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## Surface diffusion of hydrogen atoms on Cu(111) studied by optical diffraction from hydrogen density patterns formed on removable templates of xenon monolayers

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**Abstract.** – We formed grating-like patterns of hydrogen adatom density on Cu(111) by using complementary patterns of xenon adatom density as the template. The template was prepared by laser-induced thermal desorption of a Xe monolayer on Cu(111) using the interference pattern of two coherent laser pulses. By following the evolution of hydrogen density gratings on Cu(111) from 153 to 183 K with linear optical diffraction, we found that the diffusion of hydrogen atoms on Cu(111) in this temperature range is the classical over-barrier hopping characterized by an activation energy barrier  $E_{\text{diff}} = 6.4$  Kcal/mol (or 279 meV) and a pre-exponential factor  $D_0 = 2.0 \times 10^{-3}$  cm<sup>2</sup>/s. Based on the potential model proposed by Basdescu and coworkers for hydrogen on Ni(111), the WKB tunneling coefficient between the first vibrational excited states of a hydrogen adatom on Cu(111) is expected to be at least one order of magnitude smaller than that on Ni(111), indicating that the classical over-barrier hopping of hydrogen atoms could directly cross over to under-barrier tunneling between ground states on Cu(111).

Adatom diffusion on solids by classical over-barrier hopping and quantum-mechanical under-barrier tunneling continues to be a topic of great interest in condensed-matter physics [1–3]. Small masses of hydrogen and its isotopic family make them good candidates particularly for investigating quantum dissipation effect on adatom diffusion on metals, where roles of phonons, conduction electrons, disorders, and dimensionality can be systematically explored [4–9]. One of the enabling experimental techniques that are capable of measuring diffusion constants over a wide dynamic range is optical diffraction from periodic patterns of adatom density on a substrate, as first reported by Zhu and coworkers [10]. To efficiently form patterns of adsorbate density (coverage) that can be interrogated with optical diffraction, one typically employs the method of laser-induced desorption with interference patterns of nanosecond coherent optical pulses [11]. The laser interference patterns produce surface temperature profiles that are responsible for the pattern-forming desorption. The choice of nanosecond optical pulses for desorption (*e.g.*,  $\tau_p \sim 10$  ns) is to ensure the thermal diffusion length over the pulse duration, varying as  $\sqrt{\tau_p}$ , be less than the length scale of the interference pattern so that the surface temperature patterns maintain sufficient contrasts.

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Zhu and coworkers exploited the dynamic range afforded by the optical-diffraction technique and successfully studied the crossover in hydrogen diffusion on Ni(111) from a classical over-barrier hopping to a phonon-assisted under-barrier tunneling [2]. More recently using a low-temperature scanning tunneling microscope (STM), another experimental technique with an inherently large dynamic range that complements the optical-diffraction method, Ho and coworkers studied the crossover in hydrogen diffusion on a more open Cu(100) from a classical over-barrier hopping to a conduction-electron dissipation-dominated under-barrier tunneling [3]. To truly gain a comprehensive understanding of classical and quantum-mechanical behaviors in adatom diffusion, it is crucial that experimental studies based on these two techniques are extended to other metal surfaces, including application of both techniques to same metal surfaces such as Cu. The latter is necessary for arriving at a complete picture of adatom diffusion from room temperature to a few kelvin (or less) with diffusion constants varying over 20 orders of magnitude.

However, the straightforward application of optical-diffraction methods to hydrogen adatoms on low-melting-point metals such as Cu is problematic. As pointed out by Williams *et al.*, thermal desorption of strongly bonded adsorbates with nanosecond optical pulses may require to bring the surface temperature during desorption too close to or over the melting point of the substrate and cause undesirable optical damages [12]. Indeed for hydrogen on Cu(111) with a desorption activation energy  $E_{\text{des}}(\text{H}/\text{Cu}(111)) = 0.63 \text{ eV}$  and a pre-exponential factor  $\nu_{\text{d0}}(\text{H}/\text{Cu}(111)) = 1 \times 10^9/\text{s}$ , the peak surface temperature on Cu(111) needed to remove 50% of hydrogen adatoms in 10 ns is 1500 K, well over the melting point of 1358 K for Cu [13, 14].

To get around this experimental difficulty, Williams and coworkers proposed that density patterns of physisorbed species, more easily made with the laser-induced desorption technique, can be used as the template for forming density patterns of more strongly bonded species. These authors further demonstrated this idea by forming a grating-like hydrogen density pattern on Si(111) using a Xe density pattern as the template [12]. Subsequently using similar Xe templates, Kerner and coworkers successfully formed 0.65 microns wide gold wires on Ru(100) [15]. Such a templating strategy can be generally applied to any metal substrate. In this letter we report the experimental results of using Xe monolayer templates to form hydrogen adatom density patterns on Cu(111) and of using linear optical diffraction from the hydrogen density pattern to study the diffusion of hydrogen adatom on Cu(111). We show how such a method is optimized with other *in situ* optical monitoring probes.

In a recent experimental study, we find that a pre-adsorbed Xe monolayer on Cu(111) reduces the net sticking probability of subsequently arrived hot hydrogen molecules by at least a factor of 20 [13]. In the same study, we also find that the thermal desorption of Xe from Cu(111) is characterized by an activation energy  $E_{\text{des}}(\text{Xe}/\text{Cu}(111)) = 0.25 \text{ eV}$  and a pre-factor  $\nu_{\text{d0}}(\text{Xe}/\text{Cu}(111)) = 8.2 \times 10^{14}/\text{s}$ . The peak surface temperature needed to remove 50% of Xe adatoms from Cu(111) in 10 ns is 170 K, far below the melting point of Cu. These findings suggest that periodic density patterns of Xe adatoms on Cu(111) can be used effectively as templates for forming periodic hydrogen density patterns.

The experiment is carried out in an ultrahigh vacuum chamber with a base pressure of  $2 \times 10^{-10}$  Torr. A 10 mm diameter  $\times$  1.5 mm thick Cu(111) disc (prepared by Surface Preparation Laboratory, with the miscut angle less than  $0.1^\circ$ ) is used as the substrate. Prior to experiments the substrate is cleaned with cycles of ion sputtering followed by thermal annealing. We can vary the substrate temperature from 33 K to 1500 K. In addition to conventional surface preparation and analysis apparatus, the chamber is also equipped with a CreaTec Fischer Atomizer (HLC-40-200). We use the Atomizer to produce hot hydrogen molecules without fragmentation, sufficient to overcome an activation barrier for dissociative adsorption of molecular hydrogen on Cu(111). For adsorption experiment, the partial pressures of hot

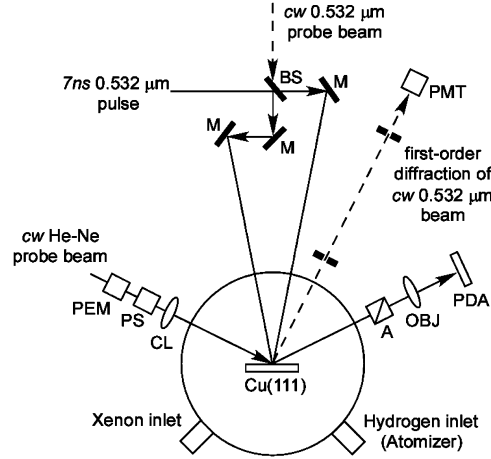


Fig. 1 – Sketch of optical set-ups for fabrication and characterization of periodic density profiles of Xe and hydrogen adatoms on Cu(111). The substrate is in an ultrahigh vacuum chamber and is held at 38 K. The hydrogen gas enters the chamber through an atomizer that heats up the gas. M: dielectric mirrors. BS: dielectric beam splitter. PMT: photomultiplier. PEM: photoelastic modulator. PS: phase shifter. CL: cylindrical lens. A: polarizing analyzer. OBJ: objective. PDA: photodiode detector array. CL focuses the He-Ne beam into a vertical line across the Cu(111) surface. OBJ images the illuminated line onto PDA.

molecular-hydrogen gas and Xe gas in the chamber are monitored with an ionization gauge, and the readings are corrected with the appropriate gas conversion factors.

We use three optical systems (shown in fig. 1) for fabrication and characterization of density patterns of adsorbates on Cu(111). To make periodic density patterns of Xe adatoms by laser-induced thermal desorption, we use a single 7 ns optical pulse at  $0.532 \mu\text{m}$  obtained by frequency-doubling the output of a pulsed Nd : YAG laser operated at  $1.064 \mu\text{m}$ . We split the optical pulse into two parts and recombine them on Cu(111) to form an interference pattern. The incidence angle is chosen to yield a pattern with a spatial periodicity of  $2a = 5.45 \mu\text{m}$ . To characterize the Xe adatom density pattern and the subsequent hydrogen density pattern, we illuminate the “patterned” region with a cw laser (also at  $0.532 \mu\text{m}$ ) and measure the first-order ( $m = +1$ ) diffraction.

To simultaneously monitor the mean density (or coverage) distributions of Xe and hydrogen adatoms on Cu(111), we use an oblique-incidence optical reflectivity difference (OI-RD) system as a probe. Such an *in situ* monitor enables us to optimize the process of density pattern formation. Details of the measurement procedures have been described elsewhere [16]. For the present experiment the OI-RD probe laser is a 5 mW He-Ne laser at  $0.633 \mu\text{m}$ , and is incident on the Cu(111) surface at  $\varphi_{\text{inc}} = 66^\circ$ . Let  $r_{p0} = |r_{p0}| \exp[i\Phi_{p0}]$  and  $r_{s0} = |r_{s0}| \exp[i\Phi_{s0}]$  be the reflection coefficients for the *p*- and *s*-polarized components of the probe beam from the bare Cu surface. Let  $r_p = |r_p| \exp[i\Phi_p]$  and  $r_s = |r_s| \exp[i\Phi_s]$  be the reflection coefficients from the surface covered with adsorbates. In oblique-incidence reflectivity difference (OI-RD) experiment, we measure the reflectivity difference defined as  $\Delta_p - \Delta_s \equiv (r_p - r_{p0})/r_{p0} - (r_s - r_{s0})/r_{s0}$  [14,17,18]. The real part,  $\text{Re}\{\Delta_p - \Delta_s\} = |r_p|/|r_{p0}| - |r_s|/|r_{s0}|$ , measures *differential magnitude change*, while the imaginary part,  $\text{Im}\{\Delta_p - \Delta_s\} = (\Phi_p - \Phi_{p0}) - (\Phi_s - \Phi_{s0})$ , measures *differential phase change*. As we have shown earlier, adsorption of Xe on Cu(111) only leads to a differential phase change (*i.e.*,  $\text{Im}\{\Delta_p - \Delta_s\}$ ) at  $0.633 \mu\text{m}$ , while adsorption of hydrogen atom on Cu(111) primarily induces a differential magnitude change (*i.e.*,

$\text{Re}\{\Delta_p - \Delta_s\}$ ) [13]. The different responses enable us to determine the coverage of both hydrogen and Xe even when they both are present on Cu(111). From the respective changes in  $\text{Re}\{\Delta_p - \Delta_s\}$  and  $\text{Im}\{\Delta_p - \Delta_s\}$ , we extract the coverage of hydrogen and Xe during the density pattern formation and subsequent processing. To obtain the profile of the mean coverage across the “patterned” region, we focus the He-Ne laser beam with a cylindrical lens to a 10 mm vertical line on the Cu(111) surface, passing through the center of the “patterned” region. The reflected He-Ne beam is focused onto a 76-element photodiode array so that we measure  $\Delta_p - \Delta_s$  and in turn determine the coverage along the 10 mm line across the substrate surface with a resolution of 0.13 mm.

The experimental procedure is as follows. The Cu(111) substrate is held at 38 K during the formation and subsequent processing of periodic adatom density patterns. We first deposit one monolayer of Xe on Cu(111) by back-filling the chamber with Xe gas at a pressure of  $3 \times 10^{-8}$  Torr. The adsorption is monitored in real time with the OI-RD system. When a full monolayer of Xe adatoms covers Cu(111) in a close-packed structure, there is one Xe atom for every three top-layer Cu atoms and therefore the saturation coverage is  $\theta_{\text{Xe},s} = 1/3$ . From a nearly linear relation between  $\text{Im}\{\Delta_p - \Delta_s\}$  and the Xe coverage on Cu(111) [13], we deduce a sub-monolayer coverage  $\theta_{\text{Xe}}$  by multiplying  $\theta_{\text{Xe},s} = 0.33$  with the ratio of the corresponding  $\text{Im}\{\Delta_p - \Delta_s\}$  to  $\text{Im}\{\Delta_p - \Delta_s\}$  at  $\theta_{\text{Xe},s} = 0.33$ . We make a grating-like density pattern of Xe adatoms by irradiating the Xe-covered Cu(111) with the interference pattern. In fig. 2a, we display the mean Xe coverage across the Cu(111) surface. The region with significant desorption covers a 3 mm diameter area. At the center, nearly 50% of Xe adatoms is desorbed, indicating that the depth of density modulation should be close to being optimal. We then expose the surface to hot molecular hydrogen at a partial pressure of  $4 \times 10^{-7}$  Torr for 60 seconds by passing hydrogen gas through the Atomizer filament held at 1473 K. The hydrogen coverage is deduced from the simultaneously monitored  $\text{Re}\{\Delta_p - \Delta_s\}$  as outlined in ref. [13]. In fig. 2b, we show the mean hydrogen coverage along the same line as what is shown in fig. 2a. As we have expected [13], hydrogen adatoms adsorb only in the region where Xe adatoms are significantly depleted. And the density of the hydrogen adatom should have a grating-like pattern, complementary to that of the Xe density pattern.

The modulation depths of the grating-like density patterns for Xe and hydrogen adatoms are revealed by the linear optical diffraction from these patterns. In fig. 3, we show the simultaneously monitored Xe and hydrogen coverage at the center of the patterned region, and the first-order diffraction signal from the region during *a sequence of pattern formation/removal/confirmation* steps. The diffraction signal displayed in fig. 3 is the ratio of the first-order diffraction intensity to the specular reflection intensity. The experimental sequence of steps is as follows: A) adsorption of a Xe monolayer on Cu(111) at  $T = 38$  K by exposing the surface to Xe gas for a length of time; B) formation of a periodic density pattern of Xe adatoms by laser-induced thermal desorption using the interference pattern of two coherent optical pulses; C) formation of a complementary density pattern of hydrogen adatoms using the Xe density pattern as the template by exposing the surface to hot hydrogen molecules for one minute; D) removal of the Xe density pattern by laser-induced thermal desorption with multiple, spatially uniform optical pulses; E) formation of a periodic density pattern of Xe adatoms again using the hydrogen density pattern as the template by exposing the surface to Xe gas; F) removal of the newly formed Xe adatom density pattern by laser-induced thermal desorption; G) further confirmation of the periodic hydrogen adatom density pattern by exposing the surface to hot hydrogen molecules for one minute.

As expected, the reduction in Xe mean coverage near the center of the “patterned” region at step B is accompanied by the emergence of the diffraction signal. The subsequent adsorption of hydrogen adatoms in the patterned region (step C) leads to the formation of a complementary

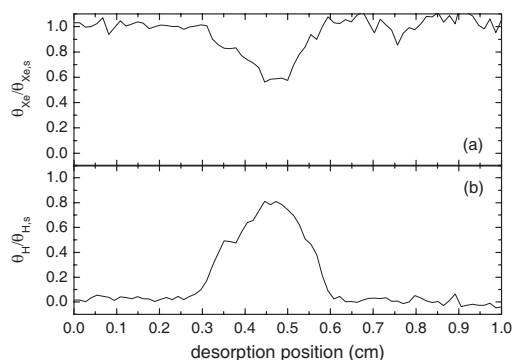


Fig. 2

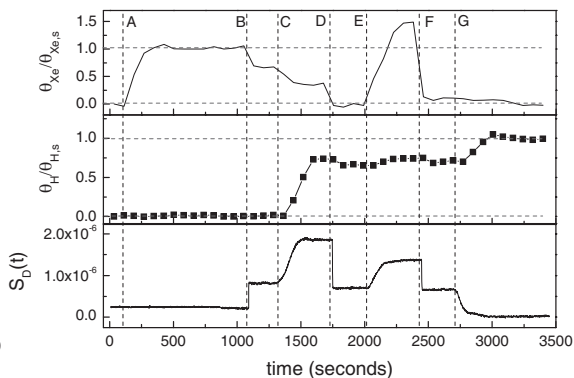


Fig. 3

Fig. 2 – (a) Mean Xe coverage along the vertical line (illuminated by the He-Ne laser for the OI-RD measurement) across the Xe-adatom-covered Cu(111) after the surface is irradiated with the interference pattern of two coherent 7 ns optical pulses at  $0.532 \mu\text{m}$ . (b) Mean hydrogen atom coverage along the same line when the surface is subsequently exposed to hot hydrogen gas. Hydrogen adatoms only adsorb in the region where Xe adatoms vacate.

Fig. 3 – Evolution of Xe and hydrogen coverage on Cu(111) at the center of the region where the laser-induced thermal desorption occurs, and the linear optical diffraction signal from the same region  $S_D(t)$  (normalized to the intensity of the specular reflection). The substrate is held at 38 K. A: adsorption of one monolayer of Xe adatoms. B: formation of a periodic density pattern of Xe adatoms by thermal desorption with the interference pattern of two coherent 7 ns pulses. C: formation of a complementary periodic density pattern of hydrogen adatoms by exposing the surface to hot hydrogen molecules. D: removal of Xe adatoms from the patterned region with multiple shots of single-beam 7 ns pulses. E: re-adsorption of Xe adatoms on the hydrogen density pattern covered Cu(111). F: removal of Xe adatoms from the patterned region again with multiple shots of single-beam 7 ns pulses. G: adsorption of hydrogen adatoms on the hydrogen density pattern covered Cu(111).

hydrogen density pattern, as indicated by an increase in the diffraction signal. By removing the Xe adatoms in the patterned region (step D), the profile of the mean hydrogen adatom density remains unchanged. Presumably the modulation depth should remain unchanged by the Xe removal process. The total diffraction signal is reduced to a level that should come solely from the hydrogen density pattern. This is confirmed by the subsequent steps. When the Xe atoms are allowed to adsorb again on the Cu(111) surface (step E), they form a density pattern similar to the original one formed at step B. This leads to the recovery of the diffraction signal. We note that the excess Xe atoms on top of both hydrogen adatoms and the first-layer Xe adatoms contribute little to the diffraction signal. When the newly formed Xe density pattern is removed again (step F), the diffraction signal returns to the level before the reformation of the Xe adatom density pattern. This confirms that the formation and removal of the Xe adatom density pattern has no effect on the hydrogen adatom density pattern, and the diffraction signal after step D indeed comes from the hydrogen density pattern alone. The final proof that the remaining diffraction signal *completely* comes off the hydrogen density pattern is the observation that when the surface is exposed to hot hydrogen molecules, not only the mean hydrogen coverage increases from 0.75 to unity, but also the diffraction signal diminishes. We note that the diffraction intensity signals (rather than the respective electric fields) from the Xe density grating and hydrogen density grating are additive. This is consistent with a separate analysis showing that the respective electric fields are roughly  $90^\circ$  out of phase.

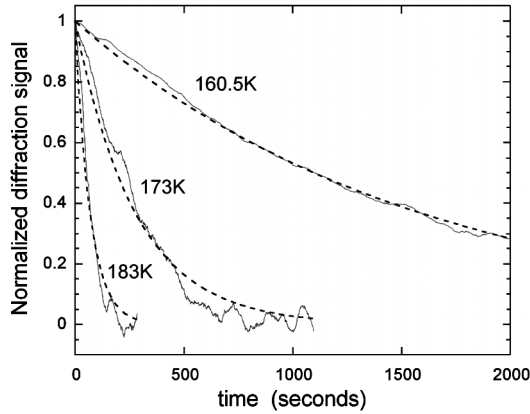


Fig. 4

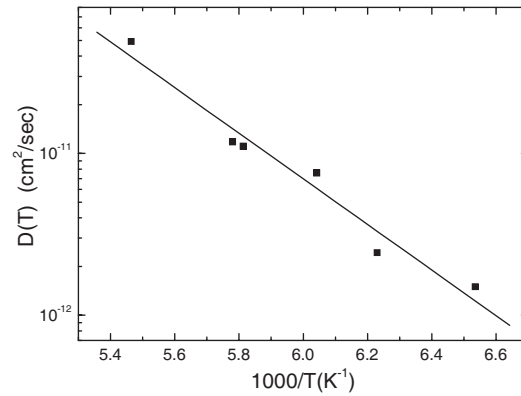


Fig. 5

Fig. 4 – Measured temporal profiles (solid lines) of linear optical diffraction (normalized to initial values) from periodic density gratings of hydrogen adatoms on Cu(111) at 160.5 K, 173 K and 183 K, respectively. Dotted lines are fits to single exponential function  $S(t)/S(0) = \exp[-\alpha(T)t]$ .  $\alpha(T)$  is related to surface diffusion constant  $D(T)$  by  $\alpha(T) = (2\pi^2/a^2)D(T)$ , with  $a$  being the half period of the density grating.

Fig. 5 – Arrhenius plot of diffusion constants  $D(T)$  for hydrogen adatoms on Cu(111) as a function of substrate temperature from 153 to 183 K. The solid line is a fit to  $D(T) = D_0 \exp[-E_{\text{diff}}/k_B T]$  with  $D_0 = 2.0 \times 10^{-3} \text{ cm}^2/\text{s}$ , and  $E_{\text{diff}} = 6.4 \text{ kcal/mol}$  (or 279 meV/atom).

As a first step toward making use of such periodic hydrogen adatom density patterns, we performed a preliminary study of hydrogen diffusion on Cu(111) by following the evolution of these patterns at elevated temperatures. To do so, we formed grating-like hydrogen density patterns using the present method with the substrate held to 38 K. We then quickly raised the substrate temperature to a value between 153 and 183 K and monitored the evolution of the optical diffraction signals. In fig. 4, we show the temporal profiles of linear optical diffraction signals measured at  $T = 160.5 \text{ K}$ , 173 K, and 183 K, respectively. These profiles (solid lines) are fit to the single exponential function  $S(t) = S(0) \exp[-\alpha(T)t]$  with the fitting curves plotted as dotted lines. By simultaneously monitoring the mean coverage of hydrogen adatoms with the OI-RD measurement, we find that there is no detectable desorption of hydrogen adatoms from Cu(111) at 183 K over 2000 seconds. As a result, the exponent is related to the surface diffusion constant by  $\alpha(T) = (2\pi^2/a^2)D(T)$  with  $a = 2.725 \mu\text{m}$  being the half-period of the density grating. As a result, we obtained the surface diffusion constant  $D(T)$ . In fig. 5, we show the Arrhenius plot of  $D(T)$  from 153 to 183 K. In this temperature range  $D(T)$  is characterized by  $D_0 \exp[-E_{\text{diff}}/k_B T]$ , with  $E_{\text{diff}}(\text{H}/\text{Cu}(111)) = 0.279 \text{ eV}$  and  $D_0(\text{H}/\text{Cu}(111)) = 2.0 \times 10^{-3} \text{ cm}^2/\text{s}$ . Compared to the activation energy for recombinative desorption of hydrogen adatoms on Cu(111) [13],  $E_{\text{diff}}(\text{H}/\text{Cu}(111)) \approx 0.5E_{\text{des}}(\text{H}/\text{Cu}(111))$ . For hydrogen atoms on Ni(111) (a surface similar to Cu(111)),  $E_{\text{diff}}(\text{H}/\text{Ni}(111)) = 0.197 \text{ eV}$  and  $D_0(\text{H}/\text{Ni}(111)) = 2.8 \times 10^{-3} \text{ cm}^2/\text{s}$  [2]. The 42% increase in activation energy barrier and 2.5% increase in lattice constant on Cu(111) in comparison with Ni(111) is significant. On Ni(111) the first vibrational excited state is 100 meV below the saddle point. Using a model potential, Badescu and coworkers showed that the WKB tunneling coefficient  $\eta$  between the vibrational excited states could be as large as  $10^{-3}$  [6]. They suggested that such a large tunneling coefficient was responsible for making tunneling between first vibrational excited states the first-phase of under-barrier tunneling diffusion of hydrogen atoms on Ni(111) as

observed by Cao, Nabighian and Zhu [2,6]. On Cu(111), the first vibrational excited state for a hydrogen adatom is 180 meV below the saddle point (the difference between the activation energy for diffusion and the excitation energy of the in-plane vibrational mode) [19]. Within the same model potential as used by Badescu and coworkers, the WKB tunneling coefficient  $\eta$  between the first vibrational excited states on Cu(111) should be at least one order of magnitude smaller. If the detail of the potential energy surface on Cu(111) is such that  $\eta$  is 2–3 orders of magnitude smaller than that on Ni(111), the classical over-barrier hopping can directly cross over into under-barrier tunneling between ground states on Cu(111) [3], an exciting scenario that deserves further experimental examination.

The present experimental work demonstrates a systematic way to use templates of physisorbed species to form spatial patterns of strongly bonded species, including characterization of adsorption and desorption kinetics of both species and optimization of spatial patterns with an array of *in situ* optical techniques. We should note that other weakly bonded species than rare gas can be used to extend the range of temperature at which the template is fabricated and utilized. Based on the theoretical model of Badescu *et al.* [6], the classical over-barrier hopping of hydrogen atoms on Cu(111) may directly cross over to tunneling between hydrogen-metal vibrational ground states, instead of tunneling between the vibrational excited states as the first step.

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