



The role of Laves phase on microstructure evolution and creep strength of novel 9%Cr heat resistant steels

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ABSTRACT

The influence of Laves phase (type Fe_2W) formation and growth kinetics on the creep strength at 650 °C of two different 9%Cr heat resistant steels of the authors' design was investigated. The microstructure evolution was characterized using transmission electron microscopy in the scanning mode (STEM). Kinetic modeling was carried out using the software DICTRA. STEM investigations revealed that the Laves phase precipitates tend to form clusters, have an irregular shape and are often located close to M_{23}C_6 carbides, along martensite lath boundaries or sub-grain boundaries. DICTRA simulations showed that the growth kinetics of Laves phase was high in the first thousand hours of creep, reaching its equilibrium volume fraction after 7000–10,000 h. Simultaneous competitive growth of M_{23}C_6 carbides and Laves phase was simulated showing that Laves phase grows very slowly to reach the final equilibrium phase fraction only after almost 13,000 h, while M_{23}C_6 reached the equilibrium phase fraction already during tempering. Best creep results were obtained for the 9%Cr alloy with low carbon content, reduced sub-grain growth, very slow coarsening of MX carbonitrides and slow growth of Laves phase.

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1. Introduction

9–12%Cr heat resistant steels are used as component materials for steam power plants. One main focus of current investigations is on design and production of advanced 9–12%Cr materials to achieve good creep properties at temperatures above 650 °C; see i.e [1].

Within the family of 9–12%Cr heat resistant steels, there have been many investigations in order to develop 9%Cr steels which present good creep properties at 650 °C [2–7]. For example, Abe et al. [3] concentrate their efforts on producing 9Cr–3W–3Co steels containing balanced B and N contents for high temperature operations. Different types of precipitates are found in the microstructure of 9%Cr heat resistant steels such as M_{23}C_6 carbides, Laves phase, MX nitrides or carbonitrides and Z-phase [8].

The increase of working temperature and pressure affects the microstructure of 9%Cr steels during service [9]. In order to improve their time to rupture the degradation of the microstructure (coarsening, precipitation of undesirable phases, subgrain growth, etc.) must be retarded [10]. M_{23}C_6 carbides precipitate on prior austenite grain boundaries, on subgrain boundaries and within

martensite laths [11]. These precipitates retard the sub-grain growth and therefore increase the strength of the materials [12].

MX carbonitrides are important as obstacles for the movement of dislocations. The small numbers of such precipitates in 9%Cr heat resistant steels may, however, not be sufficient for controlling the deformation behavior [4].

In 9–12%Cr steels Z-phase formation has been observed after long service conditions producing a drastic reduction of creep strength [13]. Z-Phase nucleates on existing MX carbonitrides (V-, Nb-, Ta-based ones) [14,15]. This effect can be retarded by reducing the Cr content of the alloy (i.e. 9%Cr) so that the kinetics of Z-phase formation is shifted to longer creep times [5].

Another important precipitate in 9–12%Cr steels is the Laves phase of the type Fe_2W . This precipitate is an important intermetallic phase that can significantly influence microstructure as well as mechanical properties [6]. Some authors state that the formation of Laves phase in 9–12%Cr is detrimental, mainly because Laves phase formation promotes depletion of W from solid solution [7,16]. However, other authors proved that the formation of fine Laves phase particles on sub-grain boundaries and grain boundaries retards the recovery of sub-grains, which improves the creep strength [17]. In this case the growth of Laves phase should be controlled during high temperature exposure to achieve a high creep resistance [18].

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Table 1
Chemical composition of alloys investigated (wt%).

Sample	B	C	Co	Cr	Mn	N	Nb	Si	V	W	Fe	Austenitisation T °C
9Cr-L	0.013	0.051	1.01	8.97	0.48	0.005	0.035	0.37	0.15	1.98	Bal.	1080
9Cr-H	0.009	0.108	0.99	8.81	0.49	0.005	0.030	0.38	0.15	2.00	Bal.	1120

Hättestrand et al. [19] showed that the precipitation of the Laves phase takes place after long-term aging or creep at 600 °C and 650 °C.

In previous investigations the authors produced and characterized two 12%Cr model steels [20–23]. Prat et al. [23] observed that the kinetics of Laves phase growth was affected by the composition of the alloy, and that the equilibrium phase fractions were reached within the first thousand hours of creep. Simulations were compared with experimental results showing good agreement.

Recently the authors produced and investigated novel 9% Cr heat resistant steels [2]. The main objective of the alloy design was to improve creep strength at 650 °C by balancing the amount of precipitates formed in the microstructure, i.e. MX carbonitrides, Laves phase and $M_{23}C_6$ carbides.

The aim of the present research is to investigate the effect of Laves phase formation and growth on creep strength of these 9%Cr alloys recently designed by the authors [2]. Two alloys were investigated. Alloy 9Cr-H ($H = 0.1\%C$) and 9Cr-L ($L = 0.05\%C$) were designed to have a fine dispersion of V-rich and Nb-rich MX precipitates, $M_{23}C_6$ carbides and Laves phase in the investigated range of temperature. The variation of carbon was mainly carried out to observe the effect of volume fraction and size of precipitates on the formation of $M_{23}C_6$ precipitate and MX carbonitrides. The composition was adjusted to allow the formation of Fe_2W Laves phases during creep at 650 °C.

The growth kinetics of the Laves phase is modeled using the DICTRA (Diffusion Controlled TRAnsformations) software [24,25]. The method is based on the assumption of local equilibrium at the moving phase interface, which means that the rate of transformation will be controlled only by diffusion. The simulation results are compared to experimental information (size and distribution of precipitates) obtained by quantitative metallography using transmission electron microscopy in scanning mode (STEM).

2. Experimental

2.1. Materials

The investigated alloys were produced by vacuum induction melting with masses of about 4 kg. The chemical compositions are listed in Table 1. The composition of the alloys were designed to produce master alloys with fine precipitates of the type MX ($M = V, Nb$; $X = C, N$), $M_{23}C_6$ carbides and precipitation of Laves phase during creep. The samples were hot-forged at 1150 °C (total area reduction of 66%) and cooled in air. In Fig. 1 Thermo-Calc [26] calculations of the phase diagram for both alloys 9Cr-H and 9Cr-L are shown. V-MX denotes V-rich precipitates which contain Nb, N and C and few Fe and Cr, whereas Nb-MX refers to Nb-rich particles with C, Cr and N and also few amounts of V. The thermodynamic calculations have shown that Z-phase is the most stable carbonitride at 650 °C. The austenitization temperatures were chosen considering the Thermo-Calc modeling of the system (Table 1). Standard heat treatments were performed, including austenitization for 0.5 h, subsequent air-cooling and tempering at 780 °C for 2 h with posterior air-cooling.

Tensile creep tests in air at constant temperature of 650 °C (± 5 °C) with constant load (100–125 MPa) were carried out to determine the creep rupture times. Standard cylindrical samples according to DIN50125 B 4×20 were used with 40 mm gauge length and 4 mm diameter. The investigated samples were creep tested for 3,400, 8,000, 10,200 and 13,700 h.

2.2. Transmission electron microscopy

The microstructures of the alloys in initial state and under creep conditions were analyzed using a TECNAI F20 G^2 operating at 200 kV in the STEM mode. The microstructure investigations after creep were carried out on samples about 15 mm from the fracture zone, avoiding the necking area.

TEM specimens were prepared from 3 mm disks, which were ground and polished down to a thickness below 0.1 mm. These disks were electropolished using a standard double jet procedure with a solution of 95 vol.% acetic acid and 5 vol.% perchloric acid as electrolyte. The electropolishing was performed at 15 °C and a voltage of 43 V. Observations were carried out in the bright field (BF) and STEM mode. Precipitates were identified by a combination of electron diffraction patterns (DP) and energy dispersive spectroscopy (EDS) analysis.

2.3. Quantitative analysis of precipitates

The Laves phase precipitates are often found to be not spherical. In order to determine a diameter for these particles a mean particle

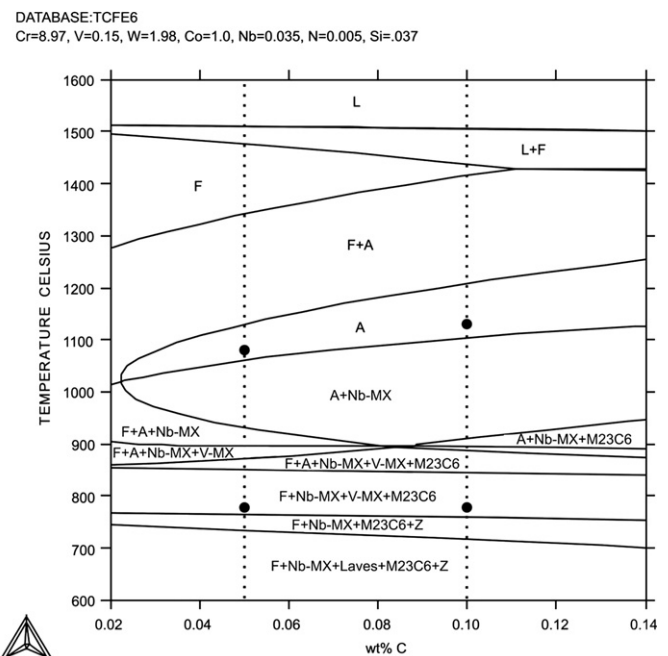


Fig. 1. Phase diagram of alloys 9Cr-H and 9Cr-L (F = ferrite and A = austenite, Thermo-Calc TCFe6). The austenitization temperature and the tempering temperature are indicated in the phase diagram by black circles for each alloy. V-MX indicates V-rich phases which contain Nb, N and C and few Fe and Cr, whereas Nb-MX denotes Nb-rich particles with C, Cr and N and also few V.

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