon energies revealed that the RA spectrum of the clean Ge(100) surface is differently affected by H termination.

We used Ge(100) substrates with a misorientation of  $6^\circ$  towards the [011] direction. Without any additional wet chemical pre-cleaning, the preparation was carried out in two different Aixtron MOVPE reactors (AIX-200-4 and AIX-200) located in Madrid and Berlin, respectively, both equipped with an *in situ* RA spectrometer (LayTec EpiRAS 200). The comparability between the experiments in both reactors was ensured by identical RA spectra for identically prepared Ge(100) samples. Temperature was measured by thermocouples placed inside the susceptor. Reactor parts (liner, susceptor) and sample carriers were cleaned of III-V residues to avoid unintentional contamination of the samples. The sample transfer system enabled contamination-free access to STM (Specs Aarhus 150), XPS (Specs Focus 500 and Phoibos 100), and FTIR spectroscopy (Bruker IFS 66v/S).<sup>22</sup> Thermal oxide removal consisted of an annealing step at 1000 K for 20 min using 14 slpm of Pd-purified H<sub>2</sub> at a pressure of 100 mbar.

As shown in Ref. 6, monohydride bonds terminate the Ge(100) surface if the surface is cooled down in H<sub>2</sub> ambient after the deoxidation process. The thin red line in Fig. 1 represents the characteristic RA spectrum of the surface. It consists of a broad and a narrow minimum around 3.0 eV and 1.9 eV, respectively, local maxima around the critical point (CP) energies of Ge(100) ( $E_1$  and  $E_1 + \Delta_1$  at around 2.2 eV and  $E_2$  at 4.3 eV), as well as two characteristic shoulders around 3.4 and 4.2 eV. The corresponding STM image (inset Fig. 1) shows a regular step structure with symmetric dimers on the terraces. The step height equals two atomic layers and the dimers are parallel to the step edges on adjacent terraces, i.e., the surface is preferentially  $D_B$ -stepped. The observed ( $2 \times 1$ ) surface reconstruction indicates a monohydride termination of the surface—in agreement with the results presented in Ref. 6—since we did not find areas with buckled dimers typical for clean Ge surfaces showing a  $c(4 \times 2)$  or  $p(2 \times 2)$  surface reconstruction.<sup>16,28</sup> However, the bright

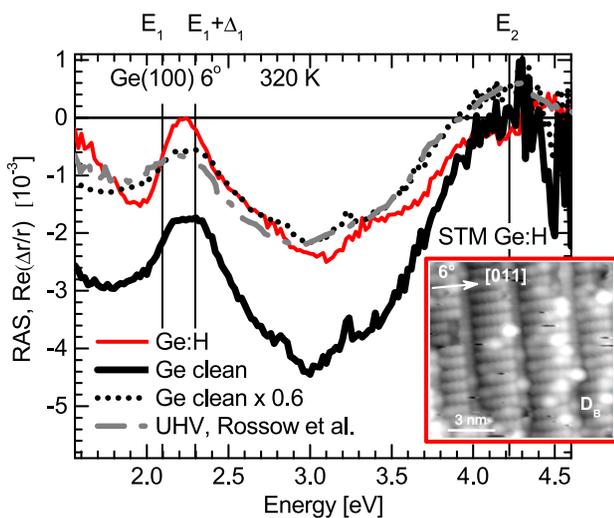


FIG. 1. *In situ* RA spectra of the monohydride-terminated (thin red line) and the clean (thick black line) Ge(100) surface with  $6^\circ$  towards the [011] prepared in MOVPE ambient. The dash-dotted thin gray line represents the RA spectrum of a UHV-prepared Ge(100) surface (see Ref. 27) and the dotted black line corresponds to the scaled RA spectrum of the MOVPE-prepared clean Ge(100) surface (i.e., thick black line). Vertical lines indicate the critical point energies of Ge(100) at 320 K. The STM image ( $V_{\text{sample}} = -1201.2$  mV,  $I_t = -0.650$  nA) corresponds to the monohydride-terminated Ge(100) surface.

ball-like features most probably correspond to missing H atoms.<sup>29,30</sup>

Fig. 1 also shows the RA spectrum after annealing the initially monohydride terminated surface under N<sub>2</sub> at 573 K and cooling to RT (thick black line). It shows features similar to the RA spectrum of the monohydride-terminated surface. The local minimum around 1.8 eV and the maximum at 2.2 eV are shifted towards lower RAS values, whereas the broad minimum around 3.0 eV is more pronounced. There is a shoulder at 3.2 eV similar to the shoulder of the RA spectrum of the monohydride terminated Ge(100) surface at 3.4 eV.

We carried out FTIR measurements in attenuated total reflection mode (ATR) to directly check for hydrogen termination after annealing in N<sub>2</sub> ambient. While FTIR measurements performed on monohydride-terminated Ge(100) surfaces displayed two characteristic absorption lines at 1986 and 1975 cm<sup>-1</sup> (see Ref. 6), no absorption lines appeared in the spectra after annealing in N<sub>2</sub> (not shown here), indicating a H-free surface. XPS measurements also confirmed the absence of any contaminants on the Ge(100) surface, in particular related to N<sub>2</sub> or C. Hence, the RAS signal measured after N<sub>2</sub> annealing corresponds to the clean and H-free vicinal Ge(100) surface, obtained by thermal hydrogen desorption from the monohydride-terminated surface in an inert gas ambient.

In Fig. 1, we include the RA spectrum (dash-dotted thin gray line) of a UHV-prepared Ge(100) surface with  $6^\circ$  offcut for comparison.<sup>27</sup> If we shift and apply a scaling factor of 0.6 to the RA spectrum obtained after annealing in N<sub>2</sub>, the spectrum (dotted black line in Fig. 1) largely overlaps with the RA spectrum of the clean UHV-prepared Ge(100) surface. The difference in RAS amplitude might be due to the coexistence of domains with mutually perpendicular orientation<sup>5,31,32</sup> or to a better surface cleanliness of the sample prepared in MOVPE ambient, since many studies report on the inevitable presence of C on UHV-prepared Ge(100) surfaces.<sup>8,33,34</sup>

The clean and the monohydride-terminated Ge(100) surfaces exhibit characteristic RAS signals, which we will apply to study the hydrogen surface coverage during preparation in the process gas ambient of a MOVPE reactor. Fig. 2(a) shows continuous RAS measurements during annealing of an initially monohydride terminated Ge(100) surface in N<sub>2</sub> ambient (100 mbar) at 523 K. The first spectrum was taken under H<sub>2</sub> process gas and shows the corresponding monohydride RAS signal (thick red line). After switching the process gas to N<sub>2</sub> (at constant temperature), we observe a successive transformation of the RAS signal with time (thin gray lines) to the characteristic RAS signal of the clean vicinal Ge(100) surface, only governed by the temperature-dependent hydrogen desorption rate. The RAS signal saturates after 60 min (thick black line), indicating the end of the H desorption from the Ge(100) surface. Upon resupplying H<sub>2</sub>, the RAS signal of the monohydride-terminated Ge surface reemerges again within a few minutes (not shown here). Consequently, the sensitivity of RAS to the hydrogen termination of vicinal Ge(100) allows us to study the hydrogen desorption and adsorption characteristics dependent on varying process conditions.

In order to perform detailed kinetic studies, it is essential to use a RAS signal (at a given photon energy) that fully reflects hydrogen termination. Features in the RA spectra are a result of an anisotropic electronic structure, which in cubic crystals may

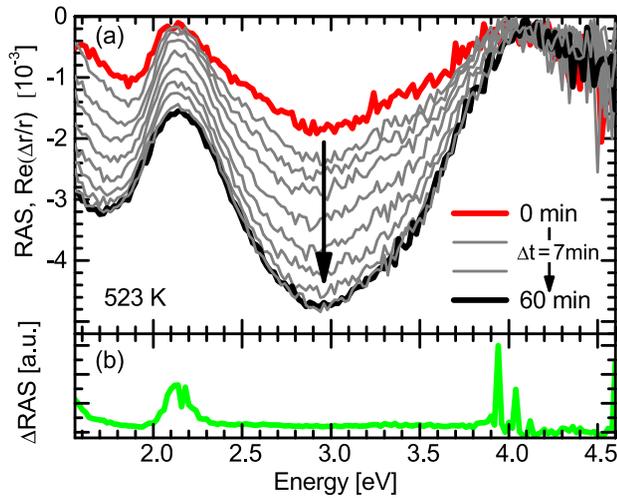


FIG. 2. (a) *In situ* RAS measurements (thin gray lines) during hydrogen desorption from the vicinal Ge(100) surface at 523 K. Switching the process gas from H<sub>2</sub> to N<sub>2</sub> ( $t = 0$  min) induces successive H desorption, which transform the monohydride RAS signal (red line) into the RAS signal of the clean Ge(100) surface (black line) after 60 min, when hydrogen desorption is complete. Acquisition time of each spectrum was 3.5 min (b) Relative difference between the RAS signal of the monohydride-terminated (red line) and the clean surface (black)  $\Delta\text{RAS} = [(\text{RAS}_{60\text{min}} - \text{RAS}_{0\text{min}}) / \text{RAS}_{0\text{min}}]$ .

arise at reconstructed surfaces and can involve both surface states and surface modulated bulk states.<sup>20,35</sup> Since there was not any corresponding theoretical analysis available for the Ge(100) surface, here, we empirically analyzed the different contributions in the RA spectrum. Fig. 2(b) shows the relative difference  $\Delta\text{RAS} = [(\text{RAS}_{60\text{min}} - \text{RAS}_{0\text{min}}) / \text{RAS}_{0\text{min}}]$  between the clean (i.e., black spectrum) and monohydride-terminated (i.e., red spectrum) surfaces in Fig. 2(a). This plot allows us to elucidate whether the presence of H equally affects the shape of the RA spectrum. In that case, we would get a constant  $\Delta\text{RAS}$  over energy (i.e., straight line in Fig. 2(b)). However,  $\Delta\text{RAS}$  shows a peak centered at 2.1 eV (around the E<sub>1</sub> critical point) and a wide plateau centered around 3.0 eV. The peaks at higher energies are an artefact of the calculation, since the RA spectra of both the monohydride and the clean surfaces are very similar and close to zero.

In UHV, hydrogen desorption from monohydride terminated Ge(100) surfaces follows a first-order reaction,<sup>15–17</sup> where the rate equation is

$$\frac{dS}{dt} = -\kappa S = -v_d \exp\left(-\frac{E_d}{k_B T}\right) S, \quad (1)$$

with  $S$  being the hydrogen related signal,  $\kappa$  the first-order rate constant,  $v_d$  the pre-exponential factor,  $E_d$  the activation energy, and  $k_B$  the Boltzmann constant. In order to estimate  $E_d$  and  $v_d$ , we conducted isothermal H desorption measurements for several temperatures in the range of 513–553 K. We analyzed the temporal development of the spectra (hereinafter RA transient mode) for a photon energy of 2.92 eV (center of the wide plateau in Fig. 2(b)), assuming that the decay of the RAS amplitude corresponds to the loss in hydrogen coverage. Below, we will comment on the same analysis performed at 2.12 eV (peak in Fig. 2(b)) and other photon energies. Accordingly, we use the normalized RA transient as the hydrogen-related signal ( $S$ ) in Eq. (1). We assume roughly 1

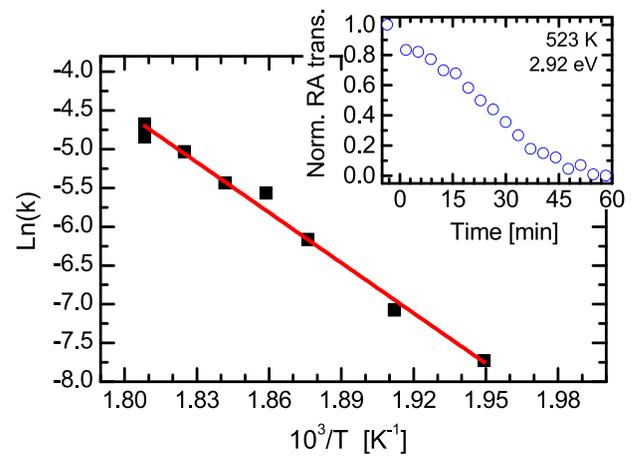


FIG. 3. Arrhenius plot of the H<sub>2</sub> desorption from Ge(100) surface in the temperature range 513–553 K, as determined from the RAS transients at 2.92 eV. The inset shows the normalized RA transient at 2.92 eV taken from the data of Fig. 2.

ML hydride coverage ( $S = 1$ ) prior to desorption in N<sub>2</sub>, since the RAS signal saturates to a constant value when H<sub>2</sub> is resupplied and neither dihydride formation nor relevant amounts of single dangling bonds have been detected with FTIR<sup>6</sup> and STM, respectively. We also assume no hydrogen coverage ( $S = 0$ ) when the RAS signal saturates after H desorption in N<sub>2</sub>. The inset of Fig. 3 shows the normalized RAS transient at 2.92 eV of the RAS evolution from Fig. 2(a). At  $t = 0$ , process gas was switched from H<sub>2</sub> to N<sub>2</sub>. The transient shows a non-single exponential decay (in contrast to Refs. 16 and 17) probably due to hydrogen readsorption,<sup>36</sup> which depends on the relative magnitudes of the chemisorption rate, desorption rate, and pumping speed.<sup>36</sup> Therefore, we used the relation  $dS/dt = -\kappa S$  to obtain the rate constants  $\kappa$  only for  $S < 0.5$ , where readsorption effects are reduced.<sup>36</sup> The resulting Arrhenius plot of  $\kappa$  for this temperature range (Fig. 3) is linear over two orders of magnitude in the desorption rate, as in Ref. 17. From the slope and intercept of the best fit to the experimental data, we calculated  $E_d = (1.9 \pm 0.1)$  eV and  $v_d = 9 \times 10^{14 \pm 1} \text{ s}^{-1}$ , respectively. These values are in good agreement with the data published in literature for hydrogen desorption from Ge(100) measured in other process environments by different experimental techniques.<sup>11,16,17</sup>

We conducted the same analysis at 1.72, 2.12, and 2.52 eV (i.e., representative energies of the features observed in Fig. 2(b)). The corresponding results are summarized in Table I. Both  $E_d$  and  $v_d$  for 1.72, 2.52, and 2.92 eV agree well with each other within the error estimation and, in particular, with values reported in literature,<sup>11,16,17</sup> whereas the activation energy obtained at 2.12 eV (around the E<sub>1</sub> CP of bulk Ge) strongly deviates. Consequently, we suggest that the change in the minima at 1.8 and 3.0 eV between the RA spectra of the clean and monohydride-terminated Ge(100) surfaces (thick black and thin red lines in Fig. 1) is directly related to hydrogen coverage. In contrast, the change in the local maximum at 2.1 eV is only partially related to hydrogen coverage. In other words, these findings indicate that the RAS signal of the clean Ge(100) surface is differently affected by H termination, as already expected from  $\Delta\text{RAS}$  in Fig. 2(b).

TABLE I. Comparison of the kinetic parameters for the H desorption from the Ge(100) surface obtained from the RAS transients at different photon energies.

Photon energy (eV)	$E_d$ (eV)	$V_d$ (s <sup>-1</sup> )
1.72	1.8 ± 0.1	4 × 10 <sup>14±1</sup>
2.12	1.4 ± 0.1	4 × 10 <sup>10±1</sup>
2.52	1.8 ± 0.1	7 × 10 <sup>14±1</sup>
2.92	1.9 ± 0.1	9 × 10 <sup>14±1</sup>

In conclusion, we observed a characteristic RA spectrum for clean and monohydride terminated Ge(100) surfaces, respectively, and demonstrated the RAS sensitivity to hydrogen coverage on vicinal Ge(100) surfaces. This enables dedicated *in situ* investigations of hydrogen termination on Ge(100) in dependence on temperature and pressure in the MOVPE reactor. Beyond showing the capability of RAS to perform H desorption kinetic studies, we indirectly showed that H affects the shape of the RA spectrum unevenly at different photon energies. Theoretical studies are desirable to understand the microscopic origins of the features in the optical *in situ* RA spectrum of the clean and monohydride terminated Ge(100) surfaces. H desorption studies by RAS, as established here for Ge(100), may also be transferred to other hydrogen-terminated surfaces such as Si(100).

Financial support from the EC (NGCPV-283798), the Spanish MINECO (TEC2008-01226, TEC2011-28639-C02-01, IPT-2011-1441-920000 and IPT-2011-1408-420000) and CAM (S2099/ENE1477), and from the German BMBF (03SF0329C) are gratefully acknowledged.

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