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Effect of long-term thermal exposure on the hot ductility behavior of GH3535 alloy

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ABSTRACT

The hot ductility behavior of Ni–16Mo–7Cr alloys (named GH3535) exposed at 700 °C for different durations has been investigated by means of tensile test. It was found that the alloy exhibited a constant low ductility within the first 10 h exposure, and then showed an increasing ductility with the exposure time until 1000 h. After that, the ductility of the alloy decreased gradually with the increasing exposure time up to 10000 h. Detailed microstructural investigations using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM) have shown that the change in the ductility of the alloy with the exposure time could be attributed to the precipitation of $M_{12}C$ carbide at the grain boundary. Such precipitates with size of 200 nm, which are formed during the thermal exposure within 1000 h, can significantly restrain the grain boundary sliding and crack initiation, resulting in the high ductility of the alloy. Further exposure will cause the coarsening of the carbides, making them as the source of grain boundary cracks, hence decreases the ductility of the alloy.

1. Introduction

Molten salt reactor (MSR) has been considered as one of the most promising next generation nuclear reactors due to its incomparable merits, such as high security, desirable online refueling properties, minimization of nuclear waste, nuclear non-proliferation, etc. [1–3]. GH3535 alloy, a nickel based superalloy, is developed for thorium-base molten salt reactor (TMSR) applications due to its superior corrosion resistance and good mechanical properties [4–6]. It is solid solution strengthened by Mo and Cr. The microstructure consists of γ matrix and primary M_6C carbides.

The GH3535 alloy would be served at about 700 °C up to more than ten years in molten salt reactor systems. Therefore, the microstructure stability of the alloy is a major concern for designers. According to the previous research, the precipitation of $M_{12}C$ carbides was observed on the grain boundary in this alloy during the long-term exposure at 700 °C, and was beneficial to the creep properties of the alloy [6]. However, the influence of $M_{12}C$ carbides on ductility evolution of this alloy was not mentioned yet. Inouye et al. [7–9] investigated the high temperature stability of a Ni–Mo–Cr alloy via tensile test, and reported that the alloy exhibited good thermal stability for long-term exposure up to 10000 h in the temperature range from 538 to 760 °C. But what they paid close attention to was the tensile property of the alloy, and the deformation mechanism was not mentioned. Therefore, the effect of $M_{12}C$ carbides precipitated during the thermal exposure on hot ductility and deformation mechanism for this alloy haven't reported yet. In order to clarify their relation, it is necessary to carry out a systematic study on the hot ductility evolution of GH3535 alloy under long-term thermal exposure.

In this paper, the effect of long-term thermal exposure on hot ductility of GH3535 alloy was investigated at 700 °C. The evolution of deformation structure, dislocation characteristics, the size and morphology of secondary carbides, and their effects on the hot ductility of this alloy have been determined and discussed in detail.

2. Experiment

In this study, the nominal chemical composition (wt%) of the investigated alloy is: Mo, 16; Cr, 7; Fe, 4; Mn, 0.5; Si, 0.5; C, 0.05; Ni, balance. The master alloy was prepared by vacuum induction melt-furnace (VIM). The obtained ingot was hot forged and rolled into rods in the temperature range of 1150–1200 °C with the diameter of 16 mm. All the specimens were heat treated at 1177 °C for 40 min, followed by water quenching, and then exposed in furnace at 700 °C for various time up to 10000 h.

The cylindrical tensile specimens with the gauge length of

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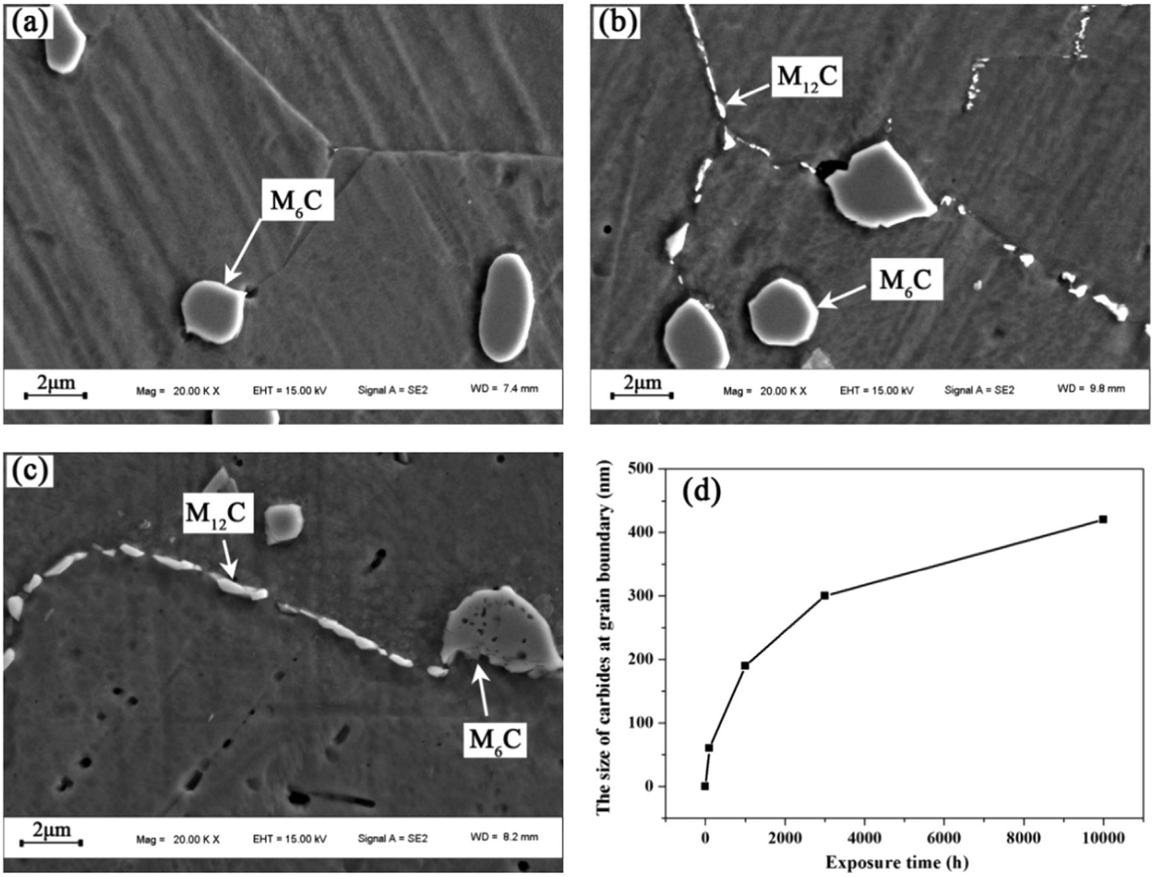

Fig. 1. Microstructure evolution of GH3535 alloy exposed at 700 °C for various time: (a) no exposure (0 h); (b) 1000 h; (c) 10000 h; (d) the size of $M_{12}C$ with exposure time.

Table 1
The evolution of chemical composition (wt%) inside the grain for the alloy exposed at 700 °C with various time.

Exposed time	Ni	Mo	Cr	Fe	Si
0 h	71.47	16.98	7.12	3.94	0.50
1000 h	71.27	16.75	7.23	4.29	0.46
10000 h	71.99	16.27	7.14	4.26	0.33

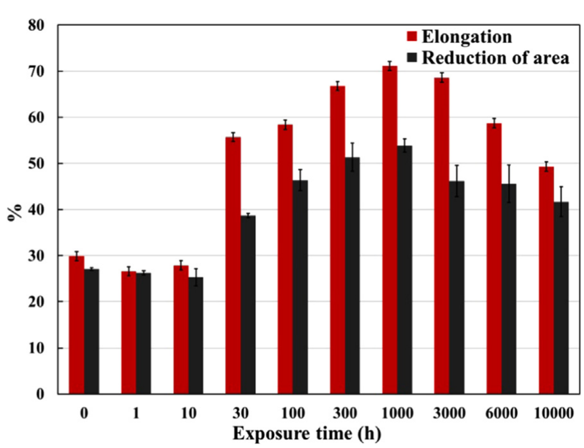

Fig. 2. Hot ductility of the long-term thermal exposure specimens tested at 700 °C.

20 mm and diameter of 5 mm were machined from the rods after thermal exposure. Tensile tests were performed at 700 °C in a universal tensile testing machine (Zwick/Roell Z100) operated at a constant strain rate of $8.33 \times 10^{-5}\ s^{-1}$ (0.005/min). The performance parameters of ultimate tensile strength, yield strength, elongation, and reduction in area were determined after the tests. At least three specimens were tested to fracture for each exposure time.

The thermal exposed specimens and tensile fractured specimens of the alloy were prepared by standard metallographic method, and etched by a solution of 3 g $CuSO_4$ + 10 ml H_2SO_4 + 40 ml HCl + 50 ml H_2O for microstructure observation. A Merlin Compact SEM equipped with EDS and EBSD was used to observe the morphology of microstructure, analyze the variation of composition and local misorientation of the alloy. A Tecnai G2F20TEM was applied to analysis the deformation structures about the dislocation configuration. The TEM specimens were cut perpendicular to the tensile direction and 5 mm distant from the fracture. After mechanically grinding the specimens to 100 μm, the foils were electrochemically thinned by twin-jet electropolishing device in a mixed solution of 10% perchloric acid and 90% ethanol.

3. Results

3.1. Microstructure evolution of the alloy during exposure process

The microstructure evolution of alloy exposed at 700 °C for various time without loading are observed and shown in Fig. 1. For the unexposed alloy, the clean grain boundary interspersed with a few M_6C carbides is observed. As the exposure time increased to 1000 h, plenty of carbides with the size of ~200 nm located at the grain boundaries can be observed, which has been identified as $M_{12}C$ carbides by Liu et al. [6]. In the case of the exposure time of 10000 h, it can be observed that the size of $M_{12}C$ carbides is increased and interconnected together along the grain boundaries.