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Anomalous yield and intermediate temperature brittleness behaviors of directionally solidified nickel-based superalloy

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Abstract: A nickel-based superalloy with good corrosion resistance was fabricated by directional solidification, and its microstructure and tensile properties at elevated temperatures were investigated. Microstructure observations reveal that the γ' precipitates are arrayed in the γ matrix regularly with some MC, Ni_5Hf and M_3B_2 particles distributed along the grain boundary. The tensile tests exhibit that the tensile properties depend on temperature significantly and demonstrate obvious anomalous yield and intermediate-temperature brittleness (ITB) behavior. Below 650 °C, the yield strength decreases slightly but the ultimate tensile strength almost has no change. When the temperature is between 650 °C and 750 °C, the yield and ultimate tensile strengths rise rapidly, and after then they both decrease gradually with temperature increasing further. The elongation has its minimum value at about 700 °C. The TEM examination exhibits that sharing of the γ' by dislocation is almost the main deformation mechanism at low temperatures, but the γ' by-pass dominates the deformation at high temperatures. The transition temperature from shearing to by-pass should be around 800 °C. The anomalous yield and intermediate-temperature brittleness behaviors should be attributed to the high content of γ'. In addition, the carbides and eutectic structure also contribute some to the ITB behaviors of the alloy.

Key words: nickel-based superalloy; directional solidification; anomalous yield; intermediate-temperature brittleness; microstructure

1 Introduction

Nickel-based superalloys, which are strengthened by γ' precipitates and solution alloying elements, are widely used in high temperature environment, due to their excellent creep properties, fatigue strength and good corrosion resistance [1−4]. Recently, with the development of the land-base and aerospace gas turbine engines, the turbine inlet temperature rises year by year to obtain high thermal efficiency, which requires the superalloy to have better creep and corrosion resistance. In order to meet these requirements, more refractory elements are added to the superalloy, which is beneficial to the creep but harmful to the mechanical properties [5−7]. To the superalloy, though the high-temperature creep and fatigue behavior are very important, having a good plastic deformation capability is also very necessary.

Generally, it can be expected a better elongation with the increase of temperature. However, for the superalloy and some other alloys, sometimes they exhibit obvious anomalous yield behavior and intermediate-temperature brittleness (ITB) characteristic, which is harmful to the application of these materials. The phenomena of ITB and anomalous yield behaviors have been reported in the last century and till now many investigations have been carried out on them [8]. The former researches [9−11] show that some polycrystal superalloys have the ITB behavior. However, the reports on the behavior of the directionally solidified nickel-based superalloy are relative few.

The experimental directionally solidified nickel-based superalloy, which consists of γ matrix, γ' precipitates, carbides, and minor borides, is designed to use in high-performance industrial gas turbines [12]. To possess excellent hot-corrosion and high-temperature oxidation resistance, the alloy contains 13% Cr and 10% Co. In addition, more W, Mo, Ta and Hf are added to increase its creep properties. The benefit can be obtained

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from the combination of refractory elements; however, they also would influence the mechanical properties of the alloy inevitably. Therefore, in the present work, the study on the microstructure and elevated temperature mechanical properties of the directionally solidified experimental alloy is carried out.

2 Experimental

The chemical composition of directionally solidified alloy used in the present study is listed in Table 1. The alloy was remelted in a VIM25F vacuum induction furnace and directionally solidified in ZGD2 vacuum induction furnace. The temperature gradient was 80 °C/cm and the withdrawal rate was 8 mm/min. The directionally solidified specimen was heat treated in a electric muffle furnace with the procedure of (1210 °C, 2 h AC)+(1080 °C, 2 h AC)+(850 °C, 24 h AC) (AC: air cooling). The standard specimens for tensile test with a diameter of 5 mm and a gage length of 25 mm were machined longitudinally from the heat-treated samples. The tensile tests were conducted on a Universal AG−250KNE test machine in air with initial strain rates of 5×10^{-5} to 1.04×10^{-4} s^{-1} at 25−1000 °C. The tensile specimens were induction heated and the temperature was measured using a thermocouples placed in the gauge length. The temperature gradient of the gauge length was not exceeded ±2 °C. Load and extension were recorded directly. The displacement rate was maintained at 0.5 mm/min up to rupture. At least three identical specimens were tested at each temperature.

Table 1 Chemical composition of directionally solidified alloy (mass fraction, %)

C	B	Cr	Co	W	Mo
0.024	0.012	12.53	9.81	5.46	1.21

Al	Ti	Ta	Hf	Ni
3.76	2.28	4.65	0.32	Bal.

Samples for microstructure observation were cut from the heat-treated and fracture specimens. The samples were fabricated by conventional method and electrochemically etched with an electrolyte consisting of 20 g $CuSO_4$+100 mL HCl+5 mL H_2PO_4+100 mL H_2O. The microstructure and fracture surface after the tensile tests were examined by an optical microscope (OM) and a scanning electron microscope (SEM) with an energy-dispersive X-ray spectroscope (EDS). The slices for transmission electron microscopy (TEM) observation were cut from the heat-treated sample and gauge part of the specimens deformed at different temperatures normal to the loading axis. The thickness of the slices was about 0.5 mm and polished to 50 μm. The polished slices were shaped into 3 mm in diameter followed by ion milling to perforation. The TEM observation was carried out by a JEM−2010 operated at 200 kV.

3 Results and discussion

3.1 Microstructure of heat-treated alloy

The typical optical metallographic microstructures of the directionally solidified alloy are shown in Figs. 1(a) and (b). The dendrites in metallographic samples cut perpendicular to the solidification direction appear as bright cross. The precipitates and γ/γ' eutectic mainly distribute in the interdendritic region. At such a withdrawal rate, the dendrite column almost parallels the direction of the solidification. Further observation on the alloy exhibits that some precipitates and chrysanthemum-like γ/γ' eutectic form along the boundary of matrix, as shown in Fig. 1(c). The EDS results reveal that they are carbide, boride and intermetallic compounds. The image of the γ' shows that the precipitates exhibit well cuboid morphology, as shown in Fig. 1(d). Moreover, there are some small γ' particles with small proportion distributing in the matrix among γ' particles.

TEM observation on the γ' precipitate exhibits that the γ' cuboids with an average edge length of 400 nm are produced after the heat treatment, as shown in Fig. 2(a). The secondary sphere γ' with scores of nanometers in diameter precipitates in the matrix. Further observation on the grain boundary and the eutectic region reveal that many precipitates exist in these regions, as shown in Fig. 2(b). Combining with the selected area electron diffraction (SAED) patterns, it can be determined that most of the particles in the alloy are MC type carbide. The EDS results reveal that they contain a lot of Ti and W element, as shown in Fig. 2(c), so the carbide can be described as (Ti, W)C. The TEM observation also exhibits small precipitates along the MC boundary. The SAED pattern reveals that the small precipitates have an orthorhombic crystal structure, and the EDS test exhibits that it has a lot of Cr element, as shown in Fig. 2(d). Due to the high content of B addition in the alloy, and based on the former studies [13,14], such precipitates should be M_3B_2 type boride. In addition, the TEM observation still finds some Hf-rich particles forming at the grain boundary, as shown in Figs. 2(e) and (f). Combining with the former researches [15,16] and the SAED pattern, the Hf-rich particle can be determined as Ni_5Hf phase, which has a cubic crystal structure with a=b=c=0.68 nm, and the space group of $F\bar{4}3m$.