

# Characterization and simulation of carbide precipitation during tempering of low silicon AISI H11 hot-work tool steel



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## INTRODUCTION

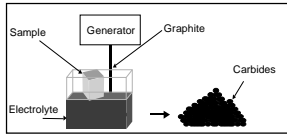
Hot-work tool steels withstand severe and complex thermo-mechanical loading in service. Generally, heat treatment is performed to obtain a good hardness and/or tensile strength with a required ductility. The microstructure parameter controlling well-balanced mechanical strength/toughness properties is the secondary precipitation and more precisely the nanometric carbides formed during tempering. The precipitation of secondary carbides during tempering of low silicon AISI H11 hot-work tool steel is investigated by transmission electron microscopy observations, X-ray diffraction and small angle neutron scattering. The experimental results are compared with results obtained by modelling precipitation sequence using a new model developed at Graz University for nucleation, growth and coarsening in multi-component multi-phase systems.

## EXPERIMENTAL METHODS

⇨ Low silicon AISI H11 : 0,35%C, 0,28%Si, 0,35%Mn, 0,06%Ni, 5,11%Cr, 1,21%Mo, 0,47%V

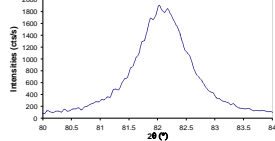
### X-Ray Diffraction

Carbides extracted by dissolution of matrix



Chemical nature of carbides

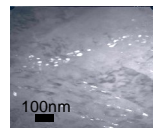
WHM and WAM methods based on X-ray peaks profile



Dislocation density

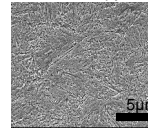
### Electronic Microscopy

Transmission Electron Microscopy



Possible carbides  
Nucleation sites

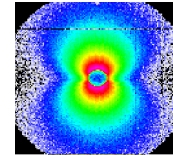
Scanning Electron Microscopy



Grain size  
Subgrain size

### Small Angle Neutron Scattering

Adjustment of the intensities diffused using a model with double distribution of size of spherical precipitates  
Scattering vector range : 0.05 to 1.6 nm<sup>-1</sup>

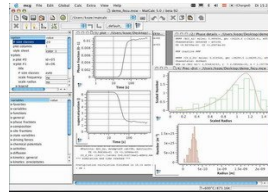


Volume fraction of carbides  
Average radii of carbides

Input parameters

### Thermodynamic and precipitation kinetics modelling

- Calculations of precipitation sequences during heat treatment in multi-component multi-phase systems with MatCalc
- Based on mean field approximation: Onsager principle



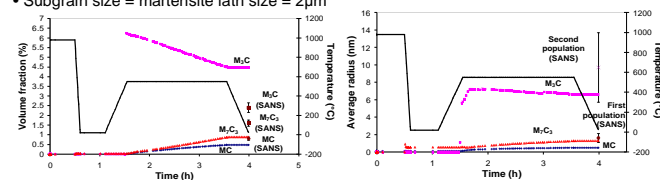
## RESULTS

### Microstructure and precipitation

- ⇨ Two populations of secondary carbides were identified by small angle neutron scattering :
- Small carbides with a radius around 1.6 nm ± 0.5nm : M<sub>7</sub>C<sub>3</sub> and MC.
- Larger carbides with a radius 9.7 nm ± 4nm : M<sub>3</sub>C and M<sub>7</sub>C<sub>3</sub>.

### Simulation

- ⇨ Input parameters :
- Dislocation density:  $\rho=3.10^{15} \text{ m}^{-2}$
- Grain size = primary austenitic grain size = 10µm
- Subgrain size = martensite lath size = 2µm



⇨ The tempering occurs in two steps:

- Rapid precipitation of paraequilibrium M<sub>3</sub>C at the beginning of tempering : the depletion of matrix in carbon exactly follows the increase of M<sub>3</sub>C volume fraction.
- Then dissolution of M<sub>3</sub>C which releases its carbon to allow the precipitation of M<sub>7</sub>C<sub>3</sub> and VC.

⇨ Comparison between calculation and experimental results :

- Same evolution of both first (MC and M<sub>7</sub>C<sub>3</sub>) and second population (M<sub>3</sub>C).
- Significant discrepancies between volume fraction and average radii measured by SANS and obtained by modelling.

## CONCLUSION

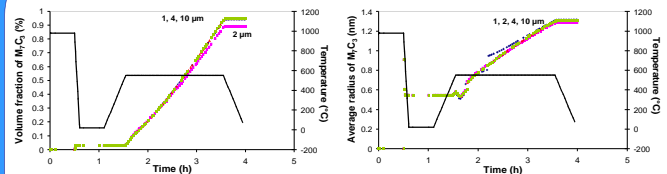
⇨ Discrepancies between experiments and simulation results can be due to an inaccurate determination of input parameters : dislocation density, shape of carbides, interfacial energy carbide/matrix.

⇨ The value of subgrain grain size has only a slight influence on precipitation of carbides.

⇨ A variation of dislocation density has a significant influence on volume fraction and radii of carbides. Low accuracy in the dislocation density determination (factor of 10) induces scattered simulation results.

## INFLUENCE OF INPUT PARAMETERS

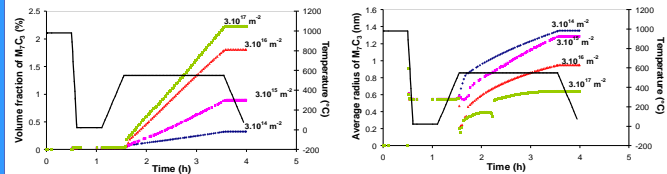
### Subgrain size



⇨ Slight influence on volume fraction and radii of carbides.

⇨ Measurement by scanning electron microscopy : 2µm is adopted for simulation.

### Dislocation density



⇨ The increase of dislocation density raises the volume fraction of M<sub>7</sub>C<sub>3</sub> carbides, and consequently enhances the dissolution of M<sub>3</sub>C .

⇨ Low accuracy in the dislocation density determination (factor of 10) induces scattered simulation results.

## FUTURE WORK

⇨ Volume fraction and radius of M<sub>3</sub>C have to be determined by small angle neutron scattering considering needle shaped carbides.

⇨ Improve thermodynamic database in order to better calculate the interfacial energy carbide/matrix.

⇨ With the up to date experimental techniques dislocations density can be adapted by a factor of 10.

⇨ Improve the simulation of MC carbides precipitation : volume fraction as well as radius are still under evaluated.