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ARTICLE

Microstructure Homogenization of GH4169 Superalloy in Shear-Compression Deformation State by Recrystallization Annealing

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Abstract: Regional microstructure characteristic always appears in shear-compression deformed GH4169 superalloy, which is detrimental to subsequent cold-rolling process in engineering. Recrystallization annealing treatments within temperature range of 1000–1080 °C and holding time range of 1–3 h were carried out to investigate the microstructure evolution behavior, and the cold-forming property of GH4169 superalloy was optimized by regulating the grain size. Results show that static recrystallization (SRX) grains are fully nucleated at 1000 °C and the original coarse grains are completely replaced by fine recrystallized grains. Bulges of high angle grain boundaries are the preferred nucleation points of SRX. At 1020–1060 °C, grain annexation takes place among adjacent SRX grains, causing partial grains to increase, while the original dynamic recrystallization (DRX) grains keeps tiny in the strain concentration region. Recrystallized grains (both SRX and DRX) uniformly grow up, with an average grain size of 87.89 μm at 1080 °C, at which the regional characteristic completely disappears, and the microstructure is significantly homogenized. Step twins appear at 1080 °C due to the SRX growth accidents, and the length fraction of twin boundaries (Σ3) reaches 35.8%, which can effectively improve the high temperature resistance of GH4169 superalloy. Ultimately, the optimal recrystallization annealing of shear-compression deformed GH4169 superalloy is determined as 1080 °C-1 h, followed by water cooling.

Key words: GH4169 superalloy; recrystallization annealing; microstructure; recrystallization; twins

Due to the excellent strength, corrosion resistance, and oxidation resistance at temperatures higher than 600 °C, GH4169 superalloy (GH4169 in short) has been selected as the preferred material for the heat transfer tubes used in the nuclear power system[1–6]. In the previous studies of Chen[7–8], the hot extrusion performance of GH4169 was investigated through shear-compression deformation (SCD), and the best SCD condition was determined as 1150 °C-0.1 s^{-1} with the highest fraction of dynamic recrystallization (DRX)[7].

However, due to the inherent strain inhomogeneity in shear-compression deformed specimen, three distinctive regions, named as slot region (SR), transition region (TR), and cylinder region (CR), appear[8]. Grain size and DRX fraction vary greatly in SR, TR, and CR, which causes a poor uniformity of microstructure. Such phenomenon is similar to the radial microstructure distribution of the industrial hot extruded GH4169 tubes, which increases the cracking possibilities in the following cold-rolling process in engineering[9–11].

To optimize the cold-forming property of GH4169, regulating the microstructure is necessary. Normally, solid solution (SS) at 960–980 °C is used as the microstructure homogenizing method of GH4169 after industrial hot extrusion[12–13]. However, the recrystallization temperature of GH4169 is about 1035 °C, which is significantly higher than the SS temperature[14]. It is difficult for static recrystallization

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(SRX) grains to nucleate during SS, and the regional characteristic of microstructure cannot be eliminated.

To effectively optimize the SCD microstructure of GH4169, recrystallization annealing (RA) experiments at 1000–1080 °C were carried out in this study. The theoretical significance is to investigate the microstructure evolution behavior of shear-compression deformed GH4169 during RA. Through promoting the nucleation of SRX, controlling the growth of recrystallized grains, and improving the length fraction of twin boundaries, the uniformity of microstructure was obviously improved. As a result, not only the cold-rolling performance but also the high temperature resistance of GH4169 can be enhanced in engineering.

1 Experiment

The experimental material was the GH4169 with a chemical composition of Ni-19.16Fe-17.95Cr-0.06C-0.18Si-0.03Mn-5.05Nb-2.93Mo-0.52Al-0.90Ti-0.12Co (at%). After receiving from Jiangsu Yinhuan Precision Steel Tube Co., Ltd, GH4169 was firstly shear-compression deformed at 1150 °C-0.1 s^{-1}. After that, RA was carried out on SCD specimen within the temperature range of 1000 – 1080 °C (20 °C gradient) with the holding time range of 1 – 3 h (0.5 h gradient), followed by water cooling. Subsequently, electron backscatter diffractometer (EBSD) and transmission electron microscope (TEM) coupled with selected area electron diffraction (SAED) analysis were used for microstructure analysis. EBSD specimens were prepared by electrolysis through the 10% perchloric acid alcohol solution with voltage of 20 V, current of 0.75 – 0.9 A, and duration of 15 – 25 s. TEM samples with a final thickness of 100 nm were prepared by twin-jet electropolishing with voltage of 5 kV.

2 Results and Discussion

2.1 Pre-selecting of RA conditions

The preliminary microstructure analysis reveals that the holding time has few influence on the microstructure of GH4169 during RA. The RA effect at a consistent temperature can be fully achieved by holding for 1 h. No grain growth is observed when the holding time is extended to 2 or 3 h. Additionally, the micro-morphologies after RA at 1020, 1040 °C, and 1060 °C are greatly similar. Therefore, shear-compression deformation state (SCDS), RA state at 1000 °C (RA-1000), RA state at 1040 °C (RA-1040), and RA state at 1080 °C (RA-1080) were selected for analysis and discussion hereinafter.

2.2 Evolution of micro-morphology

Supramaximal area scanning (3000 μm×450 μm) of EBSD was used to analyze the micro-morphology of GH4169 in different processing states, as shown in Fig. 1. In SCDS (Fig. 1a), complete DRX occurs in SR, while many coarse grains remain in TR, resulting in poor uniformity of microstructure. In RA-1000 (Fig. 1b), the DRX grains in SR hardly grow, and the coarse grains in TR are completely replaced by SRX grains. The grain of TR is significantly refined, and the microstructure uniformity of RA-1000 specimen is significantly improved compared with that of SCDS specimen. In RA-1040 specimen (Fig. 1c), the SRX grains in TR grow up in preference to the DRX grains in SR. The former continuously engulfs the latter from TR to SR, leading to obvious grain size difference again. In RA-1080 specimen (Fig. 1d), all the recrystallized (both DRX and SRX) grains grow sufficiently. No obvious difference in grain size is observed in SR, TR, and CR. The microstructure uniformity is effectively improved.

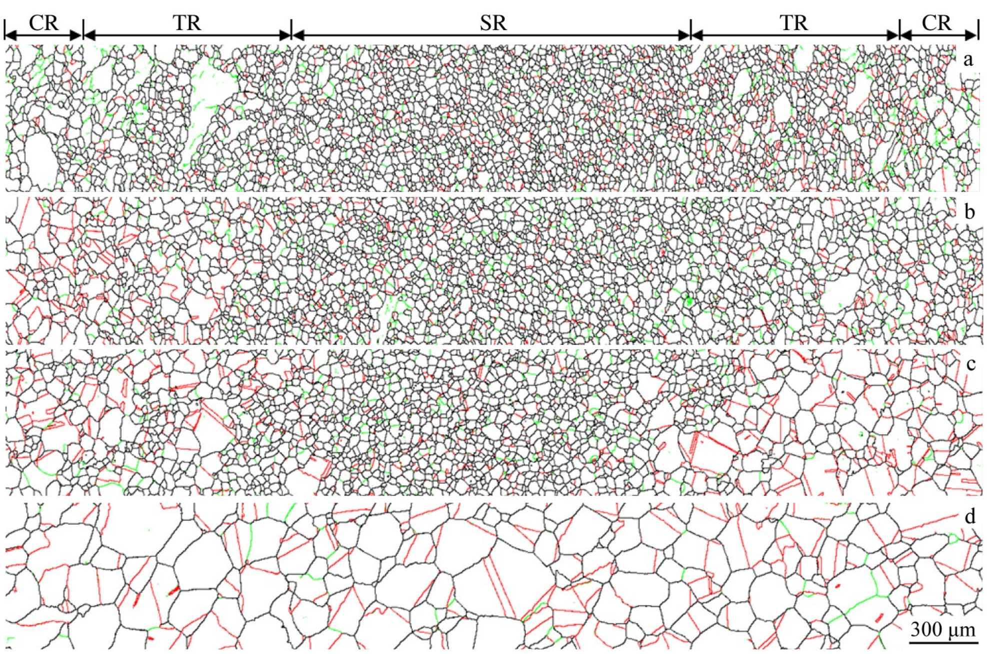

Fig.1 Micro-morphologies of GH4169 in different processing states: (a) SCDS, (b) RA-1000-1 h, (c) RA-1040-1 h, and (d) RA-1080-1 h

2.3 Statistics of grain size

Fig. 2 shows the statistics of grain size of GH4169 in different processing states. The average grain sizes (D_{avg}) of SCDS, RA-1000, RA-1040, and RA-1080 are 31.26 (Fig.2a), 29.88 (Fig. 2b), 45.30 (Fig. 2c), and 87.89 μm (Fig. 2d), respectively. The decrease in D_{avg} of RA-1000 compared to SCDS is due to the grain refining effect of SRX behavior. In different RA states, D_{avg} increases continuously with increasing the annealing temperature because of the growth of recrystallized grains. Additionally, left-skewed normal distribution characteristics occurs in SCDS, RA-1000, and RA-1040 specimens, where the fine recrystallized grains (<35 μm) occupy a high proportion (>65%). In contrast, the grain size of RA-1080 specimen shows a more standard normal distribution characteristic with the highest microstructure uniformity.

2.4 Statistics of grain boundary

Fig.3 shows the statistics of grain boundary of GH4169 in different processing states. Compared to SCDS, the length fraction of sub-grain boundaries (SGBs) and twin boundaries (Σ3) decreases in RA-1000 because of the nucleation of SRX grain, causing the increase in high angle grain boundaries (HAGBs). In the experimental RA states, HAGB and SGB decrease while Σ3 increases with increasing the temperature. The reason is that SGBs absorb nearby movable dislocations and are transformed into HAGB during the growth of recrystallized grains in RA[15–16]. HAGB will be further transformed into Σ3 due to the growth accidents[17]. In particular, the length fraction of Σ3 reaches 19.5% and 35.8% at RA-1040 and RA-1080, respectively. The HAGB network is broken, and the interfacial energy is efficaciously reduced[18–19], which is conducive to the high temperature resistance improvement of GH4169.

2.5 SRX & twinning behavior

The nano-scaled SRX grains are found in RA-1000 specimen through TEM, as shown in Fig.4a and 4b. In Fig.4a, significant dislocation plugging near HAGB results in a high

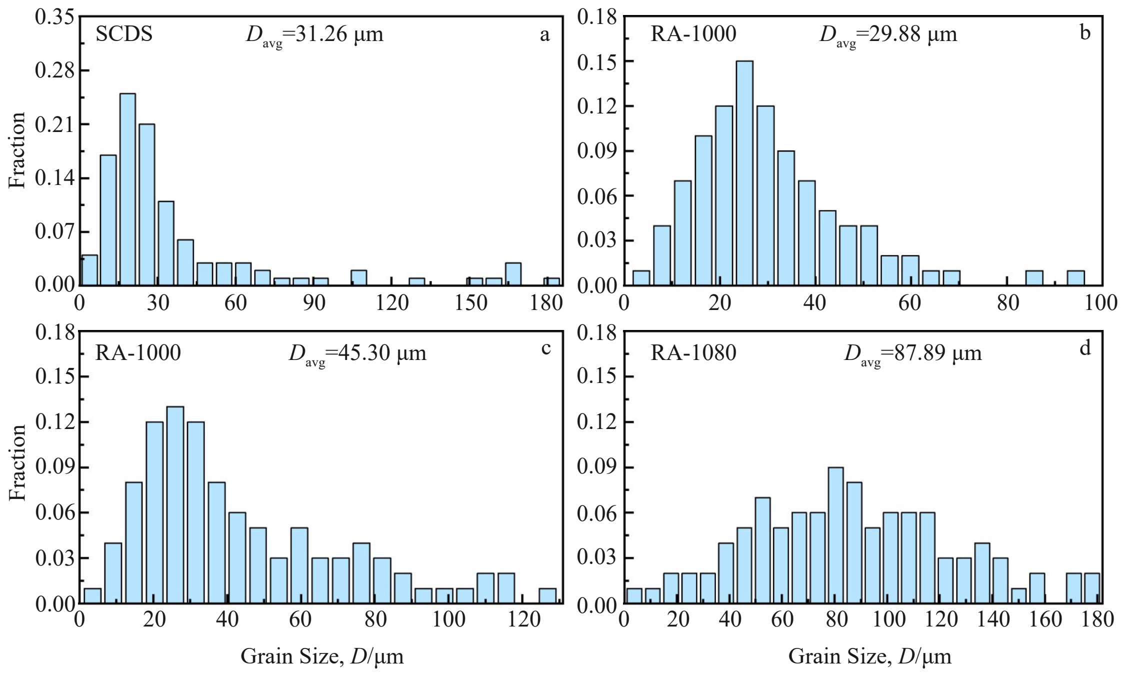

Fig.2 Statistics of grain size of GH4169 in different processing states: (a) SCDS, (b) RA-1000, (c) RA-1040, and (d) RA-1080

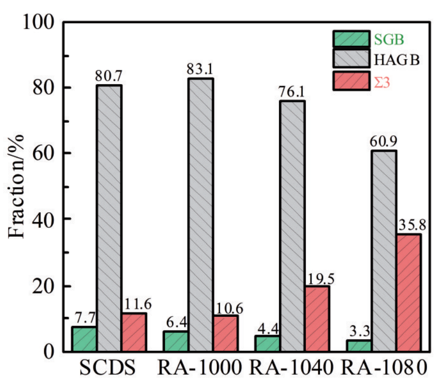

Fig.3 Statistics of grain boundary of GH4169 in different processing states

storage energy, which drives the migration and bulging of HAGB and promotes the nucleation of SRX. The SRX grains grow into the interior of parent grains. The SRX grain boundaries migrate towards the high dislocation density side and absorb the movable dislocations on the way. Such phenomenon is much similar to the discontinuous dynamic recrystallization behavior[20–21]. In Fig. 4b, the flattened SRX grains nucleate along the parallel direction of HAGB, which accelerates the disappearance of the original grain boundaries. As a result, the typical necklace structure will form[22]. In RA-1040, the annexation behavior of recrystallized grains (marked by green arrows) is observed in Fig. 4c_1. The orientations between these recrystallized grains are gradually converged during RA and the original grain boundaries gradually

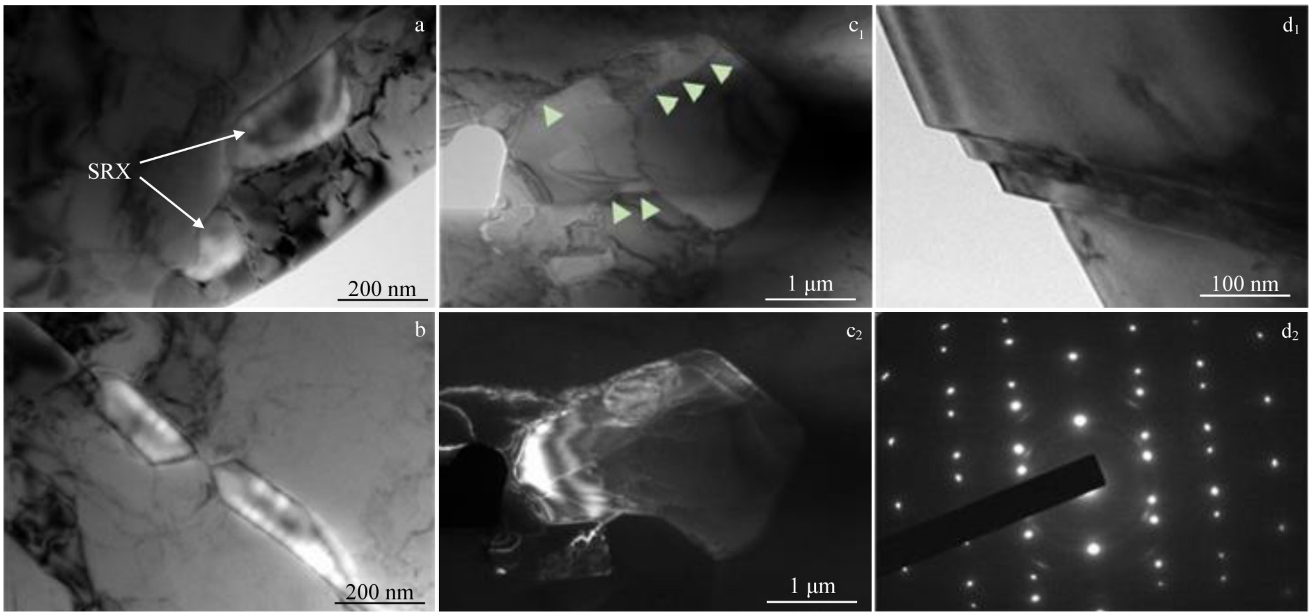

Fig.4 TEM images of GH4169 after RA: (a–b) SRX in RA-1000; (c_1–c_2) bright and dark field images of grain annexation in RA-1040; (d_1) step twinning in RA-1080; (d_2) SAED pattern of step twinning

disappear, resulting in the growth of the recrystallized grains. When the grain boundary migrates in RA-1080 specimen, the original stacking sequence of {111} crystal faces changes due to micro-accidents, and then the stacking faults form and grow into the step twins[23–24], as shown in Fig.4d_1. A clear double-dots SAED pattern is shown in Fig.4d_2. The matrix is split in steps, showing a smooth shear characteristic, which indicates that coordinated twinning occurs in GH4169[25].

3 Conclusions

1) RA within temperature range of 1000 – 1080 °C and holding time range of 1 – 3 h can eliminate the regional characteristic of SCDS and improve the microstructure homogeneity.

2) SRX grains nucleate sufficiently in RA-1000 specimen, achieving the first homogenization of microstructure. In RA-1040, the SRX grains of TR grow up in preference to the DRX grains of SR, and the mixed-grain characteristic appears again.

3) Uniform growth of recrystallized grains (both SRX and DRX) takes place in RA-1080 specimen with an average grain size of 87.89 μm. Meanwhile, the length fraction of Σ3 reaches 35.8%. That is, both the cold-rolling performance and the high temperature resistance of GH4169 can be effectively improved in RA-1080. Therefore, 1080 °C-1 h is the optimal RA condition of GH4169.

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通过再结晶退火实现剪切-压缩变形态 GH4169 高温合金的显微组织均匀化

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摘　要：GH4169 高温合金在剪切-压缩变形后容易出现区域性微结构特征，这不利于后续的冷轧加工。在1000~1080 ℃温度范围内，保温1~3 h条件下进行再结晶退火处理以研究GH4169高温合金的微观组织演变行为，并通过调节晶粒大小优化其冷成形性能。结果表明，静态再结晶（SRX）在1000 ℃时完全形核，原有的粗大晶粒完全被细小的再结晶晶粒取代。大角度晶界（HAGB）的弓出位置是SRX的首选形核点。经1020~1060 ℃退火后，部分相邻的SRX晶粒之间发生吞并，导致一部分晶粒的尺寸增大，而初始动态再结晶（DRX）晶粒在应变集中区域仍保持微小的尺寸。再结晶晶粒（包括SRX和DRX）经1080 ℃退火后均匀长大，平均晶粒尺寸为87.89 μm，此时显微组织的区域特征完全消失，组织均匀化程度明显提高。由于SRX的生长事故，经1080 ℃退火后出现了阶梯状孪晶，且孪晶界（Σ3）的长度分数达到35.8%，这有利于提高GH4169高温合金的高温持久性能。剪切-压缩变形态GH4169高温合金的最佳再结晶退火工艺最终被确定为1080 ℃保温1 h，水冷。

关键词：GH4169高温合金；再结晶退火；显微组织；再结晶；孪晶

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