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Mechanism of $\text{M}_{23}\text{C}_6 \rightarrow \text{M}_7\text{C}_3$ carbides reaction of Cr35Ni45Nb type alloy during carburization

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Abstract

The carbide transformation of Cr35Ni45Nb type alloy during service has been investigated in the present work. The primary carbide of the as-cast Cr35Ni45Nb type alloy is $\text{M}_7\text{C}_3$ (M is mainly Cr element) and NbC, which transformed into $\text{M}_{23}\text{C}_6$ type carbides (M is mainly Cr element) and G phase (Ni$_{16}$Nb$_6$Si$_7$) after service, respectively. The G phase and $\text{M}_{23}\text{C}_6$ carbides mix together and grow up with each other during service. After carburization, the crystal structure of $\text{M}_{23}\text{C}_6$ type carbide changes again and transforms into $\text{M}_7\text{C}_3$ type carbide. The mechanism of $\text{M}_{23}\text{C}_6 \rightarrow \text{M}_7\text{C}_3$ carbide reaction is an in situ transformation; the $\text{M}_7\text{C}_3$ type carbide preferentially nucleates at the interface of $\text{M}_{23}\text{C}_6$/γ and grows toward the $\text{M}_{23}\text{C}_6$ type carbide interior. With the diffusion of free carbon atoms into the $\text{M}_7\text{C}_3$ type carbides, the $\text{M}_7\text{C}_3$ type carbides are gradually surrounded by the $\text{M}_7\text{C}_3$ type carbides until they are completely changed into $\text{M}_7\text{C}_3$ type carbides.

1. Introduction

Ethylene pyrolysis furnace tubes are used in high temperature (1173 K to 1373 K) and high carbon environments, which are welded through the centrifugal cast steel tubes [1]. Therefore, the ethylene pyrolysis furnace tube alloy should exhibit good high temperature creep strength, excellent high temperature corrosion resistance, superior machinability, weldability and high-metallurgical stability [2]. The ethylene pyrolysis furnace tube materials have developed from HK40 to Cr35Ni45Nb type alloys, and now Cr35Ni45Nb type alloy is the mainly alloy, which are used widely in ethylene pyrolysis furnace tube [3–9]. The service environment of the ethylene pyrolysis furnace tube is very harsh, and the furnace tube suffers from various damages, such as overheating [10], oxidation and carburization [1, 11–13], creep [14–16].

The service environment of the furnace tube is very complicated, so the damage in different zones of the furnace tube is also various. During service, the outer wall of the furnace tube is exposed to the high temperature flame, and a pyrolysis reaction of ethane or naphtha occurs in the furnace tube [1]. Hence, the microstructure of the inner and outer zones of the pyrolysis furnace tube undergoes different transformations during service. The primary eutectic carbides are coarsened, and a large number of secondary carbides are precipitated in the dendrite interior during service [7, 17–20]. The primary carbides of pyrolysis furnace tube alloy are $\text{M}_7\text{C}_3$ and NbC type carbides [20–25] and transform into $\text{M}_{23}\text{C}_6$ type carbides and G phases after exposed to high temperature, respectively. Meanwhile, a large number of secondary $\text{M}_{23}\text{C}_6$ type carbides precipitated in dendrite interior [26–28]. Due to the pyrolysis reaction of olefins occurs inside the furnace tube, the inner wall of the furnace tube is prone to carburization during service. The crystal structure of $\text{M}_{23}\text{C}_6$ type carbide changed again during the carburizing process.

Though the transformation of $\text{M}_{23}\text{C}_6$ type carbide to $\text{M}_7\text{C}_3$ type carbide has been reported in some scientific papers [29–31], the mechanism of this carbide transformation is not perfect and clear in ethylene pyrolysis furnace tube alloy. There are a number of scientific articles on the transformation of $\text{M}_7\text{C}_3$ type carbide to $\text{M}_{23}\text{C}_6$ type carbide in Cr25Ni35Nb type alloys [27, 32–34]. Sergey Yu. Kondrat’ev has investigated the mechanism of
Although these researchers pointed out that the formation of M7C3 type carbide is caused by the diffusion of the service environments paper. Besides that, the carbide transformation of Cr35Ni45Nb type alloy ethylene furnace tube in different titanium are carbide formers and improve the metallurgical stability of Cr35Ni45Nb type alloy. Niobium can As shown in table 1, the content of Cr and C is very high in Cr35Ni45Nb type alloy. Chromium, niobium and silicon element in Cr35Ni45Nb type alloy, which doesn’t tend to form carbide. But silicon element can enhance the strength and hardness of the alloy through solid solution strengthening or controlling the grain boundary [2].

M$_2$C$_3$ carbide transformed into M$_{23}$C$_6$ carbide in HP type alloy [35]. The M$_2$C$_6$ carbides nucleate at the eutectic M$_7$C$_3$ carbide interior and grow up. The transformation mechanism of M$_2$C$_3$ type carbide change into M$_{23}$C$_6$ type carbide is an 'in situ' transformation. However, there are few scientific articles on the transformation of M$_{23}$C$_6$ type carbides to M$_7$C$_3$ type carbides after carburization in heat-resistant steel. For example, the carbide transformation of Ni–Cr–Fe Alloys in carburizing and oxidizing environments is studied by A Schnaas [30]. The transformation mechanism he proposed is that as a result of the initially present oxide layer was reduced by CO, the M$_7$C$_3$ carbides were formed on the out layer. The reduction reaction formula is as follows:

$$7\text{Cr}_7\text{O}_3 + 33\text{CO} = 2\text{Cr}_7\text{C}_3 + 27\text{CO}_2$$

Other researcher thought that free carbon atoms diffused into the M$_2$C$_6$ type carbide, and brought about M$_{23}$C$_6$ type carbides change into M$_7$C$_3$ type carbides of heat-resistant alloy during carburization [1, 31, 36, 37]. Although these researchers pointed out that the formation of M$_2$C$_3$ type carbide is caused by the diffusion of the free atom of carbon into M$_{23}$C$_6$ carbide, there is no definite evidence has been found in experiments or production, and no new transformation mechanism has been proposed. Hence, the mechanism of M$_{23}$C$_6$ carbide transform into M$_7$C$_3$ type carbide of the Cr$_{35}$Ni$_{45}$Nb type alloy during service was investigated in this paper. Besides that, the carbide transformation of Cr$_{35}$Ni$_{45}$Nb type alloy ethylene furnace tube in different service environments (the service environment of the inner and outer wall of the furnace is different), and the carbide transformation process during service also studied in this work. This research can not only guide the damage assessment of the ethylene pyrolysis furnace tubes after service, but also help understand the development law of the ethylene pyrolysis furnace tube damage during service.

2. Materials and experimental

The specimens are cut from the ethylene pyrolysis furnace tube after a five-year service. The operation pressure of the ethylene pyrolysis furnace tube is 0.2 ~ 0.4 MPa, and the working temperature is range from 1173 K to 1373 K, with ethane as feedstock. The chemical composition of specimens is determined by Optical Emission Spectrometer analyses, which are listed in table 1. Owing to carburization during service, the carbon content of the serviced tube is higher than the as-cast tube.

To characterize the microstructure, the specimens were electrolytical etched in a solution of oxalic acid (wt. 10%) at 5 V for 10 s after ground and polished. The microstructure and secondary phases are characterized by scanning electron microscopy (SEM) with an energy dispersive spectroscopy (EDS). In order to investigate the carbide transformation, electron back-scattered diffraction (EBSD) analyses were applied in order to identify the phases present. Firstly, the EBSD specimens were polished with a 1 µm diamond paste, and then vibratory polished with a 0.03 µm colloidal silica suspension for over 6 h. EBSD tests are carried out with a working distance of 13 mm and an extra high tension of 20 kV. The scanning step size is chosen as 0.5 µm.

3. Results

3.1. Microstructure of Cr$_{35}$Ni$_{45}$Nb type alloy after service

As shown in table 1, the content of Cr and C is very high in Cr$_{35}$Ni$_{45}$Nb type alloy. Chromium, niobium and titanium are carbide formers and improve the metallurgical stability of Cr$_{35}$Ni$_{45}$Nb type alloy. Niobium can significantly increase the high temperature creep strength of the heat-resistant alloy. There is a high content of silicon element in Cr$_{35}$Ni$_{45}$Nb type alloy, which doesn’t tend to form carbide. But silicon element can enhance the strength and hardness of the alloy through solid solution strengthening or controlling the grain boundary [2].

The microstructure of the as-cast and serviced furnace tube is shown in figure 1. Cr$_{35}$Ni$_{45}$Nb type alloy furnace tube has a higher C and Cr content, which are produced by centrifugal casting. Hence, there are a large number of lamellar carbides formed in the Cr$_{35}$Ni$_{45}$Nb type alloy furnace tube [12, 38]. In the as-cast Cr$_{35}$Ni$_{45}$Nb type alloy furnace tube, the skeleton shape eutectic carbides are distributed at the dendrite boundaries, consisting of γ-phase and carbides (figure 1(a)). The microstructure of ethylene pyrolysis furnace
tube has deteriorated after a long term service. The service environment of the inner and outer walls of the furnace tube is quite different; resulting in the difference in microstructure of the inner and outer zones of the furnace tube. Figure 1(b) shows the microstructure of the outer zone in Cr35Ni45Nb type alloy furnace tube after service. The lamellar shape carbides of Cr35Ni45Nb type alloy grew to blocky shape and lead them to form a network structure. A large number of secondary carbides precipitated in the dendrite interior, and some of these secondary carbides have grown to blocky shape. The microstructure of the inner zone in the serviced Cr35Ni45Nb type alloy furnace tube is shown in figure 1(c). The primary carbides formed a coarse continuous network structure and the secondary carbides agglomerated together to big blocky shape. The width of the primary carbides is approximately 8 μm, and the mean size of the blocky-shape secondary carbides is about 7 μm (figure 1(c)). After service, the lamellar and skeleton shape carbides in the furnace tube are coarsened and replaced by the continuous network structure.

The chemical composition of carbide was analyzed through EDS in figure 1. Table 2 is the chemical composition of carbides in figure 1. There are two type carbides in the as-cast furnace tube, which are M7C3 type carbide and NbC type carbide (table 2). The carbides are M23C6 type carbide and G phase with a blocky shape in the outer zone of the serviced furnace tube. While, the continuous network shape carbides in the inner zone of the serviced furnace tube are all M7C3 type carbides.

3.2. Carbides of Cr35Ni45Nb type alloy after service

The Cr35Ni45Nb type alloy furnace tube solidified rapidly and formed many non-equilibrium solid phases during casting. The eutectic carbide consists of M23C6 type carbide, M23C6 type carbide and NbC carbide, the predominant carbide is M7C3 type carbide (figure 2(a)). A small amount of M23C6 and NbC carbides are distributed on the edge of M7C3 carbides and γ matrix. The M23C6 type carbides are metastable phases and would transform into M23C6 type carbides at high temperature.

Figure 2(b) shows the carbide distribution in the outer zone of ethylene pyrolysis furnace tube. After exposed to high temperature, the metastable phase M7C3 type carbide transformed into M23C6 type carbide. Meanwhile, a lot of secondary carbides (M23C6) are precipitated in the dendrite interior, which are equilibrium phases formed in Cr35Ni45Nb type alloy during service (figure 2(b)). And NbC carbide also changed into G phase in Cr35Ni45Nb type alloy during service. At the boundary between the γ matrix and primary carbide, G phases have been precipitated and grew together with M23C6 type carbides (figure 2(b)). After a five-year service, there are only a few M7C3 carbides in the outer zone of Cr35Ni45Nb type alloy furnace tube, which have not transformed into M23C6 type carbides. After a long term service, not all NbC carbides have transformed into G phase, there are still some fine NbC carbides in the dendrite interior and mix together with M23C6 type carbides (figure 2(b)). The primary carbides formed a continuous network structure and the secondary carbides precipitation lead to the microstructure deterioration of Cr35Ni45Nb type alloy.

| Element (at.%) | C  | Si  | Mn  | Cr  | Ni  | Nb  | Ti  | Fe  |
|---------------|----|-----|-----|-----|-----|-----|-----|-----|
| A             | 54.65 | 1.35 | 0.92 | 39.86 | 3.22 |
| B             | 19.65 | 2.61 | 0.61 | 30.25 | 35.22 | 11.66 |
| C             | 29.18 | 68.66 | 0.61 | 1.55 |
| D             | 16.67 | 38.11 | 31.79 | 11.30 |
| E             | 39.69 | 55.54 | 0.50 | 4.28 |
| F             | 38.34 | 58.29 | 0.37 | 3.00 |

Table 2. The chemical composition of carbides.
The carbide distribution of the pyrolysis furnace tube inner zone is shown in figure 2(c). The primary carbides grew bigger and the secondary carbides grew to blocky shape after carburization. In the carburization zone of Cr35Ni45Nb type alloy furnace tube, most of the M23C6 type carbides have transformed into the M7C3 type carbides, but there is still a part of M23C6 type carbides have not changed, which are at the edge of the M7C3 type carbides (figure 2(c)). A large number of G phase mixed together with M7C3 type carbides, and some NbC carbides are located at the edge of massive M7C3 type carbides (figure 2(c)). The carbides undergo different crystal structure transformation in the inner and outer zones of the furnace tube during service.

3.3. Carbide transformation of Cr35Ni45Nb type alloy during carburization

Since the raw materials for producing ethylene are mainly hydrocarbons, the ethylene pyrolysis furnace tube is exposed to a high-temperature and high-carbon environment for a long time, so the inner zone of the furnace tube prone to carburization during service. The M23C6 type carbides would transform into M7C3 type carbides after the free carbon atom diffused into the furnace tube interior [1, 11, 31]. Figure 3 shows the boundary between carburization zone and non-carburization zone of Cr35Ni45Nb type alloy furnace tube after service. The primary carbides have grew to big blocky shape and formed a continuous network structure (figure 3(a)). The M23C6 type carbides have transformed into M7C3 type carbides during carburization, and the M7C3 type carbides preferentially nucleated at the boundary between M23C6 and γ matrix (figure 3(b)). A large number of M7C3 type carbide particles formed at the edge of the big M23C6 type carbide. The M7C3 type carbide particles are at the interface between M23C6 type carbide and matrix grow up around the M23C6 type carbides, and eventually engulfed the adjacent M23C6 type carbide. At last, the M23C6 type carbides are completely changed into the M7C3 type carbides.

4. Discussion

The service environment of furnace tube is complicated and various. The study of microstructure deterioration of Cr35Ni45Nb type alloy is important to ensure the safe operation of the ethylene pyrolysis furnace tube. Due to a variety of service environment, the crystal structure of carbides also transformed several times. The as-cast pyrolysis furnace tubes are made by centrifugal casting and the solidification time is very short. Hence, there are many non-equilibrium solid phases in the furnace tube alloy, such as the eutectic carbides. Owing to the high content of C and many carbide former elements (Cr, Fe, Nb), the eutectic carbides are consist of metastable carbides.
carbides ($M_7C_3$ type carbide), NbC and some $M_{23}C_6$ type carbides [20–25]. After exposed to high temperature, the metastable $M_7C_3$ type carbide and NbC transformed into $M_{23}C_6$ type carbide and G phase, respectively. Meanwhile, a large number of $M_{23}C_6$ type carbides precipitated in dendrite interior [26–28]. Naphtha and ethane, which are raw materials for the ethylene production, are almost completely cracked in a steam cracker at 1173 K ~ 1373 K [1]. Therefore, the inner zone of the pyrolysis furnace tube would be carburized during service. As the free carbon atom diffused into the alloy, the $M_{23}C_6$ type carbide changed into $M_7C_3$ type carbide [29–31]. The carbide transformation of the ethylene pyrolysis furnace tube alloy is that the primary carbide $M_7C_3$ changed into $M_{23}C_6$ after exposed to high temperature, and then the primary and secondary carbides $M_{23}C_6$ changed into $M_7C_3$ after carburization.

A variety of carbide transformations of Cr35Ni45Nb type alloy during service, which consist of $M_{23}C_6 \rightarrow M_7C_3$, NbC $\rightarrow$ G phase (high temperature) and $M_{23}C_6 \rightarrow M_7C_3$ (carburization). In order to study the transformation process of $M_{23}C_6$ type carbide to $M_7C_3$ type carbide, the interface between carburization zone and non-carburization zone of Cr35Ni45Nb type alloy after service has been analyzed through EBSD (figure 3). The schematic of carbide transformation process of Cr35Ni45Nb type alloy during service is shown in figure 4. The primary carbides in the as-cast Cr35Ni45Nb type alloy tube are mainly $M_7C_3$ and NbC type carbides. The $M_7C_3$ and NbC type carbides are metastable phases at high temperature and would change into $M_{23}C_6$ type carbides and G phases during service, respectively. The $M_7C_3$ type carbide is unstable polycrystalline structure carbide, and $M_{23}C_6$ type carbides first nucleate at the interface of $M_7C_3$ type carbides [35]. The carbon atoms of the $M_7C_3$ type carbides diffuse outward and the chromium atoms of the austenite matrix diffuse to the interface between the $M_7C_3$ type carbide and the austenite matrix to form $M_{23}C_6$ type carbide. Then the $M_{23}C_6$ type carbides grow bigger gradually. A large number of fine secondary $M_{23}C_6$ type carbide particles are also precipitated in the dendrite, and the secondary $M_{23}C_6$ type carbides grow into blocky shape as time goes on. Some small pieces of austenite are surrounded by $M_{23}C_6$ carbides, austenite remains in the carbides with the growth of $M_{23}C_6$ type carbides (figure 2). The NbC carbide also transformed into G phase during high temperature service (figure 2).

The inner part of the ethylene pyrolysis furnaces tube is exposed to the high carbon potential environment, and the carburization is inevitable during service. In the carburizing process, the free carbon atom diffuses into Cr35Ni45Nb type alloy furnace tube. When the carbon content of the austenite matrix is too high, the free carbon atom diffuse into the carbides, the $M_{23}C_6$ type carbides begin to change into $M_7C_3$ type carbides. The $M_7C_3$ type carbides first nucleate at the interface of $M_{23}C_6$/$\gamma$, and then grow toward the $M_{23}C_6$ carbides interior. With the continuous diffusion of the free carbon atom, $M_{23}C_6$ type carbides are gradually surrounded by $M_7C_3$ type carbides until they transformed into $M_7C_3$ type carbides completely.
5. Conclusion

The carbide transformation of Cr35Ni45Nb type alloy ethylene pyrolysis furnace tube after a long time service was investigated in present work. The main conclusion as follows:

(1) The M₂C₃ and NbC type carbide of Cr35Ni45Nb type alloy changed into M₂₃C₆ type carbide and G phase during service, respectively. A large number of secondary carbides (M₂₃C₆) are precipitated in the dendrite after service, and grow together with G phases. Not all NbC carbides have changed into G phases after service, and there are still some fine NbC carbides in the dendrite interior and mix together with M₂₃C₆ carbides.

(2) During service, the carbide transformation process of Cr35Ni45Nb type alloy pyrolysis furnace tube is as follows: M₂C₃ \rightarrow \text{Service at high temperature} \rightarrow \text{Carburization} \rightarrow M₂₃C₆, G.

(3) The M₂₃C₆ \rightarrow M₂C₃ carbides reaction is an \textit{in situ} transformation. The M₂C₃ type carbides first nucleate at the interface of M₂₃C₆/γ and gradually replace M₂₃C₆ type carbide during carburization.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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