	Experiment title:	Experiment number:
	Measurement of carbon content in steel martensite	01-01-656
Beamline: **BM01B**	**Date of experiment**: from: 1.3.04 to: 5.3.04	**Date of report**: 14.02.05
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Report:

The objective of this experiment is the quantitative analysis of the carbon content in the martensite of a high carbon steel. Our group is studying the relation between the mechanical properties and thermal treatment of carbon steels used for the fabrication of files. The project is supported by the Commission for Technology and Innovation and a Swiss company: Usines Metallurgiques Vallorbe, which is a major European file manufacturer. Tempering at different temperatures leads to the reduction of the carbon present in solution and to the precipitation of different carbides. Several precipitation stages are observed as a function of tempering temperature [1]. In previous experiments, it was determined that the measurement of thermoelectric power and the measurement of internal friction at room temperature give a rather precise assessment of relative carbon content variation in the martensite. However, a quantitative absolute measurement of the carbon content is missing. This experiments aims at supporting those data with the measurement of the variation of the lattice parameter of the martensite. X-ray diffraction is the only method to measure quantitatively the amount of carbon dissolved in the martensite due to the proportionality between the tetragonal distortion of the iron lattice and the carbon content. The steel used in this experiment contains 1.23wt%C and 3wt%Cr. Chromium is completely precipitated into carbides. Samples of thickness 0.2 mm are extracted from bulk martensitic samples and are aged for 16 months to have an asymptotically stable carbon content. Each sample is cumulatively tempered for 25 min at temperatures increasing by 25 K from 300 to 800 K. Samples are measured in transmission with a beam wavelength of 0.35 Å.

Another experiment aimed at analysing the carbon content at the file tooth of two types of files, which underwent different thermal treatments. One type is quenched after annealing in a cyanide salt bath and the other is quenched after annealing in a conveyor belt oven. The mechanical performances of the two types of files are different. However, up to now no analysis did show any significant difference from the point of view of the microstructure and chemical composition. In this experiment, the sample were analysed at grazing incidence. Knowing the depth of penetration of X-rays, a maximum volume 30 μm deep from the tooth tip is analysed (see fig. 1).

Experimental results

In fig. 2, part of the spectrum evidencing the peak {110}/{011} of the martensite is shown. The peak strongly sharpens with tempering, so that the reduction in peak width is followed by the increase of the peak height. Furthermore, the sharpening and later on the increase of the cementite peaks is observed (for example at 2θ = 9.99 º). For a better comparison of the peak broadening, the peak intensity is normalized in fig. 2. The peak remains unchanged by tempering up to 350 K, and the first small change is observed after tempering at 375 K. The sharp peak obtained after annealing at 800 K is the {110} of the ferrite. The concentration of carbon in solid solution in the martensitic matrix is calculated by fitting the martensite double peaks ({110}/{011}, {200}/{002}, {211}/{112} and {220}/{022}) using an iterative procedure. The calculation of the d-spacing is based on Kurdjumov equations [2] that describe the change of the lattice parameters depending on carbon concentration:

$$c = a_0 + 0.116 \cdot C(wt\%) \qquad a = a_0 - 0.013 \cdot C(wt\%)$$

The data points calculated for {110}/{011} peak are shown in fig. 3.

The experiment at grazing angle did show that cyanide treated sample produce a carbon enrichment at the surface of the file (0.47wt%) while the treatment in belt oven produces a carbon depletion (0.36wt%). This result has been correlated with the variation of mechanical performances of the respective files.

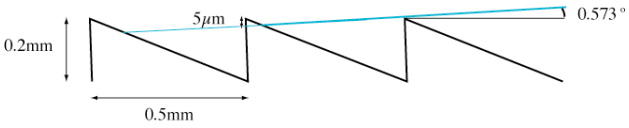

Fig. 1 Scheme of the experiment at grazing angle. Incidence angle is magnified for clarity.

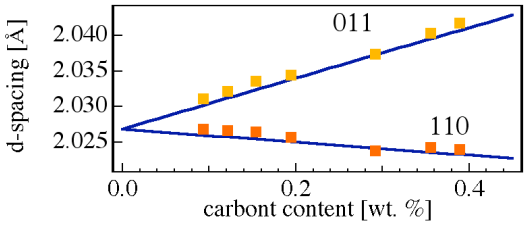

Fig. 3 Comparison of fitted d-spacing for {110} and {011} peaks with those calculated as a function of carbon concentration

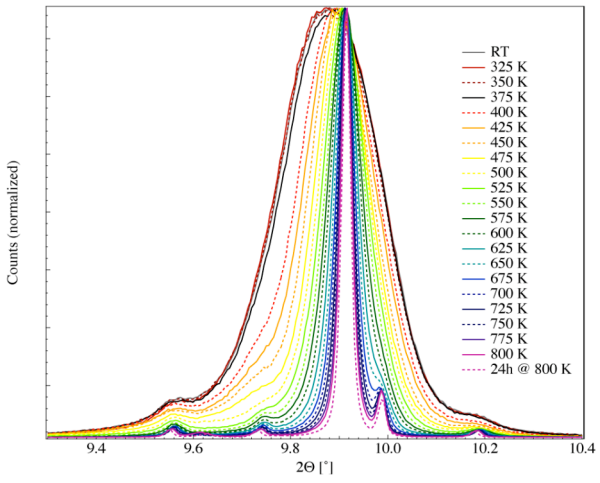

Fig. 2 Effect of tempering on the {110}/{011} peak of martensite

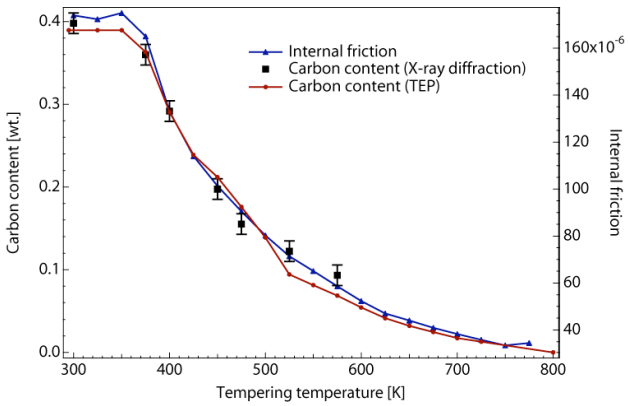

Fig. 4 Correlation between internal friction measured at room temperature, carbon content derived from room temperature thermoelectric power measurements and actual carbon content derived from x-ray measurements. All data are plotted as a function of tempering temperature.

References:

[1] I. Tkalcec, C. Azcoitia, S. Crevoiserat, D. Mari, Materials Science and Engineering A, 387-389 (2004) 352-356.
[2] G. V. Kurdjumov, Metallurgical Transactions A, 7 (1976) 999-1011.