σ -Phase Precipitation Mechanism of 15Cr–15Ni Titanium-Modified Austenitic Stainless Steel During Long-Term Thermal Exposure

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Abstract The σ -phase precipitation in 20% cold-worked 15Cr–15Ni titanium-modified austenitic stainless steel was studied during long-term aging treatment. During the isothermal aging stage, the amount of σ -phase was significantly increased at beginning and gradually became constant. The aging time only slightly affected the size and morphology of the σ -phase. Conversely, during the isochronal aging stage, the amount of σ -phase grew rapidly with the increase in the aging temperature. The σ -phase with a large amount of stacking faults was prone to nucleate around the (Ti, Mo)C particles or at the grain boundaries. The (Ti, Mo)C particles can act as effective nucleation sites, where the σ -phase directly precipitates from the austenitic matrix. From this work, the growth of σ -phase is found to be influenced by the aging temperature, and a new mechanism of σ -phase precipitation from austenite has been proposed.

KEY WORDS: Austenitic stainless steel; Aging treatment; σ -phase precipitation; Carbides

1 Introduction

The performance of core structural materials such as cladding and wrapper plays a vital role in liquid metal-cooled fast nuclear reactors (LMFR), as these materials are subjected to fast neutron irradiation and elevated temperatures (the operating temperature of cladding materials is 550–700 °C [1] in the sodium-cooled fast reactors), which induce four major phenomena: phase transformations, void swelling, irradiation creep and mechanical-property destruction [2]. According to the work environment and as a consequence of these phenomena, titanium-modified

austenitic stainless steels have been developed, which exhibit greatly improved irradiation resistance under breeder reactor conditions [3]. In these stainless steels, the formation of fine stable TiC precipitates has been reported to enhance the resistance against void swelling, helium embrittlement and in-pile creep during irradiation [4–6]. Among these austenitic stainless steels, the 15Cr–15Ni titanium-modified austenitic stainless steel was given particular attention. This alloy after 20% cold work has the advantages of high-temperature mechanical properties, weldability, chemical compatibility with sodium coolant and corrosion resistance. Therefore, it has been considered a promising candidate material for cladding and wrapper tubes [6–8].

The cladding and wrapper tubes would be subjected to prolonged exposure at an elevated service temperature of about 550–650 °C and also to short-time transients to higher temperatures. The austenitic stainless steels may experience profound changes in phase stability and microstructure during long aging treatment at 500–900 °C [9–14]. Studies on the microstructural evolution of the material due to aging at elevated temperatures are important because this may lead

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to a decomposition of the austenitic matrix, with formation of several carbides and intermetallic phases [7, 15–17], e.g., MC , M_6C , $M_{23}C_6$ and σ -phase. Among these precipitates, the σ -phase is of interest. This is a hard, intermetallic phase with a complex tetragonal structure, which is detrimental to mechanical properties and corrosion resistance of the austenitic stainless steels. Perron *et al.* [18] and Barcik [7, 17] showed that σ -phase is prone to lead to interface cracks because of its high interfacial energy and poor coherence with the austenitic matrix. It also reduces the corrosion resistance by removing chromium and molybdenum from the austenitic matrix in the 316Nb steel.

Even though the σ -phase was first observed in the Fe–Cr system, it has also been observed in Fe–V, Fe–Mo and Cr–Mo–Ni alloy systems [19, 20]. There is a scientific interest to increase the knowledge regarding the physical properties of this phase, in particular those involved in the mechanism of its formation [21]. There are two major viewpoints on the σ -phase precipitation mechanism in austenitic stainless steels among many published works [7, 22–24]. One viewpoint suggested that the precipitation of the σ -phase might occur in the residual δ ferritic phase, via two mechanisms: (1) direct σ precipitation ($\delta \rightarrow \sigma$) along the periphery of δ islands and (2) an eutectoid decomposition ($\delta \rightarrow \sigma + \gamma^*$) within these islands, where γ^* is denoted as new austenite. The residual δ ferrite phase is more susceptible to facilitate the σ -phase precipitation, which may be completed within a few hours. This is due to a close chemical composition between the δ ferrite phase and the σ -phase and to higher diffusion rates of the alloying elements in the δ ferrite phase [18]. The other viewpoint believes that the precipitation follows the reaction of $\gamma \rightarrow \sigma + \gamma^*$, where γ^* is a chromium- and molybdenum-depleted austenite compared to the original austenite [25]. The direct precipitation of the σ -phase in austenite is in general very sluggish and took thousands of hours [26]. Despite the extensive works focused on the σ -phase formation, such investigations have dealt with austenite always in the presence of residual δ ferrite. So far, very few works have addressed the σ -phase precipitation mechanism in fully austenitic stainless steels, especially in the Ti- or Nb-stabilized austenitic stainless steels between 550 and 750 °C for sustained periods of time. The σ -phase formation is still not well understood, and the relationships with other precipitates, such as NbC and TiC, are also not yet clarified.

This study used samples with 20% cold-worked reduction isothermally aging treated at 700 °C for 72–168 h and isochronally aging treated at 600–750 °C for 96 h. Firstly, various aspects of the precipitation behavior of the σ -phase, such as the composition, morphology, structure, distribution and nucleation sites, were investigated. Secondly, the influence of MC -type carbides on the σ -phase growth kinetics was studied. The objective is to delineate the

precipitation mechanism of the σ -phase in the fully austenitic 15Cr–15Ni titanium-modified stainless steel during the long-term aging treatment.

2 Materials and Methods

The original 15Cr–15Ni titanium-modified austenitic stainless steel ingot had a mass of 50 kg and was prepared by vacuum induction melting (VIM). The ingot, remelted in a vacuum arc remelting (VAR) furnace and then cast into a copper mold, had the following composition (wt%): Fe–15.32Cr–15.1Ni–2.08Mo–1.6Mn–0.46Si–0.30Ti–0.05C–0.009 N. The as-cast ingot was homogenized at 1200 °C for 24 h and then hot-forged at 1150 °C to obtain 50-mm-thick flat billets. The forged billets were then hot-rolled to 12-mm-thick plates and annealed at 1100 °C for 1 h. These plates were cold-rolled with intermediate annealing at 1050 °C for 30 min, and the 2-mm-thick plates cold-worked by 20% were obtained finally.

The cold-rolled specimens were then sealed in tubes under Ar atmosphere and annealed either isothermally at a fixed temperature of 700 °C for different time spans of 72, 96, 120, 144 and 168 h, or for a fixed time span of 96 h at different temperatures of 600, 700 and 750 °C. Thermodynamic calculations were performed by using the Thermo-Calc software package and the TCFE7 thermodynamic database. The microstructures of the annealed specimens were examined by optical microscopy (OM Observer.Z1m) after etching in a 60% nitric acid solution. The amount, morphology and distribution of precipitates in the aged specimens were examined using a scanning electron microscope (SEM Inspect F50) equipped with an energy-dispersive spectrometer (EDS). The Sic IAS image analysis system was applied to measure the volume fractions of the precipitates by calculating more than ten images of each sample. To observe and confirm the precipitates, the cold-rolled samples and the aged samples were made into transmission electron microscopy (TEM) foils by jet-beam electro-polishing at –20 °C in a solution of 10% perchloric acid and 90% ethanol. The foils were examined by TEM (Tecnai G2 F30) operated with a voltage of 200 kV. The crystal graphic analyses of the precipitates were performed using the selected area diffraction patterns (SADPs), and an energy-dispersive X-ray spectrometer (EDX) was used to precisely identify each precipitate.

3 Results and Discussion

3.1 Isothermal Aging Treatment Study

Only small white particles exist in the cold-rolled specimen without aging treatment, as shown in Fig. 1a. Based on the

corresponding TEM analysis results in Fig. 1b, these small white particles can be identified as MC-type carbides with the FCC crystal structure with a lattice constant $a = 0.432$ nm. Besides, a large amount of dislocations is observed around the MC-type carbides. The chemical analysis using EDX confirms that these MC-type carbides are rich in Mo and Ti, so that their chemical formula should be (Ti, Mo)C, as shown in Fig. 1c. The (Ti, Mo)C is very stable in Ti-stabilized austenitic stainless steels, and its presence is aimed at reducing or preventing the precipitation of carbides of the $M_{23}C_6$ type [27]. In these steels for fast breeder reactors, the MC carbides can introduce precipitate–matrix interfaces for an efficient trapping of vacancies and helium, which can improve and enhance their resistance to irradiation-induced void swelling [28]. In this work, the (Ti, Mo)C particles should be present as secondary precipitates. For these Ti-stabilized steels, the primary particles always form during solidification, in which part of the primary carbides can be dissolved during the solution heat treatment above 1050 °C and then reprecipitate as fine secondary precipitates during the annealing treatment [8].

After the age treatments at 700 °C for different periods, a new kind of precipitates formed, as shown in Fig. 2. The new phase morphology was blocky and appeared bright due to higher amounts of heavy Mo and Cr atoms, as the EDS results shown in Fig. 2f. Considering the precipitation temperature and the chemical components of the new phase, this is most likely the σ -phase. The amount of the new phase increases gradually with the aging time: however, the size of the precipitates remains the same with aging times above 96 h, according to the time-dependent backscattered electron (BSE) images shown in Fig. 2a–e.

To confirm the nature of these new precipitates, the TEM technique was used. As presented in Fig. 3, after the aging treatment at 700 °C for 168 h, some precipitates appear along the grain boundaries, others form around (Ti, Mo)C, and both of them have high-density stacking faults. The crystal structure of the precipitates is identified to be

tetragonal lattice by indexed SADPs, with lattice constants of $a = b = 0.82$ nm and $c = 0.5$ nm. Conversely, the new bright bulk precipitates are confirmed to be of σ -phase.

The precipitation occurred in the traditional manner of austenitic stainless steels, where the σ -phase always precipitates at grain boundaries (GB) and triple points of the austenitic matrix. Conversely, in austenitic steels containing residual δ ferrite, the σ -phase precipitates easily at the δ/γ interphase boundaries [29], because the δ/γ interphase is a high boundary energy place and leads to a beneficial site for the heterogeneous nucleation of the σ -phase. Besides the grain boundaries, the σ -phase particles also precipitate around the (Ti, Mo)C particles in this work. Moreover, the chemical composition (wt%) of the σ -phase at the grain boundaries was different from that at (Ti, Mo)C/matrix interphase boundaries, as shown in Table 1. The molybdenum content of the σ -phase within the matrix is much higher than that present at the grain boundaries, while the contents of other elements are almost the same. The molybdenum content of the MC phase within the matrix is lower than that at the grain boundaries, as shown in Table 2. The formation of the σ -phase in matrix with high molybdenum content is primary due to the consumption of some molybdenum element from nearby MC phase. The molybdenum plays a significant role in the formation of the σ -phase and is also an important constituent of the MC phase [18, 30]. Thus, the MC phase may have some effect on the σ -phase nucleation and growth, an effect which will be discussed latter.

To analyze the volume fraction and length variation of the σ -phase, more than ten BSE images of each sample were quantified using the Sic IAS image analysis. When samples were isothermally aged at 700 °C, the volume fraction of the σ -phase increased linearly with the aging time before 144 h and then gradually plateaued between 144 and 168 h, as shown in Fig. 4. Padilha *et al.* [21] believed that the σ -phase grown up in austenitic stainless steel had slow formation kinetics during the initial aging period. This is primarily because the crystal structure of the

Fig. 1 **a** BSE micrographs of the cold-rolled sample **BF STEM** image of the cold-rolled sample; **b** BF STEM image; **c** the TEM-line analysis of MC-type carbide

Fig. 2 BSE micrographs of the samples with different aging treatments: **a–e** samples aged at 700 °C for 72, 96, 120, 144 and 168 h; **f** SEM-EDS analysis of a new precipitate phase

σ -phase is complex and quite different from that of the austenitic matrix. In this situation, the nucleation of the σ -phase is difficult and should require extended diffusion times to grow up.

In addition, when samples were isothermally aged from 72 to 168 h at 700 °C, the average length of the σ -phase increased a little with the aging time and reached

approximately 0.44–0.47 μm , as shown in Table 3. This is mainly because that σ -phase is also very rich in substitutional elements, such as Fe, Mo and Cr, while the diffusion of these elements is very slow in austenite. According to the diffusion theory, the diffusion coefficient D given by $D = D_0 \exp(-Q/(RT))$ [31] (D_0 : diffusion constant, Q : diffusion activation energy, R : gas constant, and T :

thermodynamic temperature) exhibits an exponential relationship with temperature T . However, under isothermal conditions the diffusion coefficients of these alloying components are constant, so that their diffusion requires extended times. Hence, the σ -phase particles grow up very sluggishly and generally require hundreds of hours.

3.2 Influence of Isothermal Treatment on the Precipitation of the σ -phase

In order to monitor the growth of the σ -phase precipitates, isochronal annealing studies were carried out for 96 h at 600, 700 and 750 °C. Figure 5 shows the BSE images of samples after annealing at different temperatures. As shown in Fig. 5a, no new phase was detected when the alloy was aged at 600 °C for 96 h. The σ -phase appeared when the aging temperature reached 700 °C and quickly grew to a size of about 1.5 μm in the specimens aged at 750 °C, as illustrated in Fig. 5b and c. Figure 6 shows the

differences in the maximum amounts of σ -phase precipitated in the specimens aged at 600, 700 and 750 °C. The σ -phase particles were too small to be measured when the samples were aged at 600 °C for 96 h. When the specimens were aged at 700 °C, the fraction of σ -phase was nearly 0.3 vol%. However, in specimens aged at 750 °C, nearly 1 vol% of the σ -phase precipitated after 96 h (Fig. 6).

To further demonstrate the growth tendency of the σ -phase, Table 4 shows the average length of the σ -phase in the steel aged for 96 h at different temperatures: 600, 700 and 750 °C. The average length of the σ -phase was approximately 0.44 μm at 700 °C, while in the sample aged at 750 °C for 96 h, the average length of the σ -phase was approximately 1.5 μm , which is three times longer than that in the sample aged at 700 °C for 96 h. According to the diffusion theory, the diffusion coefficients of Cr and Mo show an exponential relationship with the aging temperature T , so that they increase sharply when the aging temperature rises from 600 to 750 °C. It can be deduced

Fig. 3 TEM micrograph of the sample aged at 700 °C for 168 h

Fig. 4 Volume fraction of the σ -phase as a function of the isothermal aging time of 72–168 h at 700 °C

Table 1 The chemical compositions (wt%) of the σ -phase from EDX measurements

Element	Ti	Cr	Mn	Ni	Mo	Si	V	Fe
σ -matrix GB	1.31	10.98	1.119	6.478	34.41	4.103	0.111	Bal.
σ -around MC	1.097	11.61	1.994	5.787	42.91	2.323	0.891	Bal.

Table 2 The chemical compositions (wt%) of the MC phase from EDX measurements

	C	Si	Ti	V	Cr	Mn	Ni	Mo	Fe
MC-in matrix	18.3	0.12	16.4	0.06	3.86	1.53	8.81	5.75	Bal.
MC-GB	29.1	–	57.5	1.05	0.29	–	–	11.46	Bal.

Table 3 Average length of σ -phase

Sample	700 °C-72 h	700 °C-96 h	700 °C-120 h	700 °C-144 h	700 °C-168 h
Length of σ -phase (μm)	0.45	0.44	0.47	0.45	0.47

Fig. 5 BSE micrographs of the samples with different aging temperatures: **a** 600 °C, **b** 700 °C, **c** 750 °C for 96 h **Fig. 6** Volume fraction of the σ -phase as a function of the isochronal aging temperature of 600, 700 and 750 °C for 96 h

that the aging temperature had a great influence on the growth of the σ -phase.

As mentioned above, the σ -phase particles precipitated intergranularly and formed around the (Ti, Mo)C particles. Taking the samples aged at 600, 700 and 750 °C for example, the σ -phase was not observed after aging at 600 °C for 96 h (Fig. 7a), and there were still many dislocations around the (Ti, Mo)C particles, the same as for the cold-rolled microstructure (Fig. 1b). In the sample aged

at 700 °C for 96 h (Fig. 7b), the σ -phase particles precipitated along the interface between the matrix and the (Ti, Mo)C particles. As the aging time was prolonged to 144 h (Fig. 7c), more σ -phase particles were formed, and their size almost did not change with an increase in the aging time. In the sample aged at 750 °C for 96 h (Fig. 7d), the σ -phase particles grew up fast along the interface between the matrix and the (Ti, Mo)C particles. The biggest σ -phase particle was almost 822 nm long, which was consistent with the SEM results (Fig. 5). Although the σ -phase has a tetragonal structure and the (Ti, Mo)C the FCC structure, the σ -phase is found to have some orientation relationship (OR) with (Ti, Mo)C, as shown in Fig. 7e. The (-1-11) direction of (Ti, Mo)C is parallel to the (022) direction of the σ -phase. A part of the σ -phase and (Ti, Mo)C is imaged under high-resolution (HR) TEM conditions, which gives a further proof of the OR between the σ -phase and (Ti, Mo)C, as shown in Fig. 7f.

The equilibrium phase mass fractions of the 15Cr–15Ni titanium-modified austenitic stainless steel studied here were calculated by using the Thermo-Calc software as a function of temperature from 500 to 1400 °C, as displayed in Fig. 8. The austenitic phase first precipitates from the liquid phase without any δ ferrite phase formation during the solidification process. The (Ti, Mo)C begins to precipitate below 1300 °C, whereas the σ -phase, Ni_3Ti and M_3B_2 precipitate from the austenitic matrix at 500–800 °C. It should be noted

Table 4 Average length of the σ -phase

Sample	600 °C-96 h	700 °C-96 h	750 °C-96 h
Average length of σ -phase (μm)	–	0.44	1.53

Fig. 7 TEM micrographs, SAED of samples with different aging treatments: TEM micrographs: **a** 600 °C for 96 h; **b**, **c** 700 °C for 96 h and 144 h; **d** 750 °C for 96 h; **e** SAED pattern of the (Ti, Mo)C and σ -phase in Fig. 7d; **f** HRTEM image of the σ -phase and the (Ti, Mo)C with the arrow in Fig. 7d

that in the 20% cold-rolled specimen (Fig. 1), the main precipitates were (Ti, Mo)C and no delta ferrite was founded before the σ -phase precipitated, which is consistent with the calculated results. The (Ti, Mo)C may play an important role

in the σ -phase precipitation. During the thermal aging treatment, the initial (Ti, Mo)C can act as nucleation sites for the σ -phase. Firstly, the (Ti, Mo)C particles formed on the grain boundaries and in the matrix during the sample

Fig. 8 15Cr–15Ni titanium-modified austenitic stainless equilibrium phase fractions of precipitates calculated by Thermo-Calc software

Fig. 9 TEM micrographs of the samples aged 750 °C for 96 h; high fault density in σ -phase precipitated on **a** grain boundary and **b** at the interface of (Ti, Mo)C and matrix

preparation process, which punched out dislocations into the surrounding matrix. The interface between (Ti, Mo)C and the matrix has a high deformation energy, which is beneficial to the σ -phase formation. Secondly, the σ -phase is a Mo- and Cr-rich intermetallic phase without C (Fig. 2f). Meanwhile, the formation of (Ti, Mo)C can decrease the C content of the adjacent matrix, which creates a feasible condition for the σ -phase formation. The major constituents of (Ti, Mo)C are Ti, Mo with sporadic inclusions of Cr, Ni and Fe (Table 2). The most important thing is that the formation of the MC phase does not need the supply of Mo. However, after the MC-type carbides has precipitated, the (Ti, Mo)C carbide has a strong affinity for Mo and accommodates this element up to 43 at% [32]. The carbides with high Mo contents can act as nucleation sites for the σ -phase precipitation, and Mo can easily diffuse into the σ -phase, which facilitates its growth. Finally, the precipitates formation can be controlled by solute

diffusion in the bulk, pipe diffusion through dislocations and interfacial reactions at the precipitate–matrix boundaries [33]. In Fig. 1b, a large amount of dislocations was observed around (Ti, Mo)C in the cold-rolled specimen. When the sample is aged at 600–750 °C, the components of the σ -particles such as Mo and Cr preferentially move along the connecting dislocations pinned around the carbides interfaces, which also form ideal sites for the σ -phase nuclei. It is worth to note that near the σ -phase, there was no new phase close when the σ -phase precipitated. Hence, the precipitation of the σ -phase followed the reaction: $\gamma \rightarrow \sigma$, which is considered as a new precipitation mechanism of the σ -phase in 15Cr–15Ni titanium-modified austenitic stainless steel.

Figure 9 shows a high density of stacking faults in the σ -phase precipitated on grain boundaries and at the interface between (Ti, Mo)C and the matrix. Faults in atomic stacking in a crystal may be caused by the movement of partial

dislocations occurring in the crystal or may have been introduced during the growth of the new crystal. In our study, the presence of stacking faults in σ -phase is in favor of the latter mechanism. TiC precipitates had large positive misfit of 21% in the austenitic matrix, and the strain energy arising out of this large misfit should be relieved by the nucleation of the σ -phase, which has a high density of stacking faults [34]. Clearly, further studies are still needed to understand the formation mechanism of the stacking faults in the σ -phase.

4 Conclusions

The σ -phase precipitation mechanism in the 20% cold-worked 15Cr–15Ni titanium-modified austenitic stainless steel during long-term thermal exposure between 600 and 750 °C has been investigated.

1. During the isothermal aging stage, the amount of σ -phase precipitated increased when the samples were aged at 700 °C for less than 144 h. After 144 h, the amount of σ -phase gradually became constant, and the average length was almost unchanged. The aging time only slightly affected the size and morphology of the σ -phase.
2. During the isochronal aging stage, the σ -phase grew rapidly with the increase in the aging temperature. In the sample aged at 750 °C for 96 h, the average length of the σ -phase was approximately 1.5 μm , which is three times longer than that measured in samples aged at 700 °C for 96 h. The aging temperature significantly affected the size of the σ -phase.
3. The σ -phase with a large amount of stacking faults was prone to nucleate around the (Ti, Mo)C particles or at the grain boundaries. The (Ti, Mo)C particles acted as effective nucleation sites for σ -phase formation during the aging treatment, when the σ -phase directly precipitated from the austenitic matrix with no other new phase. The σ -phase precipitation followed the new reaction: $\gamma \rightarrow \sigma$.

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