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Dynamic recrystallization of single-crystal nickel-based superalloy



Bing ZHANG^{1,2}, Chang-kui LIU^{1,2}, Jin-yi ZHOU^{1,2}, Chun-hu TAO^{1,2}

1. AVIC Failure Analysis Center, Beijing Institute of Aeronautical Materials, Beijing 100095, China;

2. Beijing Key Laboratory of Aeronautical Materials Testing and Evaluation, Beijing 100095, China

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Abstract: The dynamic recrystallization behavior of single-crystal(SC) superalloy SRR99 at low strain rate was investigated by high-temperature creep testing. The results show that dynamic recrystallization may take place after the uncoated samples have been creep-tested in air at high temperature and low stress for a long time. Both the threshold temperature and strain for the dynamic recrystallization of SC superalloy SRR99 at low strain rate are lower than those for the static recrystallization. Dynamically recrystallized grains with the depth less than 15 μ m are only located in the surface γ' -free layers, and the recrystallized grains are well-developed grains without columnar γ' precipitates within them. The dynamic recrystallization behavior of SC superalloy SRR99 at low strain rate is mainly related to high-temperature oxidation. Suitable protective coating can effectively prevent the dynamic recrystallization of SC superalloy SRR99 at high strain rate was also studied by high-temperature compression testing. At high strain rate, a higher temperature and larger strain are needed for the occurrence of dynamic recrystallization than at low strain rate, and the recrystallized grains have cellular structures with an amount of columnar γ' precipitates within them.

Key words: single-crystal superalloy; dynamic recrystallization; creep; compression

1 Introduction

With excellent high-temperature mechanical properties, single-crystal(SC) blades and vanes have been introduced into most of the advanced military and civil aircraft engines. SC superalloys were developed to overcome the limited mechanical performance of polycrystalline materials at high temperature. Their superior mechanical properties enable an increased service temperature and thereby an improved overall efficiency of turbines.

The superior high-temperature mechanical properties of SC superalloys mainly result from the elimination of the grain boundaries perpendicular to the main stress axis [1]. Since the recrystallized grains may introduce disadvantageous orientations and high-angle grain boundaries, they will dramatically reduce the creep rupture strength and fatigue life of SC components [2-4].

For conventional polycrystalline alloys, recrystallization is an effective approach to optimize

microstructure and mechanical performance, such as grain refining and strength improvement [5-8]. However, for SC superalloys, the aim of recrystallization researches is to find out effective measures to reduce or suppress surface recrystallization of SC even components. If SC components are deformed by improper operations, such as severe grit-blasting or polishing, and subsequently heated above the static recrystallization temperature, static recrystallization will take place [9-11]. In addition, SC components may be dynamically recrystallized because of the rigorous service condition even if the service temperature is lower than the temperature for static recrystallization [12].

Little information is available in the open literature on the dynamic recrystallization of SC superalloys, especially on the dynamic recrystallization mechanisms [12]. The present work investigated the dynamic recrystallization behavior of SC superalloy SRR99. The main purpose is to find out the dynamic recrystallization mechanism and thus to provide measures against the dynamic recrystallization of SC components.

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2 Experimental

The nominal composition of SC superalloy SRR99 was 8.5Cr, 5.0Co, 9.5W, 2.8Ta, 5.5Al, 2.2Ti, 0.02C, and balance Ni (mass fraction, %). As-cast SC bars were grown along [001] orientation through directional solidification process. The angle deviation from [001] direction was less than 15°. The as-cast SC bars were heat treated as follows: (1300 °C, 4 h) + AC + (1100 °C, 4 h) + (AC+870 °C, 16 h) + AC.

In order to investigate the dynamic recrystallization behavior at low strain rate, creep testing was carried out at high temperature and low stress. The samples with a diameter of 5 mm were machined out from heat-treated bars with their longitudinal axis parallel to the [001]-direction. Creep tests were carried out at 950–1040 °C and 100 MPa for 1000 h.

In order to investigate the dynamic recrystallization behavior at high strain rate, compression testing was carried out at high temperature. Cylindrical samples of $d8 \text{ mm} \times 15 \text{ mm}$ were machined out from heat-treated bars by electrical discharge machining(EDM) with their longitudinal axis parallel to the [001]-direction. Compression testing was carried out at the strain rate of 10^{-4} s^{-1} and the temperature of 1000, 1050 and 1100 °C, respectively, to the strain of 2%, 4% and 8%. In addition, compression testing was carried out at 1100 °C and the strain rates of 10^{-3} s^{-1} , 10^{-4} s^{-1} and 10^{-5} s^{-1} , respectively, to the strain of 4%.

The microstructures of the deformed samples were examined by optical microscopy(OM) and scanning electron microscopy(SEM). Metallographic samples were prepared by metallographic polishing. The polished samples were etched in the solution of 20 g CuSO₄, 100 mL HCl and 100 mL H₂O. Thin foils for transmission electron microscopy (TEM) were prepared from the compression-deformed samples. Discs perpendicular to longitudinal axis were cut down. Then they were mechanically ground to 50 μ m. Finally, the discs were electro-polished with a twin jet unit at -20 °C in a solution of 10% perchloric acid and 90% ethanol in volume fraction. The foils were observed using a JEOL200CX transmission electron microscope.

3 Results and discussion

3.1 Recrystallization behavior at low strain rate

The dynamic recrystallization of SC superalloy SRR99 at low strain rate is shown in Table 1. After creep testing at 950 °C, 100 MPa for 1000 h, no recrystallized grains are found. When the temperature is 1000 °C or higher, dynamic recrystallization occurs; with the increase of temperature, the depth of the recrystallized layer increases.

The dynamically recrystallized grains formed during creep testing are shown in Fig. 1. There exists a



Fig. 1 Dynamically recrystallized grains of SC superalloy SRR99 after high-temperature creep testing: (a) 1000 °C, 100 MPa, 1000 h; (b) 1020 °C, 100 MPa, 1000 h; (c) 1040 °C, 100 MPa, 1000 h

 Table 1 Dynamic recrystallization of SC superalloy SRR99 at high temperature and low strain rate

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Temperature/ °C	Stress/ MPa	Time/ h	Elongation/ %	Details of dynamic recrystallization
950	100	1000	0.28	No recrystallized grains are found
1000	100	1000	0.43	Recrystallized layer is discontinuous and average depth is about 4 µm
1020	100	1000	0.51	Recrystallized layer is discontinuous and average depth is about 5 µm
1040	100	1000	0.64	Recrystallized layer is discontinuous and average depth is about 8 µm

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