Genèse des Microstructures I

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Introduction

Characterization methods
Local : OM, SEM, EBSD, TEM, APT, …
Global : XRD, DSC, SAS, …

Mechanisms

Diffusion

Dislocation motion

Interface motion

Solute effects

Stress effects

Microstructures

Macroscale : heterogeneity
Microscale : phases, grains
Sub-micro scale : phases, dislocation structures
Nanoscale : precipitates, clusters

Disciplins

Solidification

Phase transformations

Plasticity

Recrystallisaton & grain growth

Modelling methods

Microstructure II

Outline

1. **Introduction**

2. **Microstructure at the different scales: questions and answers (characterisation methods)**

    1. Micron scale : grains and phases
    2. Sub-micron scale : fine grains, dislocation structures, coarse precipitates
    3. Nanometre scale : precipitates

3. **How to be precise and realistic**

    1. Coupling local and global techniques
    2. The virtues of in-situ

4. **“Real” materials: what kind of complexity**

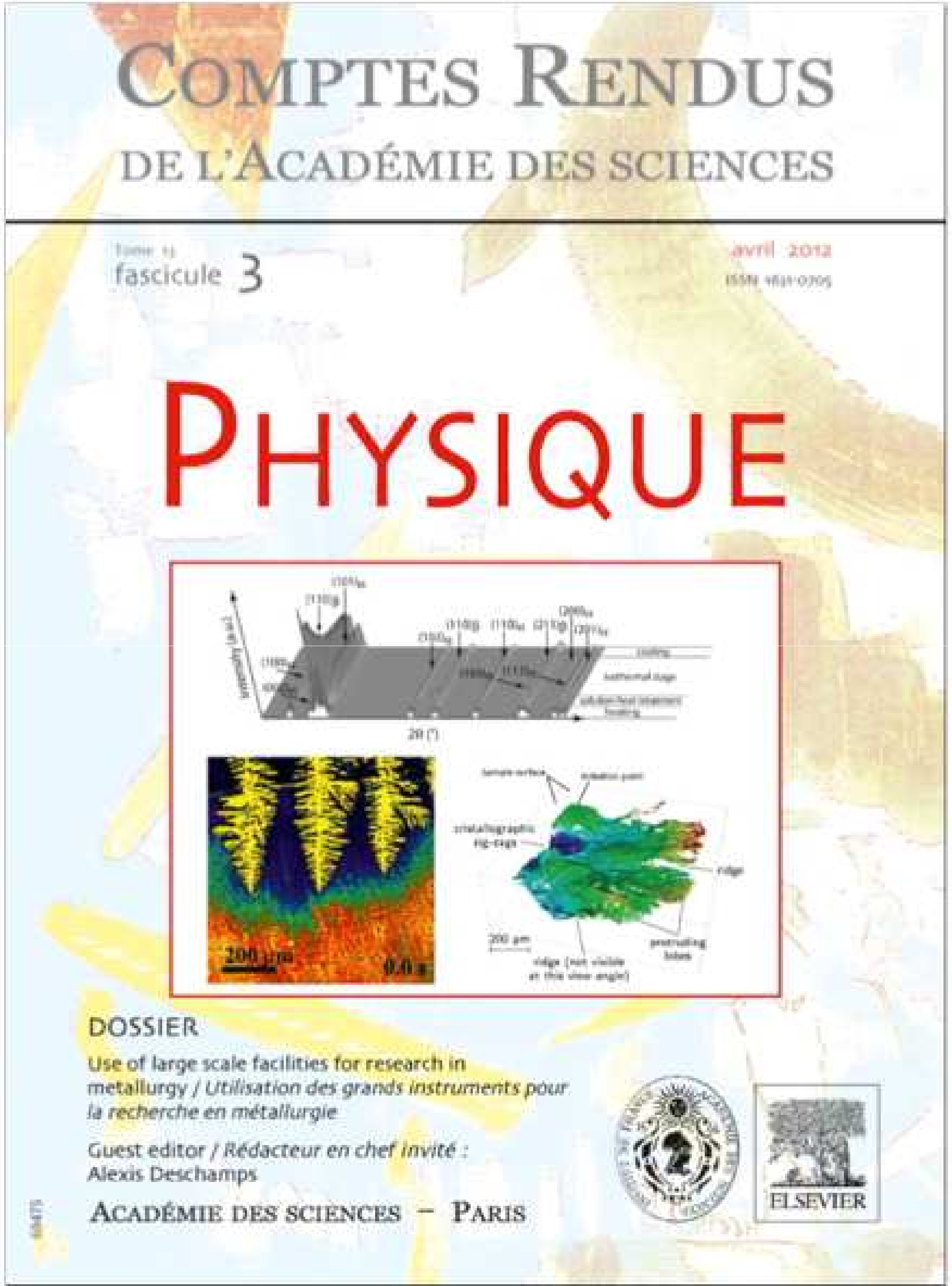
    1. Multi-constituent alloys
    2. Heterogeneous microstructures

5. **One prospect for the future : towards in-operando characterization?**

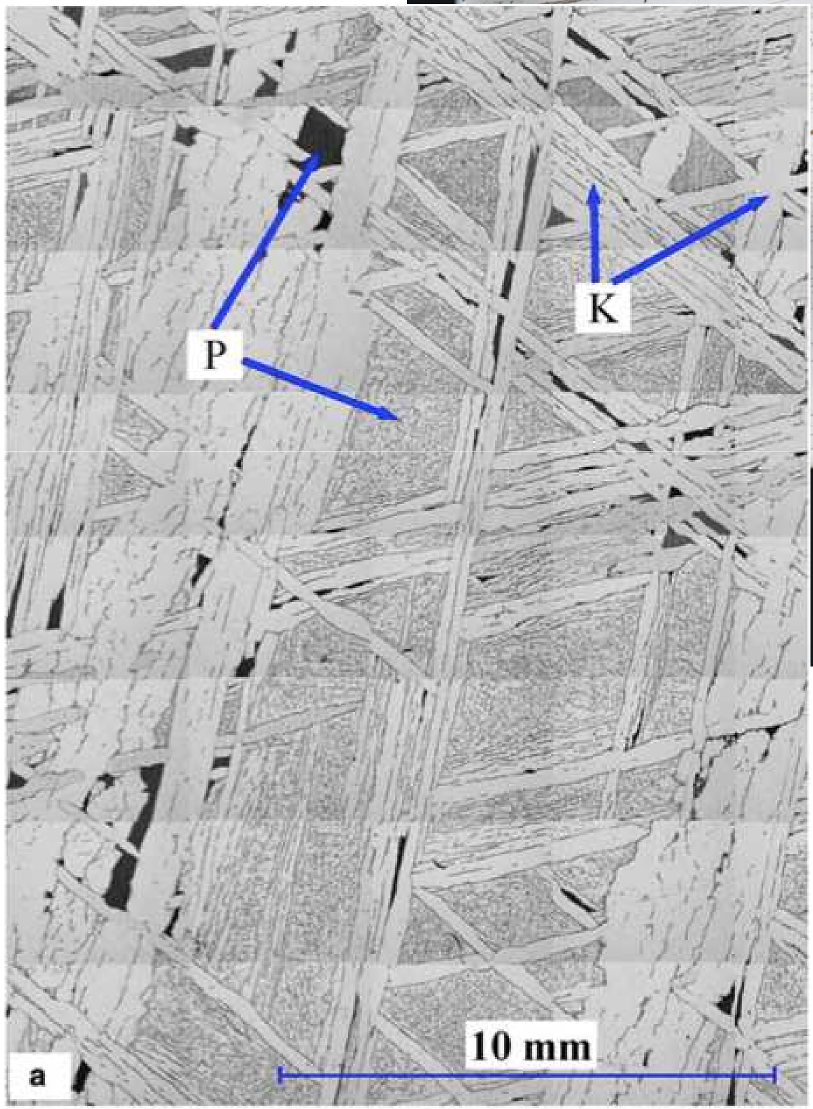
Use of large scale facilities for metallurgy

Special volume of Comptes Rendus de Physique

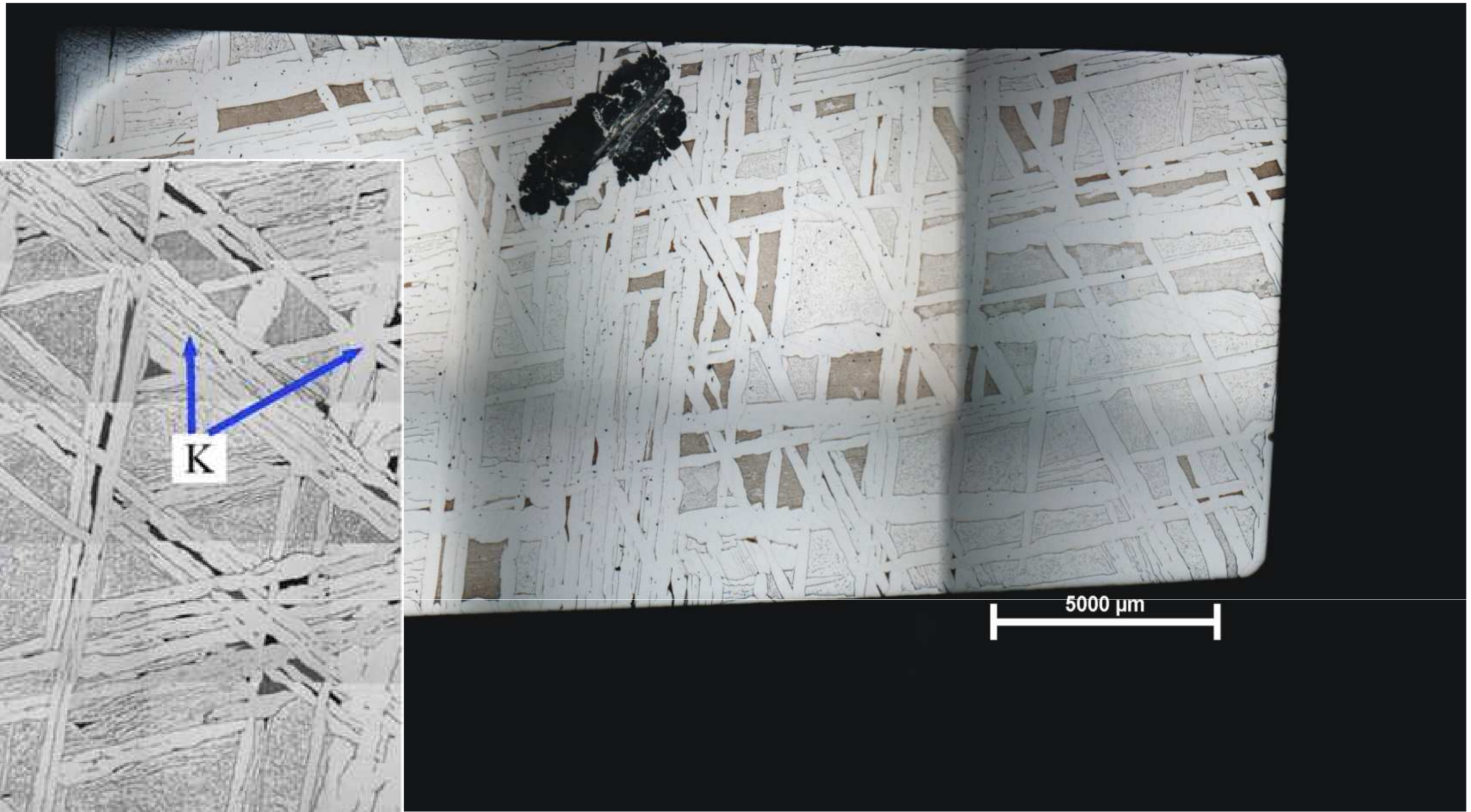
2012



Introduction



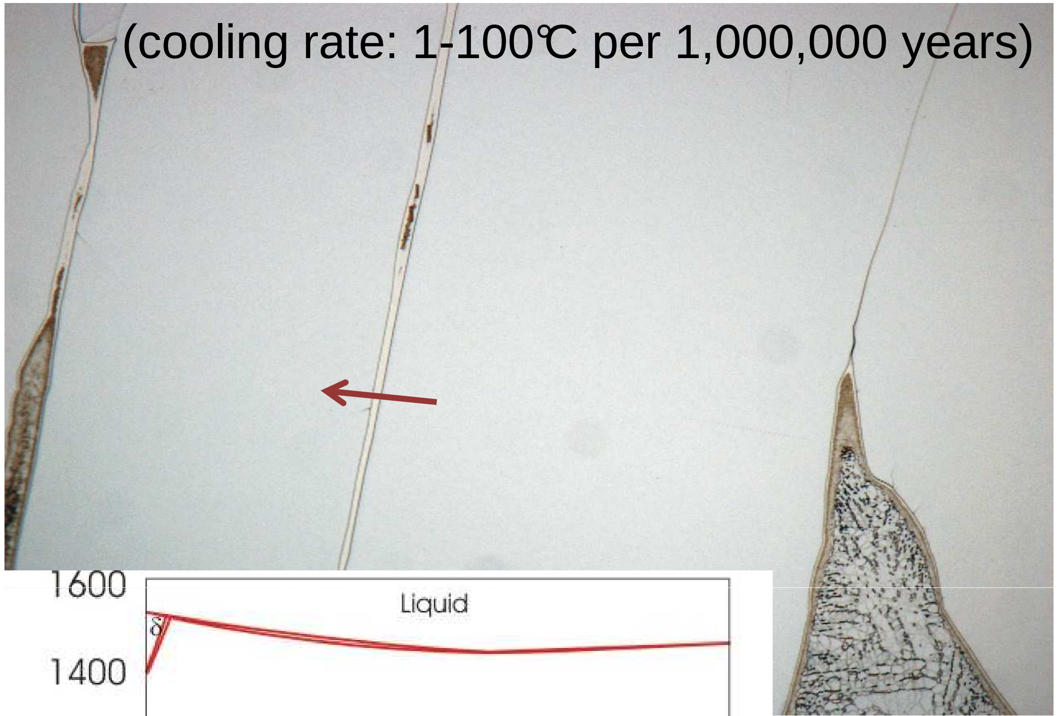
From: He et al. Acta Materialia 54 (2006) 1323–1334

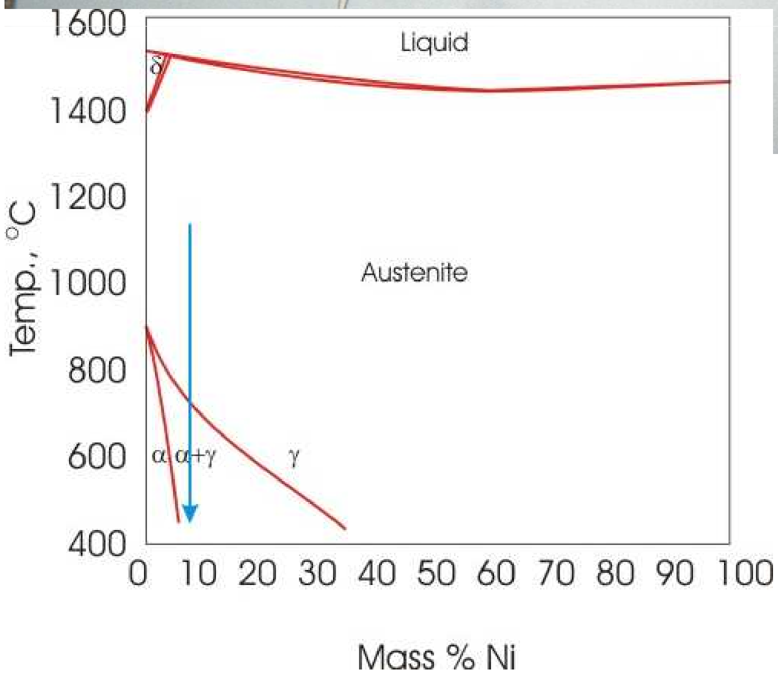


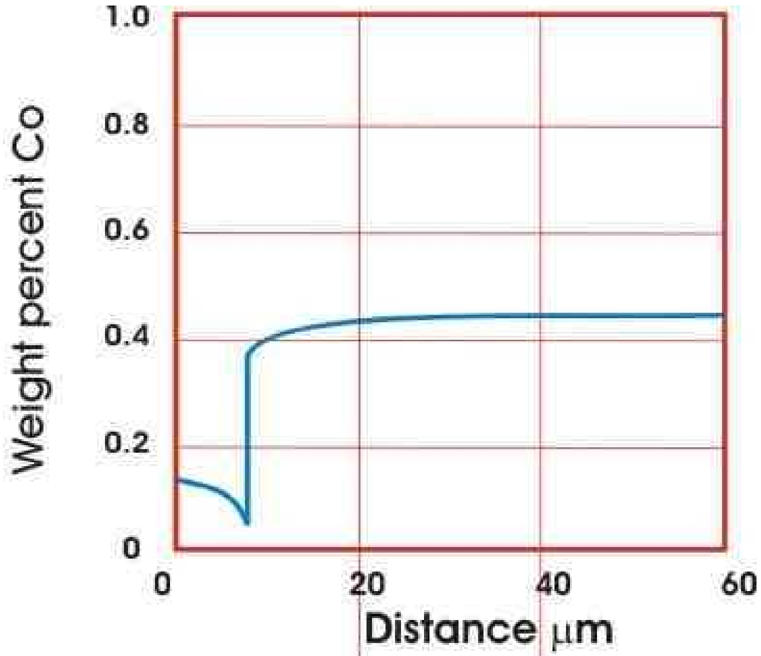
Gary Purdy, PTM 2010

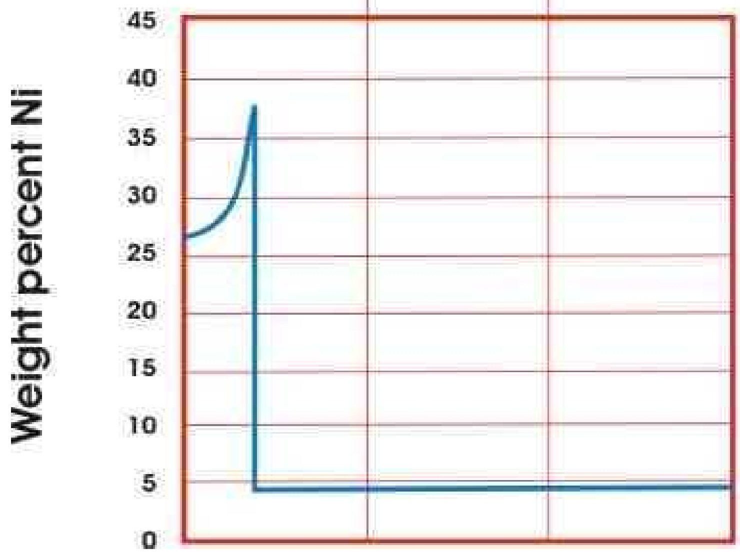
The Gibeon Meteorite, Namibia
(prehistoric, reported 1836)
Scatter field 275 km x 100 km
Fe-7.7%Ni-0.5%Co…..

kamacite (ferritic iron with up to 7.5% nickel)
taenite (fcc austenite with more than 25% nickel)
plessite









W. Rittel, Thesis, 2005 Delft University of Technology

Micro-scale

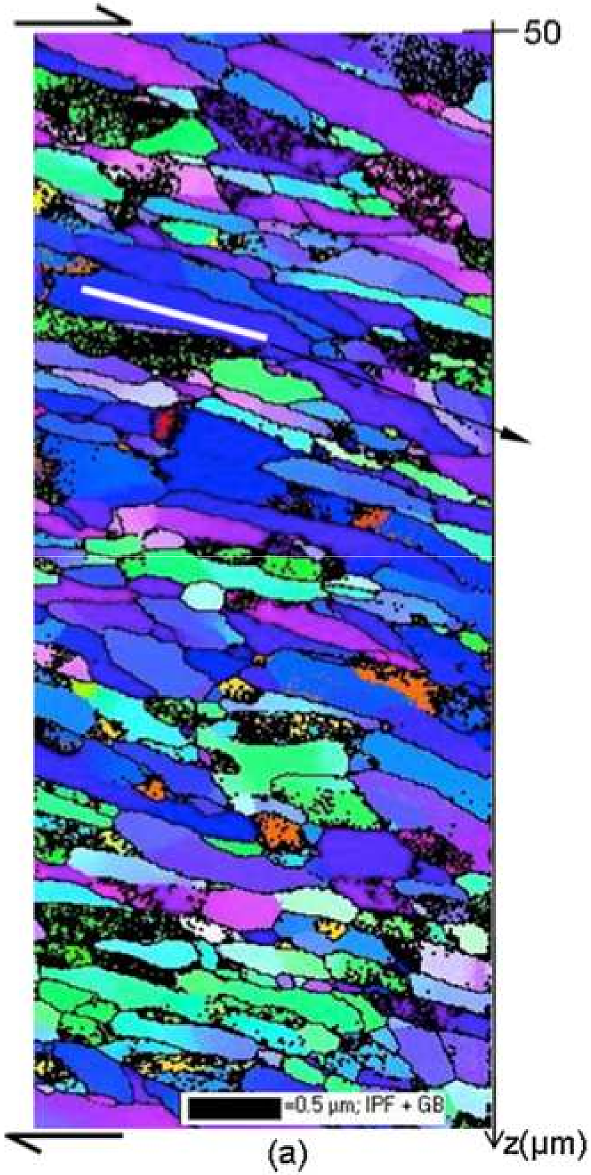
Microstructure at the microscale : grains and phases

Evolution during a long series of processes

Solidification
Homogenization
Hot working (rolling, forging, extrusion, …)
Cold working (rolling, …)
Forming (stamping, stretching, …)
Secondary processes (welding, fastening, …)
Evolution in service (ageing, creep, …)

To be characterized

Phase fractions
Shape and size
Interfaces (misorientation, orientation relationships)
Chemistry (non homogeneous)



From : P. Moeck et al. Cryst. Res. Technol. 46, No. 6 (2011)

Microstructure at the microscale : grains and phases

Phase characterization – structure – X-ray diffraction

Rietveld refinment → Phase proportions

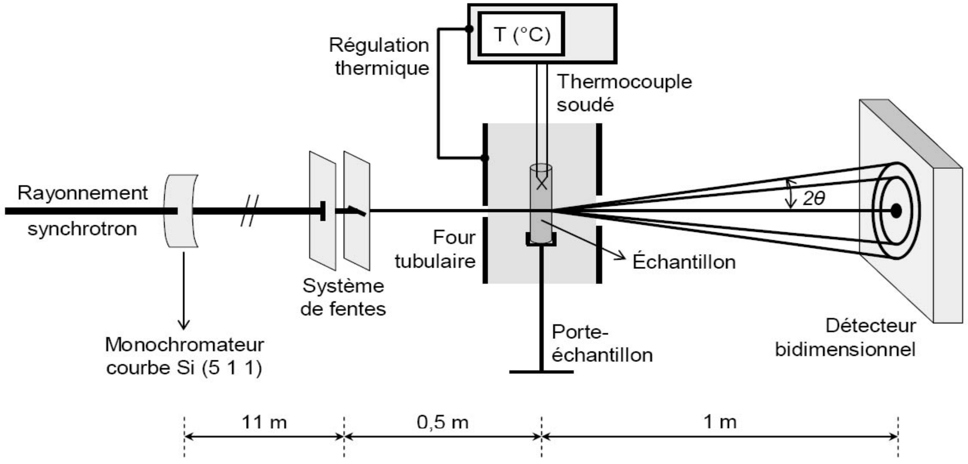
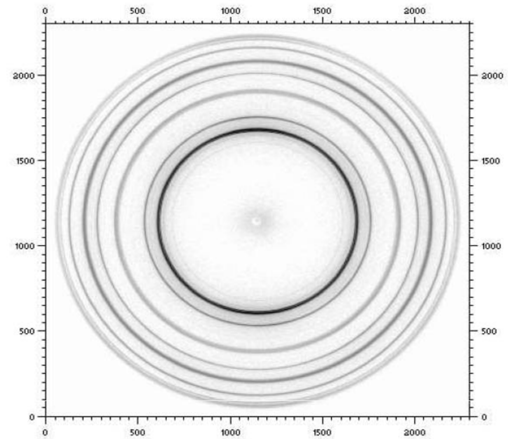
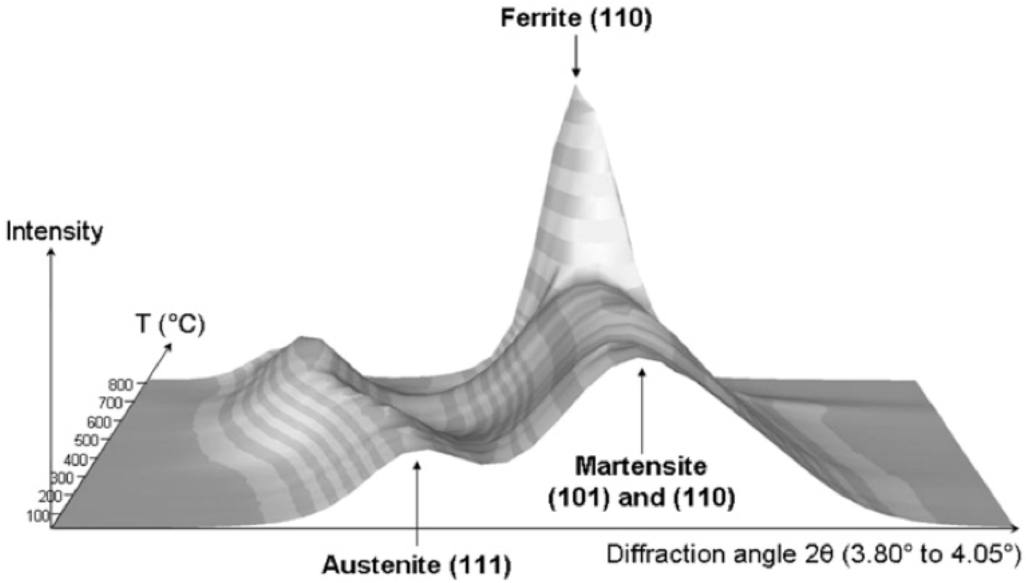


Figure 1-24. Schéma du montage expérimental avec détecteur bidimensionnel.





Phase characterization in steels

From : A. Béneteau
PhD thesis, INP Lorraine

Microstructure at the microscale : grains and phases

Phase characterization – structure – X-ray diffraction

In situ measurements - Phase fractions vs. time & temperature

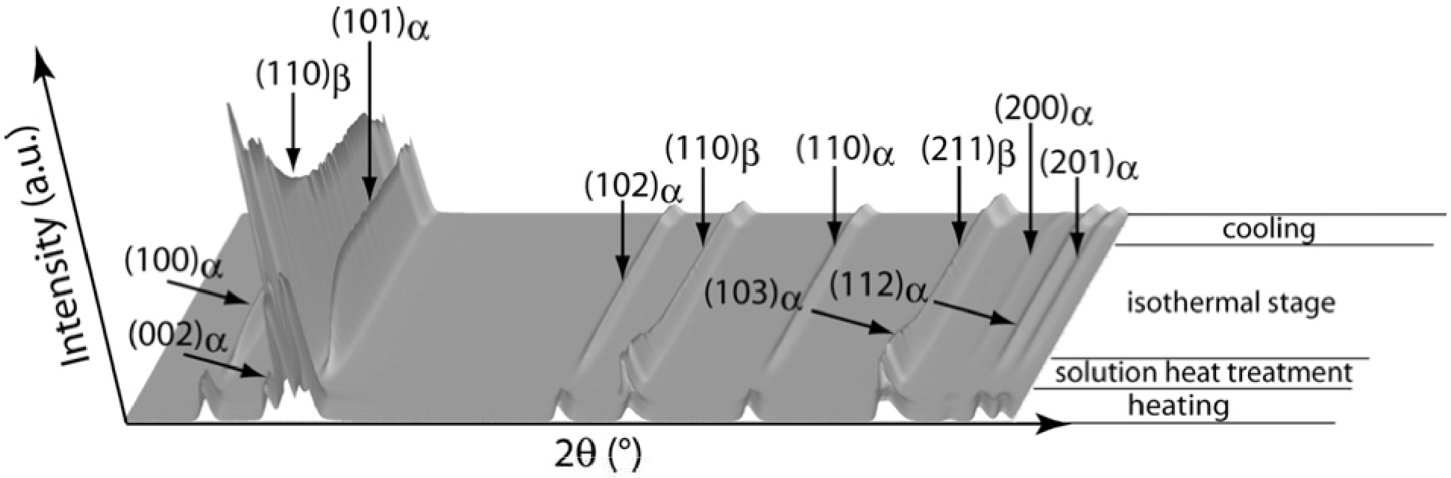
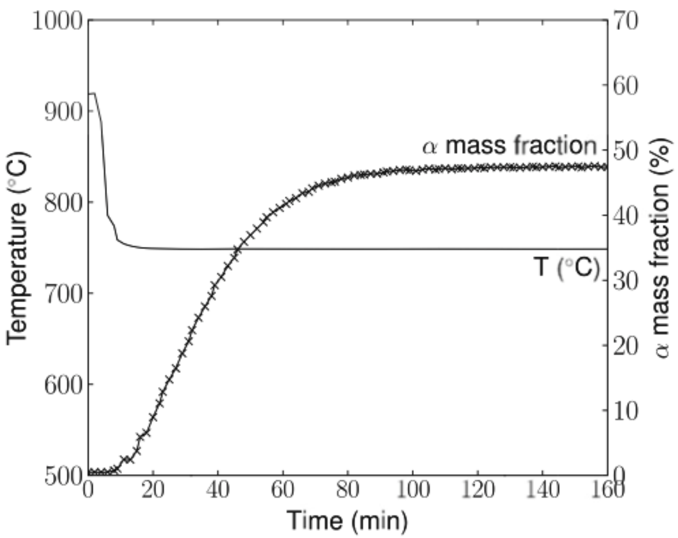
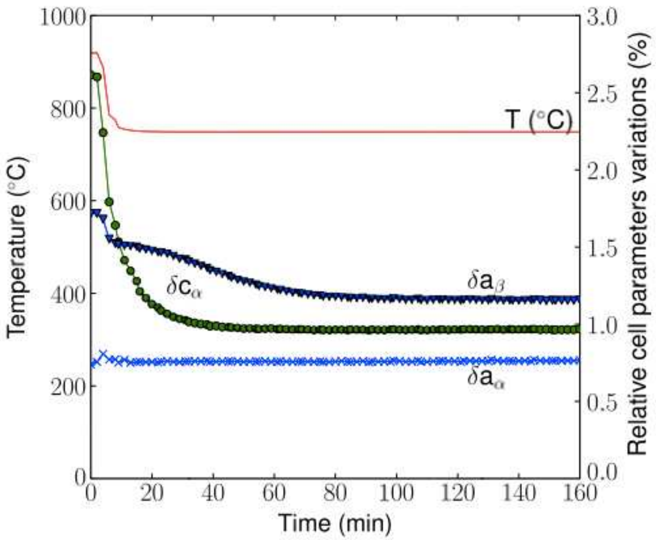


Fig. 1. Diffraction diagrams evolution over time for a β-metastable titanium alloy (Ti17).

Phase transformations in Ti alloys

G. Geandier et al.
C. R. Physique 13 (2012) 257–267



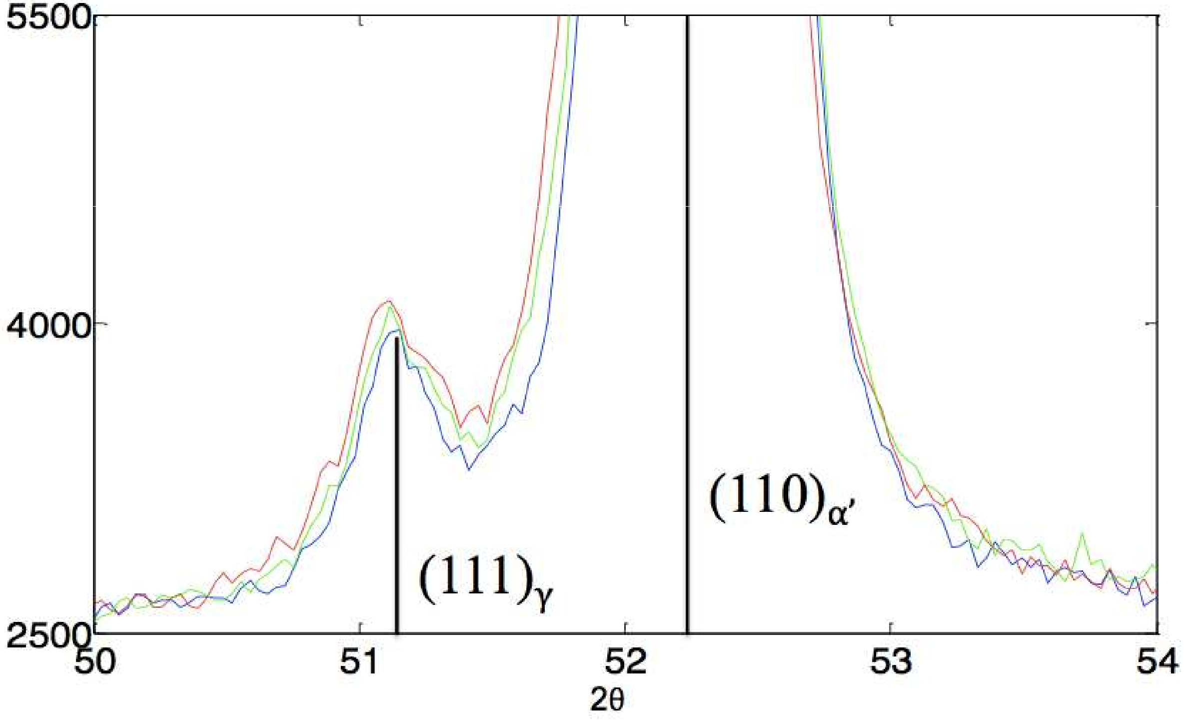


Microstructure at the microscale : grains and phases

Phase characterization – structure – X-ray diffraction

Precautions

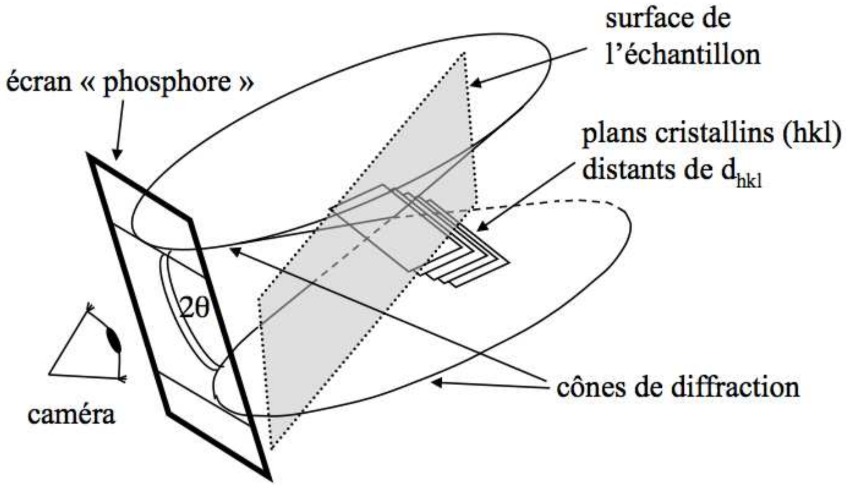
- Detection limit: synchrotron < %, lab apparatus about 1 %
- Texture effects → sample rotation, refinement
- Size effects (nanometer phases)
- Lab apparatus: penetration depth (Cu Kα for steels…), surface state

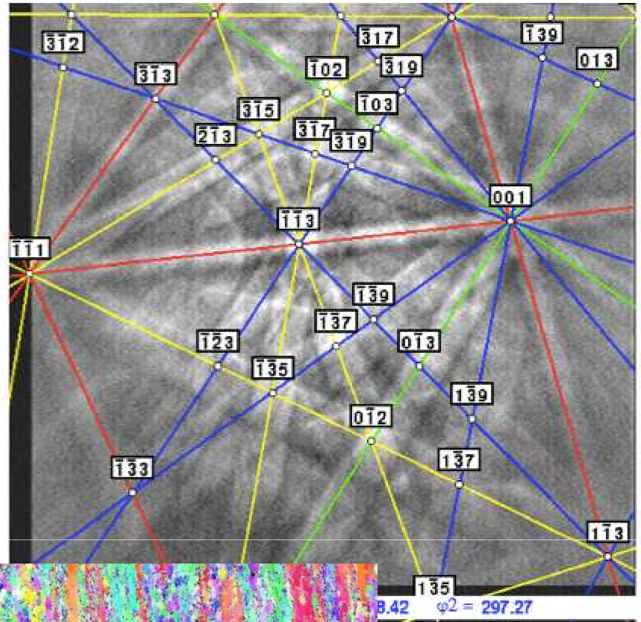


1.5% retained austenite in a martensitic steel, Co laboratory source

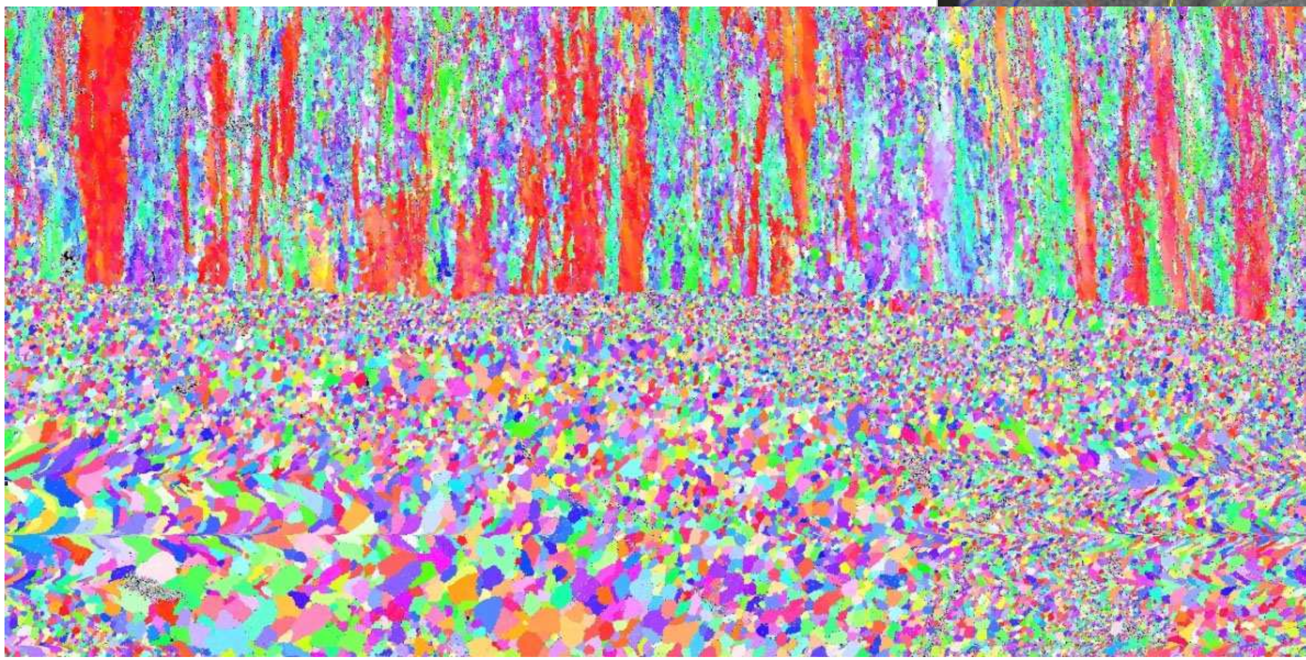
From : L. Couturier, PhD work, University of Grenoble

Grain characterization – texture – EBSD





From:
AF Gourgues
Lecture notes



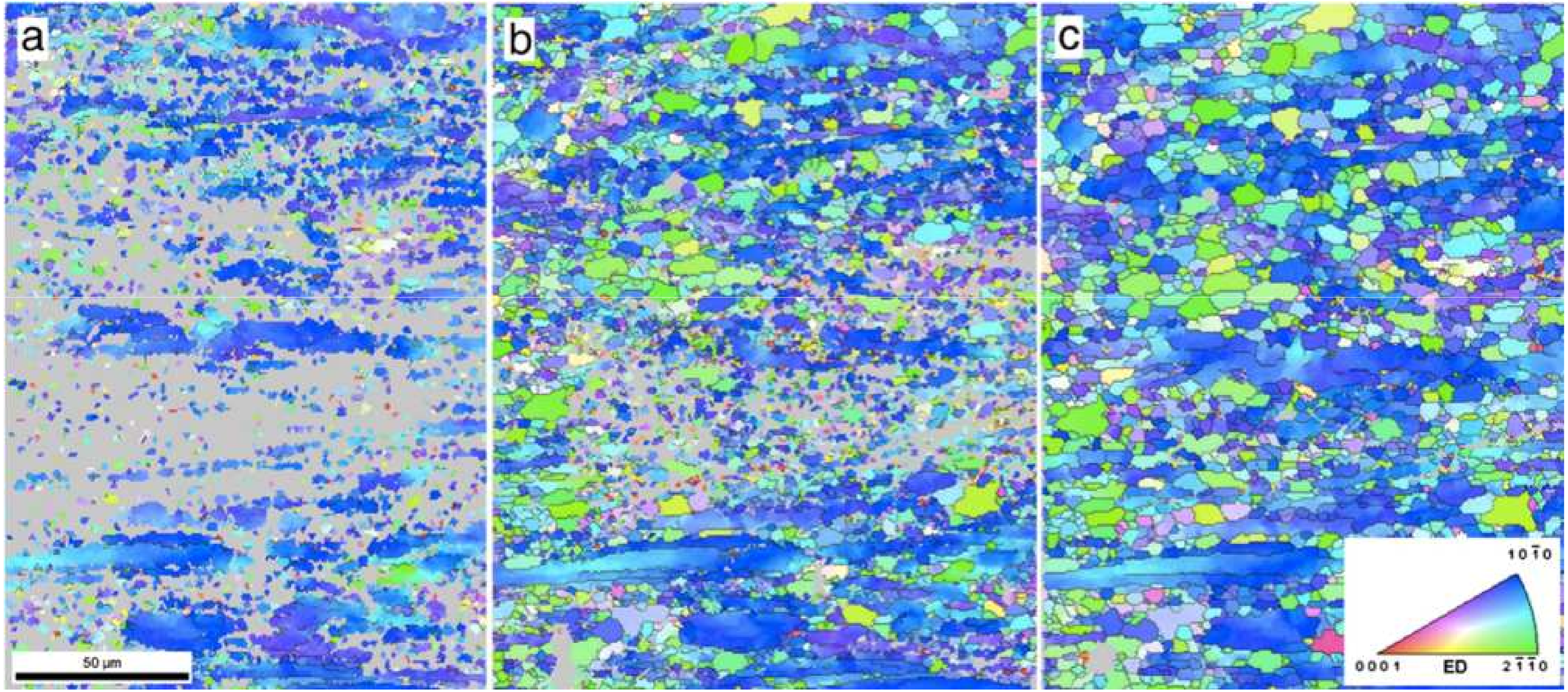
Precautions:

Surface state
Strain (dislocations)

A. Deschamps et al. / Materials Science and Engineering A 517 (2009) 361–368

Grain characterization – texture – EBSD

"in-situ" recrystallisation in the SEM

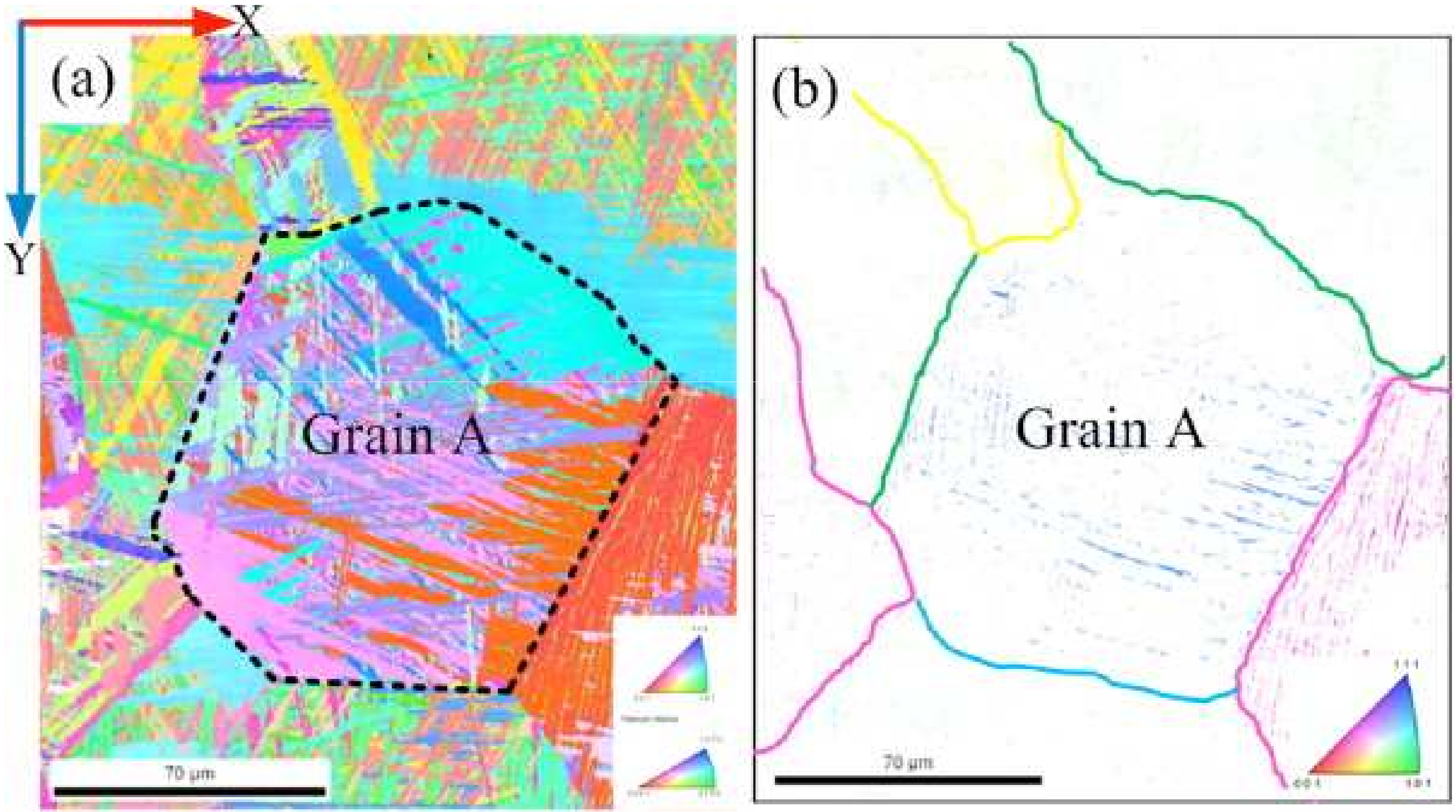


Zirconium alloy

From N. Bozzolo et al., MATERIALS CHARACTERIZATION 70 (2012) 28–32

Phase characterization – texture – EBSD

Discriminate between different crystallographic phases + study of orientation relationship



Ti alloy orientation of α+β phase

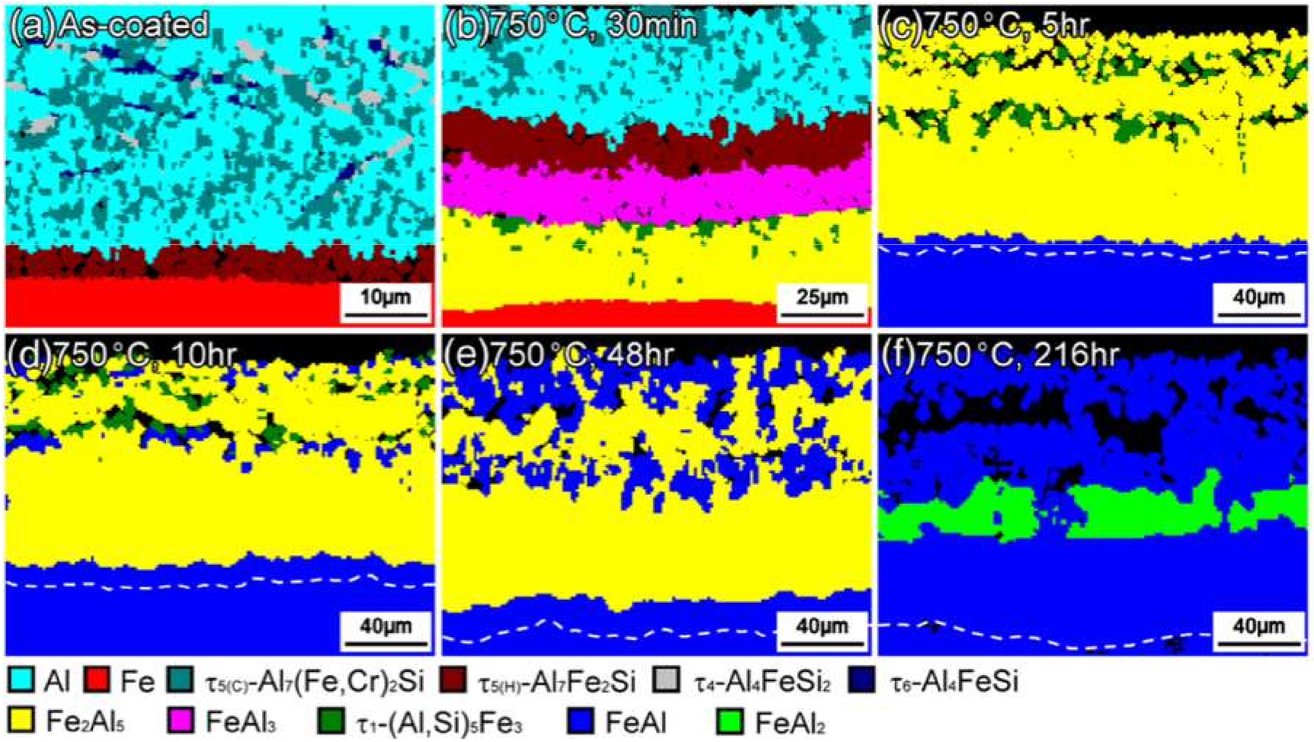
Residual β phase and sketch of former β GBs

From He et al., Materials Science and Engineering A 549 (2012) 20–29

Phase characterization – texture – EBSD

Multiple phase detection : unicity of Kikuchi line analysis?

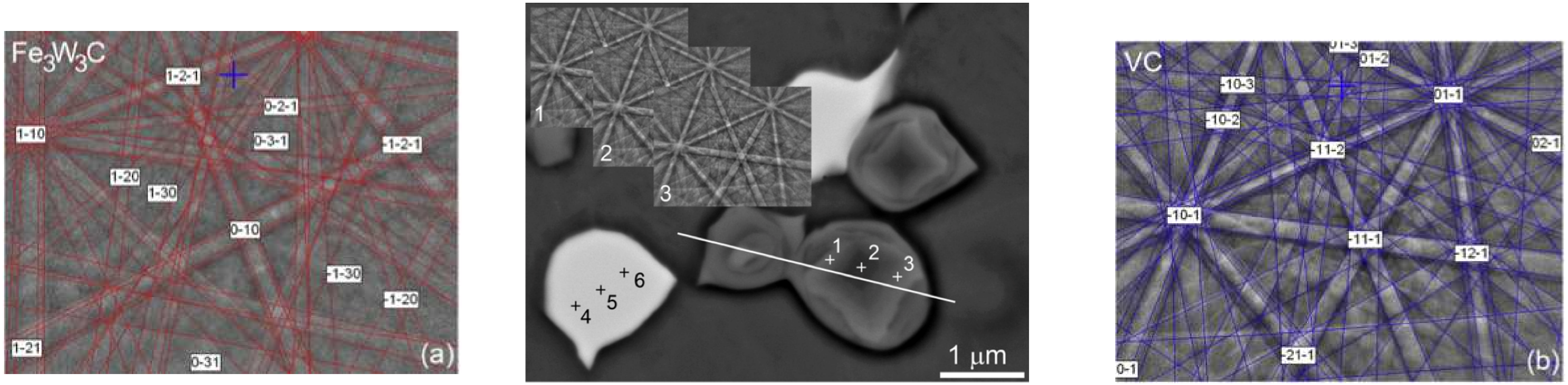
Coating of a Cr-containing steel with an Al-Si coating



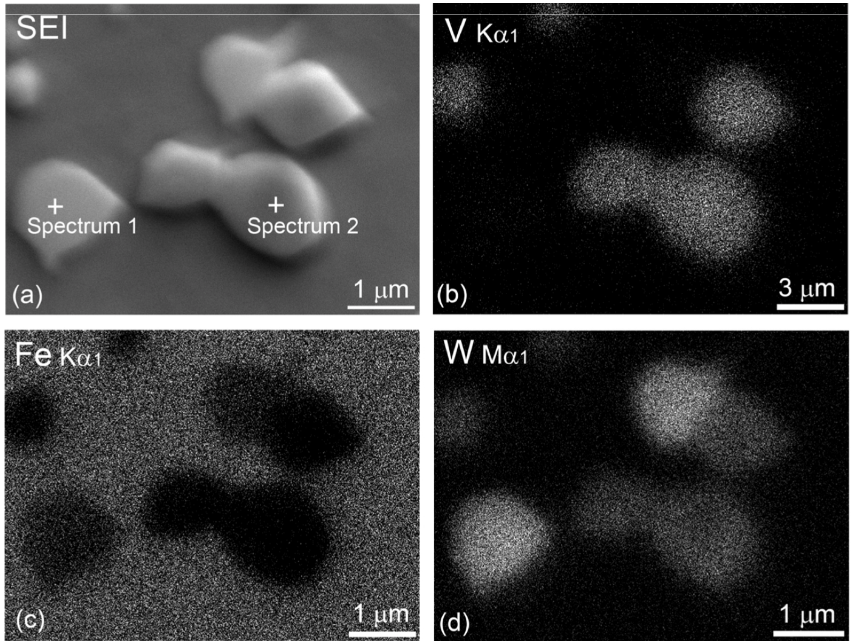
From : W-J Cheng et al. MATERIALS CHARACTERIZATION 64 (2012) 15–20

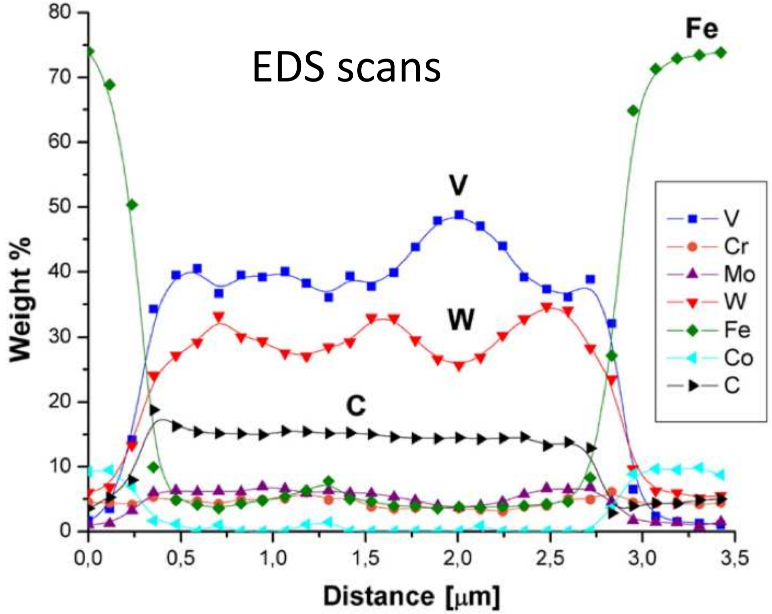
Microstructure at the microscale : grains and phases

Phase characterization – structural and chemical information



Carbide characterization in a high speed steel



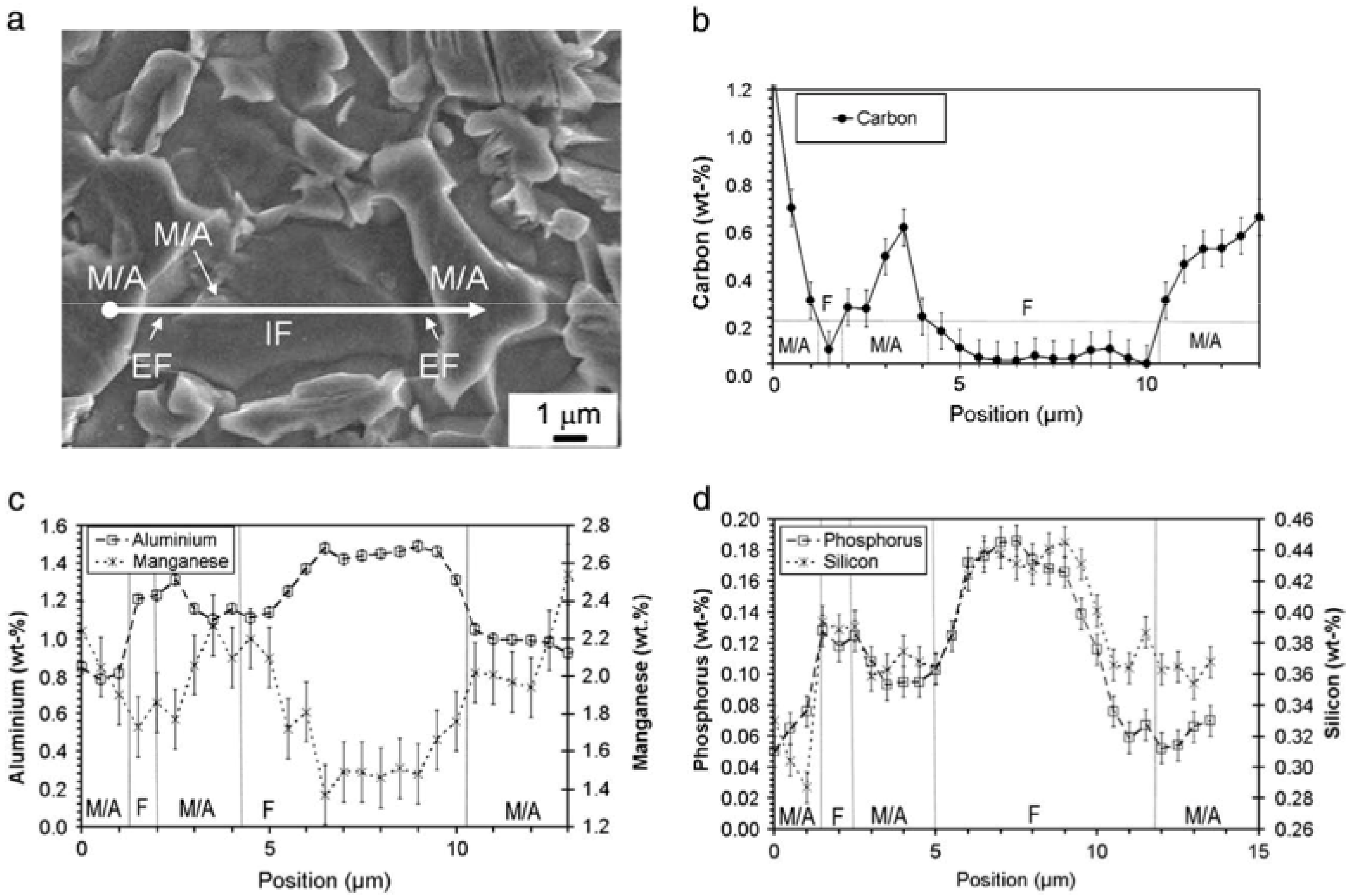


From M. Godec et al. MATERIALS CHARACTERIZATION 61 (2010) 452–458

Phase characterization – chemical information

Electron Probe Microanalysis (EPMA)

Higher sensitivity vs. EDS
More quantitative
Better suited for light elements (C, N)



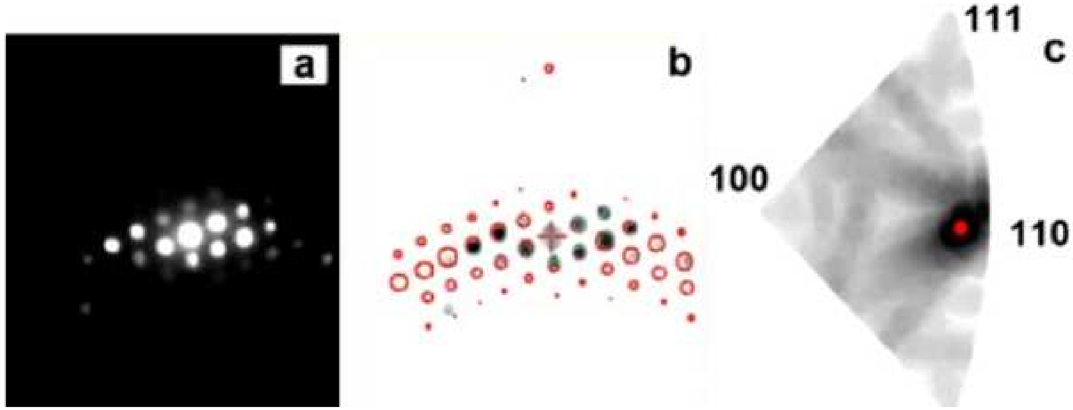
From : M.J. Santofimia et al. MATERIALS CHARACTERIZATION 61 (2010) 937–942

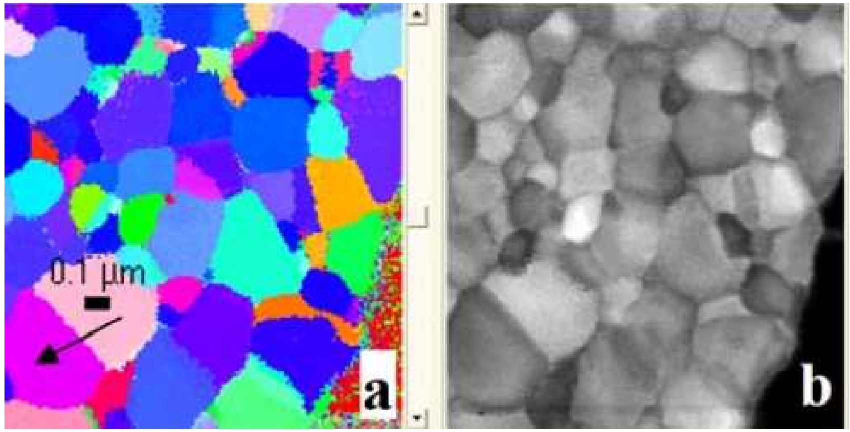
Sub-micro-scale

Grain and phase characterization – size and texture

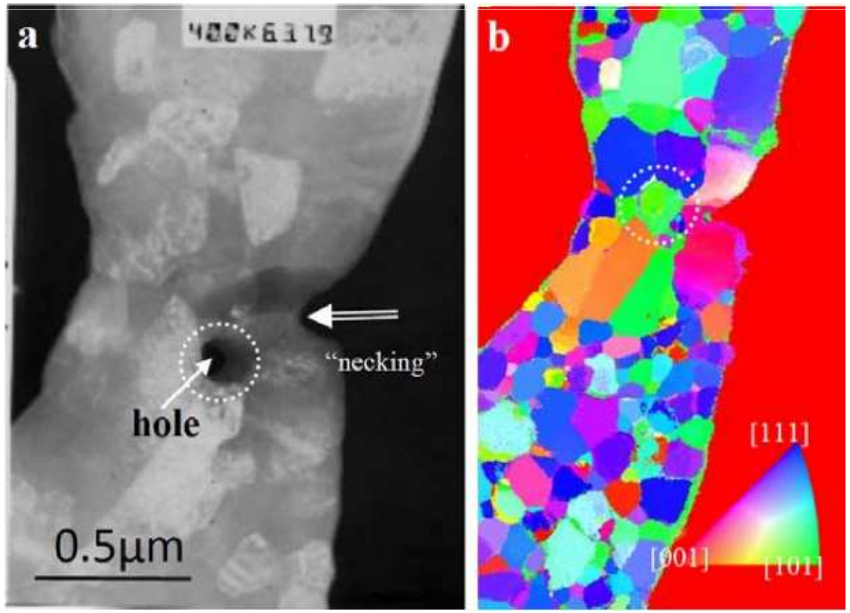
Grains at the nano to sub-micro scale,
Limits of the EBSD technique

Orientation mapping in the TEM:
ASTAR technique





Nanocrystalline Aluminium



From : P. Moeck et al. Cryst. Res. Technol. 46, No. 6 (2011)

Microstructure at the sub-microscale : coarse precipitates

Precipitate characterization – size and spatial distribution

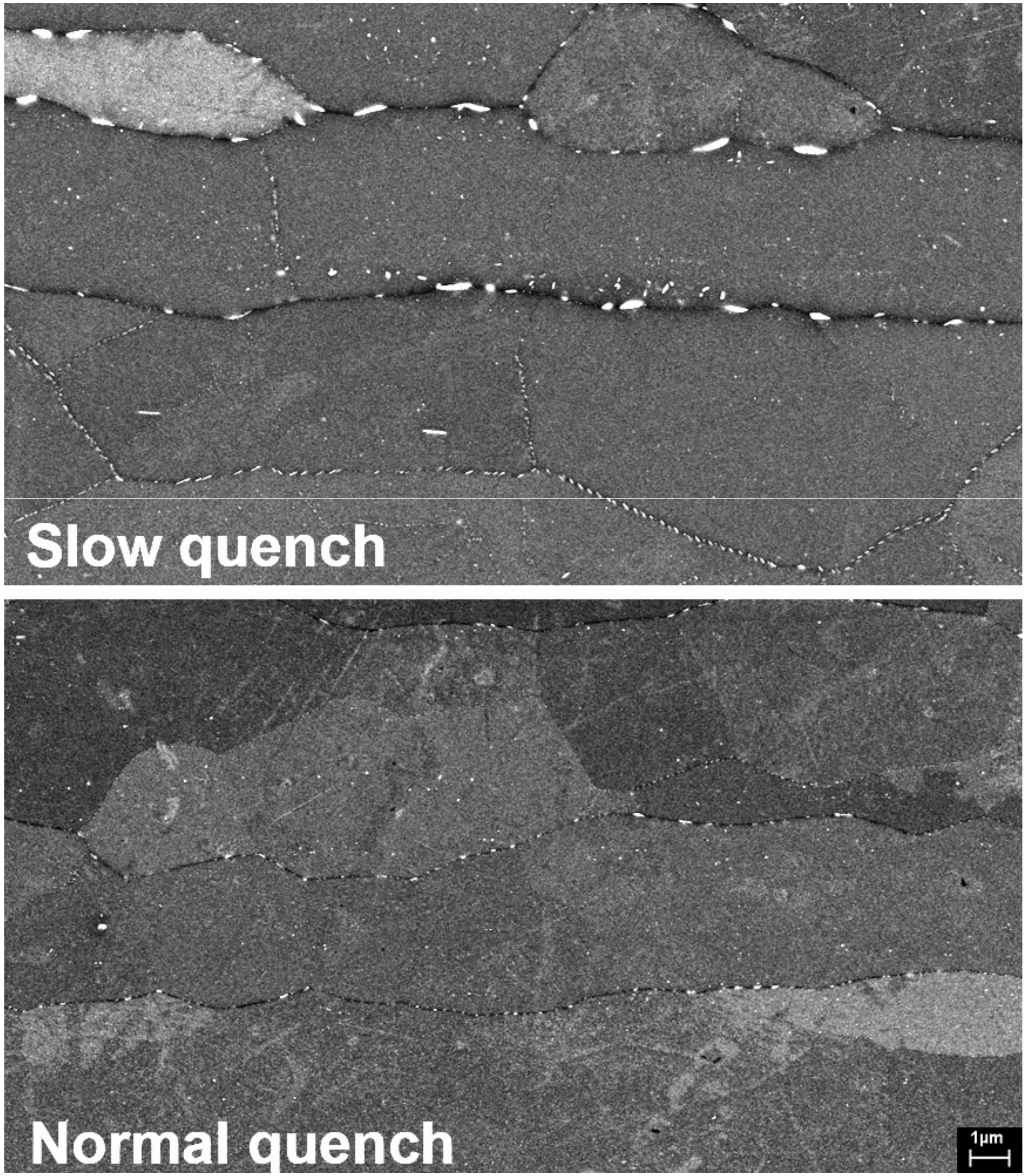
Phases at sub-micron scale :
Coarse precipitates

- Inherited from solidification
- Precipitate during high temperature processes (e.g. austenitization)
- Quench-induced

FEG-SEM standard tool for observation

If large enough volume fraction XRD suitable for structure & fraction

Else use TEM

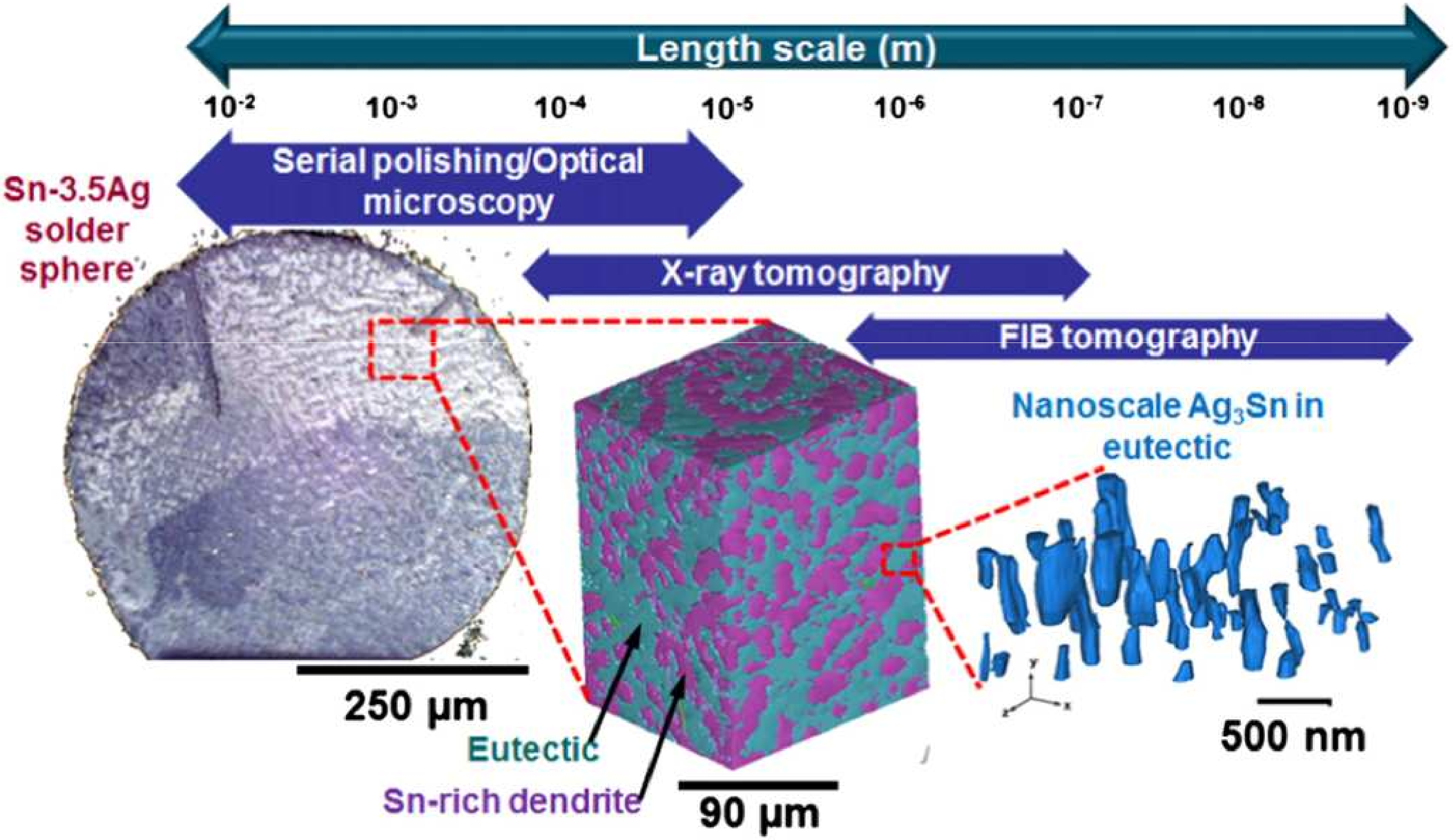


From: T. Marlaud et al.
Corrosion Science 53 (2011) 3139–3149

Microstructure at the sub-microscale : coarse precipitates

Precipitate characterization – size and spatial distribution

Many properties linked to these particles depend on 3D spatial distribution



From K.E. Yazzie et al. MATERIALS CHARACTERIZATION 70 (2012) 33–41

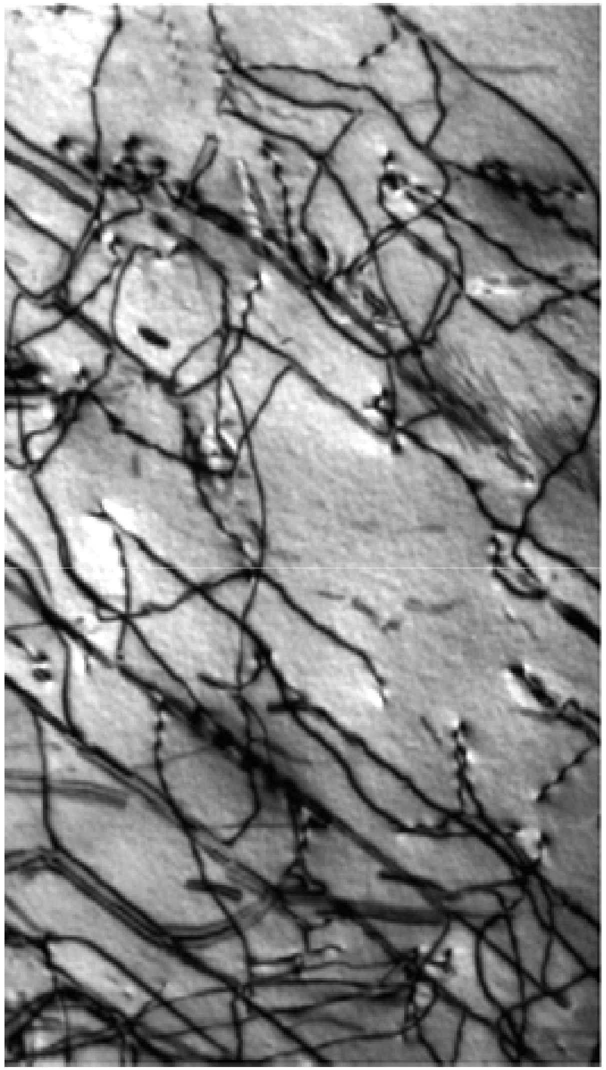
Microstructure at the sub-microscale : dislocations

Evolution during a series of processes

- Hot deformation (rolling, extrusion, forging, …)
- Cold deformation (rolling, shaping)
- Temperature changes (internal stresses – quench, welding)
- Constitutive laws for material performance
- Creep

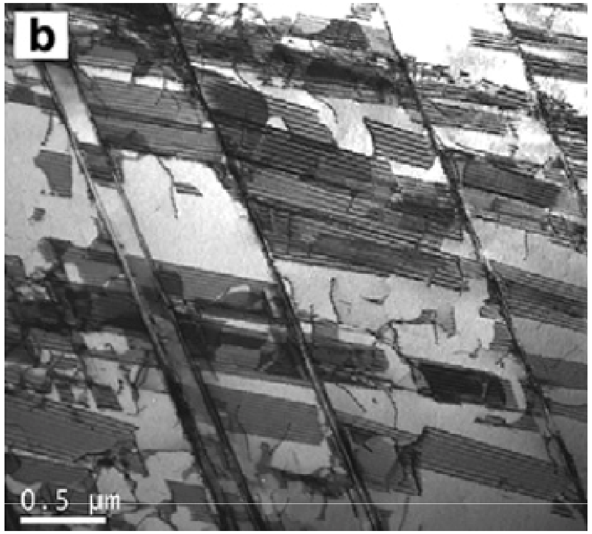
To be characterized

- Dislocation type : perfect, dissociated, super-dislocation
- Interaction with the microstructure
  - Other dislocations : recovery, storage, cross-slip, …
  - Grain boundaries : pile-up, transmission
  - Precipitates and other phases : GNDs, …
- Arrangement
  - Low energy dislocation structures
  - Competition with recrystallization



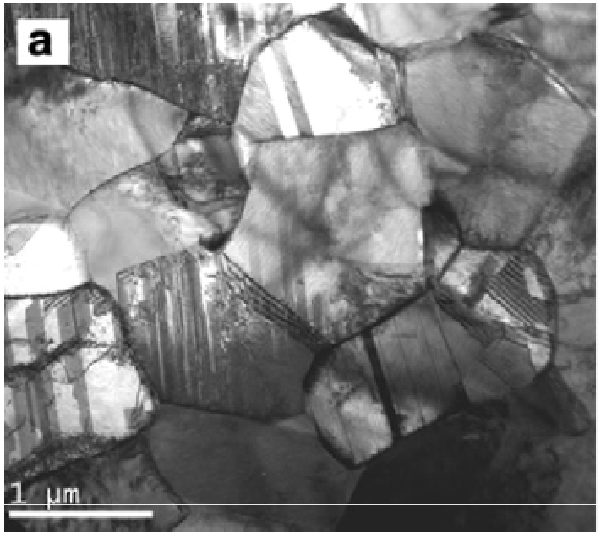
From: L. Tan et al. / Materials Science and Engineering A *528 (2011) 2755–2761*

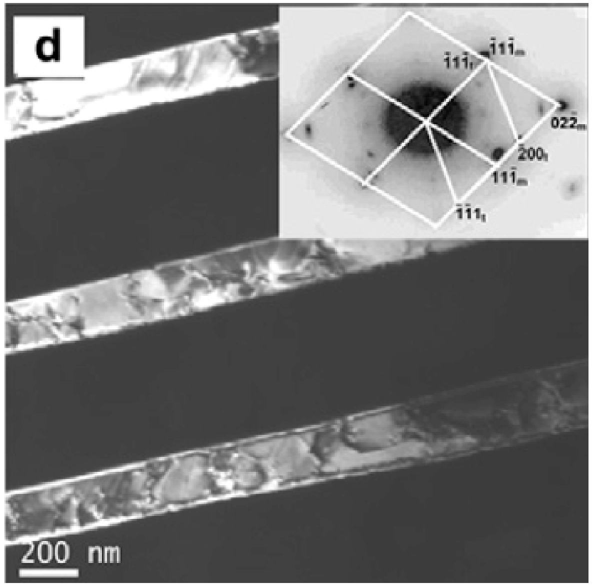
Dislocation structures : Transmission Electron Microscopy



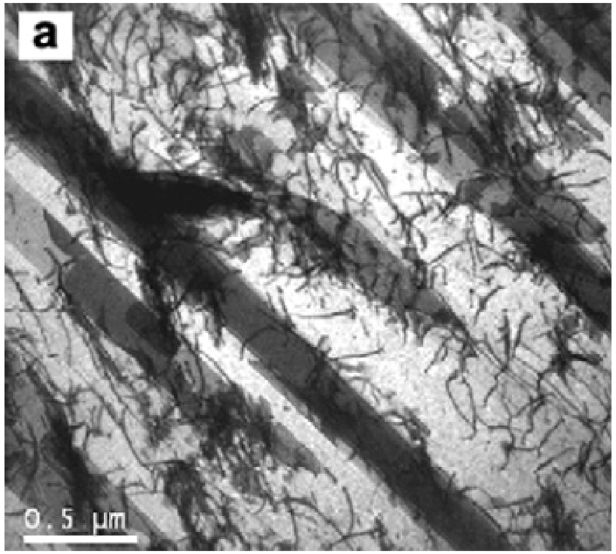
TWIP steels

- Dislocations
- Stacking faults
- Twins





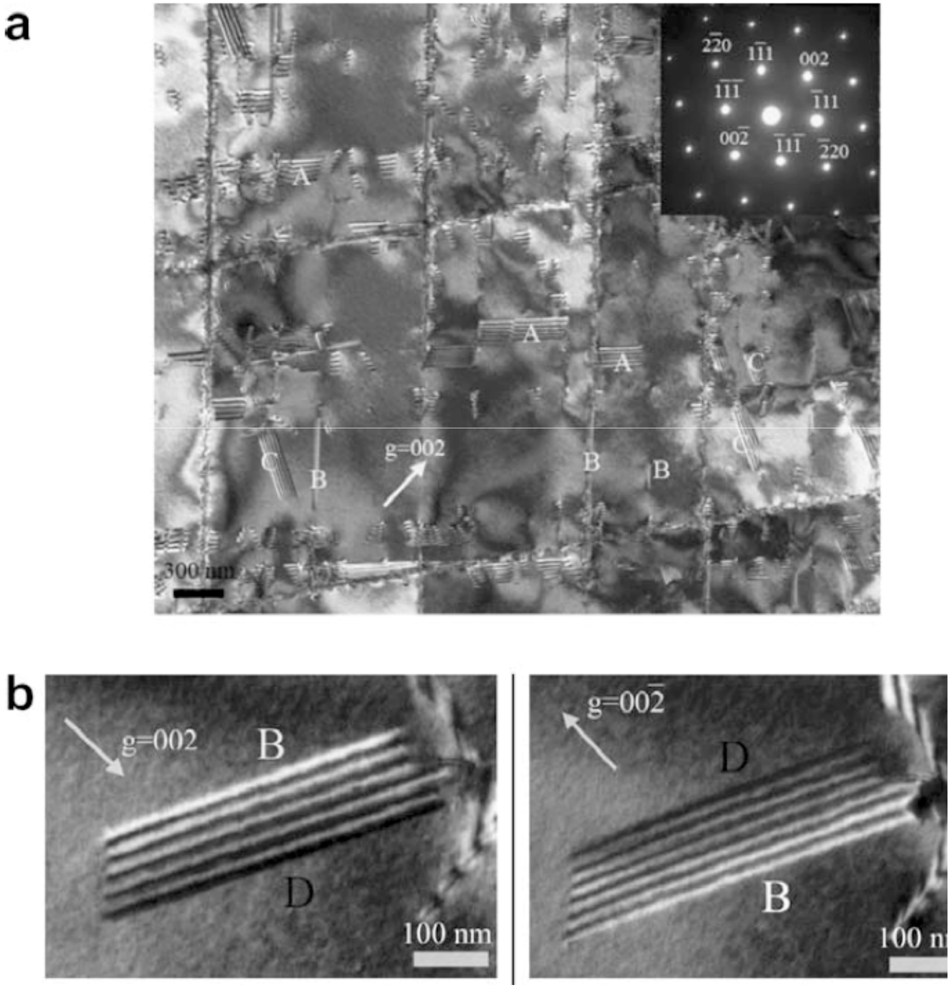
From L. P. Karjalainen et al. Scripta Materialia 66 (2012) 1034–1039

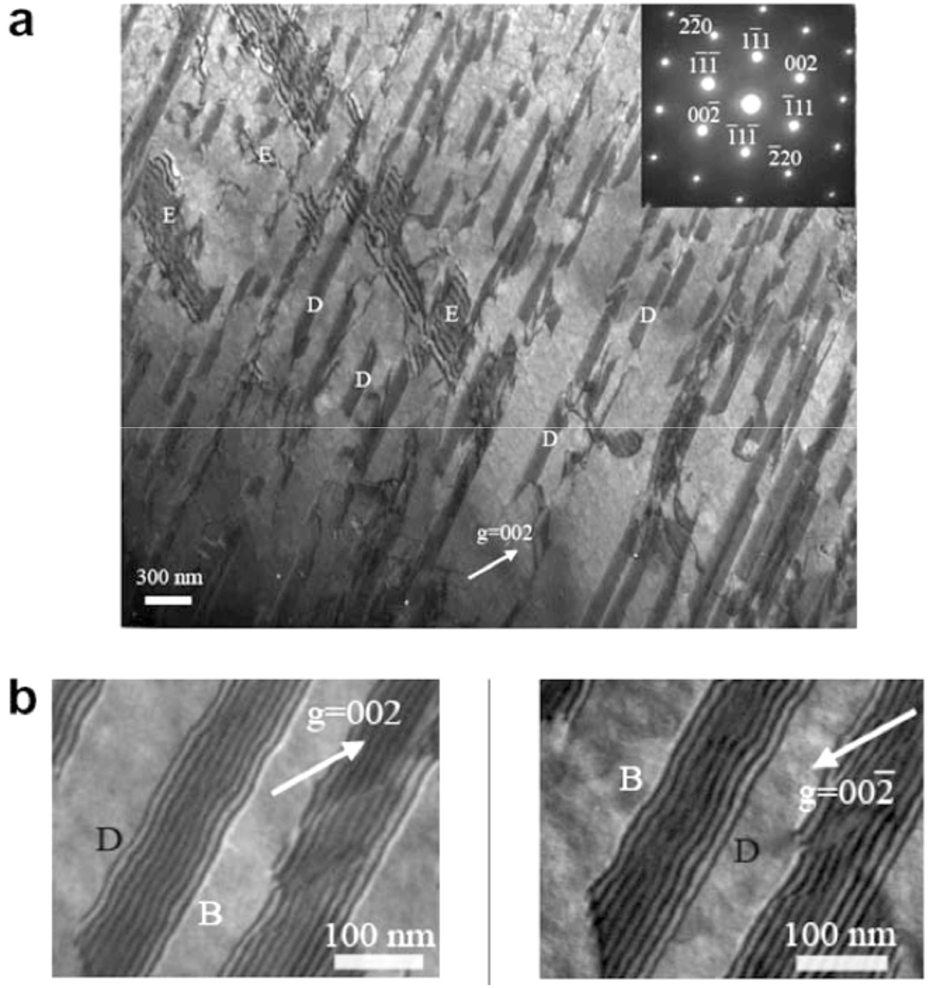


Microstructure at the sub-microscale : dislocations

Dislocation structures : Transmission Electron Microscopy

Determination of the type of stacking fault (intrinsic / extrinsic) in a TWIP steel





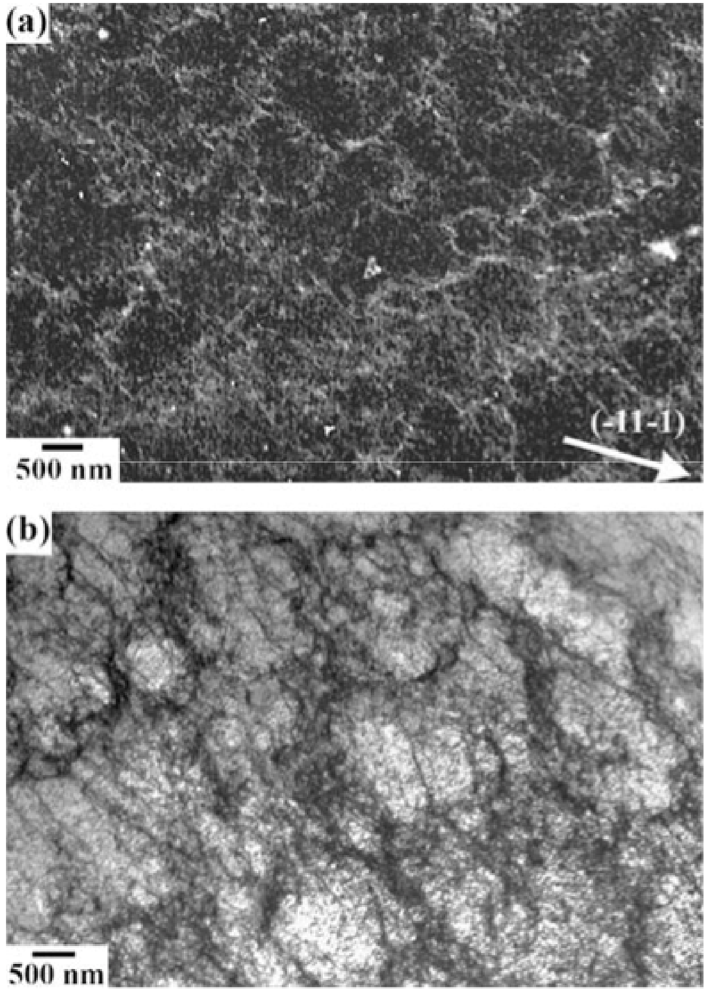
From H. Idrissi et al. / Scripta Materialia 60 (2009) 941–944

Microstructure at the sub-microscale : dislocations

Dislocation structures : other observations

SEM
Electron Channeling Contrast Imaging (ECCI)

Transmission Electron Microscopy



From I. Gutierrez-Urrutia et al. / Scripta Materialia 61 (2009) 737–740

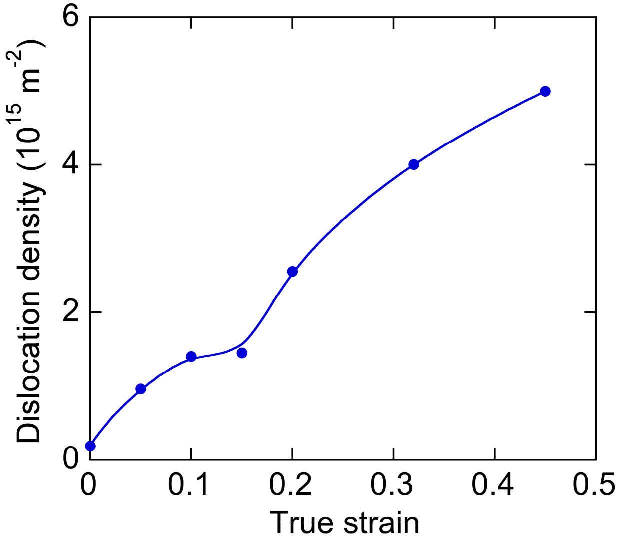
Evaluation of dislocation density

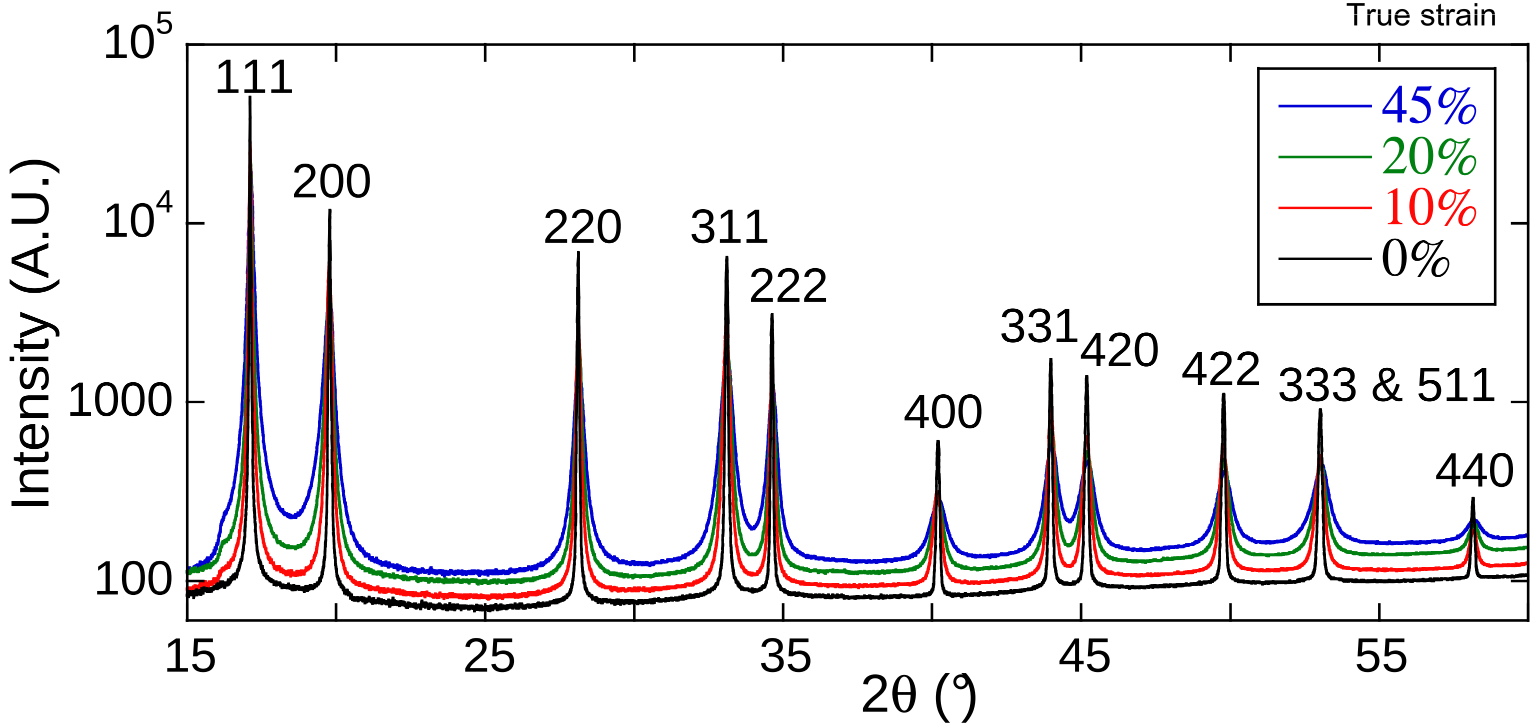
Dislocation density by XRD : quantitative… but tricky

TWIP steel
Synchrotron XRD

Dislocations + twins + internal stresses

From J.L. Collet, PhD thesis, INPG

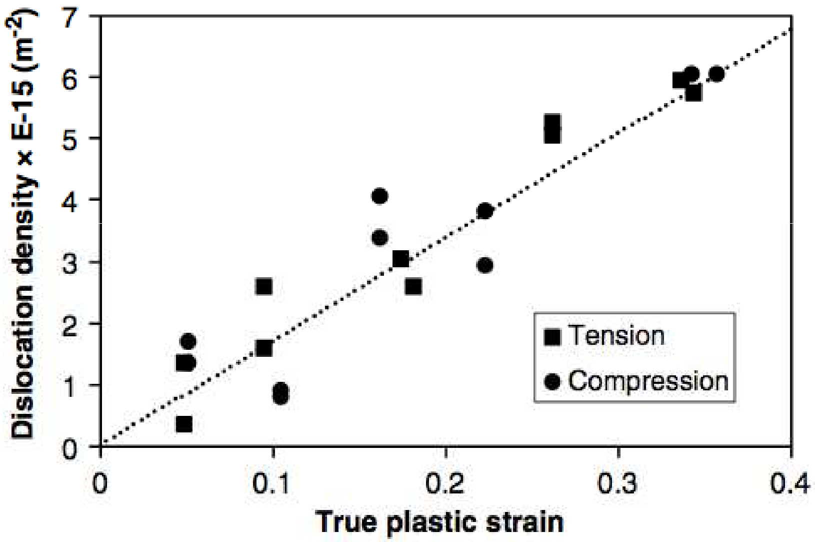




Microstructure at the sub-microscale : dislocations

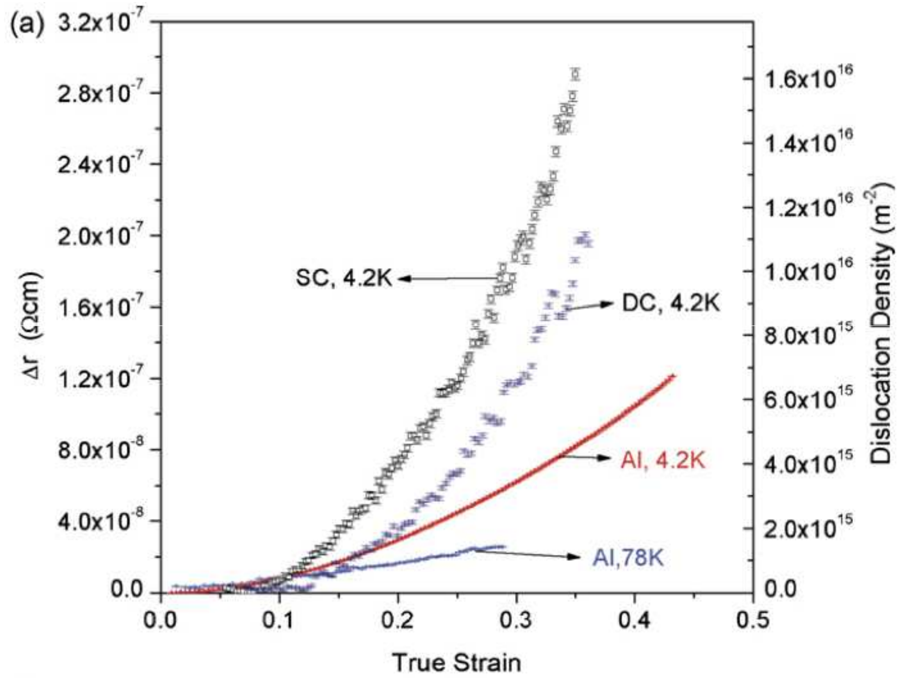
Evaluation of dislocation density

Dislocation density measurement by density
Suitable for highly deformed samples



From : B. Hutchinson, N. Ridley
Scripta Materialia 55 (2006) 299–302

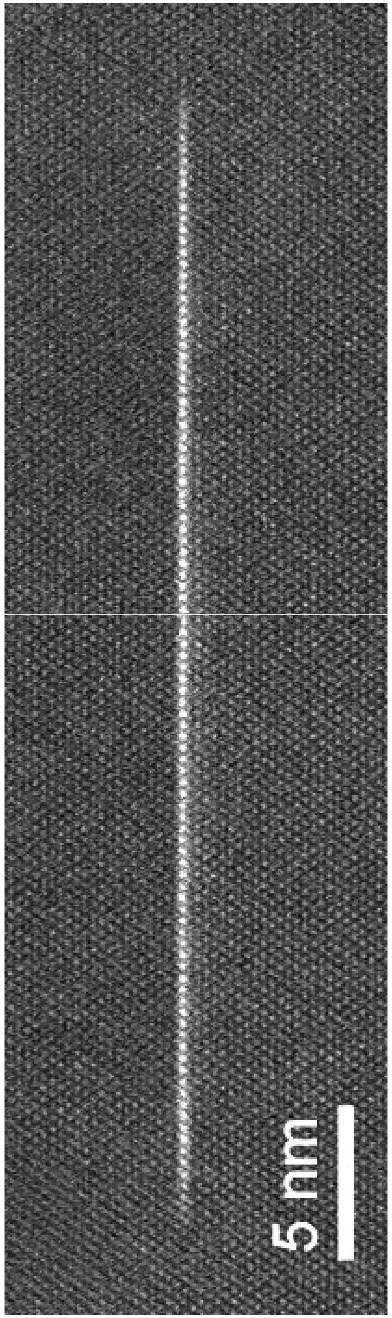
Dislocation density measurement by resistivity



From : D.-Y. Park, M. Niewczas
Materials Science and Engineering A 491 (2008) 88–102

Nano-scale

Microstructure at the nano-scale : precipitation



Evolution during a series of processes

Quench, natural ageing, multi-step ageing
Secondary processes (e.g. welding)
Evolution during use

To be understood

What forms: metastability, deviations from equilibrium
Kinetics : from nucleation to coarsening
Interaction with structural defects : GBs, dislocations, vacancies
Interaction with plasticity
Non-isothermal thermal paths

To be characterized

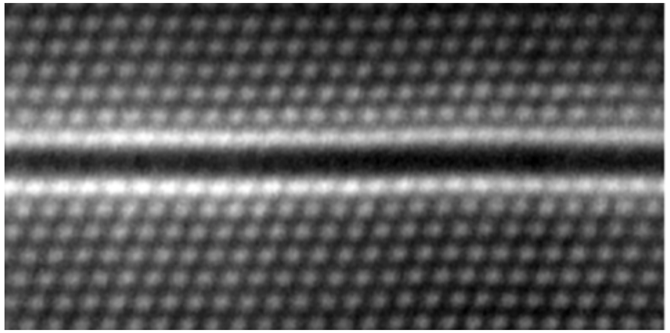
Structure
Interface with the matrix
Chemistry (deviation to stoichiometry)
Size, shape
Spatial distribution

Structure, interface with the matrix : TEM

High resolution (HREM, CS-corrected)

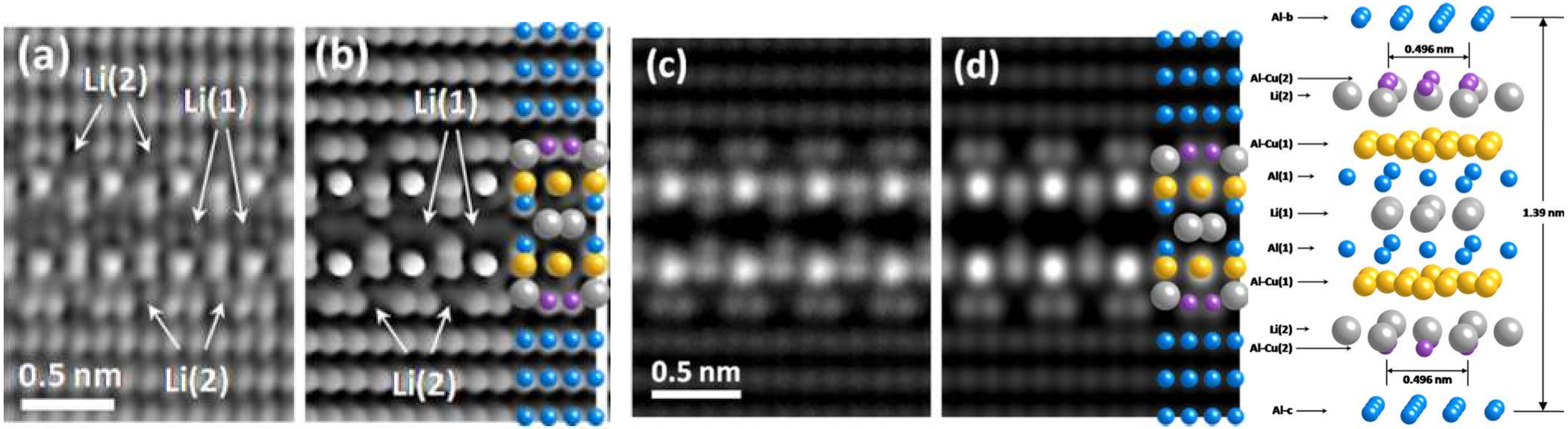
Atomic resolution HAADF-STEM (High Angle Annular Dark Field Scanning Transmission Electron Microscopy) (Z-contrast)

Diffraction



From : P. Donnadieu et al. Acta Materialia 59 (2011) 462–472

T_1 – Al_2CuLi precipitates in Al

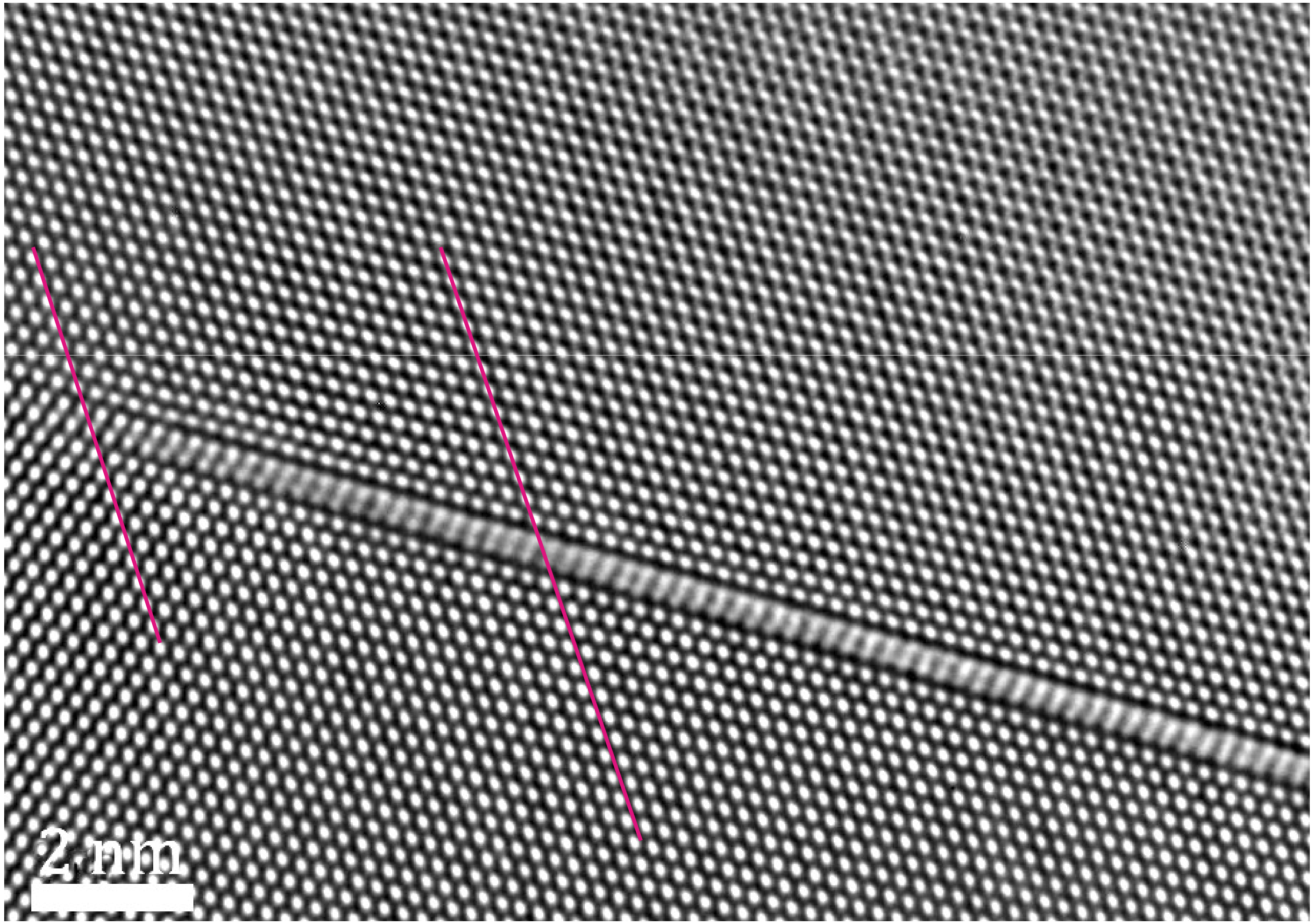


From : C. Dwyer et al. Appl. Phys. Lett. 98, 201909 (2011)

Microstructure at the nano-scale : precipitation

Structure, interface with the matrix : TEM

T_1 – Al_2CuLi precipitates in Al : presence of a stacking fault at the interface

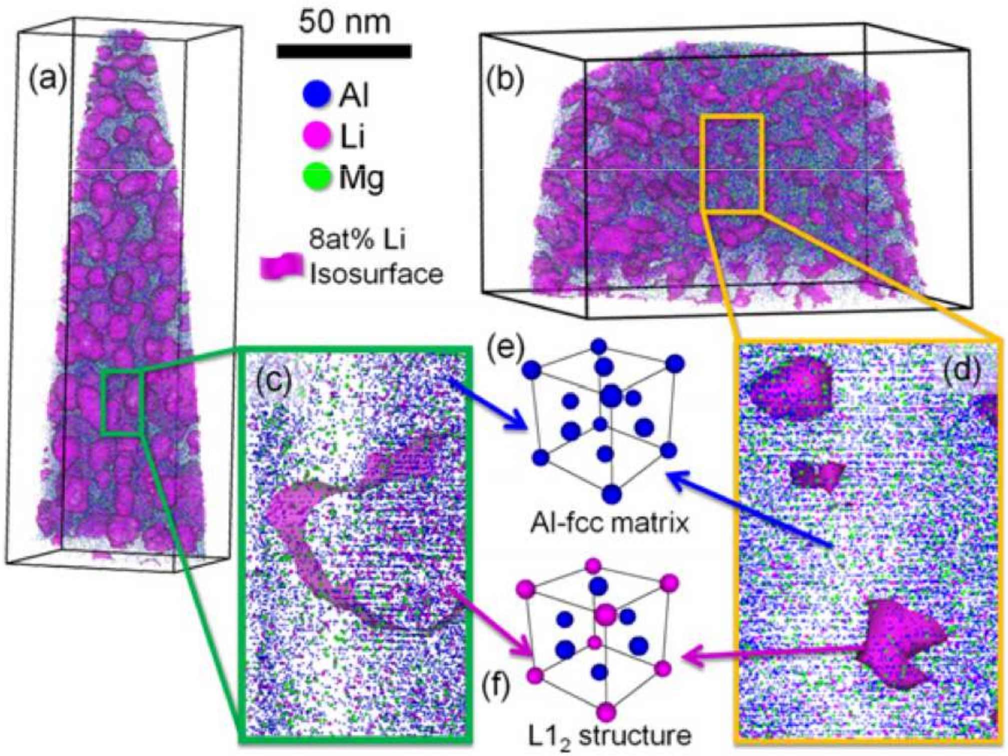


From: J. Douin, CEMES, Toulouse, France

Microstructure at the nano-scale : precipitation

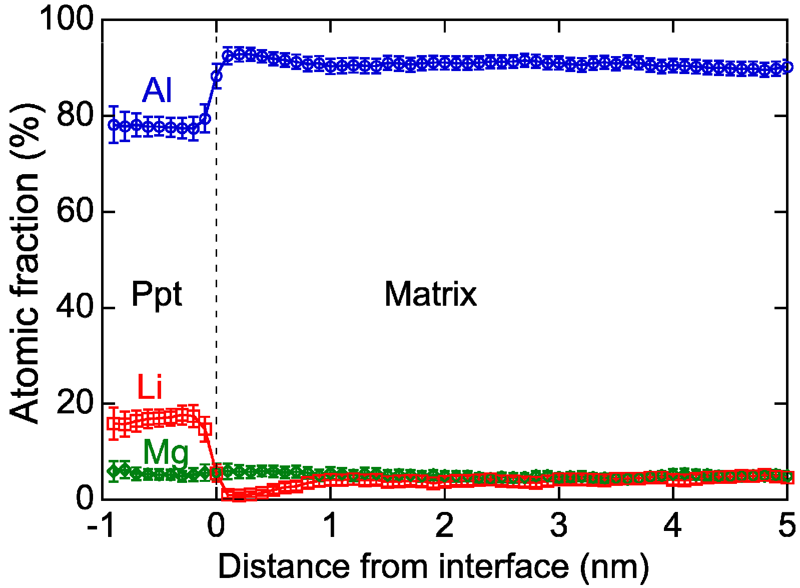
Chemistry : Atom Probe Tomography

Chemistry of precipitates
Chemistry of residual matrix
Statistical analysis of the matrix → clustering
Segregation



From: B. Gault et al. / Scripta Materialia 66 (2012) 903–906

δ' - $Al_3(Li,Mg)$ in Al

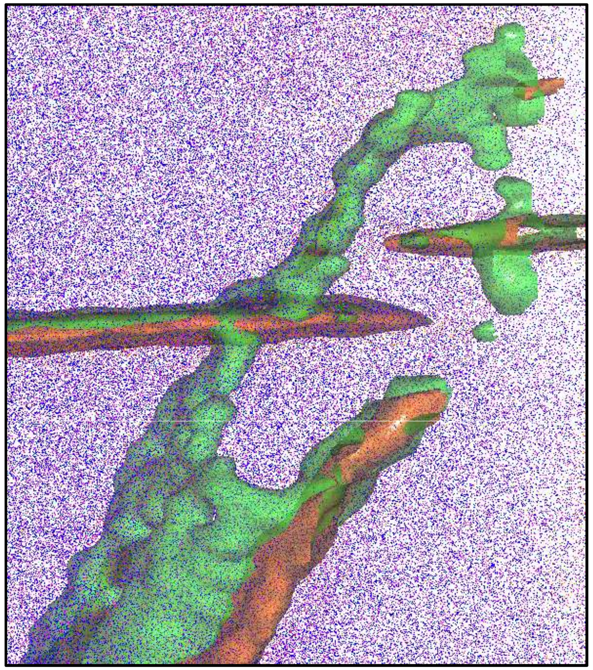


From: A. Deschamps et al.
Acta Materialia 60 (2012) 1917–1928

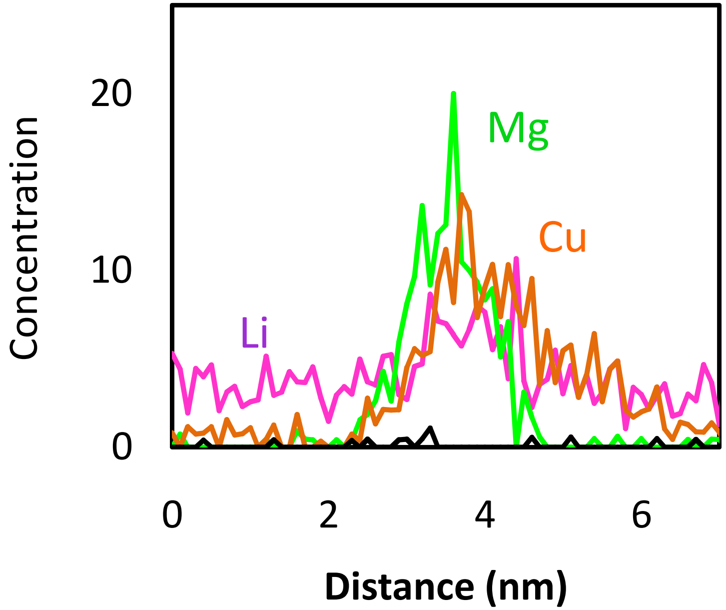
Microstructure at the nano-scale : precipitation

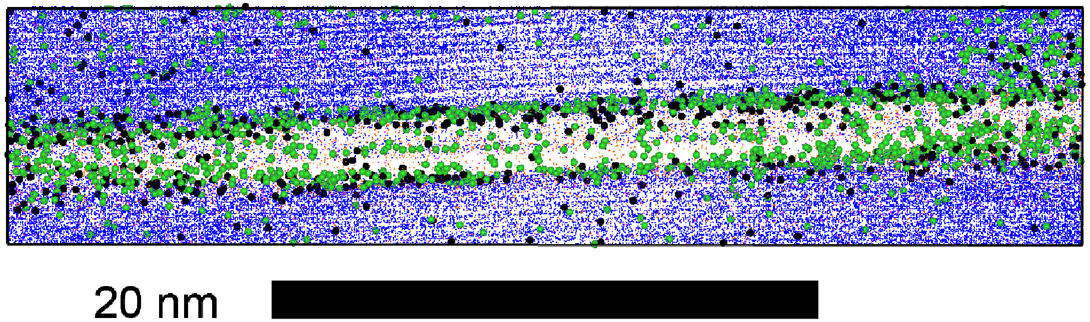
Chemistry : Atom Probe Tomography

Local chemistry of different phases



Concentration Profile S phase



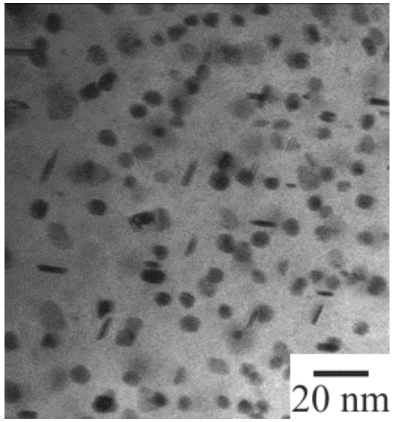


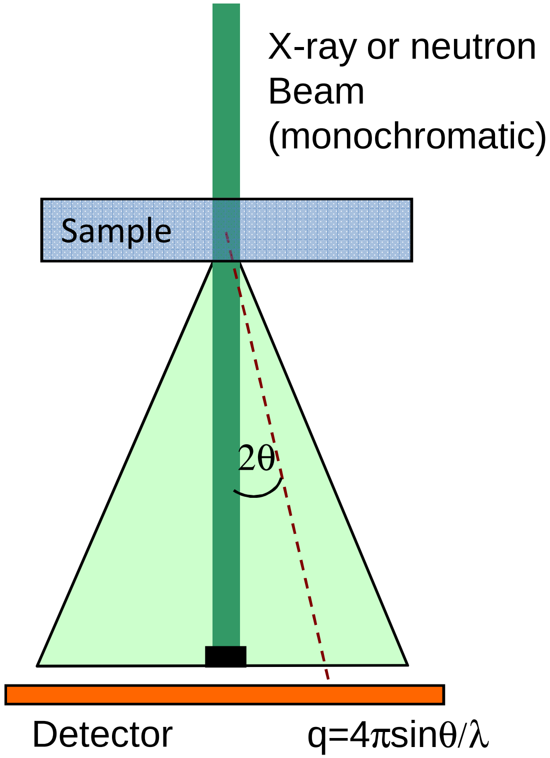
Segregation of **Mg** and **Ag** at the T_1 precipitate interface

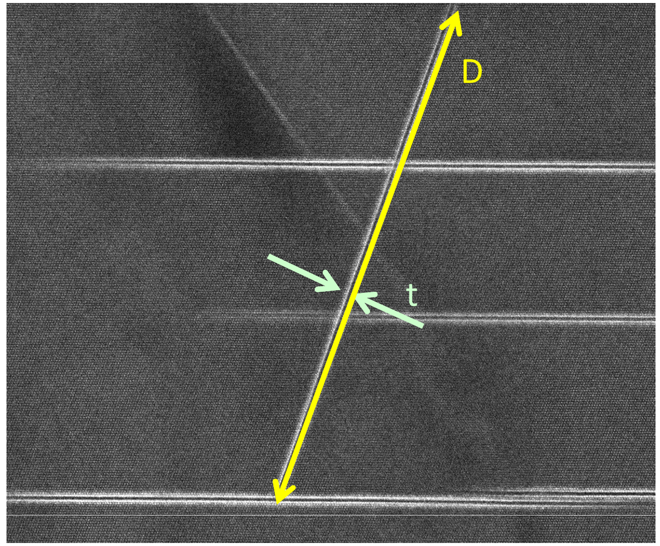
From: V. Araullo-Peters et al. , Univ. of Sydney, Australia

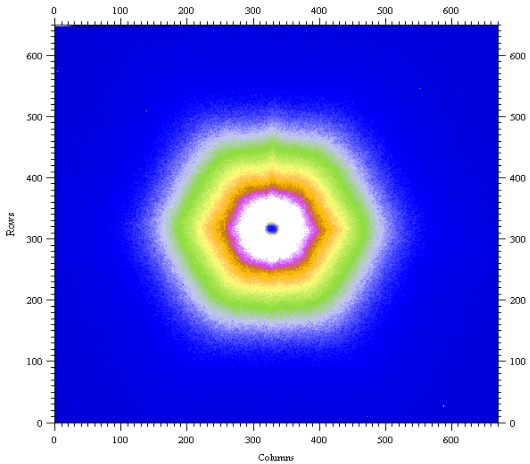
Microstructure at the nano-scale : precipitation

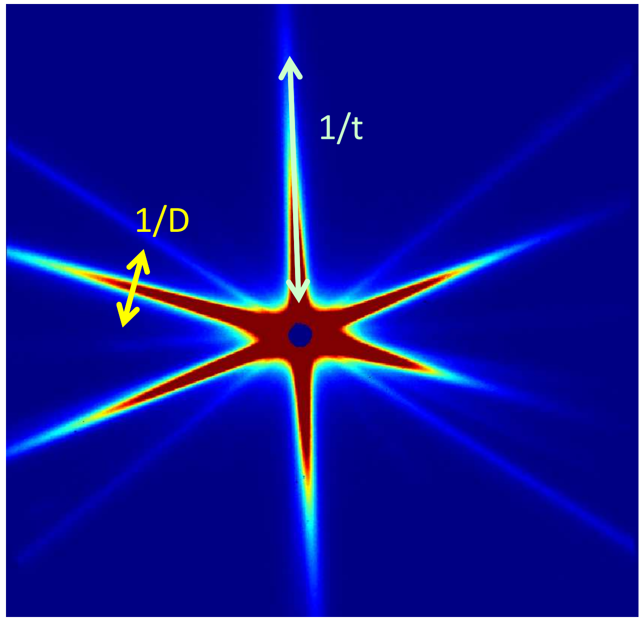
Size, shape : Small-Angle Scattering (SAXS, SANS)







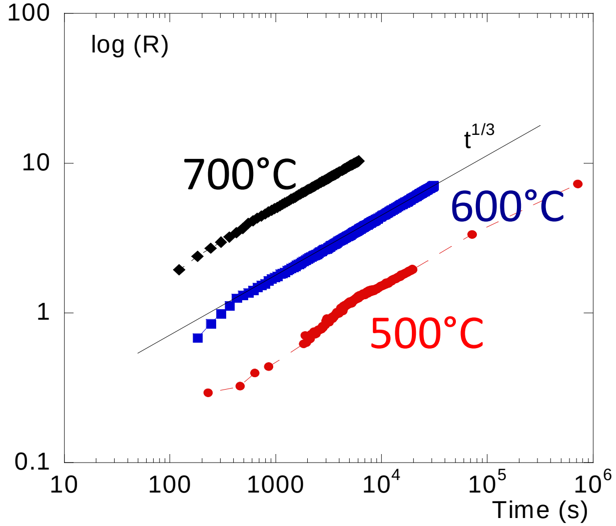


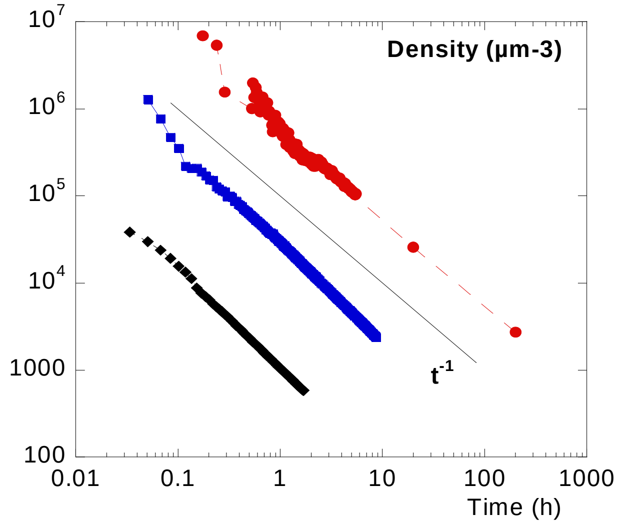


Microstructure at the nano-scale : precipitation

Size, shape : Small-Angle Scattering (SAXS, SANS)

Fe-Cu : spherical precipitates
In-situ evaluation of R, f_v, N

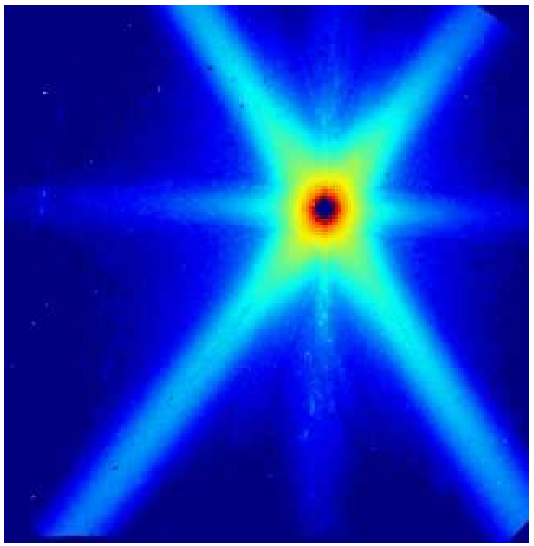


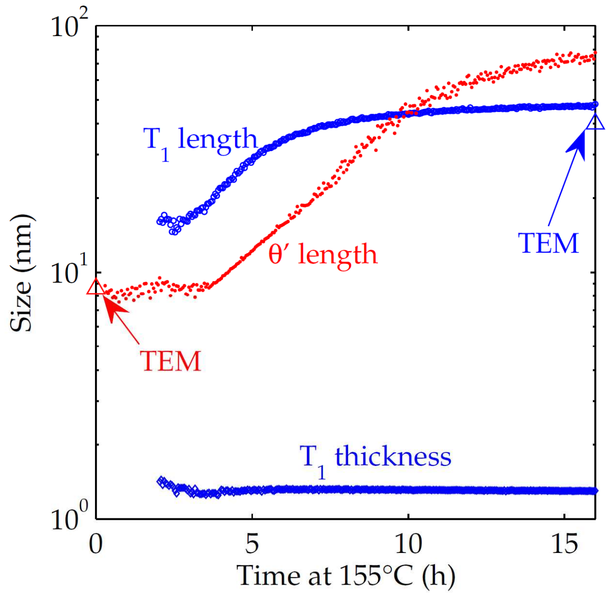


From: A. Deschamps et al. Vol. 85, 2005, 3091–3112

In-situ measurements
Temperature, strain

Al-Cu-Li : competition between θ’ and T_1



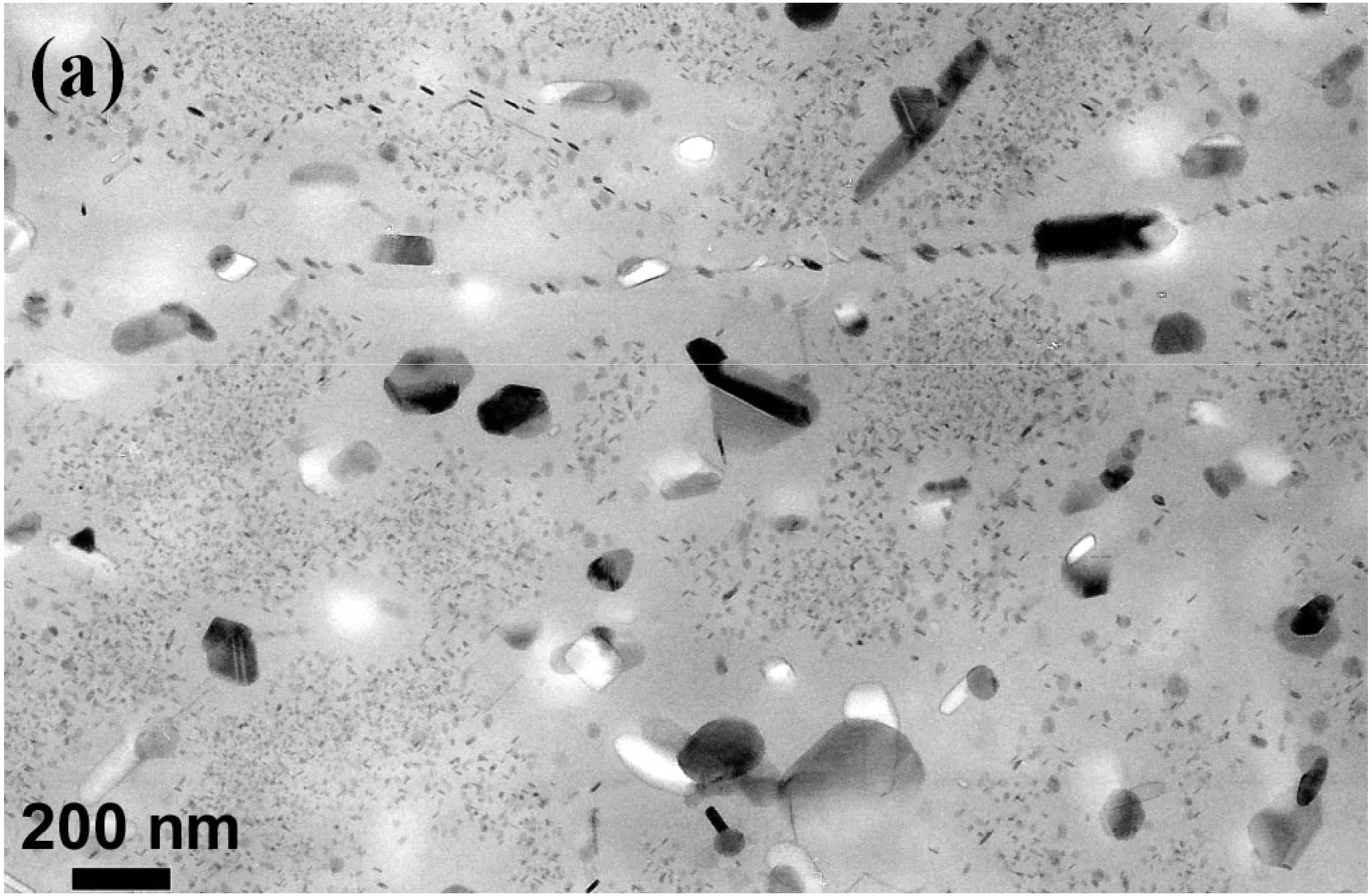


From: F. De Geuser et al. J. Appl. Cryst. doi:10.1107/S0021889812039891

Microstructure at the nano-scale : precipitation

Spatial distribution : conventional TEM

Homogeneous vs. heterogeneous (quench-induced) precipitation in an Al-Zn-Mg alloy



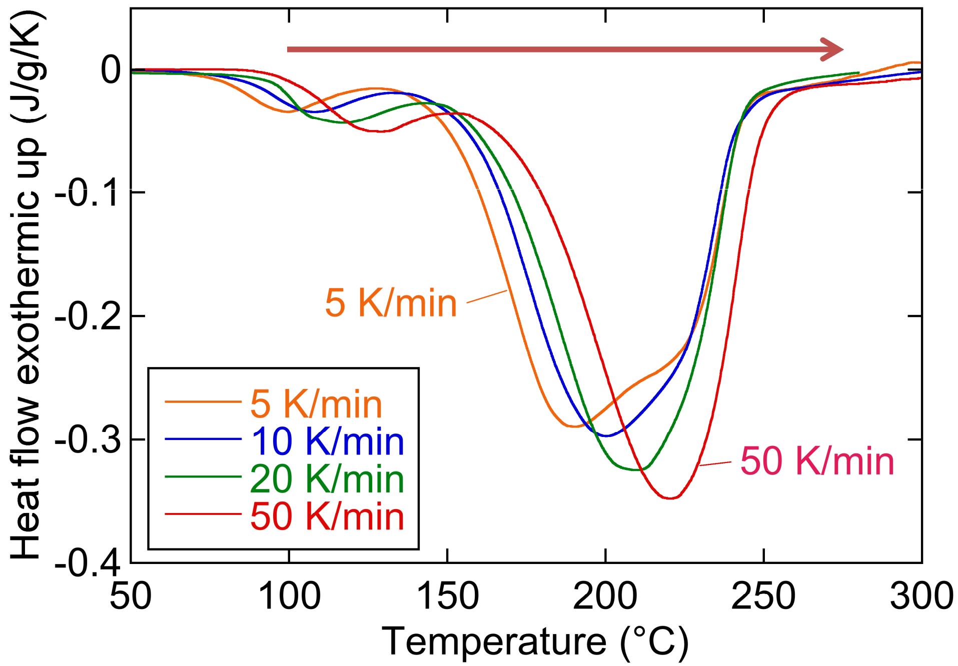
From : A. Deschamps et al. Materials Science and Engineering A 501 (2009) 133–139

Microstructure at the nano-scale : precipitation

Volume fraction, thermodynamics : DSC

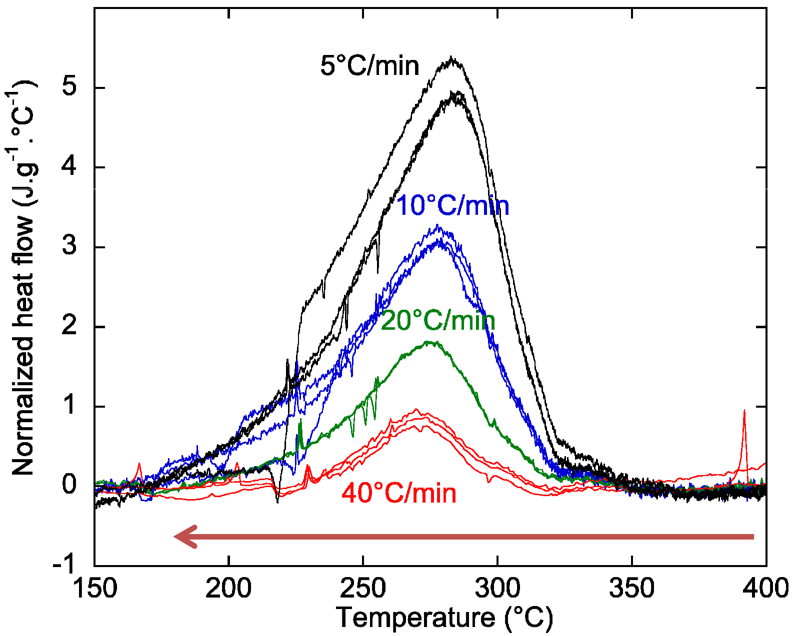
Differential calorimetry: easy quantification ΔH, f_v, solvus T, …
But interpretation can be tricky in complex systems (many phases)

Dissolution kinetics of δ′ in Al-Mg-Li



From : A. Deschamps et al.
Acta Materialia 60 (2012) 1917–1928

Precipitation kinetics during cooling
in Al-Zn-Mg



From : A. Deschamps et al.
Materials Science and Engineering A 501 (2009) 133–139

How to be precise and realistic

Kinetic process

Through process modeling
Thermomechanical effects
Non isothermal treatments
Complex chemistry

Multiple information needed

Structure
Size, fractions
Chemistry
Deformations / stresses
Spatial distributions (2D, 3D)

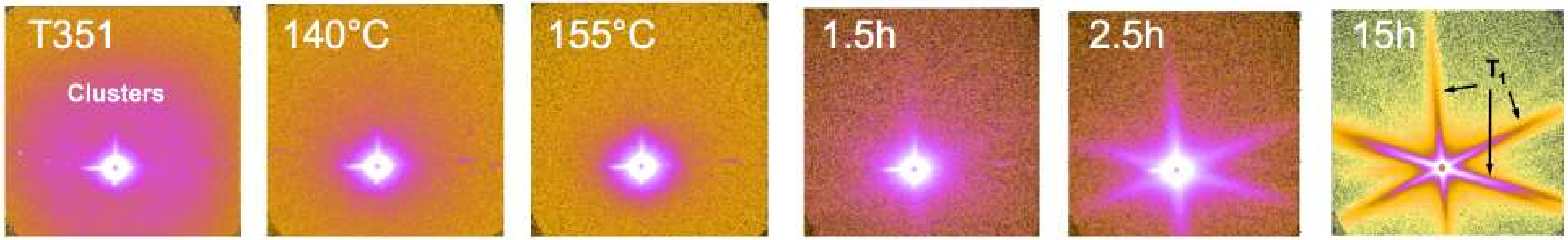
In-situ techniques (towards in operando) → non-local

Coupling between different techniques

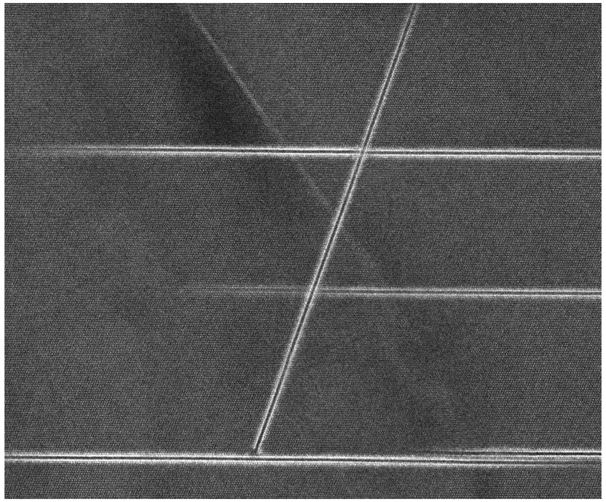
- Local & non-local
- Average (statistics, in-situ), Local (spatial resolution, local information)

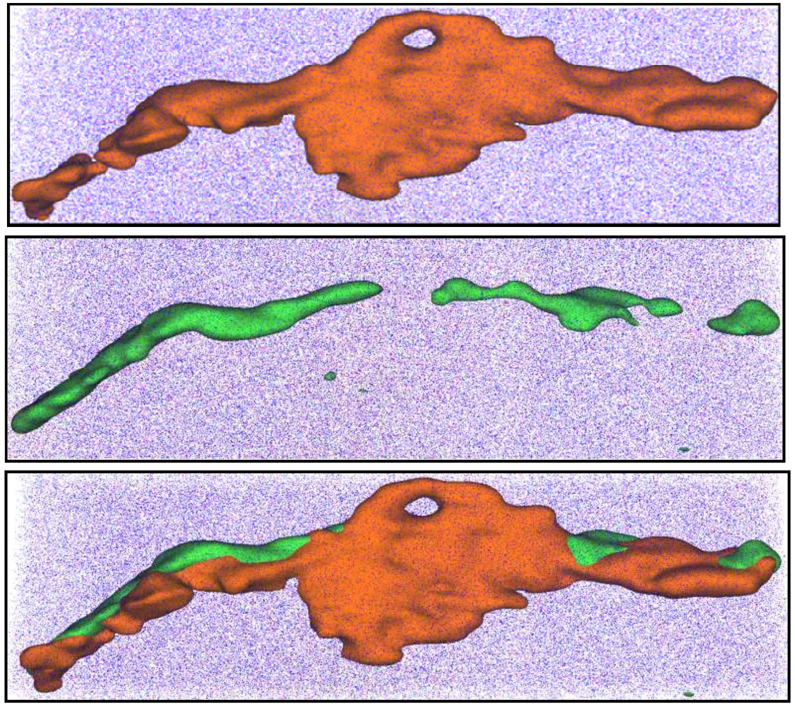
How to be precise and realistic

In-situ measurements + coupling with local technique



From B. Decreus et al. submitted to Acta Mater.





In-situ SAXS + Atom Probe Tomography

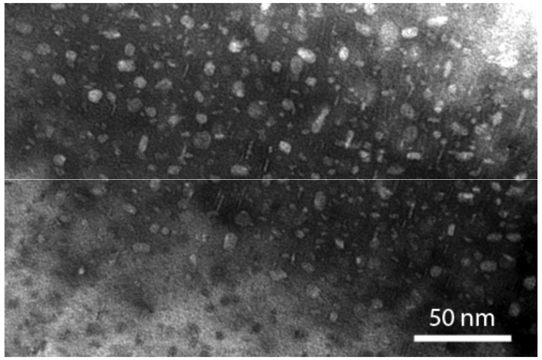
Nucleation mechanisms of T_1 precipitates in AlCuLi

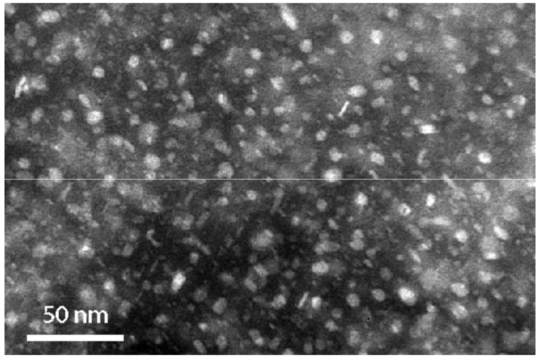
From: V. Araullo-Peters et al. , Univ. of Sydney, Australia

How to be precise and realistic

Thermo-mechanical in-situ:
Precipitation under temperature & strain

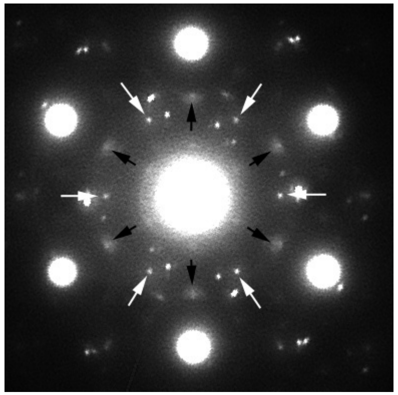
Al-Zn-Mg-Cu alloys
Small-Angle X-ray Scattering + TEM

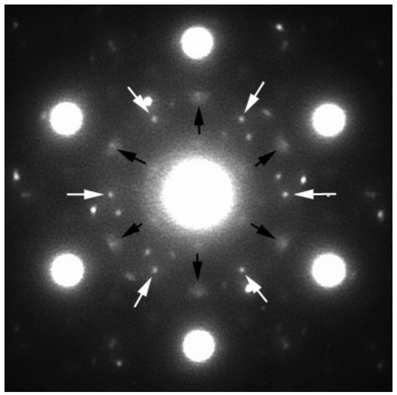


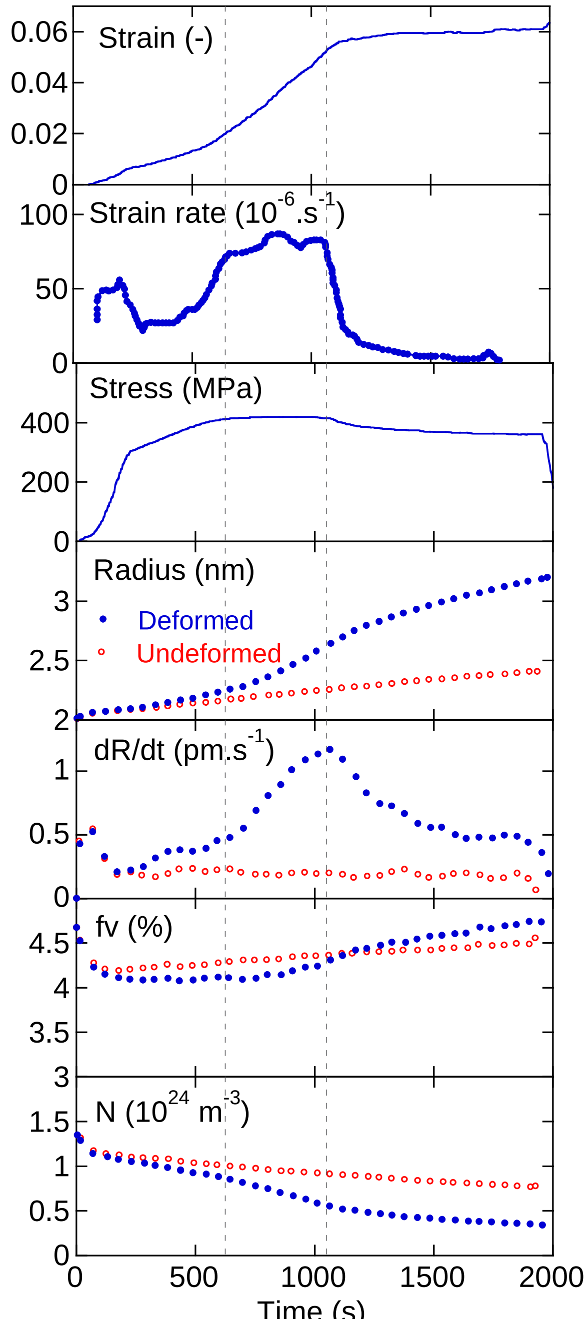


Undeformed

Deformed @ 160°C



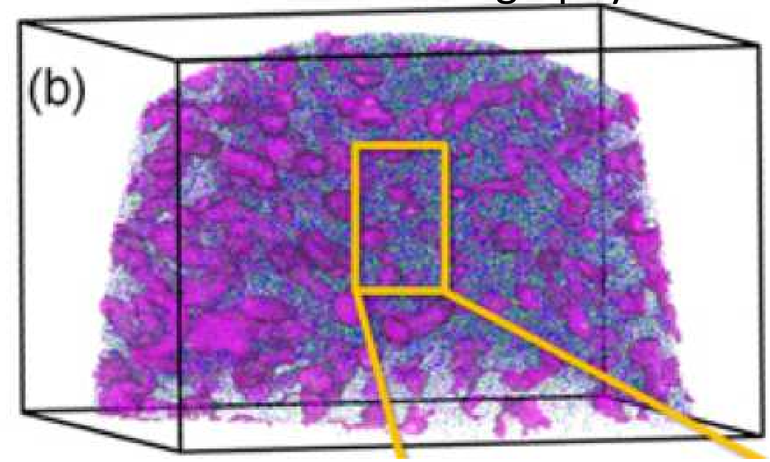




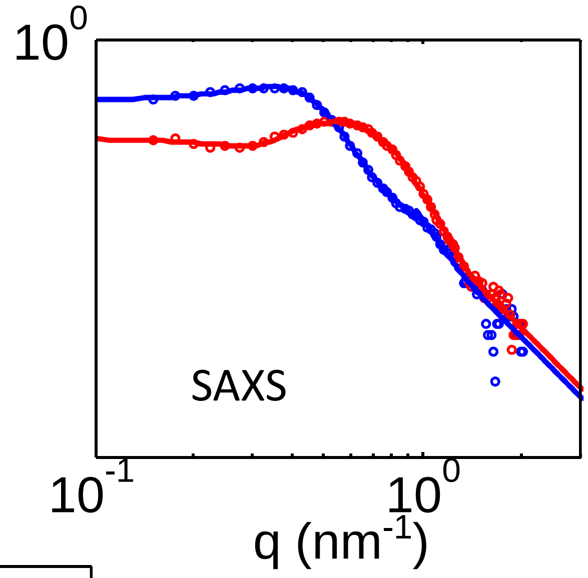
From: A. Deschamps et al. / Acta Materialia 60 (2012) 1905–1916

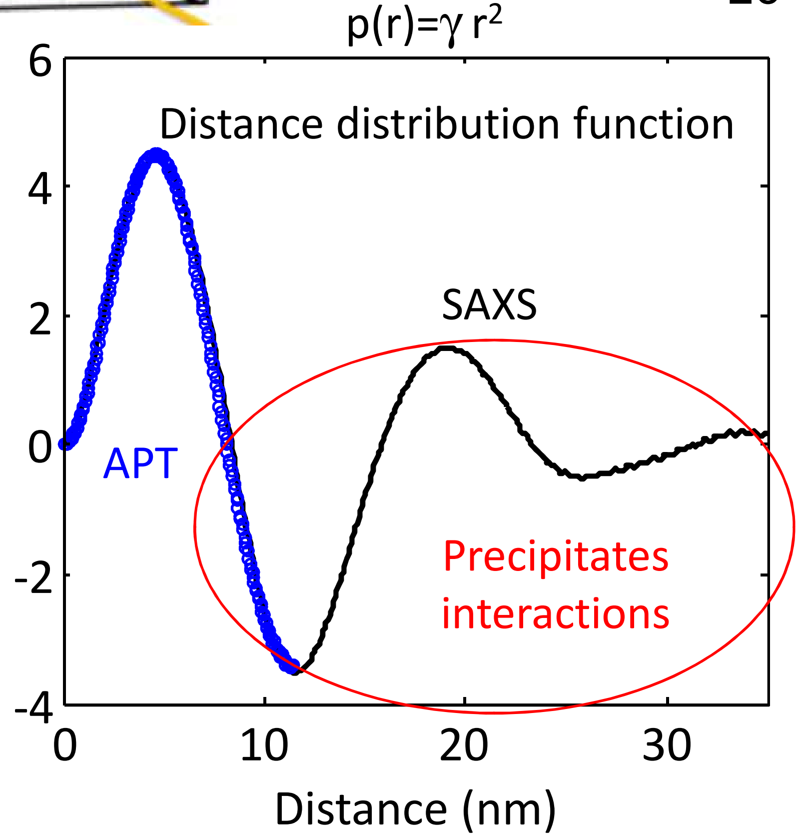
How to be precise and realistic

Atom Probe Tomography



Direct comparison between local and global techniques



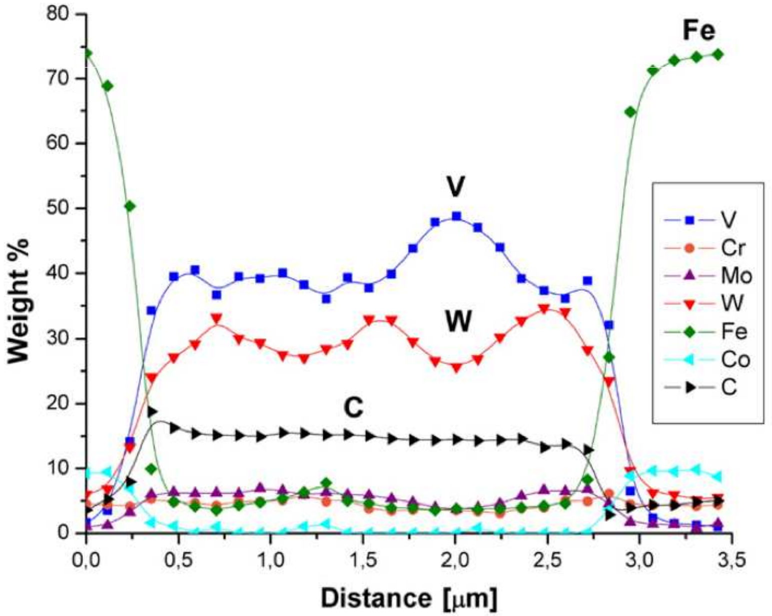


From: F. De Geuser (SIMAP)
B. Gault (Univ. McMaster)

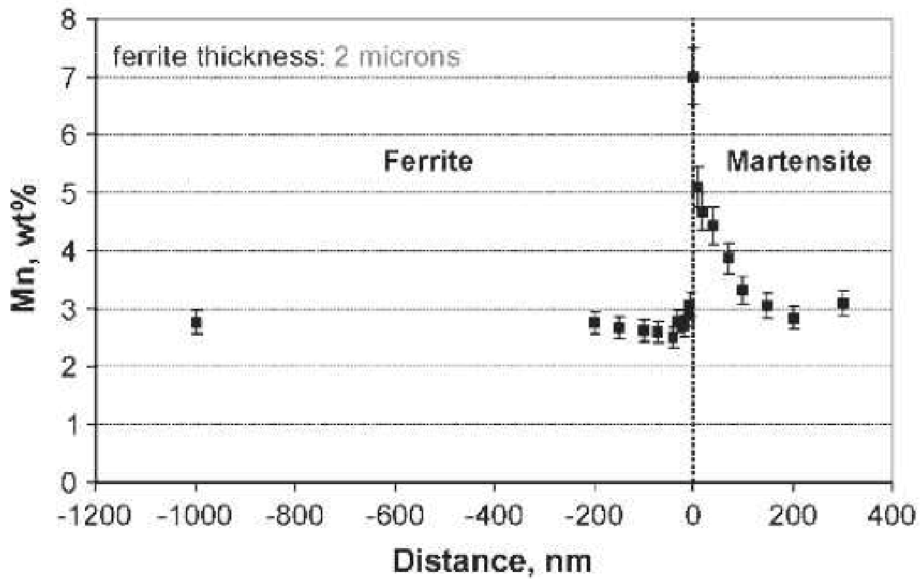
Multi-constituent alloys

Non stoichiometry
Conditions at the interfaces

Need for good thermodynamic and kinetics databases
Solving the compromise between driving force and kinetics
Interfaces : snowplow effect, segregation, precipitation, …



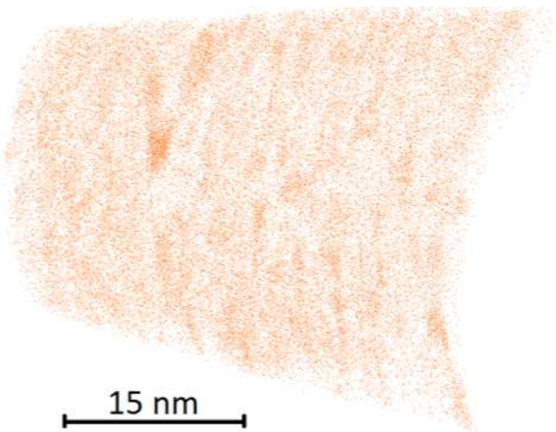
From M. Godec et al.
MATERIALS CHARACTERIZATION 61 (2010) 452–458

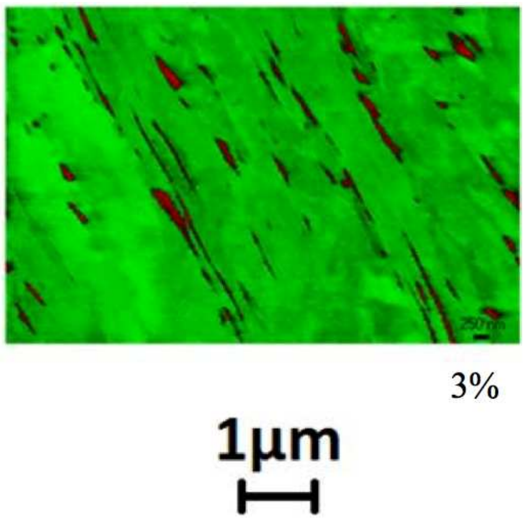


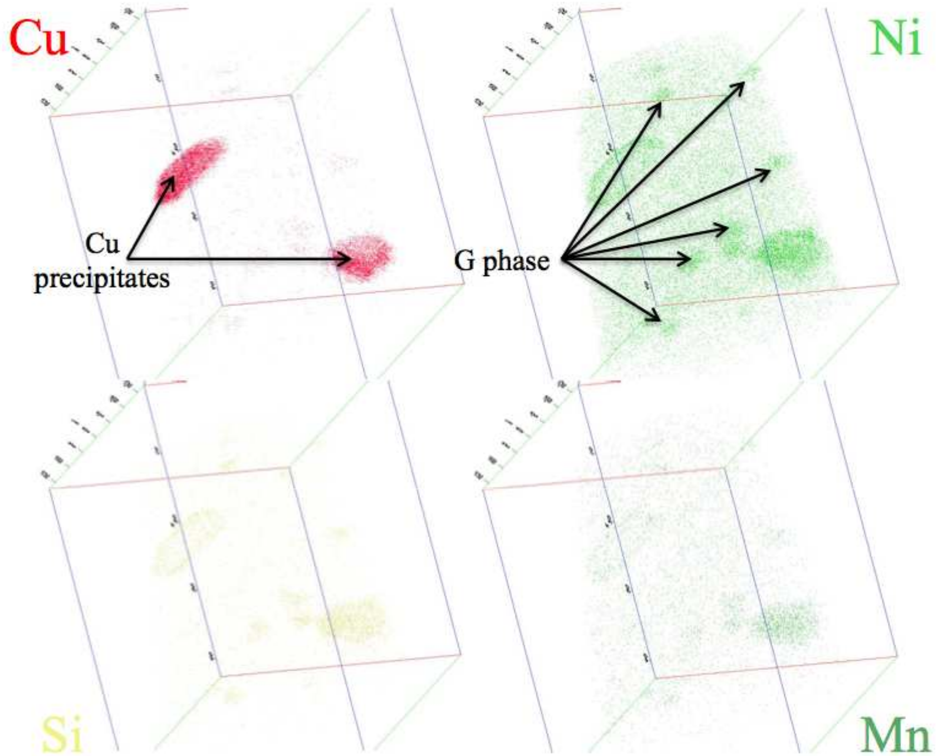
From H. Guo et al. Met Mater Trans 1724—37A, 2006

Multi-constituent alloys

Multiple features at different scales







Ageing of precipitation hardened stainless steel

- Spinodal decomposition
- Cu and G-phase precipitation
- Retained austenite

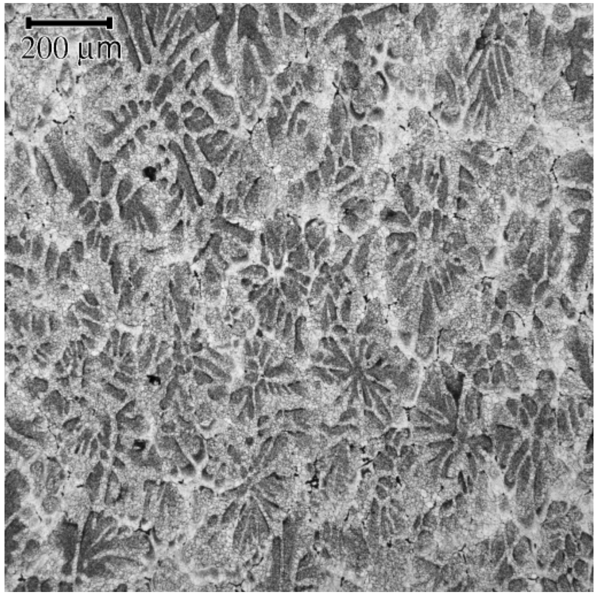
From: L. Couturier, PhD work (INPG) & CIRIMAT (Toulouse)

Heterogeneous microstructures

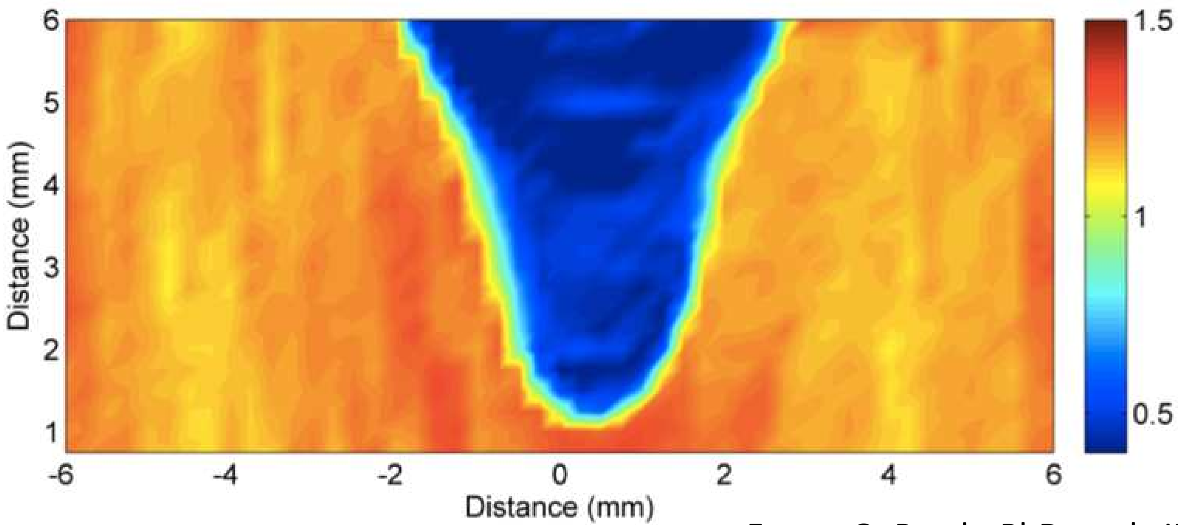
Heterogeneity due to solidification

Heterogeneity due to deformation (e.g. forming)

Secondary processes (welding)



From: D. Dumont et al.
Mat. Sci. Eng. A 2003 ; 356 : 326-336.



From : Q. Puydt, PhD work, INPG

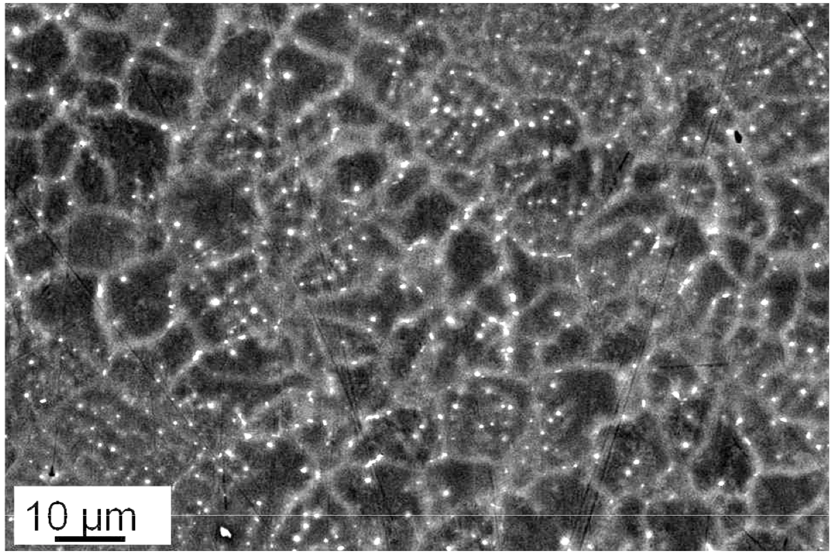
Real materials : what kind of complexity

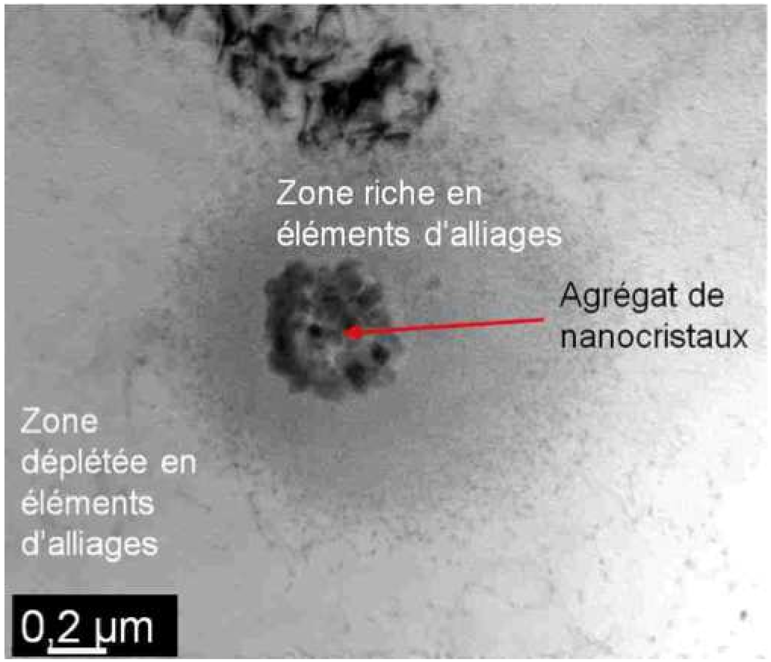
Heterogeneous microstructures

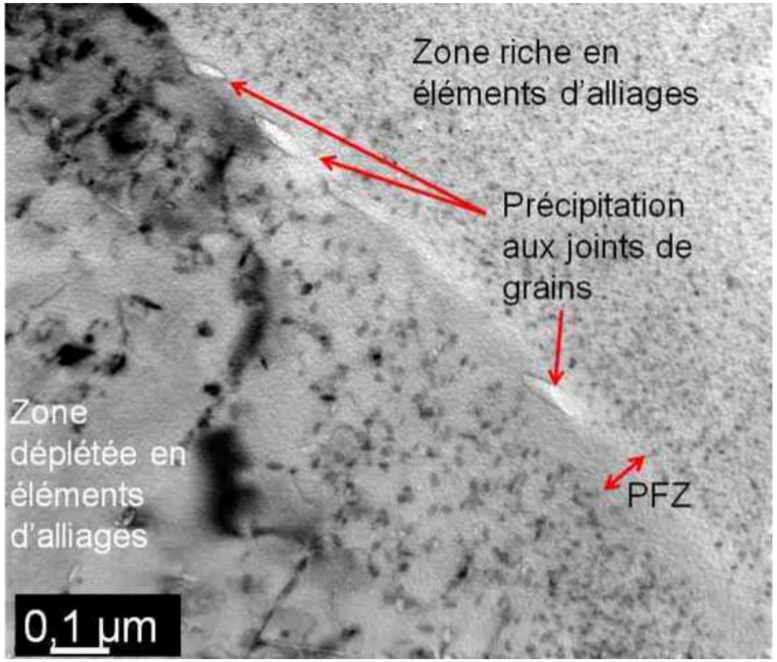
Electron beam welding of Al-Zn-Mg
+ post welding heat treatment

Heterogeneous distribution of solute and precipitates

From : Q. Puydt, PhD work, INPG

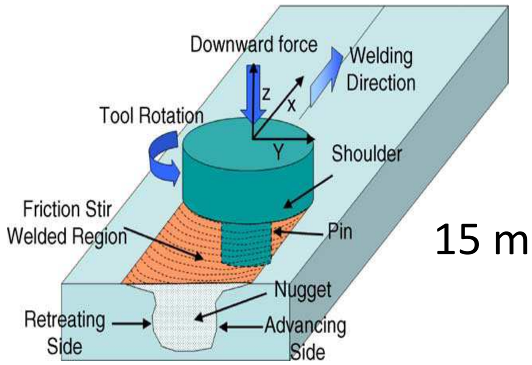


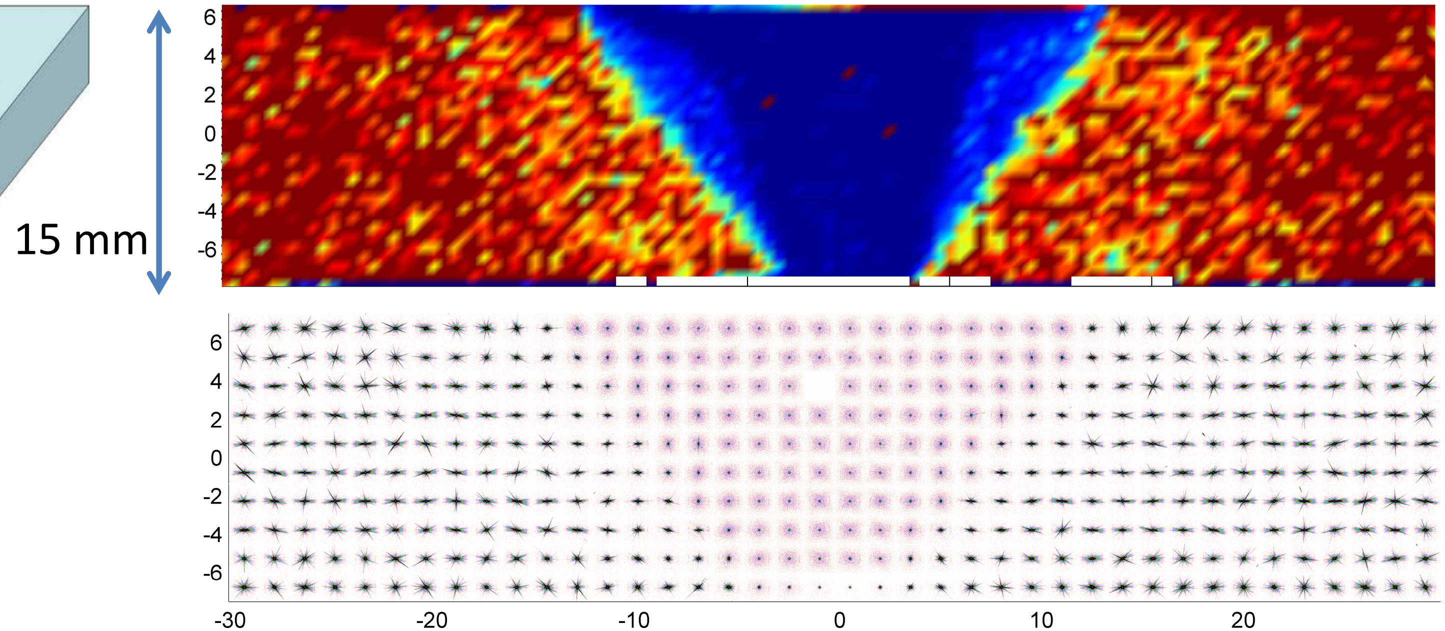


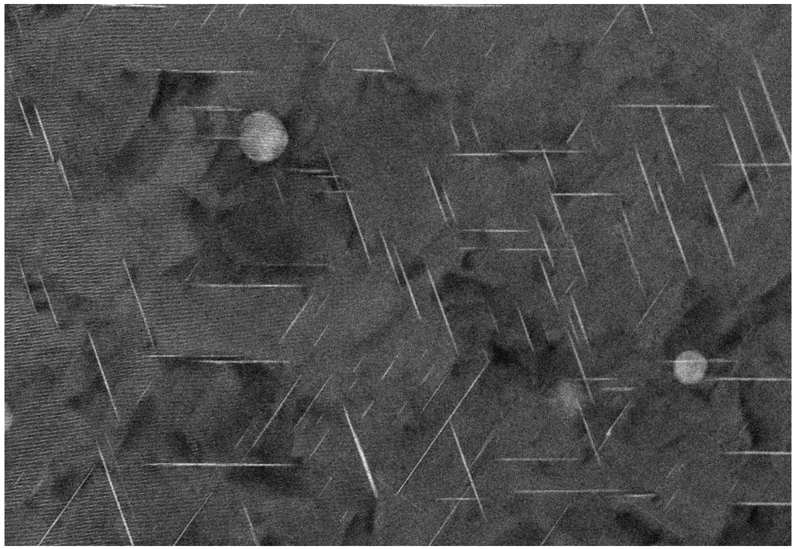


Real materials : what kind of complexity

Heterogeneous microstructures







From: B. Malard et al., Coralis project, SIMAP

Friction Stir Welding of Al-Cu-Li : SAXS mapping

Spatial distribution of precipitate microstructures

How to be realistic and precise on real materials?

Towards in-operando: a gleeble in a high energy synchrotron beamline

Deformation

Non-isothermal path

Fast kinetics

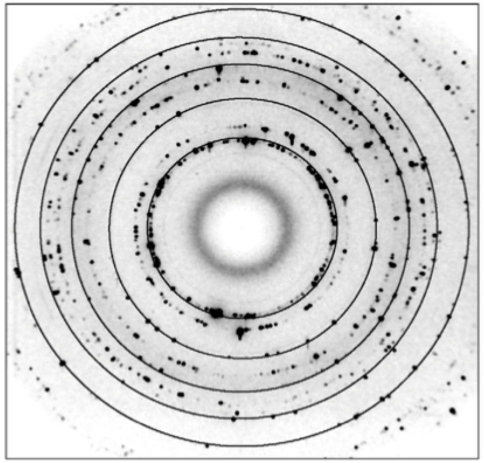
XRD
measurements

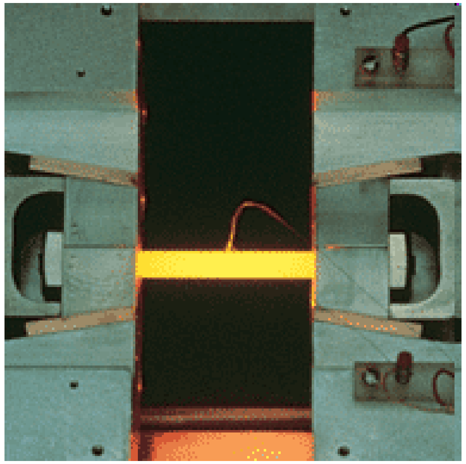
Solidification

Phase transformations

Precipitation

Recrystallization





Hermès project (Alain Jacques, IJL) :
High Energy Radiation for Metallurgical Studies