Microstructure evaluation of 12 % Cr steel using TEM technique.

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High creep resistance after long service time is a crucial parameter for martensitic 9-12% Cr steels which have to resist high temperatures and pressures in service at fossil power plants [1]. Changes in the microstructure during service deteriorate the creep properties and decrease their service life. Their microstructure is characterized by prior austenite grains boundaries, martensite laths and subgrains, with high dislocation density [2] and precipitates distributed in the matrix, at boundaries and on dislocations. According to the backstress concept [3] each precipitate phase contributes to the creep strength of the material. However during creep and aging the microstructure evolves. New phases precipitate, like mod. Z-phase, which dissolves MX precipitates and does not contribute considerably to the creep strength of the material [4]. The understanding of this microstructure evolution in these materials is necessary to develop new materials with improved creep properties.

Within this work, the microstructure of seven material conditions of a 12% Cr steel (different creep test temperature and stresses) was investigated by TEM, energy filtered TEM (EFTEM), electron loss spectroscopy (EELS) and X-ray spectroscopy (EDXS). ELNES as finger print method together with electron diffraction have been used for the identification of complex nitrides [5]. Furthermore, two different sample preparations were used: extraction replicas for elemental composition determination and ion milled specimens for particle quantification.

In the as-received condition (after heat treatment before service), the material microstructure showed a tempered martensite matrix, with small amounts of MX (Nb,V)(C,N), $M_{23}C_6$ and M_2X (Cr_2N), see figure 1. During creep and aging the precipitates nucleated, grew and coarsened, and two new phases formed, identified as Laves phase, an intermetallic phase $(W,Mo)_2Fe$ and mod. Z-phase (VCrN), see figure 1 and 2. The main contribution to the material strength is dedicated to fine dispersed precipitates like MX (Nb,V)(C,N). However, due to precipitation of mod. Z-phase, a reduction of the phase fraction of the MX precipitates was found after 24000h at 600°C of aging. Nevertheless, Cr_2N precipitates were increasing their phase fraction at the same temperature being unaffected by the formation of mod. Z-phase, and thus, indicating the possibility to use this phase for the strengthening of this material. $M_{23}C_6$ carbides showed an increase in the mean diameter, when the aging temperature was increased from 575 to 625°C, precipitating mainly at the boundaries.

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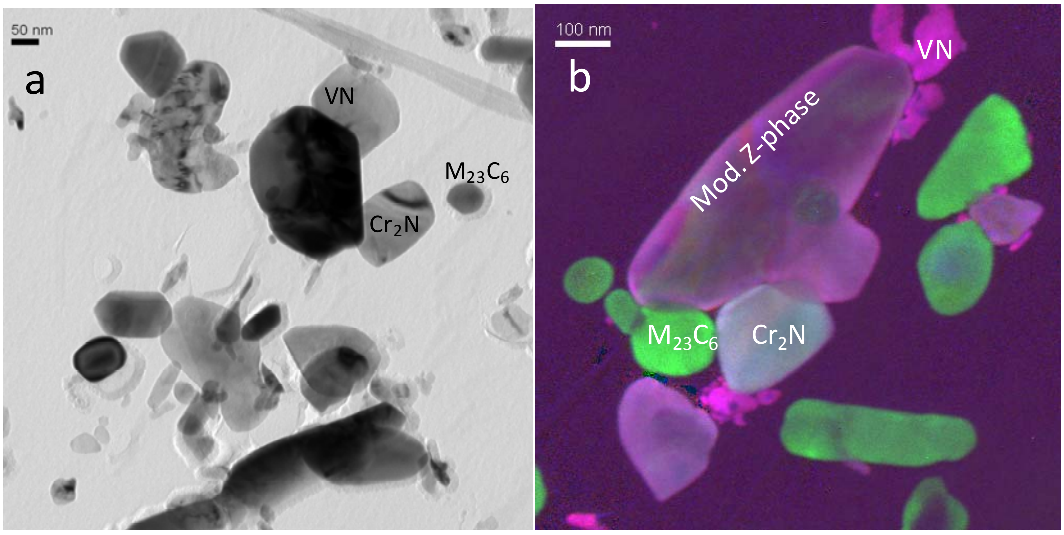

Figure 1. a) Bright Field image of a 12 % Cr steel extraction replica sample in as-received condition. b) RGB map for an extraction replica of the same material after creep exposed at 625°C and 90MPa.

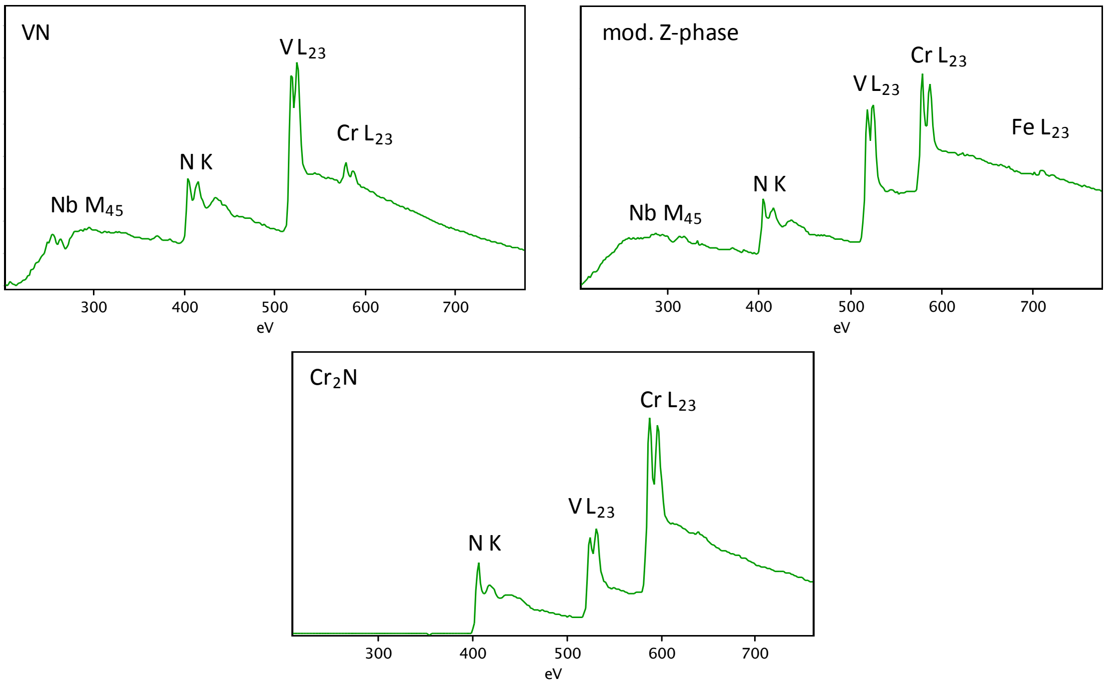

Figure 2. EEL spectra from three different complex nitride precipitates, MX (NbV)N, $(Cr,V)_2N$ and mod. Z-phase (VCrN).

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