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## Surface thermomigration of 2D voids

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In a thermal gradient, surface nanostructures have been experimentally observed to move by thermomigration. However, analytical models that describe the thermomigration force acting on surfaces are still controversial. In this work we start from the thermodynamic approach based on the Massieu function, used to describe thermomigration of single adatoms, to develop an expression of the velocity of thermomigrating 2D holes. The model can be simplified in two limiting cases: (i) when the hole motion is limited by adspecies diffusion, the velocity is independent from the hole size (as in our experiments). (ii) If the hole motion is limited by the attachment or detachment of species to/from steps, then the velocity is proportional to the hole width. We have studied by low energy electron microscopy the thermomigration of 2D monoatomic deep holes on Si(100). From the velocity measurements taken at different temperatures we find, using our model, that the sum of the migration energy and of the concentration energy is  $1.95 \pm 0.16$  eV. This value is consistent with those found by other authors and reinforce the validity of our thermomigration model.

Thermal gradients are known to affect atomic diffusion in multicomponent materials. This phenomenon, known as “Soret effect”, thermophoresis, or thermomigration (TM), has been studied since long time in gases and liquids<sup>1,2</sup>. The main known effect is that homogeneous mixtures in a thermal gradient become inhomogeneous, because the different species (atoms, molecules or particles of different kind) respond differently to the thermal gradient. The effect of TM in bulk materials, where the atomic displacement takes place preferentially along grain boundaries or by diffusion of vacancies<sup>3-5</sup>, has also been studied, especially in alloys used for soldering, where the segregation of elements due to thermal gradients should be avoided<sup>6</sup>. In nanomaterials, weak thermal gradients can lead to strong effects because of the small amount of matter, and of the importance of surface effects. TM is usually considered a detrimental effect as it can lead to the failure of microelectronic devices. In this perspective, a better understanding of how thermal gradients affect surfaces could help to develop devices less affected by TM. However, TM could also be used to move and shape nanostructures to design material surfaces. For instance, it has been reported that thermal gradients may lead to direct mass transport along nanotubes<sup>7</sup> or nanowires<sup>8</sup>.

Thermal mass transport also takes place in pure bulk materials, by displacement of vacancies and interstitials. As summarized by Huntington<sup>9</sup>, the driving forces for the thermomigration of a vacancy in a bulk material are three: (i) an intrinsic effect, where a net mass flow results from a change of atomic jump rates in a sequence of planes perpendicular to the thermal gradient, as detailed in<sup>3,10,11</sup>; (ii) a phonon contribution due to scattering of phonons with mobile atoms; and (iii) the scattering of charge carriers (via a thermoelectric effect) with mobile atoms. Within a commonly accepted framework to address TM in bulk materials, a thermal gradient implies an effective force  $F$  on diffusing species:  $F = -\frac{Q^*}{T} \frac{dT}{dx}$ , where  $T$  is

the temperature,  $\frac{dT}{dx}$  is the thermal gradient and  $Q^*$  is a scalar called heat of transport<sup>12</sup>. Under this force, atomic diffusion is biased. According to the sign of  $Q^*$ , the atomic motion can be in the same direction of the thermal gradient or against it.

We focus on the effect of thermomigration at surfaces of single component materials. The drift velocity of a single adatom in a thermal gradient has been recently modeled by Roux and Combe<sup>13</sup> for a system of about 25000 atoms. Their model perfectly reproduces adatom trajectories simulated by molecular dynamics.  $Q^*$  is found to depend mainly on the adatom/substrate binding energy and weakly on the migration energy  $E_m$  (the energy necessary for the diffusion of an isolated adatom). The interplay between the adatom migration mechanism and the velocity and shape of large migrating nanostructures made by many atoms is far from being trivial. A possible description of the motion of a surface cluster in a thermal gradient is based on considering thermomigration as a force, and a constant diffusion coefficient, as done in ref.<sup>14</sup>. In this work, based on the improved description of thermomigration of single adatoms developed by Roux and Combe, we reconsider the expression of the thermomigrating force exerted on a 2D monolayer hole. We then show our experimental observations of the motion of 2D surface holes on Si(100) under thermomigration and use the model to extract important physical parameters for the adatom diffusion on Si(100).

Our model is based on the Burton, Cabrera and Franck theory of atomic transport at surfaces<sup>15</sup>. The probability to find an adatom in a position  $x_0$  is  $p(x_0) \propto \exp(-\Phi(x_0))$ , with  $\Phi(x_0)$  a state function which is a non-dimensional thermodynamic potential. In a system with homogeneous temperature  $T$ , the thermodynamic potential  $\Phi(x)$  would be  $A(x_0)/(kT)$ , with  $A(x_0)$  the free energy:  $A(x)/(kT)$  is a Massieu function, the Legendre transform of the entropy. Roux and Combe<sup>13</sup> have shown that when the temperature is inhomogeneous, the thermodynamic potential  $\Phi(x)$  is the sum of a function that slowly increases with temperature, and an oscillatory function due to the diffusion potential. More precisely, using a simple sinusoidal potential to account for these oscillations, we write

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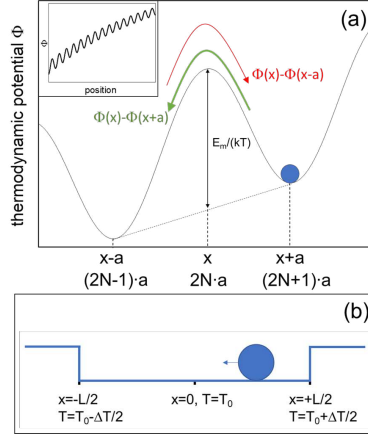


FIG. 1. (a): This graph is a qualitative sketch of the thermodynamic potential of an adatom as a function of the surface position in a thermal gradient. The temperature increases from left to right. An adatom jumping from right to left has to overcome a thermodynamic potential equal to  $\Phi(x) - \Phi(x+a)$ , while for a jump from left to right the barrier is  $\Phi(x) - \Phi(x-a)$ . The inset shows a larger view of the thermodynamic potential, traced with the expression of Roux and Combe. (b): schematics of a hole in a thermal gradient, the reference with  $x=0$  and the temperature  $T = T_0$  is taken in the center of the hole.

$\Phi(x) \approx \frac{-Q}{kT(x)} + \frac{E_m}{kT(x)} \frac{\cos(\frac{\pi x}{2}) + 1}{2}$ . The position of the relative maxima are  $x = 2Na$  with  $N$  integers.  $T(x)$  is the temperature at  $x$ ,  $Q$  is a quantity that characterizes the dependence of the thermodynamic potential on the temperature, Roux and Combe showed that it depends on the adatom-substrate binding energy. The relation between  $Q$  and the heat of transport  $Q^*$  will be addressed later;  $k$  is the Boltzmann constant,  $E_m$  is the adatom migration energy, and  $a$  is half a lattice constant ( $a = \lambda/2$ ). A schematics of the energy profile felt by adatoms in a thermal gradient is shown in figure 1(a). In the figure, the thermal gradient is aligned with the  $x$  axis (warmer regions at high  $x$ ). Notice that, with the expression above, the diffusion contribution to the thermodynamic potential depends on the position via  $T(x)$ .

Our purpose is now to describe the displacement of a 2D surface hole under a thermal gradient. In this case adatoms are generated by detachment from an edge, they diffuse inside the hole and attach to the opposite edge. We suppose an anisotropy between the exterior and the interior of the hole: this assumption is related to the presence of a Schwoebel barrier at the hole edges<sup>16</sup>. Without this anisotropy it would not be possible to observe a displacement of the hole because atomic exchanges outside the hole would be compensated by atomic exchanges inside the hole<sup>17</sup>. Similarly to the approach used by Brinkman, Shockley and Leclerc<sup>3,10,11</sup>, we

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write the diffusive flux  $J_{\text{diff}}$  of adatoms as the difference between the rate of atomic jumps from left to right multiplied by the adatom concentration on the left (position  $x-a$ , flux  $j_{\rightarrow}$ ) and the rate of atomic jumps from right to left multiplied by the adatom concentration on the right (position  $x+a$ , flux  $j_{\leftarrow}$ ):

$$J_{\text{diff}} = j_{\rightarrow} - j_{\leftarrow} = \lambda v \exp[-(\Phi(x) - \Phi(x-a))] \cdot n(x-a) - \lambda v \exp[-(\Phi(x) - \Phi(x+a))] \cdot n(x+a)$$

$x$  is the position of a relative maximum in the thermodynamic potential (see figure 1a);  $v$  is an attempt frequency taken equal for all jumps,  $n(x-a)$  and  $n(x+a)$  are adatom concentrations in the lattice positions  $x-a$  and  $x+a$ . Writing  $T(x+a) = T(x) + \frac{dT}{dx} \cdot a$  and  $T(x-a) = T(x) - \frac{dT}{dx} \cdot a$ , we obtain (with a first order approximation because we consider small and constant thermal gradients, see the development in the supplementary material):

$$J_{\text{diff}} = -\lambda^2 v \exp\left(-\frac{E_m}{kT(x)}\right) \left[ \frac{n(x) \cdot Q}{kT(x)^2} \cdot \frac{dT}{dx} + \frac{dn}{dx} \right] = -D(x) \frac{dn}{dx} - \frac{D(x)n(x)}{kT(x)} \frac{Q}{T(x)} \frac{dT}{dx} \quad (1)$$

Where  $D(x) = D_0 \exp\left(-\frac{E_m}{kT(x)}\right)$ , with  $D_0 = v\lambda^2$ , depends on the position inside the hole  $x$ . The expression for the flux of equation 1 results as a sum of a term  $(-D \frac{dn}{dx})$  implying the concentration gradient and a drift term  $\frac{FDn}{kT}$  under the effect of an effective force  $F = -\frac{Q}{T} \frac{dT}{dx}$ , and thus  $Q^* = Q$ . Other authors simply considered  $J = -\frac{d(D(x)n(x))}{dx} = -D(x) \frac{dn(x)}{dx} - n(x) \frac{dD(x)}{dx}$  which gives  $J = -D(x) \frac{dn}{dx} - \frac{D(x)n(x)}{kT(x)} \frac{E_m}{T(x)} \frac{dT}{dx}$  (see as an example<sup>18</sup>). Therefore, these authors identified  $Q^* = E_m$ , while with the more thorough approach of Roux and Combe  $Q^*$  does not depend on  $E_m$  but is related to the binding energy  $Q$ <sup>13</sup>.

Equation 1 can be rewritten as:

$$\frac{dn}{dx} + \frac{n(x) \cdot Q}{kT^2(x)} \cdot \frac{dT}{dx} = -\frac{J_{\text{diff}}}{D_0} \exp\left(\frac{+E_m}{kT(x)}\right) \quad (2)$$

In steady state, i.e. under the assumption of a flux  $J$  constant inside the hole of size  $L$  and at its edges, solving the first order inhomogeneous differential equation 2 (see supplementary material and figure 1(b)), we find:

$$J_{\text{diff}} = J = D_0 \frac{n(-L/2) \exp\left(\frac{-Q}{kT(-L/2)}\right) - n(L/2) \exp\left(\frac{-Q}{kT(L/2)}\right)}{\int_{-L/2}^{+L/2} \exp\left(\frac{E_m - Q}{kT(s)}\right) ds} \quad (3)$$

For small thermal gradients we find (see supplementary material):

$$\int_{-L/2}^{+L/2} \exp\left(\frac{E_m - Q}{kT(s)}\right) ds = \frac{2kT_0^2}{(E_m - Q) \frac{dT}{dx}} \exp\left(\frac{E_m - Q}{kT_0}\right) \sinh\left(\frac{(E_m - Q)\Delta T}{2kT_0^2}\right)$$

with  $\Delta T = T(L/2) - T(-L/2)$ . Considering the concentrations at the hole edges close to the equilibrium concentrations  $n_{eq}$ , as  $T(-L/2) < T(L/2)$  and  $n_{eq}(x) = n_0 \exp\left(\frac{-E_m}{kT(x)}\right)$ ,

where  $E_c$  is the adatom creation energy from an edge, then  $n(-L/2) < n(+L/2)$ , and since  $Q$  is positive because it corresponds to an attraction between the adatom and the substrate, the flux is negative, i.e. opposite to the thermal gradient. Notice that, in absence of a thermal gradient, equation 3 reduces to the normal diffusion equation, where the flux is determined by the concentration gradient. Therefore, atoms detach from the right hole edge (hot), diffuse inside the hole and attach to the left hole edge (cold). In order to consider the difference between  $n(\pm L/2)$  and  $n_{eq}(\pm L/2)$ , we follow the literature<sup>19-22</sup> and we introduce the attachment flux  $J_{att}$  and the detachment flux  $J_{det}$ . These fluxes depend on a kinetic

coefficient of attachment/detachment  $k_{\pm L/2}$  in  $x = \pm L/2$ , and from the difference between the real adatom concentration and the equilibrium concentration at the hole edges:

$$J_{att} = J = -k_{-L/2}(n(-L/2) - n_{eq}(-L/2)) \quad (4)$$

$$J_{det} = J = -k_{L/2}(n_{eq}(+L/2) - n(+L/2)) \quad (5)$$

Notice that the kinetic coefficients could also be written as the reciprocal of characteristic times  $\tau$  of attachment and detachment. As the detachment flux, the diffusive flux and the attachment flux are equal, we evaluate  $n(\pm L/2)$  from equations 4 and 5 and reintroduce them in equation 3 to finally get:

$$J = \frac{-2 \cdot n(T_0)D(T_0) \sinh\left(\frac{(E_c+Q)\Delta T}{2kT_0^2}\right)}{\frac{2kT_0^2}{(E_m-Q)\frac{dT}{dx}} \cdot \sinh\left(\frac{(E_m-Q)\Delta T}{2kT_0^2}\right) + D(T_0) \cdot \left[\frac{1}{k_{-L/2}} \exp\left(-\frac{Q\Delta T}{2kT_0^2}\right) + \frac{1}{k_{L/2}} \exp\left(+\frac{Q\Delta T}{2kT_0^2}\right)\right]} \quad (6)$$

If also  $\frac{Q\Delta T}{2kT_0^2}$ ,  $\frac{(E_c+Q)\Delta T}{2kT_0^2}$  and  $\frac{(E_m-Q)\Delta T}{2kT_0^2}$  are small, then we can further simplify<sup>23</sup>:

$$J \approx \frac{-2 \cdot D(T_0)n(T_0)\frac{(E_c+Q)\Delta T}{2kT_0^2}}{L + D(T_0) \cdot \left[\frac{k_{L/2} + k_{-L/2} - \frac{Q\Delta T}{2kT_0^2}(k_{L/2} - k_{-L/2})}{k_{-L/2} \cdot k_{L/2}}\right]}$$

$$J \approx \frac{-n(T_0)\frac{(E_c+Q)}{kT_0^2}\frac{dT}{dx}}{\frac{1}{D(T_0)} + \frac{1}{L \cdot k_{-L/2}} + \frac{1}{L \cdot k_{L/2}}} \quad (7)$$

As we consider constant thermal gradients,  $\frac{dT}{L}$  is equal to the thermal gradient  $\frac{dT}{dx}$ . Equations 6-7 are our main results.

If diffusion is the limiting process ( $\frac{D(T_0)}{L} \ll k_{-L/2}$  and  $\frac{D(T_0)}{L} \ll k_{L/2}$ ), which always occurs for large holes, the two last terms of the denominator can be neglected. In this case the expression is similar to that developed in<sup>14</sup>, but instead of a migration energy  $E_m$ , a quantity related to the binding energy ( $Q$ ) appears in the numerator of equation 7. In this case the flux and thus the velocity of the hole do not depend on the hole size. For small holes or slow attachment/detachment kinetics, the first term of the denominator in 7 is neglected and the flux  $J$  is directly proportional to the hole size. These findings are consistent with those obtained under an electromigration force<sup>24</sup>.

In order to study in real time the displacement of 2D clusters on a surface we have used low energy electron microscopy (LEEM) and Si(100) samples. Other authors have observed surface thermomigration phenomena of 3D droplets, without selecting the thermal gradient direction<sup>25-27</sup>. We have modified a LEEM sample holder by displacing the W heating filament on one side to heat that side of the sample more than the opposite one<sup>(28, see supplementary material)</sup>. Furthermore, a Mo plate with a hole on the side corresponding to

the filament allows good contact between the sample and the sample holder on the opposite side with respect to the heating filament. With such a sample holder, by pyrometry, we have measured a thermal gradient that increases with temperature from of  $1 \cdot 10^4$  K/m at 1100 K to  $1.7 \cdot 10^4$  K/m at 1240 K. The absolute temperatures are referred to the center of the sample where the LEEM movies are taken. Notice that we cannot change the thermal gradient without changing the overall temperature. The samples are positioned to have the thermal gradient corresponding to the [011] direction. The surfaces of the samples are prepared by successive flashes at  $\approx 1500$  K to obtain flat terraces with diameter of up to  $10 \mu\text{m}$ . The sample temperature is increased to obtain evaporation of Si atoms and formation of holes with mono-atomic depth in the terraces. Then, the temperature is decreased and the evolution of the holes in the thermal gradient is observed by LEEM in real time. The experiments are performed at temperatures higher than 1100 K to observe the displacement of the holes in reasonable times. The holes displace in the thermal gradient differently according to the orientation of the dimer rows inside and outside the hole. The effect of a force on the displacement of 2D holes and islands on Si(100) has been detailed in<sup>29</sup>, where the force was obtained with an electric field, and a short summary is given in the supplementary material. From the displacement of holes as a function of time (see figures 2a, b and c), we have measured the hole velocity at several temperatures and thermal gradients. Experimentally, we have not been able to establish a critical size  $L$  for the transition between the attachment-detachment limited regime and the diffusion limited regime: the velocity measurements are very delicate as the holes move very slowly in our relatively low thermal gradients and the hole sizes change by evaporation and by Ostwald ripening during the experiments. In a previous study of hole motion on Si(100) under electromigration, it was found that surface diffusion is the process limiting displacement<sup>29</sup> for monoatomic deep holes with sizes com-

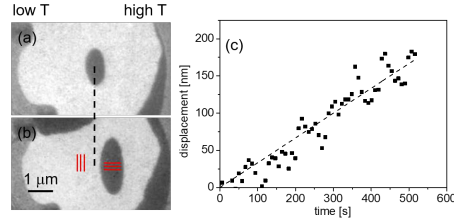


FIG. 2. (a-b) LEEM tilted bright field image of the displacement of a hole with dimer rows parallel to the thermal gradient towards the hot side. Electron energy: 6 eV, temperature: 1200 K. The shape of the holes is defined by the dimer rows outside the terrace: the holes are elongated along the axis of the dimer rows outside the hole. From the elongation of the hole is thus possible to know the orientation of the dimer rows inside and outside the holes. The direction of the dimer rows inside and outside the hole is shown by the red lines. (c) Position of the center of mass of a hole as a function of time.

parable to those of the present study, therefore we consider that in our thermomigration experiments the motion is diffusion limited. The holes studied in this work have micromet-

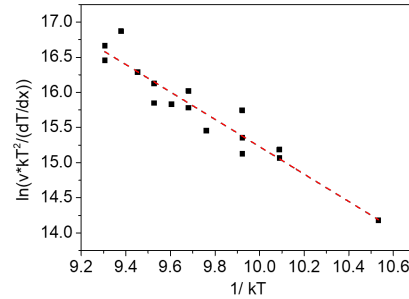


FIG. 3. Natural logarithm of the velocity multiplied by  $kT^2$  and divided by the thermal gradient, as a function of  $1/(kT)$ . From the linear fit through the data points we obtain  $E_m + E_c = 1.95 \pm 0.16$  eV.

ric sizes, the temperature difference between the back and the front hole edges is in the order of  $10^{-2}$  K at 1100 K, thus the approximations used in the equations above are valid. The 2D holes displace with velocities varying between 0.14 (at 1100 K) and 1.3 (at 1220 K) nm/s. Considering a diffusion-limited regime, as the hole velocity is:

$$v = J \cdot \lambda^2 = \lambda^2 \cdot n_0 D_0 \exp\left(-\frac{E_c + E_m}{kT}\right) \cdot \frac{(E_c + Q)}{kT^2} \frac{dT}{dx} \quad (8)$$

we plot  $\ln\left(\frac{v \cdot kT^2}{dT/dx}\right)$  as a function of  $\frac{1}{kT}$  in figure 3. The uncertainty on the intercept with the y axis for  $1/(kT) = 0$  is

too high to determine  $E_c + Q$ . From the slope of the linear fit through the data points, we obtain  $E_m + E_c = 1.95 \pm 0.16$  eV, that confirms the  $E_m + E_c$  values already reported in the literature for Si(100) (about 2 eV in<sup>29</sup> and<sup>30</sup>, 2.3 eV in<sup>31</sup>). As the  $E_m + E_c$  value that we find aligns with that found in other works, the reliability of our thermomigration model is further supported.

In order to find  $E_c + Q$ , we now consider electromigration experiments. In ref.<sup>29</sup>, we have studied the displacement of 2D holes and islands on the Si(100) surface moving by electromigration, under the effect of an electric field. In that case the velocity of surface nanostructures was  $v_{EM} = \lambda^2 \cdot n_0 D_0 \exp\left(-\frac{E_c + E_m}{kT}\right) \frac{Z^* e E}{kT}$ , with  $Z^*$  an effective valence,  $e$  the electron charge and  $E$  the electric field. As done in<sup>14</sup>, we now evaluate the ratio between the heat of transport  $E_c + Q$  and the effective valence  $Z^*$ :

$$\frac{E_c + Q}{Z^*} = \frac{v_{TM} \frac{kT^2}{dT/dx}}{v_{EM} kT \frac{1}{eE}} \quad (9)$$

where TM and EM stand for thermomigration and electromigration respectively. From the linear fits of  $\ln\left(\frac{v_{TM} \cdot kT^2}{dT/dx}\right)$  and  $\ln\left(v_{EM} \cdot \frac{kT}{eE}\right)$  as a function of  $\frac{1}{kT}$ , we obtain  $\frac{E_c + Q}{Z^*} = 4.3 \pm 0.3$  eV. Using the effective valence suggested by Stoyanov<sup>32</sup> for Si(100),  $Z^* \approx 1$ , we find  $E_c + Q \approx 4.3$  eV. The order of magnitude is reasonable, however as  $Z^*$  is only roughly estimated, the uncertainty on  $E_c + Q$  is high.

To summarize, in this study we have developed an expression to describe the thermomigration of 2D monoatomic deep holes on surfaces, starting from a recently developed description of thermomigration of single adatoms. When the process limiting the hole displacement is the diffusion of adspecies, the model predicts that the hole velocity does not depend on the hole size. In the regime where the hole motion is limited by attachment and detachment of adspecies at the hole edges, the velocity is proportional to the hole width. Studying the displacement in a thermal gradient of monoatomic deep holes on Si(100), we find that the sum of the concentration energy and of the migration energy is  $1.95 \pm 0.16$  eV. This value is similar to those found on the same system by other authors and thus supports the reliability of our model.

#### SUPPLEMENTARY MATERIAL

The supplementary material includes a figure of the LEEM sample holder used for thermomigration experiments, a summary of the mechanism of displacement of 2D holes and islands on Si(100), the derivation of the diffusive flux starting from the thermodynamic potential used by Roux and Combe<sup>13</sup>, the solution of the differential equation (equation 2 in the text), and the approximation of the integral appearing in the right hand term of equation 3.

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#### DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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