VM12 Strengthening by Precipitates - TEM Study of Precipitates in the Martensitic VM12 Steel

Martensitic VM12 steel was recently developed for advanced coal-fired power stations. Its creep resistance is dependent on a stability of microstructure. Destabilization of microstructure is caused by recovery and softening processes of a tempered martensite and depends on changes of a dislocation substructure and morphology of secondary particles during creep. Quantitative TEM analyses of VM12 steel were undertaken to determine the microstructure parameters after creep at 625°C up to 30,000h.

Experimental

The microstructure of the VM12 steel was characterized by X-ray diffraction, light microscopy (LM), scanning (SEM) and analytical transmission electron microscopy (TEM). TEM investigations were performed on thin foils prepared by conventional jet polishing and double extraction replicas. Phase identification was done by electron diffraction (SAED) and STEM-EDS. The diffraction patterns were interpreted with the JEMS software [1]. LM and TEM image analyses were performed using AnalySIS program.

Tempering of the VM12 steel (as received condition) at 780°C for 2 h resulted in a high dislocation density (6.57±1,414 m⁻²) and fine sub-grain microstructure (0.6±0.2 μm), as seen on figure 1a. The M₂₃C₆ carbides (mean diameter: 110±36 nm) and MX carbonitrides (mean diameter: 32±9 nm) were main strengthening particles of the steel. High temperature, long exposure time and stresses caused changes in VM12 steel microstructure during creep. Typical VM12 microstructure after creep deformation at 625°C for 28,983 h is shown in figure 1b. Well developed sub-grains (size increased to 1.4±0.2 μm) with low dislocation density (decreased to 1.2±0.814 m⁻²) are characteristic microstructure features of long-term exposed specimens.

Results

The size, morphology and distribution of precipitates have changed during creep deformation, what influenced creep strength. The M₂₃C₆ carbides increased
rapidly; their mean diameter is two times larger after creep for about 15,000-17,500 h, in comparison with as received condition.

These particles are very important for stabilization of sub-grain microstructure during creep (fig. 1c). Fine MX particles precipitated intergranually (fig. 2a). During creep at 625°C, the mean diameter of MX particles first increased (until 3,000 h of creep) to 41±17 nm and then slowly decreased with prolongation of creep duration (22±9 nm); simultaneously a density of MX carbonitrides decreased. It indicates that MX particles are thermodynamically unstable and during long-term exposure dissolved. Further microstructural TEM investigations revealed precipitation of a complex Cr(V,Nb)N nitrides (fig. 2b), called Z-phase [2].

Large Z-phase precipitates were found frequently in contact with VX precipitates, which might acted as nucleation sites for Z-phase (fig. 2b) [3]. After-creep during 15,000 h, only very few particles were observed. The density of these particles increased with prolongation of test duration. Beside Z-phase, prolongation of creep test resulted also in precipitation of much larger particles of intermetallic Laves phase, influencing a matrix solution hardening. Figure 2c shows coarse (up to 1 μm in diameter) Laves phase particle precipitated in the specimen deformed for 17,500 h. The precipitation of the Laves phase removed Mo and W from the matrix solid solution and the strengthening of the matrix decreased.

**Conclusion**

The formation of the large particles of the Z-phase during creep exposure causes a faster creep strength degradation because its formation consumes MX precipitates. Coarsening of secondary precipitates, dissolution of MX and precipitation of large Laves- and Z phase particles decrease precipitation hardening and influence increased recovery of the VM12 steel matrix.

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