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The highly twinned grain boundary network formation during grain boundary engineering

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ABSTRACT

Grain boundary (GB) engineering was carried out on a Ni-based alloy with pre-precipitated carbides at GBs. Microstructure with coexistence of $\sum 3^n$ boundaries formed during annealing and traces of disappeared original GBs were observed during GB-engineering. The newly formed $\sum 3^n$ boundaries are in different positions with that of disappeared original GBs, which indicates the recrystallization front GBs moved into the deformed matrix and swept away original GBs. It is a typical recrystallization process rather than GB decomposition. Strain induced boundary migration initiated the recrystallization. Grain-cluster formed with the continuous occurrence of twinning-events in the wake of the migrating recrystallization front GBs during sweeping away the deformed matrix. Finally large grain-clusters with highly twinned interconnecting $\sum 3^n$ boundaries were formed.

1. Introduction

Grain boundary engineering (GBE) for enhancing intergranular degradation resistance has been successfully applied to many alloys [1–4]. The high proportion of $\sum 3$ boundary and twin-related high order $\sum 3^n$ boundary was formed during GBE-processing. Multiple-twinning [5,6] is the basic process of $\sum 3^n$ ($n=1, 2, 3\ldots$) boundaries formation. But different models were proposed to understand the GB network (GBN) evolution. Many papers suggested that it is a local GB migration process [2,3] following strain induced boundary migration (SIBM) mechanism because the low-strain during GBE is not enough to induce full recrystallization, for examples $\sum 3$ regeneration model proposed by Randle [1], boundary decomposition mechanism by Kumar [2] and twinning emission model by Shimada [3]. Following these mechanisms, random boundary (RB) connectivity would be interrupted when $\sum 3^n$ boundary proportion is high enough [1,7]. Random percolation theory shows that about 50–75% special boundaries would interrupt the connectivity [8]. However, many experiments show that even over 80% special boundaries can't stop intergranular corrosion propagation [7], and RBs are connected still in long range [4,9] especially when Palumbo-Aust criterion was used to define CSL characters. These results are against the random percolation theory.

Our previous works [4,9] show that the GBN evolution during GBE is a recrystallization process which is featured by the formation of large grain-clusters. The grain-cluster is a twin-related-domain [4,5]: all of the inner boundaries have $\sum 3^n$ misorientations, and the outer boundaries are crystallographically random. The grain-cluster is formed by multiple-twinning [5,6] starting from a single recrystallization nucleus. This mechanism would form connected RBs inevitably, although the $\sum 3^n$ boundary proportion is more than 80% and associated with large grain-cluster GBN.

This work investigated the GBN evolution during GBE-processing. Microstructure with coexistence of $\sum 3^n$ boundaries formed during annealing and traces of disappeared original GBs was observed to understand the highly twinned GBN formation during recrystallization.

2. Experimental

Ni-based Alloy 690, which is usually used as steam-generator-tube material in pressurized water reactors, is the experimental material. Solution-annealed alloy 690 was aged at 715 °C for 15 h for carbides precipitation. The aged samples were tensile deformed with elongation of 8%. Different annealing approaches were carried out on the deformed samples, as shown in Table 1. All thermo-treatment were carried out in vacuum quartz capsules and broke the capsules simultaneously after the given time for water quenching.

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EBSD (electron backscatter diffraction, HKL/Channel 5) system, which is affiliated to a CamScan Apollo 300 scanning electron microscope (SEM), was used to investigate the GB characters. The sample surface was finally polished by electro-polishing for EBSD measurement. HKL/Channel 5 and TSL/OIM were used to analyze EBSD data. GB was defined by the misorientation more than 2°. The CSL boundaries were defined according to the Palumbo-Aust criterion.

3. Results and discussion

Fig. 1 shows EBSD maps and SEM images of sample A, B and C. The background of EBSD map was colored using gray-scale according to grain average misorientation which is the average misorientation between each neighboring pair of indexed points within a grain. The $\sum 3^n$ ($n=1$, 2, 3) GBs proportions are 48.2%, 57.9% and 74.7% for sample A, B and C respectively, As shown in Table 1. The RB density of starting state sample A is high. Sample C has high proportion of $\sum 3^n$ GBs, and large grain-clusters were formed. The highlighted zone in Fig. 1(c) is a grain-cluster. In sample B, some regions have many $\sum 3^n$ GBs while the others have high density of RBs.

Table 1
Thermo-mechanical processing and the twin-related GBs proportion of samples.

Sample ID	Thermo-mechanical treatments	Summation of $\Sigma 3^n$ boundaries (%) ($n=1$, 2, 3)
A	Solution annealing+715 °C × 15 h	48.2
B	State A+8% tensile deformation+1100 °C × 5 min+WQ	57.9
C	State A+8% tensile deformation+1100 °C × 1 h+WQ	74.7

The grain average misorientation can be used to judge whether a grain is recrystallized or non-recrystallized [9]. Fig. 1(a)–(c) shows that sample A and C have uniformly low grain average misorientation, but sample B is clearly divided into dark gray regions and light gray regions. Sample B is partially-recrystallized. The dark gray regions with high density RBs are non-recrystallized. The light gray regions are recrystallized, in which some large grain-clusters can be observed like in sample C. The change from sample B to C indicates that large grain-clusters grew into the deformed matrix during annealing.

Fig. 1(d) and (f) shows sample A has carbides at GBs, but no carbides in sample C. The pre-existing carbides disappeared during annealing. However, retained carbides are observed in sample B (Fig. 1(e)). The pre-existing carbides did not fully dissolve during the shorter time annealing. Some GBs are still pinned by carbides (red arrows pointed). They are original GBs which did not move during the annealing. The other GBs without carbides should be newly formed GBs (white arrows pointed). These results also indicate sample B is partially-recrystallized. In addition, a lot of carbides exist in line and observably not at GBs but within grains, such as the black arrows pointed. These positions should be the traces of original GBs, but these GBs were swept away owing to the recrystallization and the carbides were left at their original positions. Therefore, the retained carbides indicate the traces of the disappeared original GBs. The newly formed GBs during recrystallization and the traces of disappeared original GBs can be observed in the same SEM image. Following this method, the GBN evolution during annealing of GBE-processing can be revealed by comparing the positions of recrystallized GBs and original GBs.

Fig. 2 shows some SEM images and corresponding OIM maps of sample B. The OIM maps show the newly formed GBs during recrystallization and the retained initial GBs, and traces of the disappeared original GBs were also manually drawn. The background of these OIM maps was shaded according to grain average

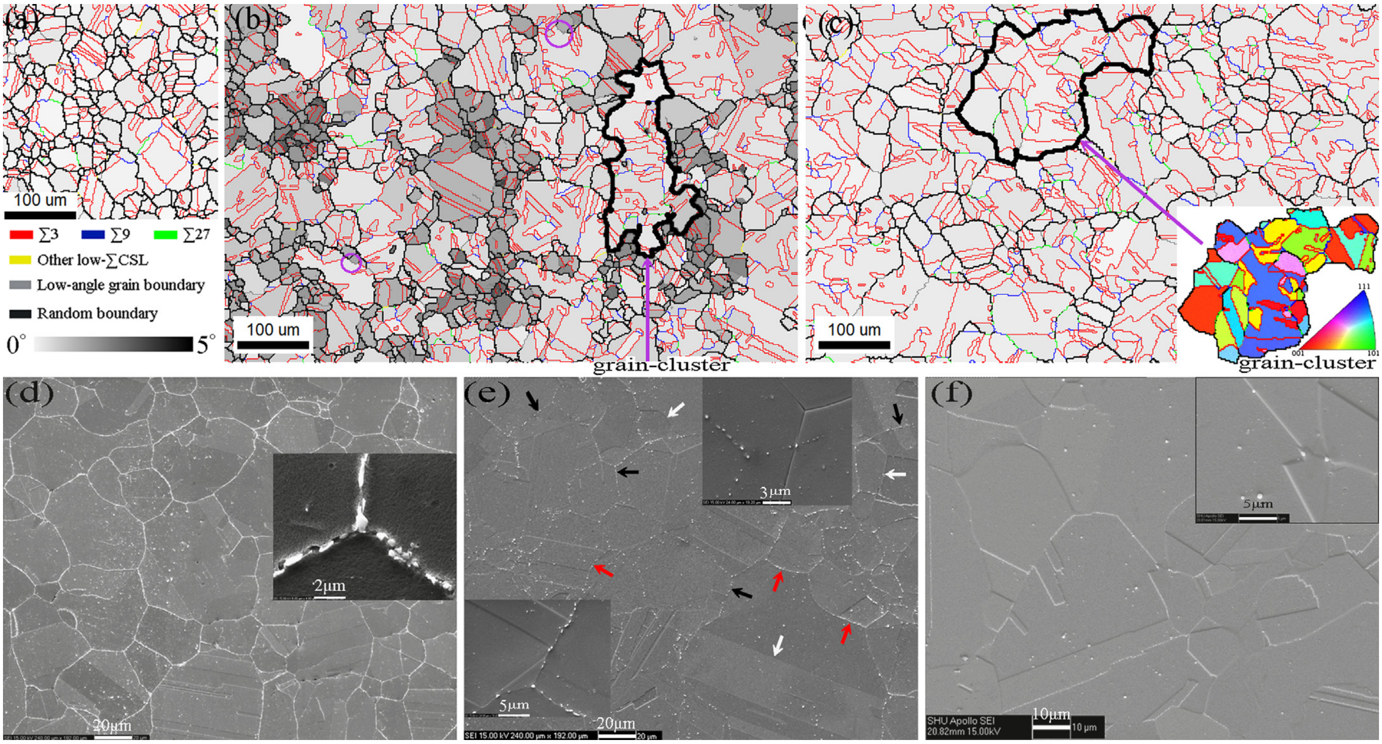

Fig. 1. EBSD and SEM images of samples A (a) and (d), B (b) and (e) and C (c) and (f). (a)–(c) were colored using gray-scale corresponding to grain average misorientation. The bold-outlines highlighted regions are two grain-clusters. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)