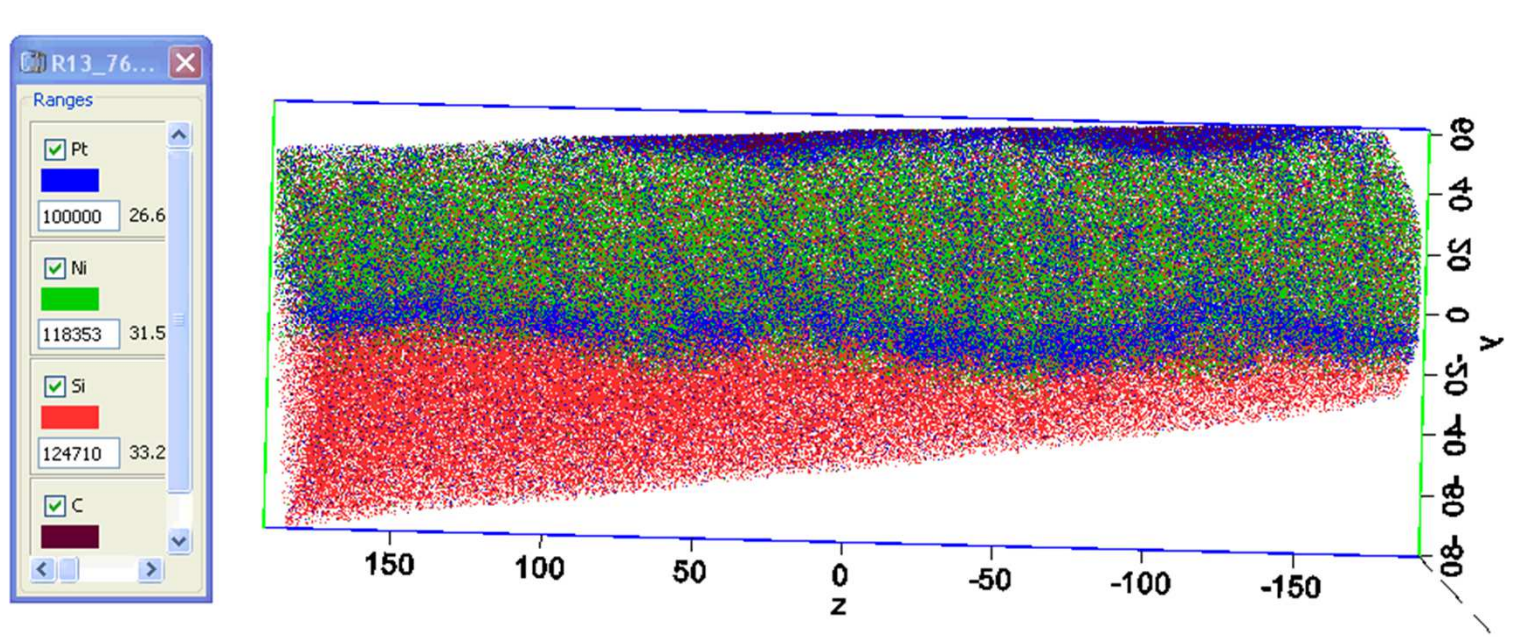


Diffusion réactive en microélectronique

Dominique Mangelinck

IM2NP, CNRS/Aix-Marseille Université, Faculté de saint Jérôme, Marseille, France



Atom probe tomography of Ni(Pt) silicide on Si(100)

- ❑ **IM2NP** : C Bergman, M Bertoglio, **I Blum** , **A. Derafa**, M. Descoins, B Duployer, L Ehouarne, P Gas, **K. Hoummada**, **F. Nemouchi** , **F. Panciera**, **G. Tellouche**, A. Portavoce

- ❑ **STMicroelectronics**: M. Gregoire, M. Juhel, R. Pantel, N. Bicais

- ❑ **MFA – Budapest** : J.L. Lábár

- ❑ **CEA–LETI Grenoble** : V. Carron, F. Nemouchi

- ❑ **IMAGO** : D Larson, R Ulfig, P Clifton

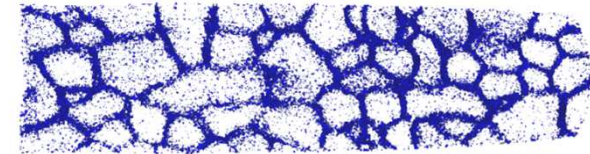
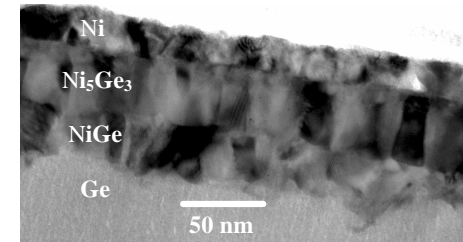
- ❑ **KTH, Kista**: S.L. Zhang, B. Swensson

- ❑ **ATMEL** : R. Coppard

- ❑ Financial support
 - ❑ ANR TAPAS
 - ❑ CNRS, FEDER, UPCAM, region PACA, CIM PACA

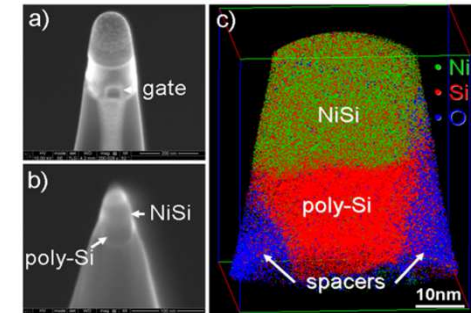
1. Basis of reactive diffusion

- a) Diffusion couple / thin films
- b) First stages : nucleation and lateral growth
- c) Deal & Grove law and silicides
- d) Sequential growth –simultaneous growth
- e) Role of grain boundaries



2. Typical example : silicides in microelectronics

- a) Contacts in microelectronics
- b) Analyses of transistors by atom probe
- c) Nucleation and alloy effect



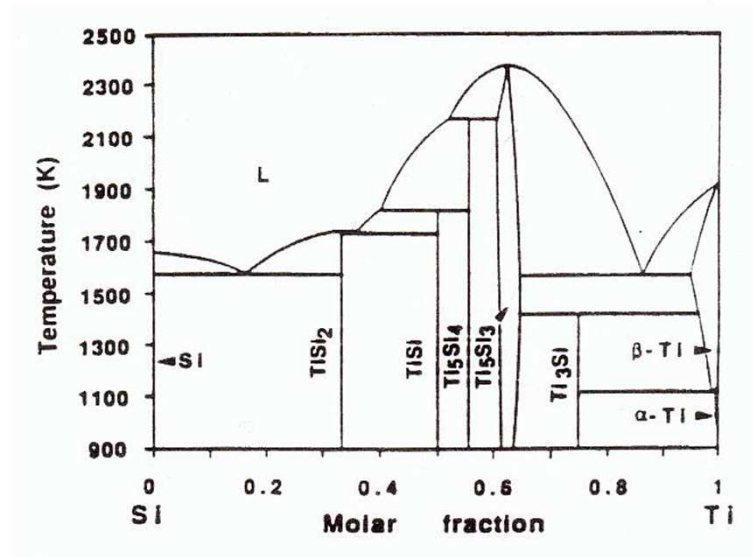
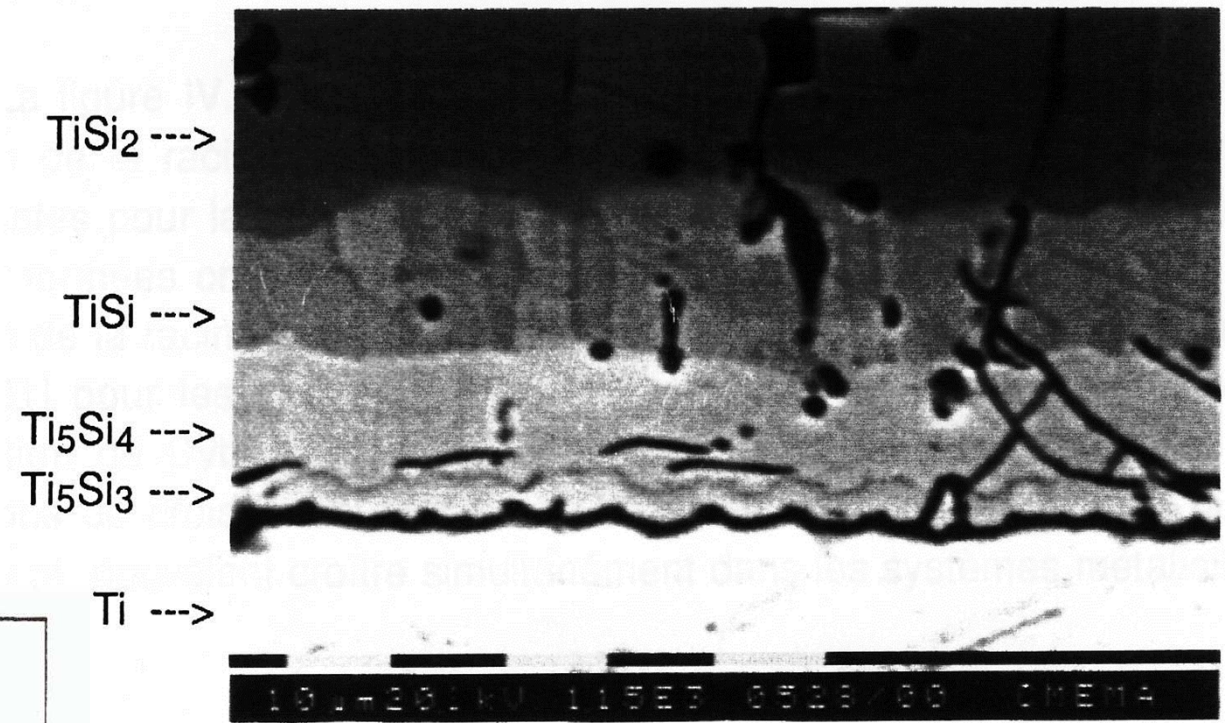
3. Challenges

- a) Encroachment / transient phase
- b) Nucleation / texture / stress
- c) Doping / defects / dislocation / precipitation / redistribution
- d) Nanoelectronics

Two bulk materials in contact
 + Heat treatment
 + Micrography

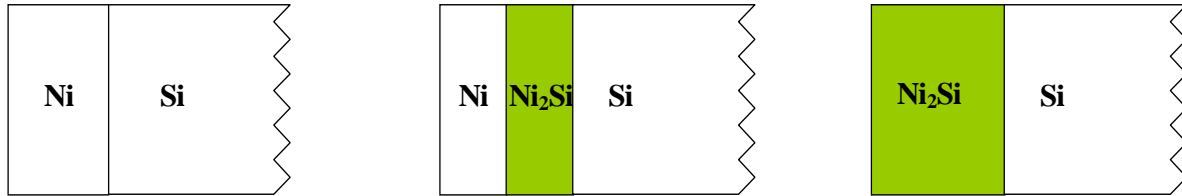
→ all the phases of the equilibrium diagramme diagramme in layers

Diffusion couple : Ti/Si

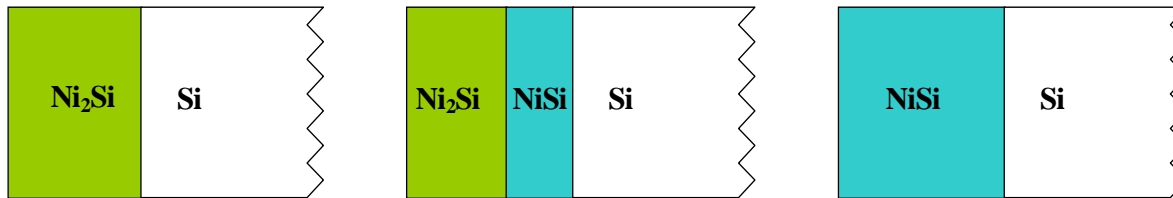


$L \propto \sqrt{t}$ → diffusion controlled growth

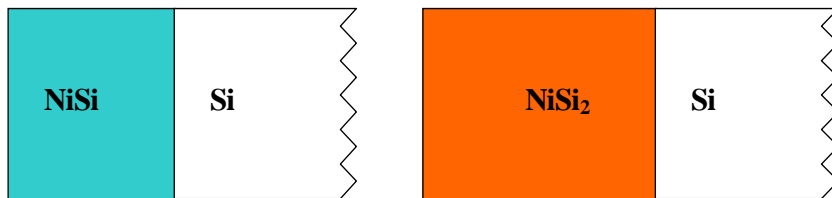
Reaction of Ni thin films with Si



T ~ 200-300°C : formation of Ni₂Si : DIFFUSION controlled , Ni diffusion

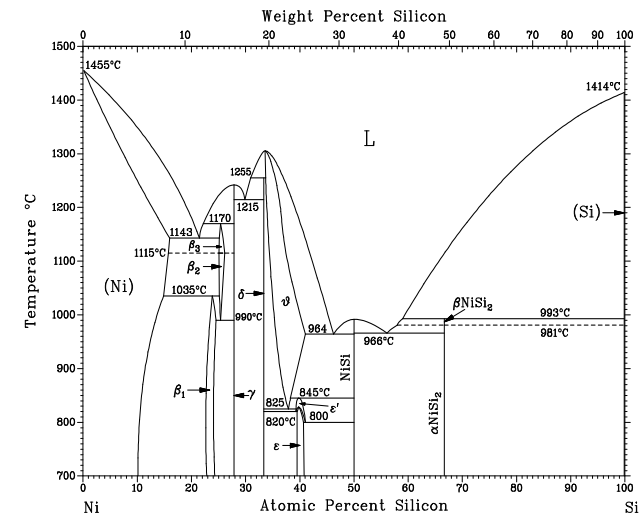


T ~ 250-350°C : formation of NiSi : DIFFUSION controlled , Ni diffusion



T ~ 750-800°C : formation of NiSi₂ : NUCLEATION controlled , Ni diffusion

Submicron thickness
 - sequential formation
 - 3 phases: Ni₂Si, NiSi, NiSi₂
 - diffusion of Ni



≠ Ni-Si phase diagram

Nanometric thickness

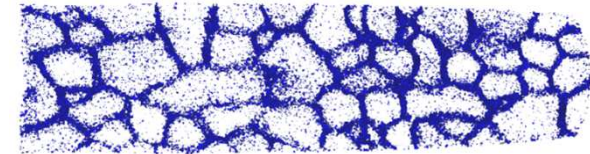
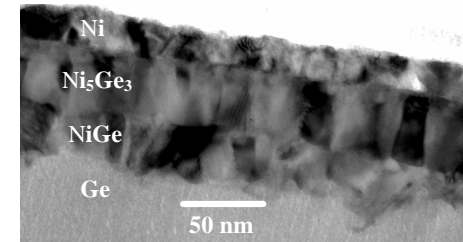
[Lavoie et al Microelec. Eng. 2003]

- new phases?
- “transient” phases
- kinetic ?

→ Better understanding of the growth mechanisms

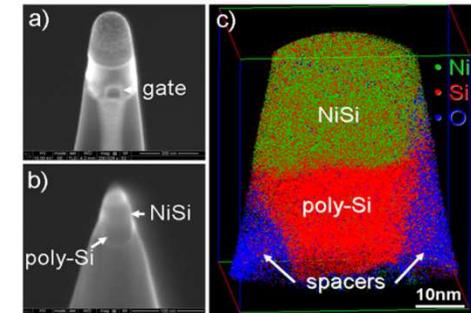
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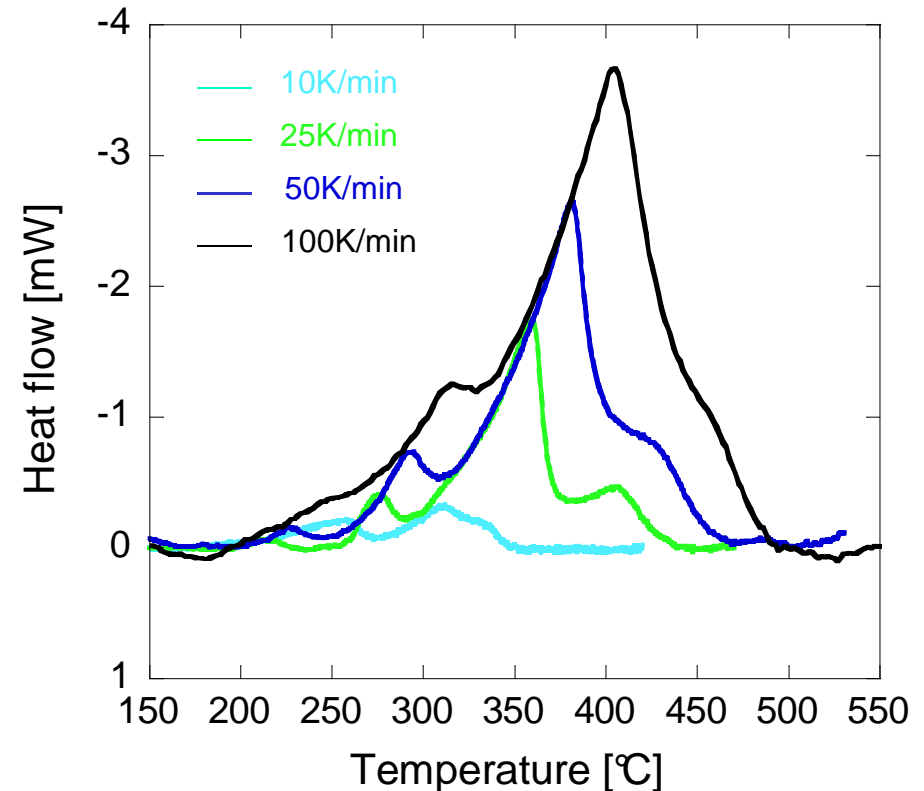
3. Challenges

- a) Encroachment / transient phase
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Differential scanning calorimetry (DSC)

- Advantages of DSC
 - Thermodynamics
 - Sensitivity to first stages of formation
- Limits of DSC
 - Quantity of materials
 - Substrate “noise”

Nemouchi et al, APL, 2005



DSC thermograms of 50 nm Ni films on a-Si with different ramps

↳ **DSC analysis of reaction of 50 nm Ni films with a-Si on a Si substrate**

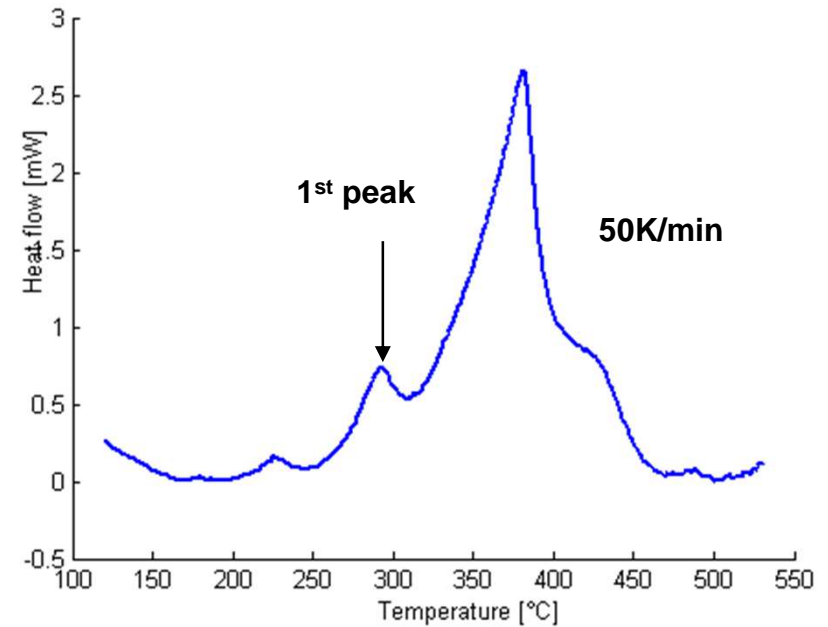
2 DSC peaks for Ni₂Si

1st Peak → lateral growth (LG) of nuclei

2nd Peak → normal growth (NG)

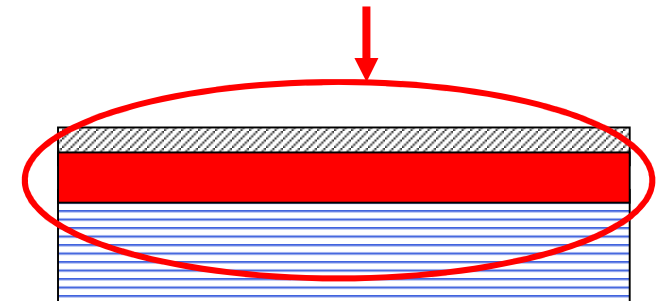
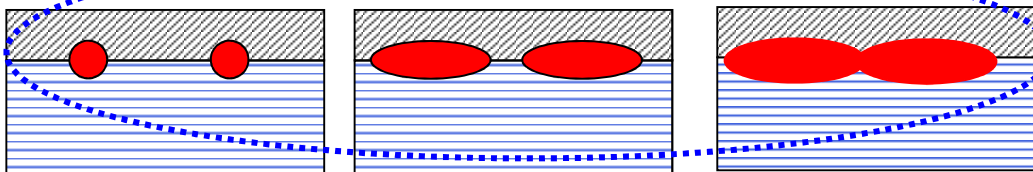
model : Coffey et al APL 1989

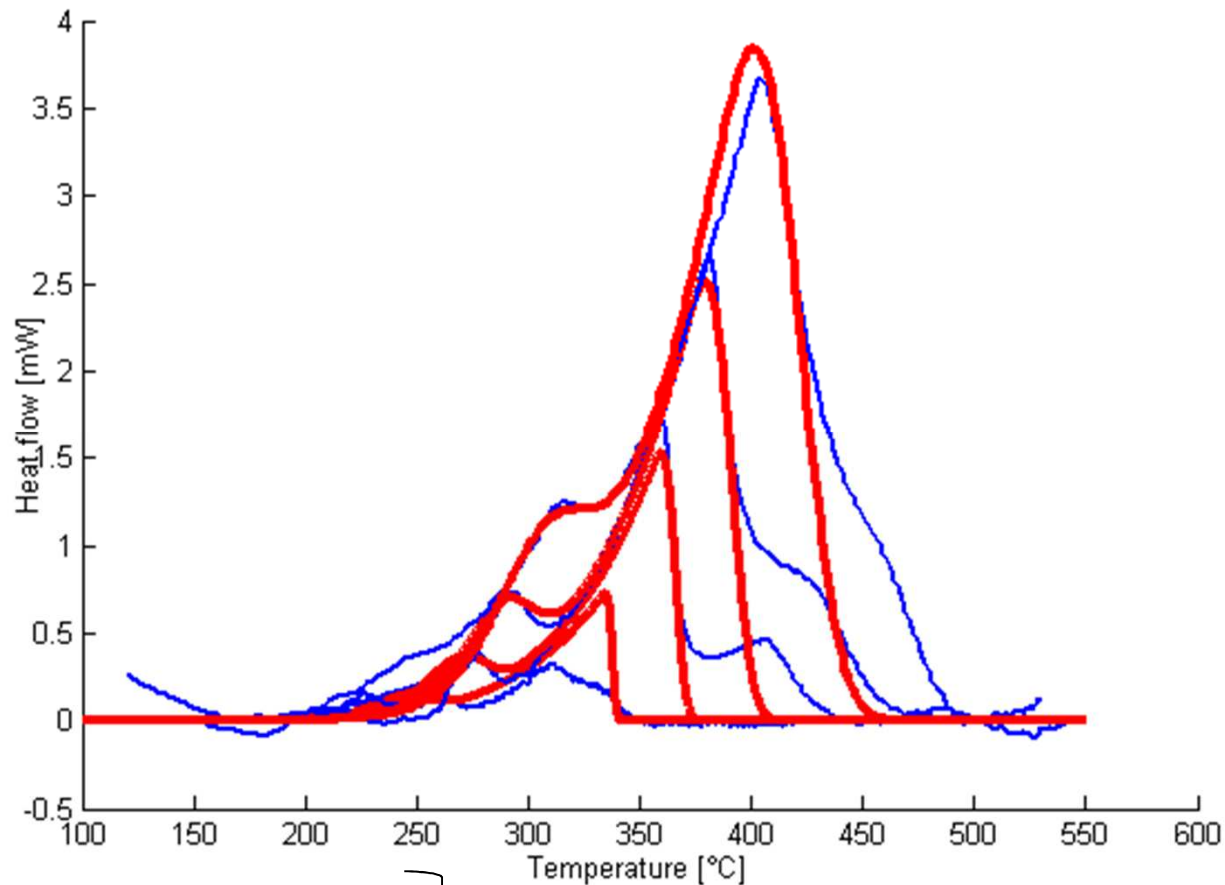
- Density of existing nuclei
- Cylindrical nuclei of radius r
- Lateral growth (LG) controlled by interface mobility



Transformed volume fraction:

$$\frac{dX_v}{dt} = \frac{dX_A}{dt} \frac{L}{L_0} + X_A \frac{dL}{dt} \frac{1}{L_{\max}}$$





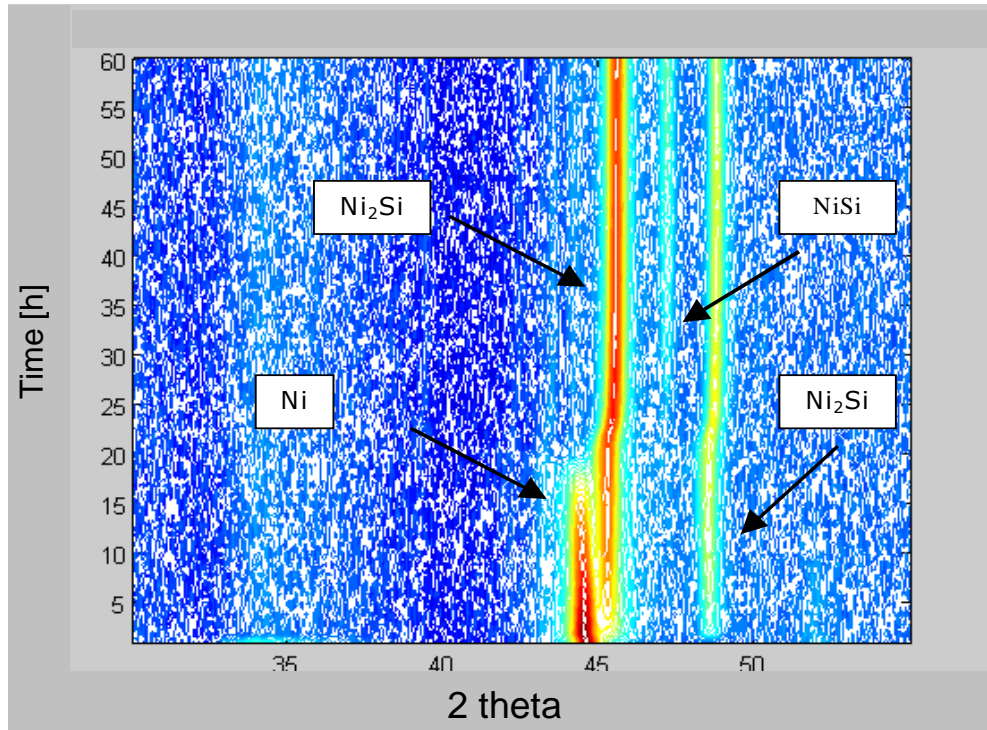
$$\text{LG: } \frac{dr}{dt} \propto K_{LG}$$

$$\text{NG: } \frac{dL}{dt} \propto \frac{D}{L + D/K_{NG}} \frac{\Delta G}{RT}$$

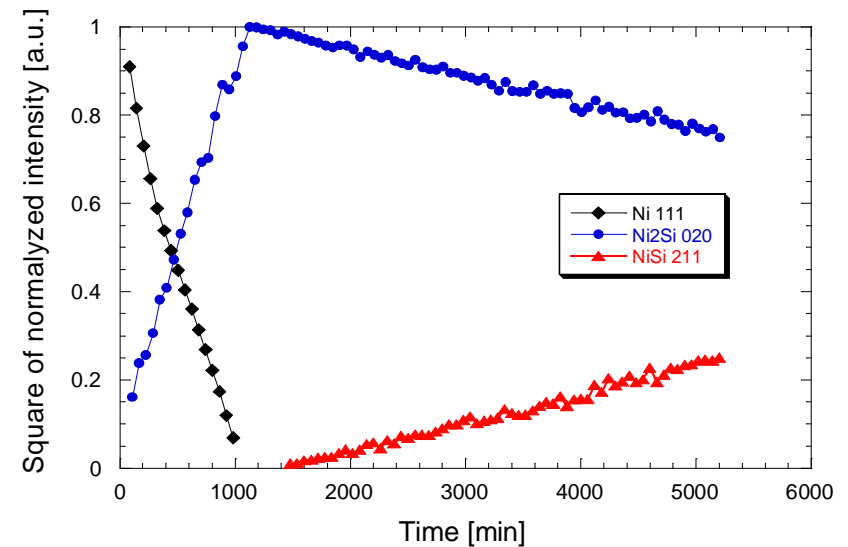
→ Interface mobility for lateral growth:

$$\rightarrow K_{LG} = 10 \exp(-0.85/kT) \text{ [cm/s]}$$

Reaction between a 50 nm film of Ni and amorphous Si



In situ X ray diffraction for the annealing at 210°C of a 50 nm Ni film on amorphous Si



Normalized intensity of X ray diffraction peaks for the annealing at 210°C of a 50 nm Ni film on amorphous Si

$$L \propto \sqrt{t} \rightarrow \text{Diffusion controlled growth}$$

P Gas - FM d'Heurle: J. Mat. Res 1986, Landolt-Börnstein 1998 ...

- Fick law:
$$J = -D \frac{dc}{dx}$$

- stoichiometric phase !
- unknown or inappropriate limits of composition !!

- **Nernst Einstein**

- $J = v C$; v = migration velocity;
 $v = -M (d\mu/dx)$; M = mobility = D/RT

- Nernst Einstein relation :
$$J = -C \frac{D}{RT} \frac{d\mu}{dx}$$

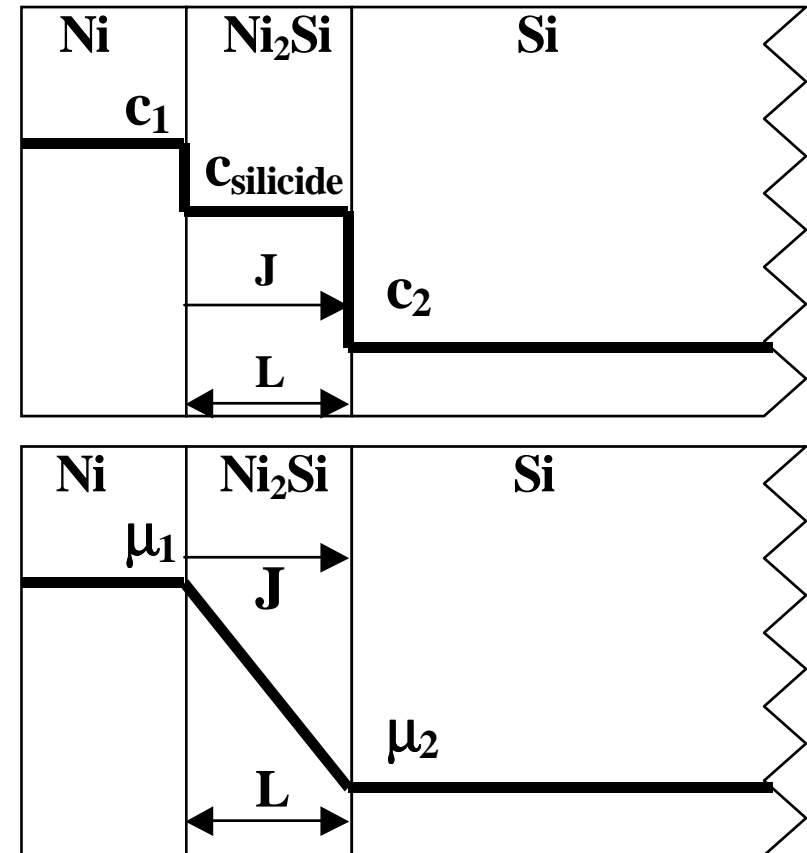
- Formation of a phase

- $d\mu/dx \sim \Delta G/L$

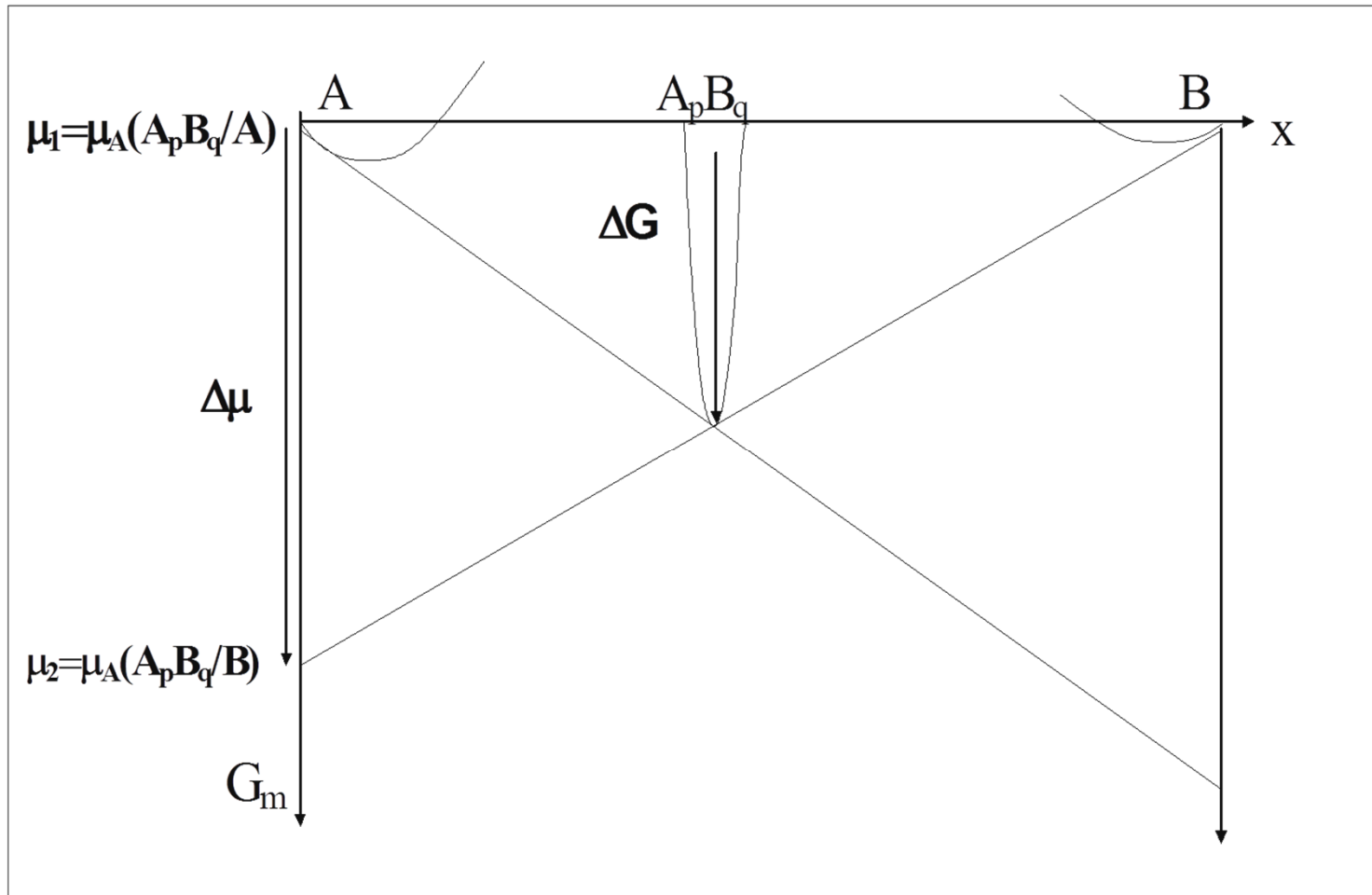
ΔG : free energy of formation

-

$$\frac{dL}{dt} = \frac{\Delta G}{RT} \frac{D}{L} \rightarrow L^2 = 2D \frac{\Delta G}{RT} t$$

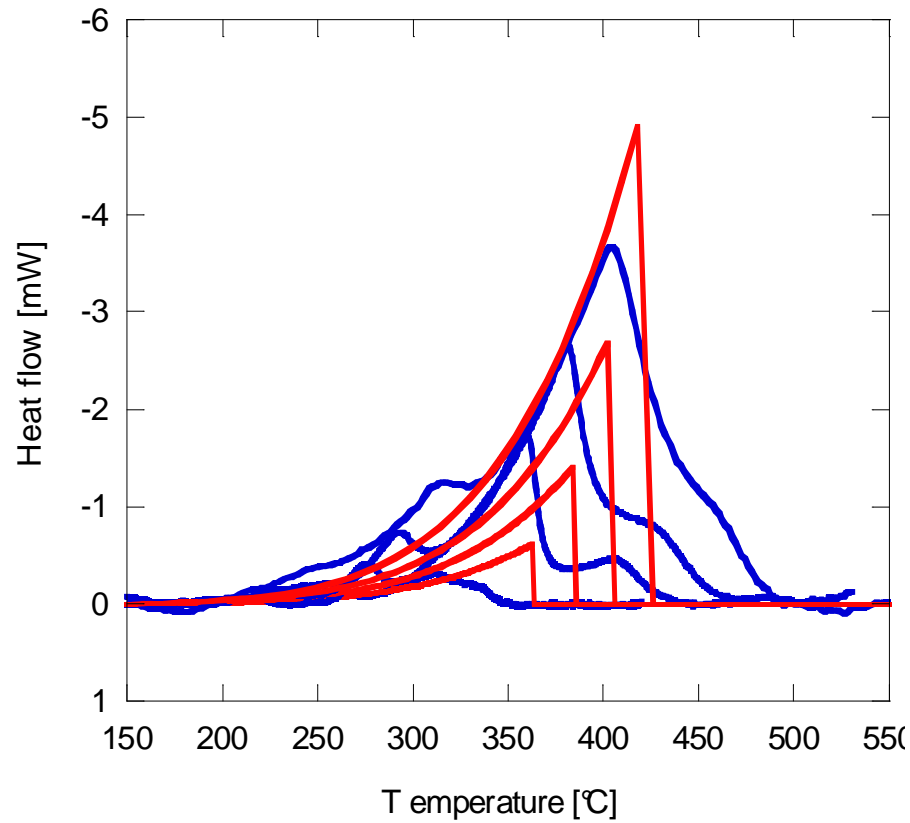


Parabolic growth

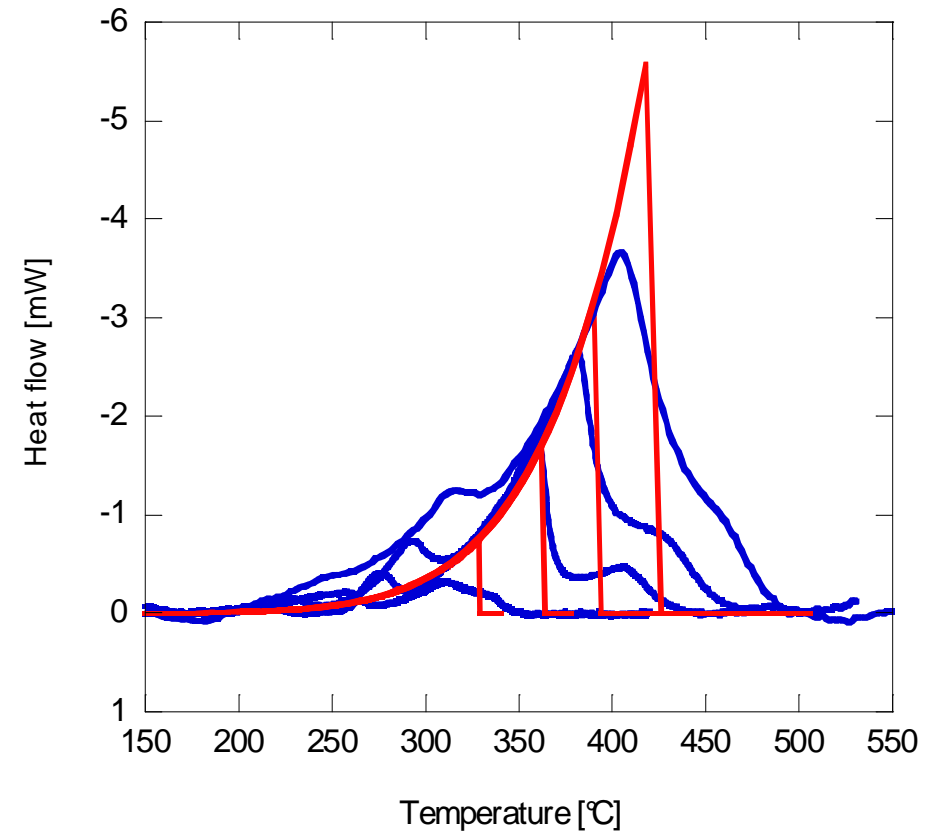


$$d\mu/dx \sim \Delta G/L$$

Simulation of the normal growth



Diffusion controlled growth



Interface controlled growth

- effective flux from grain 1 to grain 2

$$A_2 n_1 v_1 \exp(-\Delta G^a / RT)$$

- effective flux from grain 2 to grain 1

$$A_1 n_2 v_2 \exp(-(\Delta G^a + \Delta G) / RT)$$

- equilibrium $A_1 n_2 v_2 = A_2 n_1 v_1$

- net flux

$$J_{\text{net}} = A_2 n_1 v_1 \exp\left(-\frac{\Delta G^a}{RT}\right) \left(1 - \exp\left(-\frac{\Delta G}{RT}\right)\right)$$

$$J_{\text{net}} = A_2 n_1 v_1 \exp\left(-\frac{\Delta G^a}{RT}\right) \left(\frac{\Delta G}{RT}\right)$$

$$v = \Omega J_{\text{net}} = K \Delta G$$

Si $\Delta G = 0$, $v = 0 \rightarrow$ no growth

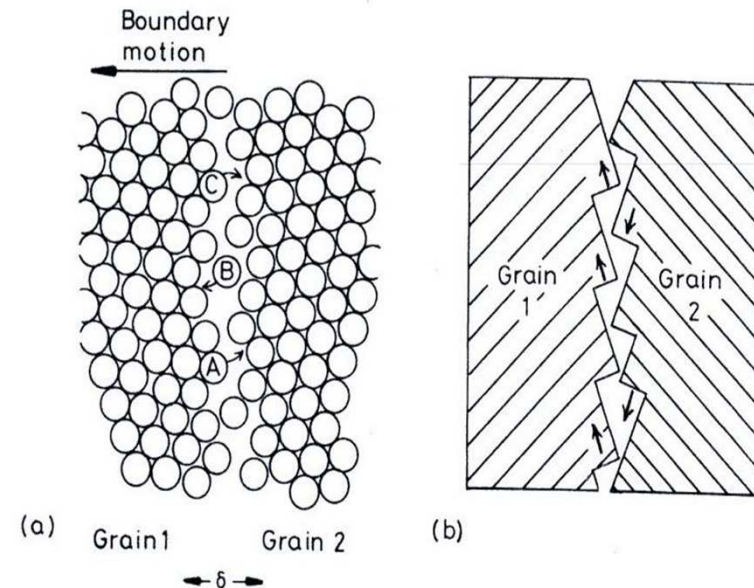


Fig. 3.23 (a) The atomic mechanism of boundary migration. The boundary migrates to the left if the jump rate from grain 1 \rightarrow 2 is greater than 2 \rightarrow 1. Note that the free volume within the boundary has been exaggerated for clarity. (b) Step-like structure where close-packed planes protrude into the boundary.

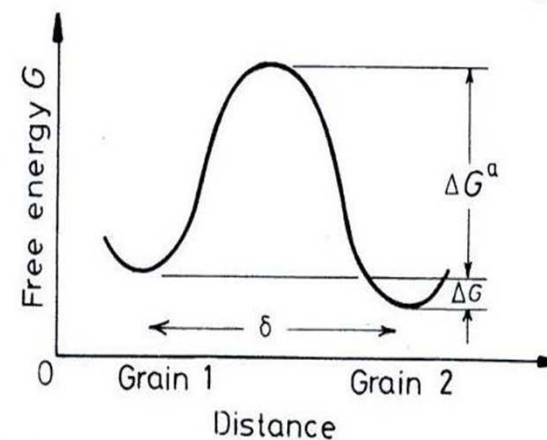


Fig. 3.24 The free energy of an atom during the process of jumping from one grain to the other.

- Growth of Ni₂Si: $\frac{dL}{dt} = -\frac{1}{\Omega} J$

- Diffusion in Ni₂Si (Nernst-Einstein)

$$J = -\frac{1}{\Omega} \frac{D}{RT} \nabla \mu = -\frac{1}{\Omega RT} D \frac{\mu_2 - \mu_1}{L}$$

$$\frac{dL}{dt} \propto \frac{D}{L} \quad \text{Isotherm} \Rightarrow L \propto \sqrt{t}$$

- Reaction at the interface

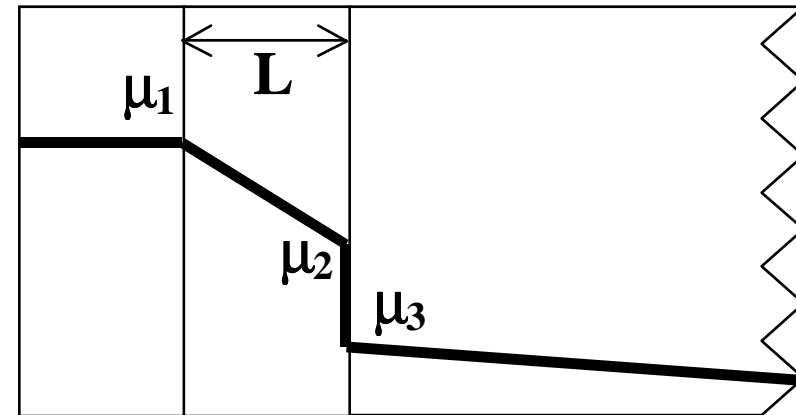
$$J = \frac{1}{\Omega} \frac{K}{RT} \Delta \mu = \frac{1}{\Omega RT} K (\mu_3 - \mu_2)$$

$$\frac{dL}{dt} \propto K \quad \text{Isotherm} \Rightarrow L \propto t$$

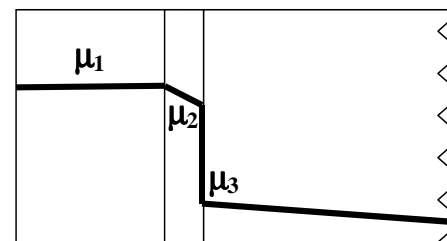
- Deal & Groove law :

$$\frac{dL}{dt} = \frac{D}{L + D/K} \frac{\mu_3 - \mu_1}{RT}$$

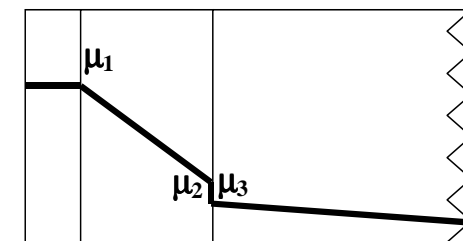
Ni Ni₂Si Si



Linear-parabolic growth

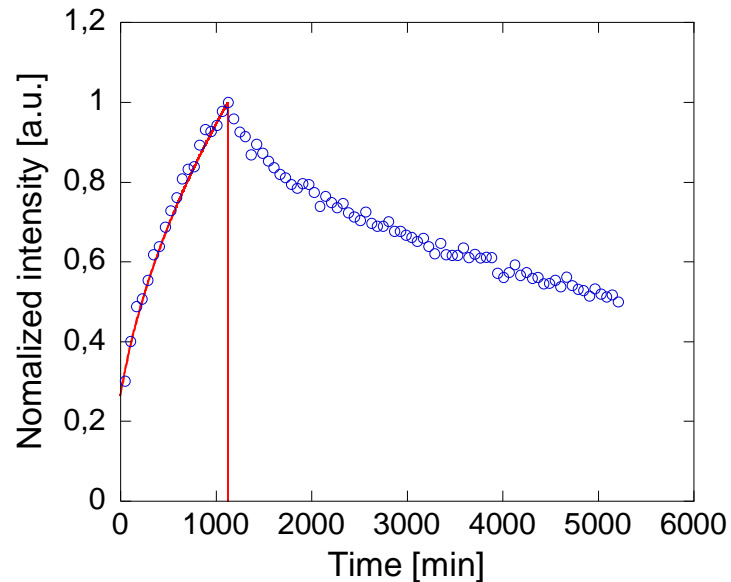


L petit

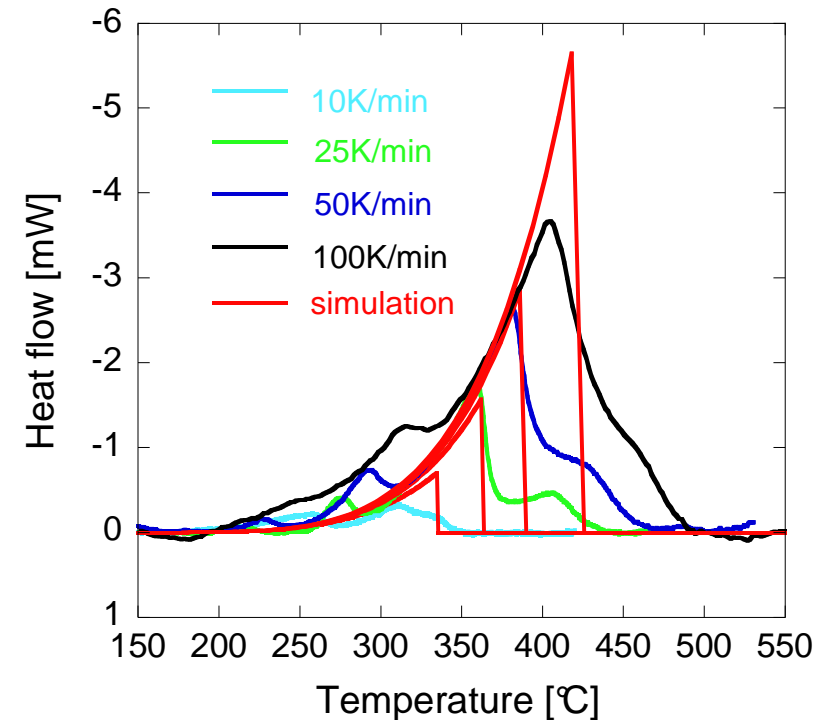


L grand

Isotherm → linear parabolic growth



Simulation of XRD results (210°C)



Simulation of DSC results

Linear parabolic growth :

→ Interface : $K_n = 0.25 \exp(-0.8/kT)$ [cm/s]

→ Diffusion : $D = 1.67 \exp(-1.5/kT)$ [cm²/s]

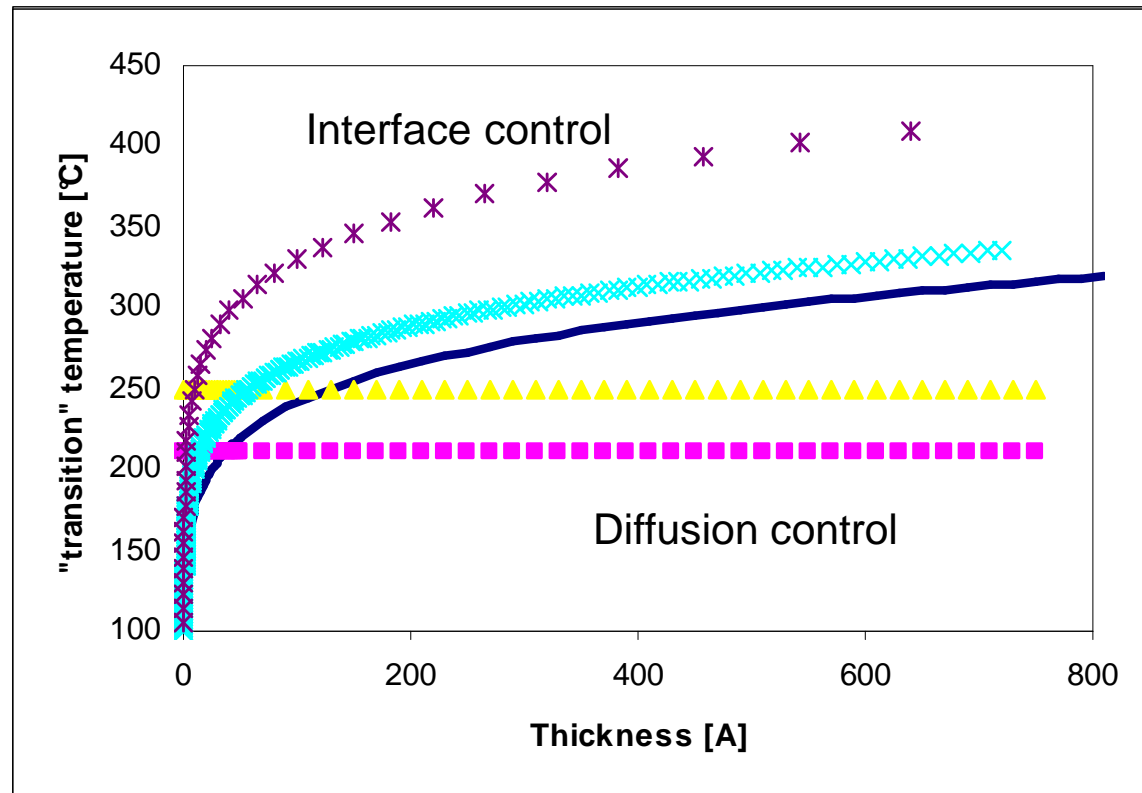
K_n = interface mobility (attachment, reaction, vacancy annihilation...)

- Deal and Groove law:

$$\frac{dL}{dt} = \frac{D}{L + D/K} \frac{\mu_3 - \mu_1}{RT}$$

- transition thickness or transition temperature :

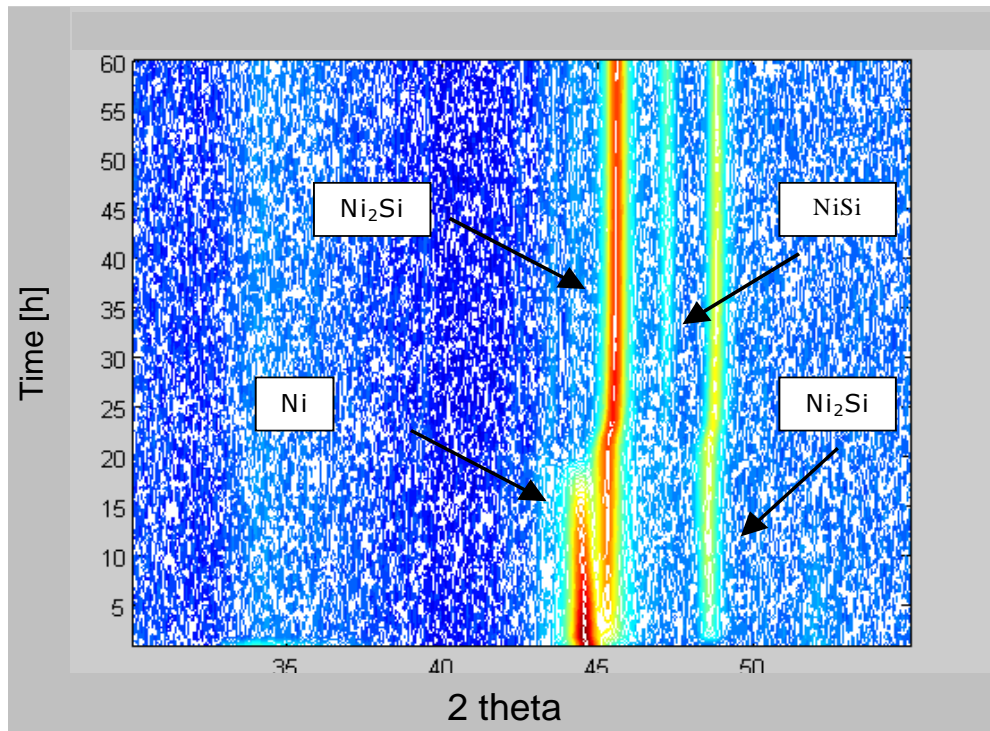
$$L_t = \frac{D}{K} \Rightarrow T_t = \frac{E_D - E_i}{k_B \ln \left(\frac{D_0}{L_t v_0} \right)}$$



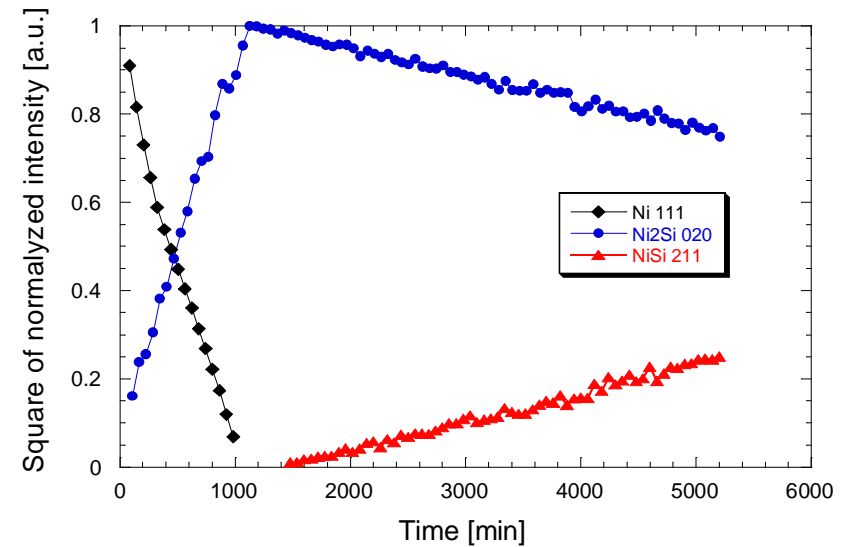
- $T \gg T_t$ or $L \ll L_t \rightarrow$ interface controlled growth \rightarrow isotherm: linear growth
- $T \ll T_t$ or $L \gg L_t \rightarrow$ diffusion controlled growth \rightarrow isotherm: parabolic growth

High temperature (RTP), small thickness \rightarrow interface controlled growth
Sequential growth linked to interface control

Reaction between a 50 nm film of Ni and amorphous Si

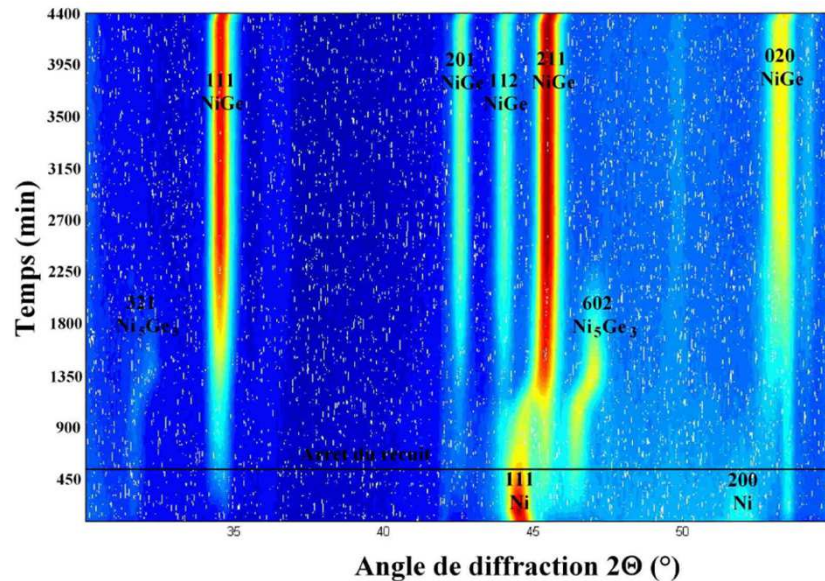


In situ X ray diffraction for the annealing at 210°C of a 50 nm Ni film on amorphous Si

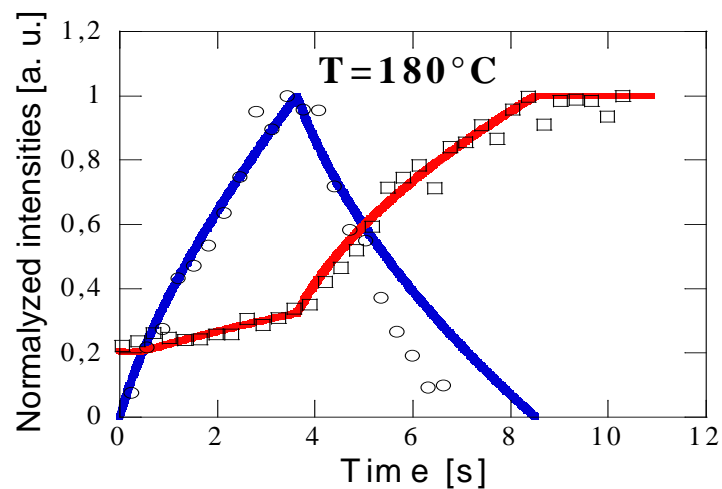


Normalized intensity of X ray diffraction peaks for the annealing at 210°C of a 50 nm Ni film on amorphous Si

→ Sequential formation

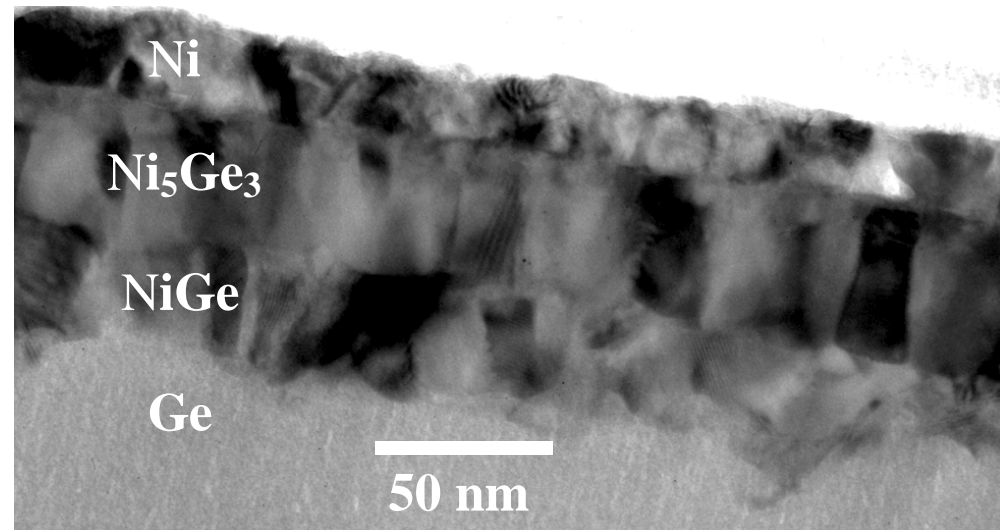


In situ XRD ($T = 180^\circ\text{C}$) of a 50 nm Ni film on amorphous Ge



XRD intensities and simulation with linear-parabolic law

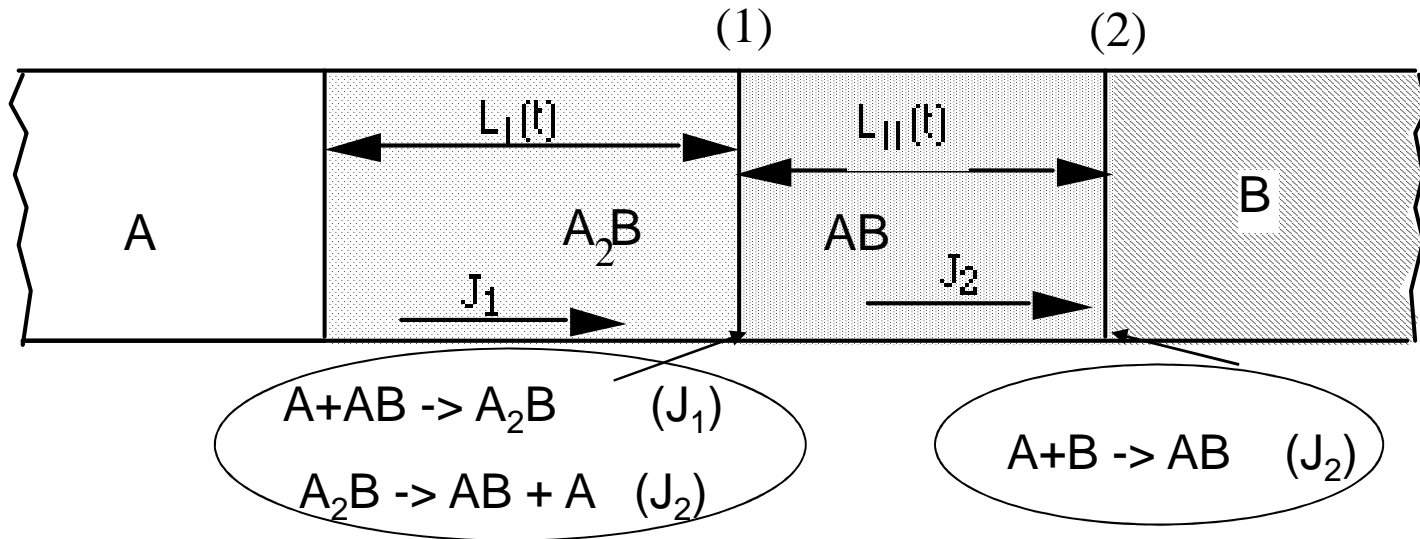
Nemouchi et al, APL, 2005



TEM images of a Ni/aGe sample annealed 100 min at 175°C

→ **Simultaneous growth of Ni_5Ge_3 and NiGe**

U. Gösele and K. N. Tu, JAP (1982) - P Gas and FM d'Heurle, Appl. Surf. Sci. 1993



$$\frac{dL_1}{dt} \propto J_1 - J_2$$

$$\frac{dL_2}{dt} \propto -J_1 + 2J_2$$

At the beginning of phase 2 growth for
 Phase 1: diffusion control
 Phase 2 : interface control

$$\frac{dL_2}{dt} \propto 2K_2 - \frac{D_1}{L_1}$$

$$\frac{dL_2}{dt} > 0 \Rightarrow L_1 > \frac{D_1}{K_2}$$

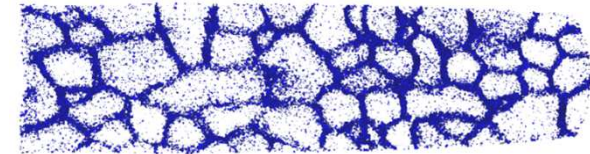
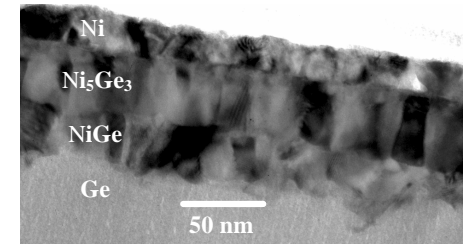
Phase 1 has to reach a critical thickness before phase 2 can grow

→ Simultaneous formation or sequential formation

$$L_c (\text{Ni/Ge}) < L_{\text{Ni}} < L_c (\text{Ni/Si})$$

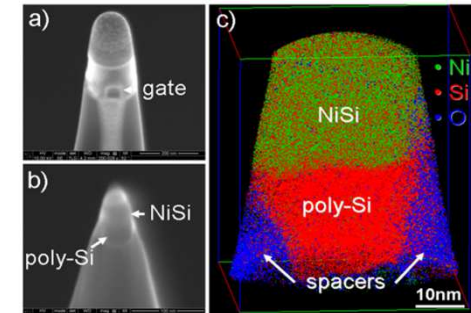
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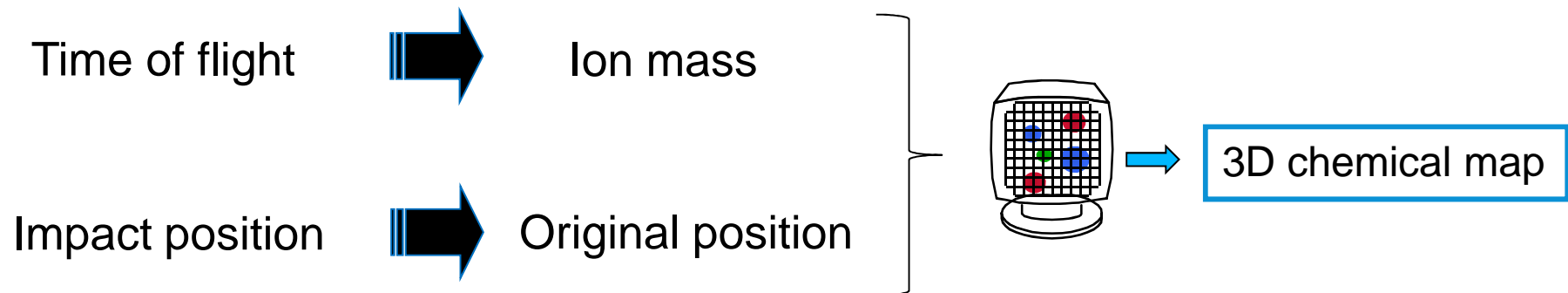
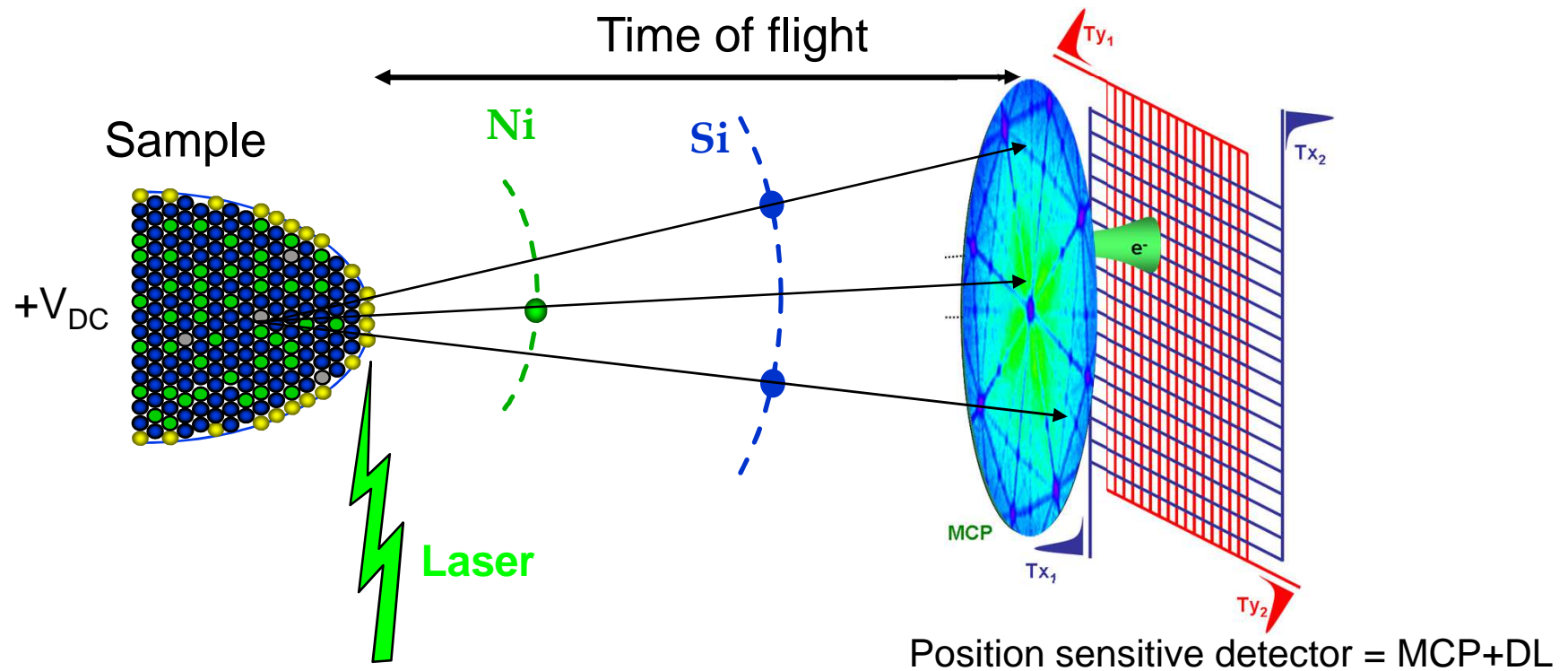
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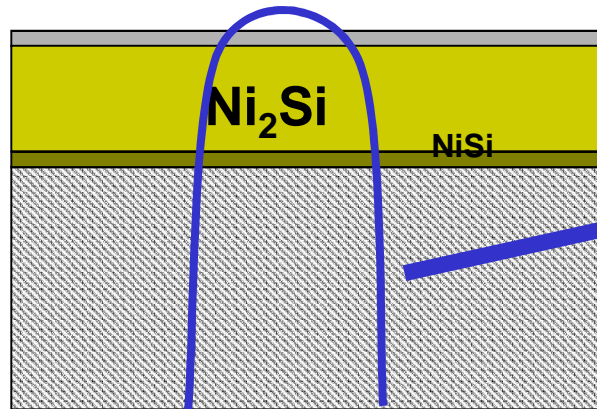
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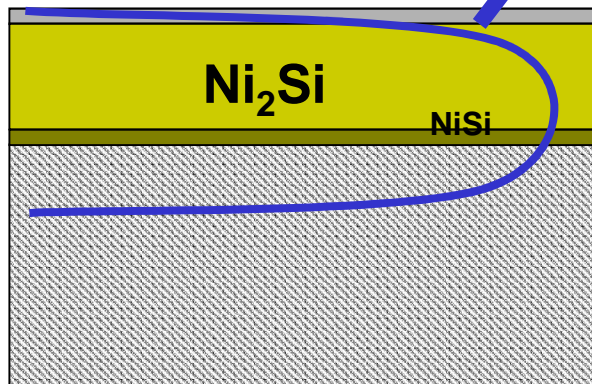
Ions field emission: high voltage (V_{DC}) + pulsed laser



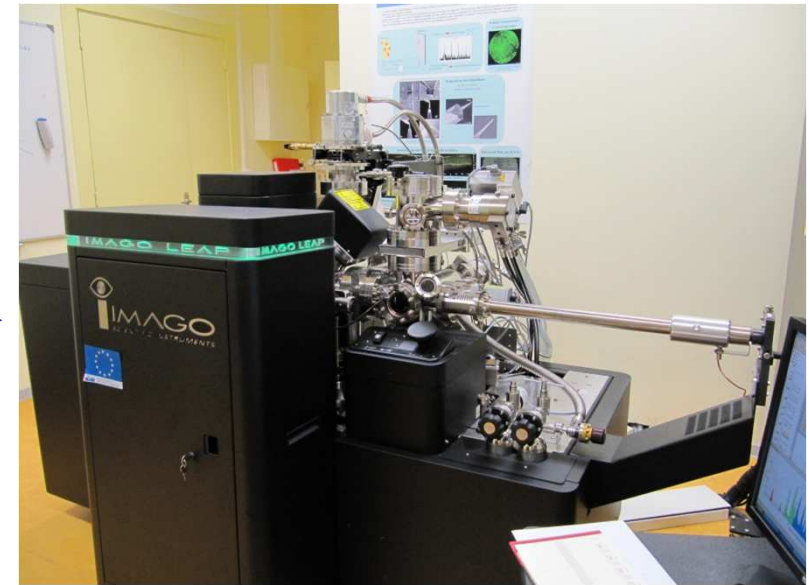
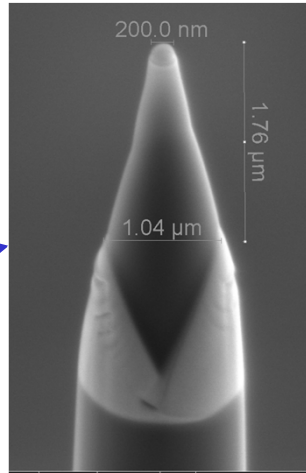
Tip preparation by FIB



Top down



Cross section



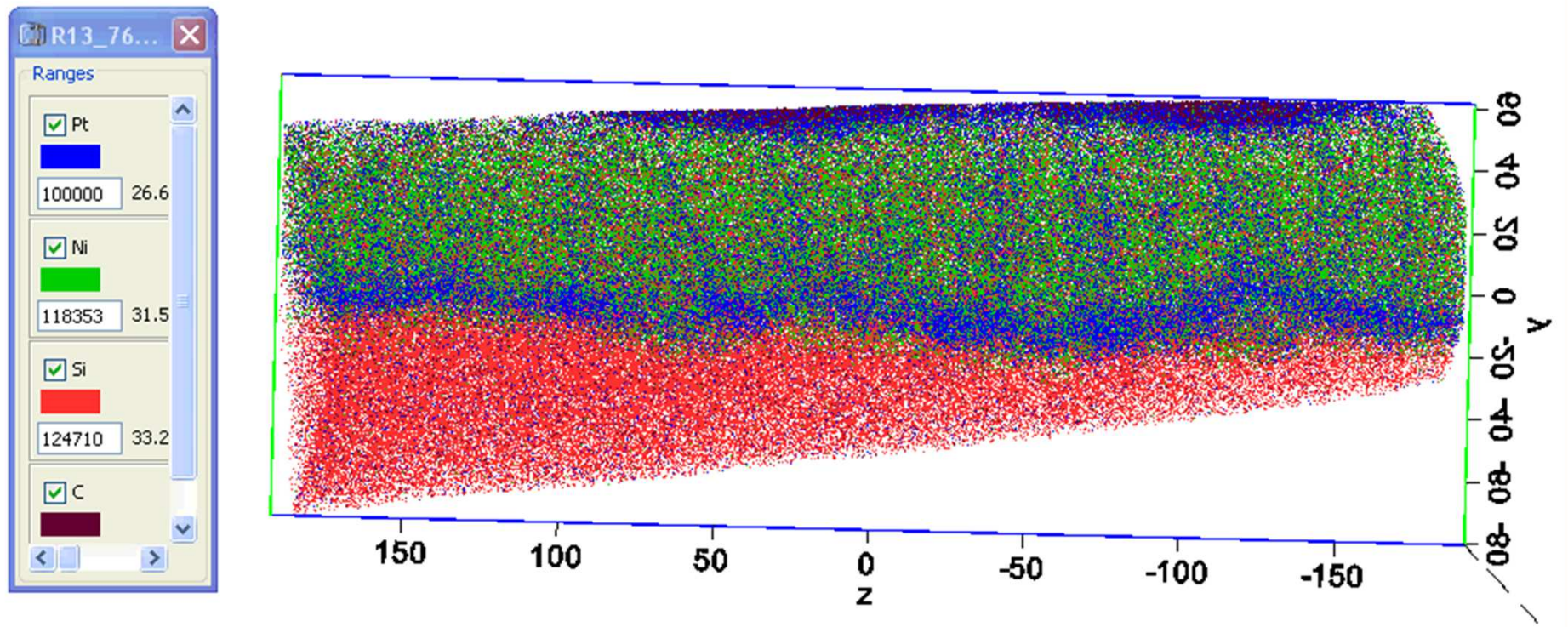
LEAP3000XHR:

• **Mass resolution :**

- $M/DM(\text{FWHM}) > 1000$
- $M/DM(\text{FWTM}) > 500$

• **Typical diameter :** 100-200 nm• **Laser:** Focalization and continuous control• **Local electrode**

- simpler tip preparation by FIB
- less rupture



Thin film reaction: redistribution of Pt and Ni silicide formation

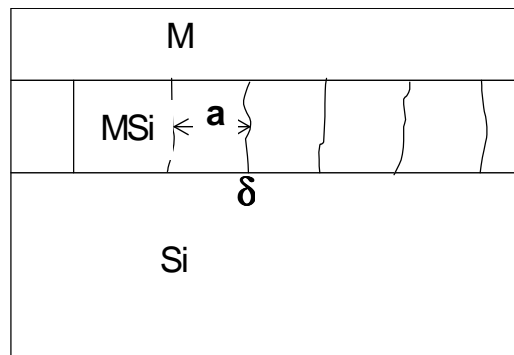
- LEAP3000XHR
 - T=50K,
 - Pulse: 200 kHz,
 - Laser energy= 0.6 nJ/pulse
 - Evaporation rate = 0.01 ions per pulse
 - **V = 120x120x400 nm³**
 - **170 Millions of atoms**
- Each point = 1 atom
 - Red = Si
 - Green = Ni
 - Blue = Pt

- Formation of CoSi_2

Difference: thin films – diffusion couple
= Different mechanisms ?

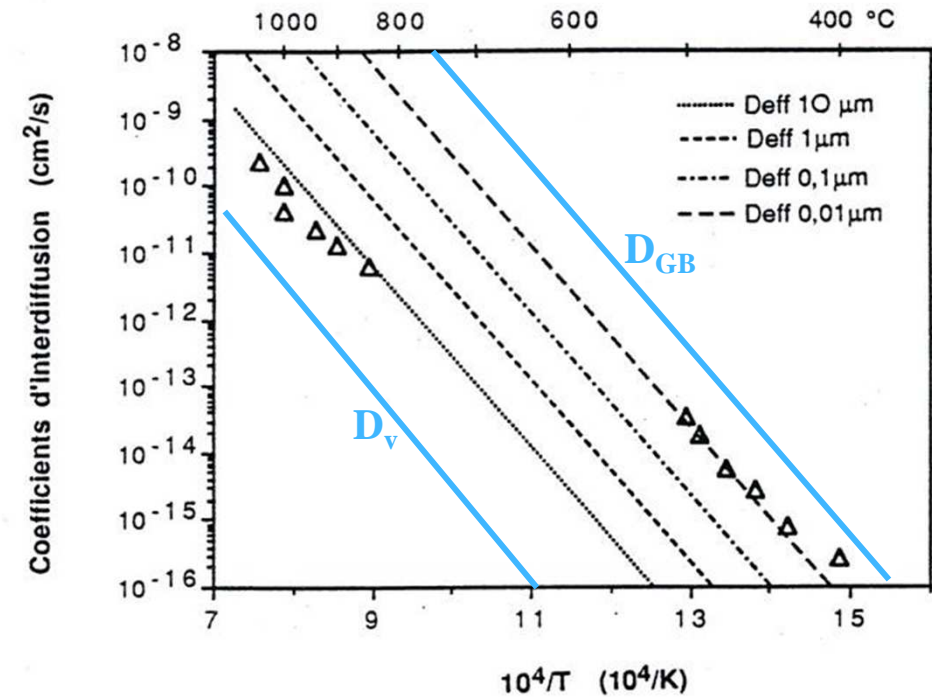
- Measurement of selfdiffusion by radiotracers (^{60}Co , $^{68}\text{Ge} \sim \text{Si}$)

- $D_{\text{eff}} = D_v + (D_{\text{gb}} \times 2d/a)$
 $d \approx 5 \cdot 10^{-8} \text{ cm}$; $a = 10\text{-}0,01 \text{ mm}$



→ Role of grains boundaries diffusion

Barge et al, J Mat Res, 1995



Comparison between interdiffusion coefficients obtained from growth rate of des taux de croissance de CoSi_2 in thin films and bulk diffusion couple

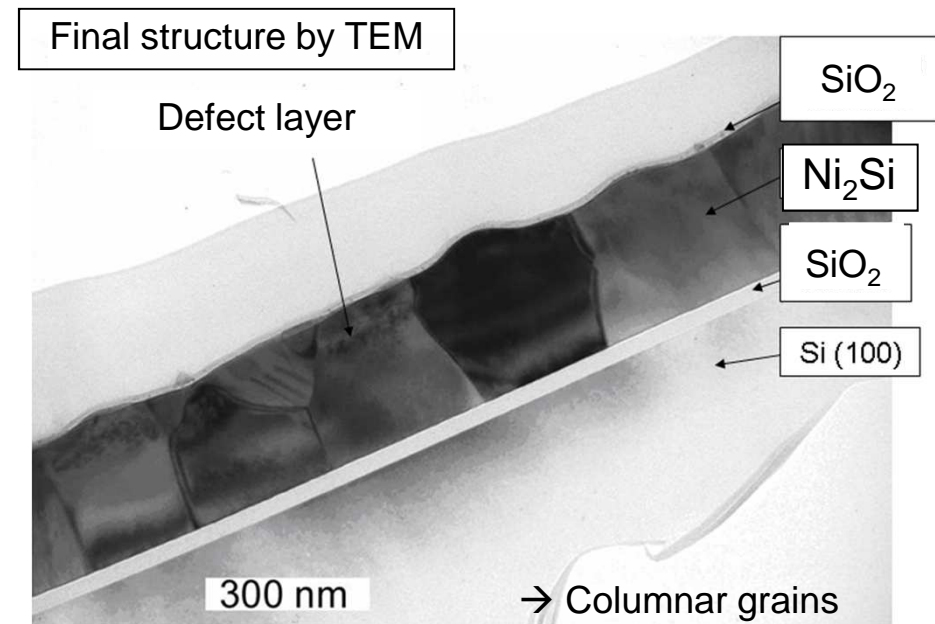
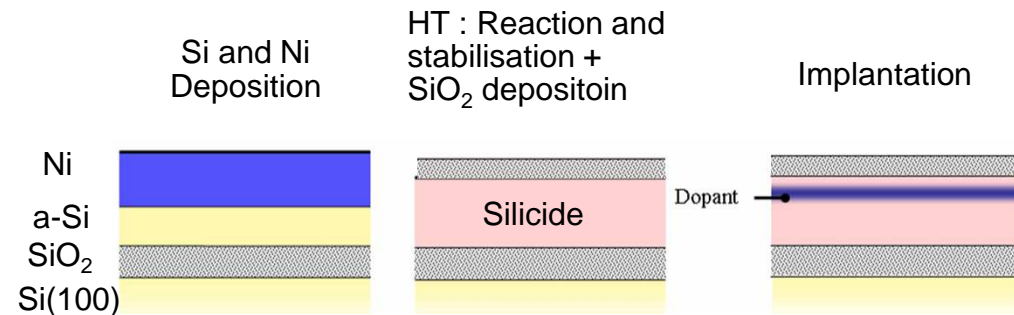
Fabrication process

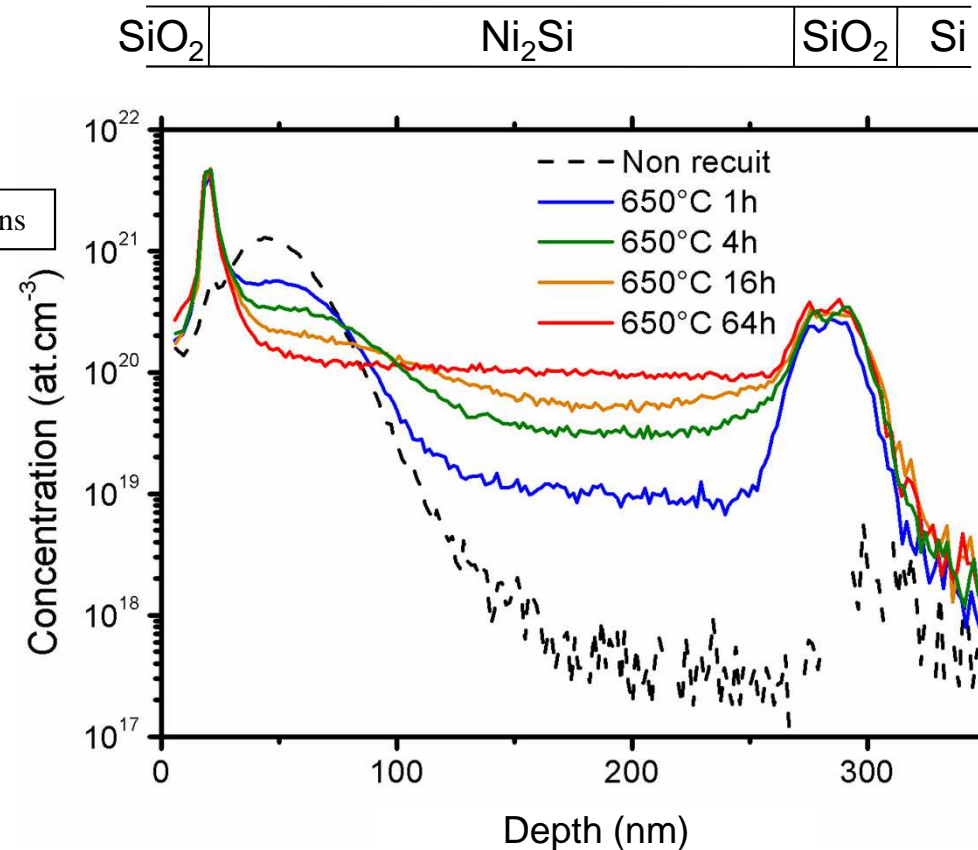
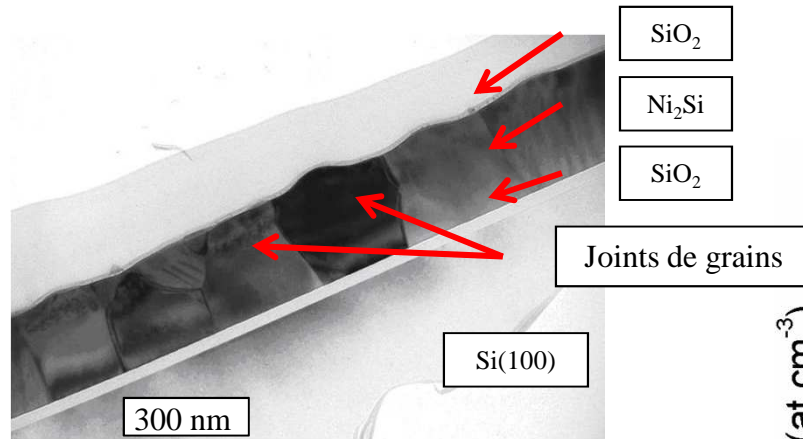
1. Deposition of Ni and Si bilayers on a SiO₂ layer
2. RTA to form the silicide
 - Si and Ni thicknesses are adjusted to form Ni₂Si or NiSi : controlled by XRD
3. SiO₂ deposition
4. Stabilization anneal : 700°C – 2h
 - Stabilize the grain size

Oxide layers act as diffusion barriers :

- No reaction between the silicide and the substrate
- No outdiffusion or evaporation of the diffusing element
- Top oxide layer : less implantation damages

5. Dopant implantation and diffusion annealing



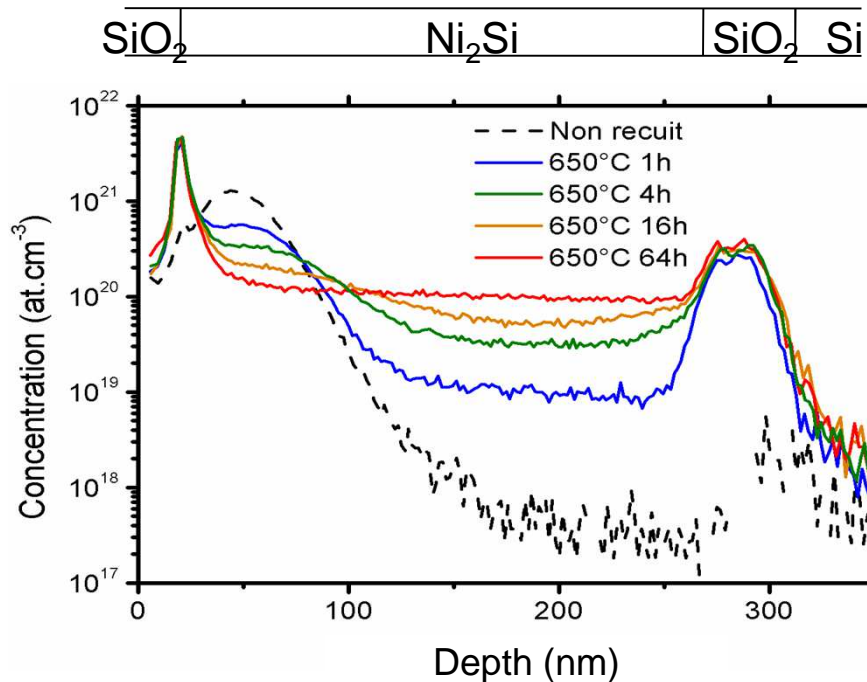
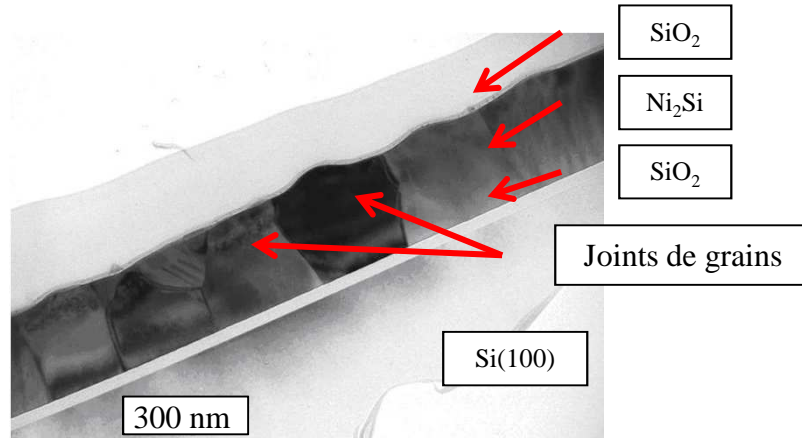
Heat treatments at 650°C + Depth profiles by SIMS

- Interface concentration has reached equilibrium after 1h.
- Flattening and broadening of the implantation peak : **Lattice diffusion**
- Diffusion tail : **GB diffusion**
- Concentration increase near the bottom interface : diffusion from the interface

↳ Significant lattice diffusion at 650°C .

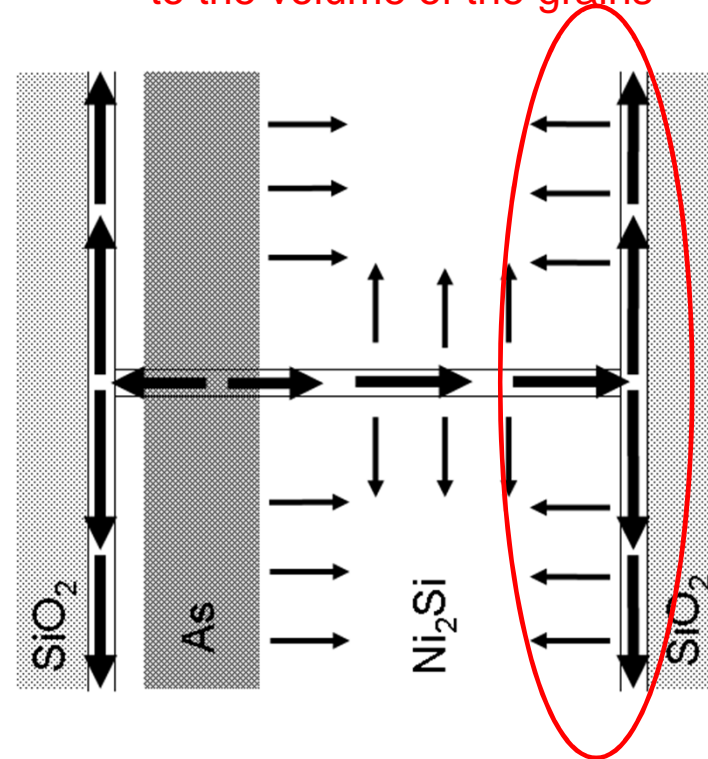
↳ Profile shape explained by diffusion along the GBs and interfaces + slow lattice diffusion

As diffusion in Ni₂Si: T = 650°C



- Flattening and broadening of the implantation peak : **Lattice diffusion**
- Diffusion tail : **GB diffusion**

Diffusion from the bottom interface to the volume of the grains

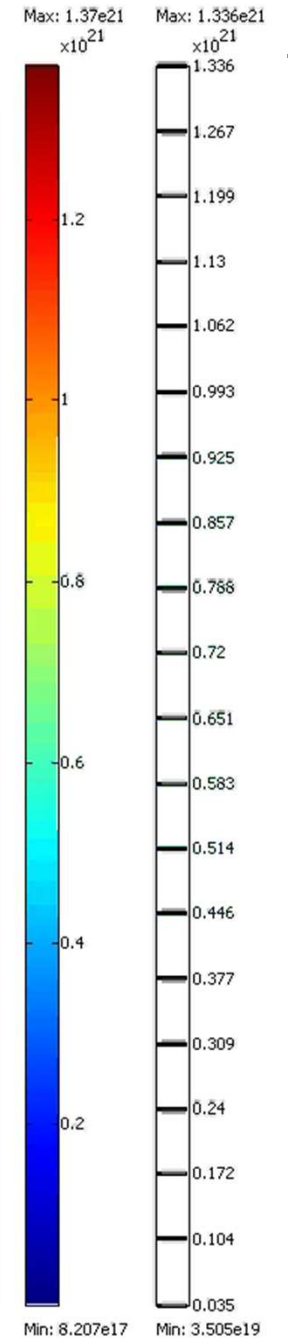
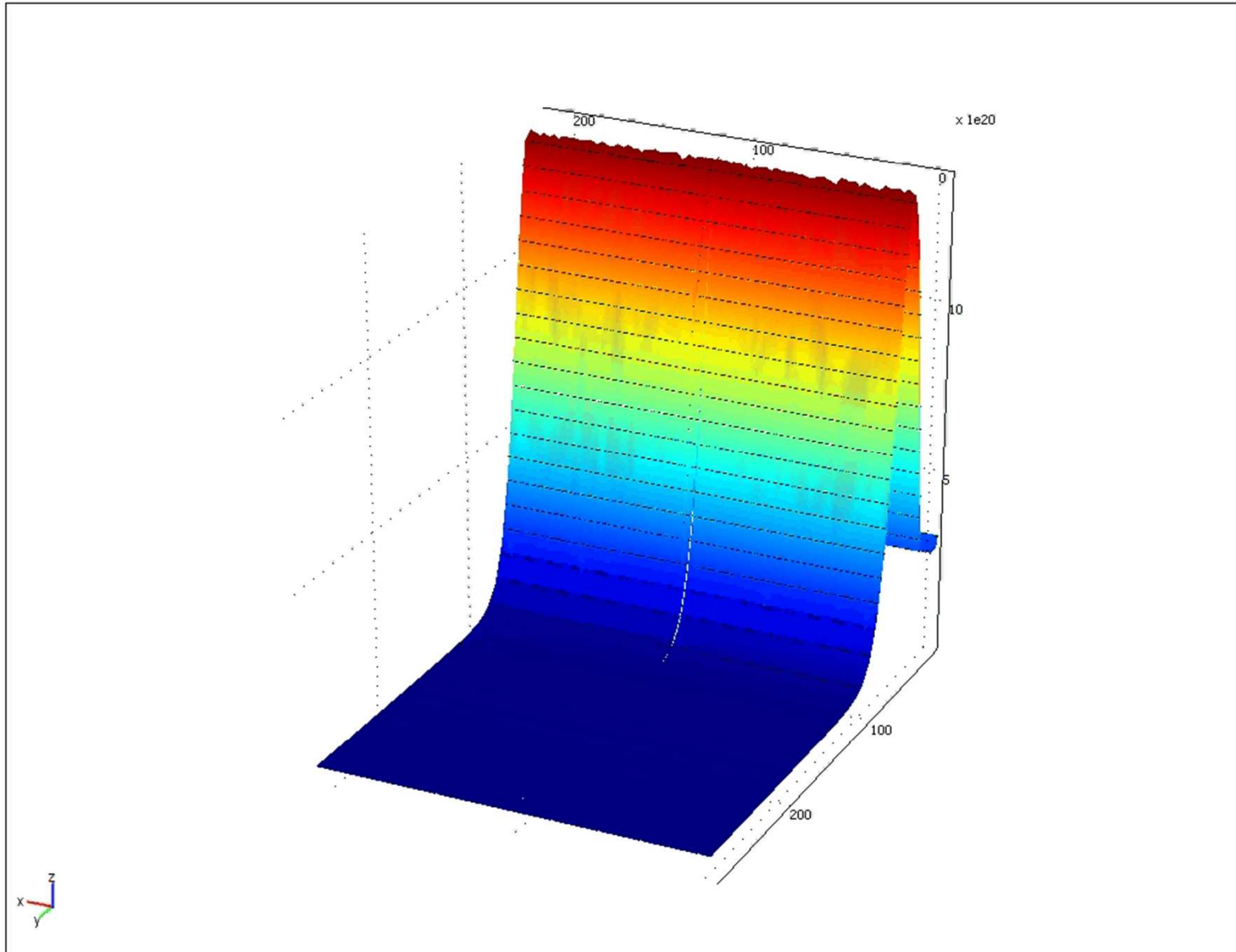


Profile shape explained by diffusion along the GBs and interfaces + slow lattice diffusion

Time=0

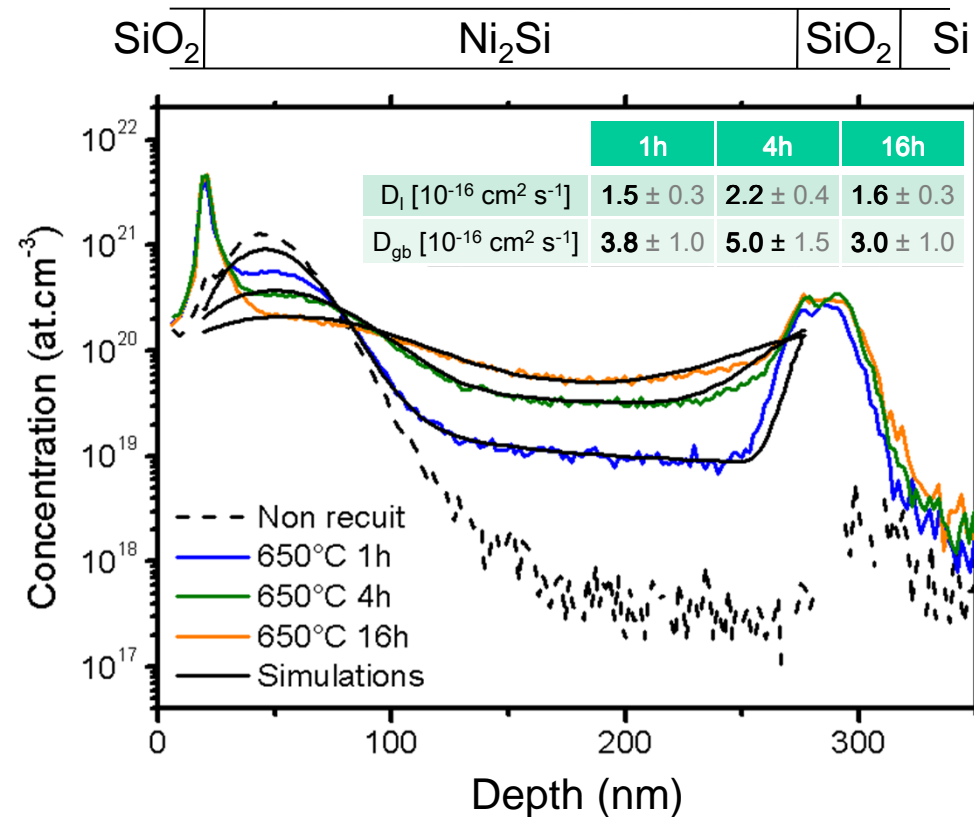
Surface: Concentration, c [mol/m³] Height: Concentration, c [mol/m³] Contour: Concentration, c [mol/m³] Height: Concentration, c [mol/m³]

Deformation: Diffusive flux, c [mol/(m²·s)]



Comparison to SIMS depth profiles

- Simulations can accurately fit the measured profiles
 - Concentration is usually overestimated close to the bottom interface : D_i is set to a too high value
 - The peaks at the interfaces are not observed
- Effect of segregation + SIMS matrix effects – not taken into account
- Measured coefficients do not depend on time
- no transient enhanced diffusion



Model can fit the measured profiles → measurements at different temperatures...

Heat treatment at different temperatures: $T^\circ = 550$ to 700°C , $t = 1$ to 186 h

- Lattice diffusion: $D_{0l} = 1.5 \cdot 10^{-1} \text{ cm}^2/\text{s}$, $E_l = 2.7 \text{ eV} \pm 0.1$
- GB diffusion: $D_{0gb} = 1.8 \cdot 10^5 \text{ cm}^2/\text{s}$, $E_{gb} = 3.1 \text{ eV} \pm 0.15$

→ higher activation energy for GB diffusion !!!

2 possible explanations :

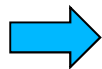
1. Impurities segregated at the GBs could occupy the sites used by As for diffusion.

Low $T^\circ =$ High impurity segregation

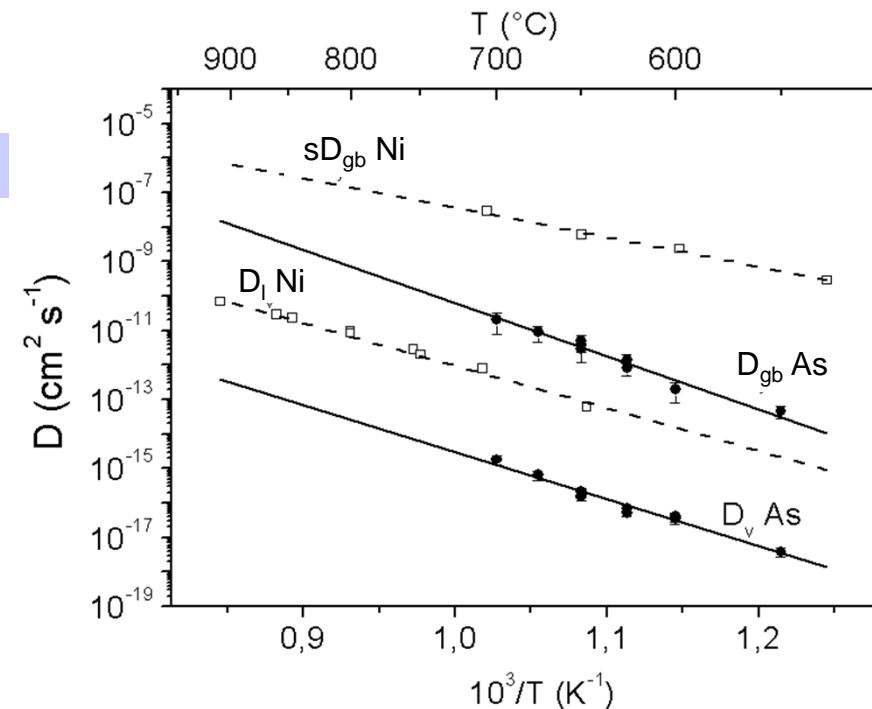
➡ Low As diffusion

2. If Si segregates in Ni₂Si GBs, As would diffuse in Si rich GBs

As in Ni₂Si lattice : 2.7 eV
As in Ni₂Si GB : 3.1 eV

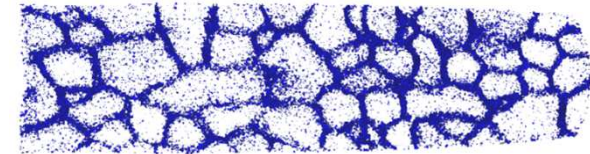
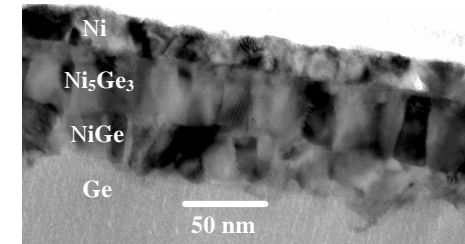


Ni in Ni₂Si lattice : 2.6 eV
As in Si GBs : 3.4 eV



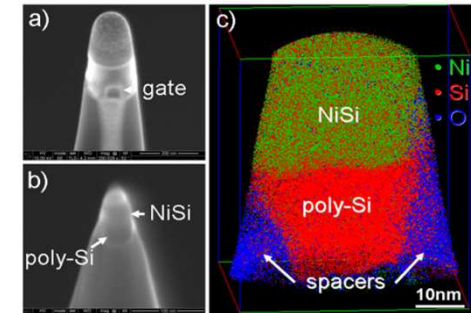
1. Basis of reactive diffusion

- Diffusion couple / thin films
- First stages : nucleation and lateral growth
- Deal & Grove law and silicides
- Sequential growth –simultaneous growth
- Role of grain boundaries



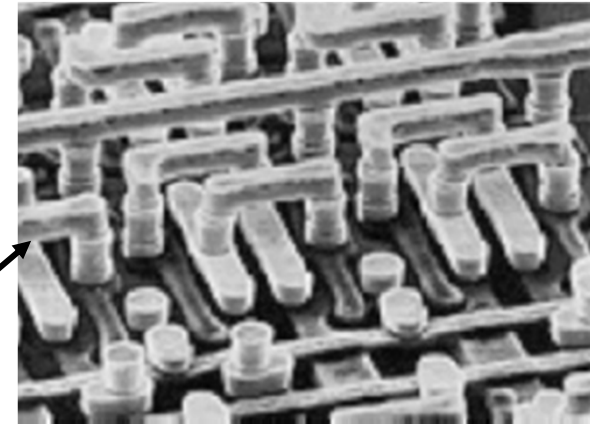
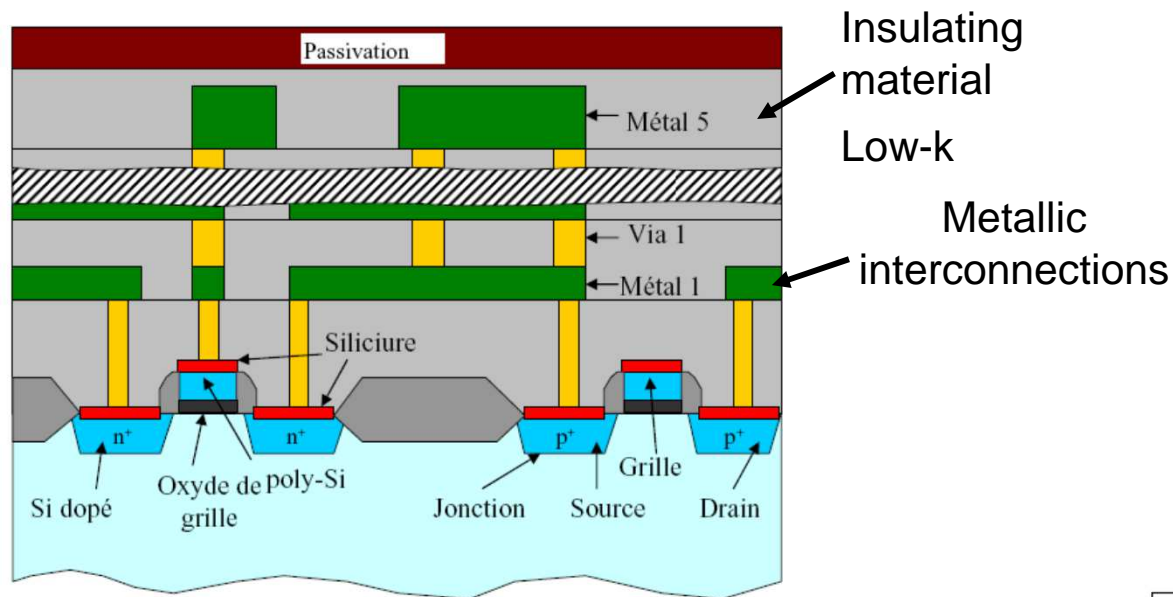
2. Typical example : silicides in microelectronics

- Contacts in microelectronics
- Analyses of transistors by atom probe
- Nucleation and alloy effect



3. Challenges

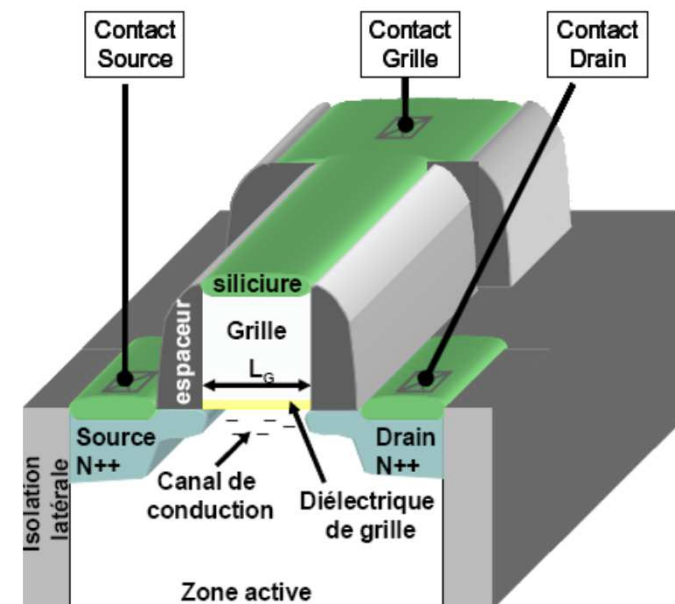
- Encroachment / transient phase
- Nucleation / texture / stress
- Doping / defects / dislocation / precipitation / redistribution
- Nanoelectronics



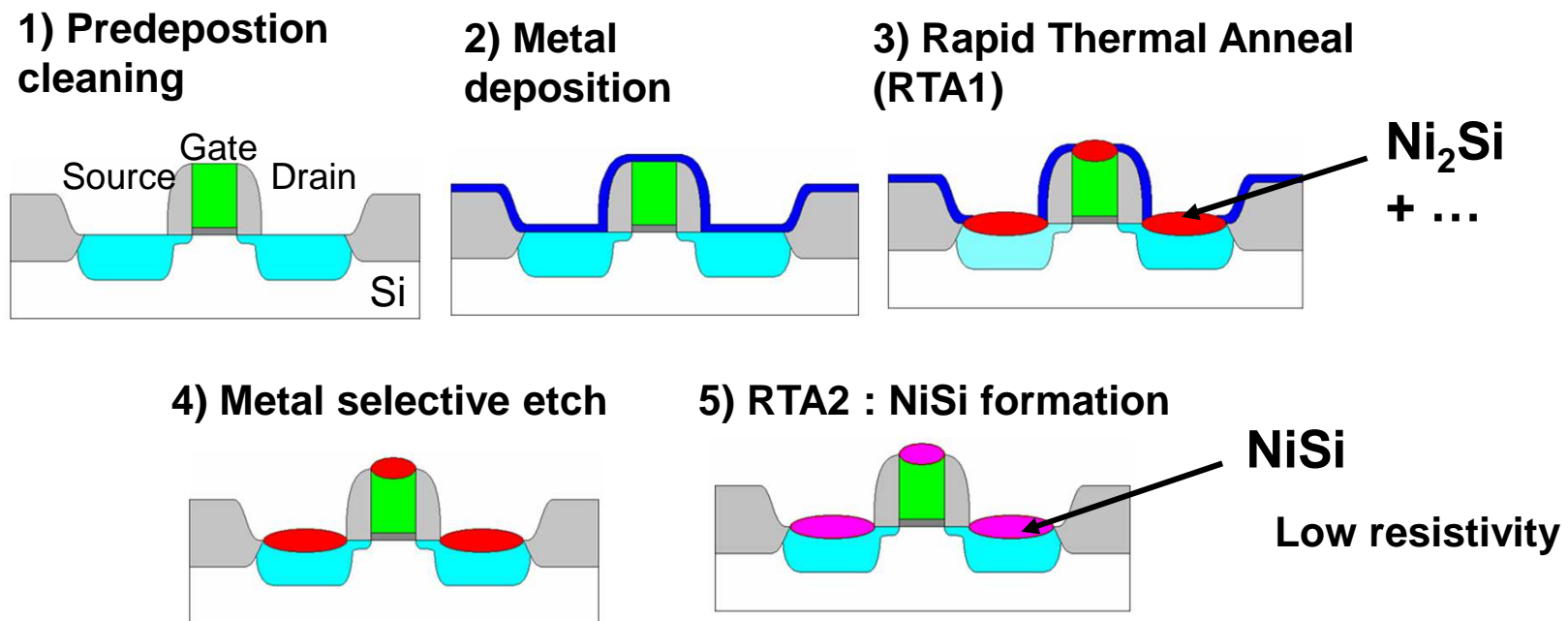
Maier, G., *Progress in Polymer Science*, 26 (2001) 3

The integrated circuit is made of :

- active components
- metallic interconnections:
 - Silicides used to reduce the contact resistance
 - Silicide = larger contact area = low contact resistance
 - Allows to spread the current along the active parts of the component

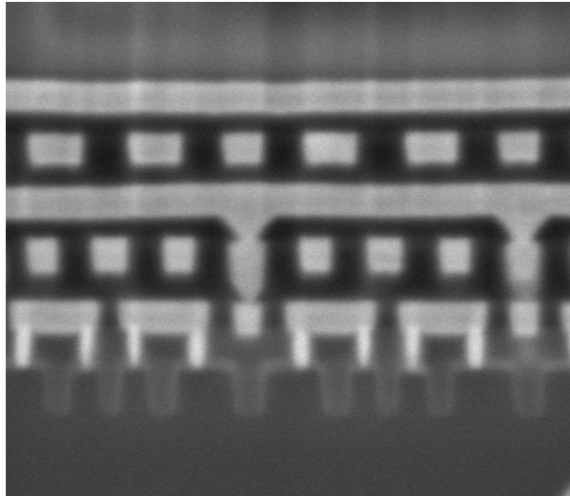


NiSi is now used for the high performance applications

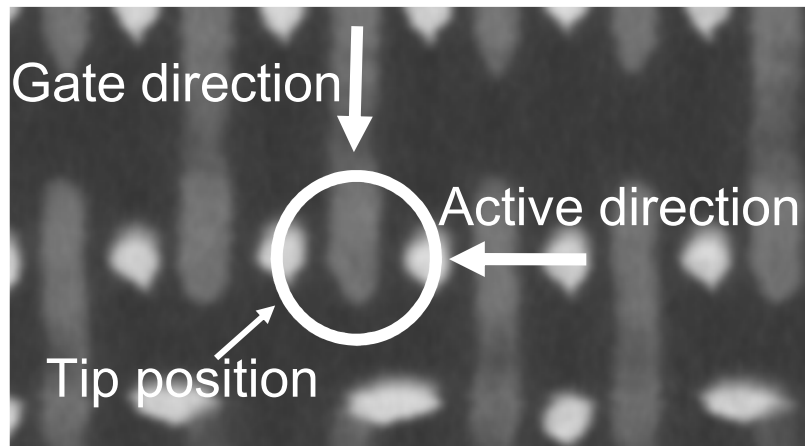


↳ Ni_2Si forms first and then reacts with Si to form NiSi

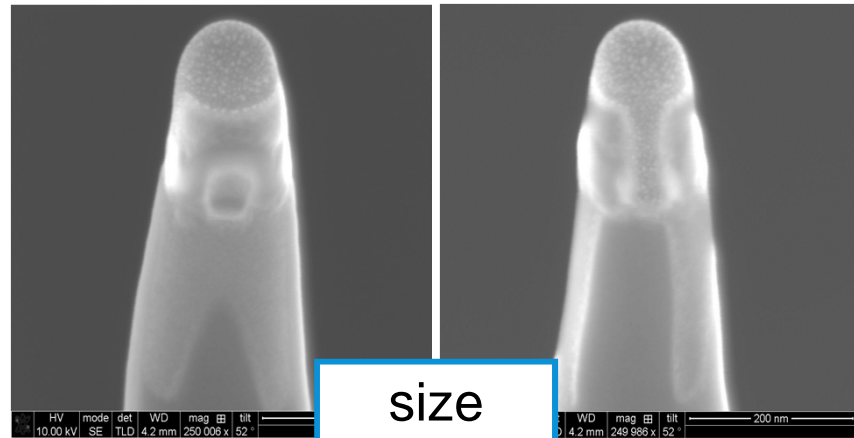
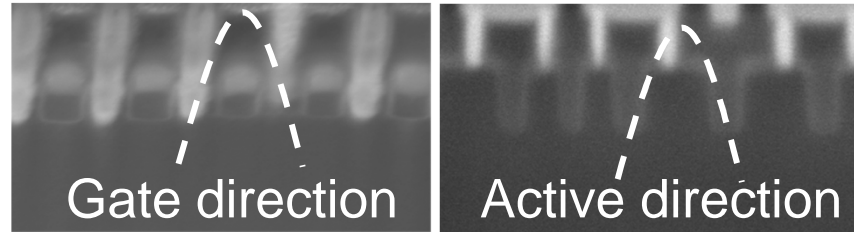
SRAM



Plan view of deprocessed SRAM

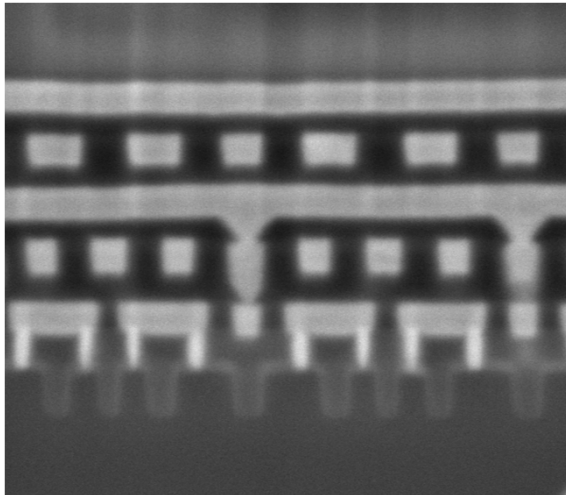


Cross section

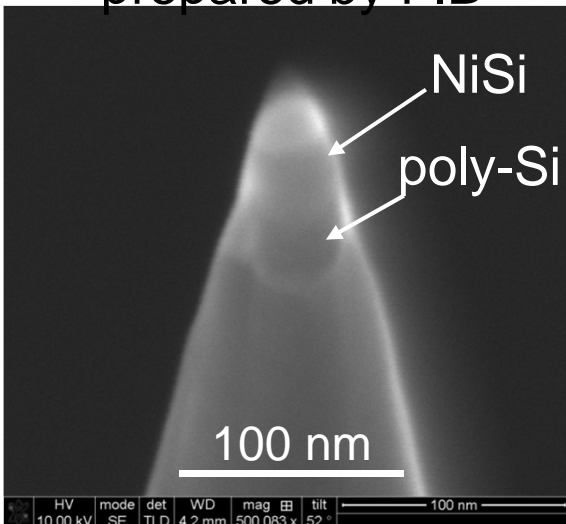


[F. Panciera et al. APL 99 (2011)]

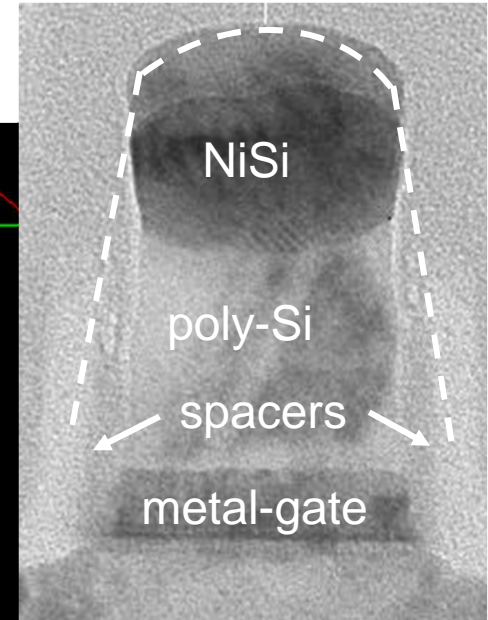
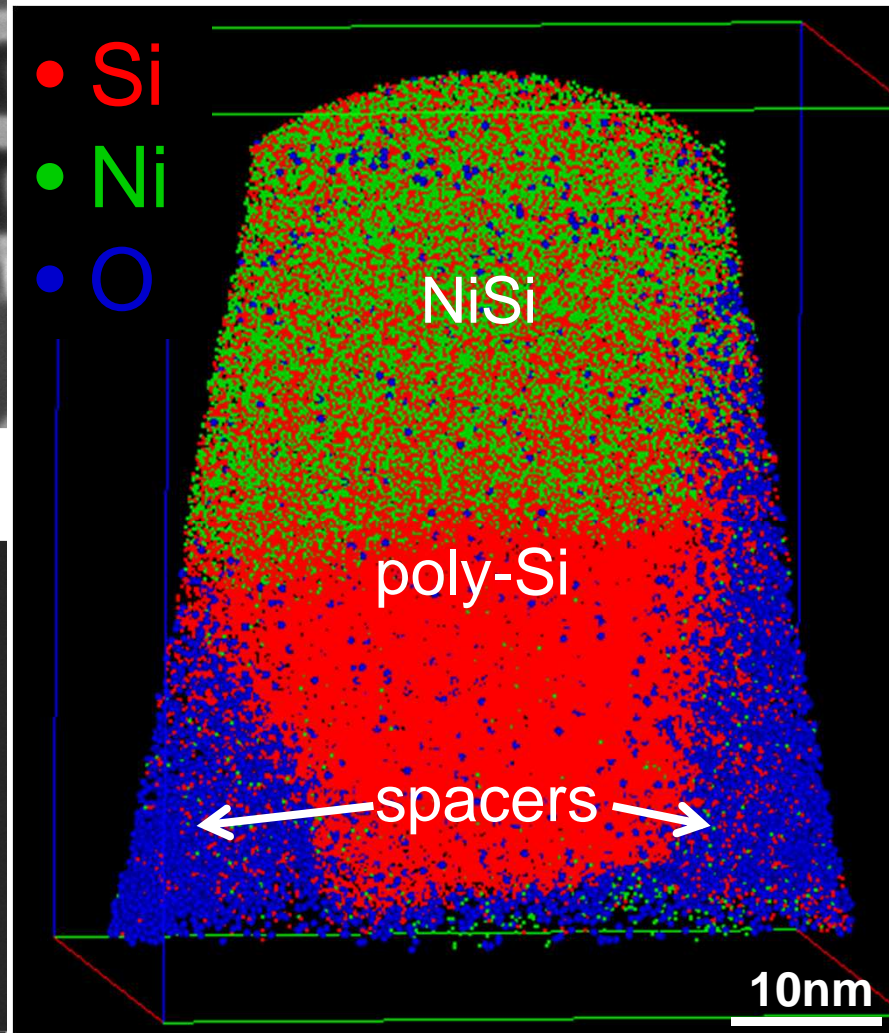
SRAM



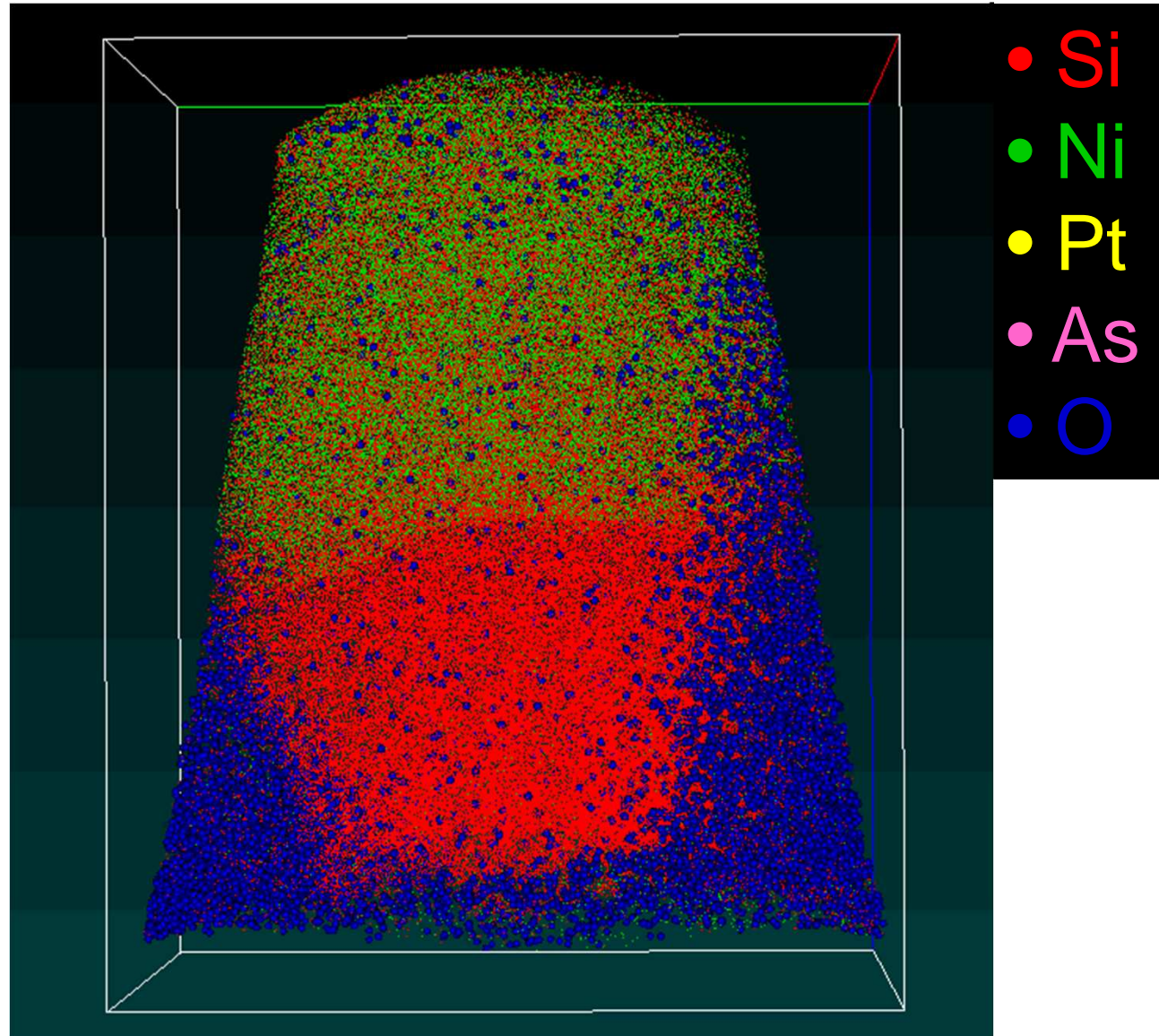
APT specimen prepared by FIB



3D reconstructed volume



LEAP 3000X HR
($\lambda = 532$ nm)

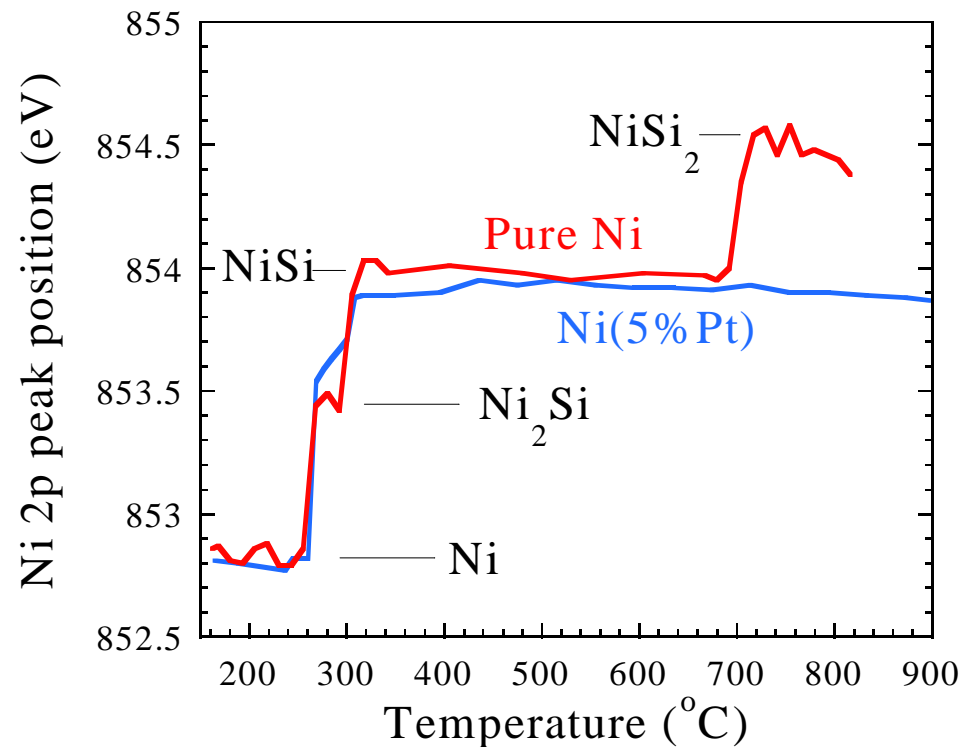


- Pb: Interface NiSi/Si not stable
- Nucleation of NiSi₂ à 750°C
- Alloy element (Co, Pt, Pd, Au)

Experiments

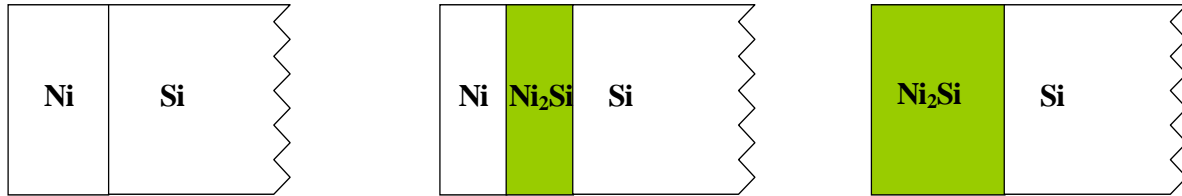
- Deposition of thin films of Ni with 5 at% Pt on Si substrate.
- Characterization by RBS, SIMS, Micro Raman, XRD, XPS, ...

→ The addition of Pt increase the thermal stability of NiSi

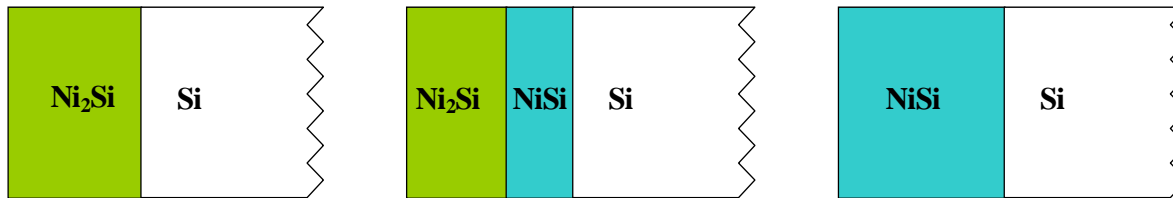


In situ annealing in a XPS system
(Mangelinck et al, APL, 1999)

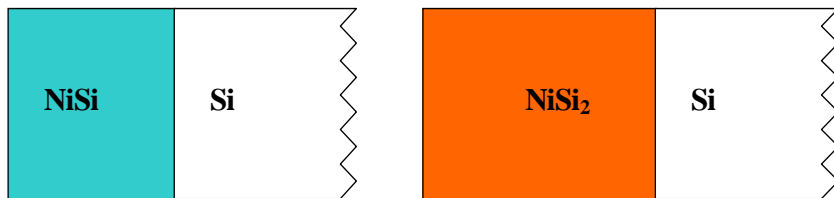
Reaction of Ni thin films with Si



T ~ 200-300°C : formation of Ni₂Si : DIFFUSION controlled , Ni diffusion

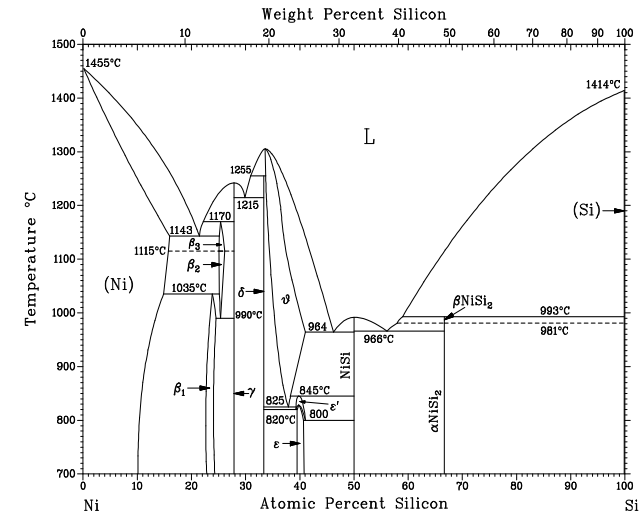


T ~ 250-350°C : formation of NiSi : DIFFUSION controlled , Ni diffusion



T ~ 750-800°C : formation of NiSi₂ : NUCLEATION controlled , Ni diffusion

Submicron thickness
 - sequential formation
 - 3 phases: Ni₂Si, NiSi, NiSi₂
 - diffusion of Ni



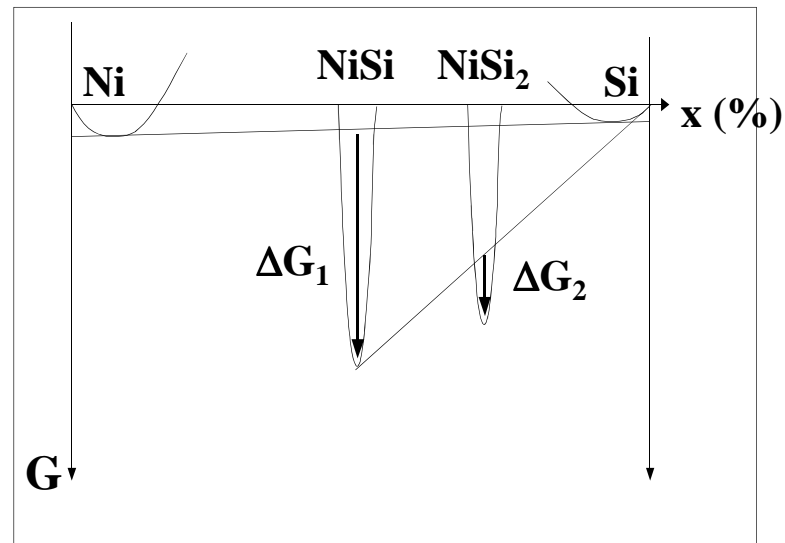
≠ Ni-Si phase diagram

Nanometric thickness

[Lavoie et al Microelec. Eng. 2003]

- new phases?
- “transient” phases
- kinetic ?

→ Better understanding of the growth mechanisms



NiSi, PtSi: same structure, misfit < 15%

- Total solid solution
- Decrease of Gibbs energy ($G_f(\text{NiSi})$)

Mixing entropy

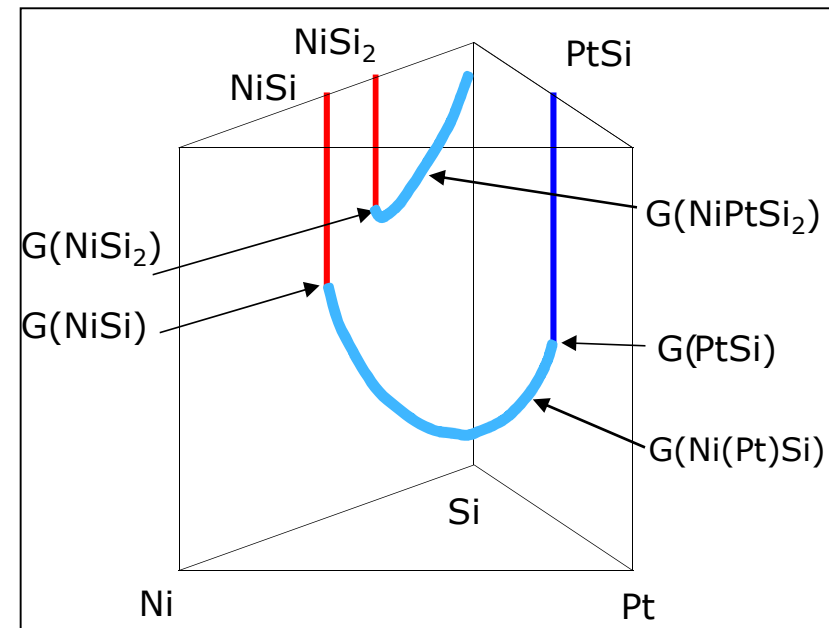
→ Nucleation barrier

→ Ni(Pt)Si is now used in microelectronics

Nucleation barrier # $\Delta\sigma^3/\Delta G^2$

$$\Delta\sigma = \sigma(\text{NiSi}_2/\text{Si}) + \sigma(\text{NiSi}_2/\text{NiSi}) - \sigma(\text{NiSi}/\text{Si})$$

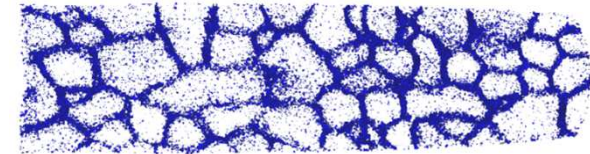
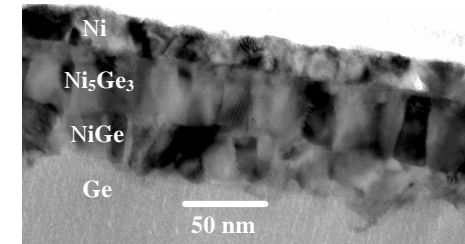
$$\Delta G = G_f(\text{NiSi}_2) - G_f(\text{NiSi})$$



Schematic of the Gibbs free energy for the ternary system Ni-Pt-Si

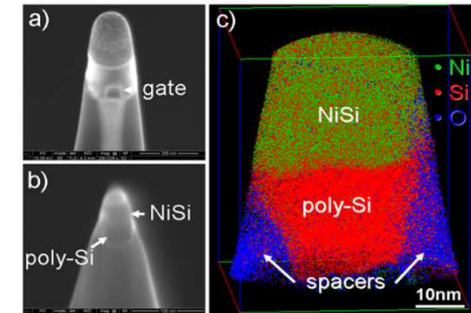
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2. Typical example : silicides in microelectronics

- Contacts in microelectronics
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- Nucleation and alloy effect

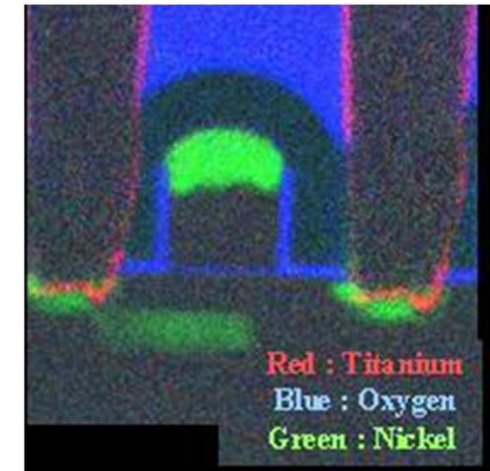
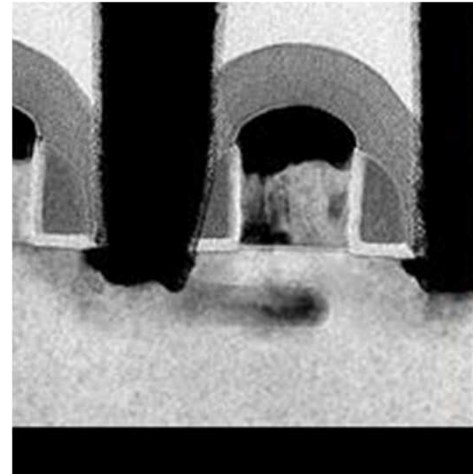
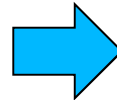
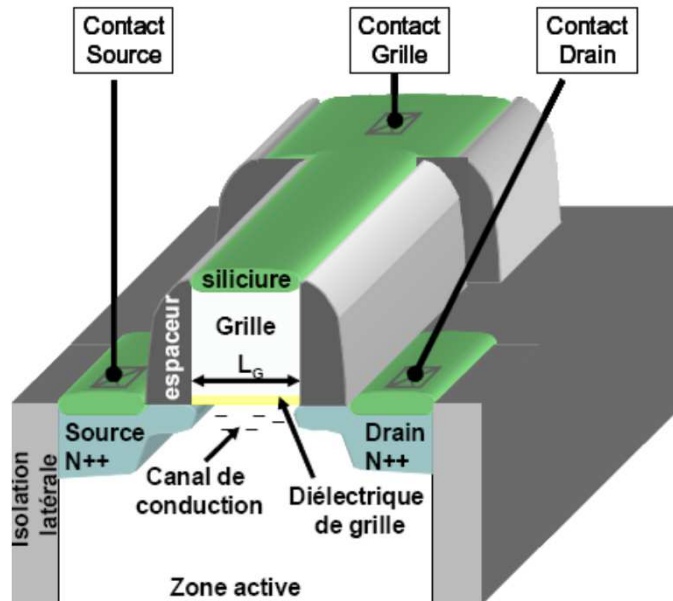


3. Challenges



- Encroachment / transient phase
- Nucleation / texture / stress
- Doping / defects / dislocation / precipitation / redistribution
- Nanoelectronics

Ni(Pt)Si is now used for the high performance applications



Ideal structures



real structures

Reliability problems: → Encroachment or ‘abnormal diffusion’

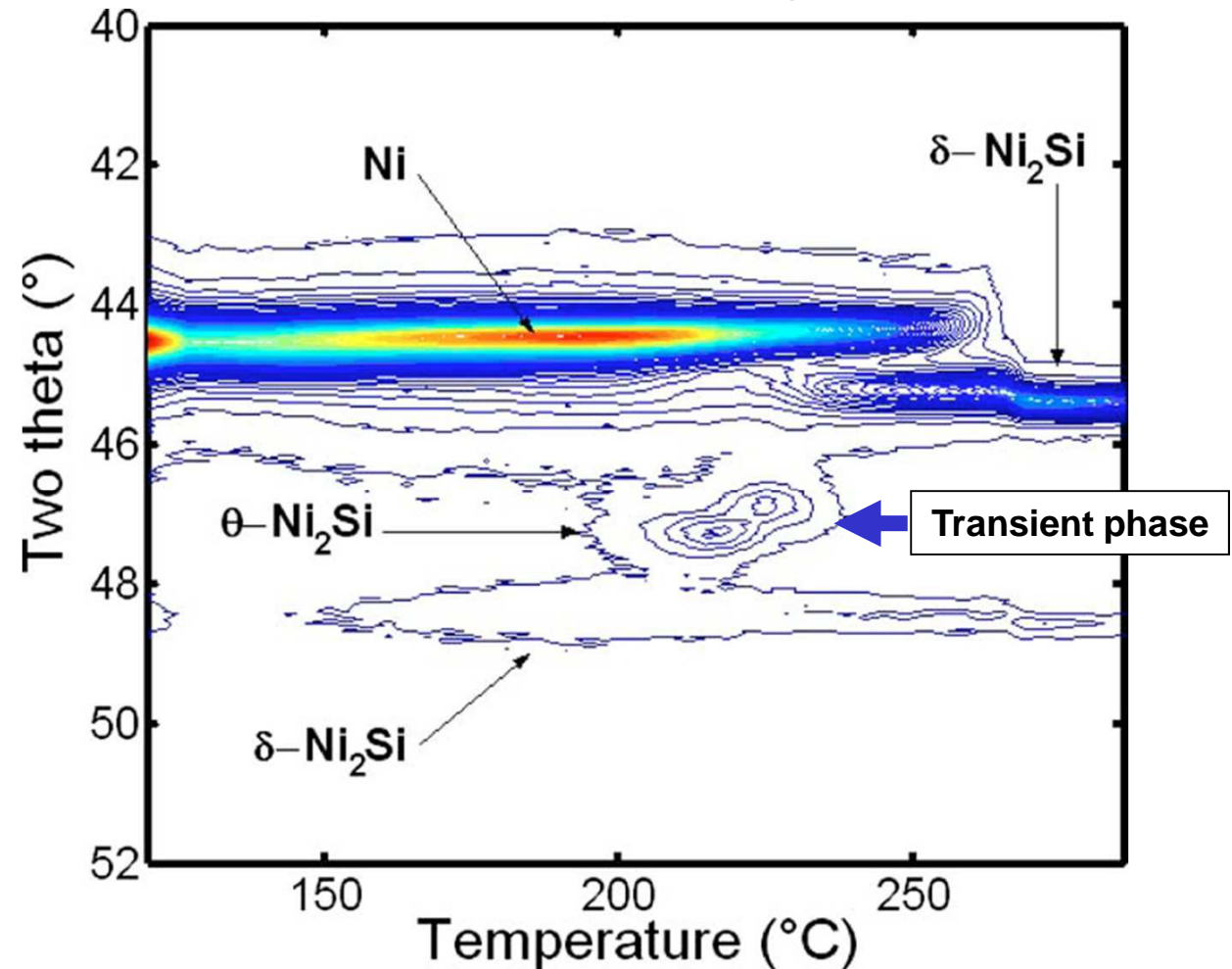
Parameters that lead to encroachment

- Stress/confinement
- Low thickness < 20nm
- Doping: N doping → As
- Effect of Pt

→ transient phase ?

Lavoie et al, *Microelec. Eng.* 2003Mangelinck et al, *APL*, 2009**Transient phase (TP) :****= unusual behaviour**

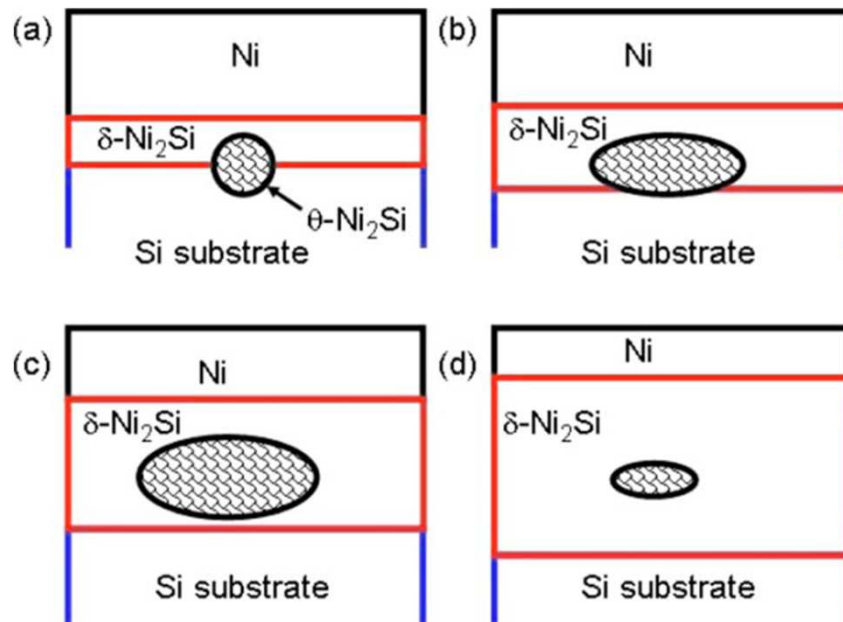
- The end members (Ni and Si) are still present
- δ -Ni₂Si is growing
- The transient phase (θ -Ni₂Si) grows and shrinks during the growth of δ -Ni₂Si

≠ sequential growth**≠ simultaneous growth**

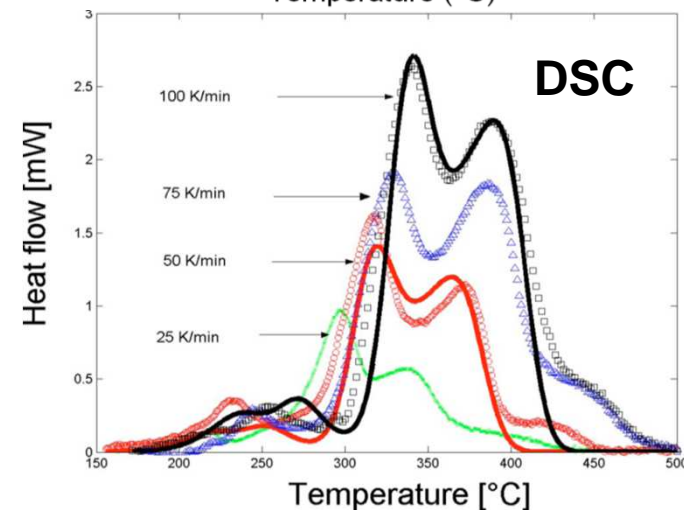
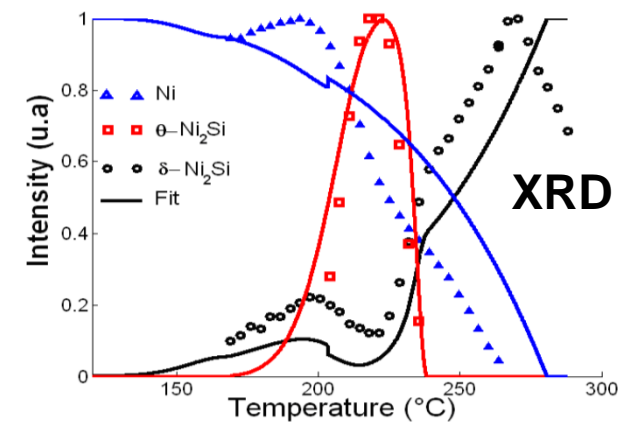
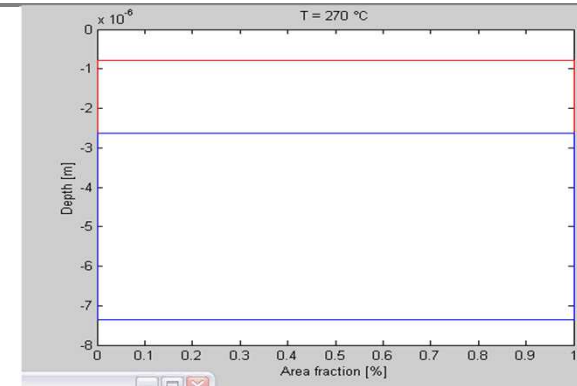
In situ X ray diffraction of the Ni thin film
reaction with a Si substrate

Possible scheme for transient phase (TP) :

- 1 – lateral and normal growth of δ -Ni₂Si
- 2 – nucleation and lateral growth of TP together with normal growth of δ -Ni₂Si
- 3 – shrinkage of TP when TP is enclosed by δ -Ni₂Si.

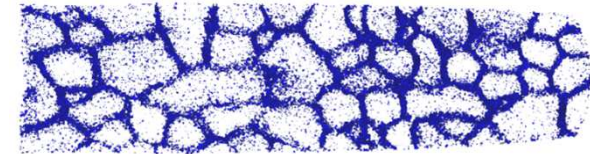
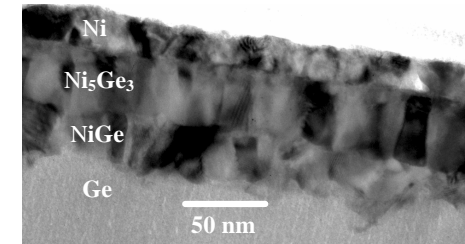


Model used to simulate DSC and XRD.



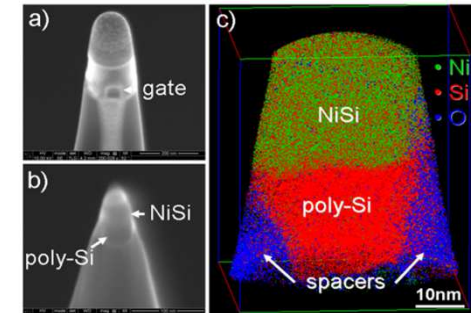
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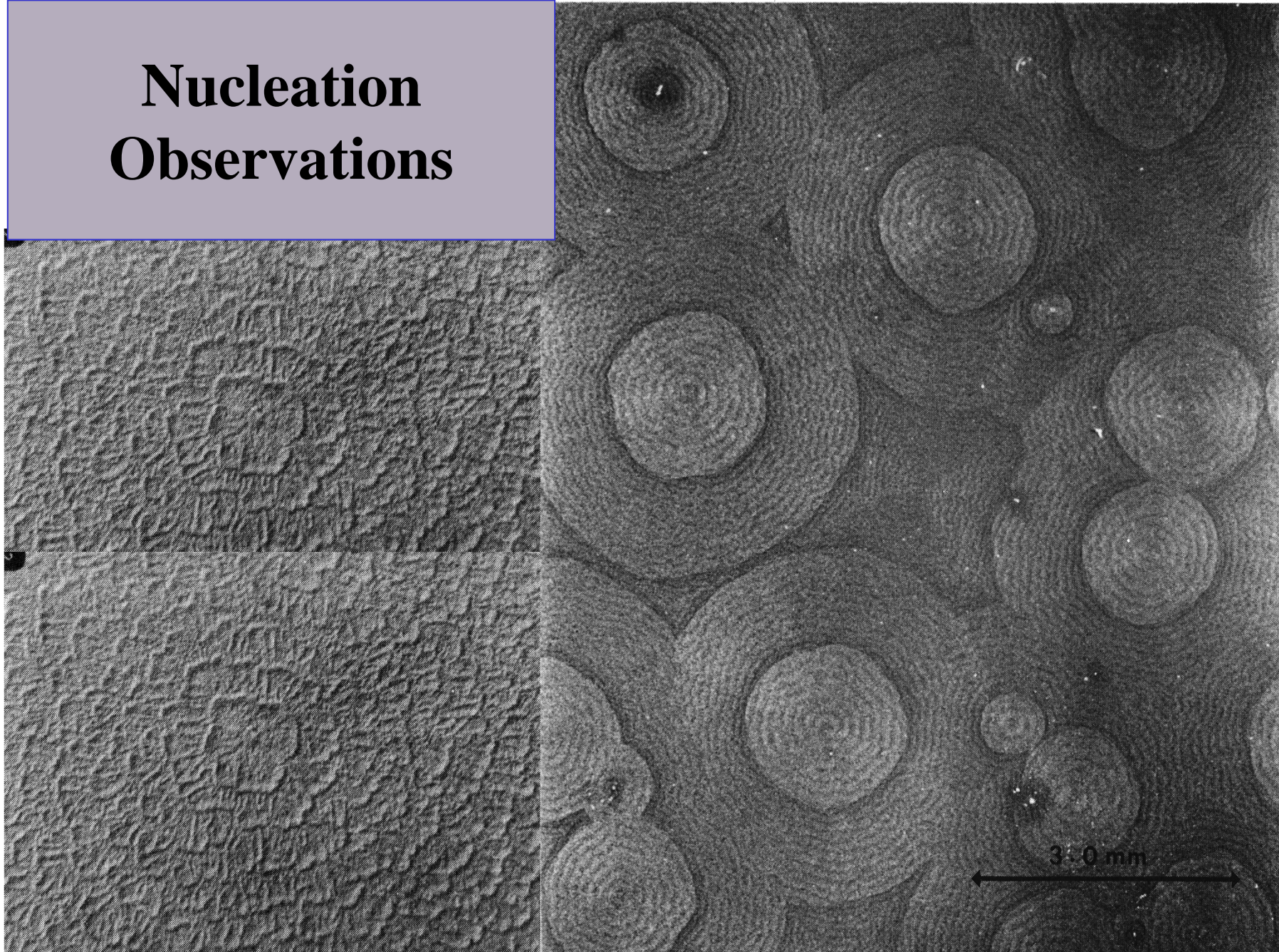


3. Challenges

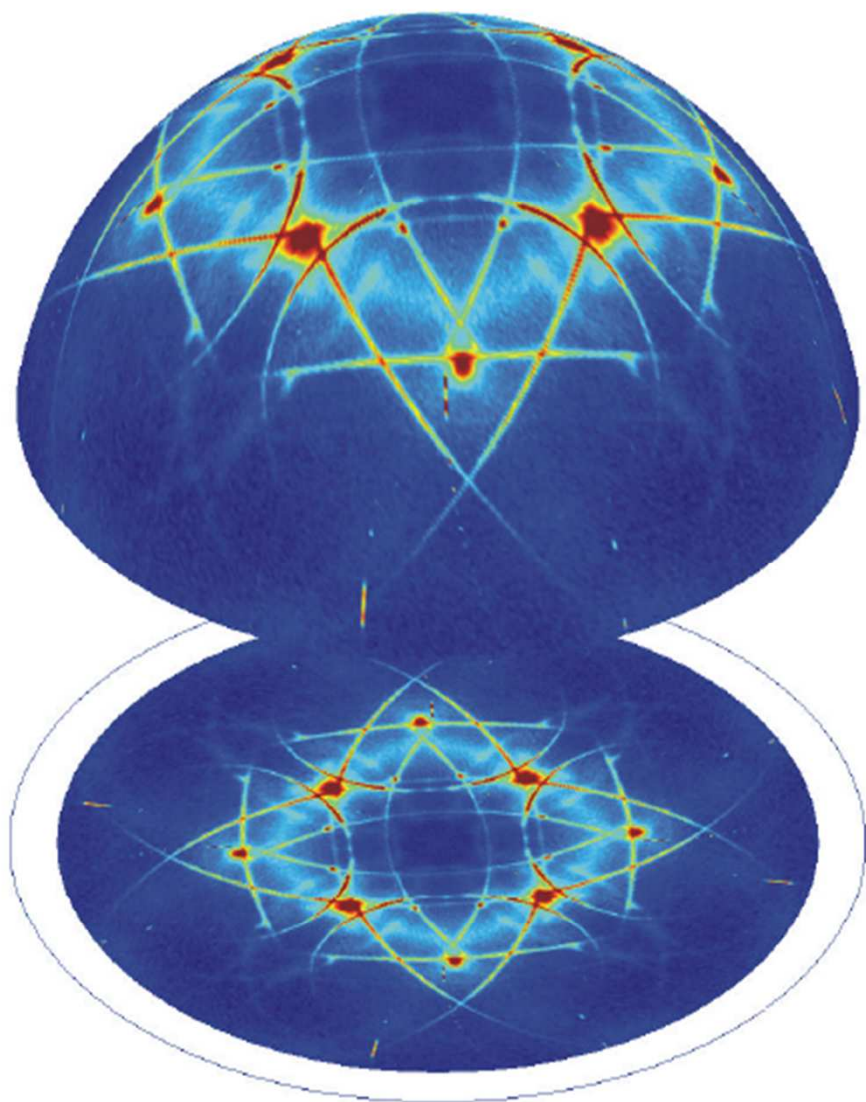
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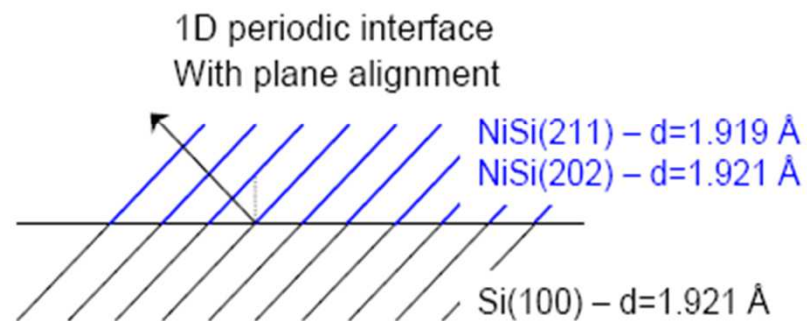
Nucleation Observations



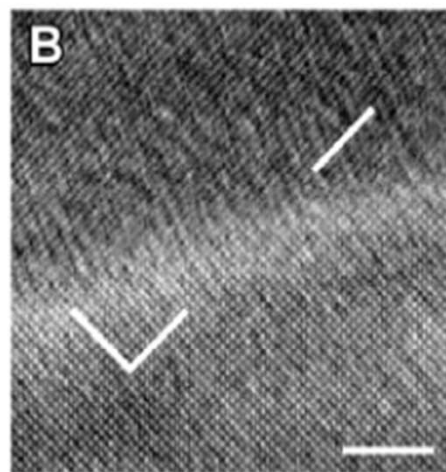
TEXTURE - Axiotaxy



(103) Pole Figure



C. Detavernier, et. al., Nature 426, 641 (2003)

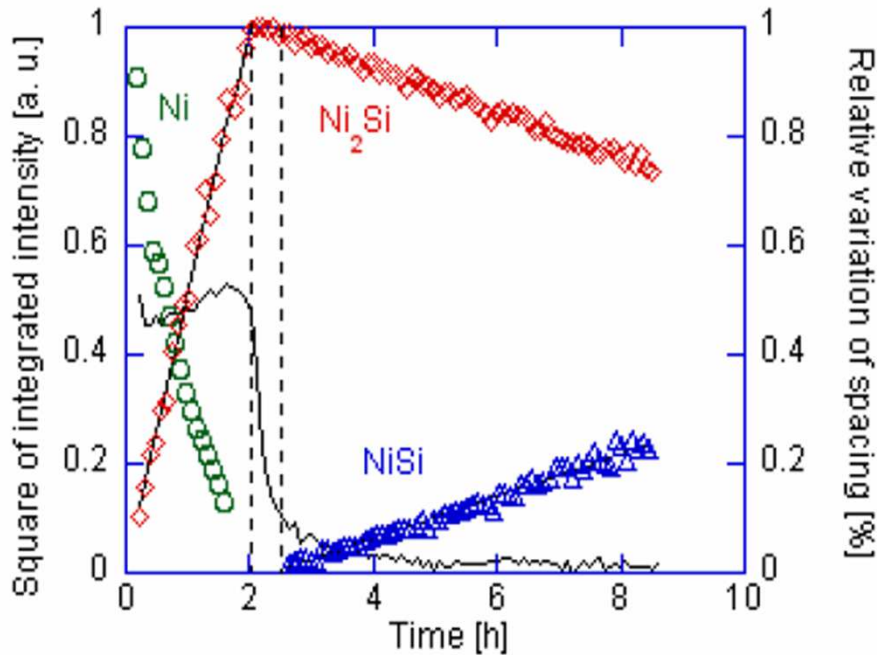


E.A. Stach,
National Center for Electron Microscopy,
LBNL, Berkeley, CA

IBM
Research

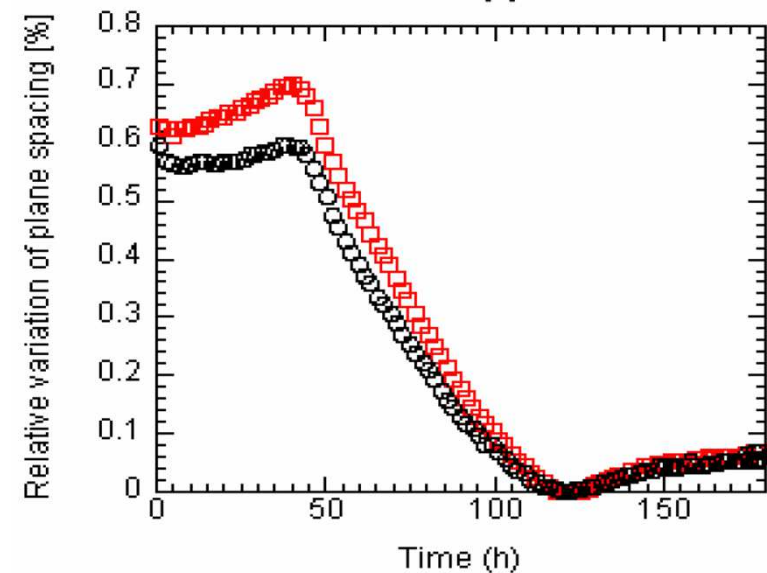
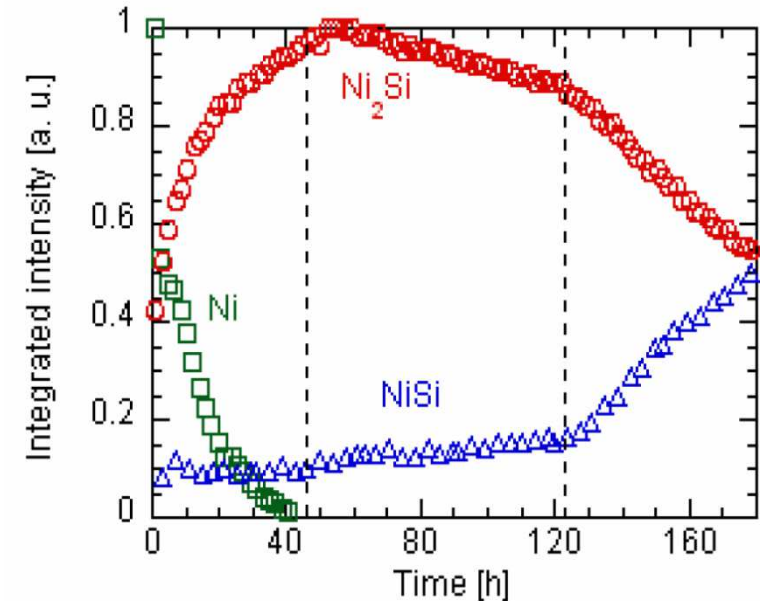
Christian Lavoie, Sept. 22 2006

Mangelinck et al, APL, 2008

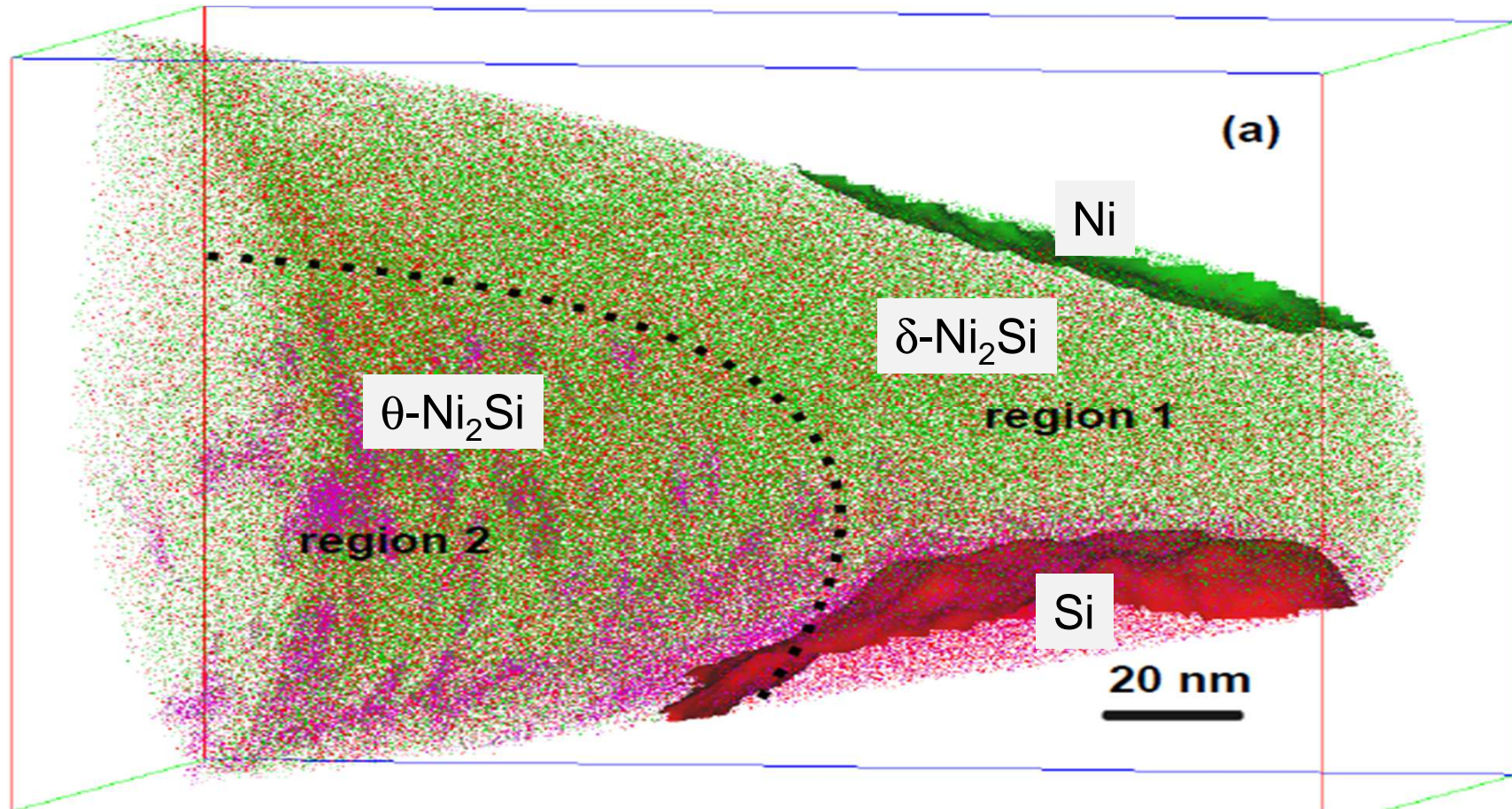


In situ XRD isotherm (210°C) of 50 nm Ni on amorphous Si

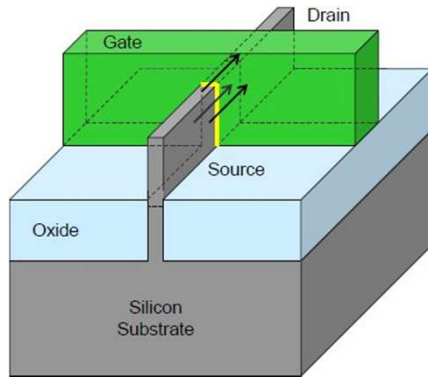
- Time delay (end of Ni_2Si - start of NiSi)
 - Nucleation ?
 - Strain/stress ?
- Ni(5%Pt)/(100)Si
 - Simultaneous growth of $\text{Ni}_2\text{Si}/\text{NiSi}$
 - Nucleation → **stress**



In situ XRD isotherm (210°C) of 50 nm Ni(5%Pt) on (100) Si

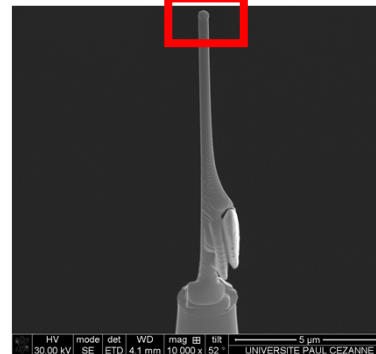
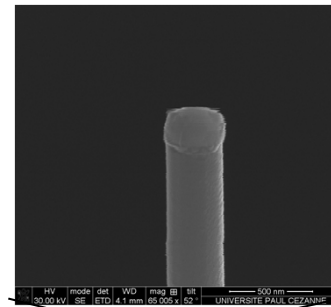
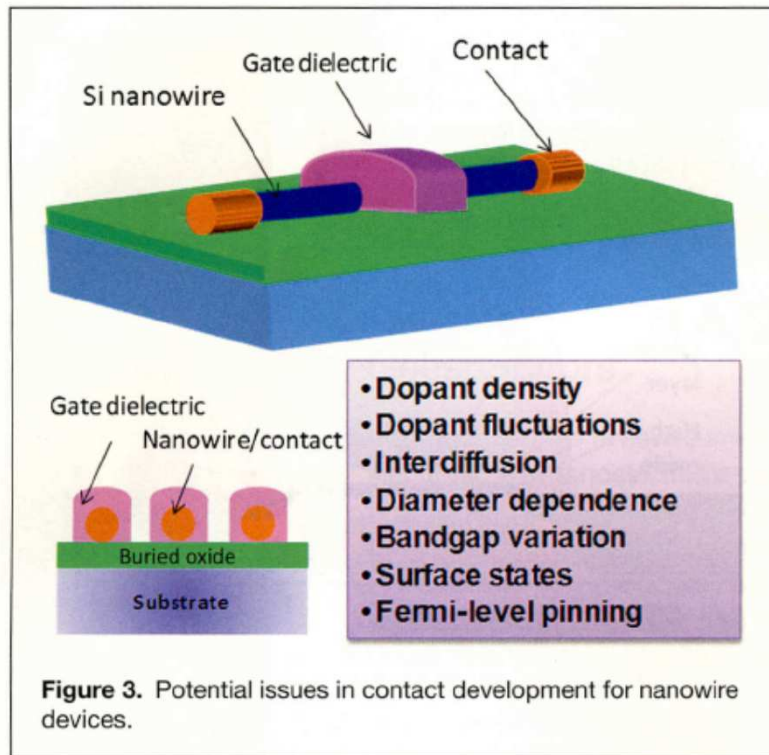


Precipitation of arsenic rich clusters at the grains boundaries of $\theta\text{-Ni}_2\text{Si}$

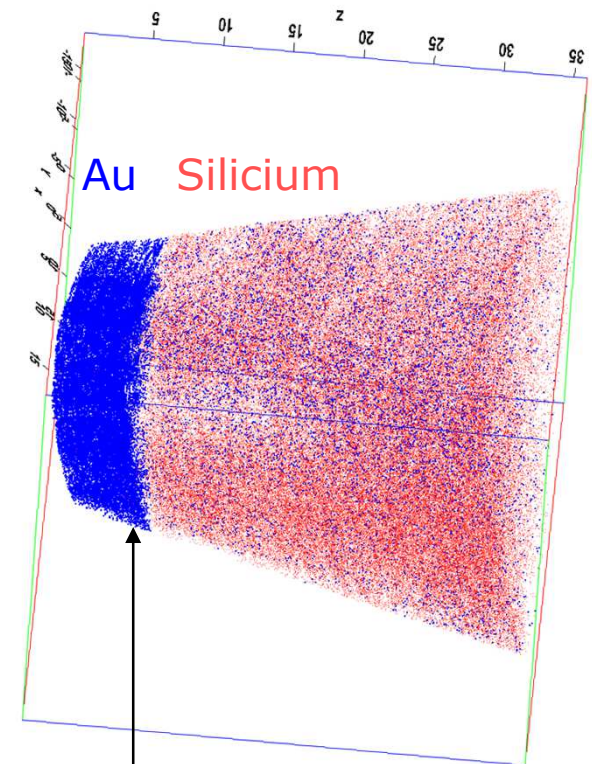


Tri-gate device

3D structures are replacing 2D planar structure in nanoelectronics



FIB Dual Beam

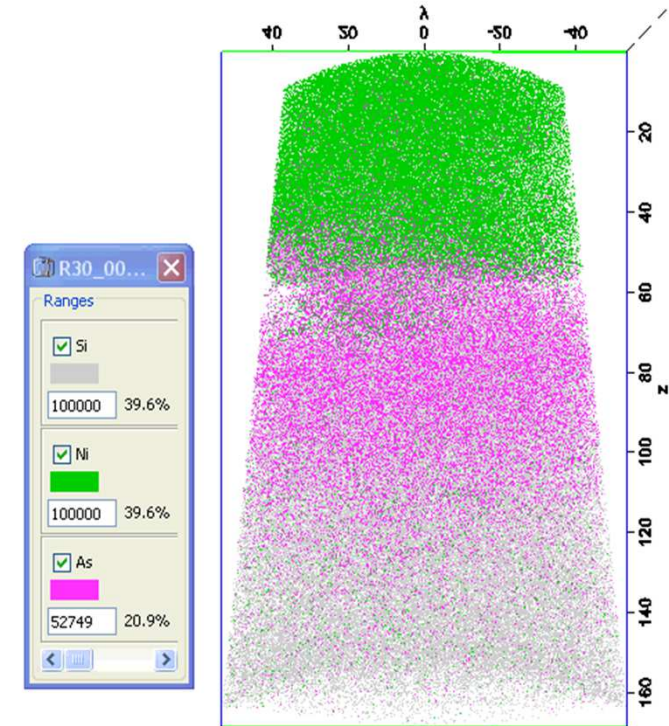


Interface Au / Silicon

mechanisms of growth, doping, metallization → metallurgy

Reactive diffusion in thin films

- Lateral and normal growth kinetics
- Nucleation
- Role of stress
- Role of grains boundaries / interface
- Sequential/simultaneous formation
- Transient phase / metastable phase
- Epitaxy/texture



The flying dislocation

Contact in microelectronics

- Salicide = complex interplay between defects, dopants and silicides
- Less than 10nm thickness in industry
 - ultra thin silicide (reactive diffusion? surface science?)
- Analysis of transistors by APT
- Thermal stability on Si, SiGe, Ge
- 3D structure / nanowires....