Diffusion, nucleation and reaction in a three-component system: Fe on Si(111)-‘5 × 5’–Cu

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Abstract. The role of an ultrathin Cu layer on the nucleation and growth of Fe/Si nanostructures on Si(111) is investigated using scanning tunneling microscopy. While for deposition on the bare Si(111)-7 × 7 surface, the diffusion and nucleation of Fe atoms are significantly influenced by the reaction with the Si substrate, the Si(111)-‘5 × 5’–Cu surface causes a decoupling of the diffusion and nucleation on and reaction with the surface. For moderate temperatures (T ≤ 350°C), this decoupling results in a diffusion behaviour, which can be adequately described by the conventional nucleation theory (CNT), using a single value for the effective diffusion barrier $E^* = 0.49$ eV and a critical nucleus size $i = 2$. Furthermore, it is evidenced that only Fe atoms contribute to the critical nucleus and $E^*$ thus describes the diffusion and nucleation of Fe atoms. On the other hand, at temperatures above 400°C, the island growth kinetics change considerably, i.e. the preferential step edge nucleation and three-dimensional growth are observed, and CNT is no longer applicable.

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1. Introduction

Triggered by their wide range of intriguing and unique properties and their potential integration in future applications, low-dimensional structures have become of great interest to the scientific and industrial community. However, the controlled fabrication of these nanoscaled systems lies beyond the limits of currently used lithography-based techniques and as such, new growth methods need to be explored. An emerging alternative for the growth of nanostructures is self-assembly. The fundamental processes that govern self-assembly on a substrate are surface migration of adatoms and island nucleation. Extensive studies of these phenomena have led to the development of adequate theories on diffusion and nucleation. In the case of adsorbed atoms diffusing on an atomically flat, inert and defect-free surface, the growth of low-dimensional structures is well described by the conventional nucleation theory (CNT) [1, 2]. This theory, also known as the classical mean-field theory, is essentially defined by atomistic parameters such as the activation energy for diffusion, the binding energy of clusters and the critical nucleus size. Knowledge of these parameters enables a complete description of the growth processes and allows one to predict the island size and density for specifically chosen experimental parameters.

CNT has been applied to numerous cases, in reactive [3]–[5] as well as non-reactive systems [6]–[8]. However, McDaniels et al. reported that the CNT-extracted atomistic parameters in a reactive two-component system (Ti/Si(100)) were not consistent with low-temperature scanning tunneling microscopy (STM) observations [9]. Hence, the model fails to adequately describe the microscopic behaviour for this system, thereby indicating the limits for the application of CNT to reactive systems. Not only the reactivity of the surface plays an important role in the correct interpretation of data, but other surface features, such as defects, surface steps, adatoms, etc may critically influence the surface diffusion and nucleation as well. Therefore, these effects should be carefully considered when modelling experimental results as they are not taken into account by CNT.

In this paper, we discuss the influence of an ultrathin Cu buffer layer on diffusion and nucleation in the reactive Fe/Si(111) system. This Cu layer induces the non-periodic and discommensurate Si(111)-‘5 × 5’–Cu surface structure, which exhibits no dangling bonds and is thermally stable up to 600°C [10]–[12]. As such, we artificially create a silicon surface with reduced reactivity compared with the bare Si(111)-7 × 7 surface, allowing to explore the applicability of CNT to reactive systems. Insight in these diffusion and nucleation mechanisms is indispensable for a better understanding of the complex interaction between adatoms and the surface.

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Figure 1. STM micrographs after deposition of 1 ML Fe on (a) the Si(111)-7 × 7 surface at 300 °C, (b) the Si(111)-'5 × 5'–Cu surface at 300 °C and (c) at 400 °C. The inset shows the remaining Si(111)-'5 × 5'–Cu surface reconstruction, while the black arrow indicates a surface depression (2 V, 0.3 nA).

2. Experimental

Si(111) substrates (FZ, P-doped, 8-12 Ω cm) were cleaned ex situ in a 2% HF solution and in situ using a two-step silicon-flux method (silicon beam clean) [13]. This procedure results in an Si(111) surface, which is free of contaminants and exhibits the well-known Si(111)-7 × 7 reconstruction, as confirmed by in situ Auger electron spectroscopy, STM and reflection high-energy electron diffraction. All substrates used in this work have an unintentional miscut of approximately 0.6° relative to the [111] direction and consequently exhibit surface steps (with a terrace width ranging from 30 to 150 nm). Following the deposition of 1.3 monolayers (ML) Cu (1 ML = 7.83 × 10^{14} at cm^{-2}) at 600 °C, the Si(111)-7 × 7 surface structure transforms to the Si(111)-'5 × 5'–Cu reconstruction. To minimize contamination, subsequent Fe depositions at temperatures between 200 and 600 °C were performed in situ and immediately after the formation of the Si(111)-'5 × 5'–Cu reconstruction. Si, Cu and Fe were evaporated in a conventional molecular beam epitaxy (MBE) set-up with a base pressure of 5 × 10^{-11} Torr. The deposition rate was monitored in situ with a quartz crystal microbalance, which was calibrated using Rutherford backscattering spectrometry and x-ray reflectivity. After Fe deposition the sample was transported in vacuo to the scanning tunneling microscope. All STM measurements were performed at room temperature and recorded in constant current mode.

3. Results and discussion

3.1. Surface diffusion

To investigate the influence of the ‘5 × 5’ reconstruction on the motion of Fe atoms on the surface (diffusion), the creation of an Fe (-Si) cluster (nucleation) and the intermixing with the substrate (reaction), a reference sample is prepared, without Cu. Upon deposition of 1.1 ML Fe at 300 °C onto a clean Si(111)-7 × 7 surface (see figure 1(a)), a rough amorphous surface is observed, covered with small and randomly distributed grains. This morphology can be understood by the high reactivity of the Si(111)-7 × 7 surface with the Fe atoms: due to the large concentration of dangling bonds the mobility of the Fe atoms is drastically
limited. As such, the three major processes, i.e. diffusion, nucleation and reaction, occur quasi-simultaneously and the Fe atoms almost instantaneously bind to the Si surface atoms upon arrival. Furthermore, the initial $7 \times 7$ reconstruction has completely vanished after the Fe deposition, as is indicated by the absence of $7 \times 7$ diffraction spots in a reflection high-energy electron-diffraction pattern taken after Fe evaporation (not shown). The disappearance of the reconstruction causes the surface structure (and thus the surface potential) to change during the diffusion and nucleation. Consequently, these processes are considerably influenced by the reaction between the surface and the diffusing species and an accurate description with CNT becomes impossible.

Depositing the same amount of Fe onto the Si$(111)$-$'5 \times 5'$–Cu surface, under the same experimental conditions, results in the formation of well defined and separated, crystalline CsCl–FeSi$_{1+x}$ nano-islands, which are homogeneously distributed over the surface, as shown in figure 1(b) and discussed in [14]. The Cu overlayer passivates all dangling bonds, thereby significantly reducing the reactivity of the surface and increasing the diffusion length of the subsequently deposited Fe atoms.

Furthermore, the initial Cu-induced `$5 \times 5'$ reconstruction remains intact in between the islands (see figure 1(b) (inset)). The sporadically appearing depressions (indicated by the black arrow in figure 1(b)) originate from Si mass transport from underneath the Cu layer to sustain the silicide formation in the islands. However, the Cu reconstruction is still present in these depressions. As such, the surface structure does not alter during the diffusion process, which demonstrates that the reaction between the Fe atoms and the Si substrate only occurs after the diffusion. If this was not the case, i.e. if reaction would occur while the Fe atoms are diffusing on the surface, traces of this process, such as defects due to missing Si atoms, (local) disappearance of the Cu reconstruction, etc would be visible over the whole surface (rather than merely a few large depressions). The instantaneous reaction would involve either the formation and diffusion of Fe–Si dimers, trimers, etc on top of the Cu layer due to the reaction with thermal Si adatoms (discussed below), or the direct penetration of the Fe atoms into the Cu layer and the subsequent diffusion along the Si–Cu interface. However, firstly, the mobility of clusters is generally much lower compared with monomers and cannot account for the large diffusion lengths observed (for a discussion on cluster mobility see, for instance, [2]). This implies that the nucleation only occurs after the diffusion as well. Secondly, the diffusion along the Si–Cu interface would occur via the vacancy or exchange mechanism, which do not allow long-range migration [15]. Based on these arguments, we argue that the Cu-induced superstructure leads to a decoupling of the diffusion on the surface and the reaction with the surface: upon arrival, the Fe atoms first diffuse on the surface before nucleating and reacting with the Si substrate. Consequently, the Si$(111)$-$'5 \times 5'$–Cu surface largely resembles an inert surface, at least from the diffusion point of view and as such, an important condition to correctly interpret our obtained results within CNT is fulfilled.

3.2. Island nucleation

In course of the diffusion process, the Fe adatoms might encounter each other and as such form a nucleus on the surface. The size of the cluster determines whether it forms a stable island or decays to a smaller cluster. The threshold is defined by the concept of the critical nucleus size $i$. It defines the largest unstable cluster which will, upon addition of one extra atom, render stable. Within the framework of CNT, the saturation density of stable islands $n_s$ for two dimensional
(2D) island growth in the complete condensation regime is given by

\[ n_x = \eta \left( \frac{E}{D_0} \right)^{(i+2)/i} \exp \left( \frac{E^*}{kT} \right). \]  

(1)

In this equation, \( \eta \) represents a constant dimensionless number near 0.1 containing the coverage dependence (see figure 6(c) in [1]), \( F \) is the deposition flux, \( D_0 \) is the surface diffusion prefactor, \( i \) is the critical nucleus size, and \( E^* = (i E_d + E_b)/(i + 2) \) is the effective diffusion barrier, which is a weighted sum of the diffusion activation energy \( E_d \) and the critical cluster binding energy \( E_b \), and essentially determines the diffusion over the surface. The energy parameter \( E^* \) is experimentally accessible from the slope of the linear relationship between \( \ln(n_x) \) and \( 1/kT \), while \( i \) can be obtained either using the \( 1/T = 0 \) intercept of the Arrhenius plot or the rate dependence of the island density. Consequently, knowledge of the atomistic parameters \( E^* \) and \( i \) provides insight in the microscopic diffusion and nucleation mechanisms on the surface. For island nucleation in the Fe/Si(111)-5'×5'–Cu system, the temperature dependence of the island density is plotted in figure 2(a) for temperatures between 200 and 500 °C. It is clear that for temperatures below 350 °C, the dependence of the island density agrees very well with the predicted exponential behaviour, while for temperatures above 350 °C, considerable deviations from equation (1) are observed.

For the low-temperature regime, a fit to equation (1) is included in figure 2(a), which yields the effective diffusion barrier \( E^* = 0.49 \pm 0.05 \) eV and the critical nucleus size \( i = 1.85 \pm 0.25 \) (using the known flux \( 0.017 \) ML s\(^{-1} \), and nominal values \( D_0 = 10^{13} \) s and \( \eta = 0.1 \) [1, 2]. In figure 2(c), the island density is plotted as a function of the Fe deposition rate at a fixed temperature (300 °C), illustrating the power law dependence, \( n_x \sim F^{i/2} \). The slope of the fit (solid line) compares best with the trend line for \( i = 2 \), which is consistent with our previously determined value.

To fully validate the model for our system, we have investigated the temperature dependence of the island density at a higher Fe flux (0.037 ML s\(^{-1} \)) as well. The results are presented in figure 2(b). The fit to the data provides the effective energy barrier \( E^* = 0.49 \pm 0.07 \) eV and the critical nucleus size \( i = 1.81 \pm 0.40 \). Both values are in very good agreement with our previous values and demonstrate the validity and consistency of our model to this reactive three-component system. Based on our results for the critical nucleus size and the fact that \( i \) represents a number of atoms—and hence an integer number—, we can conclude that \( i = 2 \), implying that a three-atom cluster forms a stable nucleus on the surface. With this value, the weighted contributions in \( E^* \) can be disentangled. The expression for the effective diffusion barrier becomes \( E_d + \frac{1}{2} E_2 = 2 E^* = 0.98 \pm 0.10 \) eV, with \( E_2 \) the binding energy of a two-atom cluster. In the following sections, we will elucidate the physical meaning of \( E^* \) and \( i \).

3.2.1. Critical nucleus size \( i \). In the Fe/Si(111)-5'×5'–Cu system, there are two (possible) moving species on the surface: the deposited Fe atoms and thermal Si adatoms, which are provided by the depressions and step edges. The Cu atoms do not enter this discussion as they provide a stable and chemically inert surface. Consequently, either of the species, or both, may contribute to the critical nucleus. This means that the critical nucleus can be (i) an Si–Si, (ii) an Fe–Si or (iii) an Fe–Fe cluster.

First of all, if the critical nucleus was made up solely by Si atoms, the island density would not vary with the Fe deposition rate. As we have demonstrated the opposite (figure 2(c)), this hypothesis is invalid.
Figure 2. (a) Temperature dependence of the stable island density after deposition of 1.1 ML Fe on the Si(111)-5×5–Cu surface (flux = 0.017 ML s$^{-1}$). The solid line represents the best fit to equation (1) for $T \leq 350$ °C. (b) Same dependence as in (a) for a higher Fe flux (flux = 0.037 ML s$^{-1}$). (c) Fe flux dependence of the island density at 300 °C. The dashed lines indicate $i = 1, 2$ and 3, while the solid line is a fit to the data.

Secondly, we consider the possibility that both species contribute to the critical nucleus, i.e. an Fe–Si cluster. To investigate whether this is reasonable, we have plotted the island volume as a function of deposition rate at a fixed temperature (300 °C) in figure 3: a significant decrease in volume is observed at high evaporation rates. This decrease can only be explained by a
lower Si content inside the islands, as the same amount of Fe is deposited. This is already a first, strong indication that the Si adatom coverage is too low to supply enough Si to the islands at high Fe fluxes. In the literature, the thermal Si adatom release rate is found to be only 0.01 ML s$^{-1}$ at 500 °C for Si/Si(100) [9, 16, 17]. Despite the different system (Si/Si(111)–Cu), this still provides an upper limit as we consider a temperature of only 300 °C. Consequently, this value is well below the Fe evaporation rates of 0.06–0.09 ML s$^{-1}$ at which the island volume decreases and underlines the rate limiting of the Si adatoms at these high Fe fluxes. Now, if the thermal adatoms would contribute to the critical nucleus, the island density at high evaporation rates (0.06–0.09 ML s$^{-1}$) would saturate as the nucleation is no longer limited by the Fe evaporation rate but rather by the supply of Si atoms. In other words, the probability for incorporation into an existing island would become much larger than the probability for nucleating a new island, which would result in a saturation of the final island density. This is definitely not the case as evidenced in figure 2(c). Considering the obtained expression for the activation energy, $E_d + \frac{1}{2}E_2 = 2E^* = 0.98 \pm 0.10$ eV, the binding energy of a free Fe–Si cluster, $E_2 = 1.93$ eV [18] would also cause the activation energy for surface diffusion to become quasi-zero. This would imply that even for slightly elevated temperatures all the islands nucleate at the step edges. As full step decoration is only observed above 450 °C (see figure 4), a critical nucleus consisting of Fe–Si is very unlikely.

Moreover, if the Si adatoms would contribute to the critical nucleus, the temperature dependence of the island density for different Fe rates would not be identical, since the ratio between the Fe flux and the Si flux has changed. The contribution of Si atoms to the critical nucleus would also be noticeable in the Arrhenius plot of the density. As the Si adatom flux is thermally activated, this would tend to flatten the ln($n_s$) versus 1/kT plot at high temperatures. Both effects are not observed and consequently, these are two experimental indications that the critical nucleus is solely made up Fe atoms. The role of the Si atoms, whether they participate in the critical nucleus or just attach when needed, can be experimentally determined by varying the Si flux. By co-depositing Fe and Si onto the Cu-covered Si surface, we artificially increase the Si adatom density on the surface and, if Si is a contributing species, drastically influence the island nucleation (i.e. induce a strong increase in island density). However, the resulting surface morphology is not significantly different from

Figure 3. Island coverage as a function of Fe deposition rate.
Figure 4. Fraction of islands nucleated at the step edge (left scale) and mean island height (right scale) as a function of deposition temperature (flux = 0.017 ML s$^{-1}$).

our previous depositions (without additional Si) and the density of islands on the surface has not increased: $3.8 \pm 0.6 \times 10^{-3}$ nm$^{-2}$ (extra Si) versus $4.6 \pm 0.5 \times 10^{-3}$ nm$^{-2}$ (no extra Si). This observation directly proves that Si does not contribute to the critical nucleus.

Altogether, we can strongly state that the Si atoms are merely required to sustain the silicide growth upon nucleation and do not contribute to the critical nucleus. By elimination we are left to conclude that the critical nucleus is an Fe–Fe cluster, which is supported by the good agreement of the observed nucleation behaviour with the nucleation theory.

3.2.2. Activation energy $E_d$. Considering the fact that the critical nucleus $i$ is a two-atom Fe cluster, $E_d$ represents the activation energy for pure Fe diffusion on the Si(111)-‘5 × 5’–Cu surface. In the literature, one can find experimental values for the binding energy of a free Fe$_2$ cluster; $E_2 = 1.14$ eV [19]. Although these are values for clusters in the gas phase and hence neglect the influence of the surface, they give at least a good indication as the surface is considered to be inert. Filling out these values yields an activation energy $E_d$ of 0.41 eV, which is of the same order as the literature values for surfactant-mediated growth on Si(111) [20].

Consequently, island nucleation in the Fe/Si(111)-‘5 × 5’–Cu system follows the CNT scaling laws well at temperatures below 350 °C, with a single value for $E^* = 0.49$ and for $i = 2$. Moreover, as the critical nucleus solely consists of Fe-atoms, we can conclude that the reaction with the substrate only occurs after the diffusion and the nucleation. For comparison, the experimental values for the critical nucleus size and diffusion barriers for Fe on the bare Si(111)-7 × 7 surface can be found in the literature. Reshöft [4] reported a total diffusion barrier $E^* = 0.81$ eV and a critical nucleus size $i = 5$, while Wohllebe et al [21] published $E^* = 0.86$ eV and $i = 3$. The effective energy barrier $E^* = 0.49$ eV determined in our study for Fe on a Si(111)-‘5 × 5’–Cu surface is considerably lower, which is consistent with the observed enhanced diffusion on the Cu-passivated surface. However, since there is no decoupling of the island nucleation and the reaction with the substrate in the Fe/Si(111) system, one has to be extremely careful when interpreting and comparing with these literature data. They are determined using a theory developed for a
non-reactive surface on the (highly) reactive Si(111)-7×7 surface, which might compromise their validity. For a fully quantitative comparison, the reliable values for Fe diffusion on Si(111)-7×7, that are currently unavailable, are essential. Still, the obtained values for the effective diffusion barrier \( E^* \) and the critical nucleus size \( i \) are of major importance since they determine the microscopic diffusion and nucleation mechanisms on the passivated surface.

At temperatures above 400 °C, the temperature dependence of the island density deviates from the predicted behaviour as illustrated by figure 2(a). The STM measurement in figure 1(c) after deposition of 1.1 ML Fe at 400 °C gives a good indication of the origin of this anomalous behaviour: lots of islands preferentially nucleate at the step edges. In figure 4, the fraction of islands nucleated at a step edge is quantified as a function of temperature. At 400 °C and above, a large (and increasing) fraction of the islands nucleate at the (lower) step edge, indicating that the diffusion length exceeds the mean terrace size at these temperatures. This effect is also reflected in the density versus inverse temperature plot in figure 2(a): at 400 °C, a small increase in island density is observed with increasing temperature due to presence of an extra diffusion barrier on the surface (the atoms are forced to nucleate at the step edge although their diffusion length is larger than the terrace size). As the temperature further increases, the diffusing atoms have sufficient energy to overcome this barrier and the island density decreases again. Consequently, the diffusion and nucleation processes are drastically influenced by the presence of surface steps, causing non-homogeneous island nucleation, whereas CNT is solely derived for homogeneous nucleation. It should be mentioned that preferential step edge nucleation is observed at \( T \leq 350 \) °C as well. However, a step decoration of 26.25% (at 350 °C) has only a minor influence on the resulting island density, i.e. a decrease from \(-8.1 \) to \(-8.2 \) in figure 2(a)\(^2\). Moreover, the mean island height rapidly increases with increasing temperature as well, as presented in figure 4. Between 350 °C (0.43 nm) and 400 °C (0.61 nm), the average island height becomes larger than the lattice parameter of the CsCl–FeSi\(_{1+x}\) structure (0.55 nm), indicating a shift from 2D to 3D growth. Equation (1), however, is derived for pure 2D growth and as such, for a correct interpretation at high temperatures, we need a 3D alternative for equation (1) [1]. Consequently, the failure of CNT to correctly describe the behaviour at 400 °C and above can be mostly attributed to the presence of surface steps. For a correct interpretation of our results an extension of the nucleation theory is required, which takes into account this phenomenon. This can be done by considering an additional place-dependent energy barrier for surface diffusion.

4. Conclusions

The presence of a thin Cu buffer layer on a Si(111) surface has a significant influence on the surface morphology of a subsequently deposited amount of Fe. Not only does the Fe diffusion on the Si(111)-5×5–Cu surface strongly increase compared with the bare Si(111)-7×7 surface, the surface passivation also causes a decoupling of the diffusion on the surface and the reaction with the surface. Due to the Cu layer, CNT can be successfully applied to describe the island nucleation in the reactive Fe/Si(111) system below 350 °C, yielding a single value for the effective diffusion barrier \( E^* = 0.49 \) eV and the critical nucleus size \( i = 2 \). It is evidenced that the critical nucleus solely consists of Fe atoms and that the thermal Si adatoms are merely

\(^2\) To calculate the difference, we assume that, due to the presence of the step, twice as many islands have nucleated in an area of one diffusion length around the step.
required to sustain the silicide formation. These parameters provide insight in the diffusion and nucleation mechanism and allow one to predict the island density, the island size and the island height for a given deposition temperature and rate, which is of major importance in controlling nanostructure growth.

Above 400 °C, however, the growth kinetics change considerably: a transition from 2D to 3D growth and step flow growth are observed. An extension to nucleation theory that takes into account the presence of surface steps is necessary to correctly describe the diffusion and nucleation processes at these high temperatures.

Obviously, for the low-temperature results, the decoupling of diffusion and reaction is essential to determine the effective diffusion barrier and the critical nucleus size using CNT on surfaces where reaction can occur. The determination of these kinetic parameters is crucial for a fully quantitative study of the influence of surface structures on surface diffusion and nanoparticle formation. Using the Fe/Si(111)-'5 × 5'–Cu system as an example, we have demonstrated that such decoupling can be achieved by using an ultra thin buffer layer. It is expected that these results can be generalized to a wide variety of other systems.

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