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Laves phase evolution in a modified P911 heat resistant steel during creep at 923 K

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ABSTRACT

The evolution of Fe_2(W, Mo) Laves phase in a 3%Co modified P911 heat resistant steel was examined during creep tests at 923 K. The tempered martensite lath structure evolved after heat treatment was characterized by dispersion of MX carbonitrides and $M_{23}C_6$ carbides. Appearance of Laves phase particles was recorded after a creep strain of 1%. The mean size of Laves phase particles increased from 190 to 265 nm with increasing strain to 18%. The Laves phase particles were spaced on various boundaries including low-angle boundaries (LABs) of laths/subgrains, but most of these particles (about 90%) were located on high-angle boundaries (HABs) at all strains studied. The size of Laves phase particles located on HABs was larger and their coarsening kinetics was faster than those precipitated on LABs. It is assumed that the evolution of Laves phase during creep is controlled by the grain boundary diffusion of tungsten and molybdenum.

1. Introduction

The 9–12% chromium heat resistant steels have successfully been used for boiler components and turbine rotors of ultra super critical power plants [1–4]. The service properties of martensitic steels are achieved by a complex alloying that provides solution and dispersion strengthening. The solid solution strengthening is provided by the additions of W, Mo, Cr, Co. On the other hand, the elements, such as V, Nb, N and C contribute to the formation of homogeneously distributed fine precipitates, which strengthen and stabilize the tempered microstructure. After tempering, the heat resistant steels usually contain two types of particles: $M_{23}C_6$ carbide and MX carbonitride [5,6]. The $M_{23}C_6$ carbides mainly precipitate along high and low-angle boundaries and they act as obstacles for migrating boundaries. Efficiency of $M_{23}C_6$ particles in stabilizing tempered martensite lath structure (TMLS) degrades with service duration, since they tend to coarsen during creep at elevated temperatures. It was recently shown [1,7,8] that this is a key factor restricting creep resistance of martensitic steels. Homogeneously distributed fine MX carbonitrides are more stable at operating temperatures as compared to $M_{23}C_6$ particles. After rather long ageing and/or creep, relatively large particles of Laves phase appear in martensitic steels [9–14]. Laves phase being thermodynamically equilibrium one precipitates at temperatures below about 973 K during creep exposure or isothermal ageing and results in removing the W and Mo from the solid solution [9,14,15]. A lot of work has been conducted to study the effect of Laves phase on stability of microstructures and strength of steels during creep [9–15]. It is assumed that the fine precipitates of Fe_2W Laves phase effectively decrease the creep rate in the transient region [14]. In other words, the fine Laves phase particles may contribute to dispersion hardening [3,14,15]. The significant contribution to dispersion strengthening from Laves phase particles can be expected in the case of their homogeneous precipitation throughout ferritic matrix. However, there is no experimental evidence for homogeneous precipitation of Laves phase. It was also pointed out that extensive coarsening of Laves phase promotes the acceleration or increase of creep rate after reaching a minimum creep rate [14]. The dispersion strengthening owing to precipitations of Laves phase compensates for the depletion of W from solid solution in the transient creep, but coarse particles of Laves phase along with W-deficient solid solution accelerate the creep rate at sufficiently large strains [9].

In spite of numerous papers, the precipitation sites for Laves phases have not been studied in sufficient detail, although this is a key issue to establish the role of Laves phase on creep resistance of martensitic steels. Sawada et al. suggested that Laves phase (Fe_2Mo) particles in Mod.9Cr–1Mo steel precipitate on high-angle boundaries (HAB) of prior austenite grains (PAG) and packet boundaries in martensitic lath structure, while Fe_2W particles in TAF650 steel precipitate on lath boundaries, thereby retarding

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the recovery of lath structure and increasing the creep rupture strength [16]. Particles of Laves phase located on HABs scarcely contribute to the dispersion hardening. However, Laves phase precipitations on low-angle boundaries (LABs) of martensite laths should improve their efficiency to act as barriers for dislocation motion, leading to additional structural strengthening. The aim of the present work is to clarify the precipitation sites for Fe_2(W, Mo) Laves phase and investigate their evolution in a 3%Co modified P911 heat resistant steel during creep at 923 K.

2. Experimental procedure

A 3%Co modified P911-type steel (0.13%C, 8.6%Cr, 3.2%Co, 1.15%W, 0.93%Mo, 0.2%V, 0.1%Cu, 0.07%Nb, 0.06%Si, 0.05%Ni, 0.04%N, 0.02%Mn, 0.005%B, all in mass%, and the balance Fe) was examined. The steel ingot was hot forged at 1373 K to a square bar of 25 mm × 25 mm. Then, the steel forging was austenitized at 1323 K for 0.5 h followed by air cooling and tempered at 1023 K for 3 h. Flat specimens having a cross section of 7 mm × 3 mm and 25 mm gauge length were used for creep tests. The creep tests were carried out at 923 K with initial stress of 118 MPa, to strains of 1% (374 h), 4% (1654 h), 6% (2960 h) and to rupture (4743 h) in order to study the evolution of Laves phases at various creep stages.

The microstructural investigations were carried out on longitudinal section of crept specimens within the gauge length by using a Quanta 600 FEG scanning electron microscope (SEM) incorporating an orientation imaging microscopy (OIM) system and an X-ray energy-dispersive spectrometer (EDS). The SEM specimens were prepared by electro-polishing method with a 10% solution of perchloric acid in glacial acetic acid. The OIM maps of dimension of 50 μm × 50 μm were obtained under the following conditions: accelerating voltage of 20 kV, beam current of 18 nA, working distance of 10 mm, step size of 50 nm. The grain boundaries with misorientations of $\theta \geq 15^\circ$ and $2^\circ < \theta < 15^\circ$ were considered as HABs and LABs, respectively. The HABs are depicted in the OIM images as black lines, and the LABs are indicated by white lines. An area of 30 μm × 26 μm was scanned with BSE (back scattered electron) detector for each sample. The Laves phase could be clearly distinguished from other precipitates by its bright contrast in the BSE image (Z-contrast) [17]. The particles of Laves phase were identified by using *Z*-contrast technique as well as by chemical composition. The amounts of Fe, Cr, W, and Mo, were measured for the chemical analysis. A particle was considered as Laves phase if the total content of tungsten and molybdenum comprised 30–35 at%. A total of 150–300 particles of Laves phase was analyzed for each data point.

3. Results and discussion

3.1. Creep behavior

Fig. 1 shows the creep rate versus creep strain curve of the P911 + 3%Co steel at 923 K and under nominal stress of 118 MPa. The curve consists of the transient creep region extending up to a strain of 1%, and after reaching a minimum creep rate at strain of about 2% the region of accelerated creep takes place at strains beyond 6%. The creep behavior is characterized by an almost constant creep rate of about 2×10^{-5} h^{-1} in the strain range of 1–6%, suggesting that an apparent steady-state flow takes place. In order to clarify the evolution of Laves phase during creep the microstructures were studied in the samples crept to some characteristic strains indicated by arrows in Fig. 1.

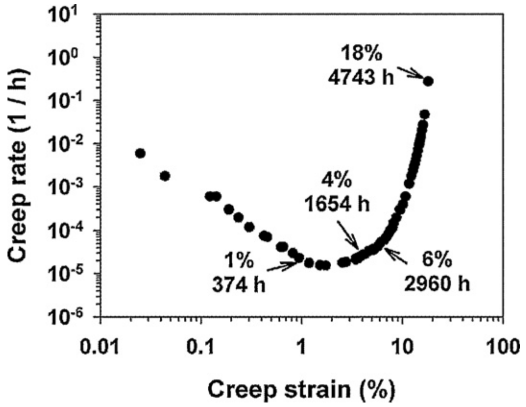

Fig. 1. Creep rate *vs.* creep strain curve for the P911 + 3%Co steel at 923 K.

3.2. Microstructures after creep tests

Typical microstructures that evolved in the gauge portions of samples subjected to creep at 923 K to different strains are shown in Fig. 2 as a series of OIM and SEM–BSE images. The SEM–BSE images correspond to the indicated portions in OIM micrographs. The microstructures of P911 + 3%Co steel consist of the PAGs, which are subdivided by HABs of packets and blocks and LABs of laths (Fig. 2). The Laves phase particles are observed after relatively small strain of 1% (the creep duration is 374 h). Therefore, the crept microstructures are characterized by a dispersion of MX carbonitrides, $M_{23}C_6$ carbides and relatively large Laves phase particles. The Laves phase particles can be uniquely distinguished from any other precipitates by their bright contrast in the SEM–BSE images (Fig. 2 b, d, f and h). Fig. 3 shows the corresponding EDS analysis for these particles, confirming that the chemical composition is consistent with Laves phase. It is seen that precipitation of Laves phase occurs in highly heterogeneous manner; interiors of martensite laths are almost free of Laves phase particles. Almost all Laves phase particles locate on various boundaries. The most of the particles (about 90%) precipitated on HABs at the all strain levels (Table 1). Precipitation of Laves phases on grain/subgrain boundaries have been observed in other studies on martensitic creep resistant steels [3,6,14]. The prevalent precipitation at HABs may be associated with somewhat increased diffusivity of constitutive elements along ordinary grain boundaries. However, this interesting topic should be further clarified.

Table 1
Laves phase particles in 3%Co modified P911 steel after creep tests at 923 K.

Creep strain	1% (374 h)	4% (1654 h)	6% (2960 h)	Ruptured (4743 h)
Size of particles (nm)	185	188	245	265
Aspect ratio of particles	1.9	1.8	1.8	1.8
Size of particles located on LABs (nm)	155	187	176	225
Aspect ratio of particles located on LABs	2.0	1.9	1.7	1.5
Size of particles located on HABs (nm)	188	188	253	272
Aspect ratio of particles located on HABs	1.9	1.8	1.8	1.8
Volume fraction of particles (%)	0.85	1.42	2.2	2.0
Volume fraction of particles located on LABs (%)	7	12	7.7	7.8
HABs (%)	62.5	60	53.7	57.6