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Microstructure and tensile properties optimization of MIM418 superalloy by heat treatment

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ABSTRACT

Solution plus ageing leads to the precipitation of finer and more homogeneous γ' precipitates in comparison to direct ageing. Solution treatment at 1220 °C eliminates the large primary γ' phase and replaces them with homogeneous secondary γ' precipitates. Coarsening of γ' particles according to Ostwald ripening mechanism is confirmed. An increase of the critical resolved shear stress (CRSS) is observed for the under-aged specimens, followed by a maximum of CRSS at γ' particle size of around 100 nm and a decrease of CRSS for the over-aged specimen. This result agrees well with the highest yield strength and microhardness of the 750 °C × 24 h aged specimens.

1. Introduction

The K418 superalloy has excellent high temperature mechanical properties, good fatigue property, prominent oxidation and corrosion resistance at the temperature below 900 °C, which is commonly used as turbine wheels in automotive turbocharger (Shi et al., 2013). Metal injection molding (MIM) is a near net shape technique by the introduction of plastic injection molding into powder metallurgy area. Contreras et al. (2008), Miura et al. (2010) and Özgün et al. (2013a,b) approved that MIM technique is suitable for the mass production of complex shaped superalloy components with high dimensional accuracy. The K418 superalloy prepared by metal injection molding is named as MIM418. Several processing procedures were involved during the preparation of MIMed superalloy components, such as sintering, hot isostatic pressing, solution and ageing treatment. HIPing process and post heat treatment on the as-sintered samples are the key steps that influence the microstructure and mechanical properties of MIM418 superalloy. Due to the complexity of processing parameters, MIMed superalloys exhibit different solution and ageing responses compared with that of the cast K418 superalloy (Özgün et al., 2013a,b). Since the excellent properties of MIM418 alloy depend greatly on the strengthening effect of γ' precipitates and carbides, it is very important to precisely control the size, distribution and morphology of these precipitates in order to further enhance mechanical properties (Martischius et al., 2009; Oh et al., 2011). Unlike the cast K418 superalloy, there are no standardized heat treatments available for MIM418 superalloy. Up till now, there are no systematic investigations on the optimization of heat treatment conditions for MIM418 superalloys.

The optimization of heat treatment cycles with respect to better understanding and adjusting the microstructure of MIM418 superalloy are the focus of this work. Two different heat treatment cycles, direct ageing and solution plus ageing, on microstructural evolution and tensile behavior together with the fracture characteristics of the superalloys were investigated. Additionally, a comparison was made between experimental critical resolved shear stress (CRSS) increment and the calculated CRSS according to the theoretical models of particle strengthening.

2. Experimental

Argon gas atomized K418 superalloy powder with spherical morphology was used as raw material, which was supplied by Beijing Institute of Aeronautical Materials. The chemical composition of the powder is given (in wt.%) as Ni-0.11C-12.62Cr-6.39Al-0.63Ti-3.83Mo-1.85Nb. Fig. 1 displays morphology of Ar atomized K418 superalloy powder. The powder exhibits spherical morphology, and

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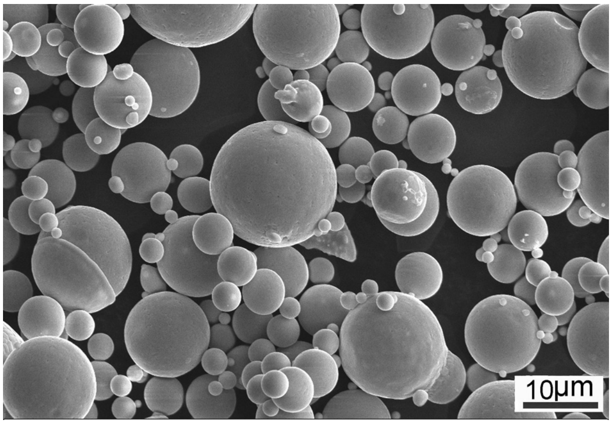

Fig. 1. Morphology of Ar atomized K418 superalloy powder.

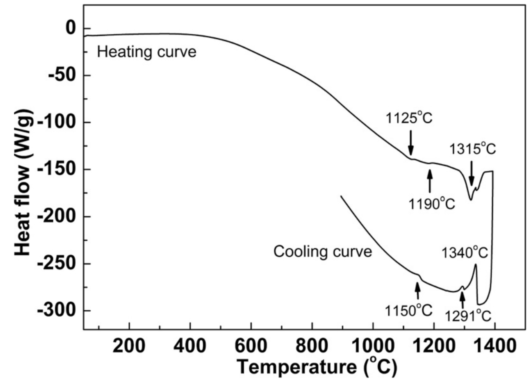

Fig. 2. DSC curve of MIM418 superalloy.

D_{10}, D_{50} and D_{90} values are 8.5 μm, 14.8 μm and 28.7 μm, respectively. The oxygen content of the powder is as low as 209 ppm.

The feedstock for MIM was produced by mixing K418 superalloy powder and binder in the double planetary mixer at 150 °C. A multi-component binder system, which consists of paraffin wax (WX), polypropylene (PP), high density polyethylene (HDPE) and stearic acid (SA), is designed. Powder loading of the feedstock is as high as 65 vol.%, which is ascribed to the spherical morphology, good dispersivity and high tap density of the powder. This optimal powder loading was selected by the incremental powder loading test based on the measurement of the mixing torque of the feedstock (Kong et al., 2012).

Tensile specimens were injected with a CJ-80E type injection molding machine at 150–160 °C. Firstly, the injection molded specimens were subjected to solvent debonding in trichloroethylene for 6 h. Then, the solvent debonded specimens were heated with a low heating rate of 1 °C/min from 20 °C to 400 °C, and the temperature was increased to 600 °C with a heating rate of 2 °C/min. Finally, the specimens were presintered at 900 °C for 90 min. The debonded samples were sintered at 1235 °C for 2 h in vacuum (5×10^{-3} Pa), and the sintered samples exhibit negligible evaporation of alloying elements. The sintered samples were HIPed at 1200 °C for 2 h under the pressure of 180 MPa. The density of the sintered specimen is 7.80 g/cm^3 (relative density = 97.5%), and the density of the HIPed sample achieves 7.96 g/cm^3 (relative density > 99.5%). Two different heat treatment cycles were designed. Some HIPed samples were directly aged at 750 °C for 4–120 h and air cooled without a prior solution treatment. The other HIPed samples were firstly solution-treated at 1180–1220 °C for 2 h, and then aged at 750–930 °C for 4–120 h and air cooled.

Tensile properties were tested on an Instron 5569 universal testing system at room temperature, and the values of mechanical properties are the mean of five measurements. Due to the high relative density (>99.5%) of the HIPed samples, the mechanical properties of the sample exhibit small scatter in data (<5%). The size of γ' precipitate was measured by drawing a circle around γ' particles using image tool software and then taking the diameter of the circle as an equivalent diameter for each precipitate. After electro-polishing and electro-etching, the morphology of γ' precipitates was observed on a LEO1450 secondary electron microscopy (SEM). Discs for transmission electron microscope (TEM) examination were cut from the tensile-tested samples close to the fracture surfaces, and the discs were observed using JEM 200 TEM microscope.

3. Results and discussion

3.1. Microstructure of directly aged specimens

In order to detect the dissolution temperature of γ' phase, DSC analysis was performed. Fig. 2 shows the DSC curve of the HIPed MIM418 superalloy. The heating curve shows a slight depression at approximately 1125 °C, which indicates the formation of γ/cabides eutectic phase. The weak peak detected at 1190 °C might be caused by γ' dissolution. The minimum on the curve at 1316 °C indicates the liquidus for the alloy. On the cooling curve, the liquidus is observed at 1330 °C. The peak detected at 1291 °C is atributed to the solidus of the alloy. The peak detected at 1150 °C is ascribed to the reprecipitation of γ' phase. According to the dissolution temperature of γ' phase, HIPing temperature of 1200 °C was selected. Subsolvus and supersolvus solution temperature of 1180 ∼ 1220 °C was designed.

HIPing is carried out at the supersolvus solution temperature of 1200 °C, and it is thought to be able to act as the function of solution treatment. Thus, directly ageing of HIPed alloy is firstly investigated. Fig. 3 depicts the microstructure of the HIPed specimen and the specimens directly aged at 750 °C for varied periods of time. The HIPed sample consists of large primary γ' phase (1.1–2.6 μm) distributed along grain boundaries, irregular-shaped intragranular secondary γ' (0.5–0.8 μm) and very fine spherical tertiary γ' (105–152 nm), as shown in Fig. 3(a) and (b). The formation of multi-modal γ' precipitates is ascribed to the slow cooling rate from HIPing temperature. Fig. 3(c) presents the morphology of γ' precipitates in the 750 °C × 4 h aged specimen. Ageing treatment shows no obvious influence on primary γ', but it shows pronounced effect on the morphology and size of the secondary and tertiary γ' phase. Aged for 8 h leads to morphological instability of the secondary γ' phase, and several protrusions based on the near-spherical γ' phase develop. Aged for 48 h leads to increased population and coarsening of the tertiary γ' phase (Fig. 3d).

3.2. Microstructure of solution plus aged specimens

Directly aged superalloy usually suffers from coarse irregular γ' precipitates, which is harmful to mechanical properties, especially the hot ductility and stress rupture property. Therefore, the as-HIPed specimens were subjected to solution plus ageing treatment. Fig. 4 shows the influence of solution treatment temperature on γ' phase of MIM418 superalloy. The 1180 °C solution treatment produces a bimodal distribution of γ' particles, and a large amount of irregular primary γ' phase remains in the microstructure (Fig. 4a). Theoretically, γ' phase should dissolve in γ matrix completely above γ' solvus temperature (1190 °C). However, the 1200 °C solution treatment still retains some undissolved coarse