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On the effect of long-term creep on the microstructure of a 12% chromium tempered martensite ferritic steel

A. Aghajani, Ch. Somsen, G. Eggeler *

Institut für Werkstoffe, Ruhr-Universität Bochum, D-44780 Bochum, Germany

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Abstract

In the present study we investigate the evolution of the microstructure of a 12% Cr tempered martensite ferritic steel under conditions of long-term aging and creep (823 K, 120 MPa, $t_R = 139{,}971$ h). We show how subgrains coarsen, that the close correlation between carbides and subgrain boundaries loosens during long-term creep and that the frequency of small-angle boundaries increases. All these elementary deformation processes have been discussed in short-term creep studies. The present study shows that they also govern long-term creep. However, during long-term creep, precipitation and coarsening reactions occur that are not observed during short-term creep. Three types of particles ($M_{23}C_6$, VX and Laves-phase) were identified after long-term creep. $M_{23}C_6$ particles coarsen at constant volume fraction and establish their equilibrium concentration after 51,072 h; VX particles are stable; and the Laves-phase particles never reach thermodynamic equilibrium.

Keywords: Long-term creep; Tempered martensite ferritic steels; Microstructure; Subgrains; Carbides

1. Introduction

Nine to twelve per cent Cr tempered martensite ferritic steels are important high-temperature materials which are used for critical components of fossil-fired power plants which operate in the creep range at temperatures between 773 and 823 K. Plant operators expect service lives in excess of 100,000 h and tempered martensite ferritic steels rely on a complex microstructure to fulfill this requirement. Tempered martensite ferritic steels have been the subject of renewed attention due to a need to improve the thermal efficiency of a new generation of fossil-fired power plants. Microstructures govern the creep strength of these materials and therefore it is important to understand the microstructures of tempered martensite ferritic steels, and especially their evolution during long-term creep.

In previous work we have studied dislocation reactions and subgrain coarsening [1], the formation of creep cavities [2], the hierarchy of different types of interfaces [3,4], the role of dislocations during heat treatment and creep [5,6], and the interaction between subgrain boundaries and precipitates [7,8]. We have also given an early account on the influence of long-term creep on the evolution of precipitates in a 12% Cr tempered martensite ferritic steel [9].

Based on our previous experimental results we have suggested microstructural scenarios which contribute to a better understanding of creep of tempered martensite ferritic steels (for a summary see Ref. [7]). These involve a number of elementary deformation and damage processes, including cavitation, dynamic recovery and an associated decrease in dislocation density. They [7] describe the interaction of free dislocations with particles and subgrain boundaries and the interaction between small carbides and subgrain boundaries. They [7] also consider the coarsening of subgrains and carbides. Carbides change their chemical composition and new intermetallic particles (e.g. Laves-phase particles) appear during long-term creep. Material scientists from all over the world (e.g. [10–30]) have worked on creep of 9–12% Cr tempered martensite

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* Corresponding author. Tel.: +49 234 32 23022.
E-mail address: gunther.eggeler@rub.de (G. Eggeler).

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ferritic steels and have provided a number of interesting results, especially concerning the formation of new phases and the coarsening of carbides during long-term creep. Our previous studies [1–9], and some of the results published in the literature, suffer from a number of drawbacks.

First, they often focus on specific elementary processes (e.g. subgrain coarsening) and miss the importance of other microstructural events which are equally important (e.g. creep cavitation). Second, some of the microstructural results were obtained from specimens subjected to accelerated creep tests (like the specimens investigated in Refs. [1–5]). Furthermore, it is well known that the temperature and stress dependence of thermodynamic driving forces and kinetic mobilities may affect microstructures. It is therefore by no means certain whether elementary deformation and softening processes which govern short-term creep also occur under long-term creep conditions. Microstructural data from long-term creep tests are moreover urgently required to provide input data and validation criteria for advanced material simulations. The third drawback is related to the fact that long-term creep testing is tedious. While efforts have been made to produce rupture data, much less emphasis has been placed on providing interrupted material states, which are equally important to assess the evolution of microstructure. Several researchers discussed the evolution of microstructure by comparing the initial material state with the material after long-term creep rupture. Every microstructural investigation must assess these two conditions. But, in addition, intermediate states from interrupted long-term creep experiments need to be taken into account to safely describe microstructural evolution. Finally, at the time when research on the effect of long-term creep and microstructure started (and this especially holds for Ref. [9]) the instruments that were available for microstructural characterization were less powerful than the equipment we have today. Most importantly, transmission electron microscopes with field emitter cathodes (FEG-TEMs) were not available and the associated advantages (such as better chemical contrast and higher lateral resolution in chemical and crystallographic analysis) could not be exploited. Equally important, scanning electron microscopes with field emitter cathodes and with the possibility of imaging orientations (OIM-SEM) were not easily accessible.

In the present work we use modern characterization techniques to study the microstructural evolution in a 12% Cr tempered martensite ferritic steel (German grade: X20) subjected to long-term creep at a stress of 120 MPa and 823 K. We investigate the initial material state, interrupted specimens which were creep loaded for 12,456, 51,072 and 81,984 h, and the ruptured specimen which failed after 139,971 h. Our study has four objectives: (i) to show that microstructural processes which govern short-term creep testing can also be identified under long-term creep conditions; (ii) to provide data which could not be obtained in earlier studies (e.g. [9]), because no interrupted long-term creep specimens were available, because TEM work was more tedious and because OIM-SEM could not be performed; (iii) to show how precipitate populations evolve during long-term creep and how our results compare to results reported in the literature; and (iv) to identify the need for a better collaboration between experimentalists who characterize microstructures and scientists developing thermodynamic and kinetic models which aim at predicting the evolution of microstructures.

2. Experiments and data evaluation

2.1. Creep testing and hardness measurements

The 12% Cr tempered martensite ferritic steel investigated in the present work was supplied by the Salzgitter Mannesmann Research Centre, Duisburg. Its chemical composition is given in Table 1. Its heat treatment consisted of austenitizing at 1323 K for 30 min followed by air-cooling and subsequent tempering at 1043 K for 2 h. Long-term tensile creep testing was performed at the Salzgitter Mannesmann Research Centre. Under an applied stress of 120 MPa at 823 ± 1 K three interrupted experiments were performed and one specimen was taken through to rupture. The rupture time was 139,971 h. The material states available for the present study are listed in Table 2. Creep strains were manually measured during interruptions of the creep tests. Fig. 1 shows the creep data which document the strain time histories of all creep specimens. Fig. 1a shows how creep strains increase with the logarithm of time in hours. Fig. 1b and c show the logarithm of strain rate in s^{-1} as a function of the logarithm of time in hours and strain, respectively. The creep data show that there is only little scatter between the different creep specimens (Fig. 1a, where the four data sets of the different specimens can hardly be distinguished) and that the creep rate minimum was established after about 30,000 h (Fig. 1b) corresponding to 1% strain (Fig. 1c). It should be noted that the interrupted material states 2, 3 and 4 (see Table 2) were all creeping close to the minimum creep rate (below 10^{-10} s^{-1}) when the tests were interrupted (Fig. 1b). With respect to the ruptured specimen (Fig. 1a and 1c), it is important to point out that less than 2% creep strain accumulated within 100,000 h. The strong increase of creep strain in the final stages of the creep test is partly related to necking. The life fractions in the right column

Table 1
Chemical composition of 12% Cr steel (German grade: X20) in wt.%.

C	Si	Mn	P	S	Cr	Mo	V	Al	Ni	Fe
0.20	0.15	0.61	0.009	0.014	11.7	0.84	0.25	0.011	0.65	Balance