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Microstructural evolution on the initiation of sub-solvus recrystallization of a grit-blasted single-crystal superalloy

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ABSTRACT

In this work, the initiation of cellular recrystallization of a grit-blasted single-crystal superalloy has been investigated by combined TEM and EBSD techniques. Based on microstructure observations of the grit-blasted surface, the formation of dislocation tangles in γ channel and stacking faults in γ' precipitates has been validated. After subsequent annealing at 1100 °C for 30 min, in the original matrix, neighboring γ' precipitates have partially dissolved and merged into localized irregular agglomerates; concurrently, dense dislocation walls have formed and transformed to subgrain boundaries. For cellular recrystallized region, the formation of twins played a significant role in lowering the energy for sub-solvus recrystallization, which facilitated the transformation process.

1. Introduction

Nickel-based single-crystal superalloys are extensively used for turbine blades of aircraft engines to withstand extreme conditions, of which the thermal stability during service is invariably a long-lasting issue [1]. However, at the start of the production route by investment casting, the impeded shrinkage of the metal during cooling caused by a stiff ceramic mold, results in local residual stresses, which is highly geometry-dependant and influenced by core and mold yielding and crushability as well as by the cooling conditions [2]. On the other hand, as an easy-processing surface finishing method, grit-blasting procedure before thermal barrier coating [3], which is often conducted to obtain a surface with roughness Ra within somewhat microns [4] for improving adhesion between the film and the substrate, is also a straining process. Thereby, accompanying subsequent heat treatments, or under rigorous long-term exposure during service, the thermal-mechanical prerequisite would facilitate the occurrence of recrystallization in the internal surfaces, specifically on the leading and trailing edges of as well as at the root or the cooling channels of gas turbine blades [5]. Besides, due to the thin wall structure [6] and thickness debit effect [7] of single-crystal components, even micron-sized recrystallization layer would lead to the failure of the whole blades. In addition, different from the well-defined deformation step such as indentation [8], the industrially practical grit-blasting causes a rather different distribution of the stored energy, and is seldom simulated or investigated. In this regard, the present study aims at providing new insights with respect to the grit-blasting induced microstructure change and its further influence on the constrained beginning stage of recrystallization events with the complicated interactive effects of initial recovery and nucleation to be considered.

2. Materials and methods

The second-generation nickel-based single-crystal superalloy DD6 [9] with tensile and creep rupture properties comparable to René N5 and CMSX-4 was used in this study. The specimens of DD6 were grit-blasted at a pressure of 0.25 MPa for 30 s. SiO_2 spheres with a radius of 75 μm were used. The grit-blasted pieces were tubed in vacuum silica glass, and then subjected to heat treatment at 1100 °C for 30 min, followed by air cooling. The bright field transmission electron microscopy (TEM) images of the specimens were obtained on a FEI Tecnai G^2 20 TEM. For TEM observation, the samples were cut along the section parallel to the recrystallized surface by electric discharge machine (EDM) followed by carefully grinding to thickness of 30 μm with the fine metallographic grinding papers from the opposite side to the free surface, and then electropolished with 8% perchloric acid solution in ethanol at 25 V and −25 °C. The local crystallographic misorientation across a section was characterized using electron

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backscattering diffraction (EBSD). Data was acquired under the scanning step size of 0.11 μm using a Zeiss-Merlin scanning electron microscope (SEM) with an Oxford Instruments *hkl* channel 5 EBSD system at an acceleration voltage of 20 kV.

3. Results and discussion

Fig. 1(a) presents a typical secondary electron SEM image of the specimen after grit-blasting. It is evident that, compared with the normal shape of cuboidal γ' precipitates in the inner matrix, severely distorted morphology of γ' precipitates formed in the cold-worked surface layer. As an easy-processing surface finishing method, the multi-directional grit-blasting procedure on the alloy surface may lead to interaction of dislocations of various slip systems through dislocation generation and multiplication. Further magnified TEM image (Fig. 1(b)) demonstrates the high density dislocation tangles residing in the continuous γ matrix channel. These entangled dislocations are randomly arranged without preferable sliding orientations. Besides, it can be noted that majorities of γ' precipitates are sheared by some ribbon-like defects.

As shown in Fig. 2, the ribbon-like defects were further determined to be stacking faults in the ordered γ' ($L1_2$) lattice, the formation of which is considered to be ascribed to the low flow stress of ~150 MPa for Ni_3Al at room temperature [10] and low stacking fault energy in the present alloy as a result of its high cobalt content (9 wt%) [11]. Both the dislocations in γ and stacking faults in γ' would provide the driving force for nucleation and growth of recrystallization during subsequent annealing.

The grain structures and the dislocation substructures of the grit-blasted alloy annealed at 1100 °C for 30 min were examined by TEM to confirm the initiation of the cellular recrystallization (CR) process. As shown in Fig. 3(a), the morphology of γ' precipitates has frequently changed from cuboidal (Fig. 1) to more oval as a result of coagulation. Since the residual stresses assist dissolution of the existing phases [12], microstructural inhomogeneities enabled partial dissolution of γ' precipitates in the grit-blasted surface layers, which has also been captured by our previous work [13]. Accordingly, it is considered that the neighboring γ' particles have partially dissolved and merged into the localized oval agglomerates. Accompanying this, the grit-basting induced stacking faults have almost disappeared in γ' precipitates with certain strain energy release. However, the stable dislocation anchoring around the γ' precipitates indicates that the driving force for recrystallization can be hardly reduced by recovery. On the other hand, the annihilation and rearrangement of dislocations have also led to the occasional formation of tangled dislocation walls, as shown in Fig. 3(b). It should be noted that the misorientation across the walls is usually smaller than 2°. The progressive increment of misorientation between neighboring regions which were separated by dislocation walls would be realized by accumulating and annihilating more dislocations in boundaries. The incidental intersecting dislocation walls indicate their different slip planes, leading to the formation of bulges which would probably provide the nucleation sites [14].

It has been acknowledged that as long as the dislocations are knitted into the γ'/γ interface, the dislocation structure is extremely resistant to recovery; these dislocations supply the mechanical energy that is released by primary recrystallization as soon as the γ' phase disappears [2]. Therefore, once the critical dislocation density for initiating the recrystallization is obtained, the instantaneous power dissipation, i.e. the nucleation takes place. As shown in Fig. 3(c), one CR aggregate, appearing to result from the movement of a distinct nucleation front into the grit-blasting affected original matrix (OM), with various curvatures along the reaction front was detected. In addition, the formation of twinning near the nucleated grain boundaries (Fig. 3(d)) also indicates its low stacking fault energy [15] as discussed above. Once the recrystallization reaction front formed, dislocations would pile up in the vicinity of the reaction front and the mobile interface would consumed them continuously as a dislocation sink for further CR progressing.

The EBSD Euler angle orientation map of the annealed alloy is shown in Fig. 4(a), in which the various colors demonstrate different orientations and annealing twins can be directly revealed. Fig. 4(b) illustrates the local misorientation map, which is interpreted in terms of density of geometrically necessary dislocations [16] to present the local strain value. As can be seen, the internal misorientation in the emphasized circles is rather large, indicating a high density of dislocations, which would contribute to further nucleation or growth process. It should be noted in the quantitative statistic information in Fig. 4(c) that, the majority (93.8%) of grain boundaries are low angle grain boundaries with the misorientation lower than 5°. Combining with above TEM analysis, it can be inferred that the subgrain boundaries (lower than 2°) with dominant fraction of 84%, mainly come from the OM region after dislocation recovery as shown in Fig. 3. Furthermore, it should also be mentioned that, at the intensely beginning stage of recrystallization, most of the high angle grain boundaries have the $\sum 3$ twin relationship. This gives strong vivid and valid evidence that the formation of twins played a significant role in lowering the energy for sub-solvus recrystallization, which facilitated the transformation process.

4. Conclusions

The initiation stage of cellular recrystallization of a grit-blasted single-crystal superalloy has been investigated by the combined TEM and EBSD techniques. Based on microstructure observations of the grit-blasted surface, the formation of dislocation tangles in γ channel and stacking faults in γ' precipitates has been validated. After subsequent annealing at 1100 °C for 30 min, in the original

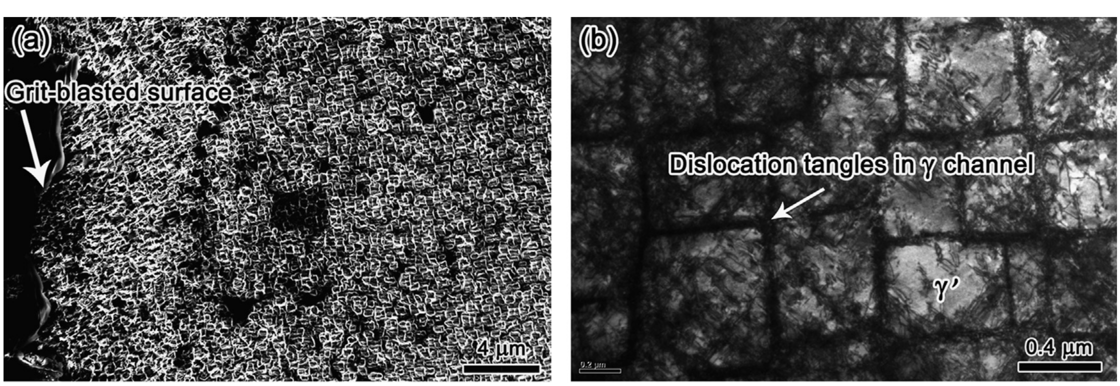

Fig. 1. Typical secondary electron SEM image (a) and bright field TEM image taken from the surface layer (b) of the grit-blasted DD6 alloy.