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Microstructural characterization, formation mechanism and fracture behavior of the needle δ phase in Fe–Ni–Cr type superalloys with high Nb content

Yongquan Ning [a,*], Shibo Huang [b], M.W. Fu [c], Jie Dong [d]

[a] School of Materials Science & Engineering, Northwestern Polytechnical University, Xi'an 710072, PR China
[b] Anshan Iron & Steel Group Corporation Bayuquan Subsidiary Company, Bayuquan 115007, PR China
[c] Department of Mechanical Engineering, Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong, PR China
[d] Inspection & Research Institute of Boiler & Pressure Vessel of Jiangxi Province, Nanchang 330029, PR China

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ABSTRACT

Microstructural characterization, formation mechanism and fracture behavior of the needle δ phase in Fe–Ni–Cr type superalloys with high Nb content (GH4169, equivalent to Inconel 718) have been quantitatively investigated in this research. The typical microstructures of δ phases with the stick, mixed and needle shapes obviously present in Inconel 718 after the isothermal upsetting at the temperature of 980–1060 °C with the initial strain rate of 10^{-3}–10^{-1} s^{-1}. It is found that the shape of the δ phase has a great effect on the mechanical properties of the alloy, viz., the stick δ phase behaves good plasticity and the needle δ phase has good strength. In addition, the needle δ phase can be used to control the grain size as it can prevent grain growth. The combined effect of the localized necking and microvoid coalescence leads to the final ductile fracture of the GH4169 components with the needle δ phase. Both dislocation motion and atom diffusion are the root-cause for the needle δ phase to be firstly separated at grain boundary and then at sub-boundary. The formation mechanism of the needle δ phase is the new finding in this research. Furthermore, it is the primary mechanism for controlling the needle δ phase in Fe–Ni–Cr type superalloys with high Nb content.

1. Introduction

Due to its excellent performance at a wide range of working temperature up to 650 °C[1,2], GH4169 (Inconel 718) is the most important Fe–Ni–Cr type superalloy used for manufacturing high temperature components in the aerospace industry. In this type of superalloy, γ (Ni) is the matrix, while γ′ (Ni_3AlTi) and γ″ (Ni_3Nb) precipitates are the strengthening phases [3]. In practice, the major strengthening phase is γ″, and only a small volume fraction (<25%) is needed to transform GH4169 into a high-strength superalloy. In contrast, the volume fraction of the γ′ phase in GH4169 is less than 5% and the strengthening contributed by this phase is incidental. And δ (Ni_3Nb) is the equilibrium phase of γ″ and is thermodynamically more stable than the γ″ phase [4,5]. The lattice parameters of γ′, γ″ and δ phases are summarized in Table 1 [6]. A typical precipitation time temperature (PTT) diagram for this type of superalloy is shown in Fig.1 [7,8]. The precipitation of the δ phase often occurs in the temperature range of about 750–1020 °C. At lower temperature (below 900 °C), the precipitation of the δ phase is preceded by the γ″ phase, but the δ phase precipitates directly from the γ matrix at higher temperature [9,10]. It has been shown that most rapid precipitation of the δ phase occurs at the temperature of about 900 °C [11]. When the temperature is higher than 980 °C, most δ phases dissolve [12]. Since the δ phase has a great effect on the mechanical properties of the superalloy, the effects of isothermal upsetting parameters on the microstructural evolution are thus explored and discussed in this research. Furthermore, the fracture behavior of the needle δ phase is quantitatively investigated to address the issue why the needle δ phase has good strength. Finally, the formation mechanism of the needle δ phase is presented in this paper.

2. Procedure

The experimental material used in this research was machined from a GH4169 drum shaft with the dimension of 50 × 50 × 80 mm. The nominal chemical compositions are as follows (wt.%): Cr, 19.10; Fe, 18.70; Nb, 4.95; Mo, 3.00; Ti, 0.95; Al, 0.50; Co, 0.30; Si, 0.10; Mn, 0.06; C, 0.03; Cu, 0.03; P, 0.004; S, 0.002; Ca, 0.0002; Ni, balance. When the ratio of (Al + Ti) to Nb (at.%) is higher than 0.66, both γ′ and γ″ phases can precipitate at the temperature between 700 and 900 °C for a short annealing time [13]. But with the increase of annealing time, γ′ and γ″ phases will transfer to the δ phase [14]. In order to ensure that only δ phase precipitating in the experimental material, delta heat treatment was conducted at 900 ± 3 °C for 24 h, followed by water quenching.

* Corresponding author.
E-mail addresses: luckyning@nwpu.edu.cn, ningke521@163.com (Y. Ning).

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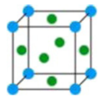
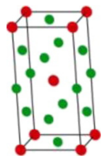
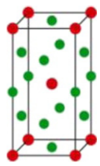
Table 1
Lattice parameters of γ′, γ″ and δ phases in Inconel 718 superalloys calculated from XRD patterns [7].

Phase	Crystal lattice	Annealing temperature (°C)	Annealing time (h)		
			4	50	100
γ′ (fcc)		680	a = 3.592 Å		
		750	a = 3.593 Å	a = 3.596 Å	
γ″ (bct)		680	a = 3.605 Å	a = 3.608 Å	a = 3.603 Å
			c = 7.437 Å	c = 7.438 Å	c = 7.474 Å
		750	a = 3.610 Å	a = 3.631 Å	a = 3.610 Å
			c = 7.439 Å	c = 7.391 Å	c = 7.457 Å
δ (orthorhombic)		680	a = 5.105 Å	a = 5.108 Å	a = 5.110 Å
			b = 4.209 Å	b = 4.220 Å	b = 4.216 Å
			c = 4.529 Å	c = 4.521 Å	c = 4.526 Å
		750	a = 5.111 Å	a = 5.106 Å	a = 5.108 Å
			b = 4.214 Å	b = 4.225 Å	b = 4.218 Å
			c = 4.531 Å	c = 4.557 Å	c = 4.530 Å

A series of isothermal upsetting were conducted on a THP-6300A hydraulic press at the temperatures of 980, 1000, 1020, 1040 and 1060 °C with the strain rates of 10^{-3}, 10^{-2} and 10^{-1} s^{-1}. All specimens were heated at a heating rate of 10 °C/s and soaked for 10 min at the deformation temperature. The upset specimens were then cooled down to room temperature by spraying with water. The microstructure examination was conducted by using an OLYMPUS-PM3 optical microscope (OM). In addition, the δ phase and boundary structure were characterized using TECNAI-G^2 F30 transmission electron microscopy (TEM). The typical microstructures of GH4169 superalloys processed after isothermal upsetting are presented in Fig. 2, which obviously shows the stick, mixed and needle δ phases presenting in the alloy.

The cylindrical tensile specimens with the gauge length of 30 mm and the diameter of 5 mm were machined from the upset sheet based on ISO 6892-2: 201 [15]. Uniaxial tension tests were carried out with an initial strain rate of 10^{-3} s^{-1} using an INSTRON-8802 testing machine at the working temperature of 20, 550, 600 and 650 °C. Fig. 3 gives the mechanical properties of GH4169 superalloys with stick and needle δ phases. In order to investigate the effect of the δ phase on mechanical properties, typical microstructure and fracture surface were observed using a ZEISS-SUPRA 55 scanning electron microscope (SEM).

3. Results

The effect of isothermal upsetting parameters on the microstructural evolution of the alloy has been discussed in present work. Fig. 2 shows the typical microstructures of GH4169 superalloys processed after isothermal upsetting at the temperature of 980–1060 °C with the strain rates of 10^{-3}–10^{-1} s^{-1}, which obviously shows the δ phases in stick (a, d, g), mixed (b, e, h) and needle (c, f, i) shapes presenting in the alloy. Fig. 2a, d and g shows the microstructure of the stick δ phase after isothermal upsetting at 980 °C with the initial strain rate of 10^{-3}–10^{-1} s^{-1}, respectively. It can be clearly seen that the stick δ phase distributes throughout the DRX grains. In Ref. [16], Zhang et al. give the schematic diagrams for the spheroidization of the stick δ phase. The results showed that the dissolution of the stick δ phase and the precipitation of spherical δ phase particles coexisted during the deformation. As a result of the deformation breakage and dissolution breakage, the stick δ phase spheroidized and transferred to the spherical δ phase. Fig. 2c, f and i presents the typical needle δ phase after the isothermal upsetting at 1060 °C. Obviously, there is no stick δ phase in the microstructure, but the needle δ phase separates at the boundaries. The needle δ phase can control the grain size of the GH4169 components. Compared with the microstructures processed at 980 and 1060 °C, the mixed δ phase (stick + needle) presents in the superalloy, as shown in Fig. 2b, e and h. The microstructure observation shows that in the mixed microstructure, the stick δ phase mainly separates at the grain boundary, but the needle δ phase always separates at the sub-boundary.

It is well known that the grain size, precipitates size, volume fraction and geometry play a role for the strength of the materials. Since the shape of the δ phase has a great effect on mechanical property, the mechanical properties of GH4169 superalloys with the δ phase in different shapes (stick and needle) are shown in Fig. 3, showing that the stick δ phase behaves good plasticity and the needle δ phase has good strength. On the other hand, since the stick δ phase has a good deformation coordination, GH4169 superalloys processed at the lower deformation temperature (980 °C) have a better plasticity. In addition, the superalloys with the δ phase and processed with the initial strain rate of 10^{-1}, 10^{-2} and 10^{-3} s^{-1} have the elongation of 10.8, 12.2 and 12.9%, respectively. In our previous research [1], an equation that describes the δ phase content as a function of temperature and strain rate is $V(\delta) = -\frac{3.92 \cdot \log(\varepsilon)}{T} + \frac{890.9}{T} + 0.02033 \cdot \log(\varepsilon) - 0.58469$, based on the microstructure observation of cylindrical specimen (Ø8 × 12 mm) processed after isothermal compression at the temperature of 940–1020 °C with the strain rate of 1.0–0.001 s^{-1}. The experimental results have a good agreement with the predicted value of the equation. Therefore, the equation can be used to predict the δ phase content for GH4169

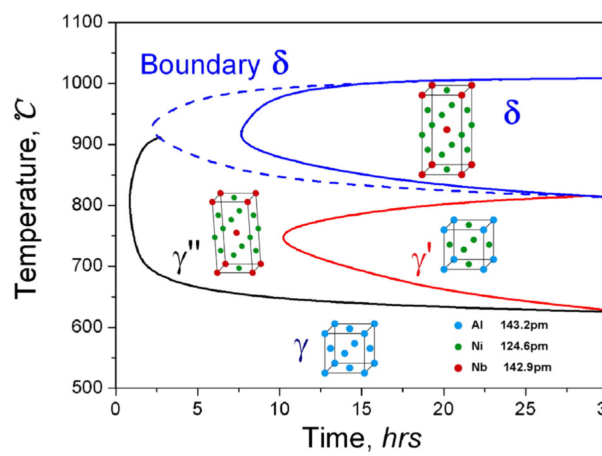


Fig. 1. Precipitation-time-temperature (PTT) diagram of different phases in high Nb content Fe–Ni–Cr type superalloys [8,9].