

# On the nucleation of Laves phase particles during high-temperature exposure and creep of tempered martensite ferritic steels

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## Abstract

This paper reports on the formation of an Mo-rich Laves phase during high-temperature exposure and creep of a tempered martensite ferritic steel with 12 wt.% Cr and 1 wt.% Mo. The material was exposed to 550 °C for time intervals between 864 and 81,984 h. For comparison, a few creep tests were carried out at 550 °C and 120 MPa (duration between 864 and 12,456 h). All tests were interrupted after specific time periods and microstructures were investigated using transmission electron microscopy and atom probe tomography. Laves phase formation occurs during both heat treatment and creep. Creep stress and strain have no significant effect on the early stages of Laves phase formation. In the present work we show that prior to Laves phase nucleation Si and Mo segregate to micrograin boundaries, where subsequently Laves phase particles appear next to  $M_{23}C_6$  carbides.

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## 1. Introduction

Tempered martensite ferritic steels are important engineering materials which are used for critical components in fossil fuel-fired power plants, where they have to withstand mechanical loads at temperatures of around 600 °C [1–3]. Creep governs the service life of such high-temperature components as steam pipes and turbine rotors. Creep is characterized by time-dependent plastic deformation and a deformation rate that strongly depends on stress and temperature [4–6]. All commercially successful members of the tempered martensite ferritic steel family contain Fe (base), Cr (between 9 and 12 wt.%) and C (between 0.1

and 0.2 wt.%). In addition, they contain carbide-stabilizing elements like Mo, W, V and Nb [3,7–12].

The microstructure of tempered martensite forms during thermo-mechanical processes. Additionally, a heat treatment which typically consists of austenitizing and tempering was applied. During austenitization at temperatures above 1000 °C, carbides dissolve and austenite grains grow. On cooling from the austenite regime, due to the presence of Cr, austenite transforms to martensite even at moderate cooling rates [13–15]. After a typical austenitization treatment, austenite grains can reach an average grain size of 30 μm [16]. During cooling, about five blocks or packets of parallel martensite laths form in a typical prior austenite grain [17,18]. Martensite is brittle, so tempering at temperatures between 600 and 750 °C after austenitization is required. During tempering, the microstructure transforms from martensite to ferrite [18]. The martensite

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laths transform into a fine elongated micrograin structure, with a typical micrograin having a length of 2  $\mu\text{m}$  and a width of 0.5  $\mu\text{m}$  [17]. Moreover, after processing, the microstructure contains a high density of free dislocations [16,19,20]. Most importantly, carbides precipitate on all internal interfaces [21]. Larger carbides, typically of type  $\text{M}_{23}\text{C}_6$ , precipitate on prior austenite grain boundaries and prior martensite lath block boundaries [16]. Finer carbides pin the micrograin boundaries [9,22]. Today, material scientists are well aware of the fact that the term *tempered martensite* reflects the heat treatment history of the material and does not imply that the material's microstructure is actually martensitic. The term *carbide stabilized micrograin strengthening* was proposed to rationalize the creep strengthening mechanism of these complex materials [17,18]. At temperatures of around 600  $^{\circ}\text{C}$ , the microstructures of tempered martensite ferritic steels are not stable. Micrograins and precipitates coarsen and the dislocation densities in the micrograins decrease [20,23]. One can also observe the formation of creep cavities [11,23–25]. These topics have been extensively studied throughout the last few decades.

After a typical heat treatment, the microstructures of tempered martensite ferritic steels are not in equilibrium and when they are exposed to elevated temperatures they strive towards this unknown equilibrium. Dislocation densities decrease [8], and it has been reported that carbides can change their chemical compositions and coarsen [23]. It is well known that Mo- and W-rich Laves phase particles can form, and Laves phase particles of types  $\text{Fe}_2\text{Mo}$ ,  $\text{Fe}_2\text{W}$ ,  $(\text{Fe,Cr})_2(\text{Mo,W})$ ,  $\text{Fe}_2\text{Nb}$ ,  $\text{Fe}_2\text{Ti}$  etc. have been observed [18,23,26–28]. In a few cases the precipitation of Laves phase particles has been reported after heat treatments [29]. It is also common to find Laves phase particles after creep exposure [18,23]. When Laves phase particles are detected, they are often observed to be large (e.g. 200 nm) [23]. This phenomenon has been presented in time temperature transformation type diagrams, where large Laves phase particles suddenly appear after 2000 h [30].

The present study builds on earlier work [16,23] in which the microstructures of deformed (gauge lengths) and undeformed parts (threads) of creep specimens (550  $^{\circ}\text{C}$ , 120 MPa) interrupted after 12,456, 51,072, 81,984 and 139,971 h were investigated. No Laves phase particles were present in the initial state, prior to thermal exposure and creep. A statistical relevant number of Laves phase particles (between 90 and 255) were investigated for each interrupted material state by electron diffraction in a transmission electron microscope. The particles were chemically analyzed by energy-dispersive X-ray analysis in the transmission electron microscope. Five facts were established: first, large Laves phase particles were already detected after 12,456 h [16,23]. Therefore, one can conclude that Laves phase nucleation can occur in time intervals smaller than 12,456 h. Second, there were no significant differences between the deformed and undeformed parts of the creep specimens, as far as the occurrence of Laves phase particles is concerned [16,23]. It is sufficient to study

the nucleation of Laves phase in specimens which were only subjected to stress-free temperature exposure. Third, the volume fraction and size of the Laves phase particles increase up to 139,971 h [16,23]. Therefore, the growth of Laves phase particles cannot be fully rationalized merely on the basis of Lifschitz–Slyozov–Wagner-type coarsening. It is also clear that the material does not reach thermodynamic equilibrium within practically relevant times of temperature exposure. Fourth, the number fraction of Laves phase particles increases up to 139,971 h [16,23]. This allows one to conclude that the nucleation of Laves phase particles is continuous. Finally, Laves phase particles always have the same chemical composition and always contain 7 at.% of Si. The slow atomic mobility of Si was invoked to rationalize the time required for nucleation and the continuous nature of precipitation [31]. This kinetic constraint also explains the fact that thermodynamic equilibrium was not reached in times of practical relevance (even after 139,971 h) [31]. These findings are significant from an engineering perspective. While these recent studies [16,23,31] have helped us to better understand the formation and growth of Laves phase particles, one question could not be answered [16,23,31]: how do Laves phase particles nucleate?

The present work focuses on a 12 wt.% Cr tempered martensite ferritic steel with 1 wt.% Mo and 0.2 wt.% C (German grade X20CrMoV12-1). High-resolution characterization techniques, such as analytical transmission electron microscopy (TEM) and atom probe tomography (APT), are used to study the combination of elementary processes which lead to the formation of Laves phase particles. The findings are rationalized on an atomistic and thermodynamic/kinetic basis. This study has four objectives. First, to identify the conditions which are required for Laves phase nucleation. Second, to document the pre-nucleation phenomena, such as segregation of Si to internal interfaces. Third, to evaluate the thermodynamic consequences of this type of chemical evolution with respect to the formation of Laves phase particles. And finally, to clarify the role of microstructural constituents which promote the nucleation and growth of Laves phase particles. The results given in the present work suggest that a gradual change in interface chemistry promotes the nucleation of Laves phase particles.

## 2. Materials and methods

In the present study experiments were performed on two tempered martensite ferritic steels, referred to as alloys S and L. Both steels are of German grade X20, containing close to 12 wt.% Cr, and were provided by the Salzgitter Mannesmann Research Center (SMRC). Their chemical composition is shown in Table 1. Two-stage heat treatments were applied, comprising austenitization at 1050  $^{\circ}\text{C}$  for 0.5 h and tempering at 770  $^{\circ}\text{C}$  for 2 h, both followed by air cooling. The investigated material states are listed in Table 2. Both aged and crept material states were

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