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Dynamic recrystallization of single-crystal nickel-based superalloy

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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 μm 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 components in service. In addition, the dynamic recrystallization behavior 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 even suppress surface recrystallization of SC 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 *d*8 mm×15 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 $CuSO_4$, 100 mL HCl and 100 mL H_2O. 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 μm. 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

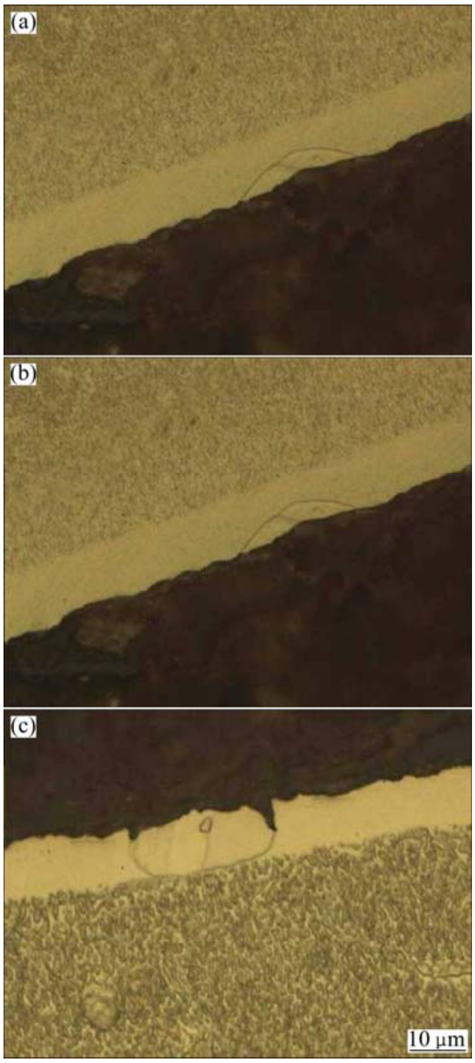

Fig. 1 Dynamically recrystallized grains of SC superalloy SRR99 after high-temperature creep testing: (a) 1000 °C, 100 MPa, 1000 h; (b) 1020 °C, 100 MPa, 1000 h; (c) 1040 °C, 100 MPa, 1000 h

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

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

γ'-free layer on the surface of each deformed sample. The dynamically recrystallized grains are only located in the surface γ'-free layers, and the depth of recrystallized grains is smaller than that of γ'-free layers. There is no doubt that the dynamically recrystallized grains nucleated on the surface and then grew up inward in the γ'-free layers. The surface selectivity of dynamic recrystallization is reasonable from the energy perspective. Nucleation of recrystallized grains on the free surface can reduce interface energy, so a much smaller deformation is needed for the nucleation of recrystallized grains on the free surface than in the center area, which has been proved by BÜRGEL et al [13], who found that at the solution temperature, the threshold strain for surface recrystallization of SC superalloy CMSX-11B was less than 1%, while that for recrystallization in the center area was over 10%.

The result of EDS analysis on the surface layer of the sample creep-tested at 1000 °C, 100 MPa for 1000 h is shown in Fig. 2. Compared with the matrix, the contents of Ti and Al in the γ'-free layer are lower, and those of Cr and Co are higher. During creep testing, Al and Ti diffuse towards the surface due to oxidation [14]. Because Al and Ti are γ'-forming elements, γ' precipitates near the surface dissolve and thus a γ'-free layer forms after a while. After creep testing at 1000 °C, 100 MPa for 1000 h, the γ' precipitates in the matrix have changed from cubical to rafted ones.

The dynamic recrystallization behavior of SC superalloy SRR99 at low strain rate has the following features. First, both the threshold temperature and strain for dynamic recrystallization are lower than those for static recrystallization. According to Ref. [15], the threshold temperature for static recrystallization of SC superalloy SRR99 is between 1000 and 1050 °C, and the threshold strain for static recrystallization at 1050 °C is

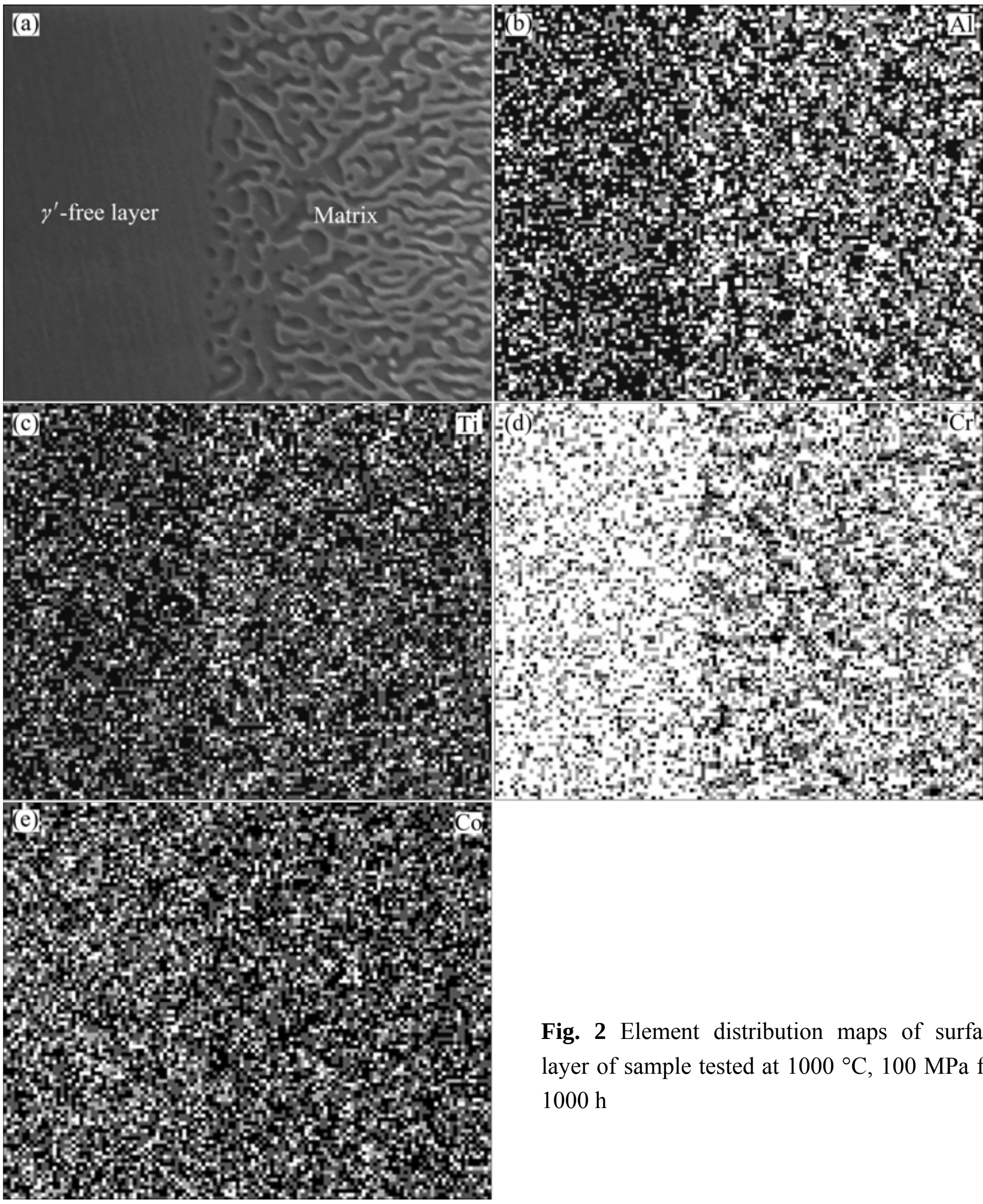

Fig. 2 Element distribution maps of surface layer of sample tested at 1000 °C, 100 MPa for 1000 h

higher than 4%. However, the threshold strain for dynamic recrystallization of SRR99 at 1000 °C is less than 1%. Second, the dynamically recrystallized grains are different from static ones formed at the same temperature. The dynamically recrystallized grains formed at 1000−1040 °C are well developed grains without columnar γ' precipitates within them, while the statically recrystallized grains formed at 1050 °C are cellular structures with an amount of columnar γ' precipitates within them [15]. Third, dynamically recrystallized grains are only located in the surface γ'-free layers, and their depth is usually less than 15 μm.

The features of dynamic recrystallization of SC superalloy SRR99 at low strain rate are mainly related to high-temperature oxidation. Under the effect of high temperature and tensile stress, there will exist a γ'-free layer near the surface. In the γ'-free layer, nearly all γ' precipitates dissolve, so the resistance to recrystallization significantly decreases, resulting in lower threshold temperature and strain for dynamic recrystallization. In addition, because few γ' precipitates exist in the γ'-free layer, few γ' precipitates are dissolved by the recrystallized grain boundaries during their movement. Low local solute concentration near the moving boundaries cannot provide sufficient supersaturation for the nucleation of columnar γ' precipitates, so there are no columnar γ' precipitates formed in the recrystallized grains [16].

In engineering, SC blades are usually coated. Surface coating can eliminate free surface so that the resistance to recrystallization increases. In addition, surface coating can relieve oxidation. So, it can be assumed that surface coating may prevent dynamic recrystallization during service. Figure 3 shows the microstructure near the surface of SRR99 sample coated with NiCrAlYSi after creep testing at 1000 °C, 100 MPa for 1000 h. No γ' free layer and recrystallized grains are seen.

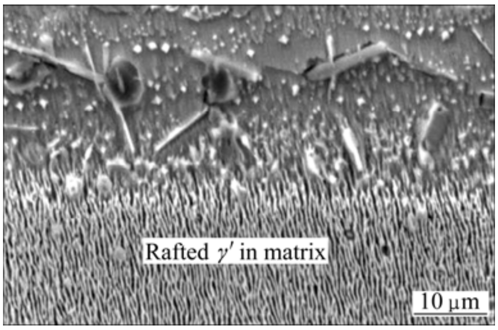

Fig. 3 Microstructure near surface layer of coated sample tested at 1000 °C, 100 MPa for 1000 h

3.2 Recrystallization behavior at high strain rate

Figure 4 shows the recrystallization tendency of SRR99 during high-temperature compression testing at the strain rate of 10^{-4} s^{-1}. From Fig. 4, it can be seen that when the strain is not greater than 8%, dynamic recrystallization didn't occur at 1000 °C or 1050 °C. At 1100 °C, the threshold strain for dynamic recrystallization is in the range of 2%−4%. Compared with the dynamic recrystallization at low strain rate, the threshold temperature and strain for dynamic recrystallization at high strain rate are significantly higher. During compression testing, oxidation is nearly negligible. The γ' precipitates retard the movement of dislocations, making the nucleation of recrystallized grains more difficult. In addition, because of the high strain rate, dislocations have no enough time to move and rearrange, which also postpones the nucleation of recrystallized grains.

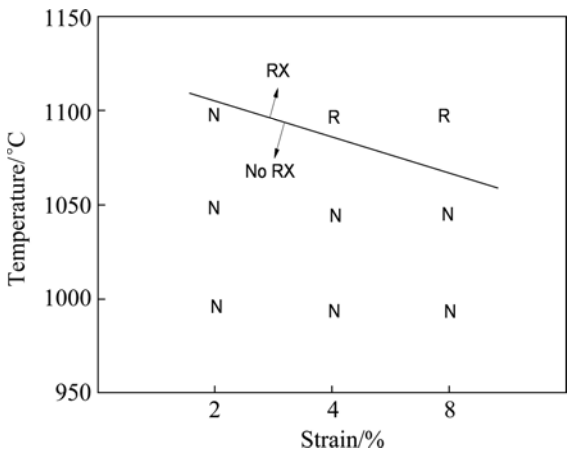

Fig. 4 Dynamic recrystallization tendency of SRR99 compressed at different temperatures and strain rate of 10^{-4} s^{-1} ('R' indicates that dynamic recrystallization occurred (RX) and 'N' indicates that dynamic recrystallization didn't occur)

The recrystallized grains formed during 1100 °C compression testing are shown in Fig. 5. The recrystallized grains have cellular structures with an amount of columnar γ' precipitates within them, similar to those formed during static recrystallization at the same temperature [15]. The recrystallized grains are only located at the surface layer, which also indicates that recrystallization tends to take place along the free surface. Cellular structure was also observed by PORTER and RALPH [16], when they studied the recrystallization of several nickel-based superalloys of different γ' volume fractions. According to their studies, the dissolution and reprecipitation of γ' phase were attributed to the high solubility and diffusivity of the moving recrystallization interface. During migration, recrystallized grain boundaries take nearly all the γ' precipitates that they encounter into solution. As a result, the moving grain boundaries soon become supersaturated with γ'-forming elements. This supersaturation is unstable and is relieved by the forming of columnar γ' precipitates.

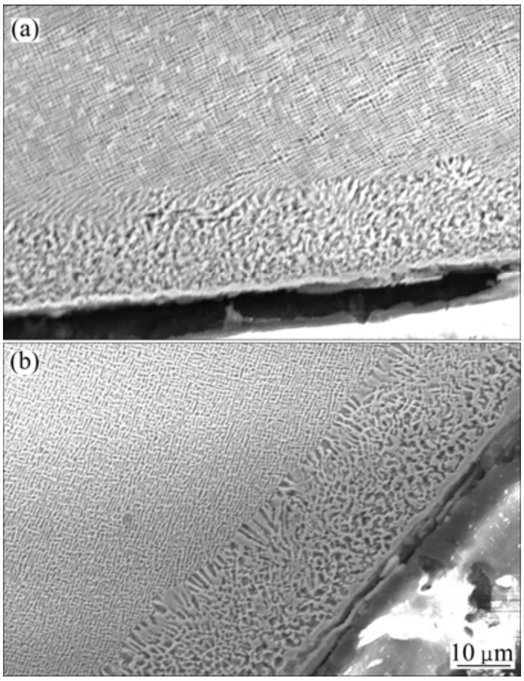

Fig. 5 Dynamically recrystallized grains of samples compressed at 1100 °C with strain rate of 10^{-4} s^{-1}: (a) ε=4%; (b) ε=8%

The effect of strain rate on recrystallization depth is shown in Fig. 6. The recrystallized depth increases with the strain rate declining. At higher strain rate, dislocations have less time to move and rearrange so that nucleation of recrystallized grains is postponed. As a result, recrystallized grains have less time to grow up, so higher strain rate results in smaller recrystallization depth. Figure 7 shows the dislocation configuration of the samples compressed at 1100 °C with different strain rates. At the strain rate of 10^{-3} s^{-1}, the dislocation at the interface of γ/γ' is relatively irregular; however, at the strain rate of 10^{-5} s^{-1}, the dislocation at the interface of γ/γ' is relatively regular, and some subgrains may be seen.

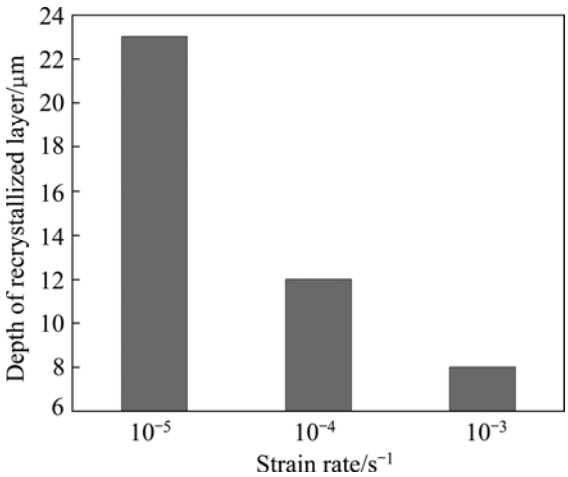

Fig. 6 Relationship between depth of dynamically recrystallized layer and strain rate compressed at 1100 °C (ε=4%)

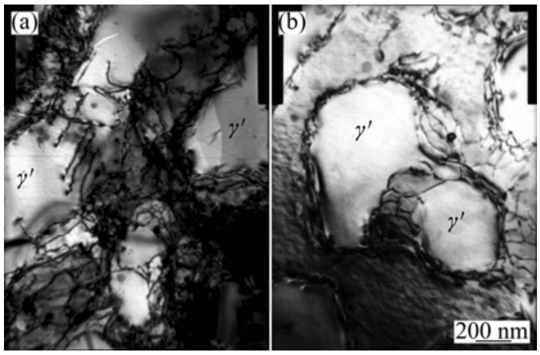

Fig. 7 Dislocation configurations of samples compressed at 1100 °C with different strain rates: (a) $\dot{\varepsilon}=10^{-3}$ s^{-1}; (b) $\dot{\varepsilon}=10^{-5}$ s^{-1}

4 Conclusions

1) Dynamic recrystallization may take place after uncoated SC superalloy SRR99 has 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 at low strain rate are lower than those for the static recrystallization. Dynamically recrystallized grains with the depth less than 15 μm are only located in the surface γ'-free layers, and the recrystallized grains are well-developed grains without columnar γ' precipitates within them.

2) 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 components in service.

3) At a high strain rate, a much higher temperature and larger strain are needed for the occurrence of dynamic recrystallization than at a low strain rate, and the recrystallized grains have cellular structures with an amount of columnar γ' precipitates within them.

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镍基单晶高温合金的动态再结晶

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摘 要：利用高温蠕变试验，研究 SRR99 单晶高温合金在低应变速率下的动态再结晶行为。结果显示，在大气环境且无涂层保护的情况下，长时间高温低应力作用会使单晶合金发生动态再结晶，动态再结晶的温度和临界应变量均低于静态再结晶，再结晶晶粒均位于试样边缘的 γ'相贫化层内，再结晶深度较浅，一般不超过 15 μm。在高温低应变速率下，再结晶以完整晶粒的形式发生，晶粒内无条状 γ'相存在。单晶合金在高温低应变速率下的动态再结晶行为主要与高温氧化有关。在实际服役条件下，表面涂层可有效抑制动态再结晶的发生。此外，还利用高温压缩试验，研究了 SRR99 单晶合金在高应变速率下的动态再结晶行为。在高应变速率下，单晶合金发生动态再结晶的温度和临界应变量均显著提高，再结晶以胞状组织的形式发生，晶胞内含有大量粗大的条状 γ'相。

关键词：单晶高温合金；动态再结晶；蠕变；压缩

(Edited by Hua YANG)