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# THERMODYNAMIC AND KINETIC MODELING OF PRECIPITATION PHENOMENA IN P9 STEELS

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# ABSTRACT

Thermo-kinetic simulation of precipitate evolution during long-term thermal exposure in four different ferritic-martensitic heat-resistant power plant steels (P91, P92, E9016, and RAFM) is carried out using MatCalc and Thermo-Calc softwares, in combination with an independently developed Gibbs energy database and a modified version of mobility database for steels that comes with MatCalc. MX and M<sub>23</sub>C<sub>6</sub> are predicted to remain as major precipitates during long-term aging in these steels. Average size of MX precipitate is found to vary between 10-100 nm during the aging, while  $M_{23}C_6$  exceeds 100 nm after 100,000 h of thermal exposure at 600°C. The simulated precipitation sequence and precipitate size evolution during thermal exposure are in general agreement with available experimental data. It is expected that the calculations presented here gives insight into long-term microstructural stability of ferritic-martensitic steels under service conditions, which are otherwise difficult establish by experiments alone.

# Introduction

There is an urgent need to improve the thermal efficiency of fossil fuel as well as nuclear power plants, mainly due to environmental considerations. This can be achieved by increasing operating temperature and pressure of the power plants. In this context pipe materials that can perform well at elevated temperature and pressure are desirable, without compromising safety aspects. Plant operations at higher temperatures inevitably requires the development of heat-resistant alloys with a higher creep rupture strength at an acceptable level of creep ductility. In this regard 9-12 % Cr ferritic-martensitic heat resistant steels with other alloying additions are ideal materials owing to their high thermal conductivity, low thermal expansion coefficient and low susceptibility to thermal fatigue.

It is generally accepted that ferritic and austenitic steels are useful up to about 620 and 675°C, respectively, purely from the creep strength point of view at a steam pressure of about 35 MPa [1]. The role of precipitation phenomenon in enhancing creep strength of heat-resistant steels is well established [2]. In order to improve the creep strength it is necessary to ensure a uniform distribution fine precipitates with good long-term high temperature stability. For example, austenitic heat-resistant steels are known to exhibit quite complex precipitation behavior involving precipitates such as carbides, nitrides and intermetallic phases [3]. In the case of 9-12 % Cr ferritic-martensitic steels, the creep strength is due to their special microstructural features. Due to diffusion processes at elevated temperature service, microstructural changes takes place leading to deterioration in strength. In ferritic-martensitic steels strength deterioration is attributed to coarsening of M<sub>23</sub>C<sub>6</sub> precipitates and transformation of MX precipitates into Z-phase (Cr(V,Nb,Ta)N). In this

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paper we have simulated long-term microstructural changes and time dependant evolution of major precipitates in some important ferritic-martensitic steel grades.

#### Materials and simulation method

Four different ferritic-martensitic heat-resistant steels are chosen for the present study. Chemical compositions of these steels are given in Table 1. They contain about 9 wt.% Cr, which is lower than the Cr content of conventional austenitic heatresistant steels. They are air-hardenable which causes austenite to transform completely into martensite. Mo imparts improved the creep rupture strength, Nb forms fine MX precipitates which is stable even at elevated temperature. Presence of W in P92 grade further enhances creep rupture strength and steamoxidation resistance at temperatures exceeding 600°C.

RAFM steels are essentially similar to medium or high chromium low carbon steels such as V, Nb modified 9Cr-1Mo steel, but with a major difference with respect to elements producing long half-life transmutants like Ni, Mo, Nb, Cu, Co, Al, N, etc. These elements are largely substituted by their comparatively lower activation counterparts, such as Mn, W, V, Ta, and C. Mo is replaced by W and Nb by Ta. Strict control is exercised on the radioactive tramp elements (Mo, Nb, B,Cu, Ni, Al, Co, Ti) and on the elements that promote embrittlement (S, P, As, Sb, Sn, Zr, O). These elements are usually restricted to ppm levels.

Simulation of long-term precipitate evolution in austenitic stainless steels has already been attempted by Shim et al. [4], using MatCalc software developed by Kozeschnik et al. [5, 6]. Simulation of precipitate evolution in certain ferritic-martensitic 9-12 % Cr steels are also reported in the literature [7, 8]. The purpose of this study is to simulate the long-term precipitate evolution in some important grades of ferritic-martensitic heat-resistant steels for applications around 600°C by MatCalc software, using an independently developed Gibbs energy database for steels and a modified version of the mobility database that is included with MatCalc.

MatCalc uses classical nucleation theory along with Onsager's extremum principle for simulating precipitate evolution. It has a numerical model to classify precipitates of same radius and composition nucleated in different intervals of time. In the precipitation kinetics approach implemented in MatCalc, the the microstructural evolution of the system is simulated within the framework of the Kampmann-Wagner model [9]. Accordingly, the total time history is broken into adequately small, isothermal segments [6]. Precipitates of equal size and chemical composition are grouped into classes, for each of which the evolution in size and composition is calculated according to the rate equations derived from the thermodynamic extremum principle [5]. Nucleation of new precipitates is taken into account in each time step based on a multicomponent extension of classical nucleation theory [10, 11]. According to this, the transient nucleation rate J defines the number of new nuclei created in the time step  $\Delta t$  as  $J\Delta t$ . *J* is given by

$$J = N_0 Z \beta^* \exp(-\frac{G^*}{kT}) \exp(-\frac{t}{\tau})$$
(1)

where  $N_0$  represents the total number of available nucleation sites, k is the Boltzmann constant, T is the temperature, Z is the Zeldovich factor,  $\beta^*$  is the atomic attachment rate,  $\tau$  is the incubation time and  $G^*$  is the critical nucleation energy given by

$$G^* = \frac{16\pi}{3} \frac{\gamma^3}{\Delta G_{vol}^2} \tag{2}$$

where  $\gamma$  is the specific interfacial energy and  $\Delta G_{vol}$  is the volume Gibbs energy change on nuclei formation.  $\gamma$  and  $\Delta G_{vol}$  and their composition and temperature dependencies are evaluated using the Gibbs energy database. Both quantities are most essential for achieving reliable calculation of nucleation rates for precipitation kinetics simulations. This issue has recently been discussed by Radis et al. [10] in a treatment of multimodal size distributions in Ni-base superalloys. All required quantities can be calculated from appropriate analytical expressions using the Gibbs energy and mobility databases. Detailed expressions for all nucleation related quantities are summarized by Janssens et al. [11]. The number of potential nucleation sites  $N_0$  occurring in equation (1) is dependent on the choice is whether nucleation is homogeneous or heterogeneous. In the present simulations, possible choices have been homogeneous nucleation in the bulk, or heterogeneous nucleation on dislocations, grain boundaries (GB), subgrain boundaries (SGB), grain boundary edges or grain boundary corners. Actual number of nucleation sites is given by the total number of atoms in the system in the case of homogeneous nucleation, or by the number of atoms located at the heterogeneous nucleation sites in all other cases. For dislocations, the number of sites is given by the number of atoms located at the dislocation lines in a unit volume. The number of atoms in the grain boundary can be estimated from the total grain or subgrain areas, which are given by the grain/subgrain diameter and the elongation ratio. Detailed expressions for calculation of nucleation sites in microstructures are found elsewhere [7]. Finally, the total number of potential nucleation sites from either homogeneous nucleation, or nucleation at dislocations, grain boundaries, subgrain boundaries, edges or corners enter equation (1).

In the kinetic simulation the matrix phase is defined as ferrite. Laves, Z-phase,  $M_{23}C_6$  and MX are considered to be likely precipitates. The transformation of the austenite matrix into martensite is not considered. Instead the precipitates are allowed to nucleate in the ferrite matrix below Ae<sub>1</sub> temperature and allowed to grow till M<sub>s</sub> temperature is reached, below which the growth of the precipitate is very sluggish. Wherever possible we have made use of dislocation density, grain size of ferrite, austenite, subgrain size and precipitate nucleating site reported in the literature [18, 21].

The interfacial energy of the precipitates is an important factor determining their nucleation and growth rates. In MatCalc, interfacial energy is calculated from thermodynamic data, based on the generalized broken bond model [12] taking into account size effects of small precipitates [13]. In this study, the interfacial energy values of coherent and semi-coherent precipitates were assumed to be 75-90 % of the ones calculated for planar and sharp interfaces, respectively. This is done in order to take into account of entropic contributions due to atomic mixing across the interface, which additionally reduces the interfacial energy as compared to the sharp interface. Nucleation considered here is heterogeneous. Quantities such as dislocation density, grain size, subgrain size and type of nucleation site, etc. have greater impact on the steady state nucleation rate [7]. Since it is known that MX, M<sub>23</sub>C<sub>6</sub>, Laves phase and Z-phase have an orientation relationship with ferrite, they are regarded as semi-coherent precipitates. For simplicity of analysis, the shape of the precipitates [14] is assumed to be spherical, although some of them develop characteristic shapes. During the simulation, precipitates of a certain size and composition are considered as belonging to a particular class. Individual size classes are created, rearranged and deleted during simulation [12], allowing to model the evolution of precipitates size distribution. In this study 25 size classes were used in order to ensure sufficient accuracy for the precipitate size distribution.

Heat treatment for these steels started with the solutionizing above Ae<sub>3</sub>. It is assumed that all constituent elements are homogeneously distributed in the matrix and no precipitates exist at the solutionizing temperature. After the solutionizing the steel specimen are cooled linearly down to room temperature at a reasonably high cooling rate. This is followed by tempering at a temperature below Ae<sub>1</sub> and then cooled to M<sub>s</sub> temperature. Finally steel specimen are heated to 600°C, which corresponds to the thermal exposure (service) temperature. The thermal exposure is done for 100,000 h.

## Results and discussion Thermodynamic calculations

Equilibrium thermochemical data and phase transformation temperatures are calculated using the Gibbs energy database for steels, employing Thermo-Calc [15] software. The database is created according to the Calphad approach. It contains 20 elements *viz*. Al, B, C, Co, Cr, Cu, Fe, Mn, Mo, N, Nb, Ni, O, P, S, Si, Ta, Ti, V and W. Main difference between the existing commercial databases for steels and the one used here is the inclusion of Ta as an alloying element.

In Table 2 calculated thermochemical data and phase trans-

formation temperatures are compared with the calorimetric data from [16]. Calculated values agree reasonably well with the experimental data. Calculated equilibrium phase fraction plots for E9016 and RAFM steels are shown in Figure 1 and Figure 2, respectively.

#### **Kinetic calculations**

Kinetic simulations are performed using the thermodynamic database together with the mobility database, employing Mat-Calc software. The mobility database was modified to take into account of presence of tantalum. All steels considered here are assumed to be in normalized and tempered condition. Normalization temperature decides the average size of prior austenite grains. The normalization temperature for the specified steels are selected based on the complete homogenization temperature. The grain sizes, subgrain sizes, and dislocation densities used in the kinetic calculations are listed in Table 3 [18, 21]. Phases considered for thermodynamic and kinetic calculation are listed in the Table 4. Since MX, M<sub>23</sub>C<sub>6</sub>, Laves, Z-phase are the major phases that are present after several hours of thermal exposure, only these phases are included in the kinetic calculations. The chosen nucleation sites for these precipitates are also given in the Table 4. These four phases were made to nucleate in the ferrite (matrix phase).

Tempered martensite has a complex microstructure that consists of various kinds of interfaces such as prior austenite boundaries, martensite packet boundaries, lath/twin boundaries and subgrain boundaries in addition to carbides along boundaries. The tempered steel retains its high dislocation density during austenite to martensite transformation. In the simulation, precipitates are assumed to grow along the grain boundaries, subgrain boundaries and dislocations. High dislocation density, grain and subgrain feature are taken care by considering ferrite as the matrix. The precipitation behavior of steels selected for this study, as revealed by the simulations, are discussed below.

**P91 steel:** Figure 3 shows the variation of the simulated phase fraction of precipitates during the heat treatment and thermal exposure of P91 steel. Four kinds of precipitates, *viz*. MX,  $M_{23}C_6$ , Laves and Z-phase, appear during the heat treatment. MX which forms on dislocation, grain boundaries and subgrain boundaries, starts its nucleation immediately while  $M_{23}C_6$  which nucleates on grain and subgrain boundaries starts nucleating after the solution treatment. Laves phase starts appearing at about 100 h. MX and  $M_{23}C_6$  attains saturation of precipitate is evident from Figure 4. Its average size remains at ~100 nm during most time. The average size of MX precipitate is ~75 nm. The amount of Z-phase is very small. Noticeable coarsening of Z-phase is seen after 10,000 h of thermal exposure and continues to increase in size, which is in contrast with the behavior of MX and  $M_{23}C_6$ 

precipitates.

**P92 steel:** Figure 5 shows the variation of the phase fraction of precipitates during the heat treatment and the thermal exposure at 600°C of P92 steel. Four precipitates, MX, M<sub>23</sub>C<sub>6</sub> and Z-phase and Laves phase appear in the microstructure. Zphase starts nucleating right after the tempering. The amount of Z-phase starts to increase slightly after 10,000 h of thermal exposure and continues to increase until the service terminates, which is in contrast with MX and M<sub>23</sub>C<sub>6</sub> exhibiting the saturation of precipitation in a short time [19]. Laves phase, though small in quantity, forms during the thermal exposure and keeps on coarsening (Figure 6). This is attributed to high amount of W and Mo in this steel. The average size of the Laves phase precipitates reaches  $\sim 1 \mu m$  at about 100,000 h. The precipitation of Laves phase can improve creep strength if its coarsening does not proceed too fast. This positive behavior of the Laves phase is seen in NF616 (similar to P92) steel. However, the presence of large  $M_{23}C_6$  and Laves phase particles above approximately 0.5  $\mu m$ are generally considered to be deleterious [20].

**E9016 steel:** Figure 7 shows the variation of the phase fraction of precipitates in the case of E9016 steel. Like in the case of P91 steel, four kinds of precipitates, MX, M<sub>23</sub>C<sub>6</sub>, Laves and Z-phase, appear during the course of thermal exposure. Phase fraction of M<sub>23</sub>C<sub>6</sub> precipitates remain constant after attaining the equilibrium value during the thermal exposure. Significant amount of Laves is seen from about 100 h onwards. The amount of Z-phase starts to increase significantly after 10,000 h of thermal exposure and continues to increase until the service terminates. It is well known that during service Z-phase grows at the expense of MX in 9-12 % Cr ferritic-martensitic heat-resistant steels containing Nb or V and a high content of nitrogen [17]. This fact is clearly evident from Figure 7. Although there is no significant coarsening seen in the case of MX precipitates, Zphase, Laves and M23C6 seem to undergo coarsening on continued thermal exposure (Figure 8).

**Reduced activation ferritic-martensitic (RAFM) steel:** Figure 9 shows the variation of the phase fraction of precipitates as a function of time for RAFM steel. The amount of  $M_{23}C_6$ , which appears during early stages of the heat treatment, remains nearly the same even after 100,000 h of thermal exposure. Phase fraction of Z-phase exceeds that of MX all throughout. Increase in fraction of Z-phase beyond 10,000 h at the expense of MX precipitate is clearly identifiable in Figure 9. Coarsening of the  $M_{23}C_6$  precipitate towards later stages of thermal exposure is evident from Figure 10. There is slight reduction in the average size of MX precipitates beyond 10,000 h. Figure 11 shows variation in the composition of  $M_{23}C_6$  as a function of time. It is seen that its Cr content increases and there is a corresponding decrease the Fe content as the thermal exposure advances [22].

#### Conclusions

Comparison of calculated phase transformation temperatures with experimental values shows that the Gibbs energy database used here is reliable in predicting phase transformation features of ferritic-martensitic steels. M<sub>23</sub>C<sub>6</sub> is a major carbide in all the steels considered here, followed by MX carbide. In P91 and RAFM, Z-phase is almost non-existent. When it is present, its amount increases at the expense of MX carbides during thermal exposure. E9016 is most seriously affected with coarsening of the Z-phase. In P92 most significant coarsening is for the Laves phase, although its amount is quite low. In most varieties of steel considered here M23C6 tends to resist coarsening up to about 10,000 h after which it tends to coarsen. It is also seen that in  $M_{23}C_6$  the amount of Cr increases and there is a corresponding decrease the Fe content as the thermal exposure advances. The kinetic simulation agrees with the evolution of elemental abundance in the M<sub>23</sub>C<sub>6</sub> phase in RAFM steel observed experimentally.

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TABLE 1. COMPOSITIONS (IN WT.%) OF SELECTED STEELS.

Element	P91	P92	E9016	RAFM
Al	0.011	-	_	0.0036
C	0.10	0.09	0.06	0.091
Cr	8.44	8.72	9.24	9.05
Cu	0.11	_	_	0.005
Mn	0.46	_	1.37	0.56
Мо	0.46	0.45	1.05	0.0036
N	0.008	0.05	0.03	0.0206
Nb	_	0.06	0.03	0.0039
Ni	0.17	_	0.095	_
Р	0.008	_	0.05	_
S	0.002	_	0.001	_
Si	0.49	0.16	0.3	0.05
Та	_	_	_	0.063
Ti	_	_	_	0.0024
V	0.001	0.021	0.17	0.226
W	_	1.87	_	1.00

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Steel	γ–s	tart	γ–fi	nish	$\Delta H^{\prime}$	$lpha  ightarrow \gamma$	Soli	dus	Liqu	idus	$T_{c}$	*	${{M_{s}}^{**}}$
	°	С	٥	С	Jg	$5^{-1}$	0	С	٥	С	٥	С	°C
	Exp.	Cal.	Exp.	Cal.	Exp.	Cal.	Exp.	Cal.	Exp.	Cal.	Exp.	Cal.	Exp.
	Ac <sub>1</sub>	Ae <sub>1</sub>	Ac <sub>3</sub>	Ae <sub>3</sub>									
P91	820	789	864	857	17	21.6	1510	1430	1524	1502	768	731	400
P92	861	857	886	898	10	16.9	1512	1448	1527	1508	741	738	420
E9016	799	673	847	799	4	20	1520	1437	1531	1497	731	720	425
RAFM	831	822	871	841	13	20.8	1457	1452	1532	1509	745	732	450

**TABLE 2.** COMPARISON OF CALCULATED THERMOCHEMICAL DATA AND PHASE TRANSFORMATION TEMPERATURES WITHCORRESPONDING EXPERIMENTAL DATA FROM [16].

\* - Curie temperature

\*\* - Martensite start temperature

**TABLE 3**.
 PARAMETERS USED FOR KINETIC SIMULATION.

Steel	Normalization	Tempering	Grain size (µm)		Subgrain size	Dislocat	tion density $(m^{-2})$
			α	γ	(µm)	α	γ
P91	1050°C	750°C	30	30	1	$10^{14}$	10 <sup>11</sup>
	1 h	2 h					
P92	1070°C	775°C	25	25	0.5	$10^{14}$	$10^{11}$
	1 h	2 h					
E9016	1050°C	760°C	25	25	1	$10^{14}$	$10^{11}$
	1 h	2 h					
RAFM	980°C	760°C	20	20	1	$10^{14}$	$10^{11}$
	0.5 h	1 h					

Steel	Phases considered	Matrix	Precipitating	Nucleation site		
		Phase	Phase	for the precipitate		
P91	$\alpha$ , $\gamma$ , Liquid, M <sub>23</sub> C <sub>6</sub> , MX	α-ferrite	M <sub>23</sub> C <sub>6</sub>	M <sub>23</sub> C <sub>6</sub> - GB,SGB		
	M <sub>6</sub> C,Fe <sub>3</sub> C, Laves, Z-phase		MX	MX - GB,SGB,Dis.		
	$M_7C_3, M_3C_2$		Laves	Laves - GB,SGB		
			Z-phase	Z-phase - GB,SGB,Dis.		
P92	α, γ, Liquid, $M_{23}C_6$ , MX	$\alpha$ -ferrite	M <sub>23</sub> C <sub>6</sub>	M <sub>23</sub> C <sub>6</sub> - GB,SGB		
	M <sub>6</sub> C,Fe <sub>3</sub> C, Laves, Z-phase		MX	MX - GB,SGB,Dis.		
	$M_7C_3, M_3C_2$		Laves	Laves - GB,SGB		
			Z-phase	Z-phase - GB,SGB,Dis.		
E9016	α, γ, Liquid, $M_{23}C_6$ , MX	α-ferrite	M <sub>23</sub> C <sub>6</sub>	M <sub>23</sub> C <sub>6</sub> - GB,SGB		
	M <sub>6</sub> C,Fe <sub>3</sub> C, Laves, Z-phase		MX	MX - GB,SGB,Dis.		
	$M_7C_3, M_3C_2$		Laves	Laves - GB,SGB		
			Z-phase	Z-phase - GB,SGB,Dis.		
RAFM	α, γ, Liquid, $M_{23}C_6$ , MX	α-ferrite	M <sub>23</sub> C <sub>6</sub>	M <sub>23</sub> C <sub>6</sub> - GB,SGB		
	M <sub>6</sub> C,Fe <sub>3</sub> C, Laves, Z-phase		MX	MX - GB,SGB,Dis.		
	M <sub>7</sub> C <sub>3</sub> ,M <sub>3</sub> C <sub>2</sub>		Laves	Laves - GB,SGB		
			Z-phase	Z-phase - GB,SGB,Dis.		

**TABLE 4**. PHASES INCLUDED IN THE THERMODYNAMIC AND KINETIC SIMULATIONS.



FIGURE 1. EQUILIBRIUM PHASE FRACTION PLOT FOR E9016 STEEL.



FIGURE 2. EQUILIBRIUM PHASE FRACTION PLOT FOR RAFM STEEL.



FIGURE 3. EVOLUTION OF PRECIPITATES IN P91 STEEL.



FIGURE 4. PRECIPITATE SIZE DISTRIBUTION IN P91 STEEL.



FIGURE 5. PHASE FRACTION PLOT FOR P92 STEEL.



FIGURE 6. PRECIPITATE SIZE DISTRIBUTION IN P92 STEEL.



FIGURE 7. PHASE FRACTION PLOT FOR E9016 STEEL.



FIGURE 8. PRECIPITATE SIZE DISTRIBUTION IN E9016 STEEL.



FIGURE 9. PHASE FRACTION PLOT FOR RAFM STEEL.



FIGURE 10. PRECIPITATE SIZE DISTRIBUTION IN RAFM STEEL.



 $\label{eq:FIGURE 11.} \ \ ELEMENT \ FRACTION \ IN \ M_{23}C_6 \ PRECIPITATE \ IN \ RAFM \ STEEL.$