Experimental report

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Proposal: **1-01-177**

Council: 4/2019

Title: Investigation of secondary precipitation during tempering of a nano-bainitic steel

Research area: Materials

This proposal is a new proposal

Main proposer: **Steve GAUDEZ**

Experimental team: Juan MACCHI
Imed-Eddine BENRABAH
Frederic DE GEUSER
Steve GAUDEZ
Guillaume GEANDIER
MURIEL BRACCINI
Lucie JACQUET

Local contacts: Ralf SCHWEINS
Nina-Juliane STEINKE
Lionel PORCAR

Samples: Fe-0.67C-1.5Si-2.5Mn-1.25Cr-1Mo-0.5V wt.%

Instrument	Requested days	Allocated days	From	To
D11	2	2	15/01/2020	17/01/2020
D22	2	0		
D33	2	0		

Abstract:

The proposed experiment aims at investigating the precipitation of secondary carbides (M2C, VC) during the tempering of nanobainitic steels. Nano-structured bainite is composed of nano-scaled laths of ferrite (20-60 nm thick) and a high proportion of untransformed austenite (15-40%), which impart very high strength and excellent ductility to these steels.To our knowledge, secondary precipitation inside a nanobainitic microstructure has scarcely been investigated so far, and SANS has never been used during nanobainite tempering. The experiments will consist in studying the secondary precipitation inside an initial microstructure of nanobainite, on samples subjected to continuous heating and tempering treatments in our laboratory. In addition, initial microstructures of martensite will be also considered prior to tempering and will serve as a basis to analyze and understand. The results will serve in developing and validating a metallurgical model of precipitation which takes into account the concomitant evolution of the matrix phases (bainitic ferrite and retained austenite), precipitation carbides but also the mechanical properties prediction (yield strength).

Investigation of secondary precipitation during tempering of a nano-bainitic steel

The proposed experiment aims at investigating the precipitation of secondary carbides of VC during the tempering of nanobainitic steels. Prior studies on these steels characterized the bainitic transformation by HEXRD and SANS. Nano-structured bainite is composed of nano-scaled laths of ferrite and a high proportion of retained austenite, which impart very high strength and excellent ductility to these steels. This microstructure is obtained after long heat treatment (16h to many days) in a temperature range from 150 to 350°C. In the current project, we attempt for the first time to combine nano-structured bainite with secondary precipitation for high temperature applications. To our knowledge, secondary precipitation inside a nanobainitic microstructure has scarcely been investigated so far, and SANS has never been used during nanobainite tempering.

The aim of the observations at ILL were to quantify the precipitation kinetics of VC carbides as well as, if possible, the cementite after interrupted tempering treatment with various time and temperature conditions.

The sample investigated for the experiment were taken from forged ingot with a composition of Fe-0.67C-1.3Mn-1.8Cr-1.7Si-1Mo-0.5V wt.%. In addition to the nano-bainite initial microstructure, a full martensite and martensite + retained austenite were also considered prior to tempering. These more conventional cases will serve as a basis to better analyze the more complex microstructure evolutions in the case of nanostructured bainite. After transformations, the initial microstructures were tempered in a temperature range from 450 °C to 650 °C after a heating at 5°C/s up to two hours.

For the ILL experiments the samples were 40 mm x 10 mm with 3 mm thickness. A magnetic field of 1.2 Tesla was used to saturate the matrix, in order to contrast with the non-magnetic precipitates. Three different distances from the detector were studied in order to scan as much q (meaning as many radius values) as possible: 2, 5, and 20 m.

Figure 1 shows examples of diffusion image of sample at the three detector distances obtained for the martensite initial microstructure tempered at 550°C during one hour after. The results were very satisfying, showing a great contrast between matrix and precipitates as shown by the signal given figure 1. On Figure 1 we can also observe that the signal is not symmetric due to the magnetic component of the matrix. In the following, both the nuclear and magnetic components were fitted simultaneously with two kinds of precipitate with a Schultz distribution in order to deduced the mean particle sizes and volume fractions.

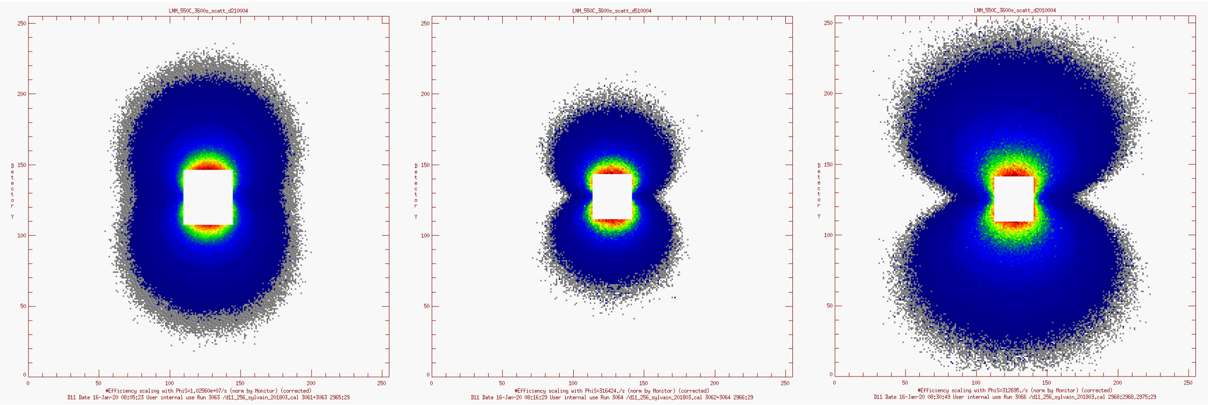

Figure 1: Diffusion image of sample at a detector distance of 2, 5 and 20 m. Martensite sample tempered at 550°C during one hour after a heating at 5°C/s.

Figure 2 shows the mean radius of the MC carbides deduced from the analysis of the SANS experiments for the three initial microstructures after a tempering at 450, 550 and 650°C during one hour. As expected, the higher the temperature the higher the mean radius. In addition, similar tendency and mean radius is observed between the three initial microstructures. Nevertheless, the SANS mean radii are under estimated compared to TEM, which could come from the volume analyzed by TEM compared to the SANS one and the difficulties to observed very small carbides especially when they are coherent with the matrix.

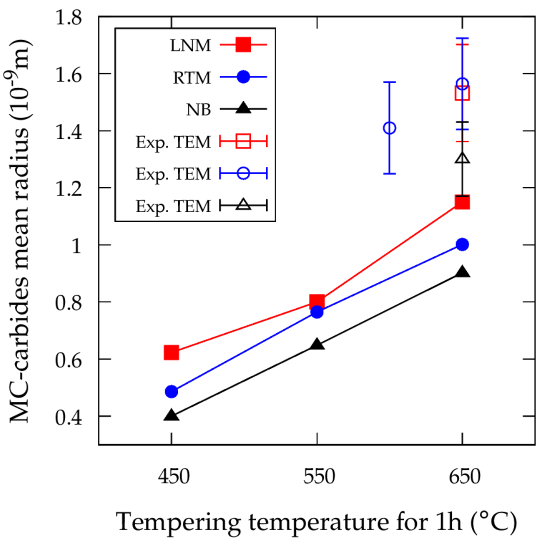

Figure 2: Mean radius deduced from the analysis of SANS experiments for samples tempered one hour at 450, 550 and 650°C. The label exp. stands for TEM experiments.

The SANS experiments performed at ILL were brought an important amount of new data to the study, which is an important step forward in the modeling of precipitation kinetics and their understanding in nano-bainite.