Interest of new observation techniques to understand and improve steel microstructures and fracture properties

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P. Barges, S. Cobo, L. Germain, J. C. Hell, M. Kahziz, M. Maziere, T. Morgeneyer, A. Perlade, J.M. Pipard, C. C. Tasan, M. Salib, M. Wang

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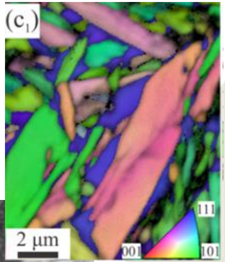
LEM3 LABORATOIRE D'ÉTUDE DES MICROSTRUCTURES ET DE MÉCANIQUE DES MATÉRIAUX
MINES ParisTech

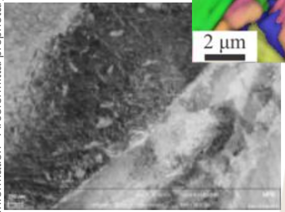
Aussois 2019 – 21-25 jan. 2019

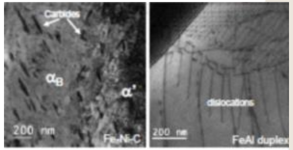
Rupture des Matériaux et des structures – Mécanismes et modélisations face aux applications industrielles

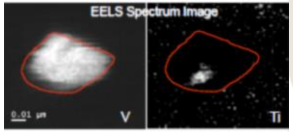
The right formula
for the steels of the future

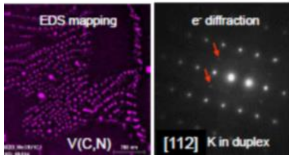
Complementary techniques

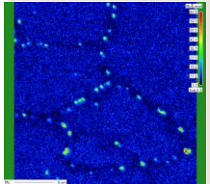










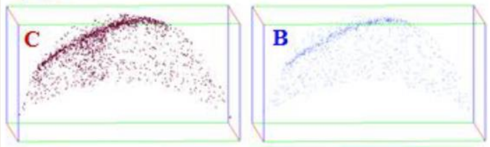


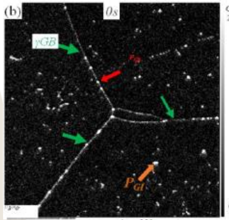
- **FEG - SEM**
- **EBSD** *(electron Back Scattering diffraction)*
  - ✓ Phase analysis
  - ✓ Reconstruction
  - ✓ Variant Analysis
- **ECCI** *(Electron channeling Contrast)*
- **TEM - ASTAR**
  (Transmission Electron Microscopy)
  (Automated orientation and phase mapping)
- **FEG - EPMA**

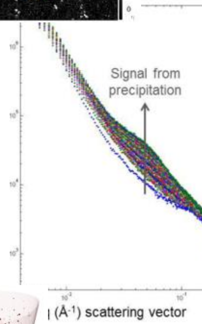
- **APT** *(Atom Probe Tomography)*
  - ✓ Interface and GB segregations
  - ✓ Clusters and precipitates
- **Nano-sims**
  - ✓ Interface and GB segregations
  - ✓ Precipitates
- **Neutron diffraction**
  (small angle neutron scattering)
  - ✓ Mean Particle size
  - ✓ Particle size distribution

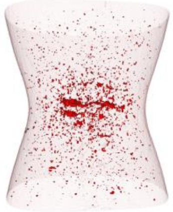
HR-XRD *(High resolution X-Ray Diffraction)*
- ✓ In situ phase analysis
- ✓ Internal stresses

X-Ray tomography
- ✓ In situ damage analysis









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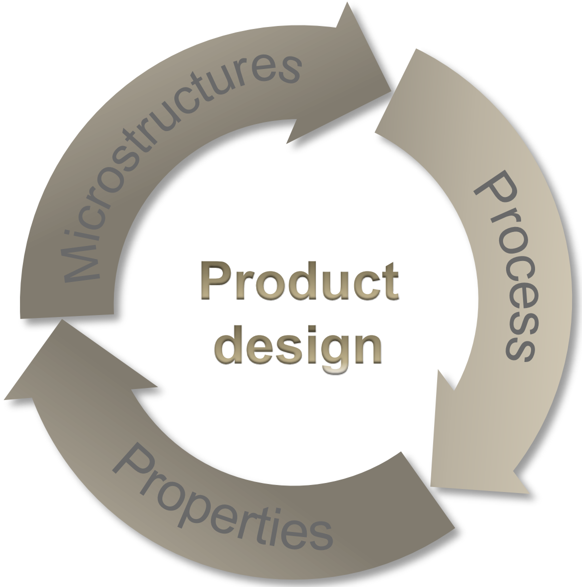
Product design for improved damage and fracture properties



- 3 case studies
  1. Product guidelines for fracture resistance of third generation Q&P steels
  2. Product guidelines for improved resistance of cut-edges in bainitic steels
  3. Root cause identification of brittleness induced by tempering

… Always starting from observations
… Using more or less recent technics

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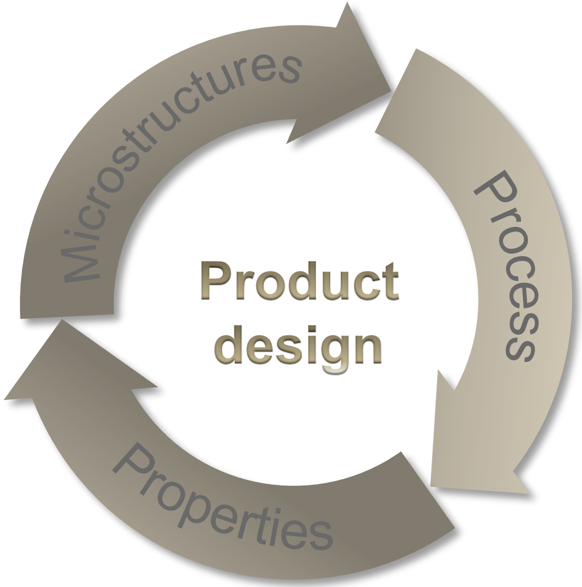
Product design for improved damage and fracture properties



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Case study 1 – Q&P steels

M. Wang, J.C. Hell, C.C. Tasan
M. Wang et al, Scripta Materialia 138 (2017) 1–5

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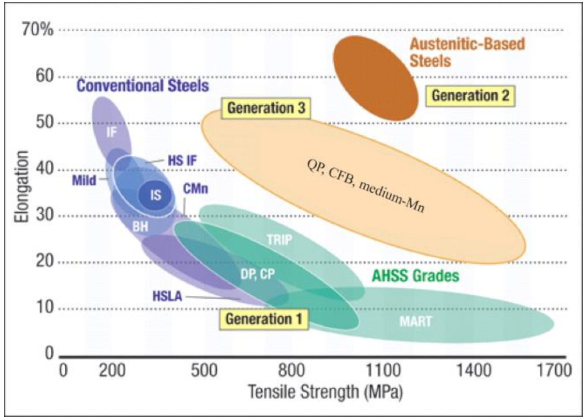
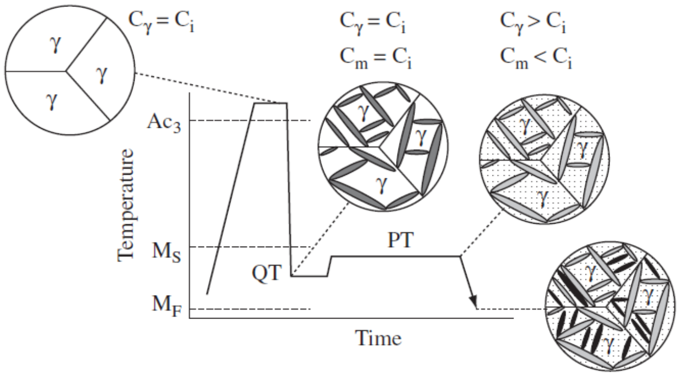
Third generation Quenched and Partitioned steels

- Q&P steels exhibit excellent combination of high strength and ductility.
- Q&P steels provide well trade-off between properties and production costs



Courtesy of WorldAuto Steel.



Schematic illustration of the Q&P process. Courtesy of J.G. Speer.

Which relationship between microstructure and mechanical properties, including fracture?

M. Wang, J.C. Hell, C.C. Tasan

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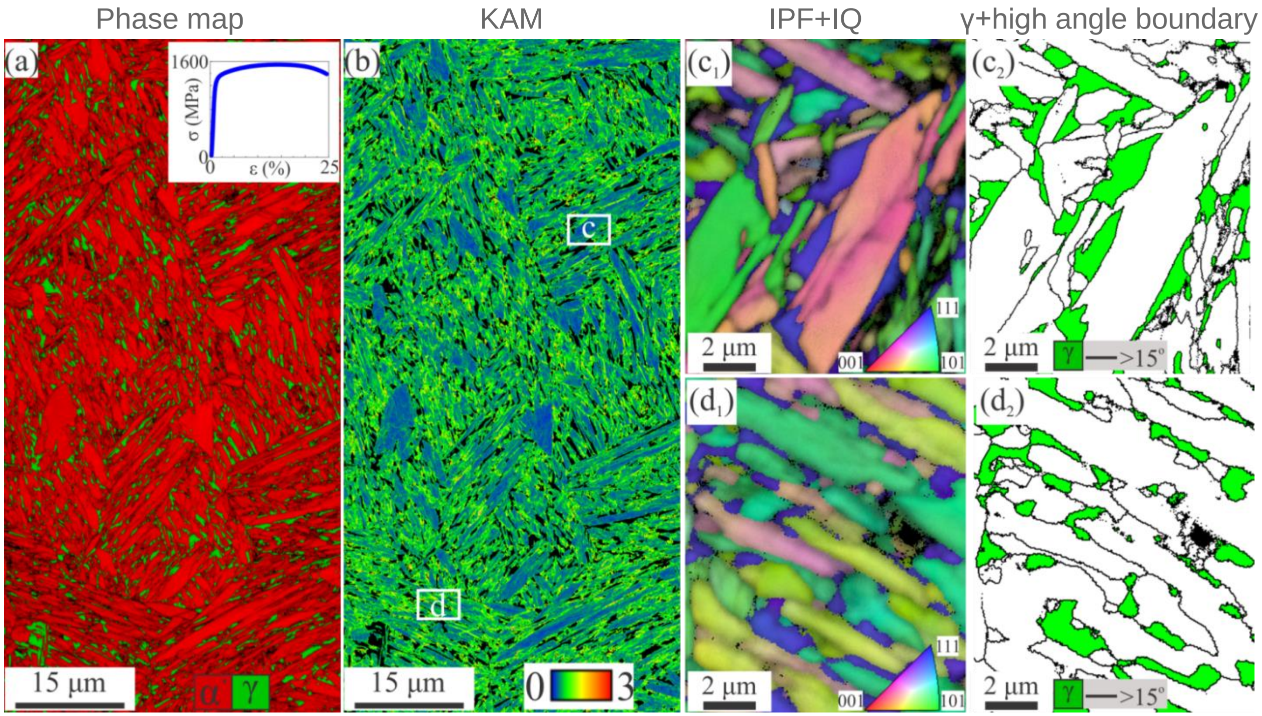
Typical Q&P microstructures

- Constituents
  - Tempered martensite (TM)
  - Retained austenite (RA)
  - Un-tempered martensite (UM)
  - Bainite
- Size distribution
  - Coarse TM laths
    ➔ 2.95 ± 1.01 µm
  - Fine TM laths
    ➔ 0.92 ± 0.35 µm



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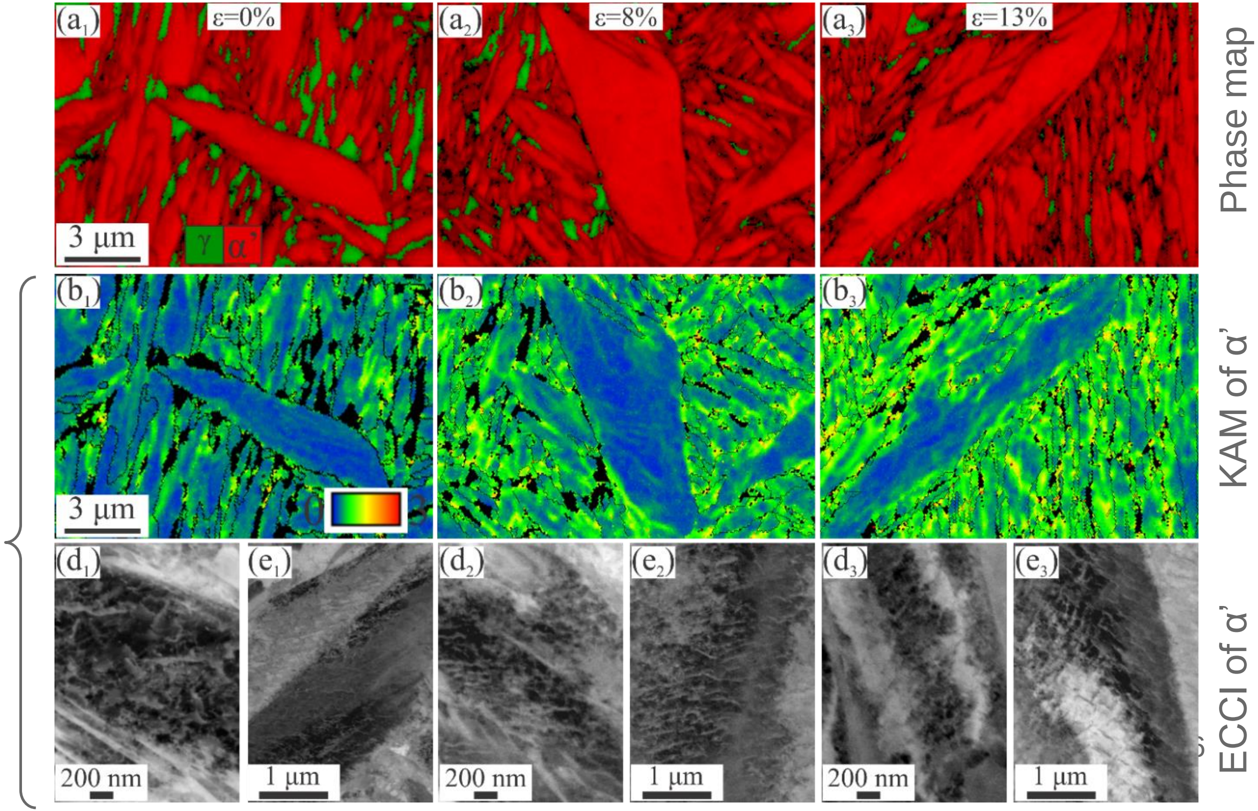
Deformation mechanisms

- Continuous transformation of γ into α' leading to TRIP

- Dislocation-mediated plasticity in α'



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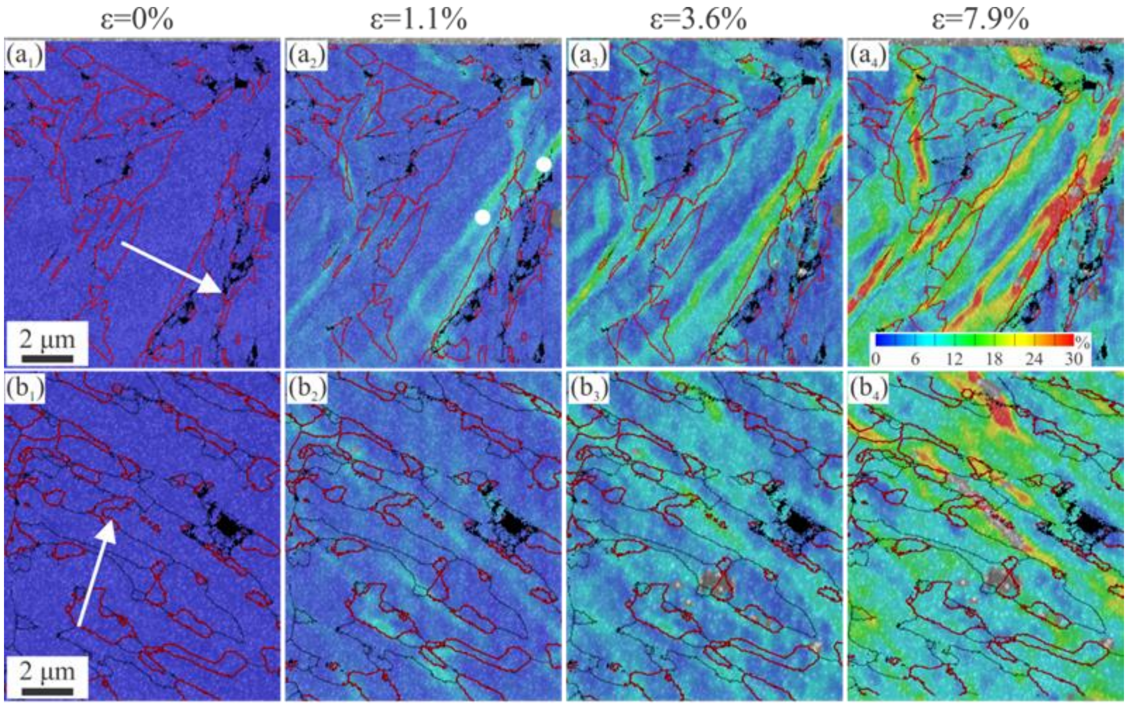
Deformation mechanisms - Strain mapping

In situ tension with High-resolution DIC based on in-lens image of silica particles



- Areas with coarse TM laths
- Heterogeneous strain distribution
- Early strain localization along coarse TM lath interfaces
  - Strongly dependent on neighboring constituents
  - Presence of UM at interfaces increase strain localization

- Areas with retained austenite and fine martensite laths
- Homogeneous strain distribution
- Plastic strain occurs first in RA

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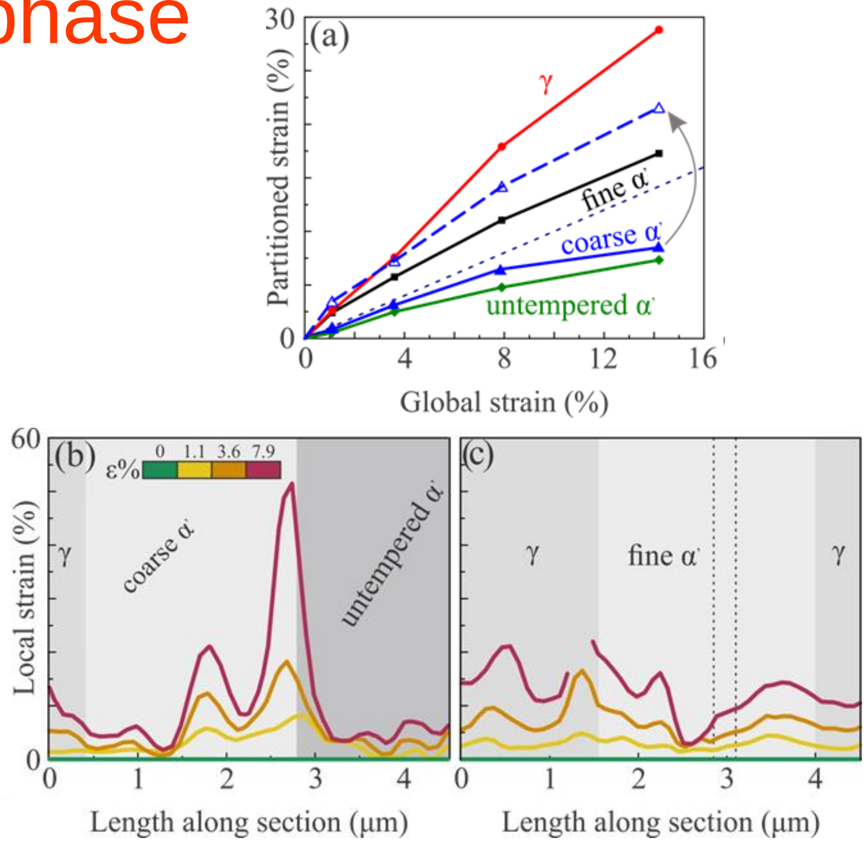
Strain partitioning of each phase

- Strain partitioning
  - Retained austenite exhibits highest level of plasticity
  - Followed by martensite laths (fine then coarse)
  - Coarse TM laths partition lower strain than fine TM laths but it depends on its neighbors
  - Untempered martensite shows the lowest level of plasticity

- Simultaneous presence of coarse lath and untempered martensite seen as most detrimental zones for damage nucleation in Q&P steels



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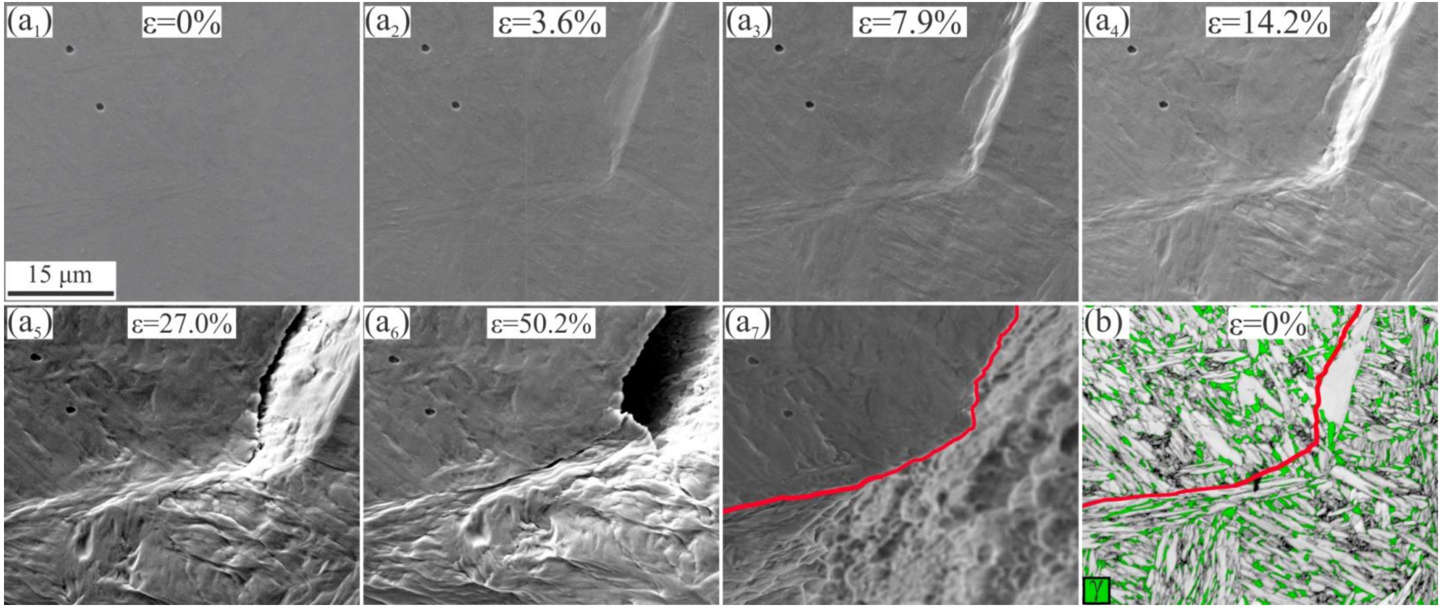
Fracture behavior

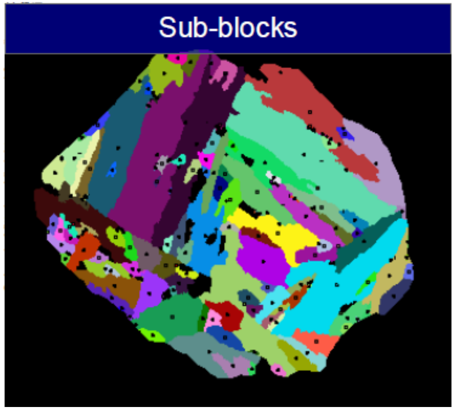


Detrimental effect of high strain heterogeneity built-up in coarse martensite laths in vicinity of untempered martensitic zones

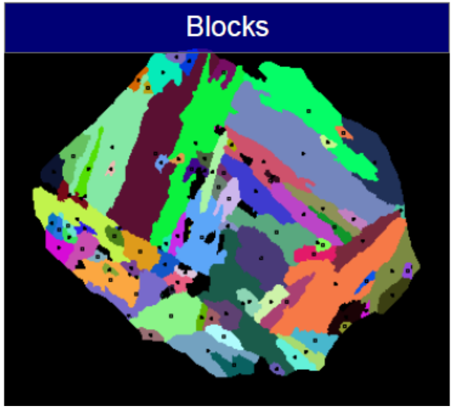
M.Salib, S.Cobo (MZ Lab), L.Germain (LEM3)

How to limit coarse martensite laths in the microstructure?

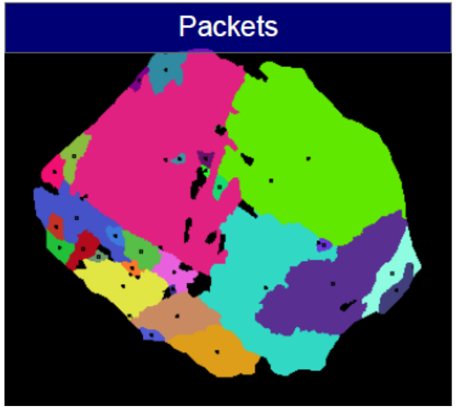
- Step 1: Clearly identify the martensite micro-constituents using EBSD
  - Grain reconstruction using **Merengue2** software
  - Automatic quantification of martensite sub-structure using **Decrypt** software



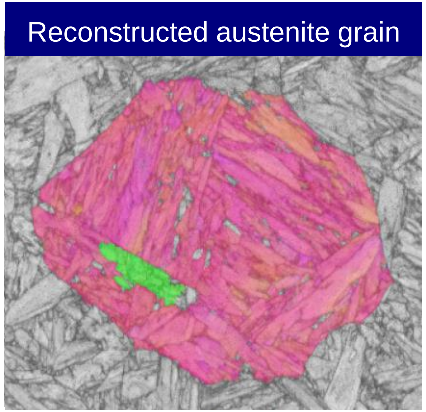
2 sub-blocks per block
1 variant of laths in each sub-block



3 blocks per packet



4 crystallographic packets



M.Salib, S.Cobo

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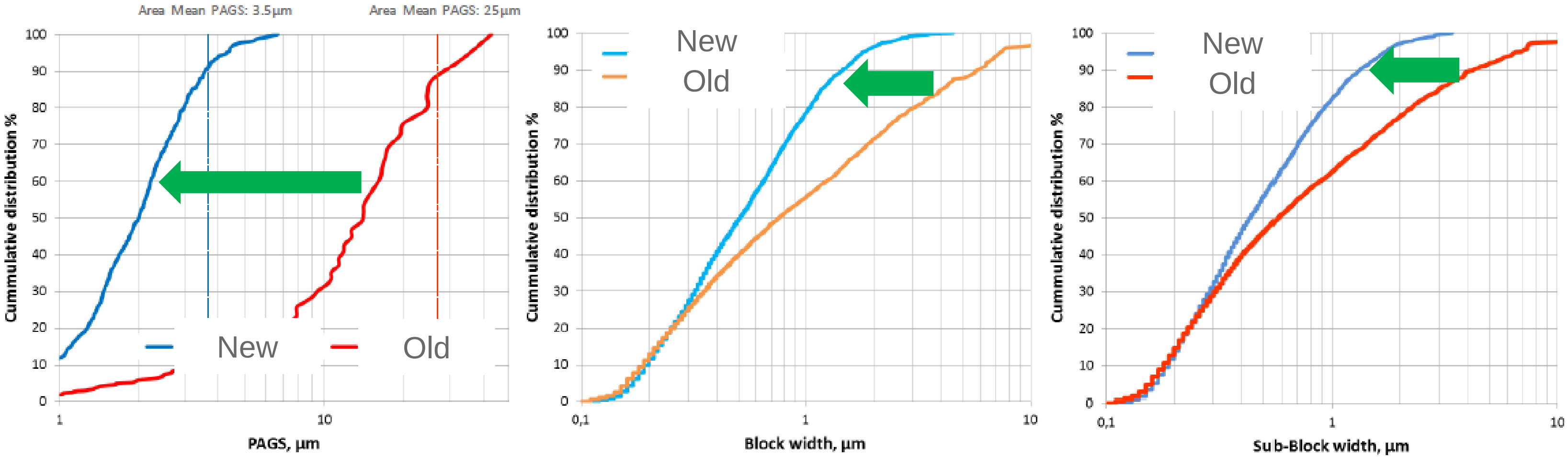
How to limit coarse martensite laths in the microstructure?

- Step 2: Find the appropriate metallurgical actuator

→ In this case, refinement of prior austenite grain size (PAGS) is an efficient actuator



PAGS refinement induces a reduction in microstructual heterogeneity and minimizes the presence of 'coarse laths'

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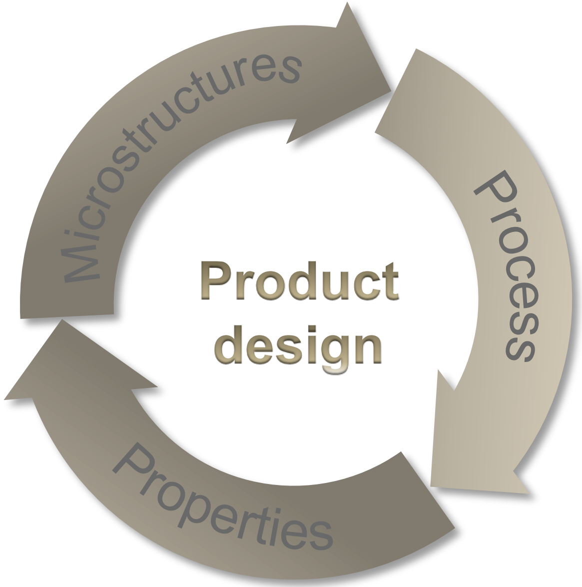
Product design for improved damage and fracture properties



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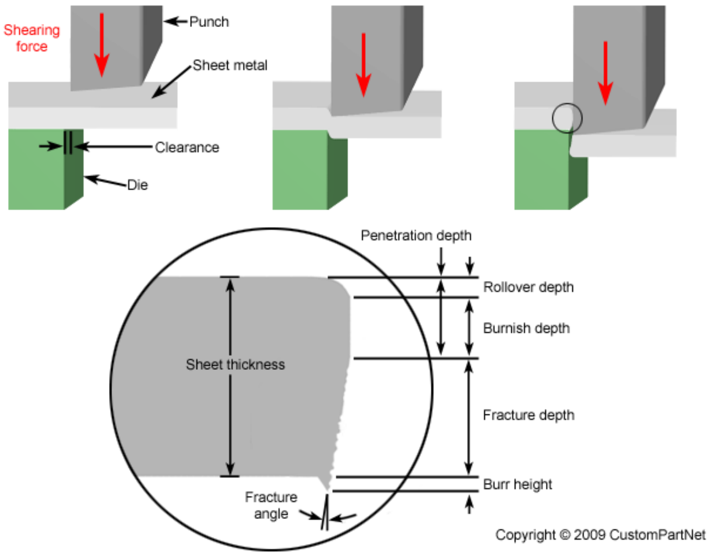
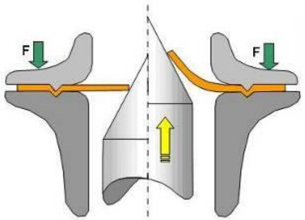
Background – Reduced formability due to cut-edges

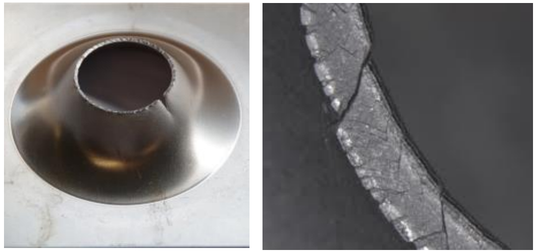
- Cutting process and cut-edge profile



- Formation of cracks from the cut-edges during forming process



$$HER = \frac{\phi_0 - \phi_f}{\phi_0}$$



- Effect of cut-edge geometry?
- Effect of strain hardening near the cut-edge?
- Effect of damage? Link with microstructure?

A. Perlade, J.M. Pipard

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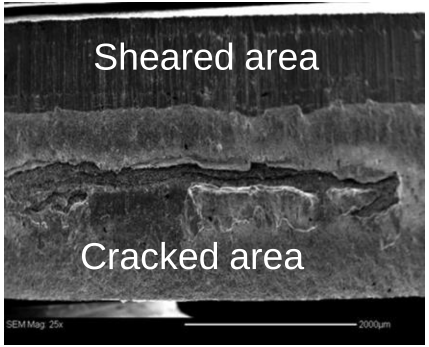
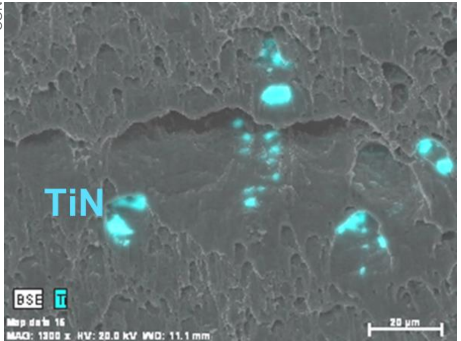
Starting with « classical » observations…

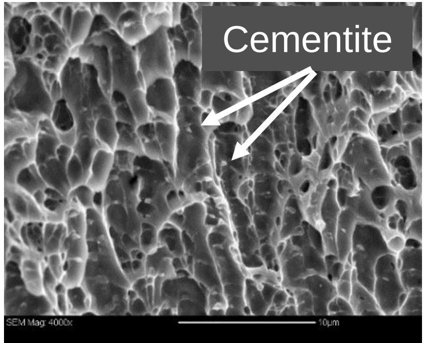
- After punching, before hole expansion

On fracture surfaces

EDS, etching, image analysis (dimple size & distribution)



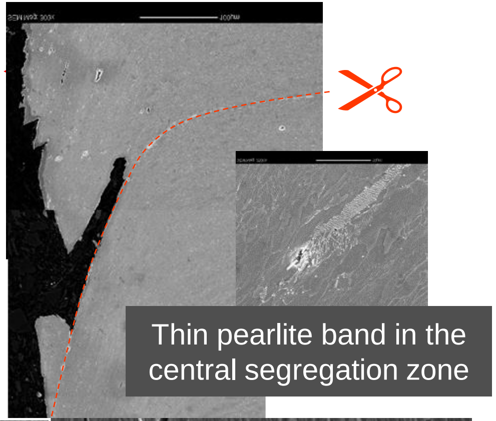


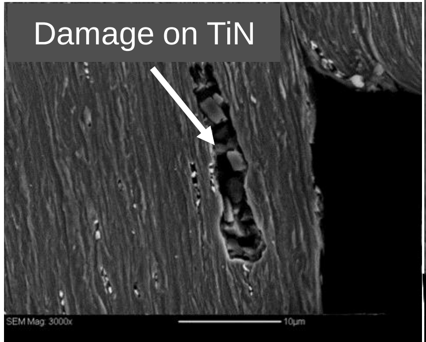


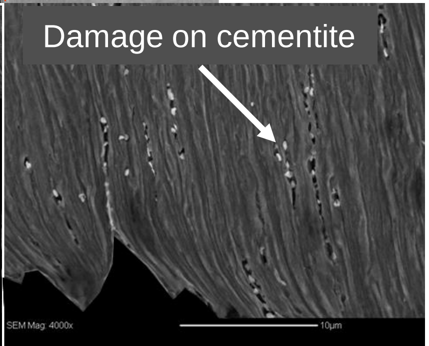
On cross-sections

Voids quantification & link with microstructural features

Damaged zone ~ 150µm







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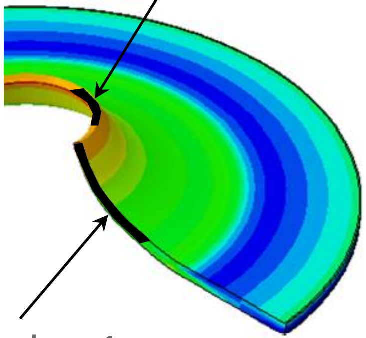
Starting with « classical » observations…

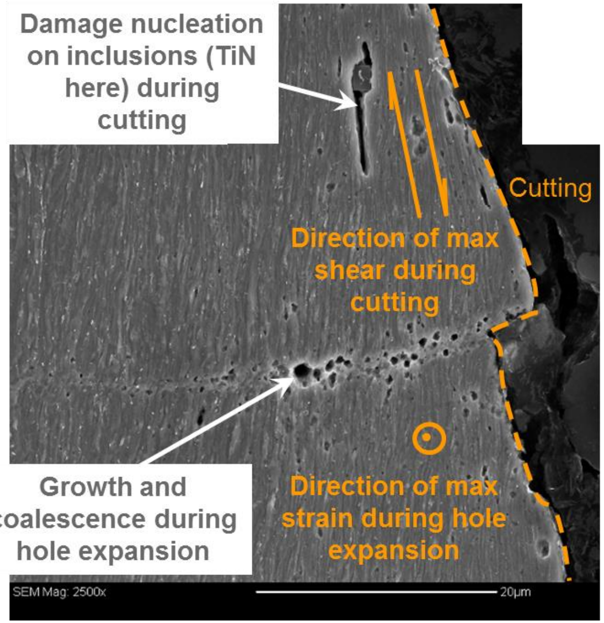
- After punching & hole expansion, on different cross sections

Section 2

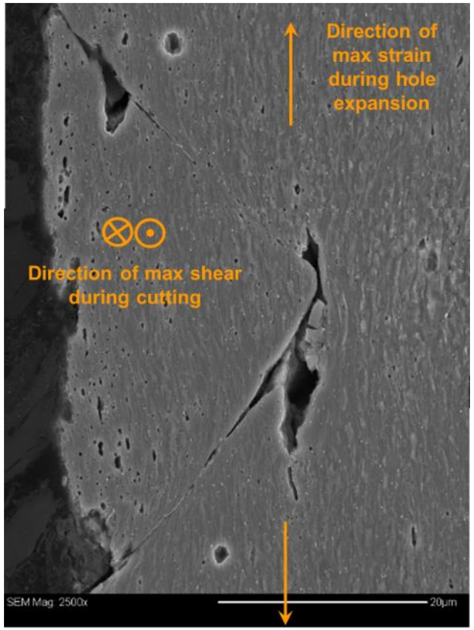


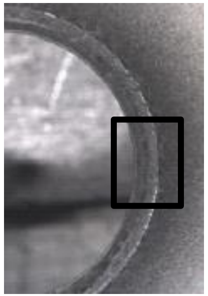
Section 1

Section 1



Section 2

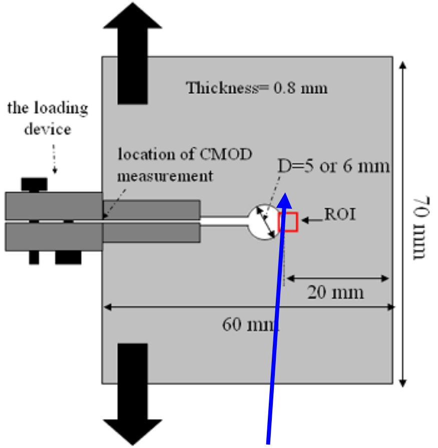




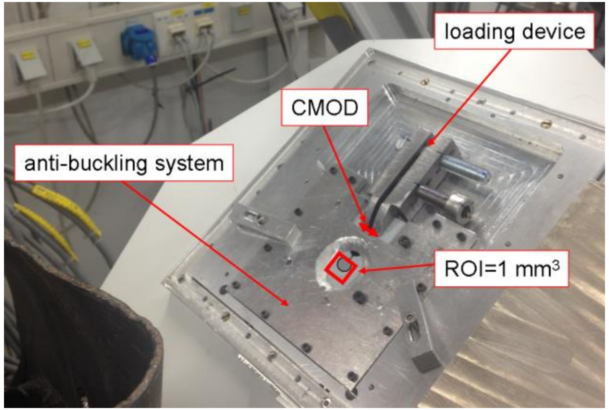
M. Kahziz, T. Morgeneyer, M. Mazière, E. Maire, A. Perlade

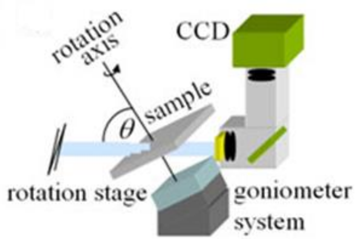
… to most advanced characterization technics

- In-situ X-ray laminography to study the cut-edge
  - An original sample designed to investigate the cut-edges and its damage in-situ.
  - 3D characterization of the damage induced by cut-edges after punching and during straining.



Punched or machined edge





M. Kahziz, T. Morgeneyer, M. Mazière, E. Maire, A. Perlade

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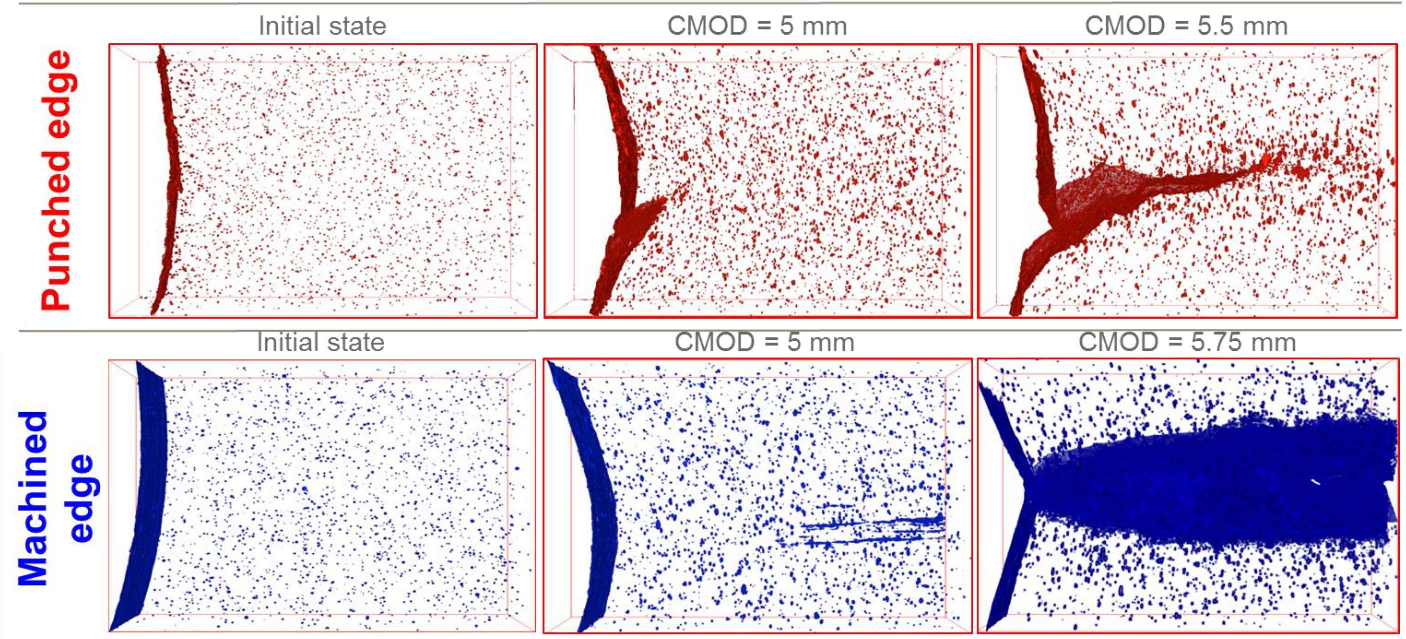
Edge fracture mechanisms

- Initiation of a crack from the cut-edge with slant propagation. Damage quantification
- Initiation from the bulk in the case of the machined edge.



1240×930×310 μm³

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Some ways to improve the edge formability

How to improve HE?	Defect modes	Design rules
1) Decrease the number of damage nucleation sites during cutting process	Shearing method	
	Central line segregation	Continuous casting conditions
	Large inclusions	Globularise the inclusions
		Limit Ti & N content to avoid any precipitation of TiN in the liquid phase
2) Decrease the propagation of micro-cracks during hole expansion	Large cementite particles and hard second phases	Apply a controlled cooling during ferrite and bainite transformation.

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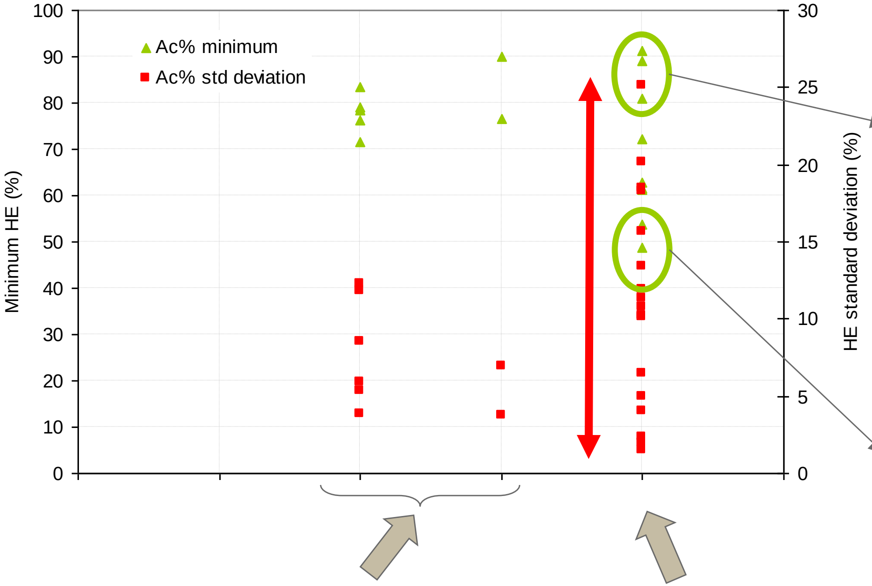
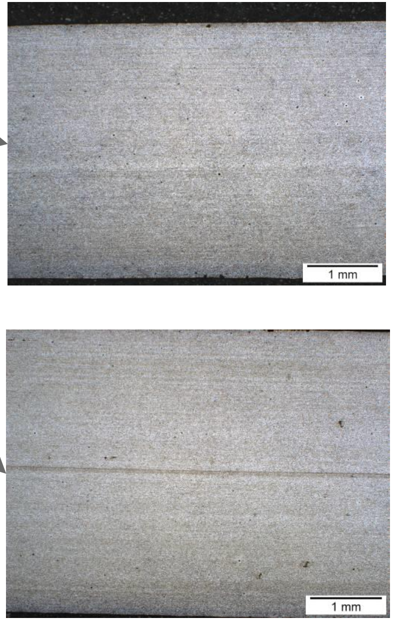
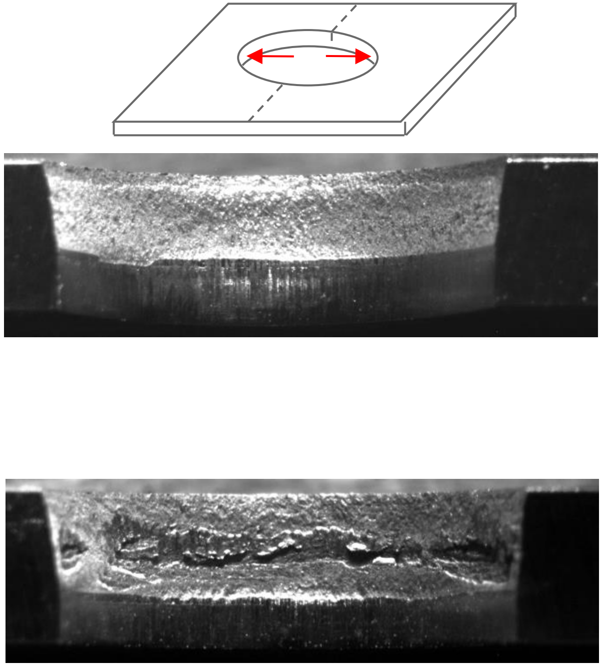
Reduce central segregation during casting is beneficial







Casting conditions 1 Casting conditions 2

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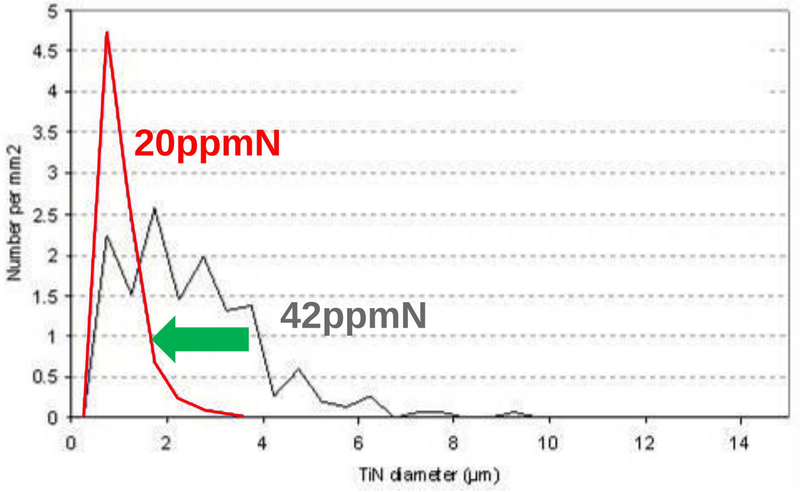
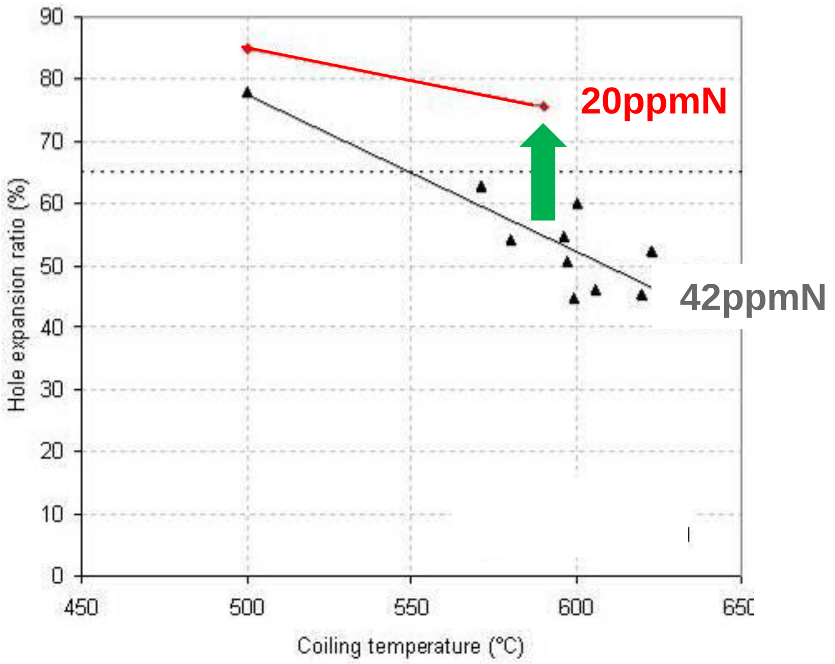
Reduce size of inclusions is beneficial





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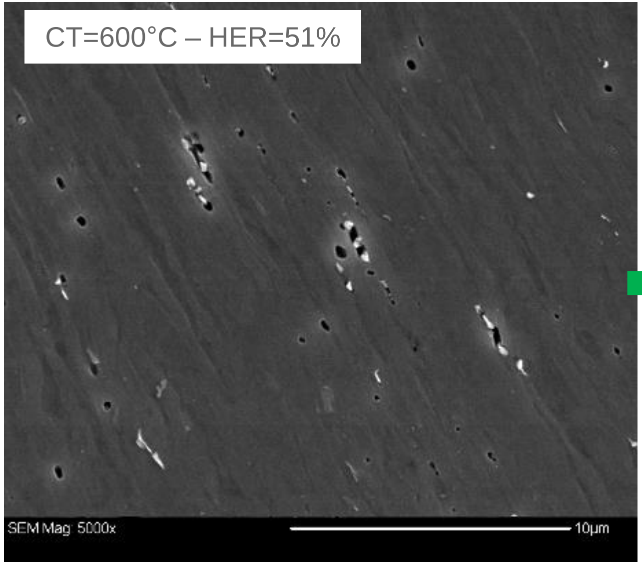
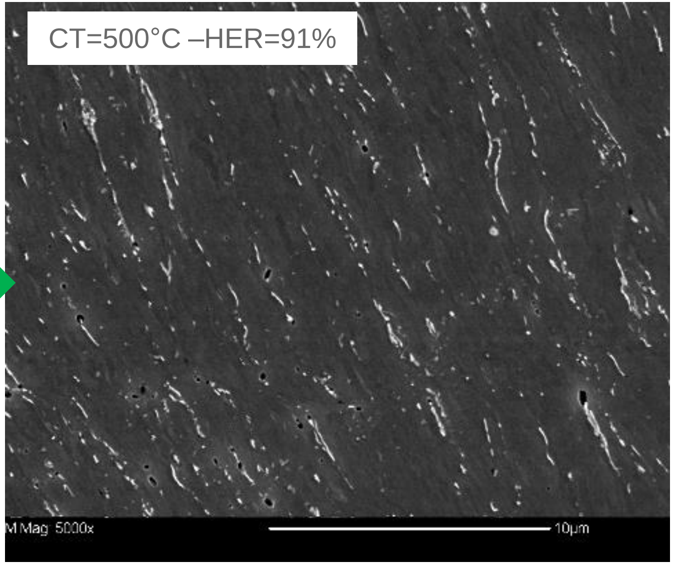
Control size and morphology of cementite is beneficial





- Decrease the coiling temperature leads to a strong change in the cementite morphology and a significant improvement in Hole expansion ratio
- Blocky cementite formed from ferrite is much more detrimental than fine cementite precipitated after bainite stasis

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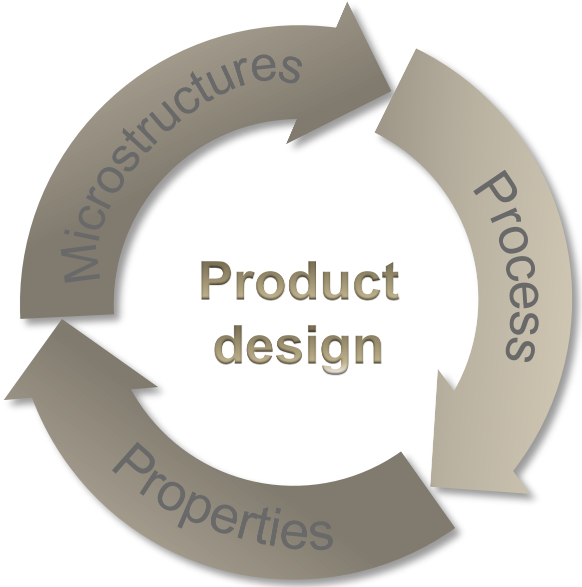
Product design for improved damage and fracture properties



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A. Perlade, K. Zhu, P. Barges

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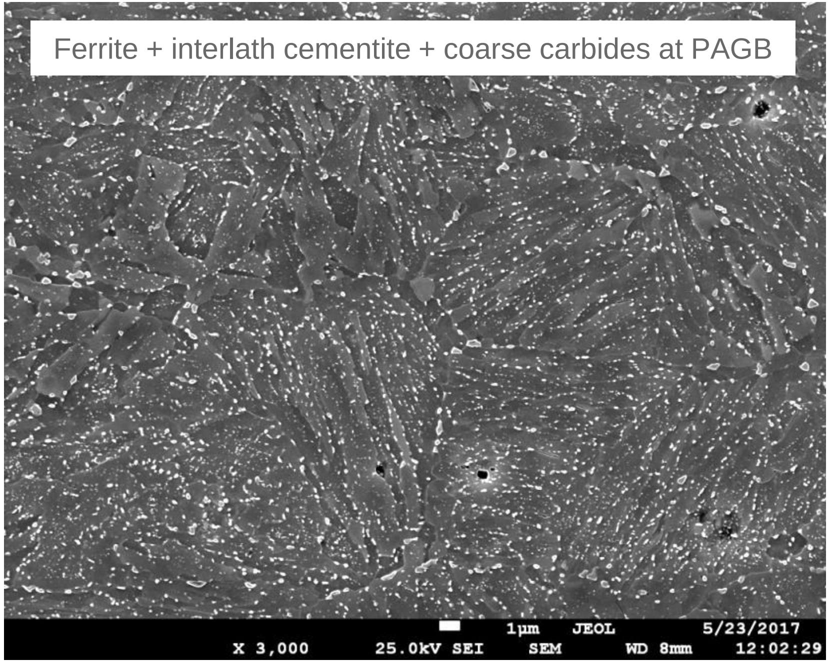
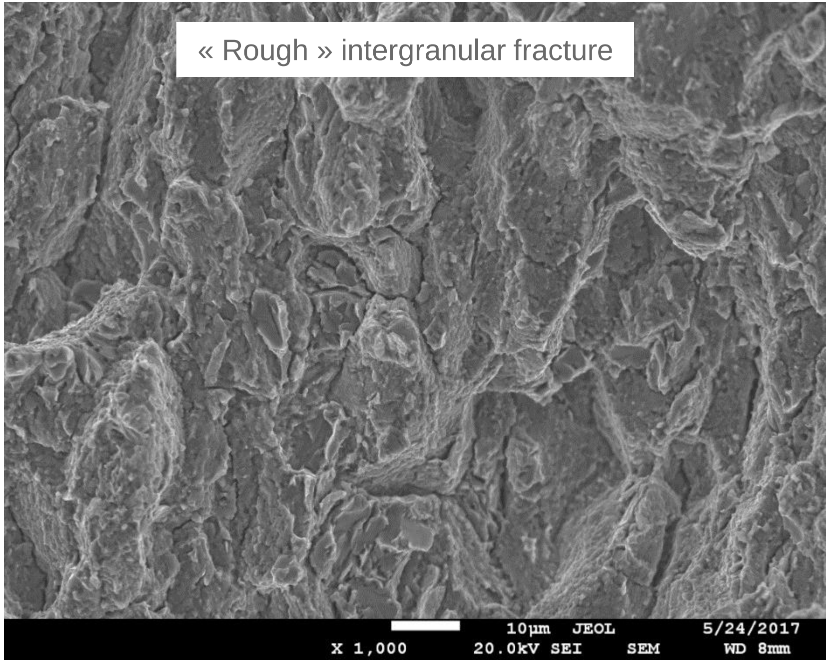
The problem:

Low Charpy toughness after tempering of AHSS





Why?

P. Barges

Indirect Rodoïd/Carbon replica on Charpy fracture surface and TEM observations

2% nital etching
of the fracture surface


Application of a polymer (Rodoïd)


Lifting of the Rodoïd


Carbon deposition

Scarifications


Dissolution of Rodoïd with aceton


Carbon replica deposition on Cu grid

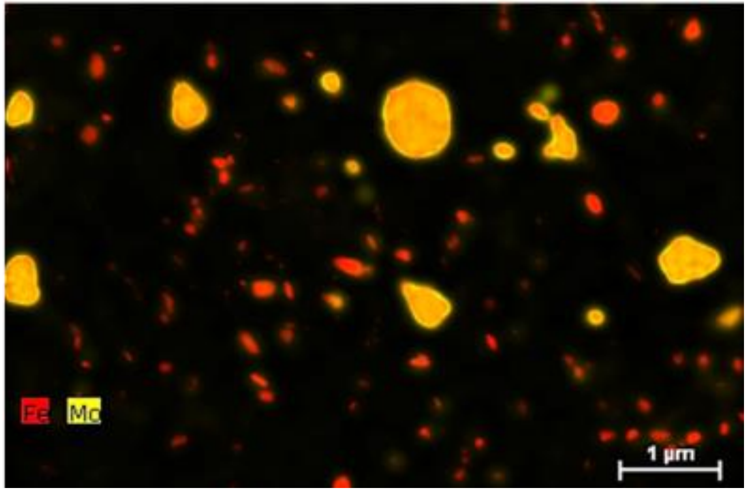
P. Barges

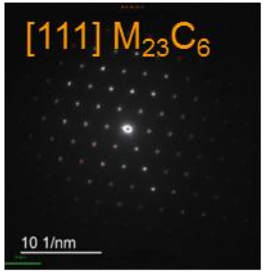
ArcelorMittal

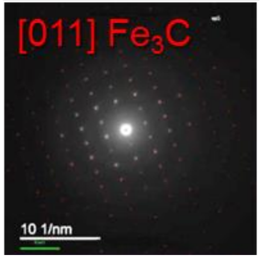
Identification of brittleness root cause

- Numerous $(Fe,Mn,Mo)_{23}(B,C)_6$ extracted on the fracture path of the tempered grade, while Fe_3C only are extracted in the bulk.

Fe_3C + $(Fe\text{-}Mo)_{23}(B\text{-}C)_6$







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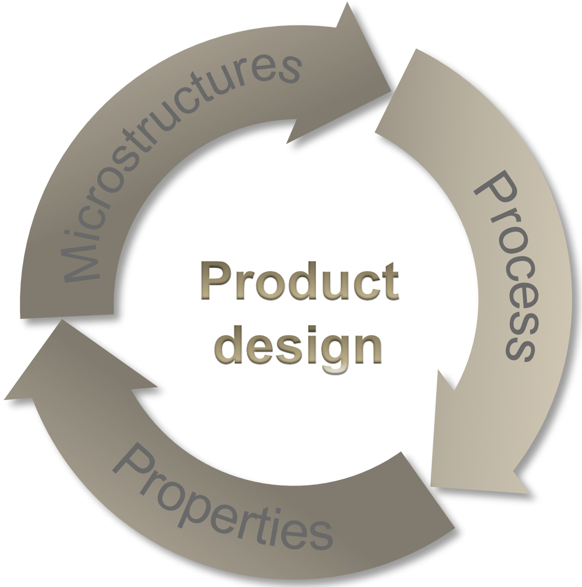
Concluding remarks



- Product design is based on the thorough understanding of the relationships between microstructure, process and mechanical properties
- Work starts from observations…
  - … Using more or less recent techniques
  - … and often crossing various complementary techniques

⇨ to **identify** damage and fracture mechanisms and **quantify** their intensity and evolution

⇨ to give **guidelines for alloy design**

⇨ to feed models to **predict** damage and fracture properties