Genèse des Microstructures I

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Introduction

**Microstructures**
- Macroscale: heterogeneity
- Microscale: phases, grains
- Sub-microscale: phases, dislocation structures
- Nanoscale: precipitates, clusters

**Characterization methods**
- Local: OM, SEM, EBSD, TEM, APT, ...
- Global: XRD, DSC, SAS, ...

**Mechanisms**
- Diffusion
- Dislocation motion
- Interface motion
- Solute effects
- Stress effects

**Disciplines**
- Solidification
- Phase transformations
- Plasticity
- Recrystallisation & grain growth

**Modelling methods**
- Microstructure II
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
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)
plexsite

Introduction

(cooling rate: 1-100°C per 1,000,000 years)

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)

Microstructure at the microscale: grains and phases

Phase characterization – structure – X-ray diffraction

Rietveld refinement → Phase proportions

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

Phase transformations in Ti alloys

G. Geandier et al.

Fig. 1. Diffraction diagrams evolution over time for a β-metastable titanium alloy (Ti17).
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
Microstructure at the microscale: grains and phases

Grain characterization – texture – EBSD

Precautions:

Surface state
Strain (dislocations)

From: AF Gourgues
Lecture notes

A. Deschamps et al. / Materials Science and Engineering A 517
(2009) 361–368
Microstructure at the microscale: grains and phases

Grain characterization – texture – EBSD

“in-situ” recrystallisation in the SEM

Zirconium alloy

From N. Bozzolo et al., MATERIALS CHARACTERIZATION 70 (2012) 28–32
Microstructure at the microscale: grains and phases

Phase characterization – texture – EBSD

Discriminate between different crystallographic phases + study of orientation relationship

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

Ti alloy orientation of $\alpha+\beta$ phase

Residual $\beta$ phase and sketch of former $\beta$ GBs
Multiple phase detection: unicity of Kikuchi line analysis?

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

Carbide characterization in a high speed steel

From M. Godec et al. MATERIALS CHARACTERIZATION 61 (2010) 452–458
Microstructure at the microscale: grains and phases

Phase characterization – chemical information

Electron Probe Microanalysis (EPMA)

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

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

Orientation mapping in the TEM:
ASTAR technique

Nanocrystalline Aluminium

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
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
Microstructure at the sub-microscale: dislocations

Dislocation structures: Transmission Electron Microscopy

TWIP steels
- Dislocations
- Stacking faults
- Twins

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
Microstructure at the sub-microscale: dislocations

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

![Graph showing dislocation density vs. true plastic strain]

From: B. Hutchinson, N. Ridley

Dislocation density measurement by resistivity

![Graph showing dislocation density vs. true strain]

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
Microstructure at the nano-scale: precipitation

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

$\text{T}_1 - \text{Al}_2\text{CuLi}$ precipitates in Al


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: A. Deschamps et al.
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)

X-ray or neutron Beam (monochromatic)

Sample

Detector

$q = 4\pi \sin \theta / \lambda$

$1/t$

$1/D$

$\theta$

$\lambda$

$D$

$t$

20 nm
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

In-situ measurements
Temperature, strain

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


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

Microstructure at the nano-scale: precipitation

Volume fraction, thermodynamics: DSC

Differential calorimetry: easy quantification $\Delta H$, $f_v$, solvus $T$, ...
But interpretation can be tricky in complex systems (many phases)

Dissolution kinetics of $\delta'$ in Al-Mg-Li

Precipitation kinetics during cooling in Al-Zn-Mg

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

How to be precise and realistic

Distance distribution function

$p(r) = \gamma r^2$

Distance (nm)

SAXS

APT

Precipitates interactions

From: F. De Geuser (SIMAP)
B. Gault (Univ. McMaster)
Real materials: what kind of complexity

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

Real materials: what kind of complexity

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)
Real materials: what kind of complexity

Heterogeneous microstructures

Heterogeneity due to solidification

Heterogeneity due to deformation (e.g. forming)

Secondary processes (welding)

From: D. Dumont et al.

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

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

Spatial distribution of precipitate microstructures

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