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# Degradation of the creep resistance of a Re-containing 10%Cr steel upon creep testing at low applied stress

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**Abstract.** New Re-containing steel is a promising material for steam blades of fossil power plants worked at ultra-supercritical parameters of the steam. This steel was subjected to creep testing at 650°C under the applied stresses ranging from 200 to 100 MPa. The steel exhibits excellent creep resistance at high applied stresses; the time to rupture comprises 10,987 hours after the creep test at 650°C/140 MPa. However, a dramatic drop in the creep strength is observed at low applied stresses. A possible reason for this decrease in the creep resistance may be a significant coarsening of the Laves phase particles.

## 1. Introduction

Creep resistant 9-12%Cr martensitic steels are widely used as materials for fossil power plants [1]. The main reason for the high creep resistance of these 9-12%Cr steels is the slowing down the transformation of the tempered martensite lath structure (TMLS) into the subgrain structure under short-term and long-term creep conditions [2,3]. The growth of martensitic laths is accompanied by the coarsening of the Laves phase particles that is one of the reasons for the decrease in creep resistance [2-5]. Fine Laves phase particles diminish the creep rate at the primary creep stage, whereas the coarsening of the Laves phase particles provides an increase in the creep rate at the tertiary creep stage [4,5]. Suppression of the Laves phase coarsening shifts the onset of the acceleration creep stage to longer time that increases the creep rupture time. However, this task is very complicated. The addition of Co reduces the solubility of W atoms in ferrite [6,7] that accelerates the growth kinetics of Laves phase [6]. The addition of Re strongly affects the precipitation of Laves phase [8-11]. Thus, alloying with Re, together with an increase in the W content to 3% and a decrease in the Mo content to 0.13% in the 10% Cr-3% Co martensitic steel with low N and high B contents [3,10-12], positively affects the creep resistance at high applied stresses, at which the creep rupture time increases from about 1800 h [12] to 10,987 h [11] for the creep test at 650°C/140 MPa. This increment is related to a low coarsening rate of  $M_{23}C_6$  carbides and the Laves phase particles and the retaining chains of these particles on the lath boundaries up to rupture [11]. However, at lower applied stresses, the creep strength breakdown is observed. The aim of the present work is to consider microstructural aspects of degradation of creep resistance of the martensitic 10% Cr-3% Co-3% W-0.2% Re steel under creep condition of 650°C/120 MPa.

## 2. Experimental procedure

A Re-containing 10% Cr-3% Co-3% W steel with a chemical composition of (in wt.%) Fe (bal.)-0.11C-9.85Cr-3.20Co-2.86W-0.13Mo-0.22Cu-0.03Si-0.14Mn-0.03Ni-0.23V-0.07Nb-0.002N-0.008B-0.17Re was prepared by vacuum induction melting. This steel was normalized at 1050°C for 1 h and



tempered at 770°C for 3 h. Creep tests were carried out at 650°C under the applied stresses ranging from 100 to 200 MPa up to rupture. Under an applied stress of 120 MPa, the creep test was interrupted after achieving strains of 1% (1001 h), 2% (5035 h) and 2.1% (10052 h) corresponding to the transient, steady-state and tertiary creep stage. The structural characterization of the crept samples was carried out using a JEOL-2100 transmission electron microscope (TEM) and a Quanta 600 FEG scanning electron microscope (SEM).

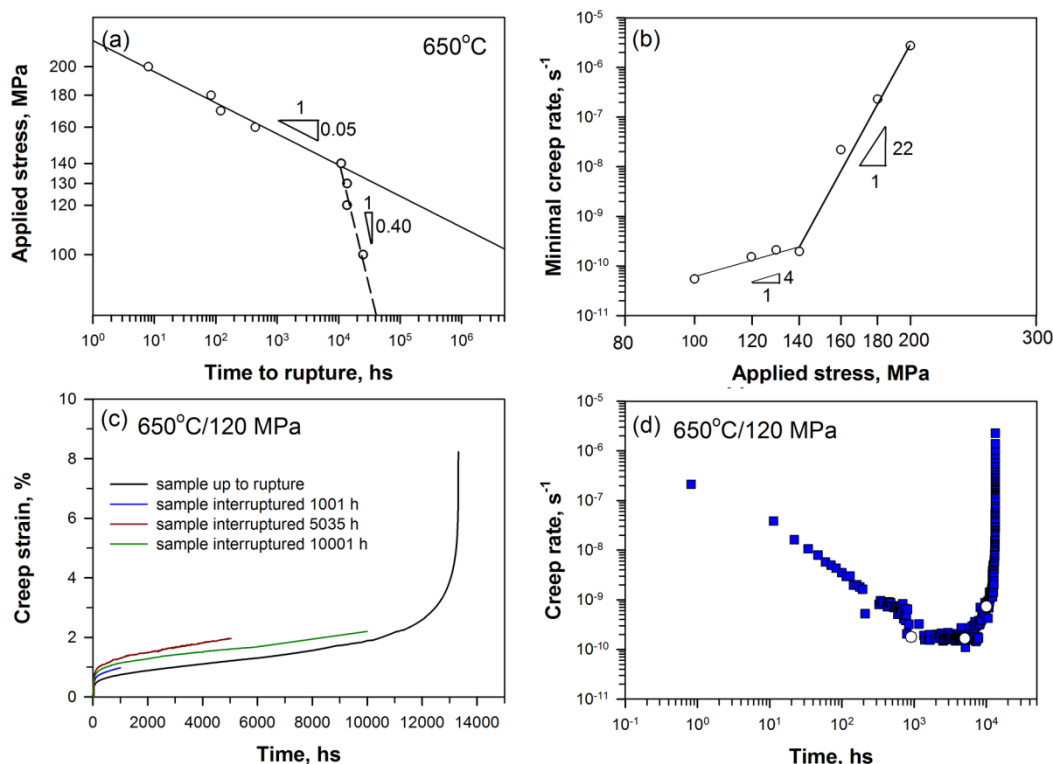
### 3. Results and Discussion

#### 3.1 Tempered state

After heat treatment, the formation of a TMLS with a lath width of 290 nm was revealed in the Re-containing 10% Cr-3% Co-3% W steel [10,11]. A high dislocation density of  $2 \times 10^{14} \text{ m}^{-2}$  was observed inside martensitic laths [10,11]. Cr-rich  $\text{M}_{23}\text{C}_6$  carbides with a mean size of 67 nm and NbX carbonitrides with a mean size of 35 nm precipitated during tempering.

#### 3.2 Creep properties

Figure 1 shows the creep rupture data of the Re-containing 10% Cr steel at a temperature of 650°C.

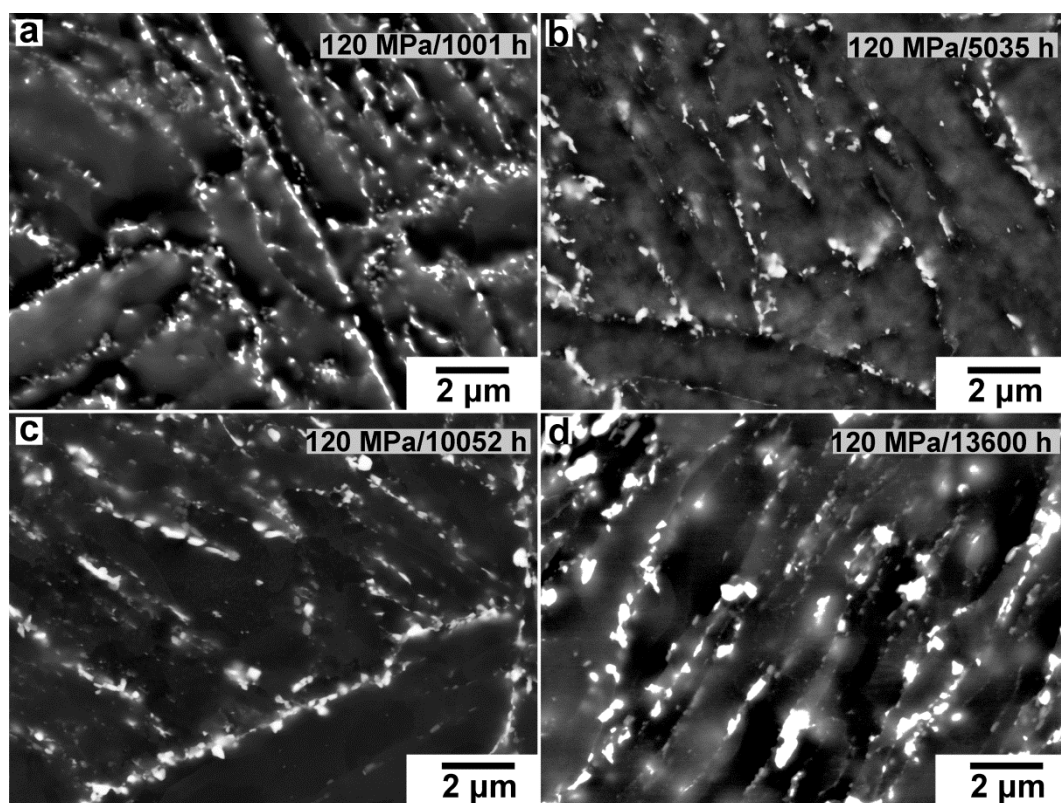


**Figure 1.** Time to rupture vs. stress (a) and minimal creep rate vs. applied stress (b) curves for creep tests at 650°C and applied stresses ranging from 200 to 100 MPa; strain vs. time (c) and creep rate vs. time (d) curves for creep test of 650°C/120 MPa.

Creep strength breakdown (Figure 1a) and power-law breakdown (Figure 1b) appeared at applied stresses below 140 MPa indicating a change in the creep mechanism from dislocation climb in the region of high applied stresses (> 140 MPa) and diffusion creep in the region of low applied stresses (< 140 MPa). The appearance of these breakdowns can be related to the growth of boundary carbides and particles of the Laves phase [13].

### 3.3 Evolution of TMLS during creep

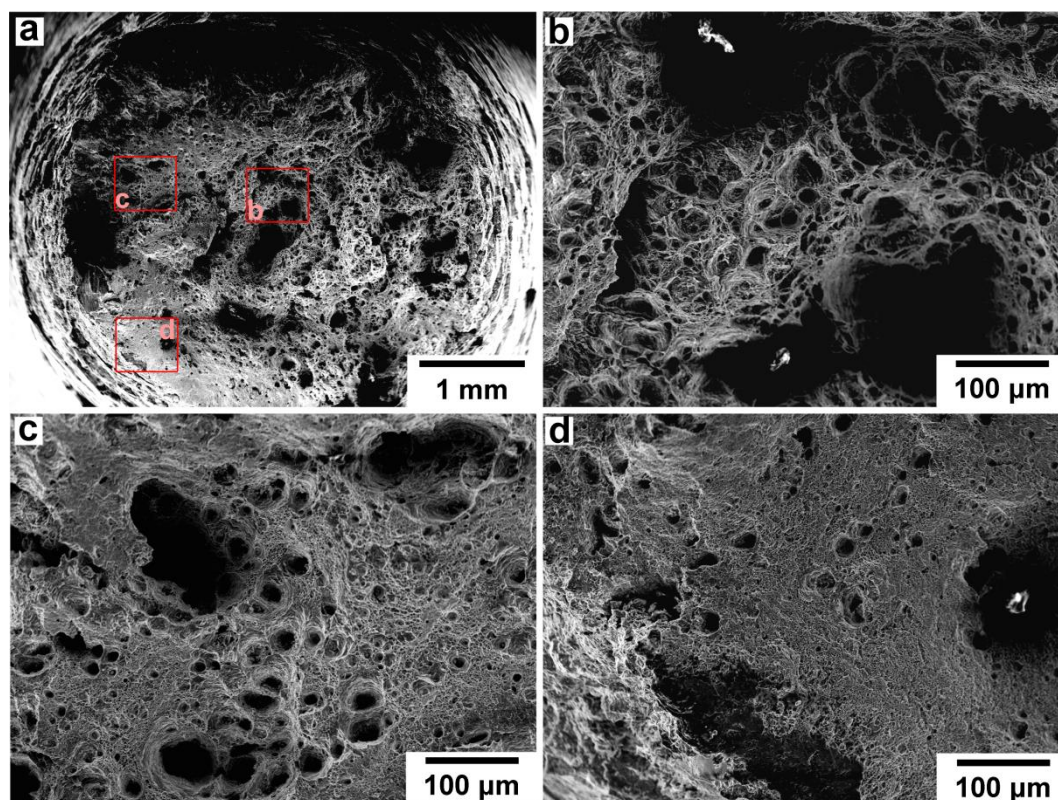
After the creep test at 650°C/120 MPa, the microstructure of the studied steel strongly evolved (Figure 2). The lath width increased to 2.5  $\mu\text{m}$  in the neck section of the ruptured specimen; the TMLS transformed into a subgrain structure with a subgrain size of 1.8  $\mu\text{m}$ . The dislocation density decreased to about  $10^{13} \text{ m}^{-2}$  after 13,600 h of creep testing. W-rich Laves phase particles were observed already after 1001 h of creep testing at 650°C/120 MPa (Figure 2a). This corresponded to the equilibrium volume fraction of 1.6% of the Laves phase in the studied steels [10]. The average chemical composition of this phase was 8%Cr-30%Fe-62%W (in wt%). When the creep time increased, the Laves phase particles grew strongly, whereas the  $\text{M}_{23}\text{C}_6$  carbides and NbX carbonitrides were dimension stable. The mean size of Laves phase in the ruptured specimen was about 250 nm, while very large particles with a size more than 2  $\mu\text{m}$ , randomly distributed in the matrix, were also observed. Their fraction was about 5% of all Laves phase particles.



**Figure 2.** SEM images of the interrupted samples tested at 650°C/120 MPa with different durations.

### 3.4 Fracture mechanism

The fracture surface of specimens tested at an applied stress of 120 MPa is shown in Figure 3. A dimple rupture occurring on the transgranular fracture path was observed. Few very large voids in the center and rims of the ruptured specimen were revealed (Figure 3a). In the center of the ruptured specimen (Figure 3b), there were few nucleation sites, in which the microvoids grew to a large size before coalescing, and this produced a fracture surface that exhibited various dimple sizes. In the rim of the ruptured specimens (Figure 3d), small dimples were formed that was indicative for the activation of numerous nucleating sites; adjacent microvoids coalesced before they started to grow. Large dimples were nucleated by particles located at the bottom of these dimples (Figure 3b,d). These particles were considered to be Laves phase, which served as nucleation sites for coarse cracks.



**Figure 3.** SEM micrographs of the rupture surface of specimens tested at 650°C/120 MPa.

#### 4. Conclusions

A strong degradation of the creep resistance of the Re-containing 10% Cr-3% Co-3% W steel was observed upon creep testing under low applied stresses. Creep strength breakdown and power-law breakdown appeared at applied stresses below 140 MPa. Significant growth of Laves phase is considered to be the reason of this degradation because coarse particles of the Laves phase with sizes of about 2  $\mu\text{m}$  can serve as nucleation sites for large voids and cracks.

#### Acknowledgments

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