Quantitative carbon determination in low-carbon steels by wavelength-dispersive X-ray spectroscopy (WDS)

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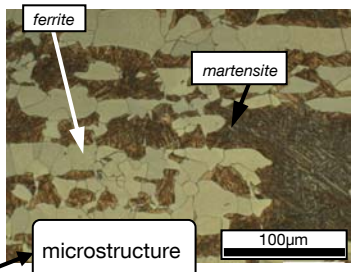
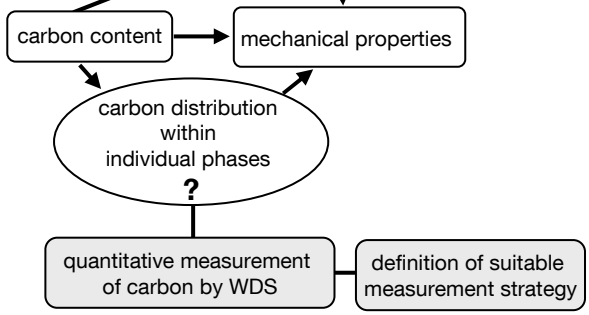
Introduction

Low-carbon steels show excellent properties and therefore are used for many industrial applications.

The investigation of the micro-structure and distribution of the individual alloying elements is essential for the under-standing and the optimization of the mechanical properties of low-carbon steels.



Dual phase steel; etching according to Le Pera



Experiments

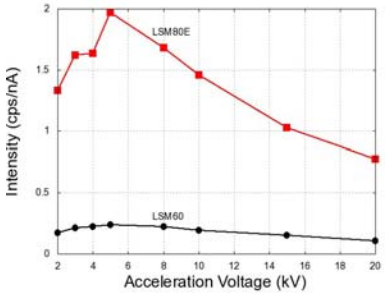
Carbon in low-carbon steel was measured with the field-emission scanning electron microscope JEOL JSM 7600F equipped with a full-focusing wavelength-dispersive spectrometer using the crystal LSM60 and LSM80E (layered synthetic material with lattice spacing around 60 and 80Å).

Light element-analysis: problems and solutions

high absorption	peak shift	carbon contamination
• definition of the optimum acceleration voltage • choice of the best suitable crystal (LSM60 or LSM80E)	• measurement of the peak position for every to-be measured sample to ensure the maximum count rate	• oil-free vacuum system • liquid nitrogen cooling trap • standard preparation method without organic solvents and diamond pastes • high probe current (to increase also the yield) • use of ferrite as internal standard material

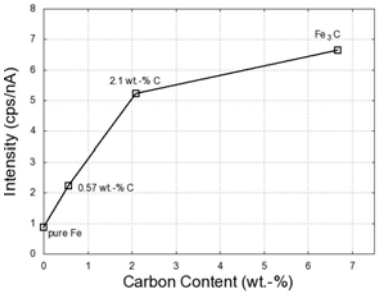
Evaluation of coarse grained cementite Fe_3C (6.67 wt.-% C) and three more standards with 0.00, 0.57 and 2.10 wt.-% C for the generation of a calibration curve for quantification!

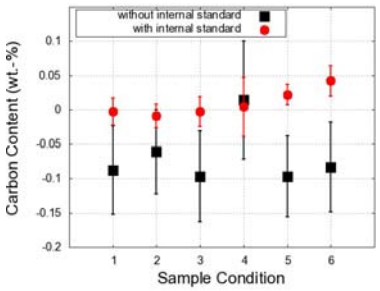
Results



Comparison between LSM60 and LSM80E: Highest detectable intensities for carbon in steel with 0.7 wt.-% C could be obtained by the use of LSM80E at 5 kV.

Measured calibration curve for an acceleration voltage of 5 kV generated by using three standard materials (pure iron, 0.57 and 2.1 wt.-% carbon) and cementite (Fe_3C, 6.67 wt.-% carbon). By the use of an internal standard the calibration curve can be shifted through the origin.





Detected carbon content with and without the use of an internal standard after the defined standard preparation method (1-3), after additional purging with ethanol (4), after additional storage for 15h under vacuum in the SEM (5), after additional storage of 30 min. in labratory atmosphere (6).

Conclusions

For carbon measurement the crystal LSM80E is recommendable due to its lower absorption. The optimum acceleration voltage for the quantification of carbon was detected to be 5 kV. Cementite (Fe_3C) was found out to be not a suitable standard material for the detection of low carbon contents within an iron-matrix due to its high absorption of the C Kα-radiation. By the use of an internal standard the variation in the carbon contamination within one individual sample as well as between different samples and the standard materials can be widely eliminated. A standard preparation method without the use of organic solvents and diamond pastes for the last polishing step is recommendable. Sample storage should be avoided.

Acknowledgments

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