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Using EELS-Carbon Measurement to Predict Hardness of V-added DP Steels

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In this paper we present a method based on EELS spectroscopy to accurately measure the amount of carbon within steel microstructure. We use a combination of second derivative and second difference to improve the signal to noise, so that carbon edge can be detected even at low concentrations.

The effect of vanadium microalloying on ultra-high strength dual phase (DP) ferrite-martensite steel microstructure was studied using TEM. It was found that the addition of 0.14%wt V to a Fe-0.18C-1.5Mn-0.3Si-0.008N reference alloy introduced very significant ferrite and martensite grain refinement in the cold rolled and annealed state [1]. Image analysis of TEM foils and direct and indirect extraction replicas after cold rolling and intercritical annealing at 735^{0}C intense showed V(C,N) precipitates (mean radius 3.7 nm) were abundant in the ferrite phase whereas precipitates were scarce in martensite (austenite) and much larger (mean radius 6.7 nm), as seen in Fig. 1. Furthermore, a detailed study of martensite islands in high strength DP steels using TEM EELS carbon measurements and nano-indentation was carried out. Figure 2a is a TEM bright field image of a thin foil from the DP-V alloy after intercritical annealing at 735°C/90s followed by water quenching. The martensite islands (darker grains) are between 0.5-1μm in size and several undissolved cementite particles (verified by diffraction) are visible. EELS data were obtained from 6 martensite islands as well as the ferrite and cementite phases indicated in the micrograph. The electron beam was defocused so that the carbon signals were averaged over the largest practical area. Selected second difference EELS spectra showing the doubly-differentiated carbon K-edges at 284 eV can be seen in Figure 2b. The data were acquired from an undissolved cementite particle, two martensite islands with different carbon levels and the ferrite matrix. The detection sensitivity of the EELS analysis can be gauged from a measurement of the total carbon content in the ferrite matrix. For the Osiris/FS1 system we found a value of 0.08 wt.% C. That is 0.06 wt.% higher than the expected solubility limit for carbon in ferrite at the quench temperature (~0.02 wt.% at 735°C). At all annealing temperatures the measured martensite (austenite) volume fractions agree well with the ortho-equilibrium values and the average EELS values for the martensite carbon content are in good accord with the concentrations predicted by the lever rule. However, the scatter in the EELS carbon data increases strongly at lower annealing temperatures. This is in agreement with the work published by Garcia-Junceda et al [2] and is what is expected if partial cementite dissolution influences the martensite carbon content. A direct comparison of the martensite hardness predicted from EELS carbon measurements and the experimental nano-hardness distributions is shown in Figure 3. For DP-Ref the nano-indentation and the EELS data concur on a value for the median martensite strength which agrees well with the continuous composite approach (CCA) stress spectrum [3], derived from the bulk tensile data. The predicted dispersion based on the EELS data can account for <25% of this variation. This implies that mechanisms other than the carbon content can significantly influence martensite island strengths in DP steels. The DP-V steel results are contradictory; EELS carbon analysis predicts that the median martensite strength and dispersion should be very similar to the DP-Ref alloy. This result is consistent with the process thermodynamics. However, both nano-indentation and CCA modelling predict a much lower median martensite strength. There is an independent softening mechanism operating in the DP-V alloy that is not present in DP-Ref. This softening process must be grain-size and/or precipitation dependent. Further work is required to determine the nature and extent of this effect.

References:

[1] C.P. Scott et al., Materials Science and Engineering A **703** (2017), p. 293.
[2] A. García-Junceda et al., Scripta Materialia **57** (2007), p. 89.
[3] S.Y.P. Allain et al., MSEA A **637** (2015), p. 222.
[4] The authors acknowledge funding from Vanitec and ArcelorMittal Dofasco. © Her Majesty the Queen in Right of Canada, as represented by the Minister of Natural Resources, 2019.

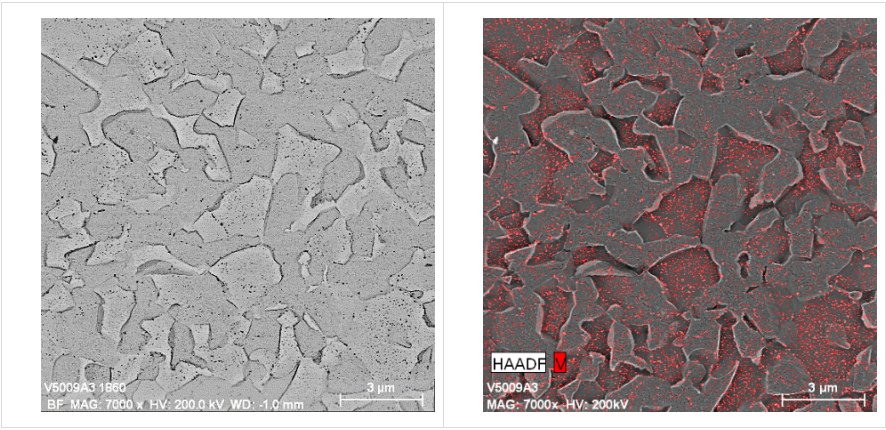

Figure 1. a) BF-STEM image of the direct replica of V-added alloy after annealing at 735^{0}C / 90s and die quenching; and corresponding composite HAADF/EDX chemical map showing V (red).

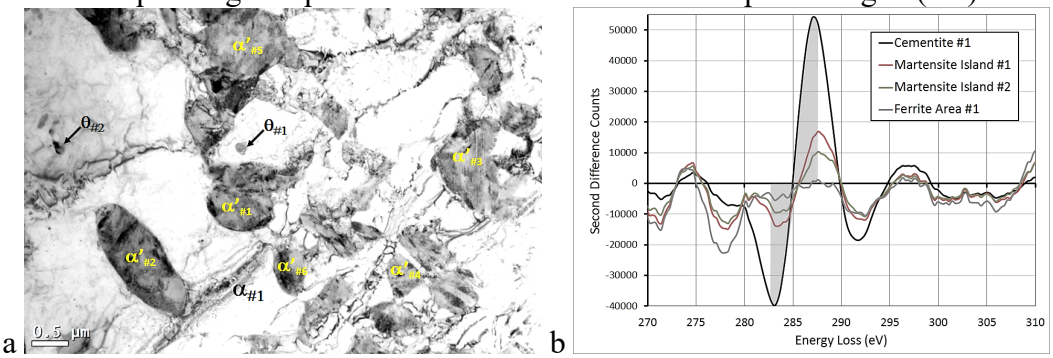

Figure 2. a) Bright field TEM image of DP-V annealed at 735°C/90s showing α, α' and partially dissolved cementite (θ) phases. b*)* Second difference EELS spectra showing C-K edges from selected regions identified in Figure 1a.

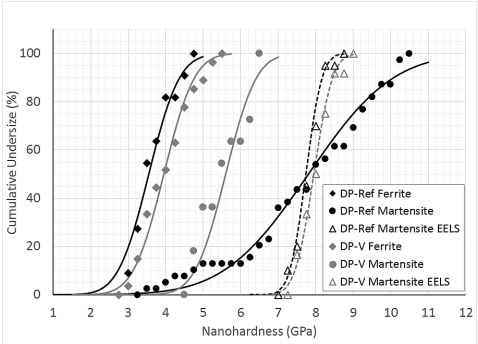

Figure 3. Cumulative undersize distributions of martensite and ferrite island hardness from nano-indentation measurements. The EELS data for martensite is included.

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