



Heat Treatment Processes

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LIQUID METAL
PROCESSING



SOLIDIFICATION



FORMING



HEAT
TREATMENT

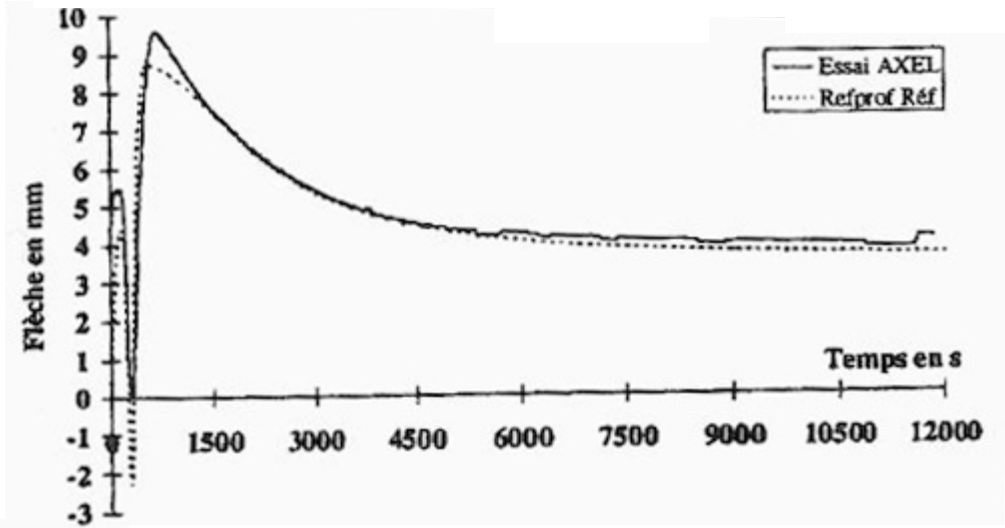
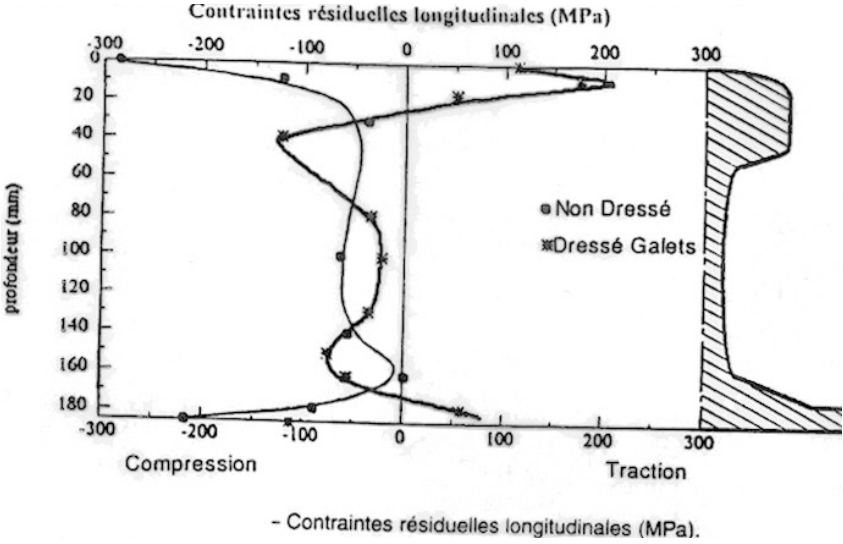
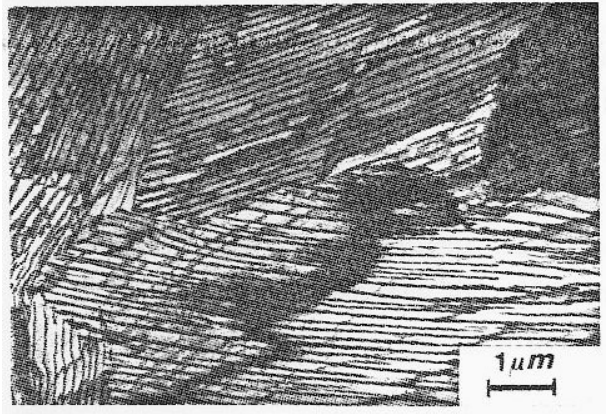
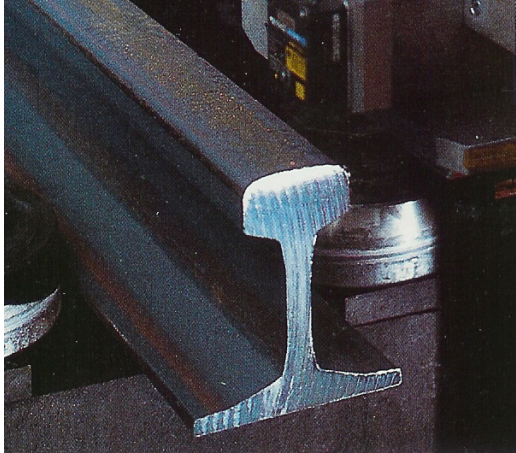


Microstructures
Mechanical properties



WELDING
CUTTING ...

Problem of heat treatment of metallic alloys



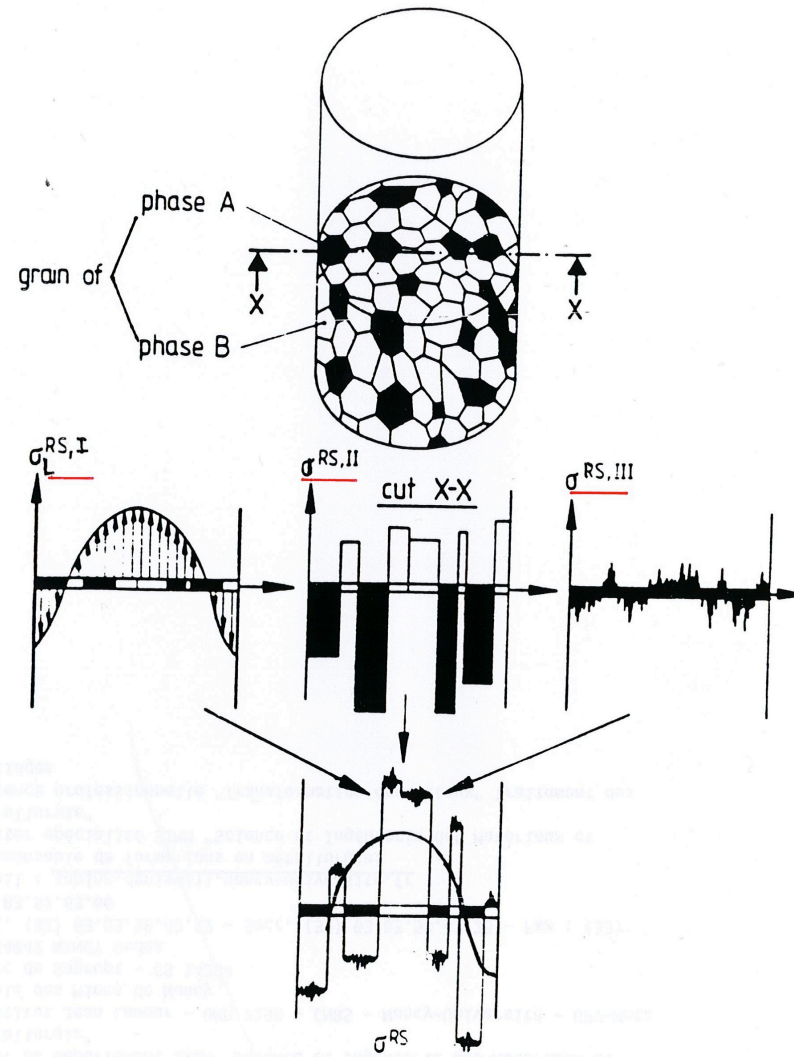


Illustration of the different stress orders

(E. Macherauch, K.H. Kloos 1986)

Heat treatment of metallic alloys

Control of microstructures

→ desired mechanical properties

Control of thermal gradients

→ avoid/limit deformations

→ avoid/limit residual stresses (quenching)

or obtain desired residual stress distributions
(surface heat treatments, thermochemical treatments)

Better optimization = modelling and numerical simulation

Development of internal stresses during cooling

qualitative approach (Rose et Bühler 1968)

Origin of internal stresses :
Heterogeneous deformations

- thermal

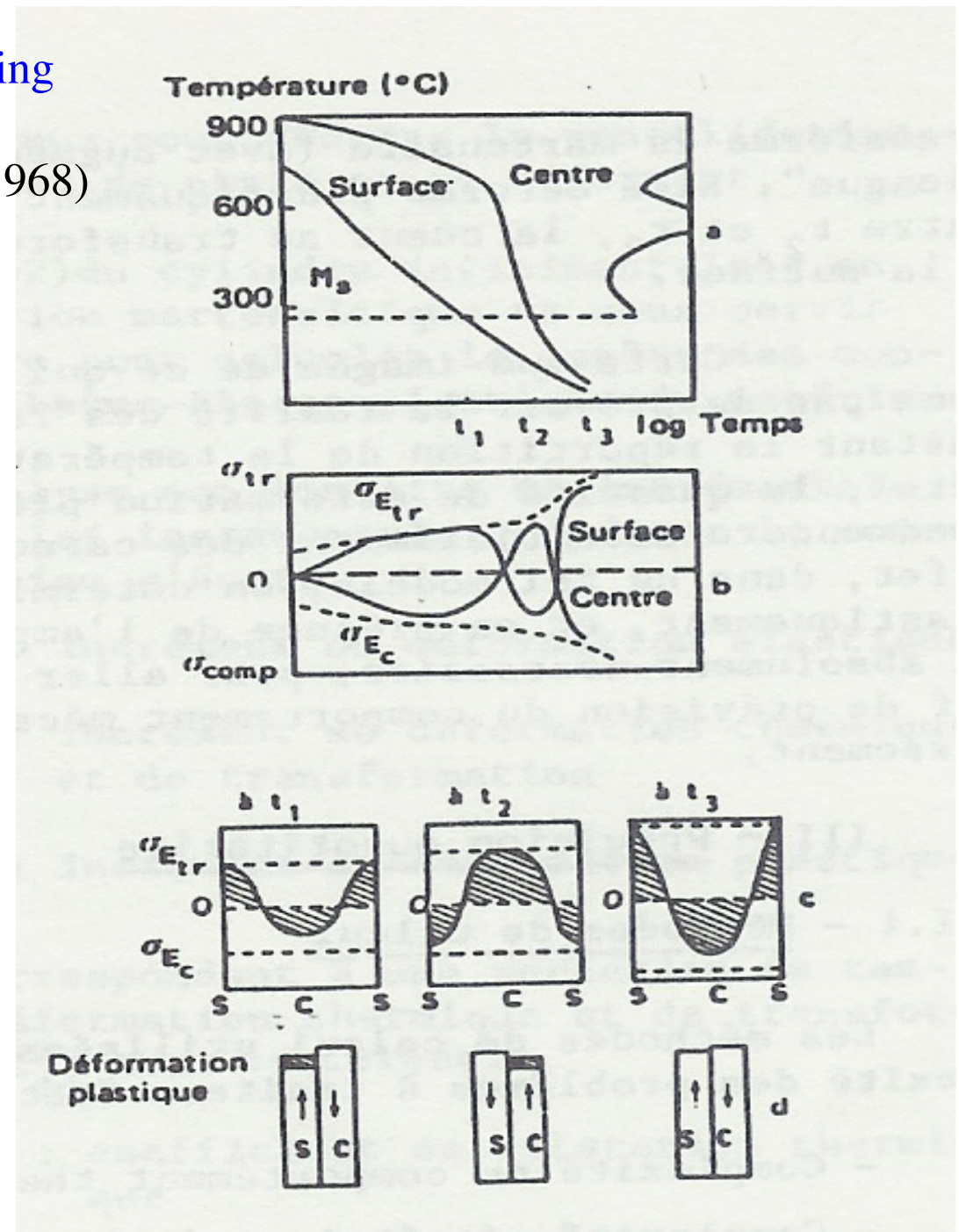
- phase transformations

Origin of residual stresses and deformations :

Heterogeneous deformations

- plastic

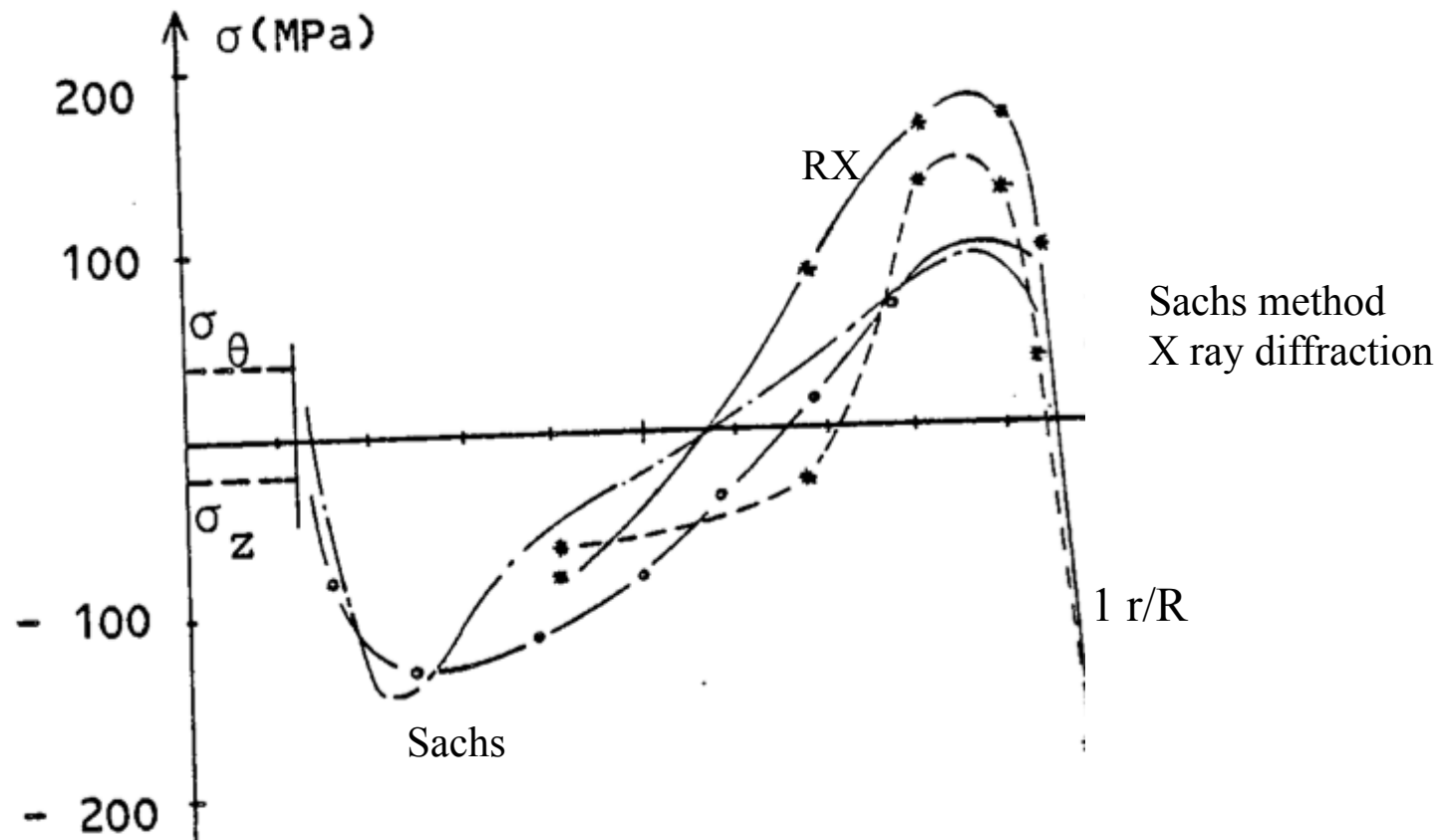
- phase transformations



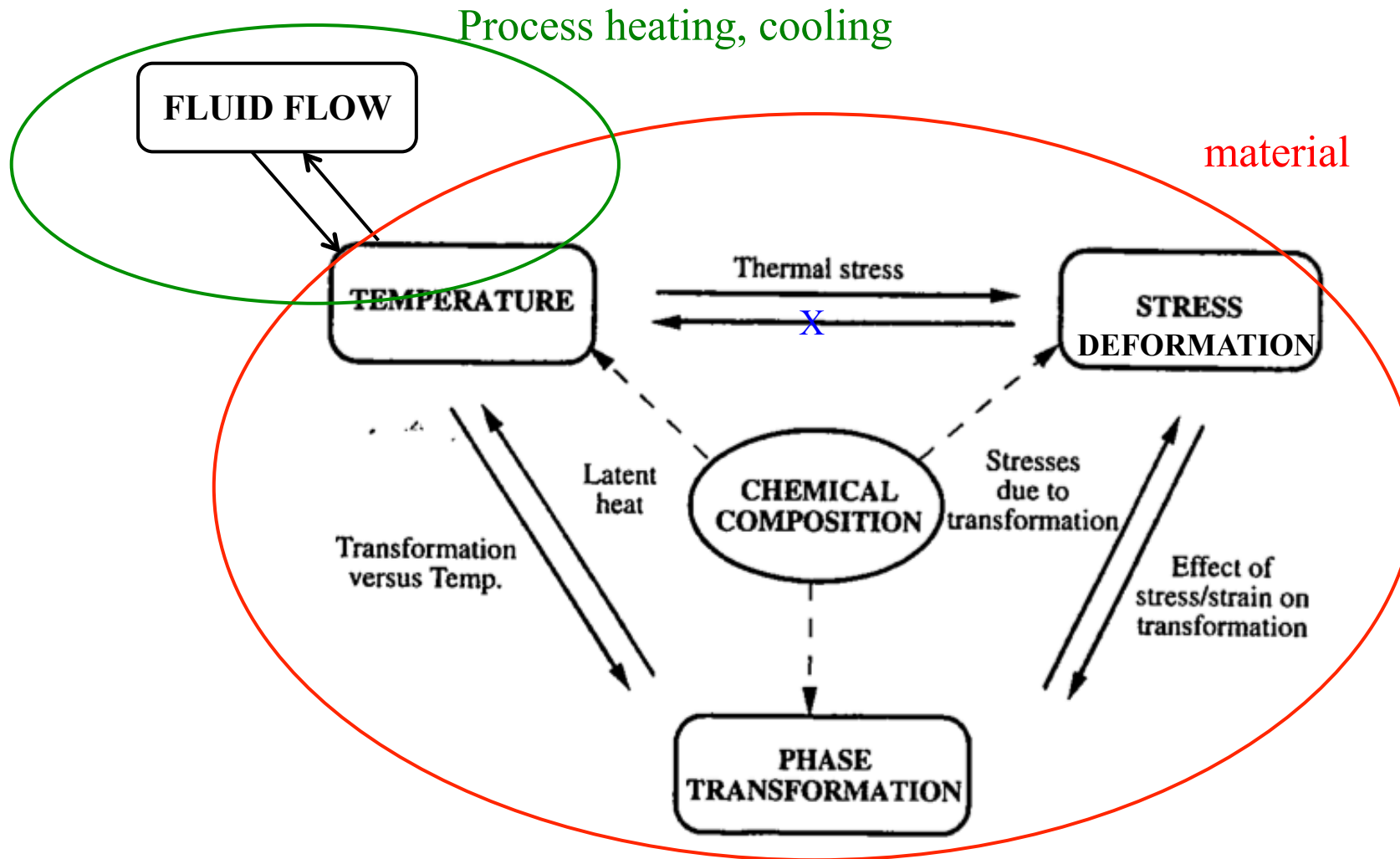
Example of experimental residual stress profiles

Water quenching of a 60NiCrMo11 steel cylinder

(diameter 40mm, length 120mm)



Fluid – thermal – metallurgical – mechanical couplings in heat treatment



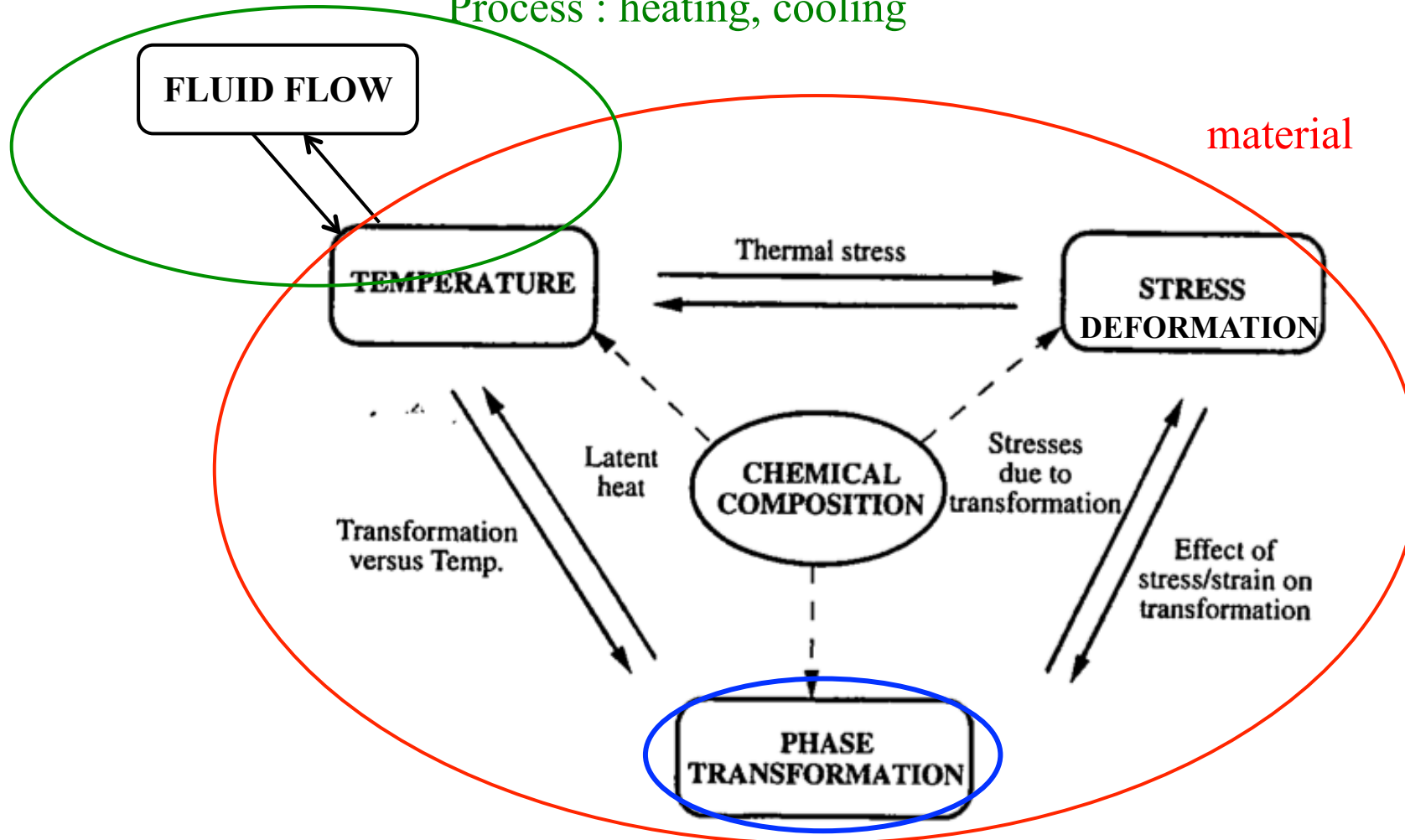
1972...prediction of thermal stresses (Al alloys, Ti alloys)

≈ 1980... main advances including phase transformations in steels

≈ 2000... precipitation Al alloys - consideration of fluid flow (gaz quenching)

Fluid – thermal – metallurgical – mechanical couplings in heat treatment

Process : heating, cooling



Overview of necessary experimental knowledge, present models, some open questions and example of results in steels

Modelling of phase transformation kinetics

(for the purpose of predicting internal stresses and deformations)

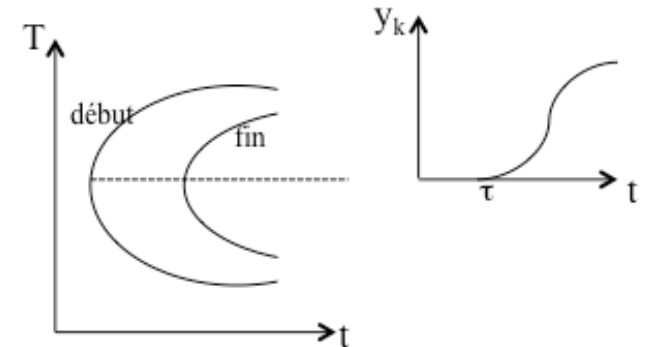
- **Global models (JMAK):** (1980 -1995)
 - Modelling anisothermal kinetics from CCT diagrams
 - Modelling kinetics from IT diagrams (additivity principle)

PHASE RC : prediction of kinetics during heating and cooling in steels from isothermal kinetics (additivity principle)

HEATING

IT diagram austenitization kinetics
carbon content of austenite and grain size

$$y_k = y_{\max k} (1 - \exp(-b_k t^{nk}))$$



COOLING : IT diagram

austenite → proeutectoid constituent
pearlite, bainite

. **incubation period : Scheil's method** $S = f_{\text{inc}} \sum \Delta t_i / \tau_i$

$$\tau_i = f(T, \text{grain size, comp., stress})$$

. **progress**

$$n, b = f(T, \text{grain size, comp., stress})$$

austénite → martensite : $y_m = y_\gamma [1 - \exp(-\alpha (M_s - T))]$

$$M_s = f(\text{grain size, comp., stress})$$

Hardness:

$$HV = \sum_{i,k} dy_k HV_k$$

$$HV_k = f(T_{\text{formation}}, \text{comp.})$$

Example : Gaz quenching of carburized cylinders

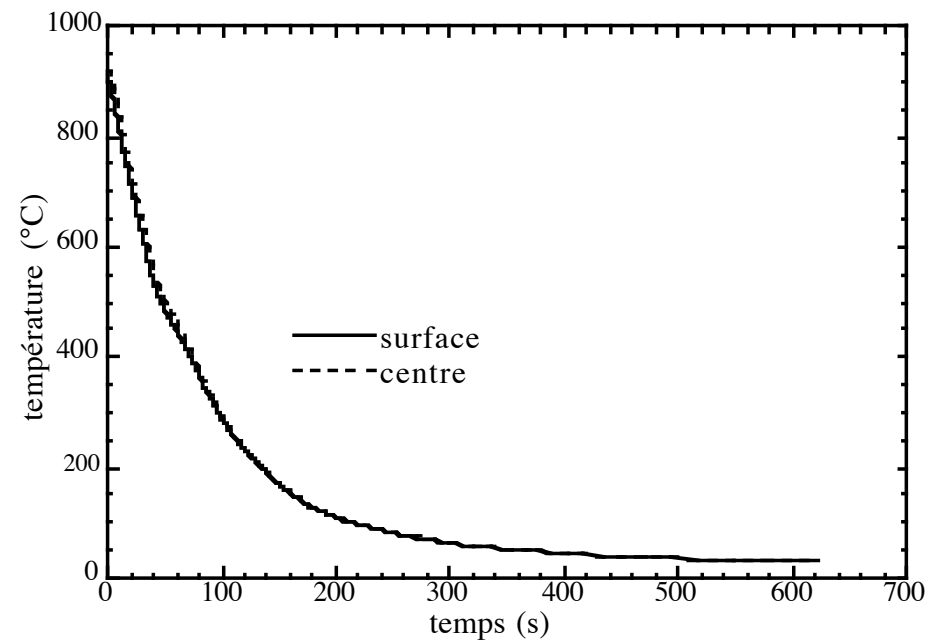
Cylinders diameter 16mm length 48mm

steel 27MnCr5

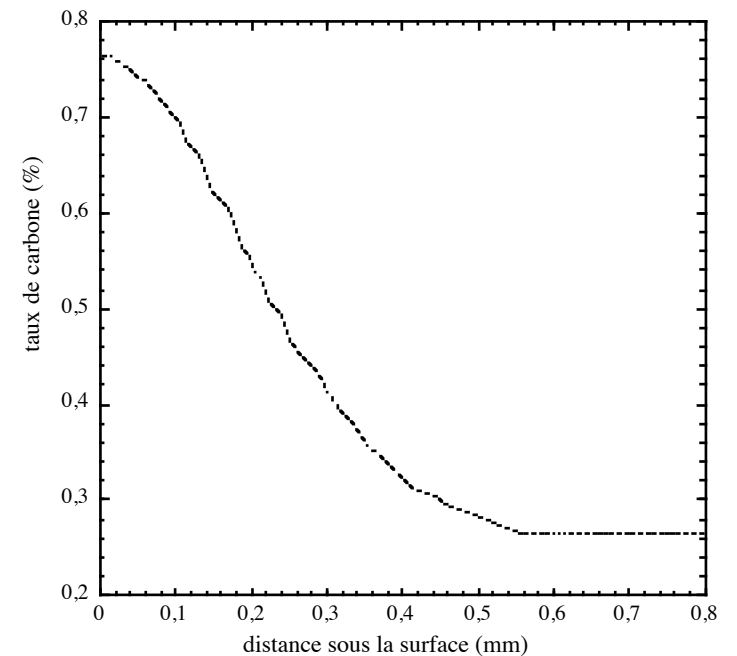
Austenitization 930°C 40min

Gaz quenching (nitrogen 4 bars)

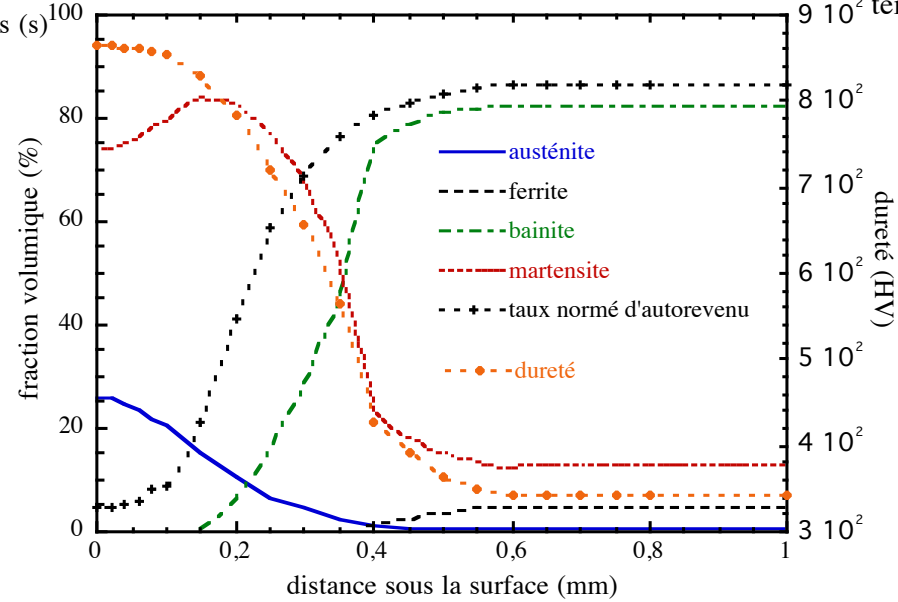
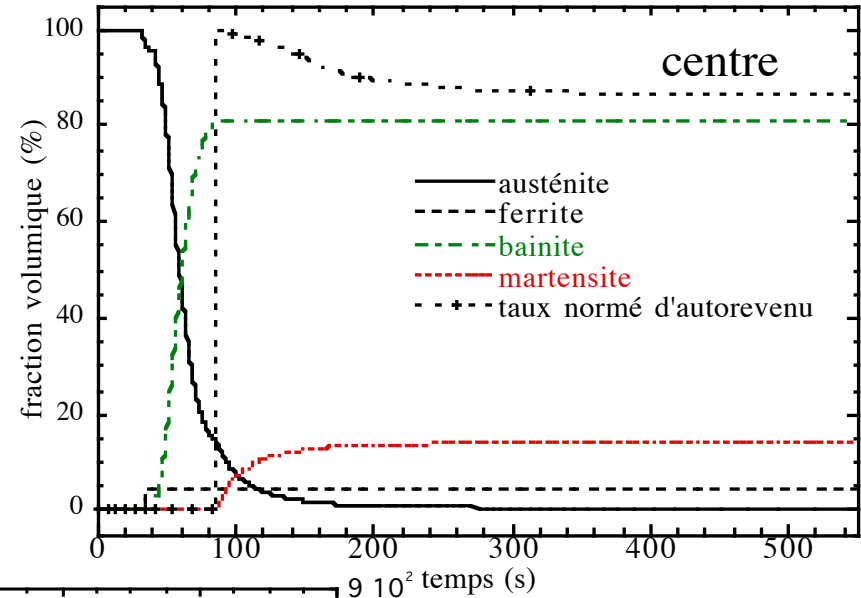
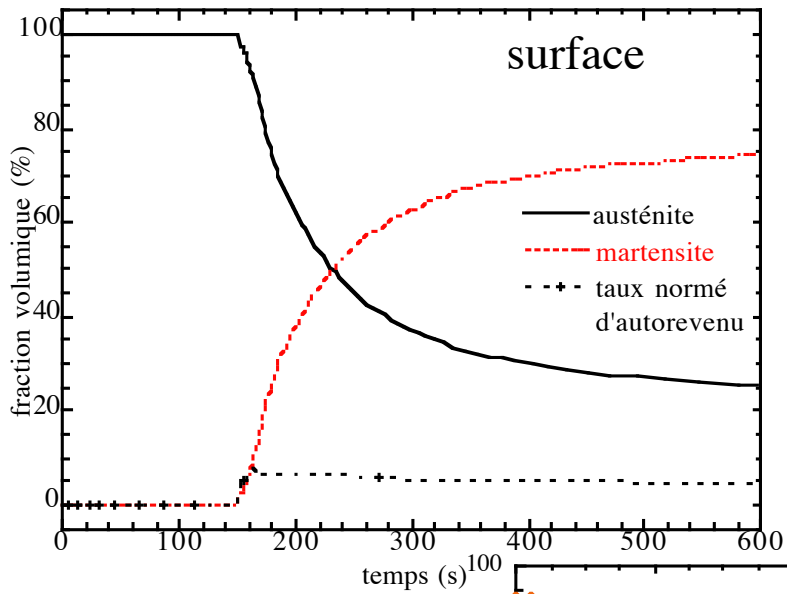
Temperature evolutions (median plane)



Radial carbon profile



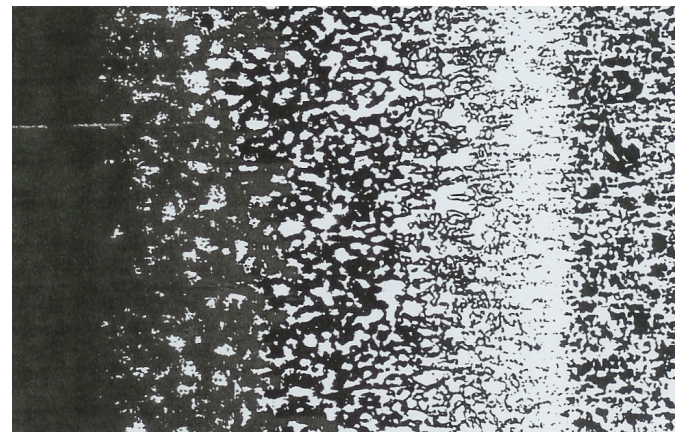
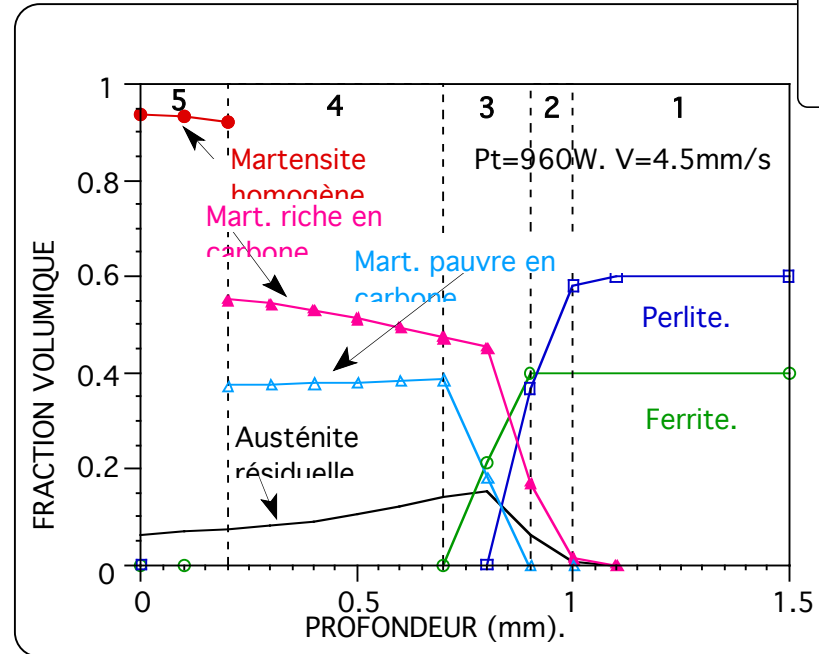
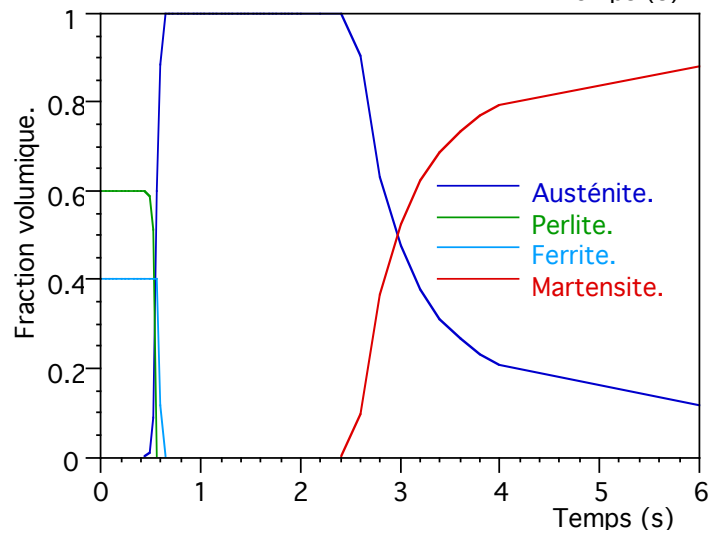
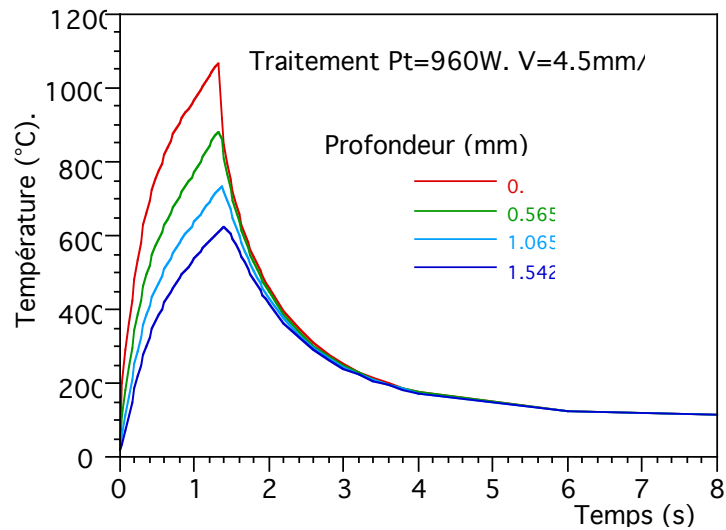
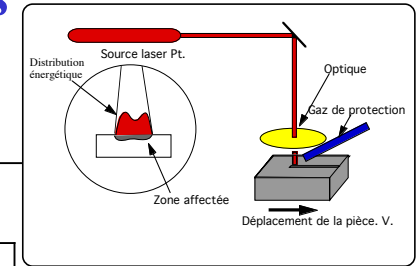
Kinetics of transformation



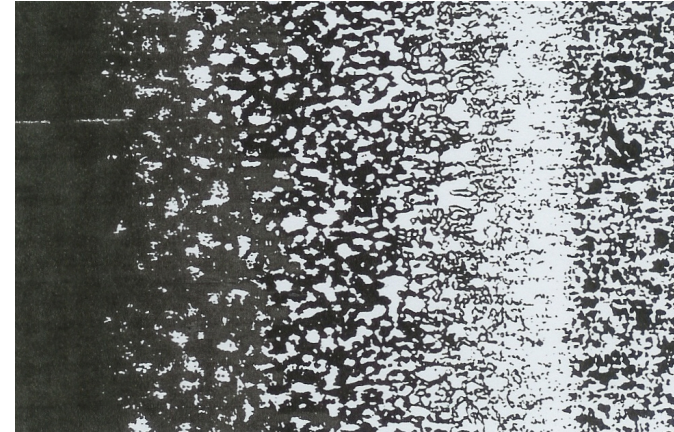
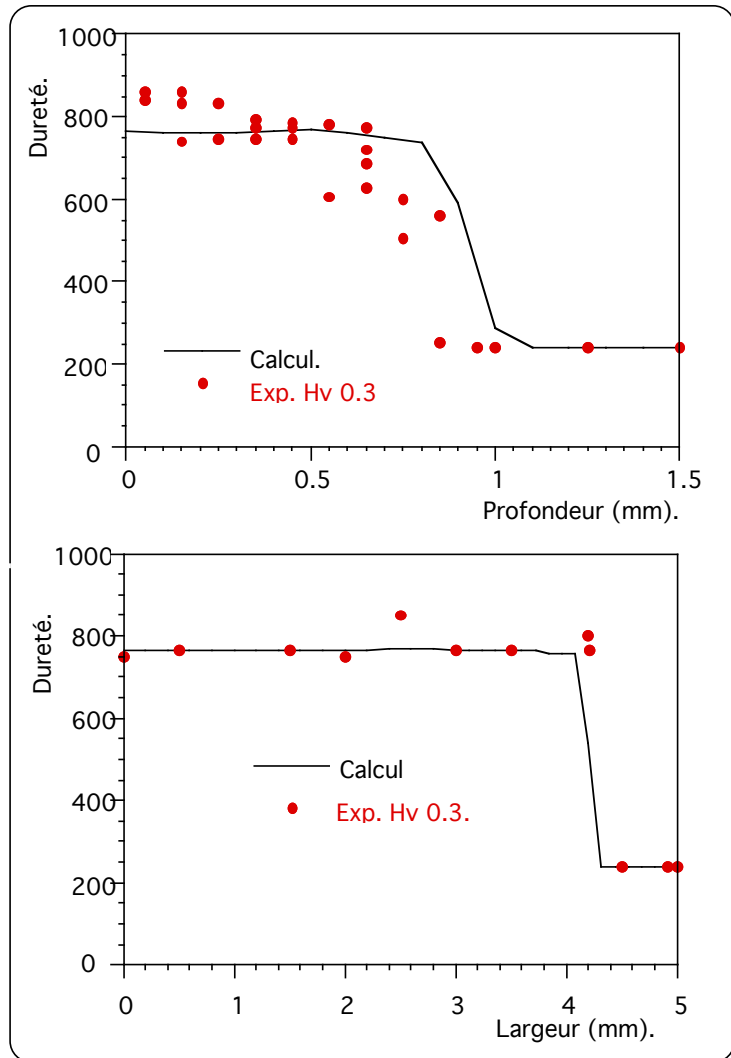
Final microstructure and hardness distributions

Ex. Prediction of microstructures during laser hardening of steels

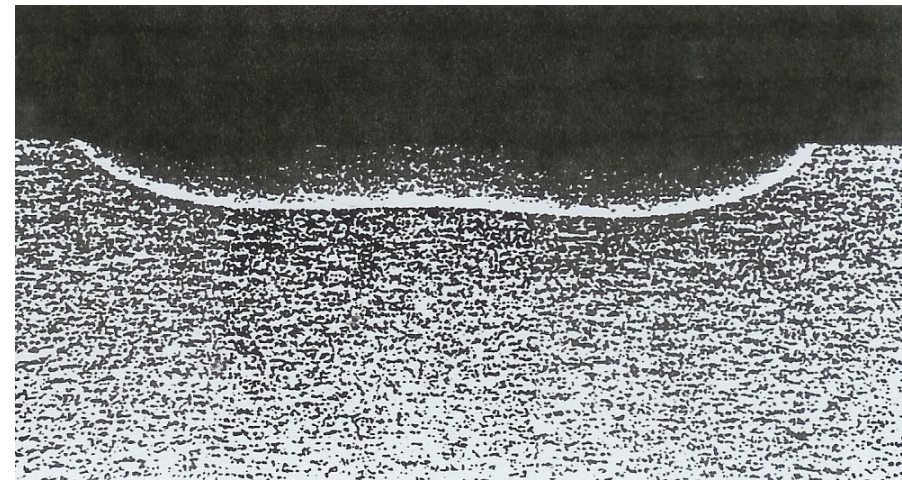
Plate moving under laser beam – steel C42



Hardness and microstructure distributions comparison calculation-experiment



depth HAZ: exp. 0,85mm calculation 1,06mm



width HAZ : exp. 8,4mm calculation 8,5mm

Modelling of phase transformation kinetics

(for the purpose of predicting internal stresses and deformations)

- **Global models (JMAK):** most commonly used for steels
 - Modelling anisothermal kinetics from CCT diagrams
 - Modelling kinetics from IT diagrams (additivity principle)

can predict volume fractions of the phases for complex situations

→ *limitations : no morphological parameters of the microstructures*

difficult to take into account prior plastic deformation effects

- **Nucleation, growth, coarsening models**

- precipitation in Al alloys *D. Godard 1999*
- **tempering of martensite in steels** *Y. Wang 2006*

*volume fractions of the precipitates, size distributions
matrix chemical composition*

Modelling of phase transformation kinetics : nucleation, growth, coarsening

Multicomponent alloys - concomittant precipitations

- thermodynamic equilibrium :

- . solubility product (stoichiometric compositions)
or coupling with THERMOCALC

- nucleation rate

homogeneous/heterogeneous nucleation, take account of elastic strain energy

- growth (dissolution) coarsening rates

diffusion controlled

local equilibrium at interfaces

Gibbs Thomson effect and effect of elastic strain energy



Results

volume fractions of the precipitates
size distributions
matrix chemical composition

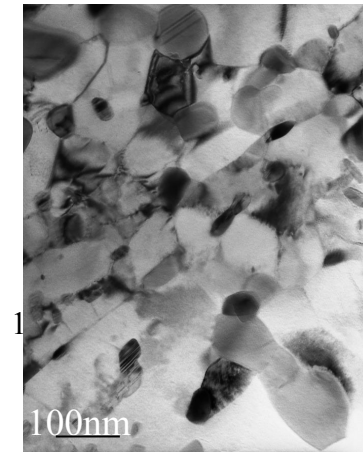
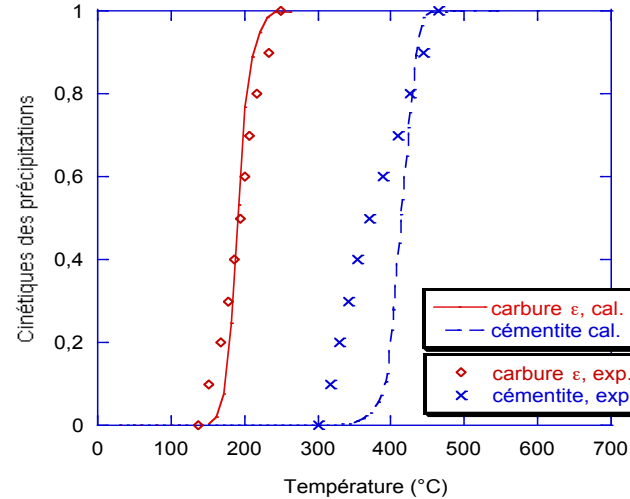
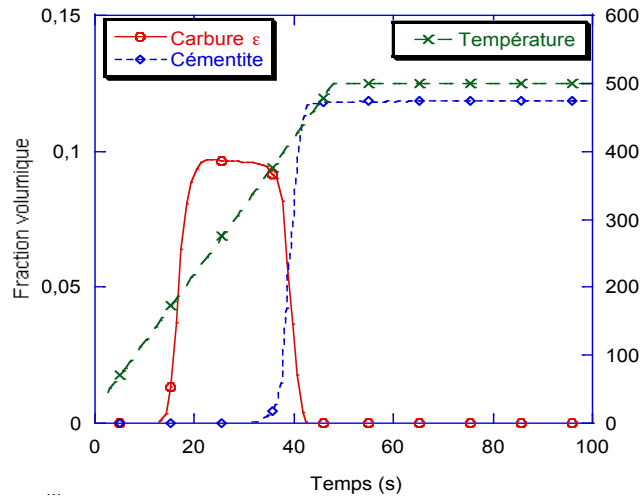


during the treatments
isothermal and anisothermal

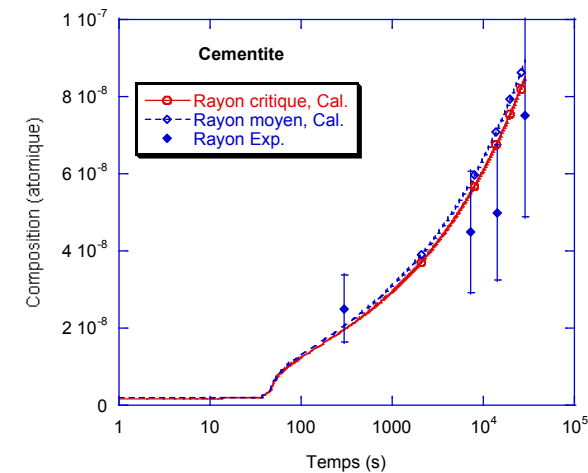
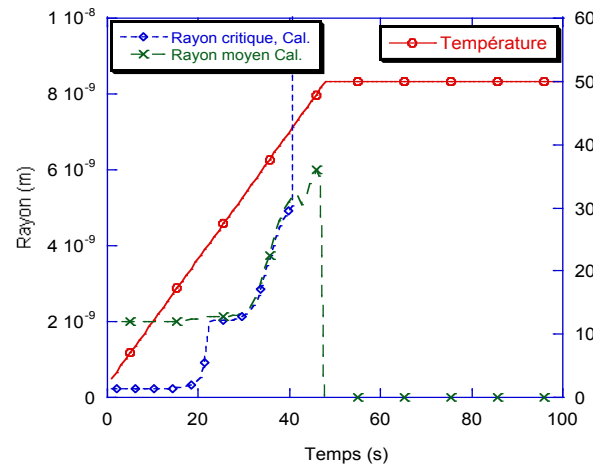
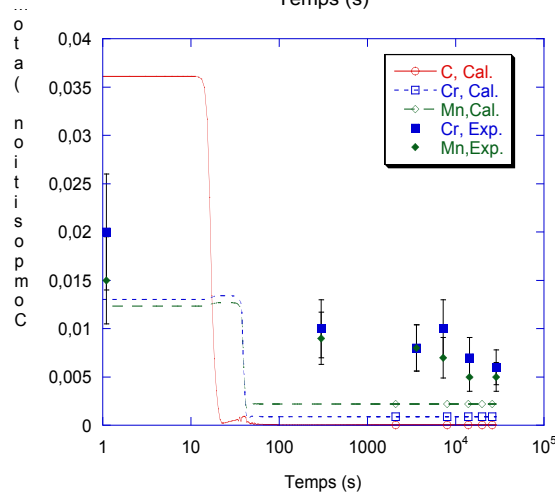
Modelling of precipitation during tempering of martensite in steels

ϵ carbides ($\text{Fe}_{2,4}\text{C}$) coherent, homogeneous nucleation,
 cementite ($\text{Fe,M})_3\text{C}$) incoherent, heterogeneous nucleation (disloc.)

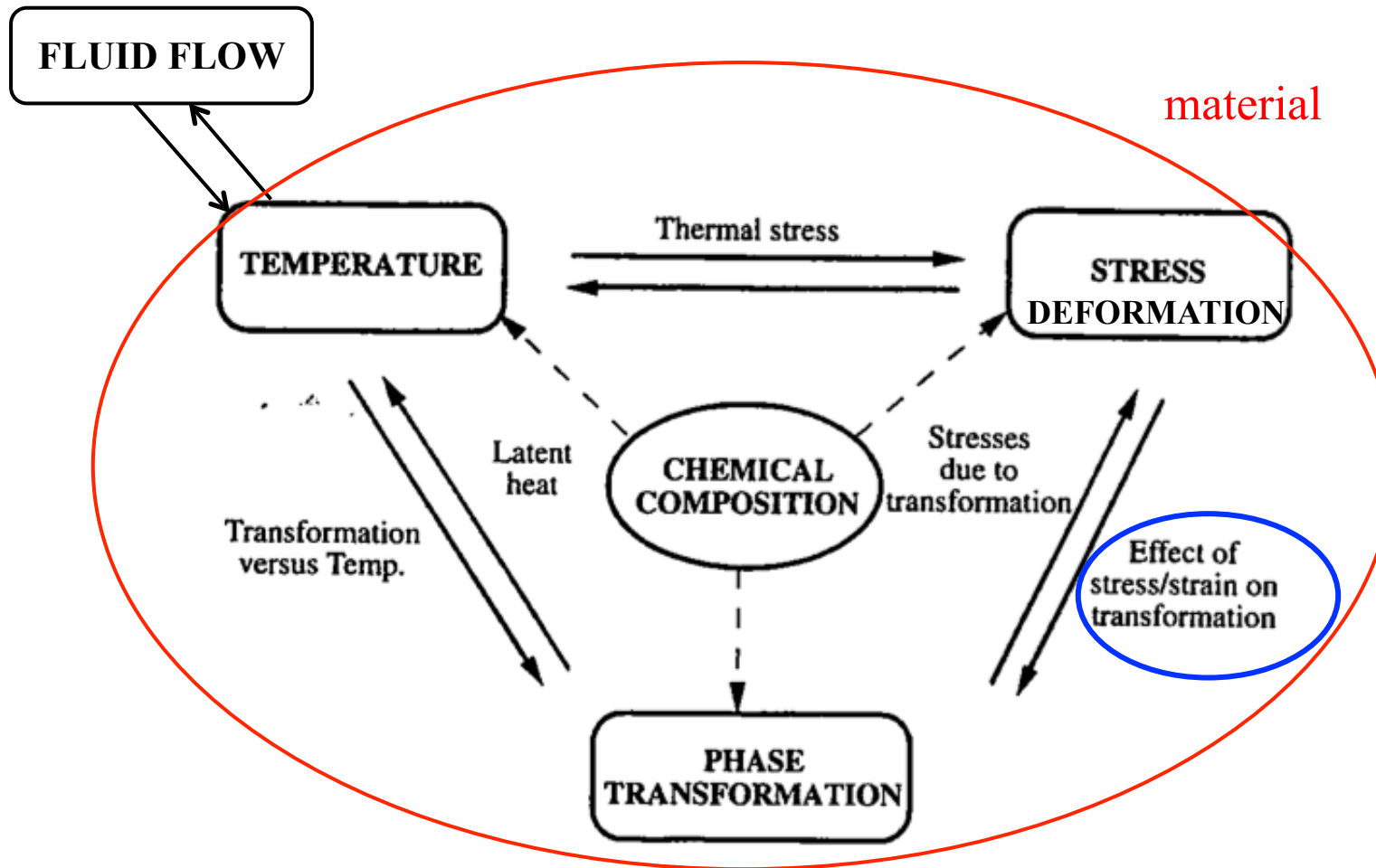
steel 80MnCr5
 heating : 10°C/s
 holding at 500°C



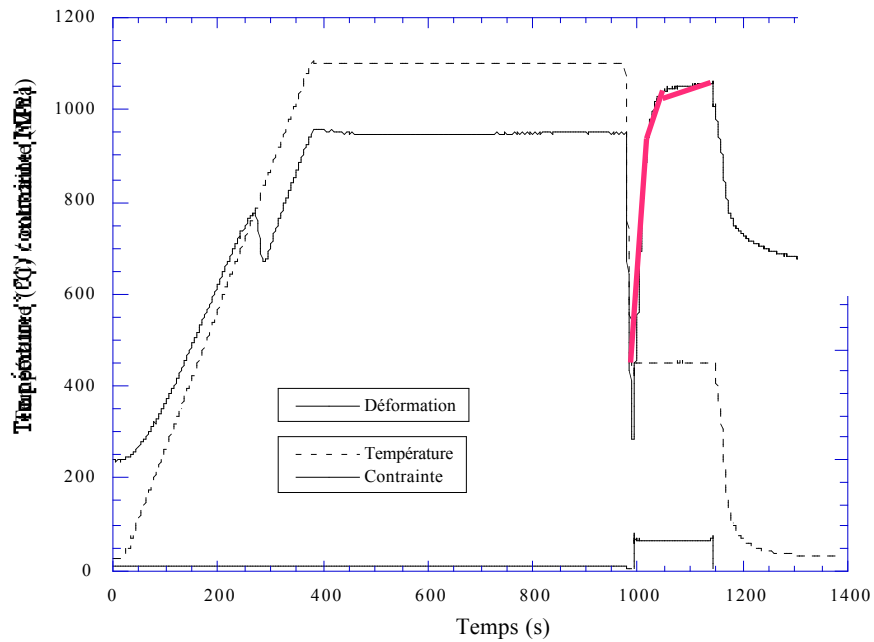
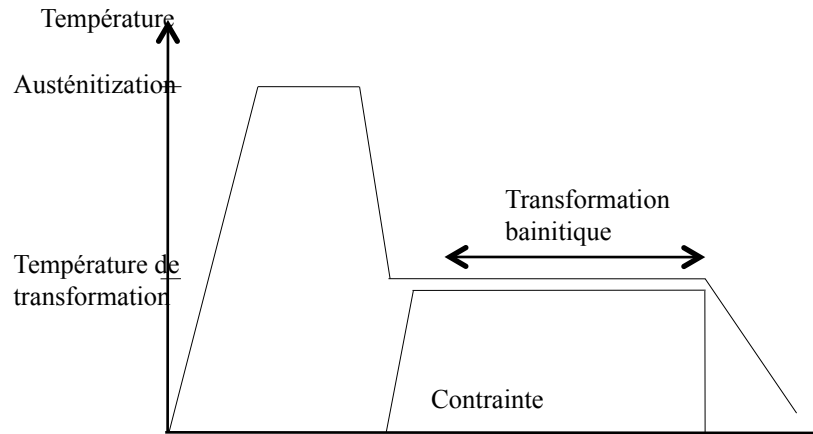
600°C 2h



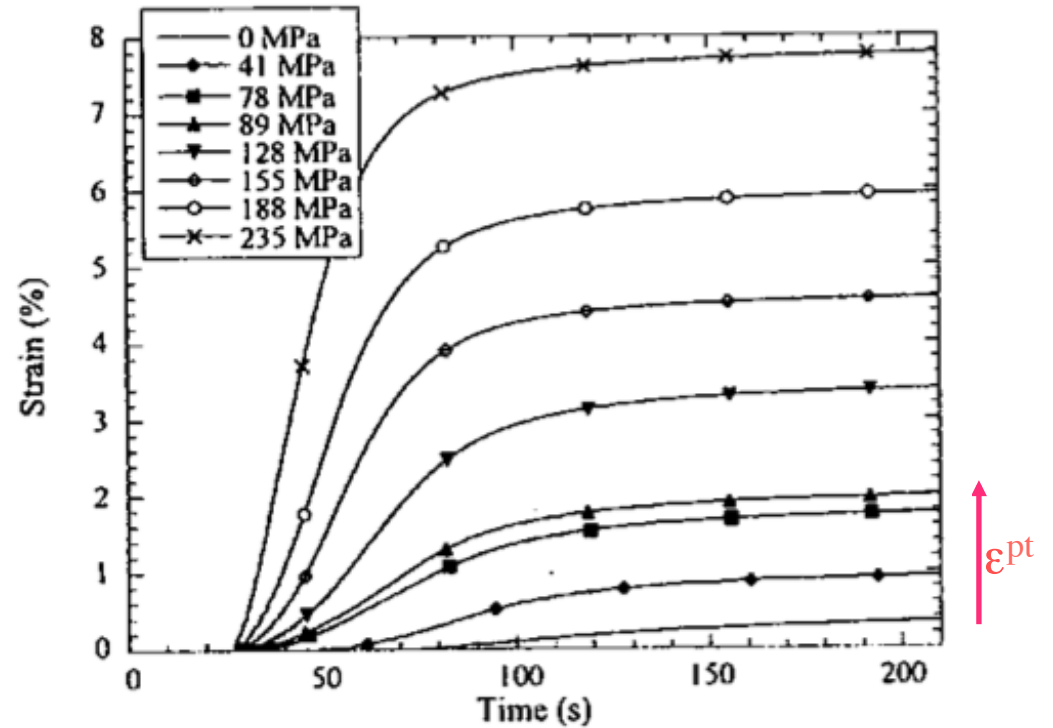
Fluid – thermal – metallurgical – mechanical couplings in heat treatment



Bainitic transformation under stress (thermomechanical simulator DITHEM)

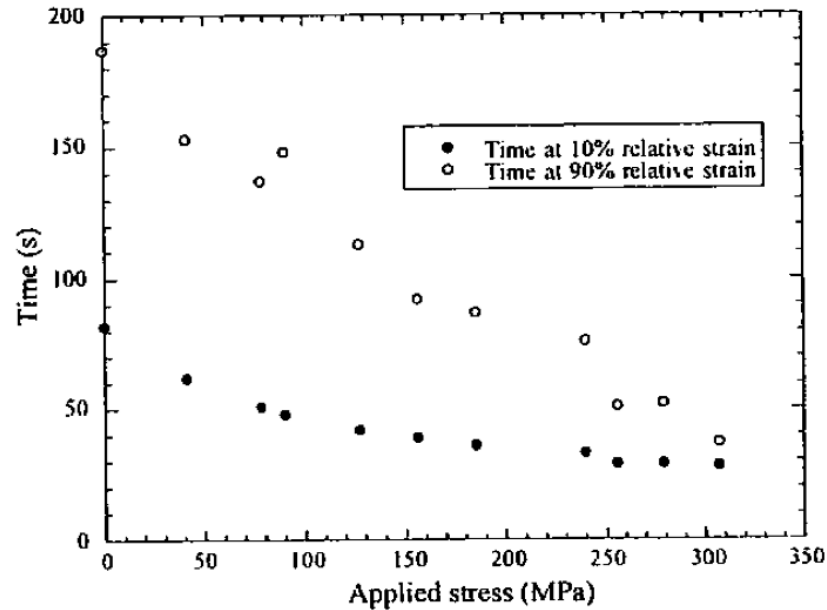


Steel 35MnV7
Transformation at 350°C



Transformation plasticity
Acceleration of the transformation

Variations of times corresponding to 10% and 90% of length variation

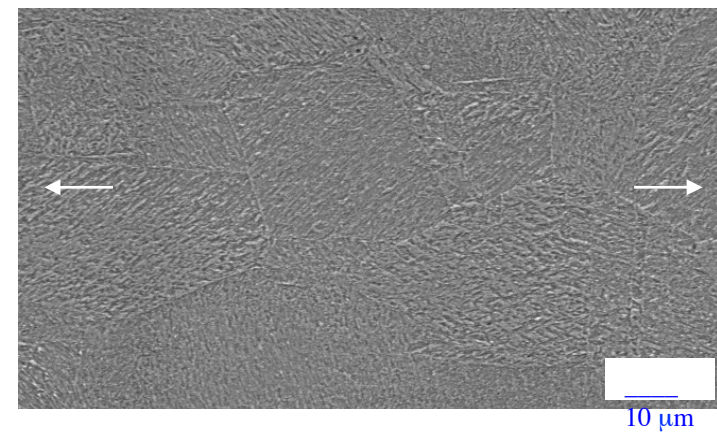
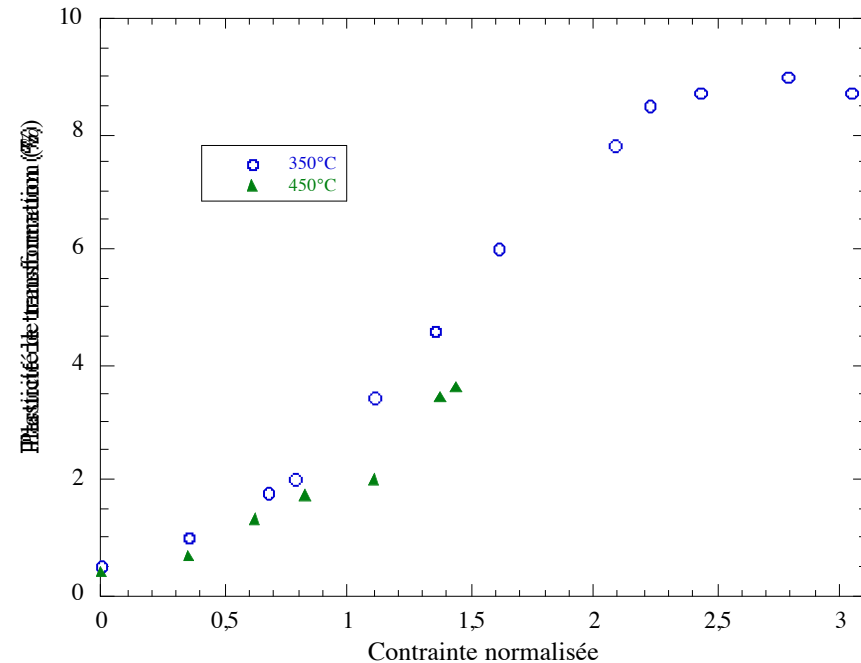


Acceleration of the transformation :
increase of nucleation rate and modification of growth rate

Mechanisms of transformation plasticity

- plastic accommodation of transformation strains
- orientation of transformation product

Transformation deformation versus applied stress



bainite formed at 350°C under 310MPa

Phenomenological modelling of the coupling between internal stress states and transformation kinetics

During heat treatment material is under triaxial stress states, small plastic deformations

For diffusion dependent transformations

effect of plastic strain and pressure can be assumed negligible
acceleration of the transformation due to stress

$$D_{\sigma} = \Delta t / t_0 = h(\sigma_e) \quad \begin{array}{l} \sigma_e \text{ Von Mises stress} \\ h \text{ experimental function} \end{array}$$

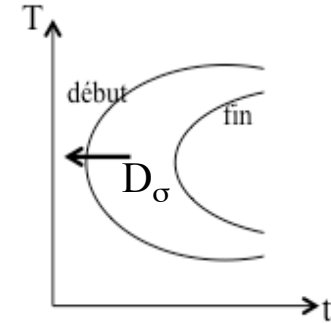
Incubation period : $\tau_{\sigma} = \tau (1 + D_{\sigma})$
(Scheil's method)

Progress of transformation : $n_{k\sigma} = n_k \quad b_{k\sigma} = b_k / (1 + D_{\sigma}) n_k$
(JMKA law)

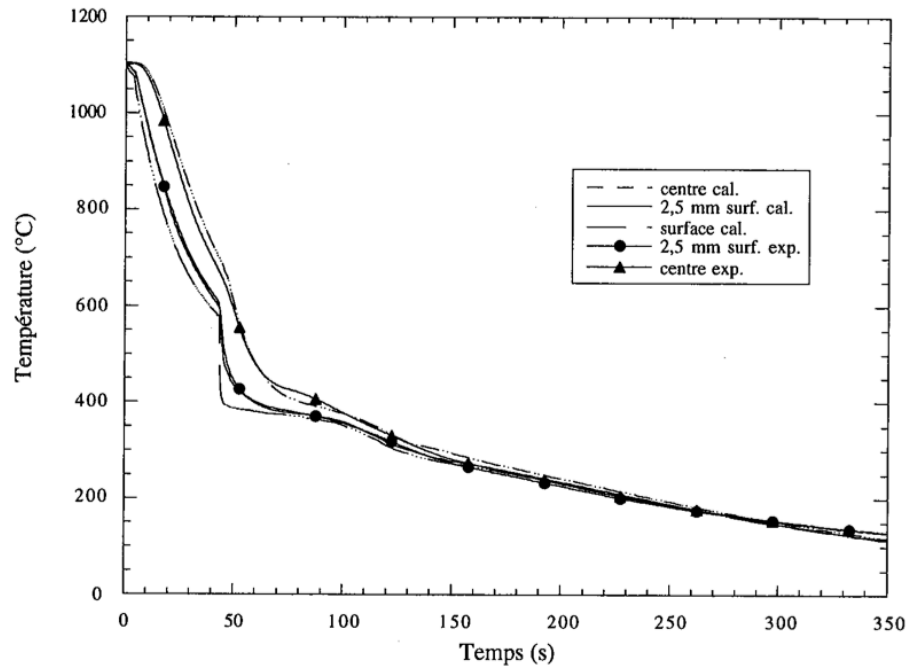
For martensitic transformation

Effect of plastic strain assumed negligible in comparison with stress effect
Variation of M_s with stress state

$$\Delta M_s = A \sigma_m + B \sigma_e \quad A \text{ and } B \text{ constants (Inoue)}$$



Ex effect of internal stress – kinetics interaction

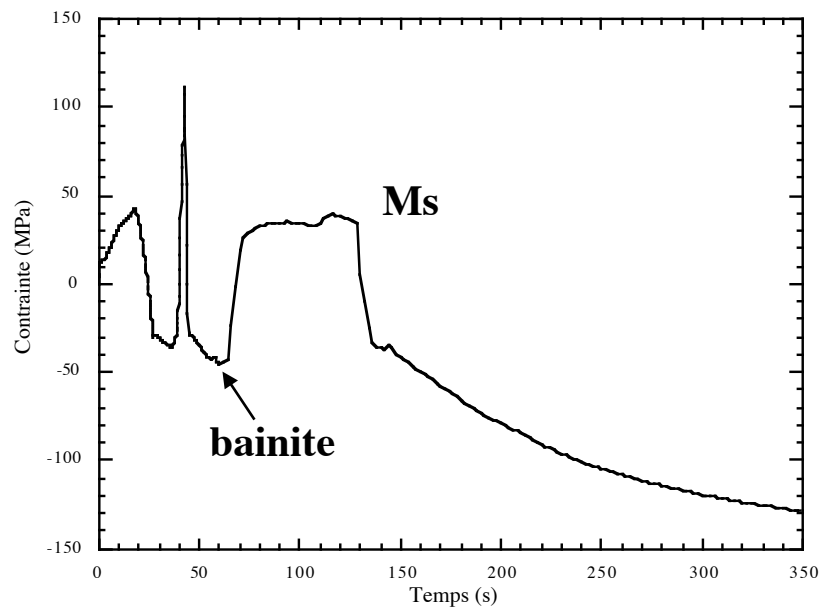


Oil quenching of cylinders
diameter 35mm length 105mm
steel 35MnV7 (*PhD M Veaux 2003*)

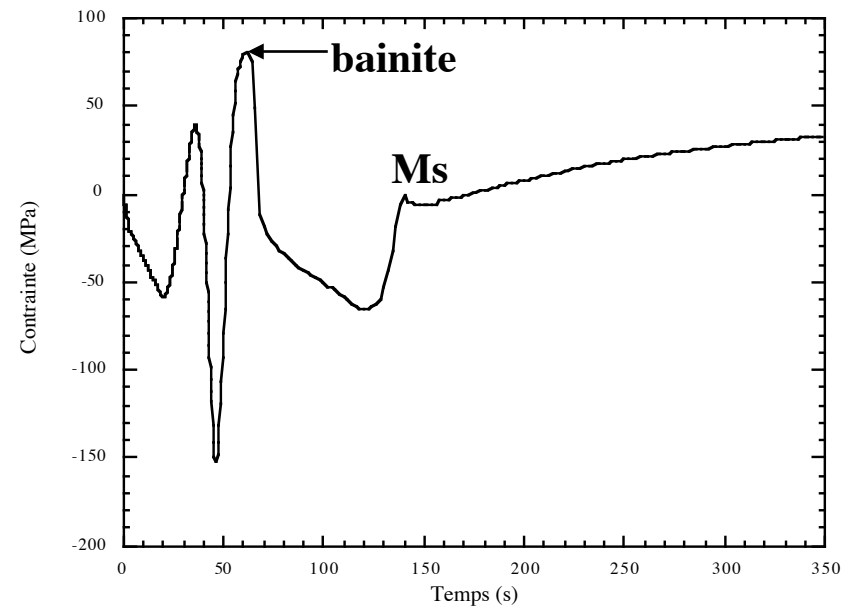
Temperature evolutions
(center and surface)

Complex stress evolutions due
to temperature gradients

Calculated axial stress versus time at the surface

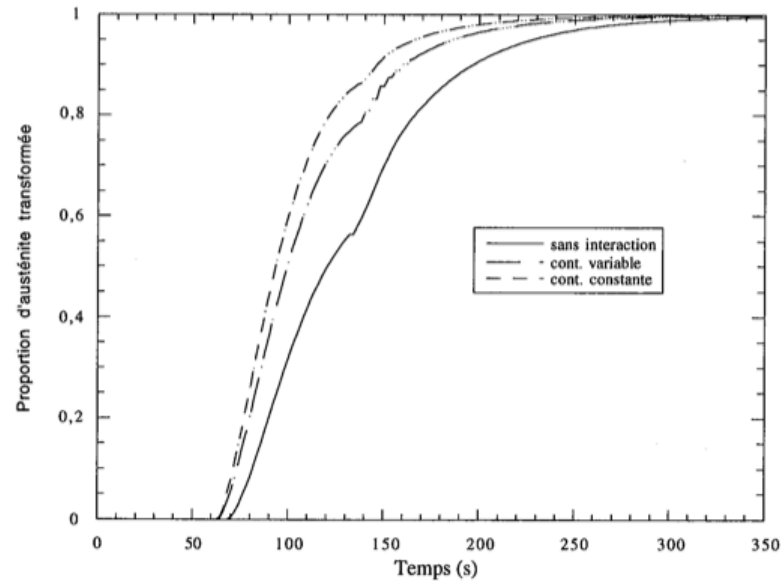


Axial stress versus time in the center

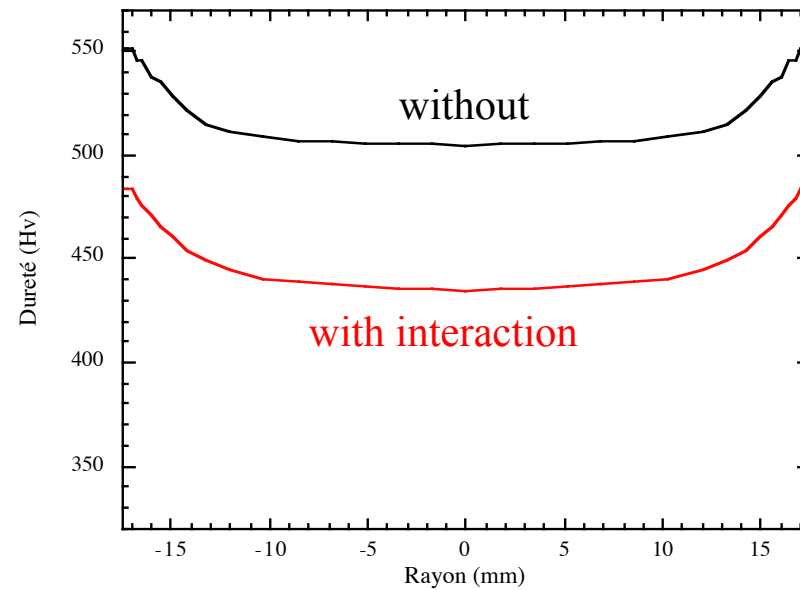


Ex. effect of the internal stress-kinetics interaction

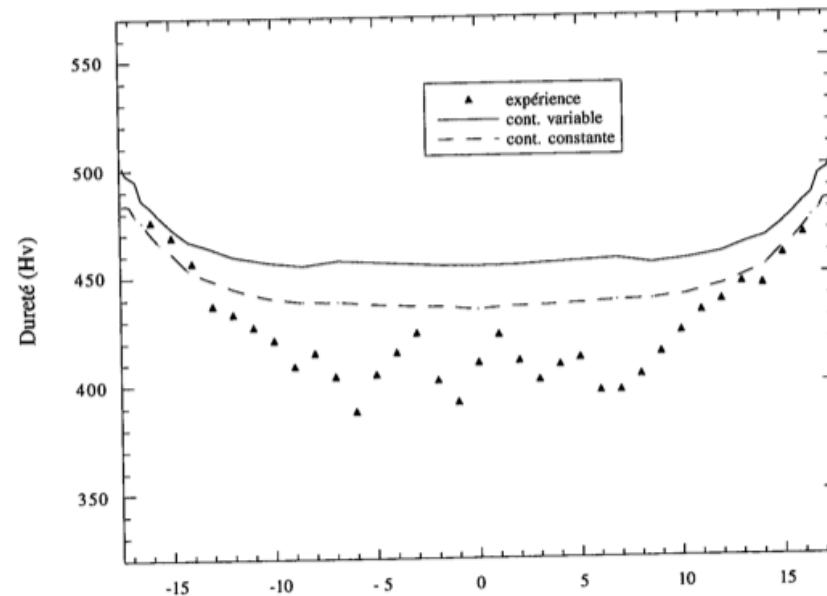
in the center



Hardness profiles



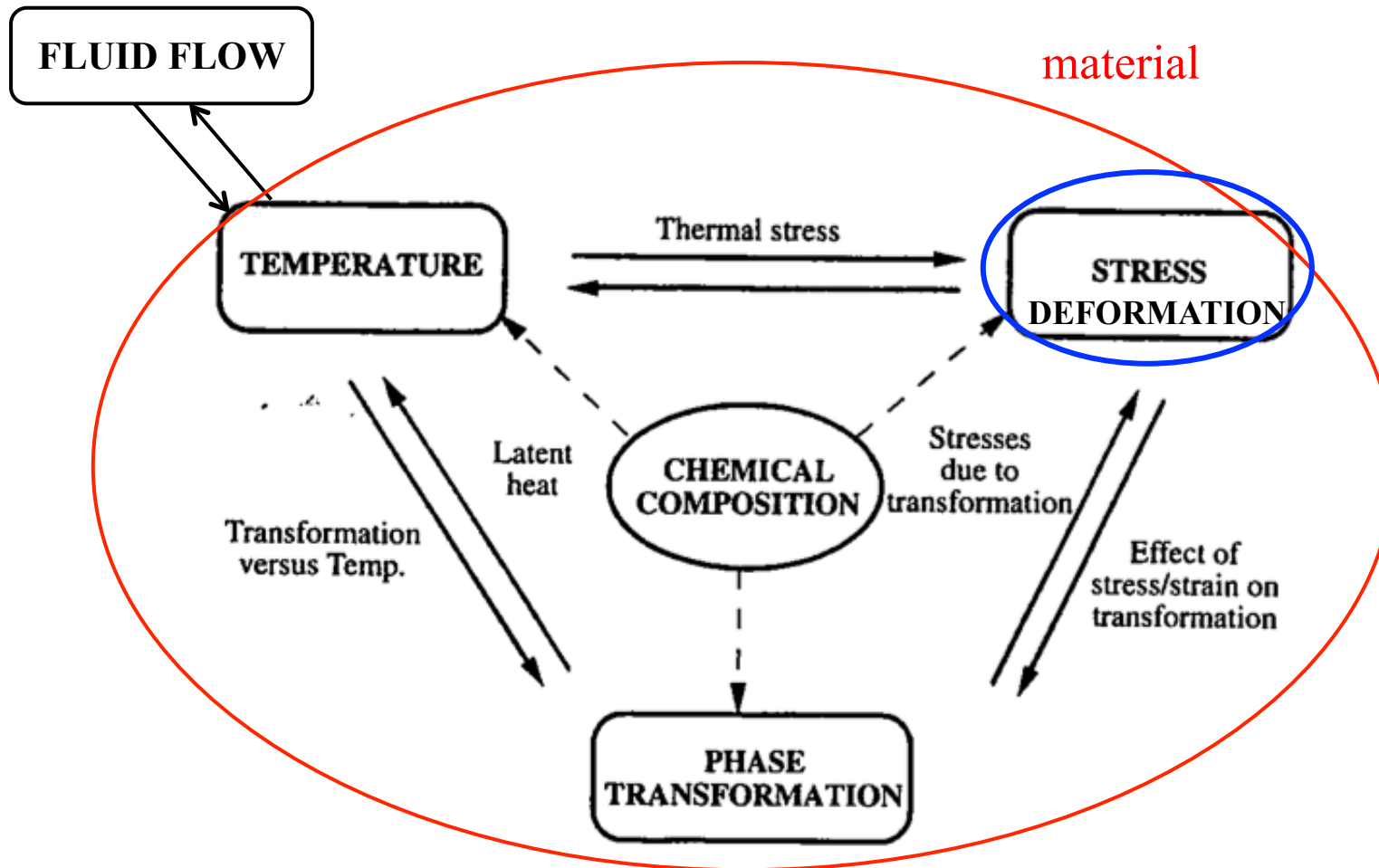
Comparison with experiment



- important effect in a massive specimen
- prediction not fully satisfactory

need of better knowledge of the effect of complex thermomechanical paths

Fluid – thermal – metallurgical – mechanical couplings in heat treatment



Calculation of stress and strain fields

Hyp. small deformations, material homogeneous isotropic

Stress equilibrium

Compatibility of deformations

Behavior law of the material



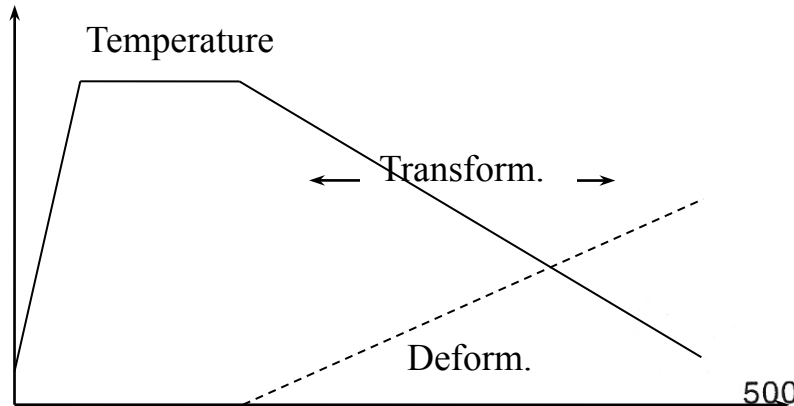
Effect of phase transformations

- change of mechanical properties
- additional deformations : volumic variations and transformation plasticity

Thermomechanical behaviour during phase transformation

Tensile test during continuous cooling

DITHEM experiment

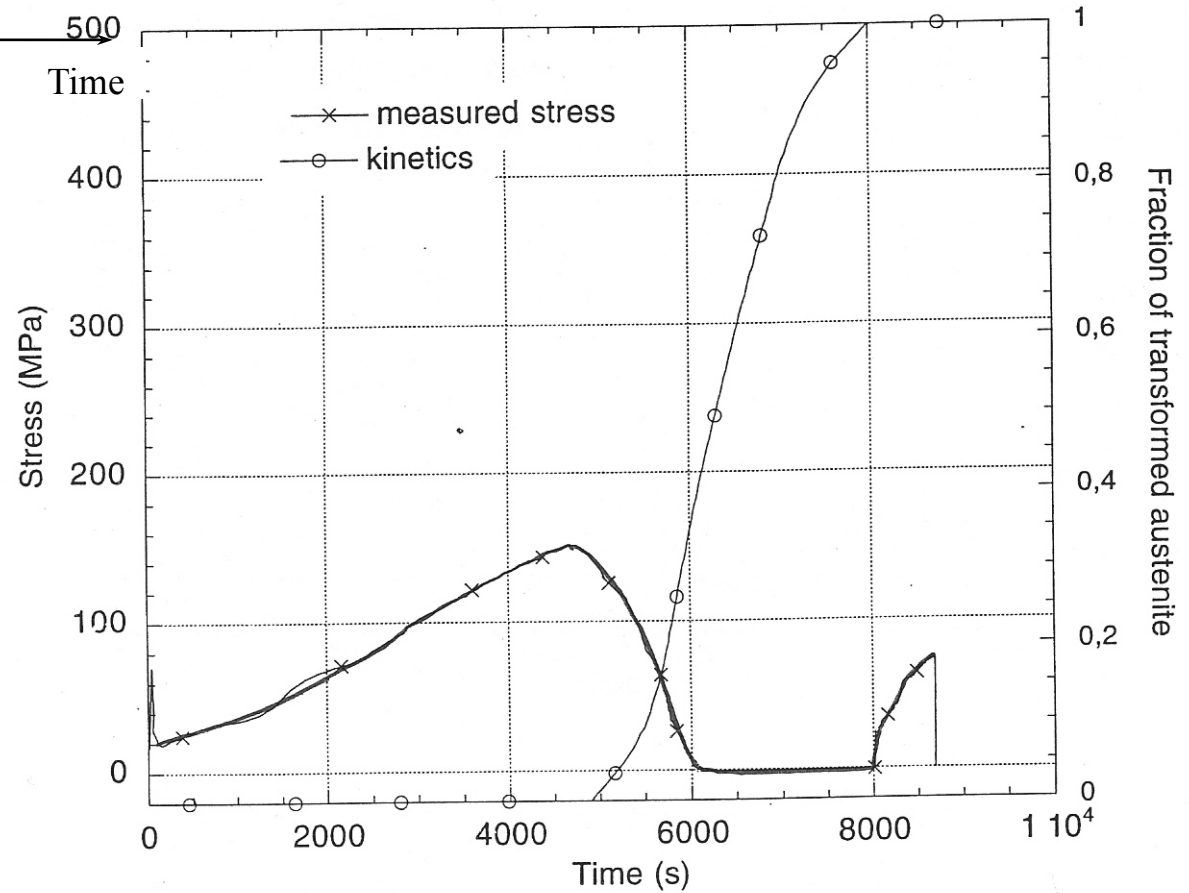


bainitic transformation

Steel 40CrMnMo8

Constant cooling rate

Deformation rate : 10^{-4} s^{-1}



Macroscopical phenomenological approach

Take into account the different sources of deformations

$$d\varepsilon_{ij}^t = d\varepsilon_{ij}^e + d\varepsilon_{ij}^p + d\varepsilon_{ij}^{th} + d\varepsilon_{ij}^{tr} + d\varepsilon_{ij}^{pt}$$

Thermal strain

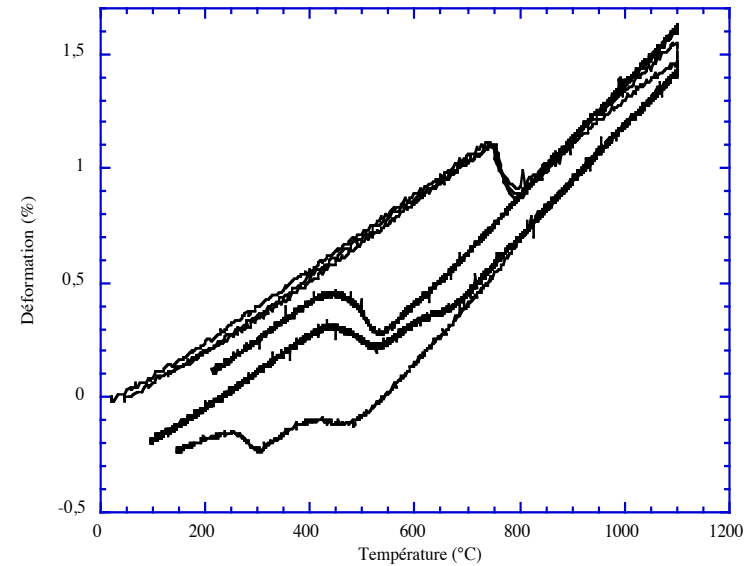
$$\varepsilon_{ij}^{th} = \delta_{ij} \sum y_k \alpha(T) dT$$

$\alpha(T)$ thermal expansion coefficients of the phases

Transformation strain (volumic variation)

$$\varepsilon_{ij}^{tr} = y_k \sum \varepsilon_k^{tr} 0^\circ\text{C}$$

Continuous cooling dilatometry
35MnV7 steel (PhD M. Veaux)



Macroscopical phenomenological approach

$$d\varepsilon_{ij}^t = d\varepsilon_{ij}^e + d\varepsilon_{ij}^p + d\varepsilon_{ij}^{th} + d\varepsilon_{ij}^{tr} + d\varepsilon_{ij}^{pt}$$

Elastic strain

$d\varepsilon_{ij}^e$ Hooke's law $E, \nu = f(\text{temperature, microstructure})$ Mixture law

plastic/viscoplastic strains :

classical theory, Von Mises yield criterion and associated flow rule, isotropic-kinematic hardening rules

For each phase

$$\sigma_k = \sigma_{0k} + H_k \varepsilon_{vp}^{nk} + K_k \dot{\varepsilon}_{vp}^{mk}$$

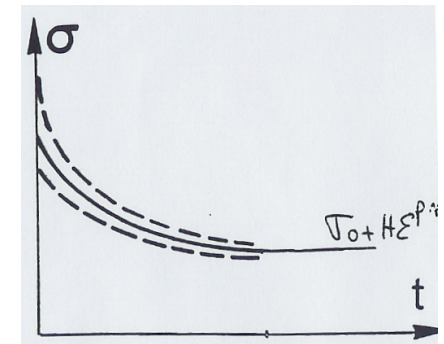
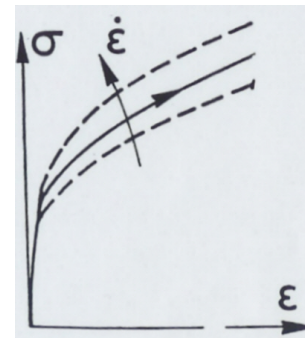
$\sigma_0(T)$ threshold stress

hardening : $H(T), n(T)$

viscous stress : $K(T), m(T)$

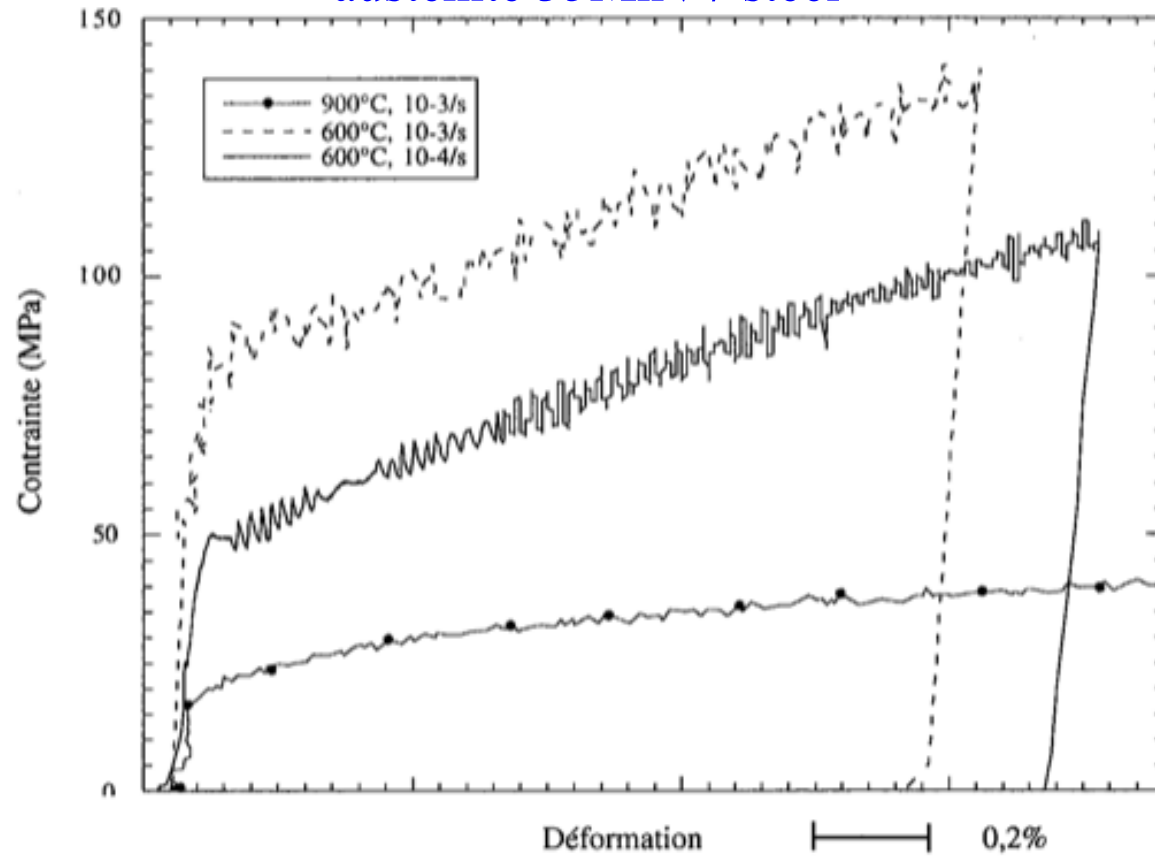
for multiphase material :

mixture law $\sigma = \sum y_k \sigma_k$



Thermomechanical behaviour of the different phases/constituents at different temperatures and strain rates

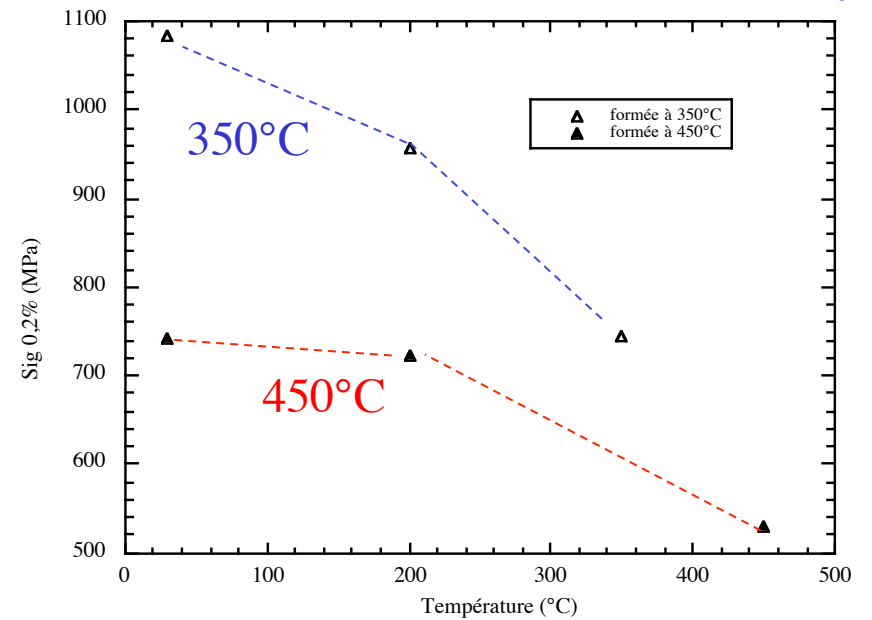
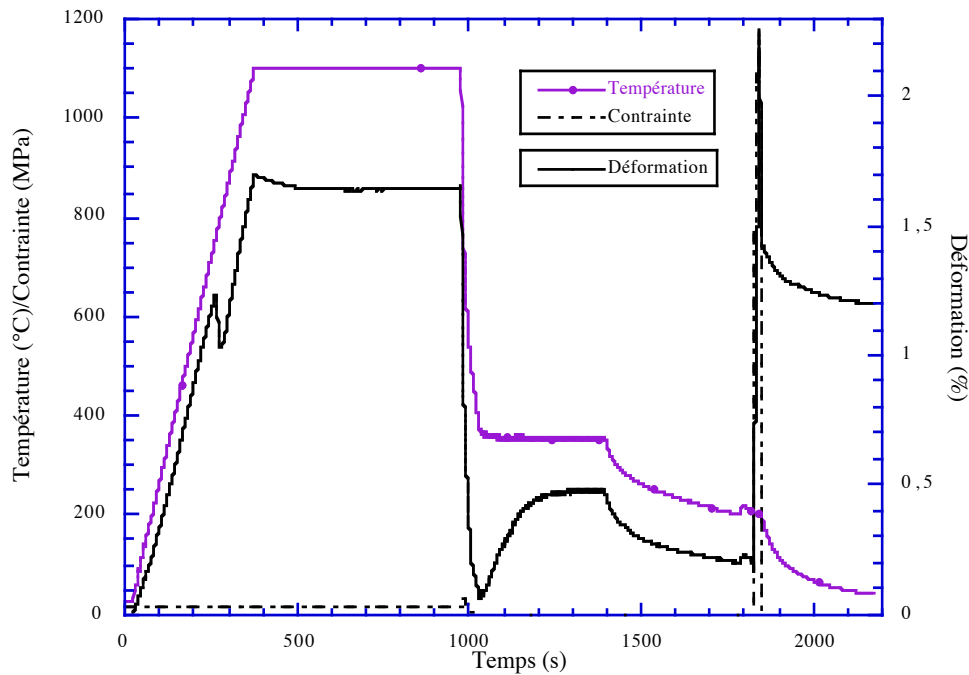
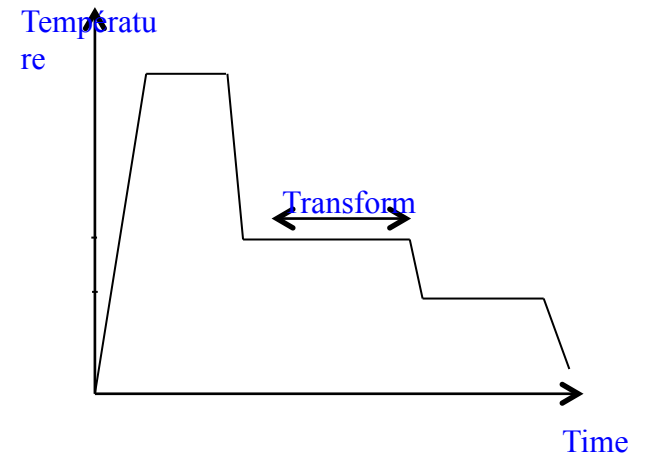
austenite 35MnV7 steel



Example : thermomechanical behaviour of bainite

Steel 35MnV7 Austenitization 1100°C 10 min

Bainite formed at 350°C/450°C tensile test at 200°C, 20°C



thickness of bainite plates
0,057 μ m at 350°C
0,239 μ m at 450°C

Yield stress (0.2%)
bainitic microstructures

Macroscopical phenomenological approach

$$d\varepsilon_{ij}^t = d\varepsilon_{ij}^e + d\varepsilon_{ij}^p + d\varepsilon_{ij}^{th} + d\varepsilon_{ij}^{tr} + d\varepsilon_{ij}^{pl}$$

Elastic strain

$d\varepsilon_{ij}^e$ Hooke's law $E, \nu = f(\text{temperature, microstructure})$ Mixture law

plastic/viscoplastic strains :

classical theory, Von Mises yield criterion and associated flow rule, isotropic-kinematic hardening rules

For each phase

$$\sigma_k = \sigma_{0k} + H_k \varepsilon_{vp}^{nk} + K_k \dot{\varepsilon}_{vp}^{mk}$$

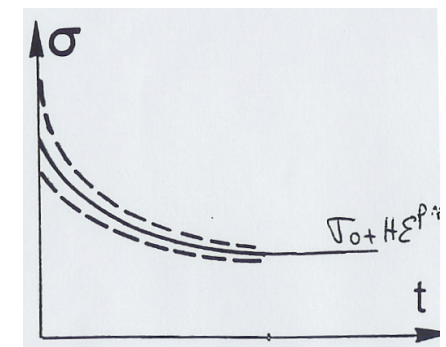
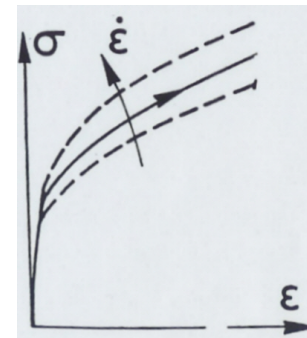
$\sigma_0(T)$ threshold stress

hardening : $H(T), n(T)$

viscous stress : $K(T), m(T)$

for multiphase material :

mixture law $\sigma = \sum y_k \sigma_k$



question : strain hardening full or partial loss of memory during phase transformation?

(Sjöström, Leblond, Taleb...)

Modelling of transformation plasticity

- Phenomenological approach:

Experimental evolution law (uniaxial stress) : $\epsilon^{pt} = K_k \sigma f(y_k)$

Generalization to triaxial stress states : **assumption $d\epsilon^{pt}$ proportional to stress deviator** *Giusti*

$$d\epsilon_{ij}^{pt} = 3/2 K_k f'(y_k) dy_k s_{ij}$$

holds when the mechanism only plastic accommodation

For diffusional transformation :

- no experimental results under mutiaxial stresses

- validation by micro - macro approaches *Leblond, Fischer, Sjöström, Ganghoffer, Barbe*

For martensitic transformation :

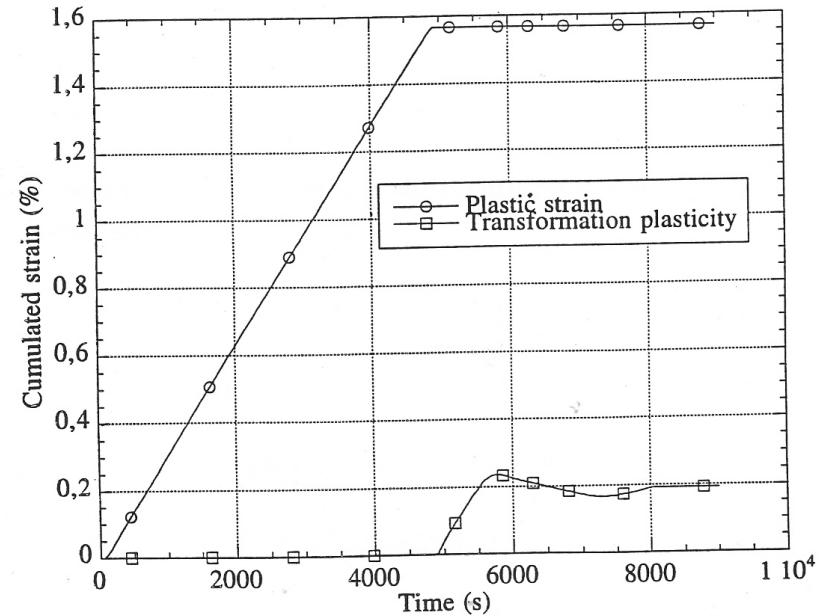
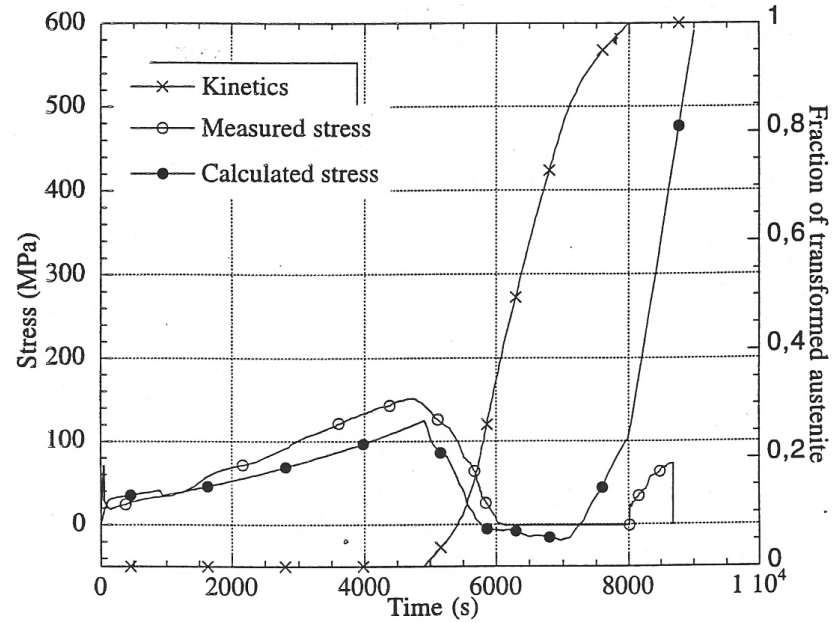
- experimental results : transform. plasticity larger **under tensile stress** than under compressive stress *Videau*

- micromechanical approach show clearly strong dependency on local stresses *Wen*

→ *formulation of a macroscopical law still an open question*
(N phase model, Cailletaud 2004)

Experimental validation of the macroscopical behaviour law for steel

$$d\varepsilon_{ij}^t = d\varepsilon_{ij}^e + d\varepsilon_{ij}^p + d\varepsilon_{ij}^{th} + d\varepsilon_{ij}^{tr} + d\varepsilon_{ij}^{pt}$$



➔ Modelling of the thermomechanical behaviour during precipitation

thermoelastoviscoplastic behaviour law

$$\sigma = \sigma_0 + H \varepsilon_{vp}^n + K \dot{\varepsilon}_{vp}^m$$

σ_0 threshold stress
 hardening = f(T)
 viscous stress = f(T)

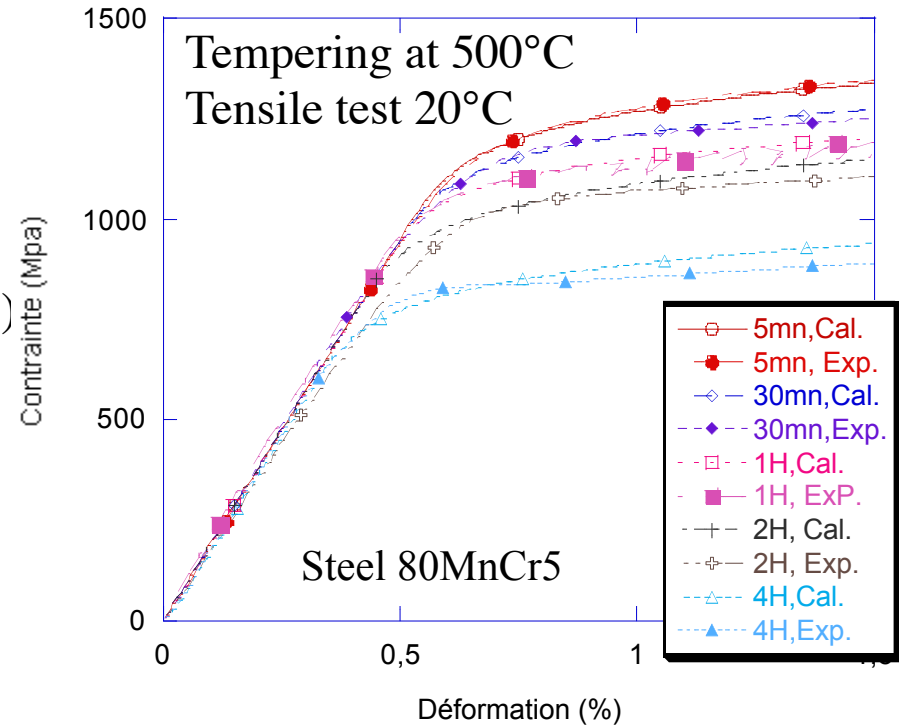
$$\sigma_0 = \sigma_{Fe} + \sigma_{ss} + \sigma_P + \sigma_{disl.}$$

σ_{Fe} friction stress f(T)

σ_{ss} solid solution hardening f(T, composition ss)

σ_P precipitation hardening f(T, volume fractions and sizes of precipitates)

$\sigma_{disl.}$ hardening due to dislocation densities f(T, t)

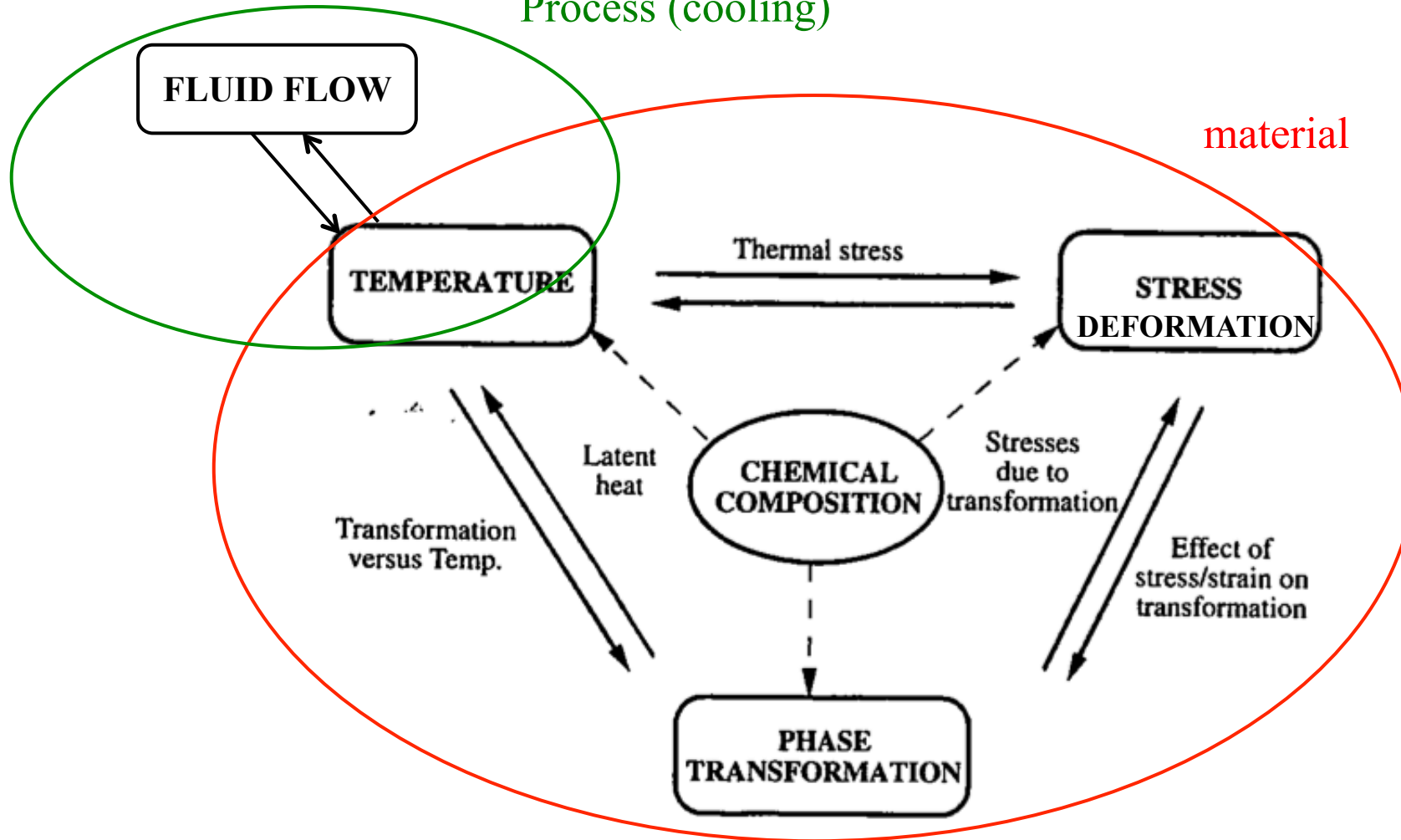


↑ Precipitation model

Fluid – thermal – metallurgical – mechanical couplings in heat treatment

Process (cooling)

material



Modelling of heat transfer in the solid

Heat conduction equation : $\text{div} (\lambda \text{grad} T) + q^{\text{tr}} = \rho c_p \delta T / \delta t$

- q^{tr} power density associated with phase transformations

$$q^{\text{tr}} = \sum \Delta H_k dy_k / dt$$

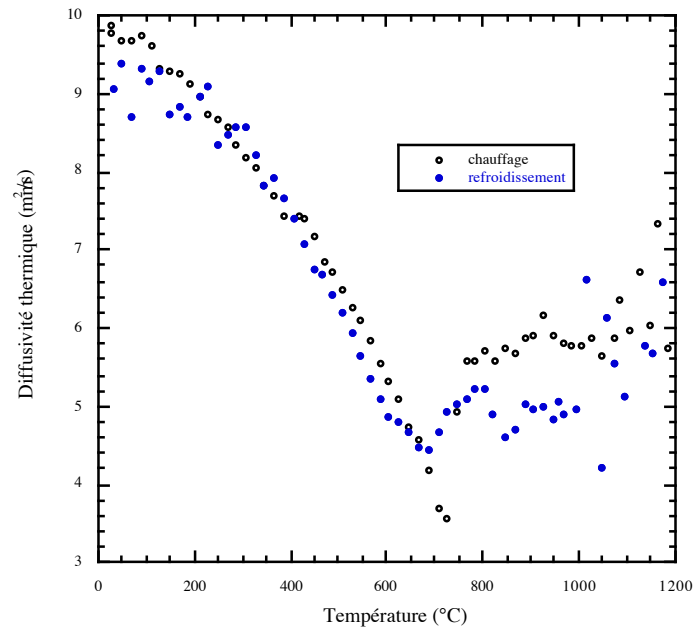
- mixture laws for thermophysical properties

$$\lambda = \sum \lambda_k y_k \quad c_p = \sum c_{pk} y_k \quad \rho = \sum \rho_k y_k \quad y_k \text{ volume fraction of phase } k$$

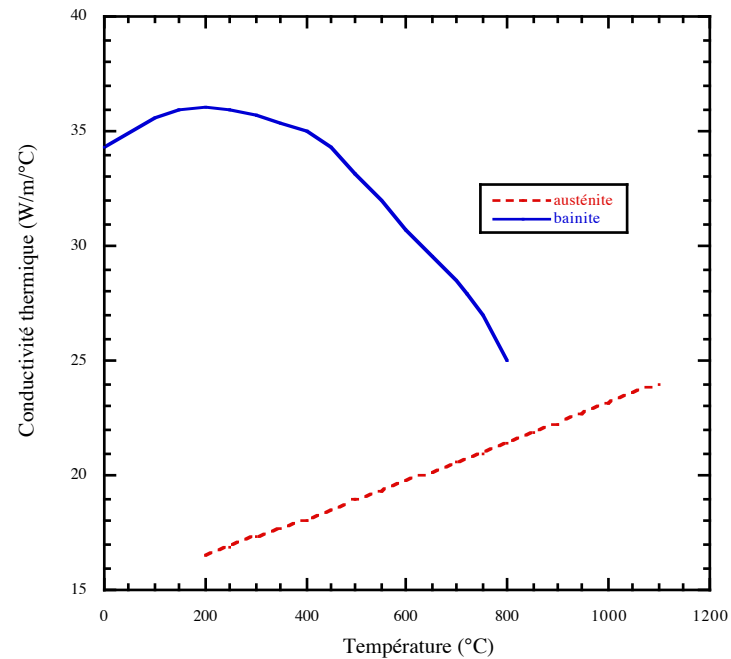
Thermophysical properties

Example : steel 35MnV7

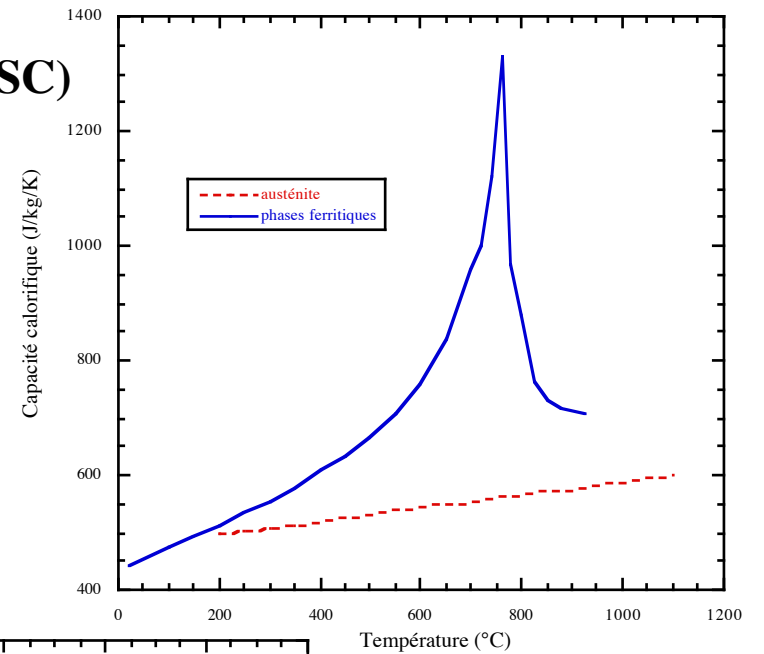
Thermal diffusivity (« flash method »)



Thermal conductivity



Specific heat (DSC)



Modelling of heat transfer in the solid

Heat conduction equation : $\text{div} (\lambda \text{grad} T) + q^{\text{tr}} = \rho c_p \delta T / \delta t$

- q^{tr} power density associated with phase transformations

$$q^{\text{tr}} = \sum \Delta H_k dy_k / dt$$

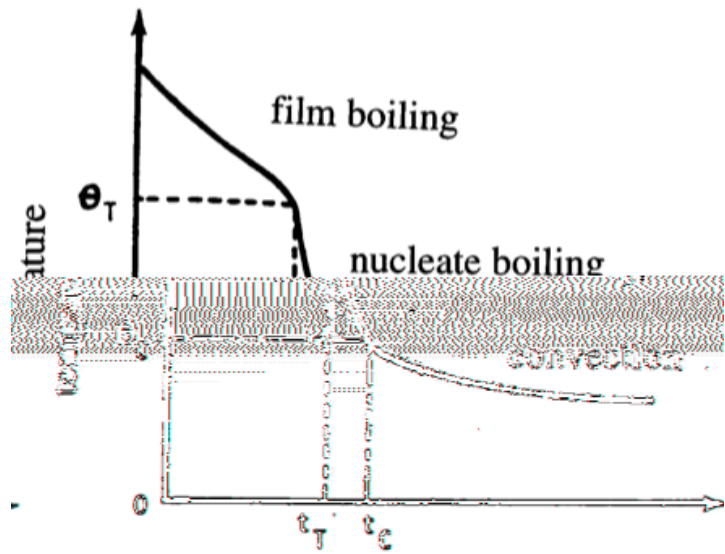
- mixture laws for thermophysical properties

$$\lambda = \sum \lambda_k y_k \quad c_p = \sum c_{pk} y_k \quad \rho = \sum \rho_k y_k \quad y_k \text{ volume fraction of phase } k$$

- surface boundary condition for quenching :

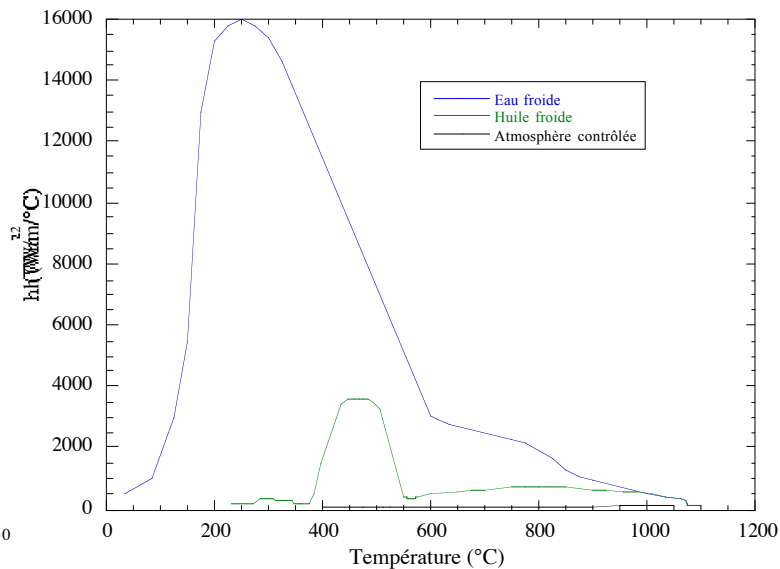
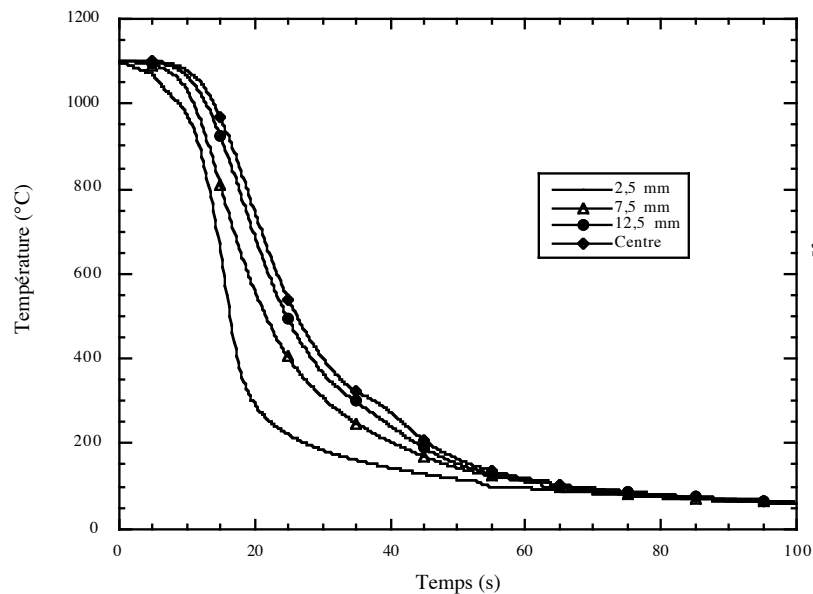
$$\Phi = -\lambda (\partial T / \partial n) = h (T_s - T_\infty)$$

Heat transfer in vaporizable fluids (water, oil, ...)



h (surface temperature, temperature and agitation of the bath, surface state, position of the piece ...) difficult to determine \longrightarrow **inverse methods**

Ex : quenching in cold water (cylinder diameter 35mm length 105 mm steel 35MnV7)



Modelling of heat transfer in the solid

Heat conduction equation : $\text{div} (\lambda \text{grad} T) + q^{\text{tr}} = \rho c_p \delta T / \delta t$

- q^{tr} power density associated with phase transformations

$$q^{\text{tr}} = \sum \Delta H_k dy_k / dt$$

- mixture laws for thermophysical properties

$$\lambda = \sum \lambda_k y_k \quad c_p = \sum c_{pk} y_k \quad \rho = \sum \rho_k y_k \quad y_k \text{ volume fraction of phase } k$$

- surface boundary condition for quenching :

$$\Phi = -\lambda (\partial T / \partial n) = h (T_s - T_\infty)$$

most often : inverse methods to determine $h(T_s)$
(due to the complexity of heat transfer mechanisms particularly in vaporizable fluids)

Promising approaches : coupling with fluid flow simulations

→ gaz quenching

→ heating in furnace

Modelling of flow and heat transfer in the gaz (code Fluent)

- Solution of equations
- Navier-Stokes
 - continuity
 - heat transport
 - turbulence transport : chosen model : $k-\omega$

thermophysical properties of gaz: dynamic viscosity, thermal conductivity, density, specific heat

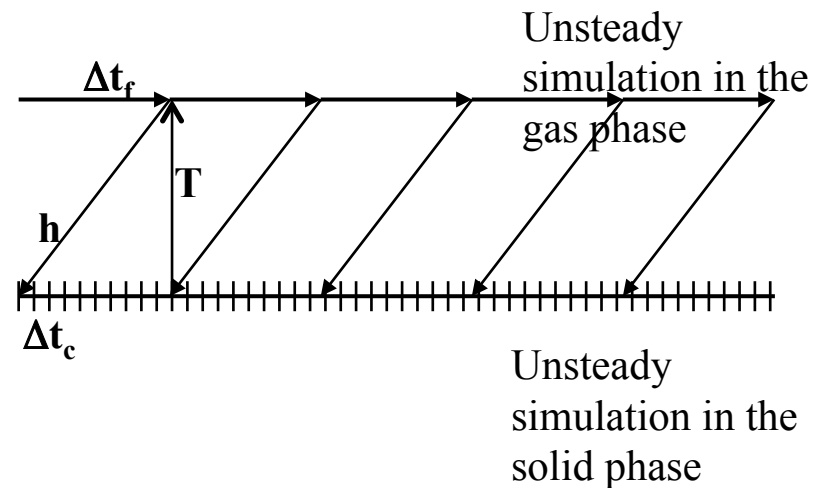
Prediction of the local wall heat transfer coefficient distributions

surface boundary condition for the solid :

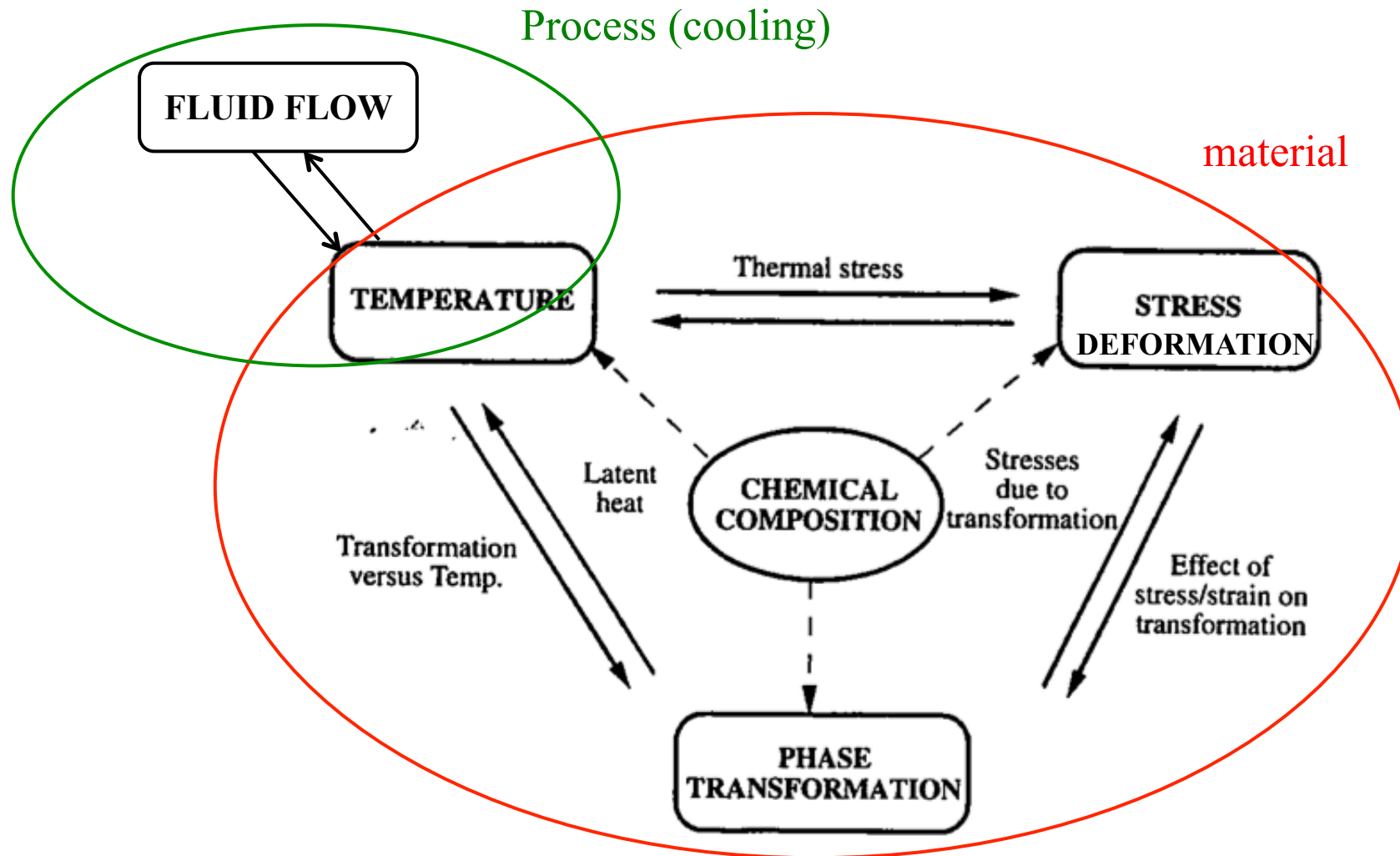
$$\Phi_{loc} = -\lambda (\partial T / \partial n) = h_{loc} (T_p - T_{\infty})$$

Coupling with simulations in the solid

Coupling between FLUENT
and SYSWELD



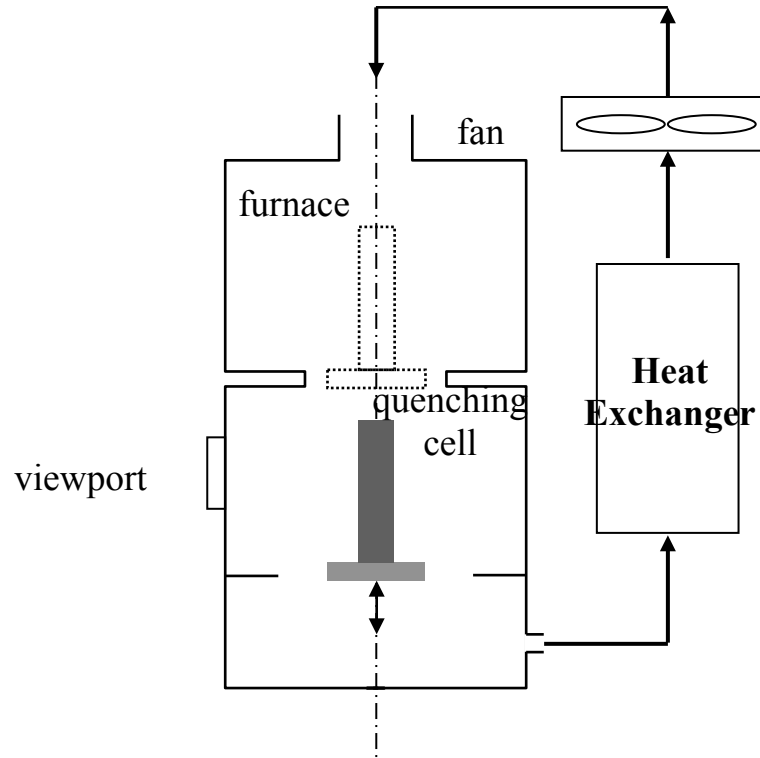
Fluid – thermal – metallurgical – mechanical couplings in heat treatment



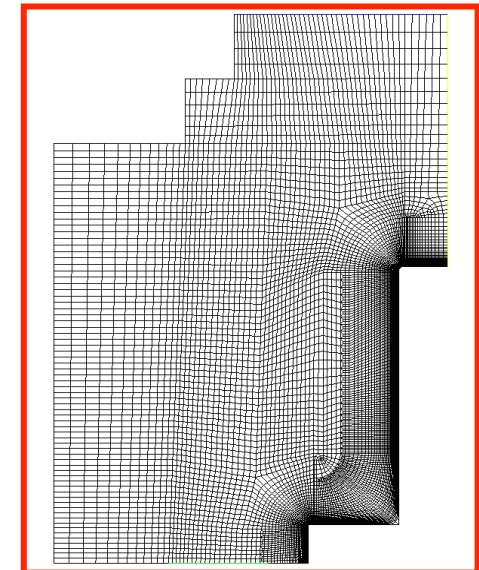
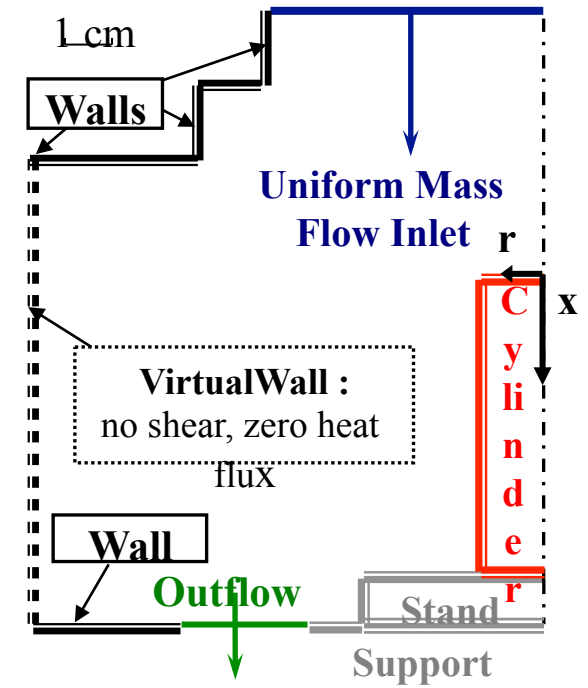
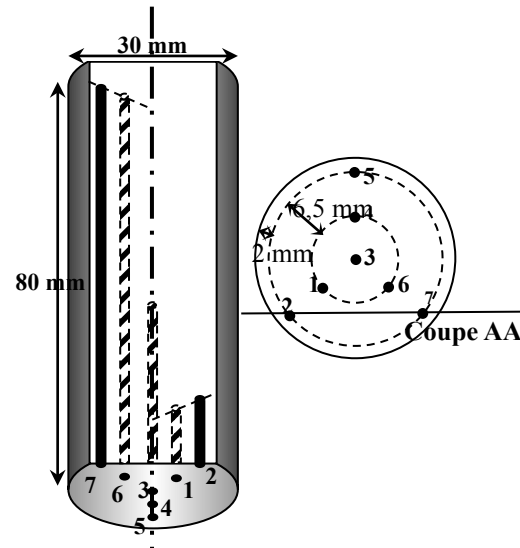
2 examples : gaz quenching
quenching and tempering

Application example : gaz quenching

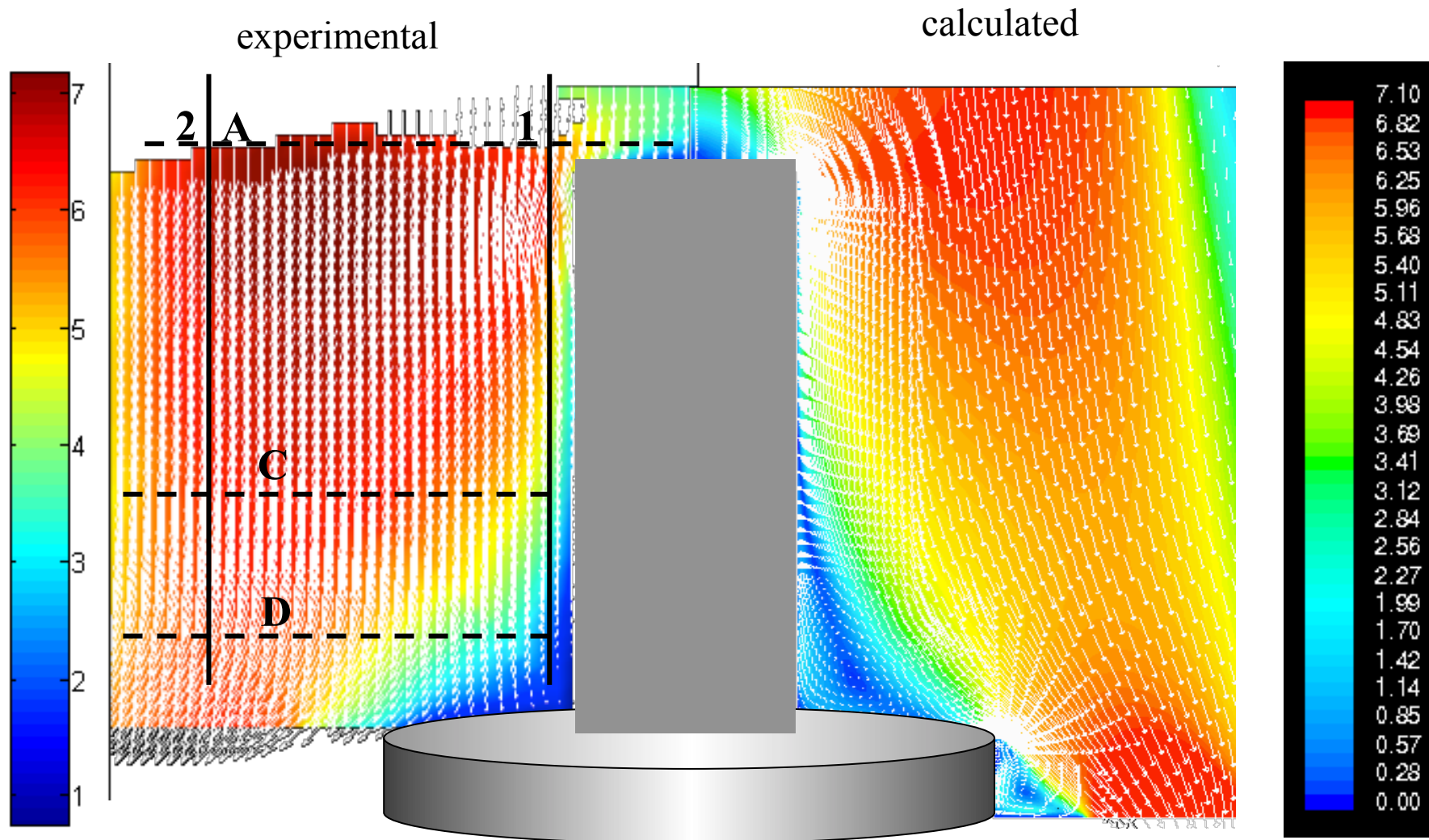
Experimental device from Ecole des Mines d'Albi



- nickel
- 27MnCr5 steel

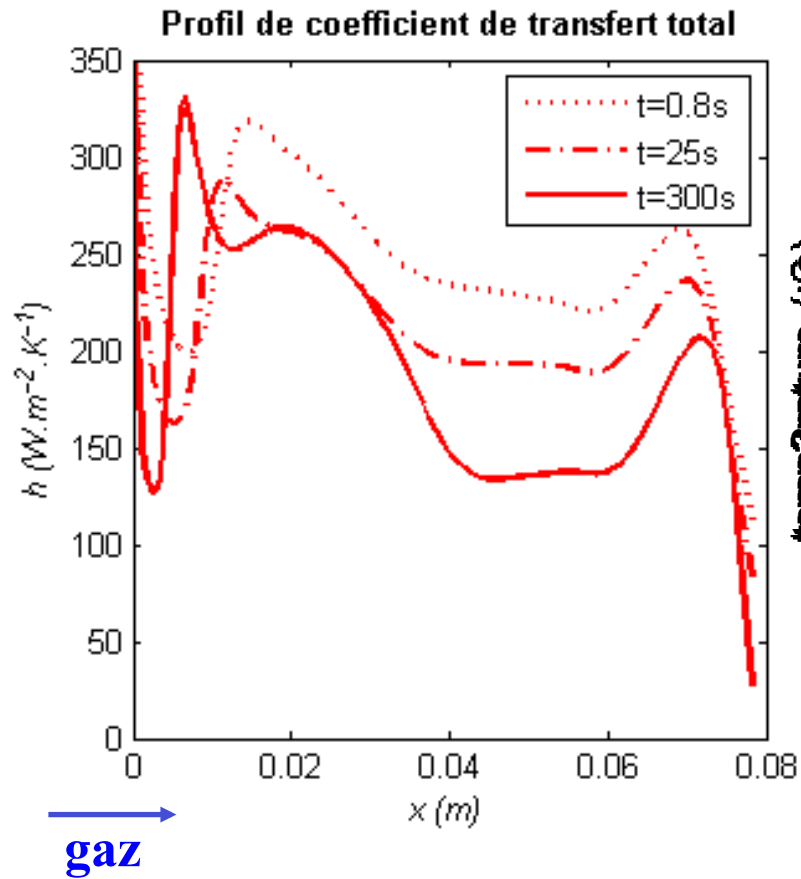


4.5 bar helium gas quenching velocity vectors

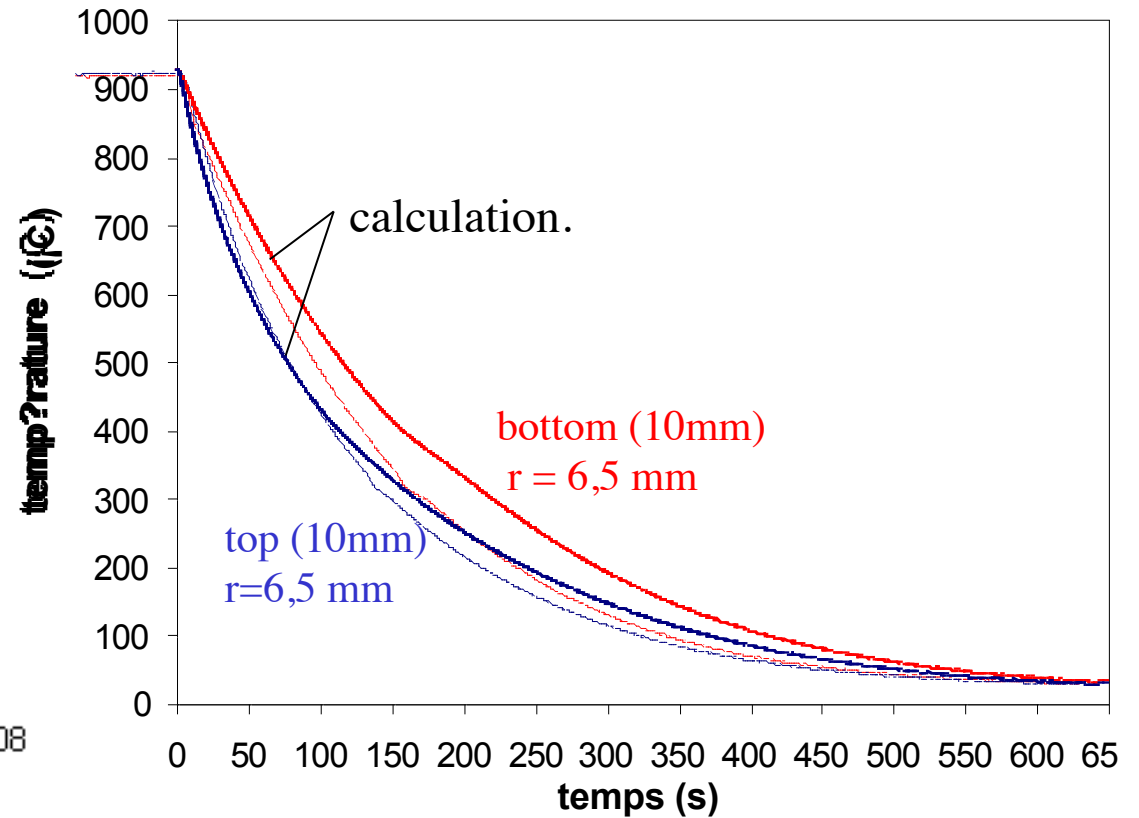


4.5 bar helium gas quenching of a nickel cylinder

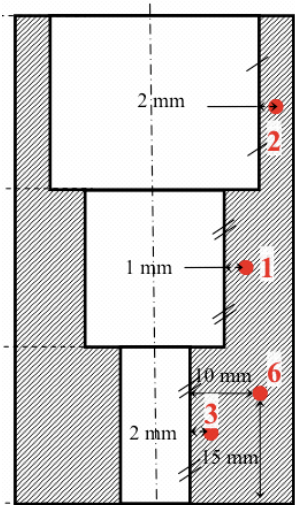
Heat transfer coefficient distribution
along lateral surface



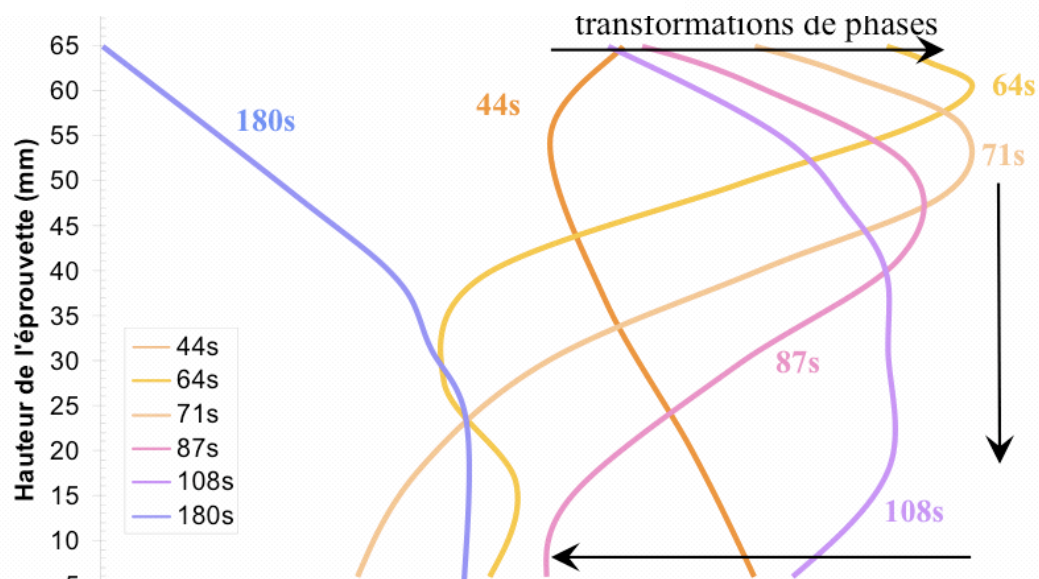
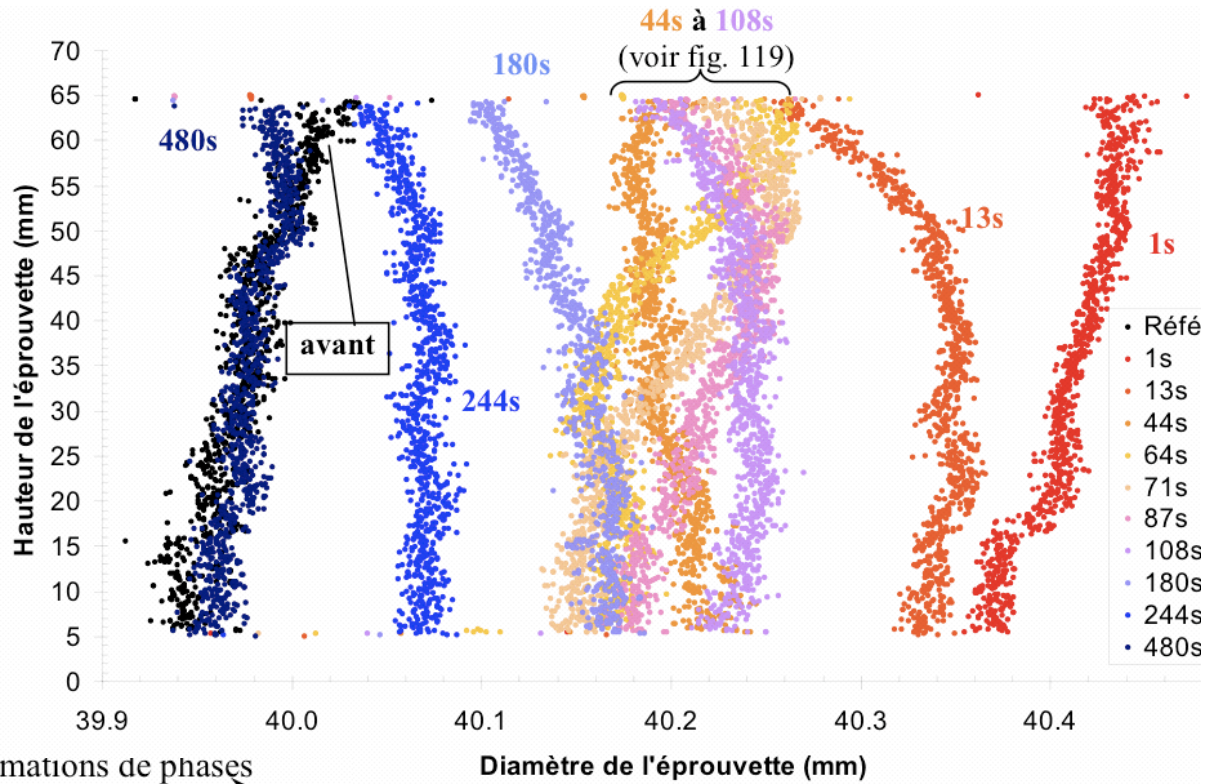
Temperature evolutions
measured- calculated



In-situ measurements of the deformation



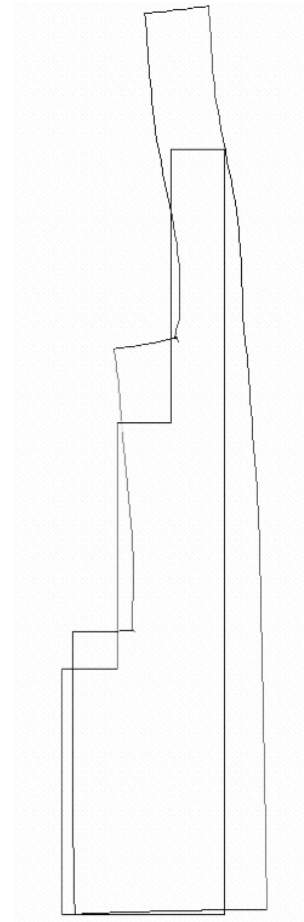
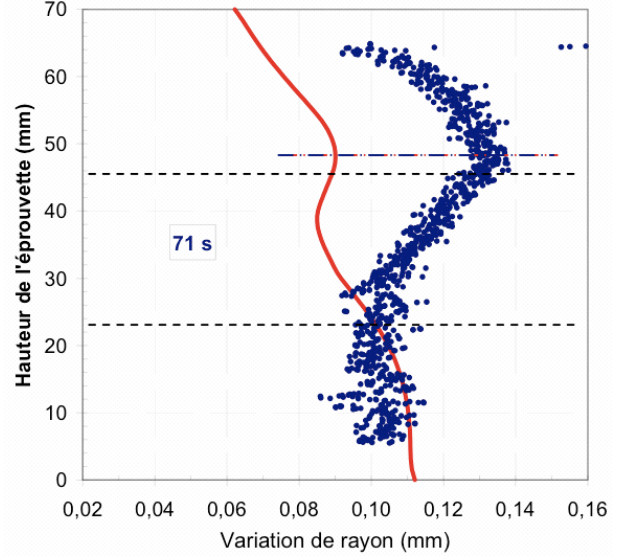
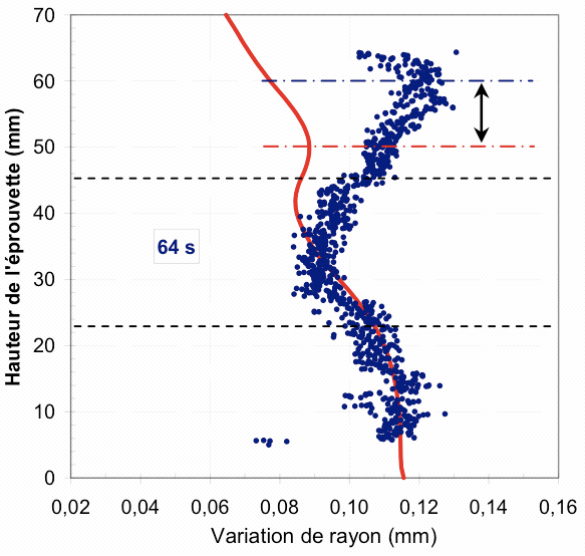
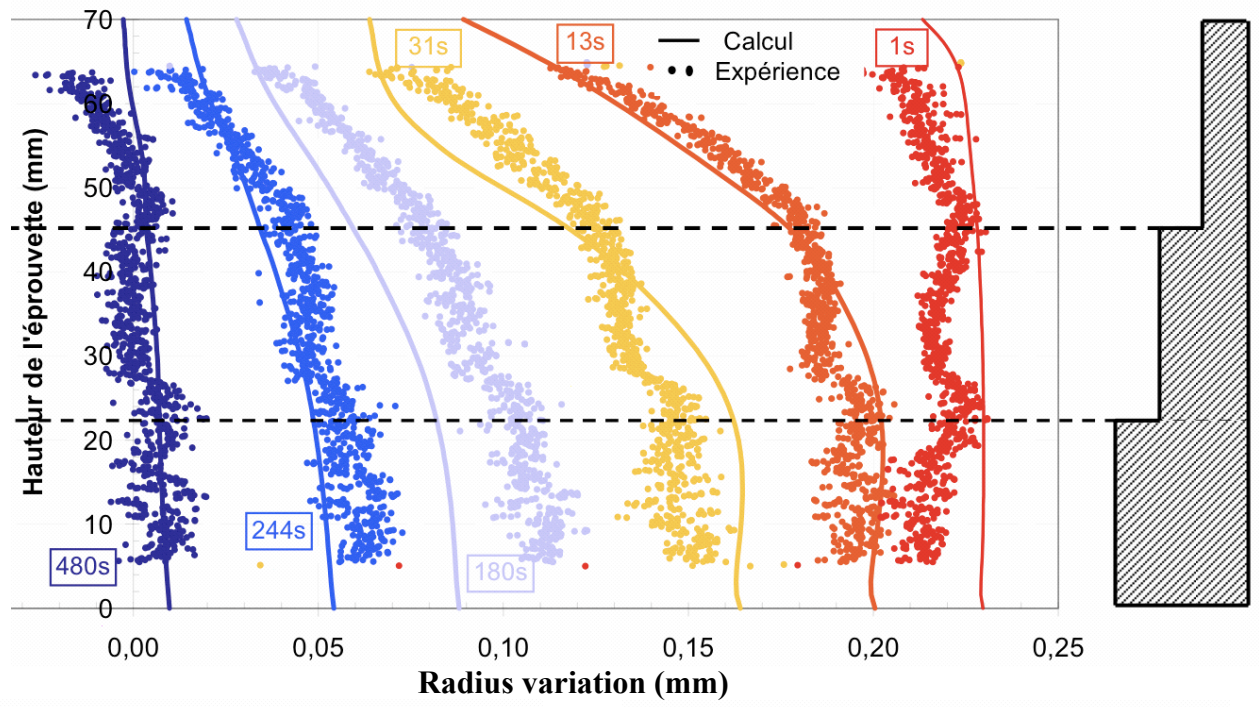
Ext. diam. : 40mm
Height : 70mm



27MnCr5 steel

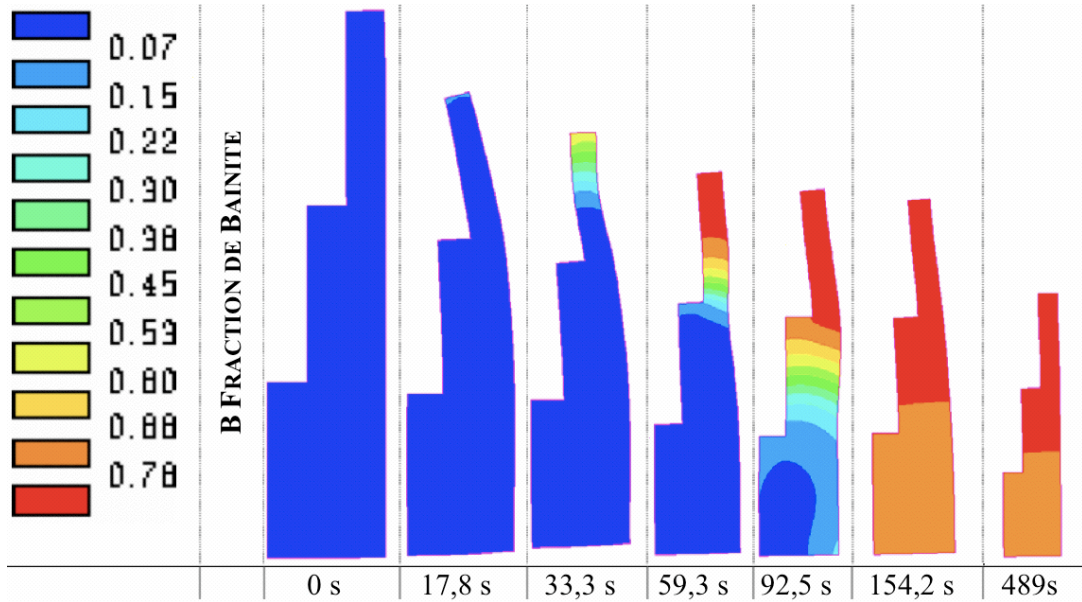
Dissertation J.F. Douce, 2008

Comparison measurements - calculation

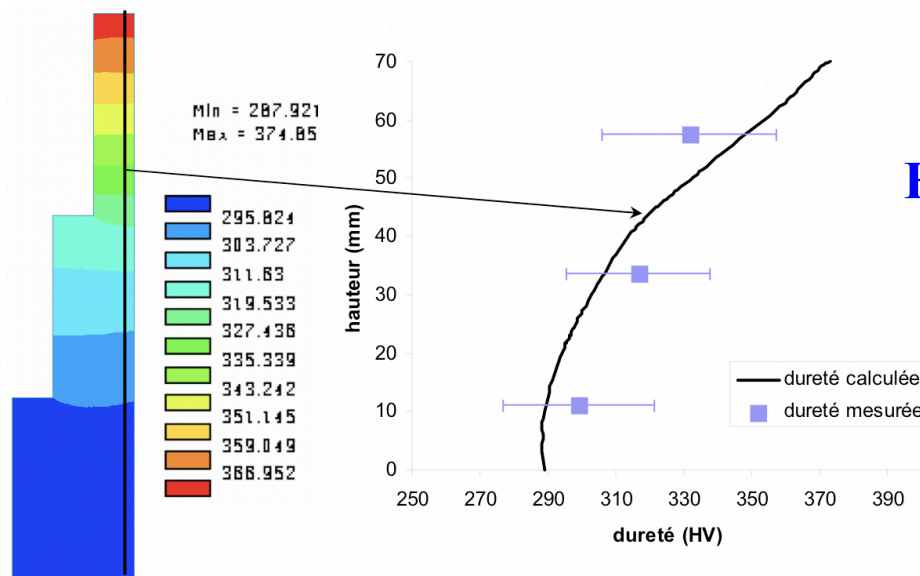
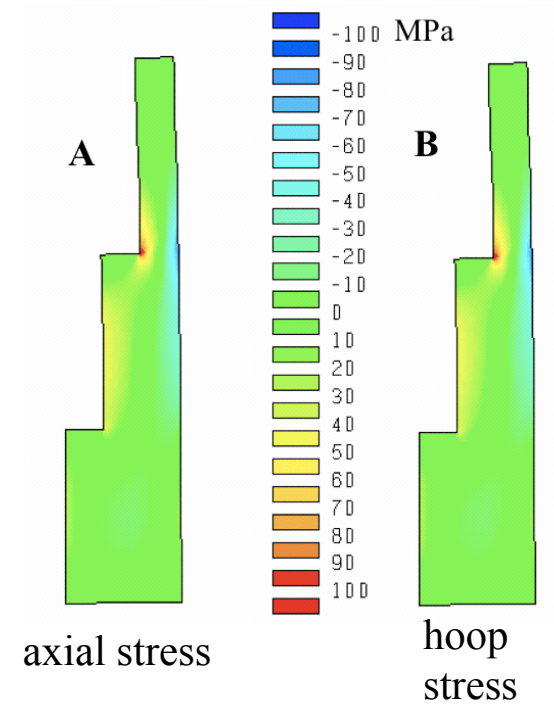


Final shape

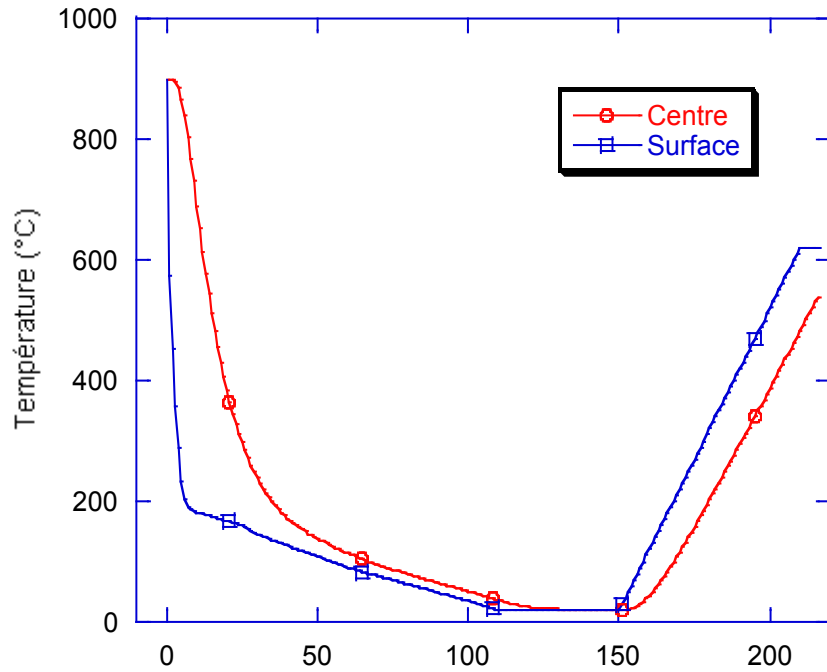
Distributions of microstructures



Residual stresses



Chain different processes : quenching + tempering (FE code ZeBuLon)

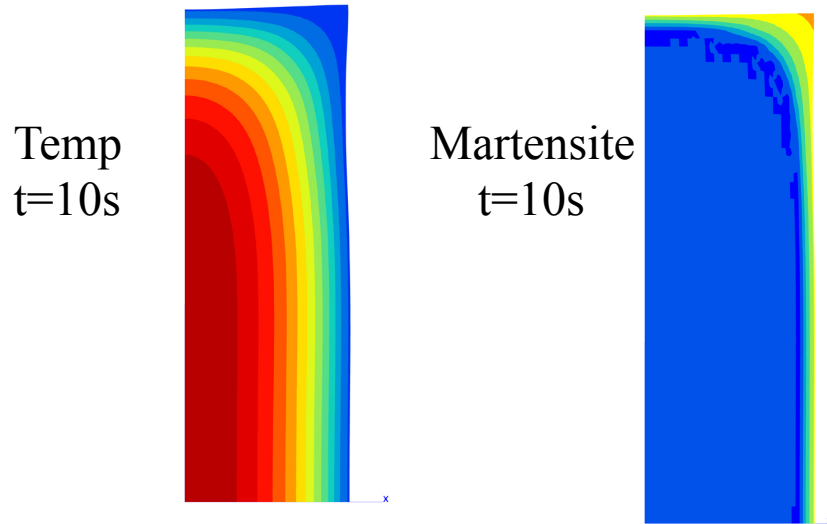


cylinder Φ 35mm L 105 mm

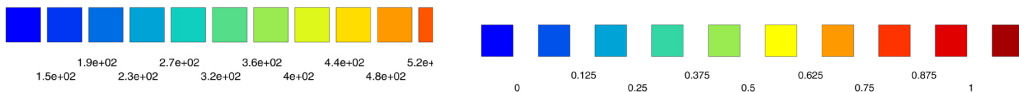
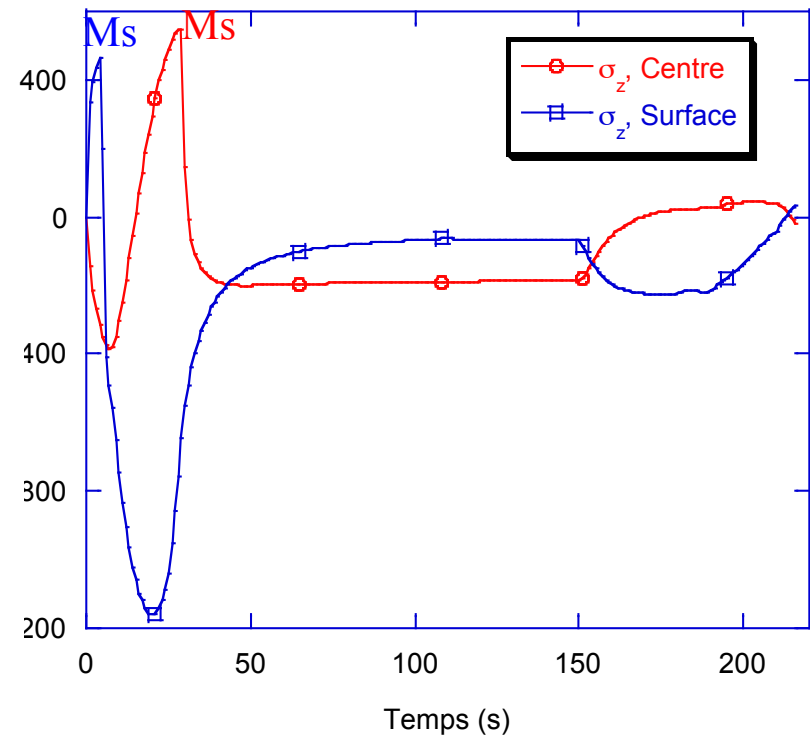
steel 60NiCrMo11

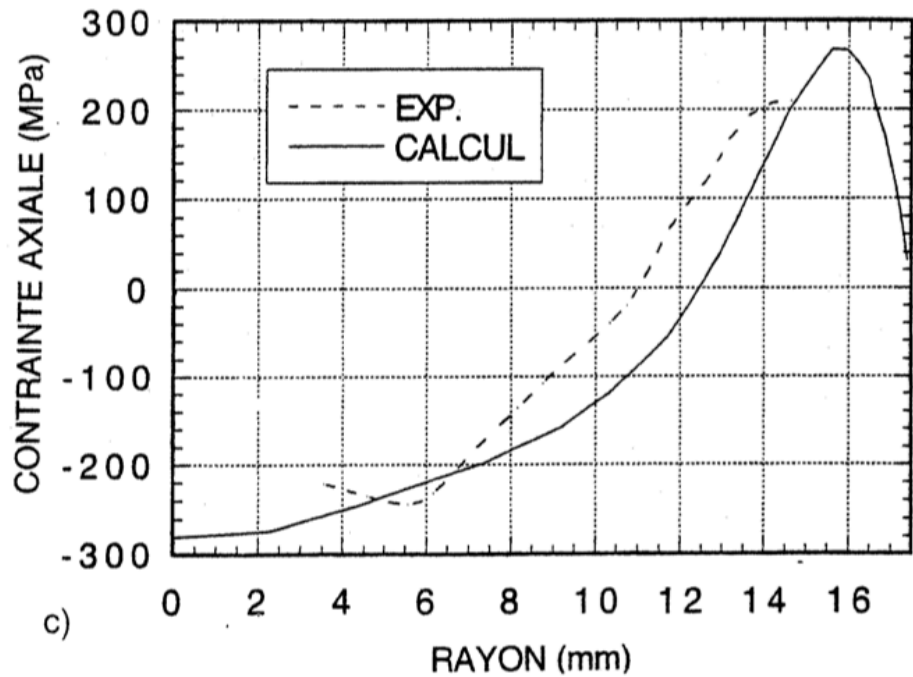
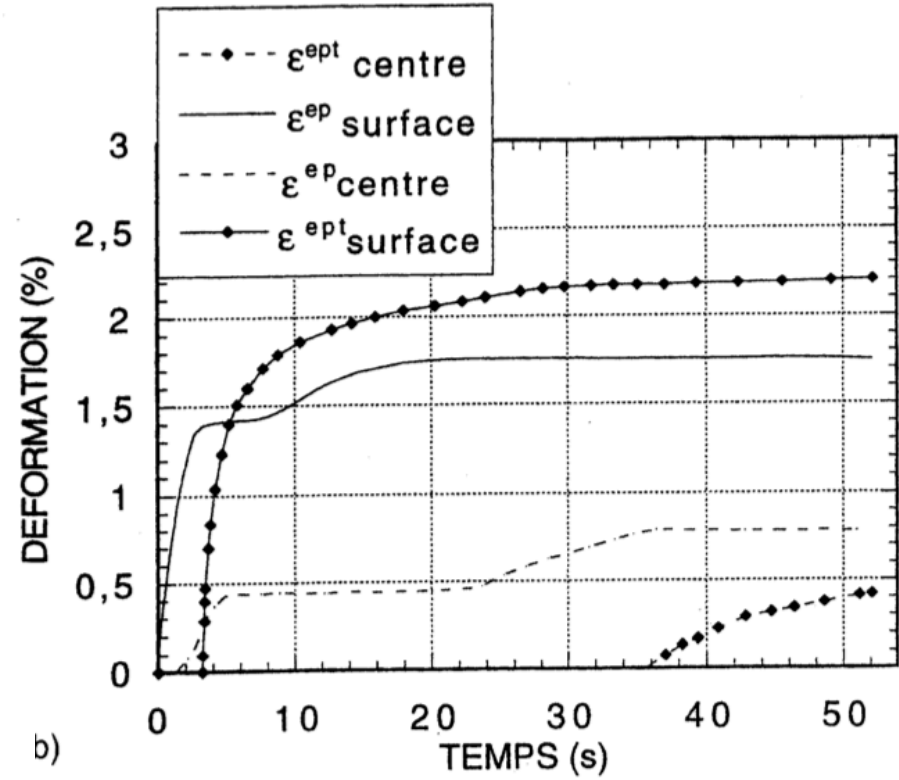
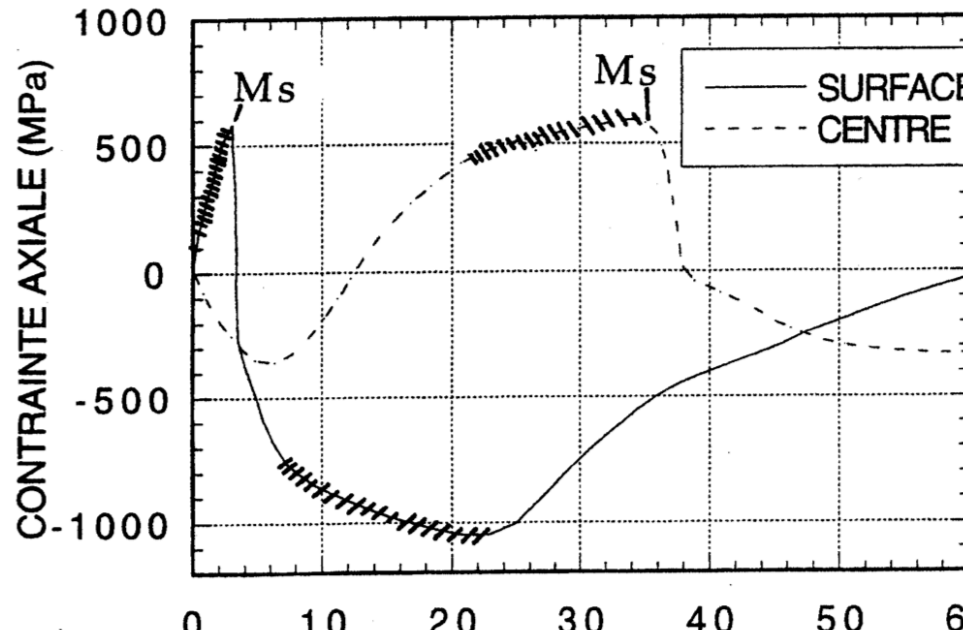
martensitic quenching from 900°C to 20°C

tempering 10°C/s 600°C

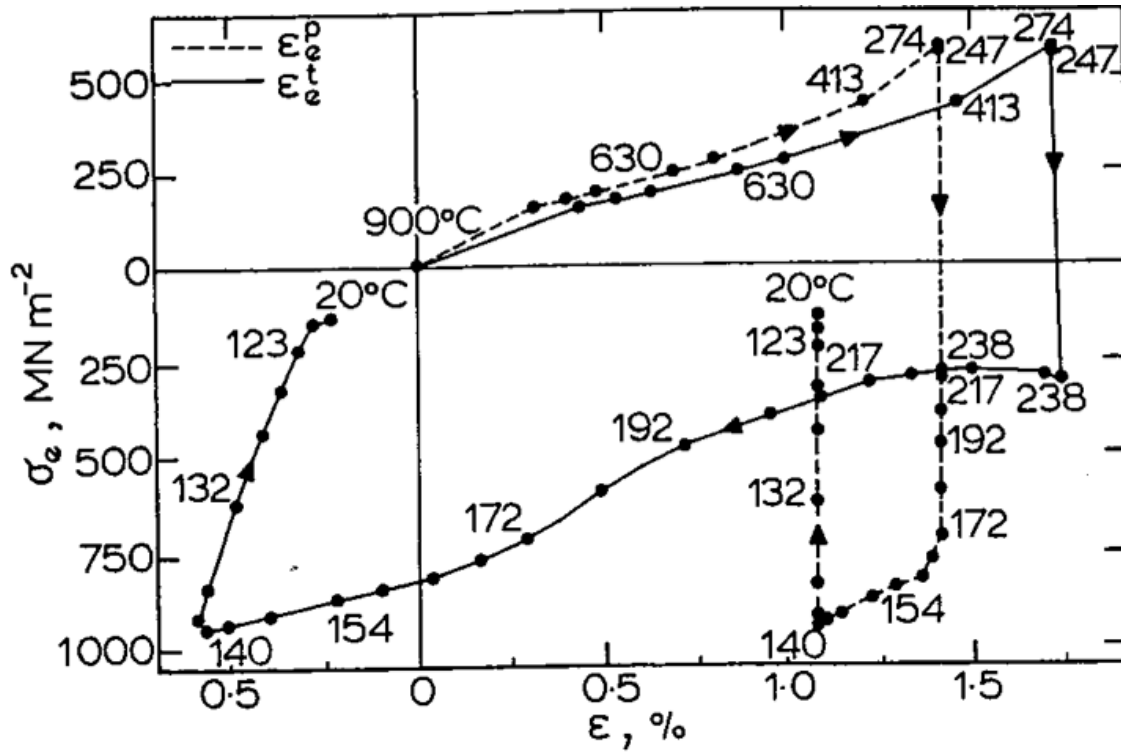


Axial stress evolution





Quenching



Calculated loading paths

Von Mises stress σ_e

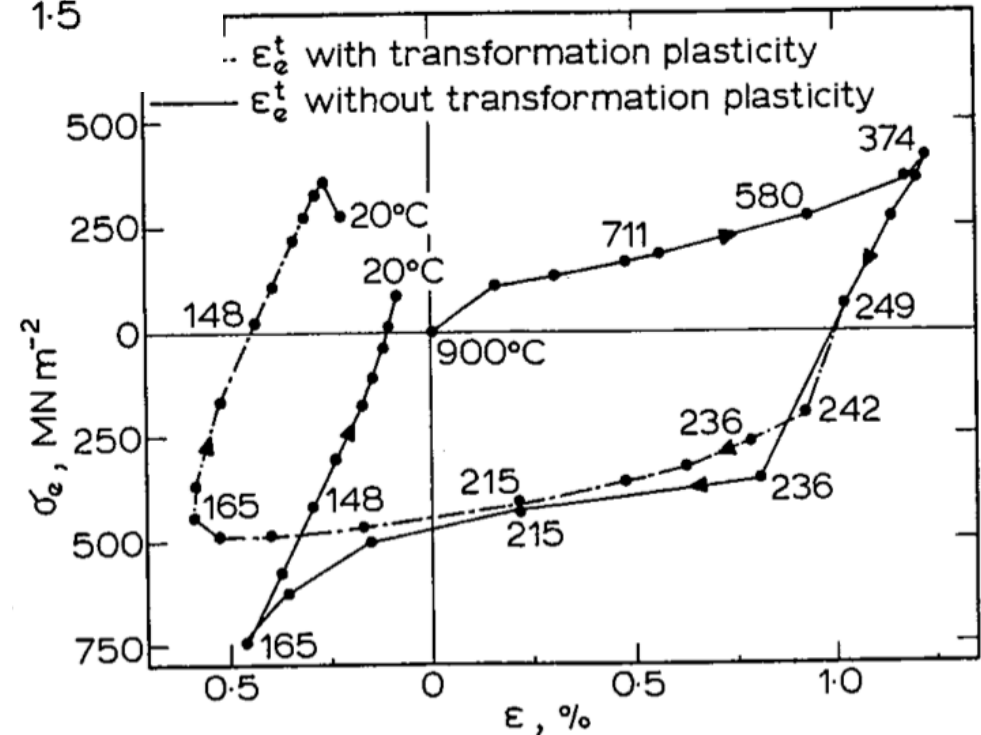
equivalent plastic strain

$$\epsilon_e^p = \sqrt{2/3} \epsilon_{ij}^p \epsilon_{ij}^p$$

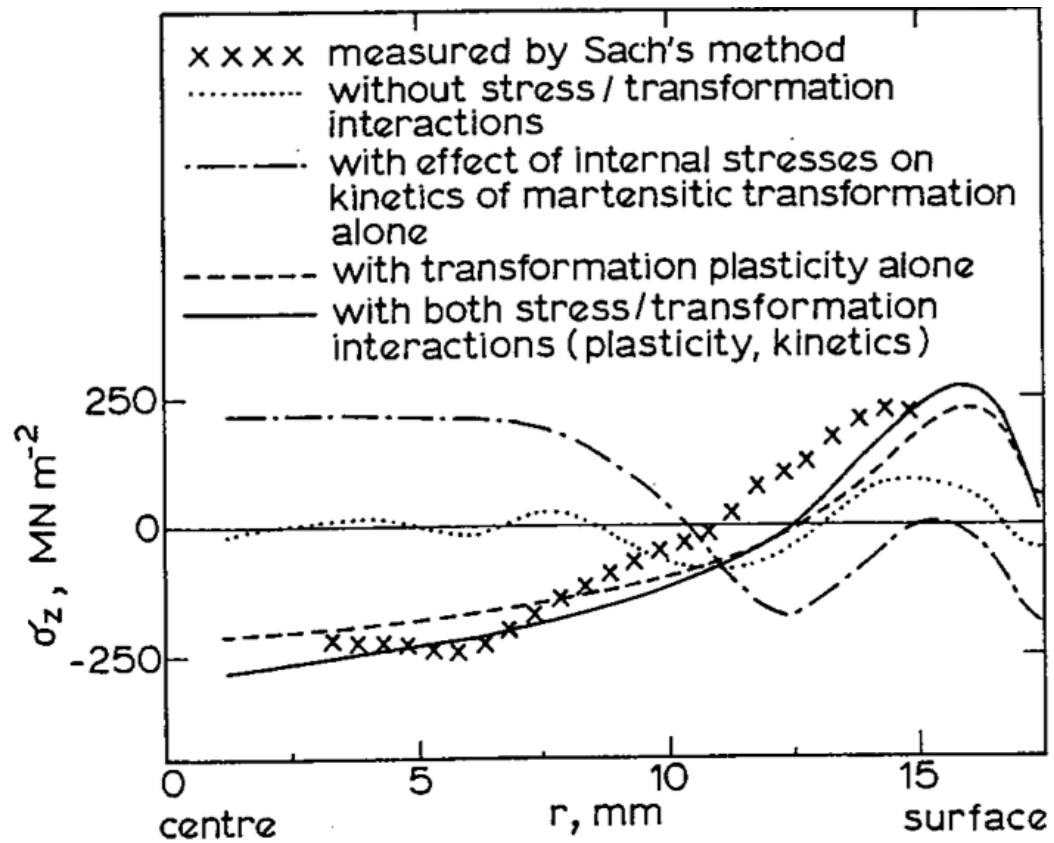
equivalent total strain

$$\epsilon_e^t = \sqrt{2/3} \epsilon_{ij}^t \epsilon_{ij}^t$$

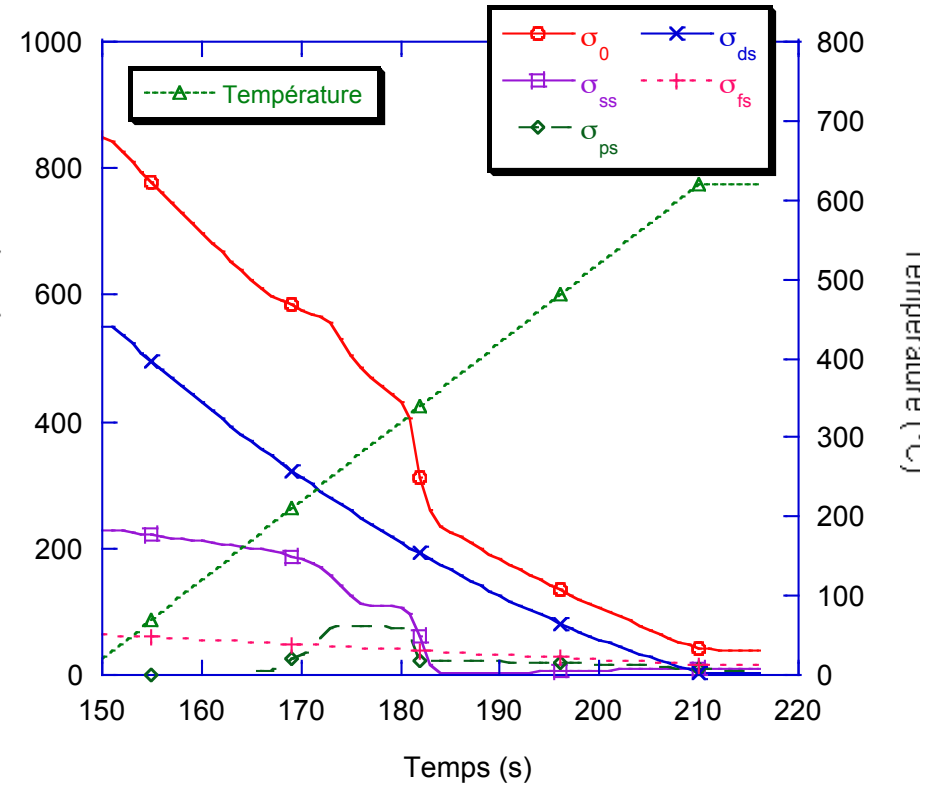
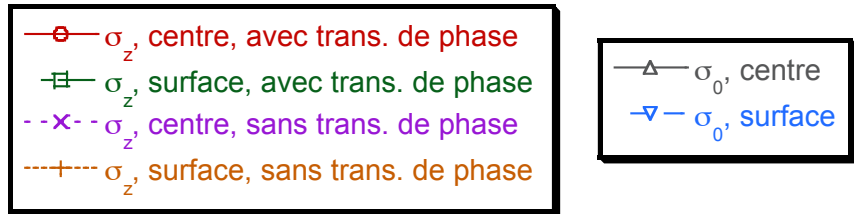
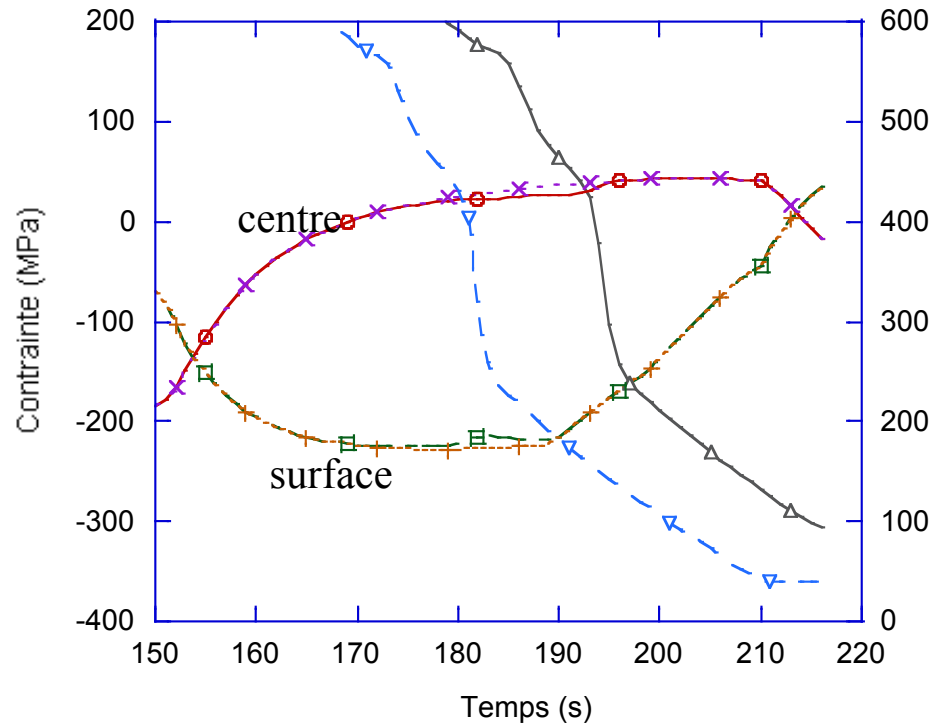
Role of transformation plasticity during quenching



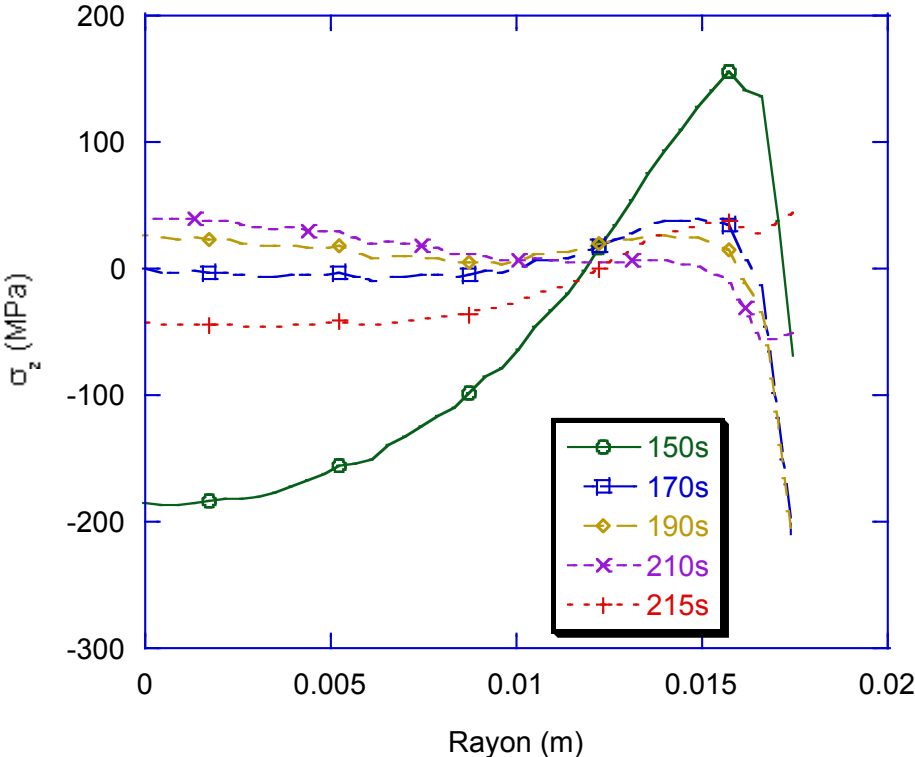
Residual stress profiles after water quenching
Role of stress – phase transformation
interactions



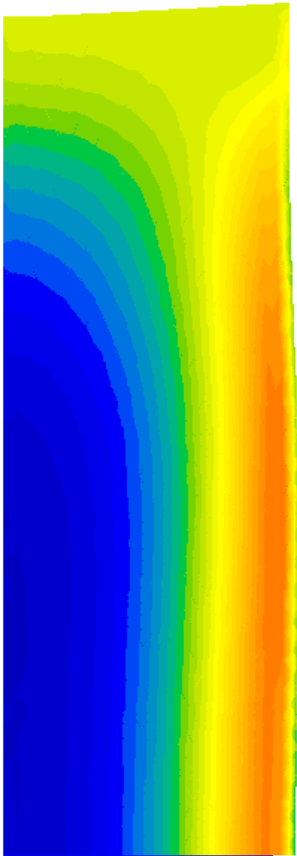
Relaxation of quenching residual stresses during tempering



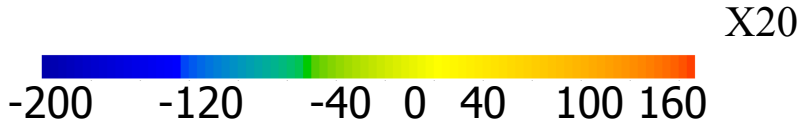
Evolutions of stress profiles during tempering



End of quenching



End of heating



Conclusion - Future developments

- **Material point of view**

- **metallurgical models**

- . global models work for industrial metallic alloys and complex situations → prediction of volume fractions

- . models including nucleation and growth develop more and more for multicomponent alloys

- volume fractions and morphological parameters allow prediction of mechanical properties

- (Al alloys, Ti alloys...)

- . *take into account chemical composition gradients (carburizing, carbonitriding, solidification segregations)*

- . *effects of prior plastic deformations, effect of stresses*

Conclusion - Future developments

- thermomechanical behaviour with phase transformations

. phenomenological laws exist

extension to other metallic alloys (Ti alloys...)

better description of multiphase material (morphology)

micro-macro approaches must be further developed

• Heat treatment processes

- coupling between fluid and solid :

developments for vaporizable fluids (experiments and modelling)

- include heating processes (furnace, induction...)

- *in situ measurements (solid + fluid) for rapid processes*

- *chain different processes :*

liquid metal processing + solidification + forming + heat treatment...

• Numerical aspects

Two review papers

S. Denis, Revue de Métallurgie CIT/SGM, février 1997, pp. 157-176

S. Denis, P. Archambault, E. Gautier, A. Simon, G. Beck, JMEPG Vol 11 (1), 2002, pp. 92-102

Common work with

A. Simon, E. Aeby-Gautier, P. Archambault, J.P. Bellot, B. Appolaire

PhD students :

FMB Fernandes, C. Basso, J.P. Josserand, D. Farias, F. Saliou, M. Boufoussi, J.F. Ganghoffer, Y. Wen, L. Massicart, M. Zandona, A. Mey, P. Brunet, C. Aubry, J. Ch. Louin, M. Veaux, Y. Wang, Y. Renault, J.F. Douce, M. Haering, L. Mangin, S. Devynck, S. Catteau...

In the frame of industrial collaborations

CETIM, ARBED, UNIMETAL RECHERCHE, PSA, RENAULT, Creusot Loire Industrie, French Programme SIMULFORGE, European programme VHT, Ascometal, Air Liquide, SNR, Vallourec, ...