

**Experiment title:**

Investigation of self-tempering and tempering effects as well as phase specific densities during heat treatment of different steel grades

Experiment number:

MA-965

Beamline: ID11	Date of experiment: from: 22.10.10 to: 25.10.10	Date of report: 10.12.11
Shifts: 9	Local contact(s): Dr. Loredana Erra	<i>Received at ESRF:</i>

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Report:

The experiments performed on ID11, were executed with an ETMT machine allowing a controlled heating of small samples (40mm in length) by resistivity and also the application of tensile or compressive stresses, which function has not been used. In order to reach different cooling curves, three different samples' cross sections were used: $1 \times 1\text{mm}$, $1.5 \times 1.5\text{mm}$ and $2 \times 2\text{mm}$.

Two different kinds of steel were investigated: the case hardening grade 20MnCr5 (as cast and after spray forming), 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 950 °C with 5K/s, followed by a soaking period of 15 minutes. Afterwards, the samples were cooled down quickly to room temperature. During the entire heat treatment cycles, diffraction frames were recorded with the FRELON camera using an exposition time of 0.4 seconds. The energy of the beam used was 70 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 a thermocouple welded at the surface of the samples.

In order to get the information about the instrumental contribution on the diffraction patterns, standard material (LaB6 powder) was measured. As the samples's thickness also contributes to the broadening, 3 different LaB6 samples were prepared with thicknesses of 1, 1.5 and 2mm.

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 in a similar way as for previous experiment (MA-574). The refinement was done with a four phase structure composed by non-transformed austenite (retained austenite) non dissolved cementite and two modifications of the martensitic phase: the expected tetragonal martensitic structure (bct) and an additional cubic phase (bcc martensite).

In the previous experiment (MA-574), it was postulated that first 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 approaching zero. As reported in the experimental report of (MA-574), carbon atoms can be trapped along the dislocation during tempering as the density of dislocations in martensitic is very high (about 10^{12} cm^{-2}). As self-tempering effects can occur during quenching, similar phenomena are expected to happen.

Variations of austenitizing temperature of 100Cr6 steel, lead to a varying amount of dissolved carbide, and so to a different carbon content in solution in the austenite at high temperature. This will directly affect the Martensite start temperature which decreases with increasing carbon content in solution. Results of experiments performed with an austenitizing temperature of 825 °C (a) and 940 °C (b) are presented in Fig. 1.

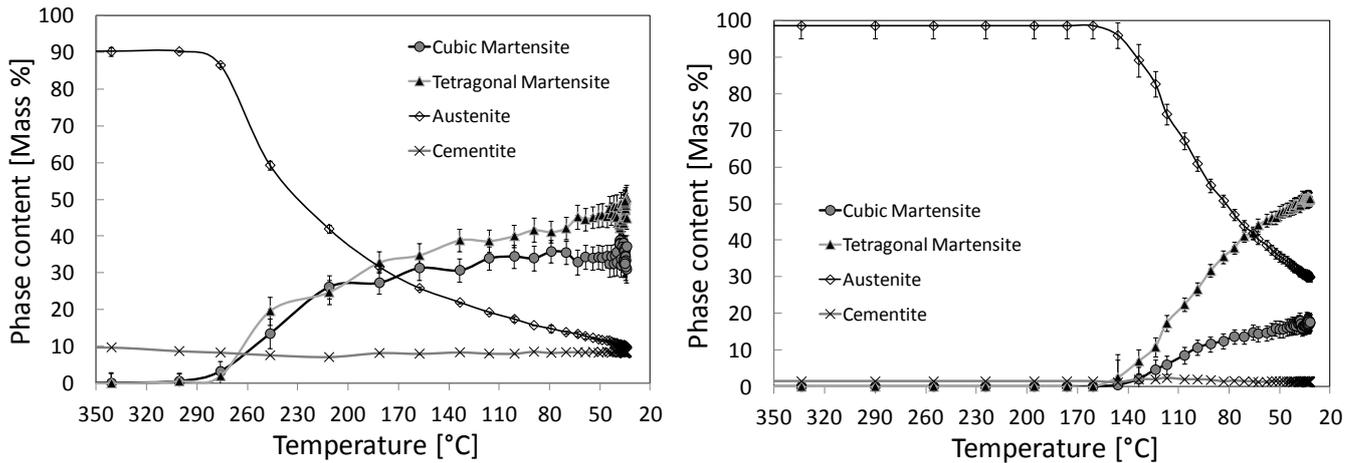


Figure 1: Evolution of phase contents during quenching of 100Cr6 samples with a thickness of 1.5 mm after austenitizing at different temperatures for 15 minutes: a) 825 °C; b) 940 °C

It can be noticed that beside the much lower Martensite start temperature for higher austenitizing, the amount of formed cubic and tetragonal martensite seem also to depend on the austenitizing temperature, and so on the carbon content in solution.

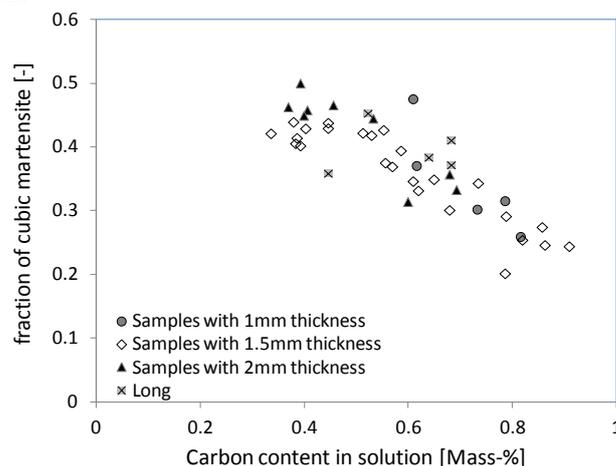


Figure 2: Fraction of martensite that has a cubic structure at room temperature as a function of the carbon content in solution

The results presented in Figure 2, show clearly that an almost linear dependence of the fraction of cubic martensite over the carbon content in solution is present. However, no significant influence of the samples' thickness, and so of the cooling rate, seem to be present. This might indicate, that the process of self-tempering occurs mainly in a very short time period and that longer times do not lead to significantly larger effects.

Investigations of several samples with the method of atom probe tomography are ongoing to show the carbon distribution within the microstructure.