Characterisation of phases in equilibrium in quaternary Fe-C-Mn-Al duplex steels

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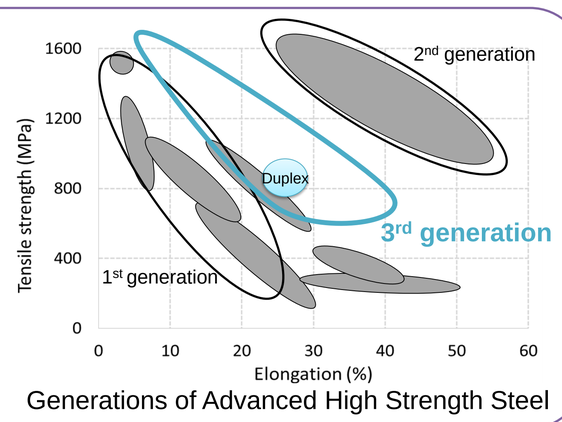
Context

A lightening of automotive weight contribute to CO_2 emission reduction while improving the mechanical properties
⇨ solution : 3rd generation of Fe-C-Mn-Al

Challenge: The knowledge of the corresponding quaternary phase diagram serves as a roadmap in the choice of the compositions and the optimisation of the elaboration conditions

Requirement: An accurate characterisation of the phases in equilibrium, typically ferrite (α), austenite (γ) and carbide $(Fe,Mn)_3AlC$ (κ) ⇨ obtain a thermodynamic database reliable and precise

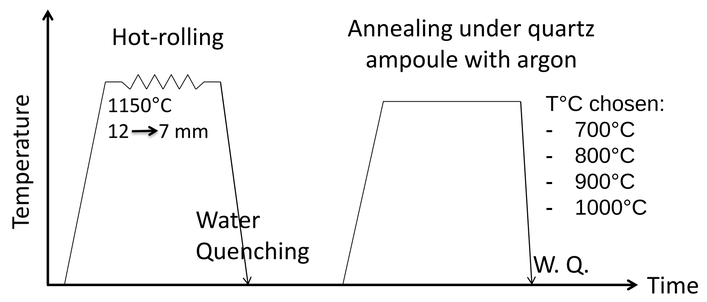


Generations of Advanced High Strength Steel

Experimental procedure

➢ **Preparation of specimens**

- Heat treatments : to obtain the two-phase or three-phase alloys at equilibrium



Characterisation methods

- X-Ray Diffraction: identify crystallographic nature of the phases
- Scanning Electron Microscopy: microstructure and identification of phases
- Electron probe microanalysis: composition of each phase
- Image analysis: phase fraction

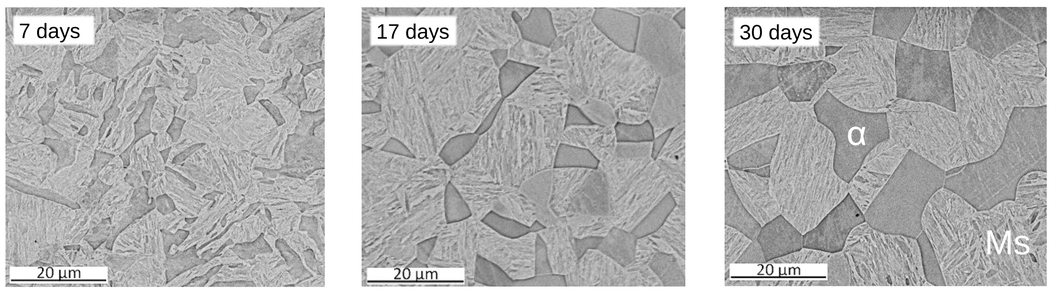
➢ **Calibration of EPMA**

Al & Mn : Calibrated using high purity references

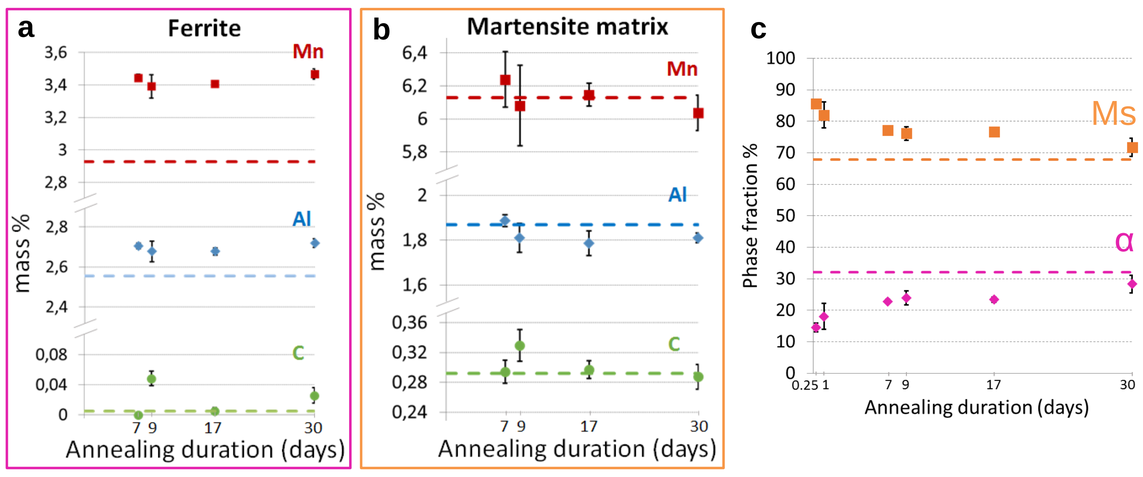
Fe : To 100% of the other elements analyzed

C : Specific methodology [1]
- Trap with liquid nitrogen } reduce C contamination
- Oxygen micro-leak } reduce C contamination
- Calibration curve : different references of carbon steels (0.2 to 1 mass%C)

Composition evolution with annealing duration



Microstructure SEM-BSE Fe-0.2C-5Mn-2Al annealing **at 800°C** – Nital 2%, observation perpendicular to the rolling direction



Evolution of ferrite phase (a) and martensite matrix (b) composition and phase fraction (c) of Fe-0.2C-5Mn-2Al alloy as a function of annealing duration **at 800°C.** Experimental data (symbols) in comparison with the calculated values [2] (dotted lines)

⇩

17 days minimum annealing duration for the thermodynamic equilibrium (stabilization of the composition and phase fractions)

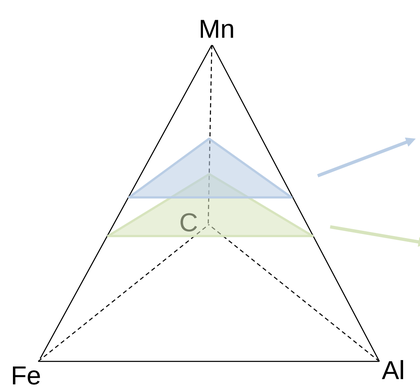
Results : example of phase equilibria at 800°C

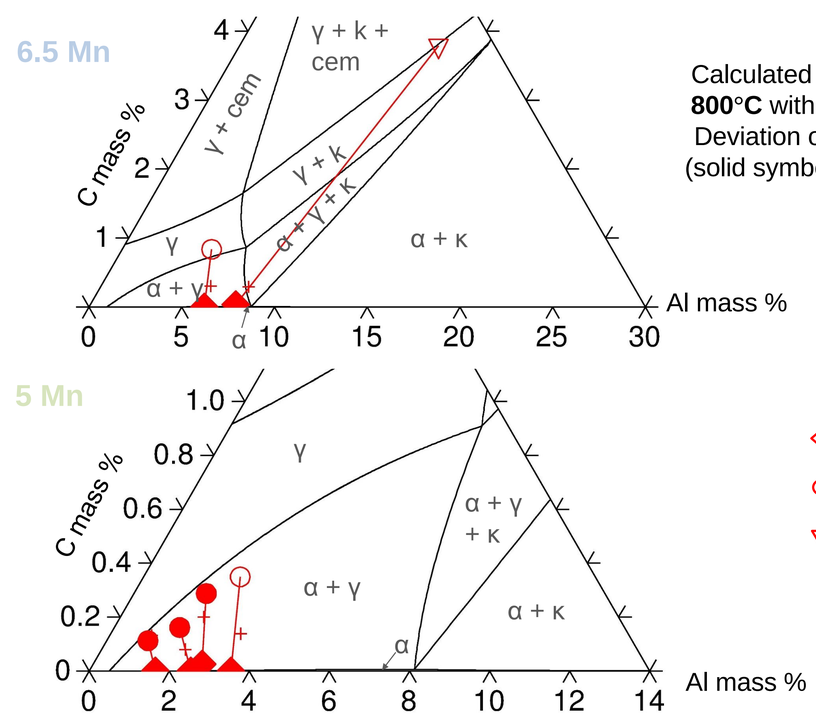
➢ **Isothermal section at 6.5 mass% Mn**

Two phases α + κ are evidenced in the 0.3C-6.5Mn-8Al alloy whereas the calculation provides the presence of a tri-phase domain α + γ + κ

➢ **Isothermal section at 5 mass% Mn**

The experimental boundary γ / α + γ area is slightly lower than the calculated data





Calculated isothermal sections [2] **at 800°C** with experimental data in red. Deviation of Mn content within ± 2% (solid symbol), larger than ±2% (open symbol)

+ Nominal composition
◇◆ α - ferrite
○● γ - austenite
▽▼ κ - carbide

Conclusions and perspectives

- Good measurement accuracy for the phase composition
- Determination of α /γ, α /k and α /γ/k boundaries between 700 and 1000°C
- Location of the domain stability of γ and κ

Next steps : Optimization of a thermodynamic database of the Fe-C-Mn-Al system

[1] Robaut F, Crisci A, Durand-Charre M and Jouanne D 2006 *Microsc. Microanal.* **12** 331-334
[2] Database PrecHiMn-03, coll. B. Hallstedt, RWTH Aachen University