TEM study of boron effect on the microstructure of 9CrWTaVB martensitic steel

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Martensitic tempered steels are established high-temperature materials for use in turbines or power plants. The 8-10%Cr steels are also developed for future application in a fusion reactor. The European reduced-activation ferritic-martensitic (RAFM) reference material EUROFER 97 [1] was alloyed with 83 and 1160 wt.ppm ^{10}B to study the influence of He on the mechanical properties and microstructure of irradiated material [2]. Helium will be generated inside the material by the nuclear reaction $^{10}B(n, \alpha)^{7}Li$ during neutron irradiation. Alloying of steels even with a low boron concentration influences distribution of precipitates and hence the steel microstructure. TEM characterisation of microstructural changes, new precipitate types and their location is an important step towards understanding the boron effects on steel properties.

The precipitates were studied using combined electron energy loss spectroscopy (EELS) and energy-dispersive X-ray analysis (EDX) spectroscopic imaging. EELS spectroscopy conducted in the transmission electron microscope (TEM) represents a powerful tool not only for the detection of light elements, such as boron or nitrogen, but also for determination of chemical composition and, in some materials with orientation dependence of ionization edges like BN, its crystallographic orientation to the electron beam. The chemical phase of precipitates was determined by a combination of analytical results with electron diffraction.

The combined EDX-EELS elemental mapping of specimens with different boron contents shows the distribution of different precipitates in the specimen (Figs. 1 and 2). Altogether, four types of precipitates have been detected: $M_{23}C_6$ precipitates are well visible in the Cr maps, TaC and VN particles are visible in the Ta and V maps, respectively. EELS maps show the formation of nitrides or carbides in the specimen (N and C maps in Figs. 1-2). The boron nitride precipitates of 300nm-500nm size are distributed randomly in the specimen (Fig. 3). Detailed analysis shows that the precipitates are surrounded by the TaC and VN precipitates.

VN precipitates located on the grain and lath boundaries (Figs. 1e,g) in specimens with 83 wt. ppm boron contents and EUROFER 97 without boron [3] were not detected in the specimen with 1160 wt. ppm boron (Figs. 2e,g). Instead of VN, the significantly higher amount of BN precipitates was detected. Based on this result, it may be assumed that nitrogen in the alloy was gettered by boron for formation of BN phase. This phase shows the lower free formation energy at high temperature than VN. The deficiency of nitrogen prevents the formation of VN precipitates on the lath boundaries and, hence, influences the formation and stabilisation of the lath structure. The average lath width increases from 350nm in specimen without boron to 570 nm in the specimen with 1160 wt. ppm boron. In the specimen with 1160 wt. ppm boron the new precipitate type with $(Ta_{0.89}V_{0.11})C$ composition and cubic structure was detected (Figs. 2e,f,g). The preferred location of this precipitate type on the lath boundaries assumes its influence on the stabilisation of lath structure.

The TEM investigations of 9%CrWVTa steel specimens with different boron contents show that high boron concentrations prevent VN precipitation, which results in a lower stability of the lath structure at higher temperature [2].

References

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[2] E. Materna-Morris *et al.* Journal of Nuclear Materials 386-388 (2009) **422**
[3] M. Klimenkov *et al*. Progress in Nuclear Energy 57 (2012) **8**

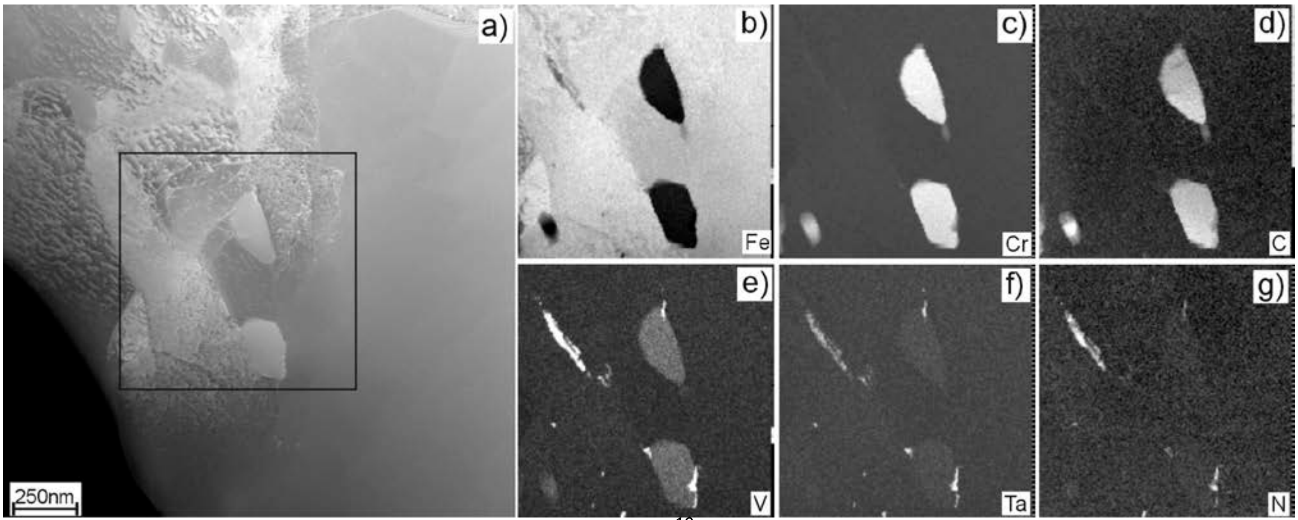

Figure 1. *EDX-EELS mapping of the steel with 83 appm ^{10}B. (a) HAADF image of the investigated area, (b) – (g) Fe, Cr, C, V, Ta, N elemental maps.*

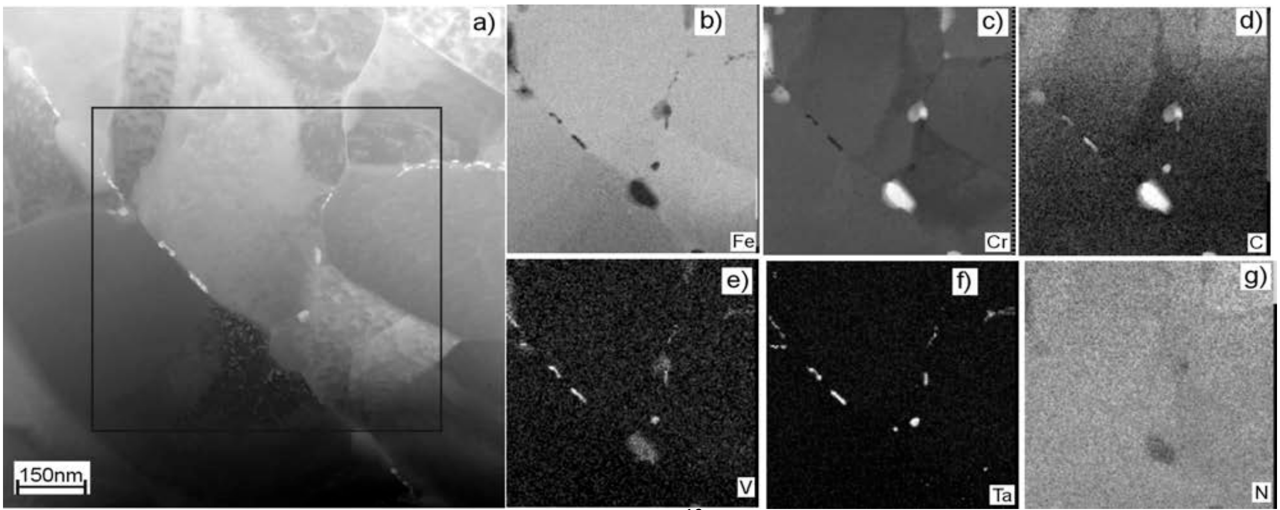

Figure 2 *EDX-EELS mapping of the steel with 1160 appm ^{10}B. (a) HAADF image of the investigated area, (b) – (g) Fe, Cr, C, V, Ta, N elemental maps.*

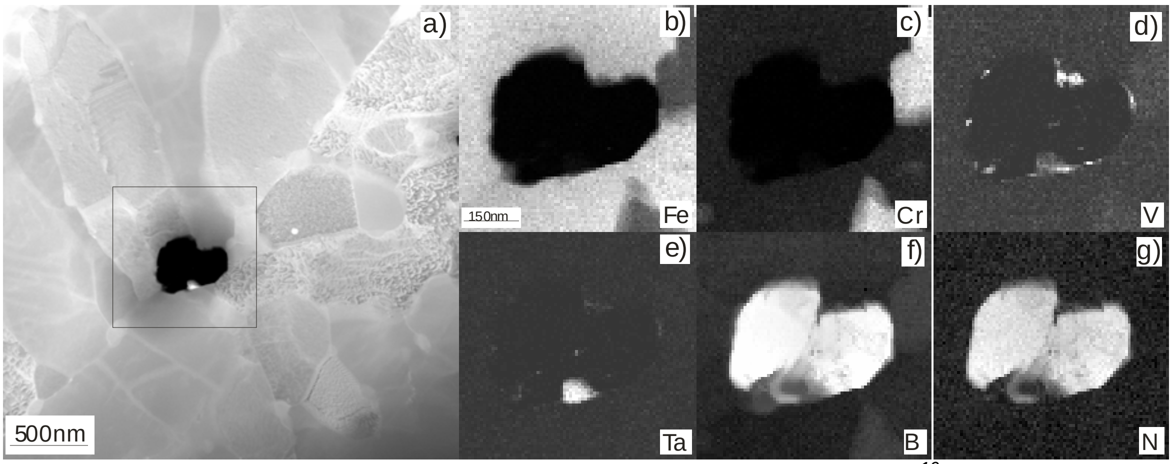

Figure 3 *EDX-EELS mapping of a BN precipitate in the steel with 1160 appm ^{10}B. (a) HAADF image of the investigated area, (b) – (g) Fe, Cr, V, Ta, B, N elemental maps.*