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Effect of cooling rate on microstructure and tensile properties of powder metallurgy Ni-based superalloy

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Abstract: The effects of size distribution, morphology and volume fraction of γ' phase and grain size on tensile properties of powder processed Ni-based superalloy were investigated by using two different quenching methods. Oil quenching and air cooling were adopted with cooling rate of 183 °C/s and 4−15 °C/s, respectively. The experimental results show that the average size of the secondary γ' after oil quenching is 24.5 nm compared with 49.8 nm under air cooling, and corresponding volume fractions of γ' are 29% and 34%, respectively. Meanwhile, the average grain size remains nearly equivalent from both oil-quenching and air-cooling specimens. The tensile strength at room temperature is higher for the oil-quenched specimen than the equivalent from the air-cooled specimen, but the difference approaches each other as the temperature increases to 650 °C. The fractography clearly demonstrates that transgranular fracture governs the failure process at ambient temperature, in contrast to the intergranular fracture at 650 °C or even higher temperature. These two mechanical responses indicate the strengthening effects of γ' precipitates and grain boundary for polycrystalline Ni-based superalloys at different temperatures.

Key words: powder metallurgy Ni-based superalloy; cooling rate; tensile properties; γ' phase precipitate; fracture mechanism

1 Introduction

Polycrystalline Ni-based superalloys have been widely used for gas turbine disk of aircraft due to their excellent high temperature strength, fatigue and creep resistance, oxidation and corrosion resistance, superior microstructure stability and service reliability [1,2]. These striking mechanical performances upon elevated temperature have been primarily attributed to the ordered and coherent γ' precipitates with a disordered γ matrix. The microstructure of disk superalloys, especially in terms of the morphology, as well as the spatial and size distribution of γ' precipitates, plays a very important role in determining the mechanical properties [3,4].

The heat treatment of the turbine disk components is a crucial process for achieving an optimum microstructure and mechanical properties [5−7], which can be achieved by the typical processing of turbine disk blanks, termed as solution and aging treatment. The grain size and γ' precipitates are significantly affected by heat treatment. Specimens heated to a supersolvus solution temperature usually can obtain coarse grain size bringing better crack growth resistance, but lower tensile strength [8]. So, it is necessary to improve the tensile property through the process of cooling or ageing treatment. Previous researches [9−11] show that the size distribution, morphology and volume fraction of γ' can be modified by cooling pathways in a controllable manner. Wherein, controlling of γ' precipitation through quenching greatly affects the final tensile properties, because the nucleation and growth kinetics of the precipitates strongly depend on the cooling rate during quenching. Many researchers [12−14] have studied the effect of different steady cooling rates on the γ' precipitates characteristics, showing that the higher cooling rate can obtain higher strength despite the tendency to crack.

In practice, the typical quenching media, i.e., water, oil, air, are utilized to cool down the alloys and produce the desirable mechanical properties. However, the cooling rate from near solvus temperature to low

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temperature is nonlinear and hard to be quantitatively described, leading to the difficulty to establish the relationship between cooling rate and mechanical strength. In this work, we quantitatively investigated the cooling process by means of two kinds of cooling media, and systematically characterized the tensile properties corresponding to two different cooling rates in order to explain the mechanism underpinning the tensile failure process.

2 Experimental

2.1 Materials

A powder metallurgy (P/M) processed Ni-based superalloy, designated as CSU-A1, was used in this work. Table 1 lists its nominal composition. The master alloy was melted by vacuum inducing melting followed by gas atomization and hot isostatic pressing (HIP) compaction. Later, the billet bar was hot-extruded (HEX) with a reduction ratio of 12:1. The tensile specimens were cut from the extruded bar and the geometry of specimen is depicted in Fig. 1.

Table 1 Nominal composition of CSU-A1 alloy (mass fraction, %)

Co	Cr	Mo	W	Al	Ti
26	13	4	4	3.3	4.0
Nb	Hf	C	B	Zr	Ni
0.9	0.2	0.04	0.03	0.04	Bal.

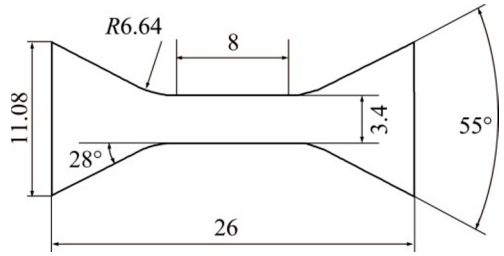

Fig. 1 Geometry of tensile sample (unit: mm)

2.2 Solution temperature tests

Prior to the heat treatment, it was critical to determine the solvus temperature of γ' phase for a new P/M superalloy. Different thermal analysis (DTA), thermodynamic calculation and metallographic analysis method were utilized to determine the phase precipitation temperature separately. The DTA test was conducted using an ultra-high temperature thermo gravimetric analysis (Setsys Evo). The sample sizes used were 2.5 mm × 2.5 mm × 2.5 mm and the heating/cooling rate during the test was 10 °C/min. The thermodynamic calculation was performed using the Thermo-Calc software with the TTNi8 database. Metallographic analysis method was conducted by heating up the samples to a serious of temperature including 1110, 1130, 1140, 1150 and 1170 °C and exposing for 1 h in a electric-resistance tube furnace, and finally followed by water quenching to remain the original solution structure.

2.3 Heat treatment and cooling rate tests

Tensile samples were cut to sizes of about 26 mm × 11 mm × 2 mm shown in Fig. 1 by a wire electrolytic-discharge machine and divided into two groups by different cooling approaches. The specimens were enclosed in a silica tube and back-filled with nitrogen to avoid oxidation and elemental evaporation. Heat treatment consists of a supersolvus treatment of 1165 °C for 1 h and a subsequent quenching in the oil or air, respectively. Both two groups of specimens were aged for 8 h at temperature of 815 °C finally. Monitor thermocouples were welded on the specimen surface before heating up. The measurement of cooling rate was performed using a temperature monitor (RDXL8, OMEGA) with a record frequency of 2 Hz.

2.4 Tensile property tests

The tensile tests were performed at the temperatures of 25, 400, 650, 700 and 750 °C. Prior to tensile test, the samples were heated to target temperature inside the furnace and then soaked for 15 min. The loading strain rate is set up to 10^{-4} s^{-1} which was controlled by crosshead using the electronic universal testing machine (UTM5105, SUNS). Each test was repeated at least twice to guarantee the reliability of the experimental measurement. The tensile failure for all the specimens occurs within the gage length.

2.5 Microstructure characterization

Prior to the microstructure observation, the specimens were manually ground up to 1000-grits sand paper and polished with 0.05 μm alumina suspension. Subsequently, vibratory polishing was used to achieve high-quality surface. In order to observe the grain boundary and precipitates, the specimens were etched using different etchants, as shown in Table 2. Microstructure analyses of the samples were performed by optical microscopy (OM, DM4000M, Leica) and scanning electron microscopy (SEM, QUANTA 650 FEG, FEI). The grain size was measured using electron back-scattered diffraction (EBSD, QUANTA 650 FEG, FEI) technique. Image post-processing software was

Table 2 Imformation for etching methods [15]

Enchant	Etching time/s	Microstructure
50 mL lactate + 30 mL nitrate + 2 mL HF	10~15	γ'
100 mL HCl +100 mL alcohol + 5 g CuCl	60~90	Grain boundary