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Effects of hot compression on carbide precipitation behavior of Ni−20Cr−18W−1Mo superalloy

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Abstract: The effect of hot compression on the grain boundary segregation and precipitation behavior of M_6C carbide in the Ni−20Cr−18W−1Mo superalloy was investigated by thermomechanical simulator, scanning electronic microscope (SEM) and X-ray diffraction (XRD). Results indicate that the amount of M_6C carbides obviously increases in the experimental alloy after hot compression. Composition analyses reveal that secondary M_6C carbides at grain boundaries are highly enriched in tungsten. Meanwhile, the secondary carbide size of compressive samples is 3−5 μm in 10% deformation degree, while the carbide size of undeformed specimens is less than 1 μm under aging treatment at 900 and 1000 °C. According to the thermodynamic calculation results, the Gibbs free energy of *γ*-matrix and carbides decreases with increase of the compression temperature, and the W-rich M_6C carbide is more stable than Cr-rich $M_{23}C_6$. Compared with the experimental results, it is found that compressive stress accelerates the W segregation rate in grain boundary region, and further rises the rapid growth of W-rich M_6C as compared with the undeformed one.

Key words: Ni−20Cr−18W−1Mo superalloy; hot compression; grain boundary segregation; carbide precipitation

1 Introduction

The very high temperature gas-cooled reactor (VHTR) is being developed rapidly as one of the Generation-IV nuclear systems due to its much higher heat-electricity conversion efficiency than current light water reactors (LWRs) [1]. Currently, the outlet temperature of the VHTR has been designed as approximately 750 °C, but follow-up designs raise it to about 950 °C. These conditions are extremely demanding on the materials [2,3]. Nowadays, the Ni−Cr−W-based solid-solution-strengthened superalloys (especially HAYNES 230 alloy) become the primary candidate materials of high temperature components of intermediate heat exchanger (IHX) due to high temperature oxidation and corrosion resistance, excellent creep resistance at elevated temperatures [4−8].

The harsh environment of IHX causes the most vital damage, such as stress corrosion cracking [2] and hot corrosion [9,10], which primarily appear at the grain boundaries and on the surface, respectively. Therefore, much attention has been given to the grain boundaries quality for better service performance of superalloys. In the Ni−Cr−W-based superalloys, carbide is the major precipitate which affects mechanical properties due to its type, shape, size dimension and distribution [11]. For instance, PATAKY et al [12] have indicated that intense carbide−dislocation interactions are observed providing substantial resistance to dislocation motion and the M_6C-type carbide has far less interaction with dislocations than the $M_{23}C_6$ carbide. Experimental results in Refs. [13,14] show that the decrease of both tensile and yield strengths of the aged Ni−Cr−W-based superalloy is mainly caused by the breaking of the lamellar $M_{23}C_6$ carbide. XU et al [15] have found that the addition of silicon can rise the dissolved temperature of M_6C carbide up to 1335 °C. The silicon-rich M_6C carbide particles, which act as cracking origin sites, always account the deterioration of mechanical properties at elevated temperatures. However, W-rich M_6C type carbides have been proven to increase the strength and creep resistance at evaluated temperatures by blocking the movement of dislocations effectively and pinning grain boundaries to prevent sliding. It has been proven that the W-rich primary M_6C carbides always

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form during solidification process [16,17] and the secondary M_6C carbides form during heat treatment and thermal processing [18−20] in Ni-based superalloys.

Elements segregation at grain boundaries becomes a major factor in affecting mechanical properties of Ni-based superalloys [21]. Previous researches [22,23] have shown that high temperature plastic deformation can induce grain boundary segregation of Cr, Mo and P in an interstitial free steel, and this deformation-induced segregation increases with increasing deformation strain. ALLART et al [24] have indicated that plastic deformation accelerates the kinetics of sulphur grain boundary segregation. For Ni-based superalloys, thermomechanical working plays an important role in the fabricating process of thermostability components. Different components of Ni-based superalloys are generally fabricated by hot forging, hot rolling, and extrusion. Hence, it is necessary to study grain boundary segregation of Ni-based superalloys during hot deformation process. It is expected to illustrate the effect of grain boundary segregation on the carbide precipitation behavior and mechanical properties of Ni-based superalloys.

The present work aims to illustrate the deformation-induced grain boundary segregation in a solid-solution-strengthened Ni−20Cr−18W−1Mo superalloy. The effect of deformation conditions including temperature and deformation degree on grain boundary segregation of W and Cr elements was investigated by isothermal compression tests and deliberately focused on low levels of deformation (2.5% to 10%) to avoid dynamic recrystallization. The comparison of experimental data and thermodynamics calculation was discussed to illustrate the deformation effect on carbide precipitations at the serviceability temperature in nuclear power field.

2 Experimental

A nominal Ni−20Cr−18W−1Mo alloy was melted in the vacuum induction melting (VIM) furnace (with 1.3 MPa pressure) and remelted twice by vacuum arc remelting (VAR). The mass of the ingot was around 30 kg. The chemical composition of the alloy (mass fraction, %) was 19.82 Cr, 18.48 W, 1.24 Mo, 0.46 Al, 0.11 C, 0.0028 B, (0.026) La, <0.004 P, <0.004 S and balance Ni. The cast ingot was homogenized at 1200 °C for 24 h with furnace cooling and then hot forged and rolled at 1150 °C. Hot-rolled sheet with 10 mm in thickness was solution-annealed at 1260 °C for 0.5 h, followed by water quenching (WC). Cylindrical specimens of 8 mm in diameter and 12 mm in height were machined from the hot-rolled sheet. The isothermal compression tests were carried out on a Gleeble−3500 thermomechanical simulator with a constant strain rate of 10^{-4} s^{-1} at 900 and 1000 °C. The temperature control was within ±1 °C in the strain range of 2.5%−10%. All the specimens were heated at a heating rate of 20 °C/s and held for 30 s to secure temperature uniformity prior to deformation. Deformation conditions such as temperature and displacement velocity were automatically controlled by a computer system. The relevant data such as true stress and strain were also automatically recorded. The adiabatic temperature rise in the specimen during testing was measured by a thermocouple wire embedded in a 0.8 mm hole machined up to the center at mid-height of the specimen. In order to reduce the friction, a tantalum sheet was applied to specimen surface to ensure sufficient lubrication during each test. All the tests were carried out in high vacuum (133.322 mPa). After isothermal compression, the deformed specimens were quenched to room temperature immediately by water quenching.

The specimens for microstructure observation were sectioned parallel to the compression axis, and metallographic samples were prepared by standard metallographic techniques and the polished specimens were etched with aqua regia (V(HCl):V(HNO_3)=3:1) for 60 s to reveal the microstructure. Microstructures were examined by a scanning electron microscope (SEM, Vega Tescan) equipped with an energy dispersive spectrometer (EDS, Oxford INCA PentaFET×3). The phase composition of this alloy was analyzed by DX−2700 X-ray diffraction (XRD) with a Cu K_α radiation source. Voltage and current were 40 kV and 30 mA, respectively. Rietveld method [25] based on the XRD patterns was used for qualitative and quantitative phase analyses. The full spectrum fitting was carried out to obtain a easy quantitative phase analysis by Rietveld refinement.

3 Results and discussion

3.1 Microstructure

3.1.1 Microstructure before thermal deformation

Figure 1 illustrates the SEM image of as-rolled Ni−20Cr−18W−1Mo alloy in the solution-annealed condition. The as-rolled microstructure preserves an average grain size of 60 to 70 μm. The plate-like and globular shaped primary carbides (bright color in SEM image in Fig. 1), which are typically fine with an average size below 10 μm, distributed randomly within the grain. In previous studies, these carbides have been proven as the primary W-rich M_6C-type carbide in the form of Ni_3W_3C through TEM/SAD and EDS analyses [14,20,26]. Most of grain boundaries were observed to be straight or smoothly curved in their morphology and a low fraction of carbide precipitations along grain boundaries (see Figs. 1(a)). EDS analysis as shown in