European Synchrotron Radiation Facility

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ESRF

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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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.

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The experiments perfomed on ID11, were executed with an ETMT machine allowing a controlled heating of small samples ($1.5 \times 1.5 \times 40$ mm) by resistivity and also the application of tensile or compressive stresses, which function has not been used.

Two different kinds of steel were investigated: the case hardening grade 20MnCr5, and the ball bearing grade 100Cr6. The heat treatment cycles were principally similar for all samples: a controlled heating up to a temperature between 700 °C and 1000 °C, followed by a soaking period of 10 to 60 minutes. Afterwards, the samples were cooled down quickly to room temperature (within 20 seconds).

During the entire heat treatment cycles, diffraction frames were recorded with the FRELON camera using an exposition time of 0.4 or 0.8 seconds depending on the velocitity of the ongoing process. The energy of the beam used was 71 keV and the beam size was set at the maximum (about 100µm high and 300 µm width) in order to obtain the best statistical conditions as possible in terms of diffracting domains. The temperature was controlled by the use of two thermocouples welded on the surface of the samples. One type R thermocouple was used for the temperature regulation (imposed by the machine) and one type K thermocouple was used for a more precise temperature measurement in the low temperature range. In order to get the information about the instrumental contribution on the diffraction patterns, a standard material (LaB6 powder) was measured.

The recorded frames were integrated after background correction with the help of an automatic macro file.
The analysis of the diffraction patterns has been performed with the Rietveld refinement software TOPAS© from Bruker AXS. First of all, the LaB6 measurement has been refined in order to receive the instrumental function. Then, some time has been spent on the refinement of one pattern obtained at room temperature after cooling (as it is the most complicated microstructure). Several models of peak broadening were tested, including anisotropic crystallite size and strain broadening as well as the effect of stacking faults on the broadening, in order to obtain the most reliable fit.

During the evaluation of the best refinement the best fit could only be observed with a four phase structure composed by non-transformed austenite (retained austenite) non dissolved cementite and two modifications

of the martensite phase. Indeed, if the fit of the austenite (fcc structure) was quite good, the refinement with only the expected martensitic structure (bct) could not give a reliable fit of the peaks. Only with the addition of an additional phase (bcc martensite) it was possible to obtain a nearly perfect fit of the data.

In literature, authors mostly refer to "martensite with bcc or bct structure", but never give a clear statement. It actually seems that the question of the martensite structure is still not clear although martensitic steels are widely used for industrial applications.

The extension of the developed refinement model to the high temperature data allowed the description of the transformation kinetics and also give a possible explanation for the presence of two different martensite modifications at room temperature.

Figure 1 shows the phase fraction changes during quenching of one sample made from 100Cr6. The sample was first heated up to 880 °C and hold for 10 minutes. As it can be seen, the transformation of austenite (fcc) to martensite (bct) begins at the temperature of about 170 °C. The cubic martensite (bcc) appears at a temperature slightly lower. The austenite content decreases consitnuously while the phase fractions of bct and bcc martensite increase. The bcc martensite reaches its maximum content at a temperature of about 90 °C while the content of bct martensite still increases strongly. Non-dissolved cementite still remains in the sample but its content do not change during quenching.
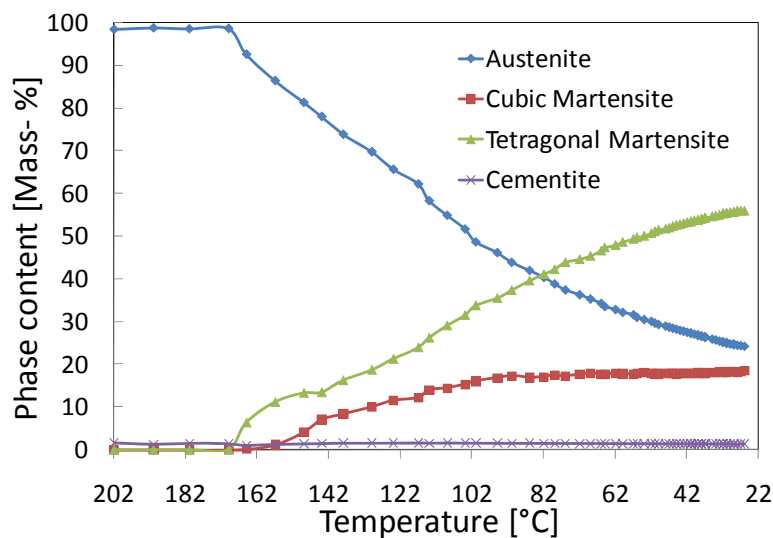

Figure 1: Phase content evolution during quenching of a sample from 880 °C to room temperature.

The reported behaviour led to some of the following assumptions and conclusions:

First of all, bct martensite forms from the austenite. Immediately after the transformation, some regions of the newly formed martensite reach a more stable energetic state by ejection of carbon atoms out of the lattice and its structure becomes more or less cubic. Further on, transformation of austenite into bct martensite continues with decreasing temperature, but the thermal energy for the carbon movement also decreases, so the transformation rate of bct to bcc is approachuing zero at the temperature of 90 °C. One other reason for this is that almost all defects available for carbon movement may already be filled by carbon atoms.

As found in [1], the possibility that carbon atoms are trapped along the dislocation line is possible during tempering as the density of dislocations in martensitic is very high (about 10^{12} cm^{-2}) [1]. As self-tempering effects can occur during quenching, it is possible that similar phenomena happen.

However, these are only assumptions and further experiments will be needed to clarify some points and confirm these tendencies.

[1] M. Maalekian, E. Kozeschnik : A thermodynamic model for carbon trapping in lattice defects. Computer Coupling of Phase Diagrams and Thermochemistry 32 (2008) 650 - 654