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Effect of aging temperature on structural evolution of HP-Nb heat resistant steel

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Abstract

In this study, the changes in microstructure of HP-Nb cast heat resistant steel after 8 years of service at high temperatures were investigated. Study of microstructure using optical microscopy (OM) and scanning electron microscopy (SEM) equipped with an energy dispersive spectrometry (EDS) showed that the microstructure of this steel in as-cast condition includes an austenitic matrix, and that continuous eutectic-type precipitates in grain boundaries. These deposits include $M_{23}C_6$ chromium carbide and NbC. Aged sample microstructure at intermediate temperature (700-900 °C) consists of an austenitic matrix, $M_{23}C_6$ chromium carbides and blocky G-phase The reason for this microstructure is the instability of NbC during aging at medium temperature 700-1000 °C which, as a partially phase transformation, is transformed to G-phase. But in the samples aged at higher temperatures (1000-1100 °C), there are only two types of NbC and agglomerated $M_{23}C_6$ carbide precipitations, and the skeletal morphology of NbC carbides has become spherical.

Keywords: HP-Nb cast heat resistant steel, microstructure, chromium carbide, G-phase.

1- Introduction

Cast heat resistant steels are generally used at service temperatures above 650°C [1)]. Due to the metallurgical stability of these steels at high temperatures, their corrosion resistance, excellent creep strength and low cost compared to the nickel-based superalloys, cast heat resistant steels have many applications in different industries [2)]. One important application of cast heat resistant steels is radiant reformer tubes employed in petrochemical industries and direct reduction unit of steelmaking plants. Reformer tubes of direct reduction unit in Mobarakeh Steel Company are made from two types of cast heat resistant alloys with centrifugal casting method one of which is the HP-Nb.

HP-Nb alloy (25Cr-35Ni-1 Nb) is the most widely used and most successful steel grade in this category [1)]. Although this alloy is selected and designed for use in high temperature condition, after long-term service at temperatures above 800°C, a significant change occurs in its microstructure and mechanical properties [3)].

In this study, microstructural changes of HP-Nb heat resistant alloy aged after 8 years of service exposure at different temperatures have been investigated. In addition, the types of phases and carbides in this alloy at different temperature conditions were determined and compared with the as-cast microstructure.

2- Experimental procedure

HP-Nb cast heat resistant alloy was investigated. Chemical composition of this alloy is given in Table 1. For metallographic evaluation, the three samples with dimensions 2×2×2cm at high temperature aged (1000-1100° C), intermediate temperature aged (700-900 ° C) and as-cast conditions, were prepared from a HP-Nb reformer tube. All specimens were machined at the inner tube surface, then they were ground on silicon carbide paper of 80-1200 grit and polished on a nylon cloth with 0.3 μm alumina. The Marbles reagent (10 g $CuSO_4$ + 50 CC HCl + 50 CC H_2O) was used for revealing the microstructure.

Table1. Chemical composition of HP-Nb cast heat resistant steel (wt %).

Element	C	Si	Mn	Cr	Ni	Nb	Fe
Amount (wt%)	0.42	1.04	1.01	23.09	34.41	1.25	bal.

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To evaluate the microstructure of samples, optical microscopy (OM) and scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy system (EDS) were used.

3- Results and discussion

Microstructure of as-cast HP-Nb heat resistant steel at two different magnifications are shown in Fig. 1.

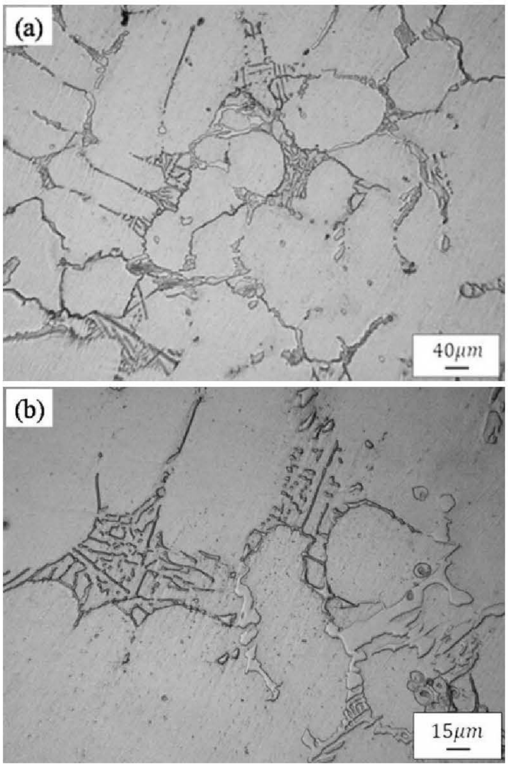

Fig. 1. (a), (b) Microstructure of as-cast HP-Nb steel at two different magnifications using OM.

As seen in this figure, the microstructure consists of an austenitic matrix with an almost continuous network of carbides in the grain boundaries.

SEM image of as-cast HP-Nb steel, and EDS analysis results of precipitates are shown in Fig. 2.

Investigation by SEM with backscattered electrons (BSE) detector showed a network of primary carbides in the grain boundaries which consists of two types of carbides as dark and bright locations. EDS analysis shows that dark carbides are chromium-rich. With consideration of the Nb content percentage in the alloy and high cooling rate in centrifugal casting, this type of carbide is $M_{23}C_6$ carbide. EDS analysis of bright precipitates also shows that these particles are NbC carbides. The difference in color of these two types of carbides is due to the fact that niobium has greater atomic weight than chromium [3, 4, 5].

Fig. 3 illustrates the microstructures and EDS analysis results of phases in the aged sample at intermediate temperature (700-900 ° C). As can be seen, three types of phases with different chemical compositions have been induced in this sample during 8 years of service exposure. The reason for this microstructure is the instability phenomenon of NbC during aging at medium temperature 700-1000 °C which, as a partially phase transformation, is transformed to G-phase. Transformation of NbC carbide to Ni-Nb silicide known as the G phase occurs at temperatures from 700 to 1000 °C. G phase with FCC crystallographic structure and lattice parameter equal to 1.13 nm has the stoichiometry of $Ni_{16}Nb_7Si_6$. EDS analysis also shows that iron and chromium can be solved in this silicide [6, 7]. However, due to the fact that temperature is not high enough, NbC carbides have not been transformed completely. Also, the chromium carbides somewhat have missed their grain boundary distribution mode.

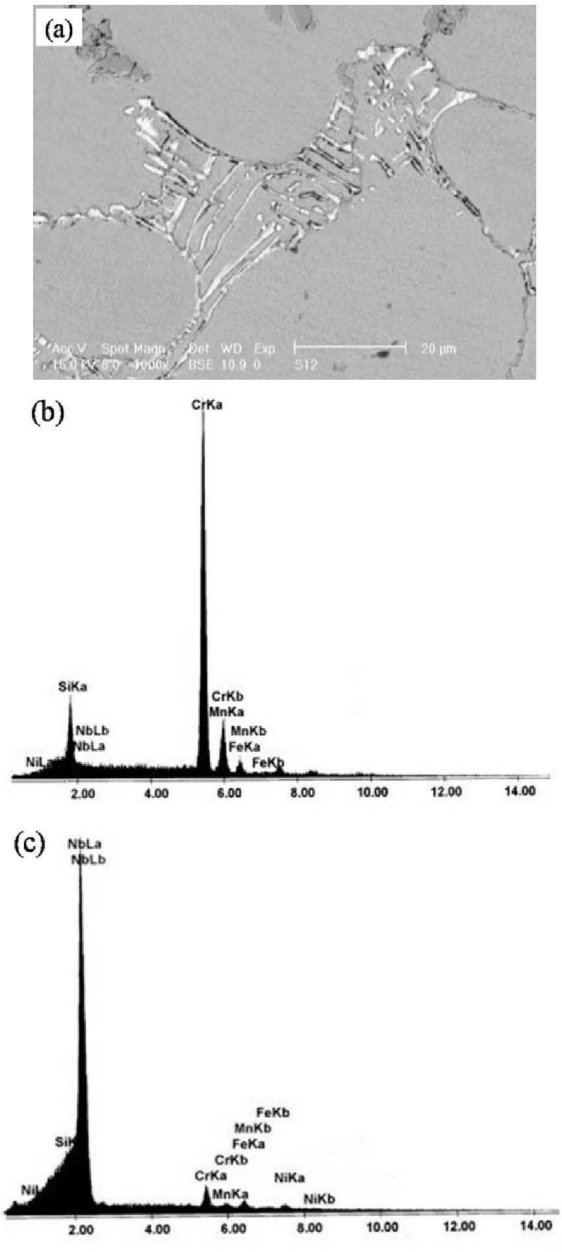

Fig. 2. Microstructure and existent phases in as-cast HP-Nb steel: (a) SEM image (BSE), (b) EDS micrograph of chromium carbide, (c) EDS micrograph of niobium carbide.

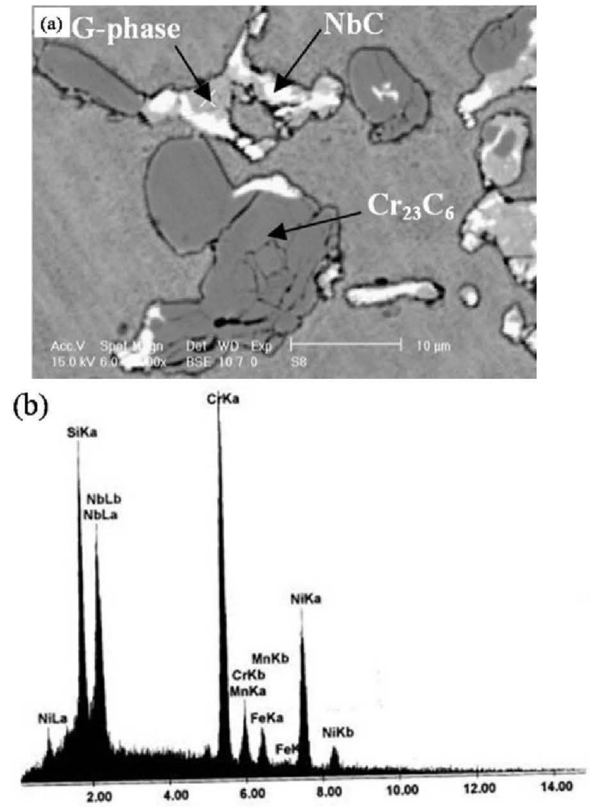

Fig. 3. Microstructure and existent phases in intermediate temperature (700-900 °C) aged HP-Nb steel: (a) SEM image (BSE), (b) EDS micrograph of G-phase.

In the microstructure of the sample aged at high temperatures (1000-1100°C), only two types of precipitates can be seen that include $M_{23}C_6$ carbides and NbC. Since the niobium carbide at service temperatures above 1000°C is stable, carbides NbC have not been transformed, and therefore no G-phase is seen [7].

In Fig. 4, microstructure and EDS elemental analysis results of samples aged at high temperatures is shown. According to SEM image of Fig. 4, dimensions of $M_{23}C_6$ carbides are much greater than NbC. In addition, the skeletal morphology of NbC carbides has become spherical. The reason for agglomeration and growing of $M_{23}C_6$ carbides during 8 years is the atomic hydrogen diffusion into the matrix crystal lattice of HP-Nb alloy during reforming operation of methane gas, that leads to an increase in diffusion rate; and ultimately coarsening of chromium- rich carbides is followed [8].

4- Conclusion

- Microstructure of HP-Nb cast heat resistant steel consists of an austenitic matrix with a continuous network of $M_{23}C_6$ and NbC primary carbides in grain boundaries.
- In the sample aged at intermediate temperature (700-900°C), there are three types of precipitates: $M_{23}C_6$, NbC and G-phase after 8 years of service. The reason for this microstructure is the instability phenomenon of NbC during aging at medium temperature 700-1000 °C which, as a partially phase transformation, is transformed to G-phase
- For samples aged at high temperatures (1000-1100°C), there are only two types of carbides: $M_{23}C_6$ and NbC. The skeletal morphology of NbC carbides has become spherical. The reason for agglomeration and growing of $M_{23}C_6$ carbides during 8 years is the atomic hydrogen diffusion into the matrix crystal lattice of HP-Nb alloy during reforming operation of methane gas that leads to an increasing diffusion rate; and ultimately coarsening of chromium- rich carbides is followed.

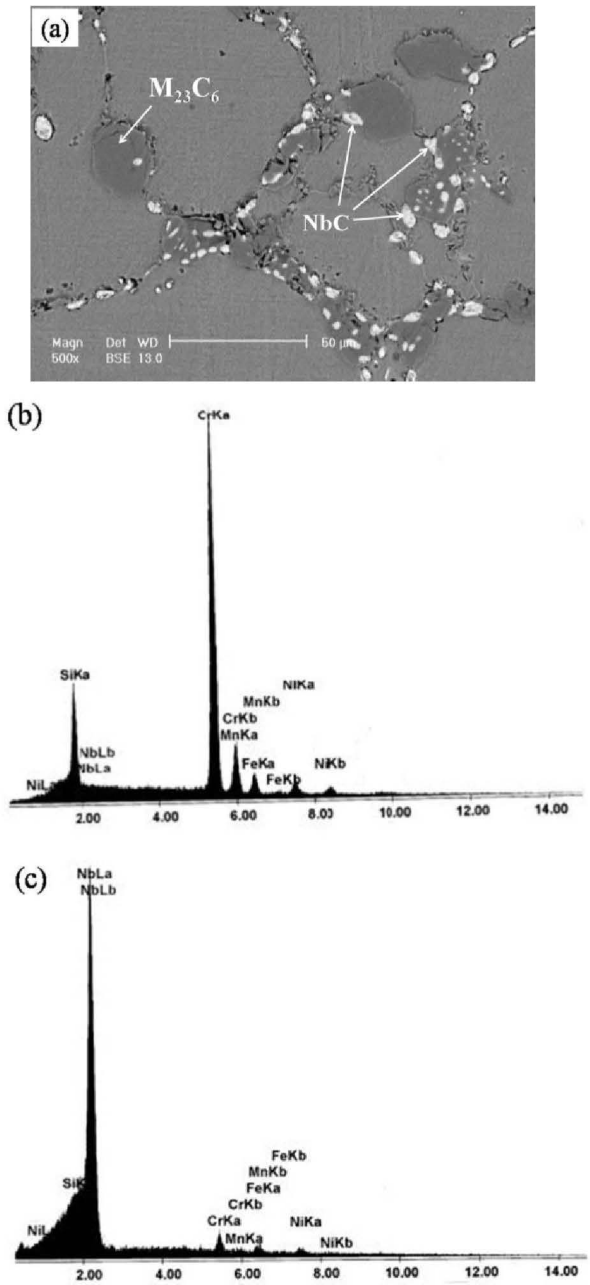

Fig. 4. Microstructure and existent phases in intermediate temperature (1000-1100 °C) aged HP-Nb steel: (a) SEM image (BSE), (b) EDS micrograph of chromium carbide, (c) EDS micrograph of niobium carbide.

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