CRYSTAL GROWTH IN SOLUTION
A VOLUME DIFFUSION PROBLEM

STAGNANT LAYER

\[ d \approx 5 \sqrt{\frac{x \eta}{V \rho}} \]

\( \eta \) - viscosity, \( \rho \) - fluid density,
\( T = 300K \), \( \eta = 0.01 \) g/cm sec,
\( \rho = 1 \) g/ml, \( V = 40 \) cm/sec,
\( x = 0.1 \) cm, \( d \approx 0.25 \) mm

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RATE OF ADVANCE OF A SINGLE STEP

\[ C(r) = C_\infty - (C_\infty - C_{st}) \cdot \frac{\ln \left( \frac{r}{d} \right)}{\ln \left( \frac{a}{\pi d} \right)} \]

\[ \nu_\infty = \nu_c D \left( \frac{dC}{dr} \right)_{r = \frac{a}{\pi}} = \chi \sigma \frac{\pi \nu_c DC_0}{a \ln \left( \frac{\pi d}{a} \right)} \]

\( \nu_c \) – molecular volume of crystal

\( D \) - bulk diffusion coefficient

\( C_0 \) – equilibrium concentration

at a temperature \( T \)

\[ \sigma = \frac{C_\infty}{C_0} - 1 \] - supersaturation in bulk

\[ \sigma_{st} = \frac{C_{st}}{C_0} - 1 \] - supersaturation at step

Solution:

\[ \frac{d^2 C}{dr^2} + \frac{1}{r} \frac{dC}{dr} = 0 \]

Boundary conditions:

\[ r = \frac{a}{\pi} \rightarrow C = C_{st} \]

\[ r = d \rightarrow C = C_\infty \]
\[ v_\infty = \frac{\beta_{st} \sigma C_0 v_c}{1 + \frac{a \beta_{st}}{\pi D} \ln \left( \frac{\pi D}{a} \right)} \]

\[ \beta_{st} = a v \frac{a}{\delta_0} \exp \left( -\frac{\Delta U}{kT} \right) \]

1. Diffusion control - \( \frac{D}{a} \ll \beta_{st} \)

\[ v_\infty = \frac{\pi D C_0 v_c}{a \ln \left( \frac{\pi D}{a} \right)} \sigma \]

2. Kinetic control - \( \frac{D}{a} \gg \beta_{st} \)

\[ v_\infty = \beta_{st} C_0 v_c \sigma \]

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$\Delta C = 0$

$$\Delta = \frac{d^2}{dx^2} + \frac{d^2}{dy^2} + \frac{d^2}{dz^2}$$ - Laplace operator

$y = 0$ and $z = d \rightarrow C = C_\infty$

$y = 0$ and $z = a/\pi/ \rightarrow C = C_{st}$

$$C = A \ln \left[ \sin^2 \left( \frac{\pi}{y_0} y \right) + \sinh^2 \left( \frac{\pi}{y_0} z \right) \right]^{1/2} + B$$

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\[ v_\infty = \frac{\beta_{st} C_0 \nu_c \sigma}{1 + \frac{a \beta_{st}}{\pi D} \ln \left( \frac{y_0}{a} \sinh \left( \frac{\pi d}{y_0} \right) \right)} \]

1. \( \frac{\pi D}{a} \ll \beta_{st} \) (diffusion control)

\[ v_\infty = \frac{\pi D C_0 \nu_c \sigma}{a \ln \left( \frac{y_0}{a} \sinh \left( \frac{\pi d}{y_0} \right) \right)} \]

2. \( \frac{\pi D}{a} \gg \beta_{st} \) (kinetic control)

\[ v_\infty = \beta_{st} C_0 \nu_c \sigma \]

Growth of \( \text{NH}_4\text{H}_2\text{PO}_4 \) (ADP)
Prismatic face
\( T = 300K, a = 4 \times 10^{-8} \text{ cm}, \delta_0 = 4a, \nu = 1 \times 10^{13} \text{ sec}^{-1}, D \cong 1 \times 10^{-6} \text{ cm}^2 \text{ sec}^{-1} \)
\( \Delta U = 10 \text{ kcal/mol} \)

\( \beta_{st} = 4 \times 10^{-3} \text{ cm sec}^{-1} \)

\( \frac{a \beta_{st}}{\pi D} \cong 5 \times 10^{-5} \ll 1 \) (kinetic regime)

\( C_0 = 3.5 \text{ mol/l}, \quad C_0 \nu_c = 0.2, \quad \sigma = 0.03 \)

\( v_\infty = 2.4 \times 10^{-5} \text{ cm sec}^{-1} \)
GROWTH IN MELT

According to Jackson’s criterion a crystal surface will be smooth when

\[ \alpha = \frac{\Delta s_m Z_l}{k Z} > 2 \quad (Z_l < Z) \]

Metal surfaces should be rough as \( \Delta s_m/k \approx 1.2 \). For semiconductors \( \Delta s_m/k \) has larger values:

<table>
<thead>
<tr>
<th>Material</th>
<th>( \Delta s_m/k )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>3.6</td>
</tr>
<tr>
<td>Ge</td>
<td>3.7</td>
</tr>
<tr>
<td>InP</td>
<td>5.7</td>
</tr>
<tr>
<td>InSb</td>
<td>7.4</td>
</tr>
<tr>
<td>InAs</td>
<td>7.6</td>
</tr>
<tr>
<td>GaAs</td>
<td>8.5</td>
</tr>
</tbody>
</table>

and their surfaces should be smooth.

Mathematics of diffusion is precisely the same as that of heat conductivity. The diffusion coefficient is replaced by the temperature conductivity \( \kappa_T = k_T/C_P \rho \) (cm² sec⁻¹) (\( k_T \) - coefficient of thermal conductivity, \( \rho \) - density, \( C_P \) - specific heat capacity,).

\[ \beta_{st}^T = a_v \frac{\Delta s_m}{kT} \frac{a}{\delta_0} \exp \left( -\frac{\Delta s_m}{k} \right) \exp \left( -\frac{\Delta U}{kT} \right) \]

\[ v_\infty = \frac{\beta_{st}^T T_m \sigma}{1 + a \beta_{st} T_q \frac{\pi \kappa_T}{\kappa_T} \ln \left[ \frac{y_0}{a} \sinh \left( \frac{\pi d}{y_0} \right) \right]} \]

\[ T_q = \frac{\Delta H_m}{C_P^{liq}} \] - the temperature which will be reached if the heat of crystallization is not taken away.

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RATE OF STEP ADVANCE ON Si(001)

\[ k_T = 0.356 \text{ cal/cm sec K} \]
\[ C_P^{\text{sol}} = 5.455 \text{ cal/g - atom K} \]
\[ C_P^{\text{liq}} = 6.5 \text{ cal/g - atom K} \]
\[ \rho = 2.328 \text{ g/cm}^3 \]
\[ \Delta H_m = 12082 \text{ cal/mol} \]
\[ \kappa_T = 0.787 \text{ cm}^2 \text{ sec}^{-1} \]

(heat conductivity is 6 orders of magnitude larger than the diffusion coefficient in solutions)

\[ T_q = 1860K \]
\[ \Delta U \approx 5000 \text{ cal/mol} \]
\[ \delta_0 = 3.5a \]
\[ \Delta s_m / k = 3.6 \]
\[ \beta_{st}^T = 3.7 \text{ cm sec}^{-1} \text{ K}^{-1} \]
\[ a\beta_{st}^T T_q \approx 1 \times 10^{-4} \ll 1 \]
\[ \pi \kappa_T \]

(kinetic control)
\[ \nu_\infty = \beta_{st}^T (T_m - T) \approx 3.7 \text{ cm sec}^{-1} \]

GaAs

\[ \beta_{st}^T = 0.1 \text{ cm sec}^{-1} \text{ K}^{-1} \]

Two orders of magnitude smaller than for Si due to the larger entropy of melting.

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1. The step spacing is proportional to the radius of the critical 2D nucleus.
2. The length of the step in the origin is always equal to the diameter of the 2D nucleus.
SHAPE OF A ROUNDED SPIRAL

\[
v(r) = \frac{dr}{dt} = \frac{dr}{d\theta} \frac{d\theta}{dt} = \omega r'
\]
\[
\omega - \text{angular velocity}
\]
\[
v(\rho) = v(r) \cos \gamma
\]
\[
\cos \gamma = \frac{r}{\left(r^2 + r'^2\right)^{1/2}}
\]
\[
v(\rho) = \frac{\omega r r'}{\left(r^2 + r'^2\right)^{1/2}}
\]
\[
v(\rho) = v_\infty \left(1 - \frac{\rho_c}{\rho}\right)
\]

GOVERNING EQUATION:

\[
v_\infty \left(1 - \rho_c \frac{r^2 + 2r'^2 - rr''}{\left(r^2 + r'^2\right)^{3/2}}\right) = \frac{\omega r r'}{\left(r^2 + r'^2\right)^{1/2}}
\]

\(r, \theta - \text{polar coordinates}\)

\(\rho - \text{radius of curvature}\)

\[
\rho = \frac{(r^2 + r'^2)^{3/2}}{r^2 + 2r'^2 - rr''}
\]

\[
r' = \frac{dr}{d\theta}, \quad r'' = \frac{d^2 r}{d\theta^2}
\]
We cannot solve this equation. That is why we neglect all $r^2$ terms. The equation is still unsolvable and we neglect all $r$ containing terms. What remains is

$$r' = \frac{dr}{d\theta} = 2\rho_c$$

$$r = 2\rho_c \theta$$ - ARCHIMEDEAN SPIRAL

STEP SPACING:

$$\lambda = r(\theta + 2\pi) - r(\theta) = 2\rho_c (\theta + 2\pi - \theta)$$

$$\lambda = 4\pi\rho_c$$

The step spacing is proportional to $\rho_c$

CABRERA and LEVINE solution

$$\lambda = 19\rho_c = \frac{19\kappa a^2}{kT\sigma}$$

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PROPERTIES OF THE SPIRAL

The radius of curvature:

\[
\rho = \frac{(r^2 + r'^2)^{3/2}}{r^2 + 2r'^2 - rr''}
\]

\[r = 2\rho_c \theta, \quad r' = 2\rho_c, \quad r'' = 0\]

\[
\rho = \rho_c \frac{(1 + x^2)^{3/2}}{1 + \frac{1}{2}x^2}; \quad x = \frac{r}{2\rho_c}
\]

\[x \to 0, \quad r \to 0, \quad \rho = \rho_c\]

The curvature in the center of the spiral is always equal to the curvature of the critical nucleus

\[x \to \infty, \quad r \to \infty, \quad \rho = 2\rho_c x \to \infty\]

\[
v(\rho) = v_\infty \left(1 - \frac{\rho_c}{\rho}\right) = v_\infty \left(1 - \frac{1 + \frac{1}{2}x^2}{(1 + x^2)^{3/2}}\right)
\]

1. \(x \to 0, \quad r \to 0, \quad v(\rho) \to 0\)
   The growth in the center is slow and the spiral rotates slowly (slow kinetics).

2. \(x \to \infty, \quad r \to \infty, \quad v(\rho) \to v_\infty\)
   The growth far from the center is fast and the spiral rotates rapidly (fast kinetics).
GROUPS OF SCREW DISLOCATIONS

DISLOCATIONS WITH OPPOSITE SIGNS MAKE LOOPS WHEN THE CENTERS DISTANCE $\mathbf{AB}$ IS GREATER THAN THE RADIUS OF THE CRITICAL RADIUS

Ivan Markov, Bulgarian Academy of Sciences
IF THE NEIGHBORING DISLOCATIONS HAVE ONE AND THE SAME SIGN BUT $l = AB \gg 2\rho_C$ THE STEP SPACING IS EQUAL TO $l$ AND DOES NOT DEPEND ON THE SUPERSATURATION

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IF THE NEIGHBORING DISLOCATIONS HAVE ONE AND THE SAME SIGN BUT $l = AB \ll 2\rho_C$ THE STEP SPACING IS TWICE SMALLER THAN THE DISTANCE $\Lambda$ ORIGINATING FROM A SINGLE DISLOCATION

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WE HAVE A SOURCE OF $n$ DISLOCATIONS WITH A LENGTH $L$

$L = nl$

1. $l \gg 2\rho_c$ ($L \gg \lambda = 19\rho_c$)

$$y_0 = l = \frac{L}{n}$$

2. $l \ll 2\rho_c$ ($L \ll \lambda$)

$$y_0 = \frac{\lambda}{n}$$

IN THE GENERAL CASE:

$$y_0 = \frac{\lambda}{N},$$

where $N = \frac{n}{1 + \frac{L}{\lambda}}$ is the strength of the dislocation source.

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SPIRAL GROWTH FROM A VAPOR
THE FAMOUS PARABOLIC LAW OF BURTON, CABRERA AND FRANK (BCF)

\[ R = p v_\infty = a \frac{v_\infty}{y_0} \]

\[ \frac{v_\infty}{y_0} - \text{flux of steps passing in a direction parallel to the terraces} \]

\[ v_\infty = 2 \sigma \lambda_s \nu \exp\left(-\frac{\varphi_{1/2}}{kT}\right) \tanh\left(\frac{y_0}{2\lambda_s}\right) \]

\[ y_0 = \frac{\lambda}{n} = \frac{19\kappa a^2}{nkT\sigma} \]

\[ R = C \frac{\sigma^2}{\sigma_c} \tanh\left(\frac{\sigma_c}{\sigma}\right) \]

\[ C = a \nu \exp\left(-\frac{\varphi_{1/2}}{kT}\right) \]

\[ \sigma_c = \frac{19\kappa a^2}{2nkT\lambda_s} \]

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\[ \kappa \approx 3 \times 10^{-5} \text{ erg cm}^{-1} \]
\[ a \approx 3 \times 10^{-8} \text{ cm} \]
\[ \lambda_s \approx 2 \times 10^3 a \]
\[ T = 1000K \]
\[ n = 1 \]
\[ \sigma_c \approx 3 \times 10^{-2} \]

1. \[ \sigma \gg \sigma_c (\lambda_s \gg y_0 / 2) \]
\[ R = C \sigma \]

2. \[ \sigma \ll \sigma_c (\lambda_s \ll y_0 / 2) \]
\[ R = C \frac{\sigma^2}{\sigma_c} \]
THE CLASSICAL EXPERIMENT IN CRYSTAL GROWTH

ELECTROLYTIC GROWTH OF SINGLE FACES OF Ag

Budewski, Kaischew, Bostanov (Bulg. Acad. Sci. Sofia)
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Current (rate) of growth of Ag(111) (top) and Ag(001) (bottom) vs the square of the overpotential (supersaturation).

Bostanov et al. 1969.

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THE “BACK-STRESS” (FEEDBACK) EFFECT

The supersaturation in the spiral center will be smaller than predicted by

\[
\Lambda_0 = \frac{19\kappa a^2}{kT\sigma} \quad \text{or} \quad \frac{\Lambda_0}{\lambda_s} = 2 \frac{\sigma_c}{\sigma}
\]

due to the steps around the center. The higher the supersaturation, the smaller the radius of the first turn, the smaller the supersaturation, the greater the radius of the first turn.

\[
\Psi(r) = \Psi(\rho) \frac{I_0\left(\frac{r}{\lambda_s}\right)}{I_0\left(\frac{\rho}{\lambda_s}\right)} \quad (r \leq \rho)
\]

\[
r = 0, \quad I_0(0) = 1
\]

\[
\sigma_s(0) = \sigma \left[ 1 - I_0^{-1}\left(\frac{\Lambda_0}{\lambda_s}\right) \right]
\]

\[
I_0^{-1}(x) \approx 1 - \frac{1}{4} x^2
\]

\[
\frac{\Lambda_0}{\lambda_s} \approx 2 \left(\frac{\sigma_c}{\sigma}\right)^{1/3}
\]

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\[ \frac{\Lambda_0}{\lambda_s} \approx 2 \left( \frac{\sigma_c}{\sigma} \right)^{1/3} \]

\[ \frac{\Lambda_0}{\lambda_s} = 2 \frac{\sigma_c}{\sigma} \]
SPIRAL GROWTH IN SOLUTION

\[ R = C \frac{\sigma^2}{\sigma_c} \ln^{-1} \left[ \frac{d}{\pi a} \frac{\sigma_c}{\sigma} \sinh \left( \frac{\sigma}{\sigma_c} \right) \right] \]

Growth of ADP (\(NH_4H_2PO_4\))
\[ \kappa \approx 5.5 \times 10^{-7} \text{ erg cm}^{-1}, \]
\[ \rho_c \approx 0.95 \times 10^{-6} \text{ cm}, \]
\[ \Lambda = 18 \times 10^{-6} \text{ cm}, \]
\[ n = 1, \]
\[ p = \tan \theta = 2.4 \times 10^{-3}, \]
\[ \sigma = 0.03 \]
\[ \nu_\infty = 2.4 \times 10^{-5} \text{ cm sec}^{-1} \]
\[ R = 6.2 \times 10^{-8} \text{ cm sec}^{-1} \]

1. \(\sigma \ll \sigma_c\)  \((\sinh(x) \approx x)\)
\[ R = C \frac{\sigma^2}{\sigma_c} \ln^{-1} \left( \frac{d}{\pi a} \right) \]

2. \(\sigma \gg \sigma_c\)  \(\sinh(x) \approx \frac{1}{2} \exp(x)\)
\[ R = C \sigma \]

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GROWTH BY 2D NUCLEATION

LAYER-BY-LAYER GROWTH

BILAYER GROWTH

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1. $J_0(2D) = \text{const}(t)$, $\nu_\infty = \text{const}(t)$  

LAYER-BY-LAYER GROWTH

$\tilde{J}_0 = J_0 L^2$ - frequency of nucleation 

$T = \frac{L}{\nu}$ - time for complete coverage 

$N = \tilde{J}_0 T = J_0 L^2 \frac{L}{\nu} = J_0 \frac{L^3}{\nu}$ - number of nuclei formed during the period $T$

$N \leq 1$ - LAYER-BY-LAYER GROWTH

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2D nucleation growth of ADP

\[ T = 300 K, \]
\[ \kappa \approx 1 \times 10^{-6} \text{ erg cm}^{-1}, \]
\[ a = 8 \times 10^{-8} \text{ cm}, \]
\[ K_1 \approx 1 \times 10^{19} \text{ cm}^{-2} \text{ sec}^{-1}, \]
\[ R_c \approx 1 \times 10^{-9} \text{ cm sec}^{-1}, \]
\[ L = 1 \times 10^{-4} \text{ cm}, \]
\[ \sigma_c = \frac{\Delta \mu_c}{kT} = 0.38 \]
\[ \frac{C}{C_0} \approx 1.5 \]

This result was the reason to develop the theory of spiral growth because growth was observed at supersaturations much smaller than 50%.

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RHEED intensity oscillations of Si(001) at 800K

Sakamoto et al. 1986
Ivan Markov, Bulgarian Academy of Sciences
DECAY OF THE NORMALIZED INTENSITY IN THE GROWTH OF Cu/Cu(111)
COMSA ET AL. 1994

Ivan Markov, Bulgarian Academy of Sciences
MULTILAYER GROWTH

THE PROBLEM OF SECOND LAYER NUCLEATION

\[ \tilde{J}_0 = J_0 l^2 \]

\( l \) - critical island size for second layer nucleation

\[ T = \frac{l}{\nu} \approx \frac{1}{\tilde{J}_0} = \frac{1}{J_0 l^2} \rightarrow l \approx \left( \frac{\nu}{J_0} \right)^{1/3} \]

\( T \) - time elapsed from nucleation of the 1st island to the nucleation event of the second island

\[ R = J_0 l^2 a = a \left( J_0 \nu^2 \right)^{1/3} \]

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\[ J_0 = K_1 \exp\left( -\frac{\Delta G^*}{kT} \right) \]

\[ K_1 \propto \sqrt{\Delta \mu} \]

\[ \nu \propto \Delta \mu \]

\[ R = C\Delta \mu^{5/6} \exp\left( -\frac{\Delta G^*}{3kT} \right) \]

Growth of ADP prismatic face

\[ T = 300 \text{K} , \]

\[ \nu = 2.4 \times 10^{-5} \text{ cm sec}^{-1} , \]

\[ K_1 \approx 1 \times 10^{19} \text{ cm}^{-2} \text{ sec}^{-1} , \]

\[ R_c \approx 1 \times 10^{-9} \text{ cm sec}^{-1} , \]

\[ \sigma_c = 8.6\% \text{ vs } 50\% \text{ in LbL growth} \]

\[ \therefore \text{THE CRITICAL SUPERSATURATION IN ORDER GROWTH TO TAKE PLACE IS SMALLER IN MULTILAYER RATHER THAN IN LAYER-BY-LAYER GROWTH, BUT THE RATE OF GROWTH IS GREATER.} \]

\[ \text{SMALL FACES - LbL GROWTH} \]

\[ \text{LARGE FACES - MULTILAYER GROWTH} \]

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$R_1 = a \frac{d \Theta_1}{dt}$

$\Theta_1 = 1 - \exp \left( -\frac{\pi}{3} J_0 c^2 t^3 \right)$

$R_1 = a \pi J_0 c^2 t^2 \exp \left( -\frac{\pi}{3} J_0 c^2 t^3 \right)$

$Ivan Markov, Bulgarian Academy of Sciences$
MULTILAYER MULTINUCLEAR GROWTH
FORMATION OF MOUNDS

\[
\Theta_1 = 1 - \exp \left[ -\pi J_0 c^2 \int_0^t \left( \int_{t'}^t k(\tau - t') d\tau \right)^2 dt' \right]
\]

\[
\Theta_n = 1 - \exp \left[ -\pi J_0 c^2 \int_0^t p_{n-1}(t') \left( \int_{t'}^t k(\tau - t') d\tau \right)^2 dt' \right]
\]

\( p_{n-1}(t') \) - probability to find in moment \( t' \) of nucleation a crystallized part in the underlying \( n \)-1st layer

\( J = J(t) \) - transient nucleation

\[
\Theta_n = 1 - \exp \left[ -\pi c^2 \int_0^t J(t') p_{n-1}(t') \left( \int_{t'}^t k(\tau - t') d\tau \right)^2 dt' \right]
\]

\[
\frac{d\Theta_n}{dt} = \left[ 1 - \Theta_n(t) \int_0^t J(t') p_{n-1}(t') 2\pi \rho_n(t') \nu_n(t') dt' \right]
\]

\( \rho_n(t') \) - radius of the growing 2D island

\( \nu_n(t') = d\rho_n/dt \) - rate of lateral growth

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MEAN FIELD APPROXIMATION
BOROVINSKI and ZINDERGOSEN

\[ p_{n-1}(t') = \Theta_{n-1}(t') \]

\[ \Theta_{n+1}(t) = \Theta_n(t - T) \]

\[ T = 0.63(J_0c^2)^{-1/3} \]

\[ R = \frac{a}{T} = 1.59a(J_0c^2)^{1/3} \]

\[ R = \sum_{n=1}^{\infty} R_n = \sum_{n=1}^{\infty} a \frac{d\Theta_n}{dt} \]

\[ T' = 2.6(J_0c^2)^{-1/3} \]

\[ \frac{T'}{T} \approx 4 \text{ monolayers grow simultaneously.} \]
MEAN FIELD MONTE CARLO

SUBMONOLAYER

RATE OF 2D GROWTH $R \tau / a$

TIME

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GROWTH TRANSIENT OF Ag(111)

THE FIRST EVER OBSERVED GROWTH OSCILLATIONS
BOSTANOV ET AL. 1981

Ivan Markov, Bulgarian Academy of Sciences
SUMMARY

\[ J_0 = \text{const} \cdot 2D \text{ nucleation rate (cm}^{-2} \text{sec}^{-1}) \]
\[ \nu = \text{const} \cdot \text{step advance rate (cm sec}^{-1}) \]
\[ \tilde{J}_0 = J_0 l^2 \] - frequency of nucleation
\[ T = \frac{l}{\nu} = \frac{1}{\tilde{J}_0} \] - time elapsed from nucleation of the lower to nucleation of the upper island
\[ N = \tilde{J}_0 T \] - number of nuclei formed in the period \( T \)
\[ N = 1 \] - condition for layer-by-layer growth
\[ N = 2 \] - condition for simultaneous growth of 2 MLs