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Influence of intermediate annealing on the microstructure and texture of Ni-9.3at%W substrates

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Abstract: The effects of intermediate annealing (IA) on the microstructure and texture of Ni-9.3at%W substrates have been investigated by using electron backscattering diffraction and X-ray diffraction. Results suggest that IA can optimize the homogeneity of deformation microstructure. Higher IA temperatures (without undergoing recrystallization during IA) will increase the copper-type components of deformation texture and improve the content of cube texture after recrystallization. Sharp cube texture (97.2%) can be obtained at the optimum IA temperature of 650°C. The mechanism underlying the transition of deformation texture can be interpreted as that IA increases the dislocation slipping ability and suppresses the twinning deformation of Copper orientation in the subsequent rolling process. The observed strengthening of cube texture as a result of IA treatment is presumably attributed to the reduction of noncube nucleation and the optimization of preferential growth surrounding the cube nuclei.

Keywords: nickel tungsten alloys; annealing; textures; rolling

1. Introduction

Rolling-assisted biaxially textured substrates (RABiTS) method is a promising approach for the manufacturing of high-temperature superconducting (HTS) YBCO tapes that can carry large currents in high magnetic fields at 77 K. Among the different textured substrates prepared by using RABiTS, Ni-W alloy is considered to be the most promising material owing to its superior strength, beneficial magnetic properties, and oxidation behavior [1-4]. In particular, Ni5W, which is a Ni alloy with 5at% W, is widely used as the substrate material for coated conductors [2]. However, the main drawback associated with Ni5W is its ferromagnetic property at 77 K, which is the typical operating temperature of high-T_c superconductors. In principle, the fabrication of a nonferromagnetic substrate demands a W content of about 9.5at% [1].

However, the mere increase in the content of W leads to two main disadvantages: (1) the rolling texture transforms from copper-type to brass-type, (2) the cube texture decreases dramatically after recrystallization annealing, due to the decreased stacking fault energy (SFE) [5]. Thus far, several efforts have been proposed to overcome this problem, including the preparation of Ni-W alloy by powder metallurgy, increasing the rolling temperature, and adopting stage annealing [6-8]. It has been recently demonstrated that the intermediate annealing (IA) during cold-rolling process can optimize the deformation texture and also result in strong cube texture after recrystallization in high W content Ni substrates [9-11]. However, the detailed mechanism pertaining to the effects of IA is still unclear.

Therefore, in the present study, we have elucidated the effect of IA temperature on the microstructure and texture of Ni-9.3at%W substrates.

2. Experimental

In the typical experiment, Ni and W ingots (purity of 99.96%) were melted in an induction furnace and then cast into a round bar with a diameter of 30 mm in argon atmosphere. The round rod thus obtained was then hot-forged to a

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rectangle-shaped rod of dimension 20 mm× 40 mm at 1200°C. Subsequently, the rod was cold rolled from 20 to 0.2 mm. The thickness reduction was less than 10% per pass, and the total reduction of thickness by cold rolling was 99%.

After the rolling process of 90% total reduction, each sample was intermediate annealed (IA) eight times at the same temperature for 45 min. In this study, different samples were annealed at different IA temperatures. The reduction of thickness between two annealing steps was less than 30%. After each annealing, the substrate was cooled down to room temperature to handle the subsequent cold-rolling process. Finally, the rolled strip was treated by a two-step annealing (TSA) process under Ar atmosphere, in order to obtain the recrystallized texture. The first step of TSA involves annealing at 700°C for 30 min, while the second step in the process involves annealing at 1100°C for 2 h [8].

The rolling texture was investigated by using X-ray diffraction (XRD). Furthermore, the single component deformation texture of the crystals was calculated by the orientation density functions from the X-ray pole figures, using the MTEX toolbox [12]. The microstructure, recrystallization texture, and the local deformation texture were examined by using a field-emission scanning electron microscope (FE-SEM, Tescan Mira3) equipped with the electron back-scattering diffraction system (EBSD, Oxford Instruments).

3. Results and discussion

3.1. Microstructure of 99% reduced substrates

Figs. 1(a) and 1(b) show the typical microstructure of 99% reduced samples without and with IA treatment. In the EBSD maps, the grain boundaries of >2° and >15° are represented in gray and black lines, respectively, while the Σ3 twin boundaries are shown in red lines. In addition, the colors indicate different ideal orientations with a maximum deviation of 15°. As can be seen from Fig. 1(a), the microstructure of substrate without IA is inhomogeneous. The lamellar boundaries (LBs) are not exactly parallel to the rolling direction (RD). In addition, shear bands are observed. On the other hand, the typical microstructure of Ni-9.3at%W substrate with IA at 650°C (Fig. 1(b)) indicates a homogeneous deformation microstructure. The LBs are basically parallel to RD, with a broader average width. This could be attributed to the release of stored energy during the IA treatment.

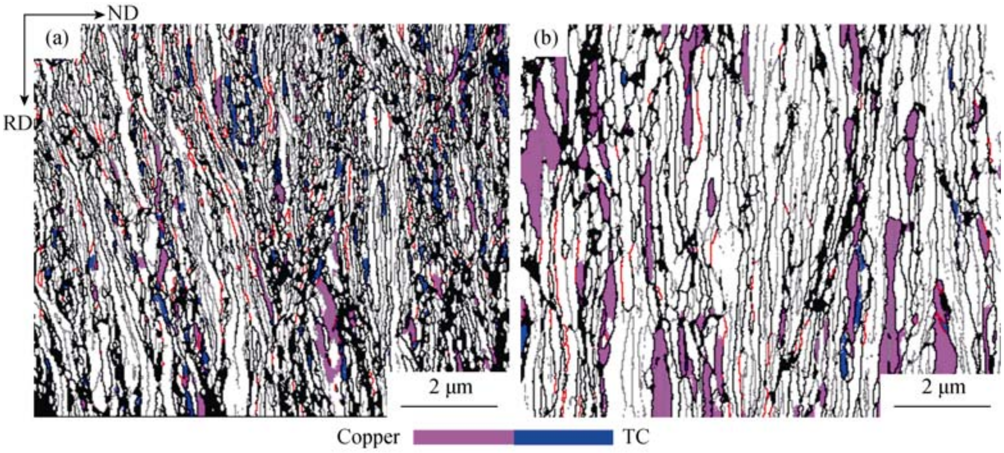

Fig. 1. Microstructure of 99% reduced cold-rolled samples without IA (a) and with IA (b) at 650°C.

3.2. Deformation texture of 99% reduced substrates

In general, the main rolling textures for large deformed fcc metals are Copper $\{112\}<111>$, S $\{123\}<634>$, Brass $\{110\}<112>$, and Goss $\{110\}<001>$ [13]. Table 1 lists the volume fraction of these deformation texture components, as determined by using XRD analysis. For IA temperature below 650°C, it can be seen that the contents of Copper $\{112\}<111>$ and S $\{123\}<634>$ components increase significantly with the increase in IA temperature. On the other hand, the Brass $\{110\}<112>$ component increases initially, followed by a slight decrease with an increase in IA temperature from 500 to 650°C. Meanwhile, the Goss $\{110\}<001>$ component shows a drastic decrease, and almost disappears in the 650°C IA sample. The increase in Copper and S components suggests that the deformation texture changes from brass-type toward copper-type. To gain further insights on this transformation in the deformation texture, we performed the α- and β-fiber analyses, and the corresponding results are shown in Figs. 2(a) and 2(b). In case of fcc metals, the α-fiber is an orientation tube in the Euler angle space from Goss orientation to Brass orientation, while the β-fiber basically runs from the Copper orientation, through the S orientation to the Brass orientation. Because the rolling texture was destroyed during IA at 750°C, only samples with IA temperature below 650°C were characterized. It is found from the α-fiber that the orientation intensity of Goss orientation decreases with the increase in IA temperature, while that of the Brass orientation increases. This result indicates that more Goss-oriented grains are rotated into Brass orientation at higher IA temperatures. As for the β-fiber, all the orientations along the β fiber increased with IA temperature. Accordingly, it can be concluded that the grain orientation mainly gathers from α-fiber to β-fiber with the increase in IA temperature.

Table 1. Volume fractions of rolling texture components in 99% reduced Ni-9.3at%W tapes processed at different IA temperatures

Texture component	Cold rolled	430°C	500°C	650°C	750°C
Copper	5.93	7.14	9.75	11.47	3.55
Brass	16.4	17.91	24.58	24.25	1.23
Goss	4.69	5.18	2.41	0.44	0.73
S	18.43	18.96	24.63	26.97	5.27

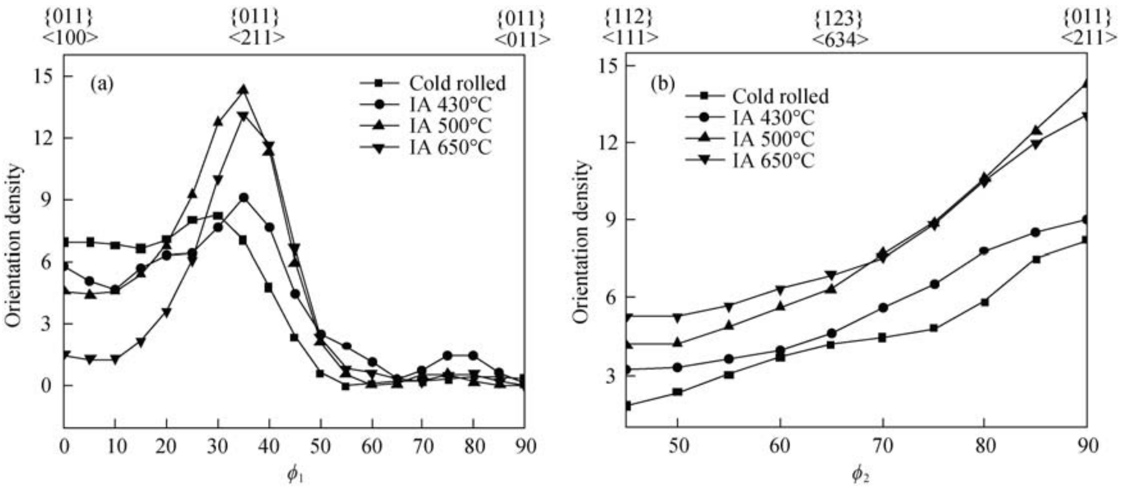

Fig. 2. The α-fiber (a) and β-fiber (b) of the 99% deformed Ni-9.3at%W substrates obtained at different IA temperatures (φ_1, ϕ, and φ_2 are the three axes of orientation space).

It is commonly believed that the transition of rolling texture from copper-type to brass-type with lowering SFE can be attributed to mechanical twinning and shearing. Hirsch *et al.* [14] proposed that the twinning in Copper-oriented grains leads to TC $\{552\}<115>$ orientation. Subsequently, the resulting TC component is rotated to Goss orientation by slipping or rotated through $\{111\}<112>$ orientation to Goss orientation by shearing. Since the Goss orientation is metastable [15], the grains with Goss orientation are finally rotated to Brass orientation. This transition mechanism is confirmed in the cold-rolled sample. As shown in Fig. 1(a), very few LBs with Copper orientation are observed, and many Copper components are adjacent with TC component by Σ3 boundary. This indicates that the Copper-oriented LBs are destroyed as a result of twinning.

When IA is carried out during the cold-rolling process, the texture of 99% reduced substrate changes from brass-type to copper-type. This transition is considered to be due to the following two reasons. On one hand, the twinning of Copper orientation is suppressed, and the flow of Copper-oriented grains into TC orientation is reduced. Thus, the TC components nearly disappear, leaving behind lots of Copper-oriented LBs in the cold rolling (Fig. 1(b)). On the other hand, the slipping of TC and Goss-oriented grains is promoted, as the deformed structure is relaxed during IA. Consequently, the TC component can be easily rotated through Goss orientation into Brass orientation, during the follow-up rolling process. Therefore, both the TC and Goss components are reduced, whereas the Brass component is increased.

3.3. Recrystallization texture

The fraction of recrystallized cube texture (within a misorientation angle of 15°) in samples with different IA temperatures after TSA is shown in Fig. 3. It can be seen that the cube component increases with the increase in IA temperature. Fig. 4 provides further insights on the recrystallized texture, obtained after the two-step annealing process. The color of orientation indicates the deviation from the ideal cube orientation. Here warmer colors indicate larger deviation. In addition, several noncube and cube twin grains could be observed in the cold-rolled sample, which correspond to the peaks beyond 15°. In contrast, these grains nearly disappear in the 650°C IA sample, and only the peaks within 15° are observed. The cube component reaches 97.2%, which is 26% higher than that in the cold-rolled sample.

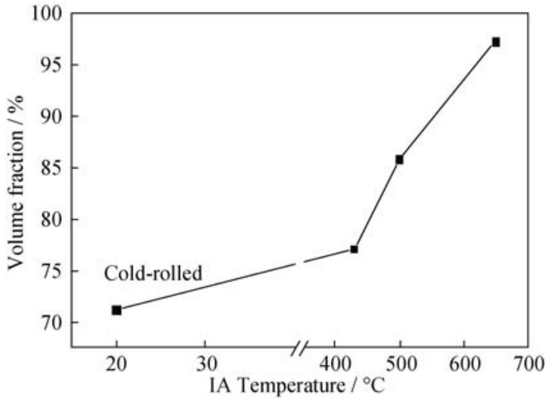

Fig. 3. Content of cube texture plotted as a function of IA temperature in recrystallized Ni-9.3at%W substrates.

Furthermore, we annealed the cold-rolled and IA 650°C samples at 700°C for different periods, in order to further gain a comprehensive understanding of the recrystallization process. Fig. 5 shows the evolution of recrystallization microstructure in the cold-rolled sample. Upon annealing at 700°C for 45 min, we could obtain a partly recrystallized microstructure with cube and noncube grains. On the other hand, in case of the sample annealed for 60 min, the deformed regions are continuously consumed by the recrystallized grains. Upon annealing for 90 min, the microstructure is recrystallized almost completely, leaving behind few deformed regions. Further annealing at 700°C leads to the coarsening of recrystallized grains. During the whole annealing process, the fraction of cube texture was below 12%. Fig. 6 shows the evolution of recrystallization microstructure in the IA 650°C sample. Upon annealing for 45 min, we could observe only a few recrystallized grains, mostly with cube orientation. Further annealing leads to the growth of recrystallized grains. However, cube texture is always the predominant recrystallization texture. After 120 min of annealing, the fraction of cube texture reaches 51.2%.

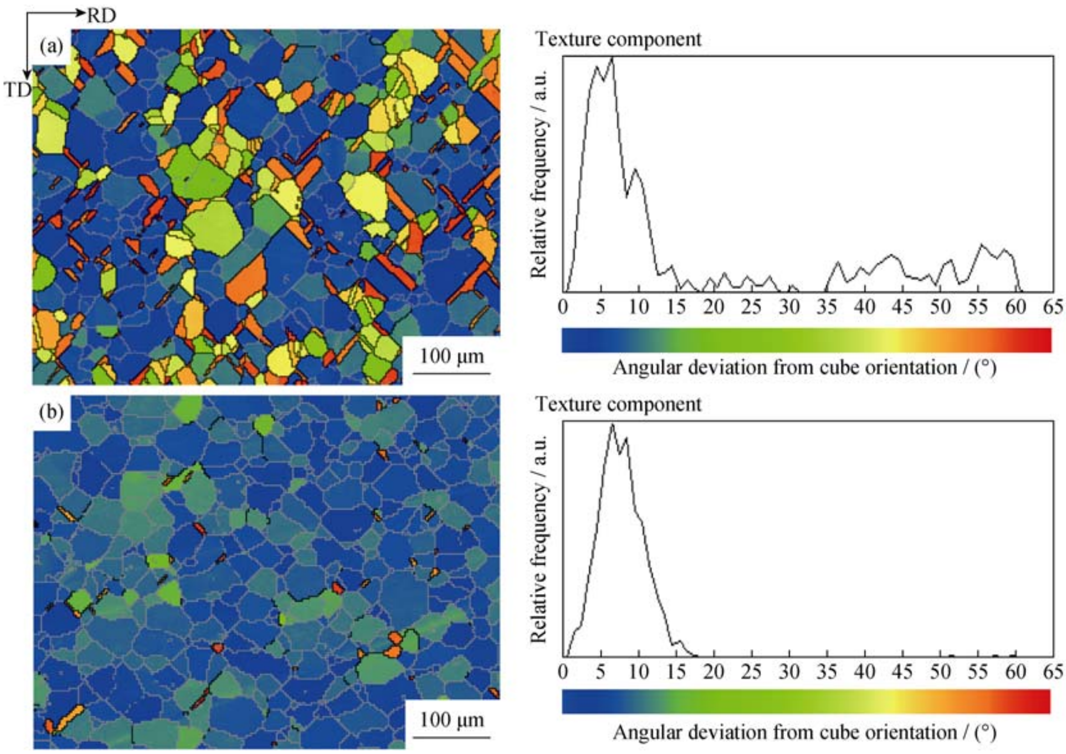

Fig. 4. EBSD maps of 99% reduced Ni-9.3at%W tapes after TSA: (a) cold rolling; (b) IA at 650°C.

Fig. 7 shows the average grain size of the cold-rolled sample and the IA 650°C sample annealed at 700°C. It could be observed that the cube-oriented grains have a size advantage during the annealing process, in case of both the cold-rolled and IA 650°C samples. However, the size advantage of the cold-rolled sample is relatively lesser, since the average size of cube-oriented grains is only about 1.2 times as large as that of the noncube grains. On the other hand, in case of the IA 650°C sample, the size advantage of cube-oriented grains is larger. The average size of cube-oriented grains is about 2 times as large as that of the noncube grains.

It is well known that the evolution of recrystallization texture depends on the competition between cube-oriented and noncube grains. It has previously been reported that the cube grains originate from the preexisting cube band in the deformed matrix [4], while noncube grains originate from the high-deformed regions, e.g., shear bands [16-17]. In case of the cold-rolled sample, the microstructure is inhomogeneous due to the twinning and shearing deformation (Fig. 1(a)). Thus, the noncube nucleation can be promoted, and the nucleation of cube-oriented grains is suppressed accordingly. When IA is performed during the cold-rolling process, the microstructure exhibits homogeneous deformation (Fig. 1(b)). Thus, the nucleation of noncube grains is suppressed. The cube-oriented grains can nucleate early and offer significant size advantage over noncube grains (Fig. 7), due to the special dislocation arrangement of the cube band [18].

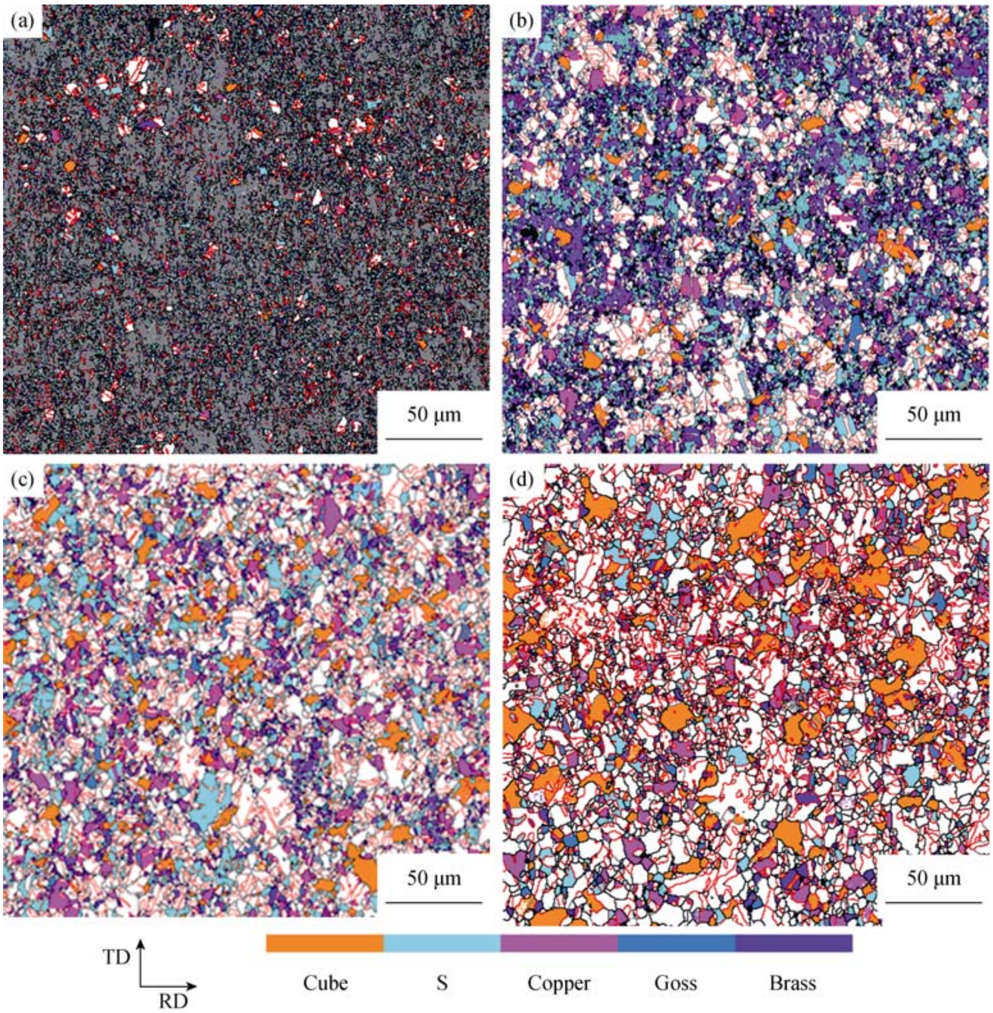

Fig. 5. Microstructure and texture evolution of the cold-rolled sample annealed at 700°C for different time: (a) 45 min; (b) 60 min; (c) 90 min; (d) 120 min.

On the other hand, some researches have shown that the cube-oriented grains grow faster than noncube grains [19], which is explained by the highest rate of boundary migration of 40°<111> orientation relationship between S and cube orientation [20]. Fig. 8 shows the axis-angle misorientation distribution between ideal cube orientation and the deformation structure shown in Fig. 1. It can be seen that the rotation axes between ideal cube orientation and deformation structure in the cold-rolled sample are mainly close to <111> axis. Additionally, there are few other axes near <001> axis due to the existence of Goss component. Thus, the growth of cube-oriented grains will be restricted, when the cube-oriented nuclei grows into the Goss component. In contrast, the Goss component in the IA 650°C sample nearly disappears. Then, almost all the rotation axes assemble near <111> axis. Moreover, the S component in rolling texture is significantly increased by IA (Table 1). The cube nuclei can thus grow rapidly by consuming the S component.

4. Conclusions

In summary, IA can result in the formation of homogenous deformed microstructure. Under the premise that deformation texture is not destroyed during IA, higher IA temperatures promote the copper-type components of deformation texture and the content of cube texture after recrystallization. The transition in the deformation texture can be interpreted as that, IA increases the dislocation slipping ability and suppresses the mechanical twinning of Copper orientation. The reason for the observed improvement in cube recrystallization texture as a result of IA is presumably attributed to the reduction in nucleation sites of noncube orientation and the optimization of preferential growth surroundings of cube-oriented nuclei.

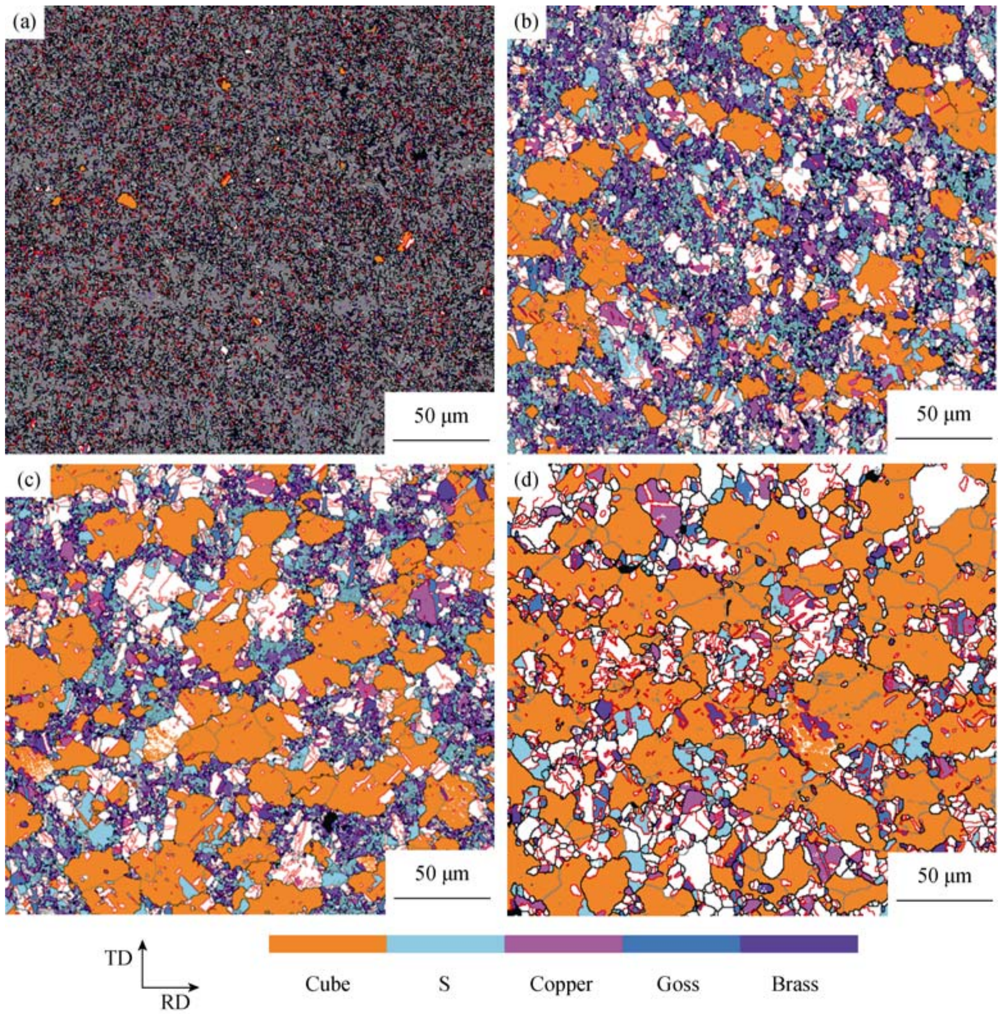

Fig. 6. Microstructure and texture evolution of the IA 650°C sample annealed at 700°C for different time: (a) 45 min; (b) 60 min; (c) 90 min; (d) 120 min.

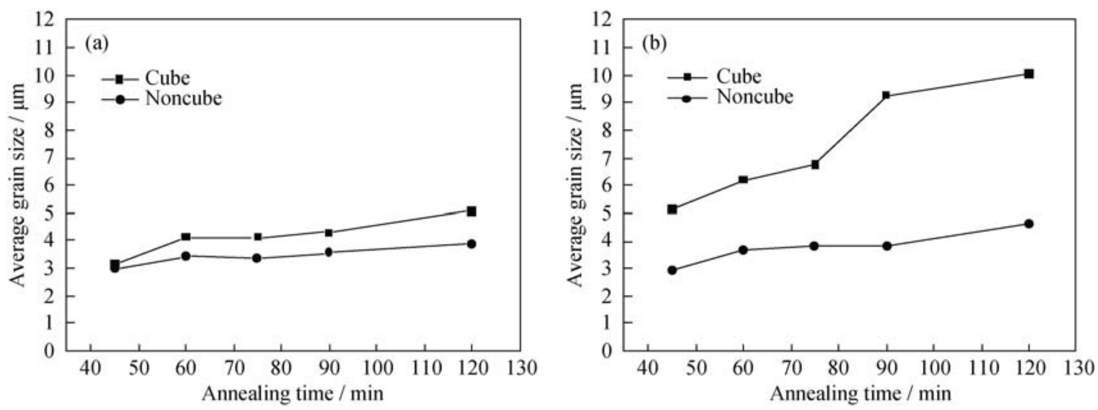

Fig. 7. Variation of average grain size as a function of annealing time: (a) cold-rolled sample; (b) IA 650°C sample.

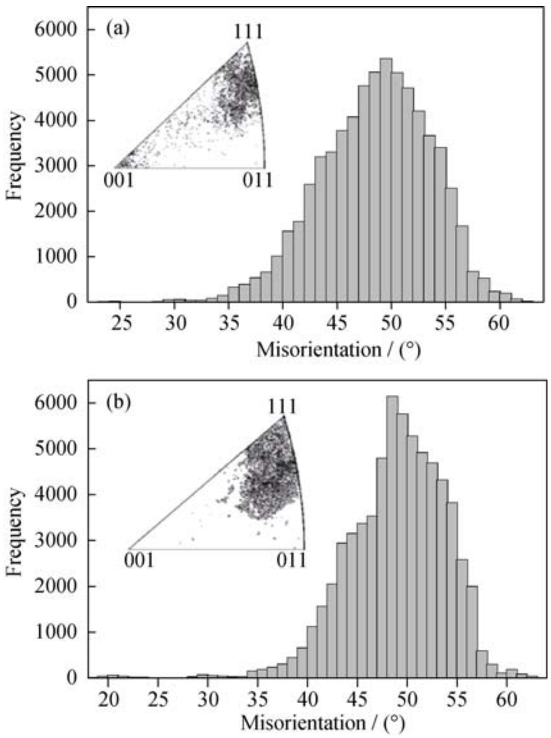

Fig. 8. Axis-angle misorientation distribution between the ideal cube orientation and deformation structure shown in Fig. 1: (a) cold-rolled sample; (b) IA 650°C sample.

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