EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

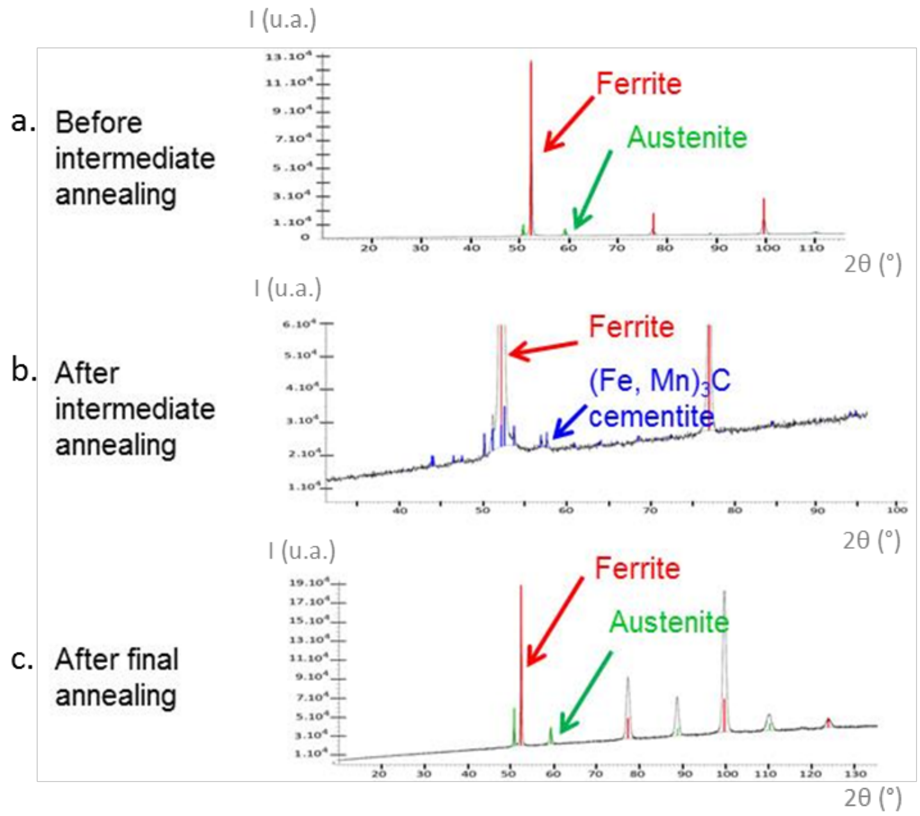
- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** HEXRD study of the effect of microalloying on phase transformations in medium Mn 3rd generation high strength steels	**Experiment number**: MA3387
Beamline: ID11	**Date of experiment**: from: 20 / 07 / 2017 to: 24 / 07 / 2017	**Date of report**: Feb 28 2018
Shifts: 12	**Local contact(s)**: Thomas Buslaps	*Received at ESRF:*
Names and affiliations of applicants (* indicates experimentalists): Alexis Deschamps, SIMAP – Grenoble INP Hugo Van Landeghem, SIMAP Zelie Tournoud, SIMAP Patricia Donnadieu, SIMAP		

Report:

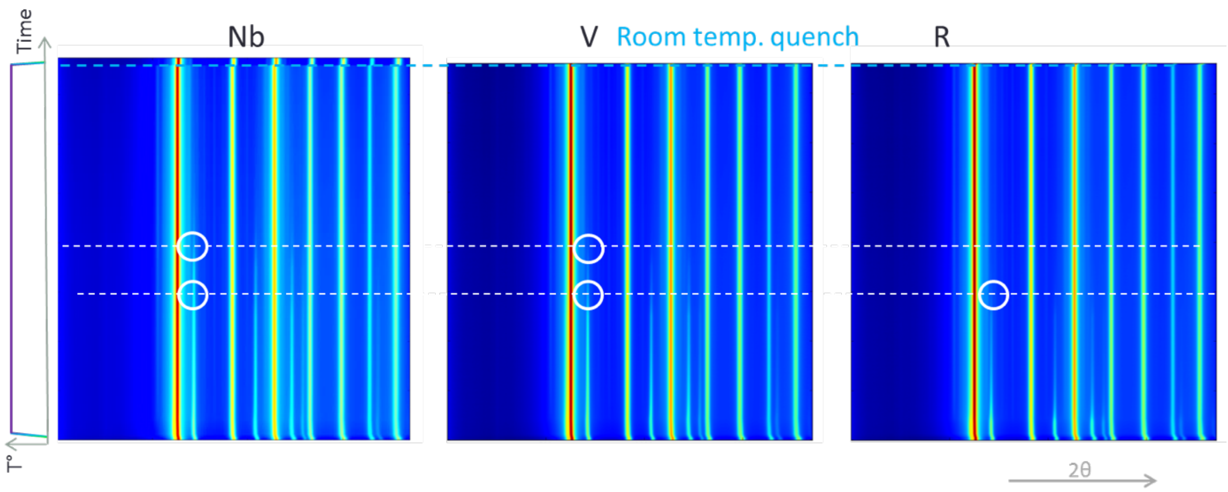
This experiment aimed at characterizing in-situ the phase transformtions occuring in 3rd generation advanced high strength steels of the “carbide-free bainite” type, based on the Fe-Si-Mn-C system, and more specifically to describe the effect of microalloying additions on the phase transformation kinetics. For this purpose, three steels have been studied, with a constant Fe-Si-Mn-C basis, and respective addition of V and Nb microalloying. These steels have been subjected in-situ to a first heat treatment of isothermal (softening) annealing, and subsequetly to an austenitization treatment at 950°C, followed by rapid and controlled cooling to the bainite formation temperature (350, 400 or 450°C) and holding at this temperature. In parallel, samples heat treated ex-situ have been evaluated as well to compare the in-situ results with heat treatment cycles more relevant to industrial conditions.

Overall the experiments have been very successful, with a very consistent performance of the ETMT heating device enabling to perform precise and reproducible heat treatments. Most conditions that were initially considered haven been successfully measured, providing for the first time a complete viw of the effect of microalloying on the phase transformations in these systems. The interpretation of the results is still under way, however, it will clearly result in a significant part of the PhD thesis of Zelie Tournoud (co-proposer) and several publications.



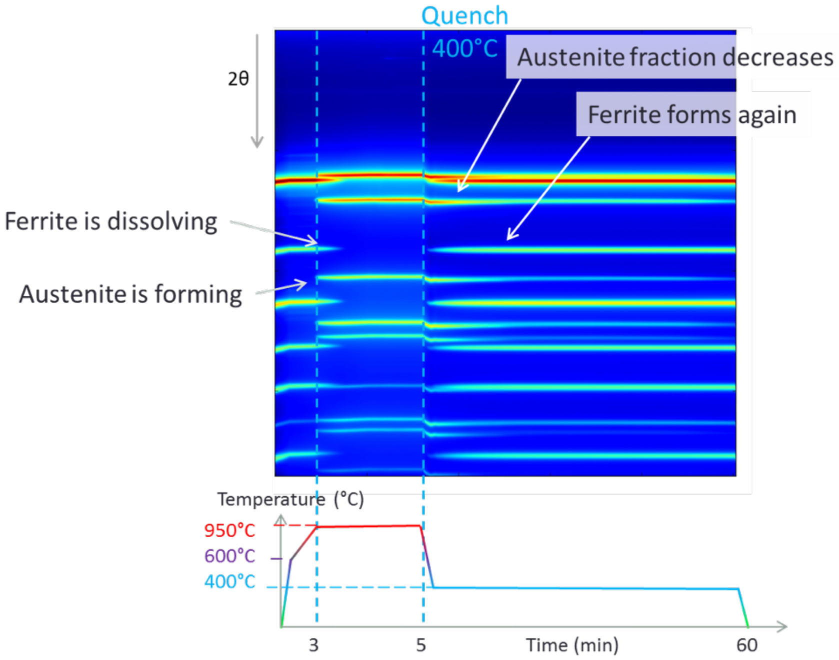
The figure above shows characteristic diffractograms in the initial state, after the intermediate annealing and after the final heat treatment. The intermediate annealing results in the disappearance of austenite and the formation of cementite. During the final annealing, carbide-free bainite formation occurs together with the carbon enrichment of the remaning austenite. The main parameters that are important to follow from an aplication point of view are the fraction of residual austenite and its carbon content, which can be accessed through the austenite lattice parameter evolution.

1. Phase transformations during annealing



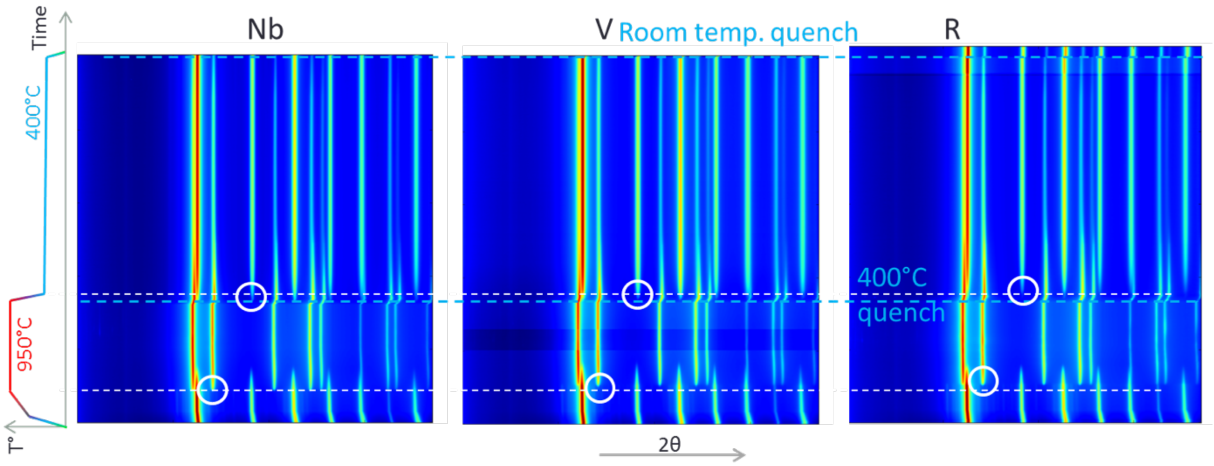
The effect of microalloying on intermediate annealing can be observed in the figure above which plots as a color map the diffarctograms as a fucntion of annealing time at 550°C. The white circles denileate the characteristic times for dissolution of austenite. Clearly, this process is strongly slowed by the microalloying addition.

2. Phase transformations during final heat treatment



The above graph shows qualitatively the evolution of phase transformations during the different steps of the final annealing. During austenitization at 950°C, ferrite quickly dissolves and a fully austenitic structure is obtained. Immediately after quenching (to 400°C in the presented case), ferrite nucleates and quickly grows, while the residual fraction of austenite stabilizes.

Qualitatively, the effect of microalloying can be observed in the diffractograms / time maps:



The processes are observed to be similar in the three materials with however some small differences linked to the microalloying content. In order to appreciate better these differences, the diffractograms have been fitted with the Fullprof package to extract variations of phase fractions and lattice parameter.
While we have performed experiments at three annealing temperatures (350, 400 and 450°C), and for the three materials, the folllowing data is presented only for one case, Nb, at the three temperatures.
The fraction of remaining austenite is observed to decrease initially very fast and then more slowly, reaching of the order of 10% at the end of the experiments. Its evolution is strongly correlated to the redistribution of carbon since carbon stabilizes the remainng austenite. The carbon content of austenite can be estimated from the ratio of lattice parameters of austenite and ferrite. Initially it is observed to increased very fast, which represents the carbon enrichment of austenite, and then to decrease, despite the fraction of austenite continuing to decrease. This effect must be due to carbide precipitation, which actually has been observed by SAXS in a parallel experiment performed on BM02. It limits the amount of enrichment of austenite and

therefore these results are very important findings from a practical point of view since the stability of austenite is the main factor controlling the end properties of the steel.