Rare Metal Materials and Engineering
Volume 47, Issue 10, October 2018
Online English edition of the Chinese language journal

Available online at www.sciencedirect.com
ScienceDirect

Cite this article as: Rare Metal Materials and Engineering, 2018, 47(10): 2936-2941.

ARTICLE

Microstructure and Texture of Ni7W/Ni12W/Ni7W Composite Substrates at Different Intermediate Annealing Temperatures

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Abstract: Ni7W/Ni12W/Ni7W composite substrates for $YBa_2Cu_3O_{7-\delta}$ coated conductors were prepared by rolling assisted biaxially textured substrates route. A threefold intermediate annealing at different temperatures was introduced in the rolling process of Ni7W/Ni12W/Ni7W composite substrates to optimize the deformation texture. The recovery microstructure and the deformation texture of latitude and longitude section were studied with X-ray diffraction and electron backscattered diffraction to reveal the possible mechanism of temperature effects on rolling and strong cube texture formation processes. The results show that the highest cube area fraction is found in sample intermediately annealed at 600 °C/60 min; in this way Ni7W/Ni12W/Ni7W composite substrates with high cube texture content of 95% (<10°) are obtained at a low recrystallization annealing temperature. Crystal orientation maps reveal that a higher amount of cube orientation exists in its recovery and initial recrystallization samples. Point to point misorientation files confirm the high grain boundary angle of these cube grains, which strongly boost the cube texture formation during subsequent recrystallization.

Key words: high temperature superconductor; NiW substrate; intermediate annealing; deformation texture; cube orientation

Second generation high temperature superconductor $YBa_2Cu_3O_{7-\delta}$ (YBCO) is known to exhibit high critical current densities and low irreversible fields, which is interesting in view of higher operation temperatures and magnetic fields[1,2]. In YBCO coated conductors (CCs), the YBCO superconductor layer is generally deposited on textured buffer layers: this configuration allows a high fraction of low angle grain boundaries [3,4], resulting in an optimization of the critical current density. In the RABiTs (rolling assisted biaxial textured substrates) approach [5,6], the textured substrate undertakes the role of transferring texture to the superconducting layer and bearing certain stress. NiW alloys are considered as a good choice and have been studied for years. At present, the institute of IFW Dresden has realized the commercialization of textured Ni-5at%W (Ni5W) tapes. However, in spite of its good ability of cube texture formation, Ni5W is ferromagnetic at 77 K[7, 8], which leads to hysteresis losses in alternating current (AC) applications. Furthermore, its yielding strength (160 MPa) is too low to withstand the mechanical load occurring in various applications. Increasing the tungsten content of the NiW alloy is efficient in reducing the Curie temperature and simultaneously improving the mechanical strength; however, when the fraction of tungsten in NiW alloy exceeds 5%, the lowered stacking fault energy (SFE) results in work hardening during deformation and poor cube texture after recrystallization[9,10].

Composite substrates are effective in avoiding the disadvantages of both kinds of NiW alloys. In 2007, Zhao et al. proposed to fabricate tri-layered composite ingots by means of Spark Plasma Sintering (SPS)[11]. The cube texture fraction of Ni5W/Ni9W/Ni5W, Ni5W/Ni12W/Ni5W composite substrates prepared by this way reached 98.6% and 95.4% at 1250 °C/60 min, respectively[12]. As for Ni7W/Ni12W/Ni7W, the SFE of the outer layer is decreased, Zhao et al. produced a cube texture of 97.5% at 1350 °C/60 min[13], as a result the high recrystallization annealing temperature leads to thermal

Received date: October 25, 2017
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etching and weak surface quality.

This work introduced intermediate annealing to optimize the deformation texture of Ni7W/Ni12W/Ni7W, and thus it can obtain cube texture at a relatively low recrystallization temperature. What is more, the difference of samples with and without intermediate annealing is obvious[14,15], but many details remain to be further explored. In this study, the recovery microstructure and the deformation texture were studied to reveal the influence of intermediate annealing temperature and the possible mechanism of strong cube texture formation due to intermediate annealing was particularly discussed.

1 Experiment

The Ni7W/Ni12W/Ni7W composite substrates were prepared by powder metallurgy. Powders of Ni (99.9%) and W (99.8%) were mixed, with the atomic fraction of Ni and W being 93% and 88% respectively, after which they were ball-milled in a protection atmosphere (Ar/4%H_2) for 6 h to homogenize powders and also to refine the particle size. The mixed powders were synthesized in the order of Ni7W, Ni12W and Ni7W by SPS at 800 °C/5 min. The as-sintered ingot was homogenized at 1200 °C/24 h to avoid the segregation of tungsten in nickel, cold rolled from 8 mm to 80 μm with the reduction ratio of 5% per pass, and then recrystallized using two step annealing[16]: the first step was performed at 700 °C/90 min, and the second step at 1200 °C/120 min. During cold rolling, a threefold intermediate annealing at different temperatures was added when it was rolled at 2, 0.8 and 0.3 mm, respectively: for sample #1 at 550 °C/60 min, sample #2 at 575 °C/60 min and sample #3 at 600 °C/60 min. In addition, the final cold-rolled substrates (80 μm) were annealed at 700 °C/30 min to investigate the influence of different intermediate annealing temperatures on initial recrystallization.

The global texture of final rolled substrate (80 μm) was determined by means of X-ray diffraction (Bruker D8 Advance, Cu Kα)[17]. The {111}, {200} and {220} pole figures were collected and then transformed to orientation distribution functions (ODFs) for the calculation of the volume fractions of deformation orientation components. The microstructure of recovery and recrystallized samples were characterized by means of electron backscattering diffraction (EBSD) in a scanning electron microscope (QUANTA FEG 450). The obtained data were analyzed by orientation image micrograph (OIM), where low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs) were defined as boundaries with misorientations of 2°~10° and >10°, respectively. The threshold for Σ3 twin boundaries and all components were 15 deviation; for strong cube texture formation specimens it was10 deviation.

2 Results and Discussion

2.1 Recovery microstructure

For low SFE materials, heavily cold-rolled deformation results in serious work hardening and massive dislocation in grain scales, even twinning or shearing happens at higher strain rate, which causes inhomogeneous deformation. In terms of orientations, the content of Brass ({110}<112>) will rise and those desired ones like S ({123}<634>) Copper ({112}<111>) decreases. Intermediate annealing below recrystallization temperature is thought to be efficient in stress relief and thus the copper-type texture can be guaranteed. Fig.1 shows the grain boundary maps of recovery specimens after the third intermediate annealing. LAGBs and HAGBs are represented as silver and black lines and the red lines are Σ3 grain boundaries. It can be seen that after intermediate annealing, all specimens retain a structure consisting of elongated thin ribbon and high aspect ratio grains. The lamellar grains are almost parallel to the rolling direction (RD), which is typical structures of severely deformed alloys[18-20].

Intermediate annealing of rolled sample at relatively low temperatures results in recovery and structural coarsening[21]: Spacing along the normal direction (ND) increases from 149 nm to 172 nm as the annealing temperature increases (see Table 1). Correspondingly the aspect ratio of the samples turns

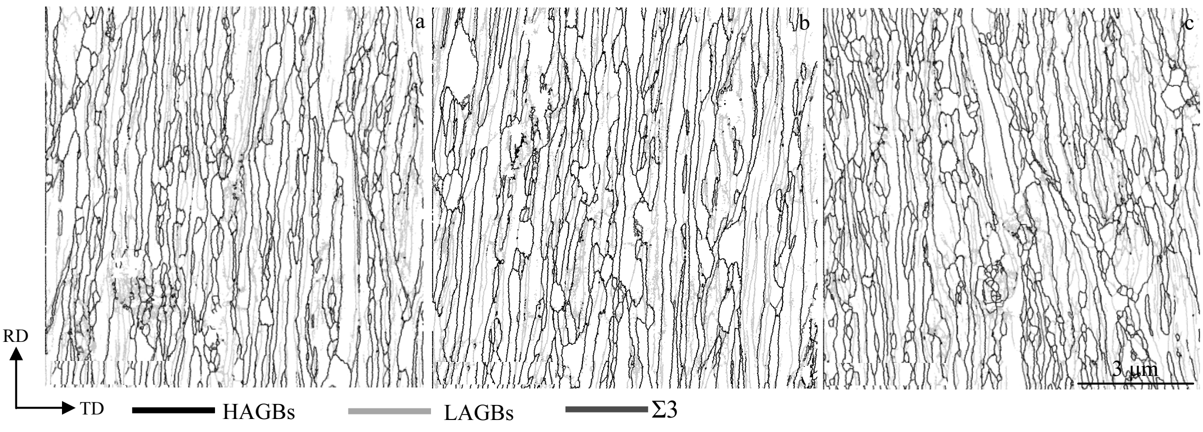

Fig.1 Grain boundary maps of the longitudinal section beneath the surface in Ni7W area: (a) sample #1, (b) sample #2, (c) sample #3 (measured by EBSD in a region of 10 μm×10 μm with a step size of 0.02 μm)

Table 1 Values of d_{ND}, and aspect ratio of recovery samples

Sample	d_{ND}/nm	Aspect ratio
#1	149	0.430
#2	158	0.425
#3	172	0.387

out to be decreased, to 0.430, 0.425 and 0.387, respectively. When structural coarsening fraction (f_{HAGBs}) decreases[22], the greatest reduction of f_{HAGBs} occurs in sample annealed at 600 °C/60 min, f_{HAGBs}= 60.1%. These results indicate that sample #3 shows the most recovery softening.

When it comes to orientation, it can be seen from the crystal orientation maps (Fig.2a~2c) that most part of the samples are occupied by deformation texture. A small quantity of Goss ({110}<001>) can be seen. The Copper is within 15.1% (see Table 2) and the Brass is higher than Copper in all three samples, which is common in low SFE materials[20]. Hirsch proposed that the transformation of Copper in low SFE materials contributes to the formation of brass-type deformation texture, Copper twin and rotate to Goss, Goss rotate to Brass at last[23]. The low fraction of Copper and relatively high fraction of Brass here may be resulted by the rotation of Copper during intensive deformation

Besides, it is significant that 0.8% of long thin cube orientation bands appear in sample #3 (blue), while it is 0.2% in sample #1 and sample #2. Previous reports ever showed that cube orientation bands greatly facilitate the cube texture formation in the subsequent recrystallized annealing[24,25]. Fig.2d~2f are the point to point misorientation values between different grains, as we know the point to point misorientation within a grain is quite low, but on the boundary it goes up apparently. The misorientation between Cube and S is 32.5°, Cube and copper being 52.2°; however it is 24.4° between Copper and S. This result corresponds well with the conjecture that cube grains have a greater growth rate, because cube orientation grains migrate faster during the subsequent recrystallization owing to the higher grain boundary angles.

2.2 Deformation texture

It is known that deformation texture has great influence on the development of recrystallization texture. Face centered cubic materials of high SFE tends to form copper-type texture after heavy rolling deformation, and it is easier for these kinds of materials to obtain strong cube texture which is a decisive factor in the manufacture of CCs[22,26]. We can see from Fig.3 that the addition of intermediate annealing greatly optimizes the deformation texture of Ni7W/Ni12W/Ni7W composite substrates. The {111} pole figure has two rather than four maxima in the central region. Volume fractions of typical deformation texture components measured by XRD are listed in Table 3. The value of (f_{Copper}+ f_S)/2f_{Brass} is used to distinguish

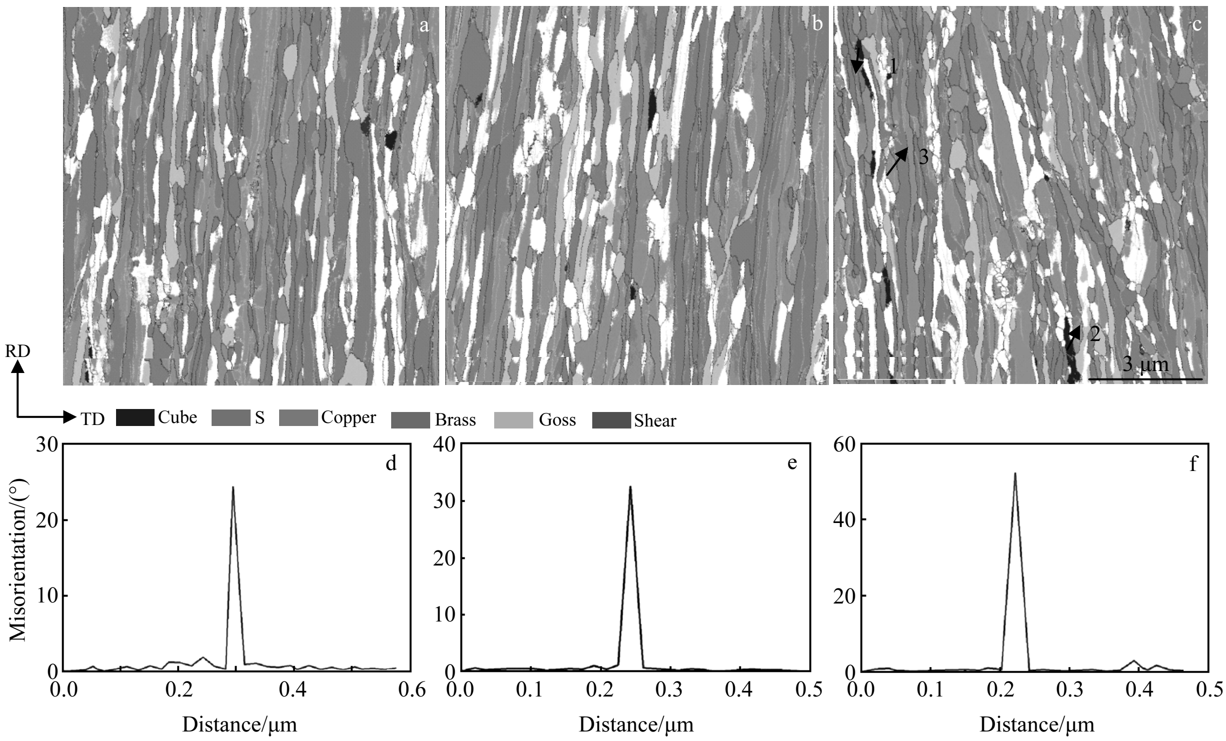

Fig.2 Crystal orientation maps of the longitudinal section beneath the surface in Ni7W area (a~c): (a) sample #1, (b) sample #2, and (c) sample #3, measured by EBSD in a region of 10 μm×10 μm with a step size of 0.02 μm; the point to point misorientation between different grains (d~f): (d) Cube and S, (e) Cube and Copper, (f) Copper and S (along the black arrow marks numbered by 1, 2 and 3 in Fig.2c)

different types of textures, and copper-type texture is considered when the numerator is higher than denominator[27]. Here for these three samples it is 1.043, 1.077 and 1.099, respectively, and S appears to be the strongest among the four deformation orientations. Therefore the deformation texture of three samples can be classified as mixed rolling texture which is between copper-type and brass-type.

Sample #2 has the strongest rolling texture component, reaching 92.99%, while the rolling texture of sample #3 is obviously lower, which is 85.72%. A possible reason may be that the intermediate annealing causes certain stress release, during which the dislocation mobility is enhanced[28]. The higher the temperature is, the more stored energy will be consumed. Consequently, the rolling texture in sample #3 is lower after the same reduction rate. In addition the cube orientation of sample #3 continues to be the highest, at 0.65 vol%.

2.3 Cube texture formation

The cube texture formed during the initial recrystallization will be further strengthened in the process of grain growth, and higher cube texture content results in greater cube grain

Table 2 Volume fractions of deformation and cube orientations of recovery samples measured by EBSD (vol%)

Sample	f_S	f_{Coppe}	f_{Brass}	f_{Goss}	f_{Cube}
#1	43.2	7.4	23.8	7.2	0.2
#2	36.3	15.1	17.9	10.0	0.2
#3	35.5	12.8	24.4	6.5	0.8

size and larger growth rate as confirmed by Y. B. Zhang[29] with Monte Carlo simulation. Fig.4 represents the {111} pole figures of the substrates annealed at 700 °C/30 min. It appears that the four sharp high intensity maxima are concentrated around the ideal cube orientation corresponding to {001} parallel to the rolling plane and <100> parallel to the rolling direction. Since the strength of sample #3 is the highest, reaching 12.067, it means that samples annealed with the intermediate temperature of 600°C/60 min have the priority of cube texture formation during initial recrystallization. This is probably because of the cube orientation grains found in recovery and deformation specimens.

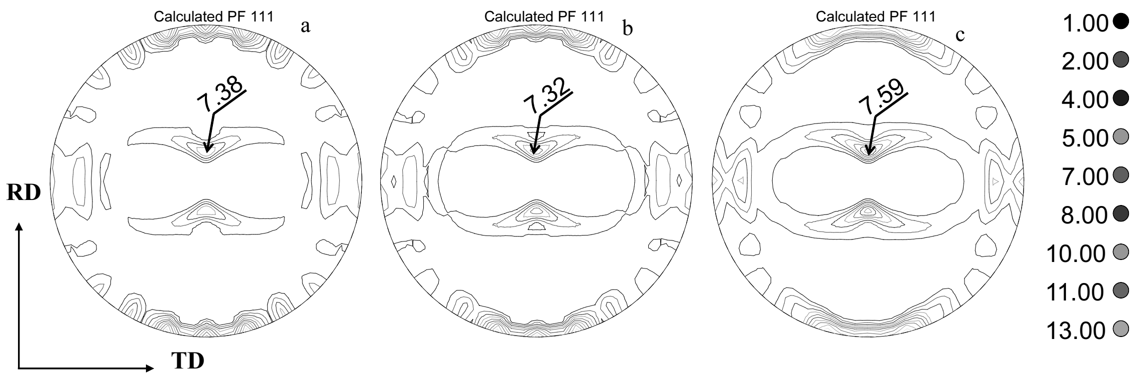

Fig.3 Pole figures of final-rolled substrates (80 μm): (a) sample #1, (b) sample #2, and (c) sample #3 measured by XRD on RD-TD section

Table 3 Volume fractions of deformation and cube orientations of final-rolled substrates (80 μm) measured by XRD

Sample	f_S/vol%	f_{Copper}/vol%	f_{Brass}/vol%	f_{Goss}/vol%	f_{Cube}/vol%	Rolling texture/%	$(f_S+f_{Copper})/2f_{Brass}$
#1	39.20	19.23	27.99	4.36	0.44	90.78	1.043
#2	40.80	20.46	28.42	3.31	0.36	92.99	1.077
#3	37.89	18.35	25.58	3.90	0.65	85.72	1.099

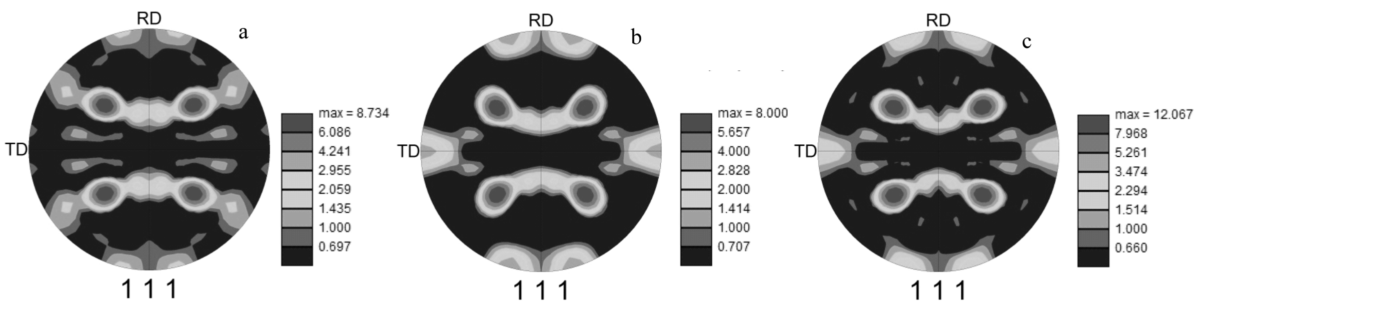

Fig.4 Pole figures of substrates annealed at 700 °C/30 min: (a) sample #1, (b) sample #2, and (c) sample #3 measured by EBSD on RD-TD section

A clear separation between the cube grain size and the average grain size can be seen in Fig.5, confirming that cube oriented grains demonstrate a size advantage compared to other grains[30]. The size of cube orientated grains in sample #3 is larger than that of other sample, which is 9.49 and 13.14 μm for sample #1 and sample #3, respectively. Also, its cube size advantage is larger too, for sample #1 the cube grain size is about 2.5 times larger than average grain size, while for sample 3#, however, the value is about 4.5 times. We can see that sample #3 has the stronger cube texture than sample #1 and #2.

Further annealing of the microstructure promotes grain growth and leads to the expansion of cube texture. Two step annealing at 700 °C/90 min and 1200 °C/120 min were launched to verify whether sample #3 is superior to others in strong cube texture formation or not. Fig.6 shows that the cube fraction of sample #1 is 91% (<10°), while that of sample #3 is higher by 4%, with the content of LAGBs and Σ3 being 77.8% and 2.5%, respectively. The performance of sample #2 is intermediate, with a highest cube fraction of 93% (<10°). This is perhaps because, for sample #3, an optimized deformation texture with higher cube orientation boosts the cube texture formation during initial recrystallization.

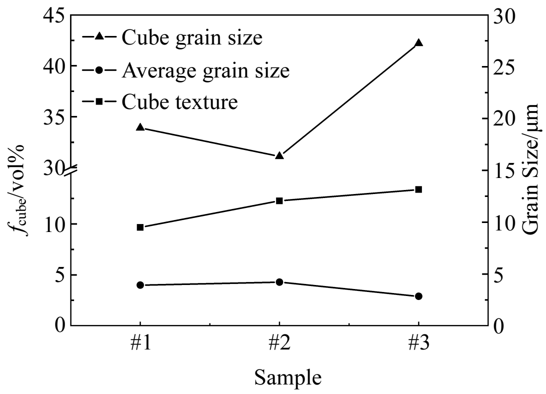

Fig.5 Grain sizes and cube texture of samples annealed at 700 °C/30 min

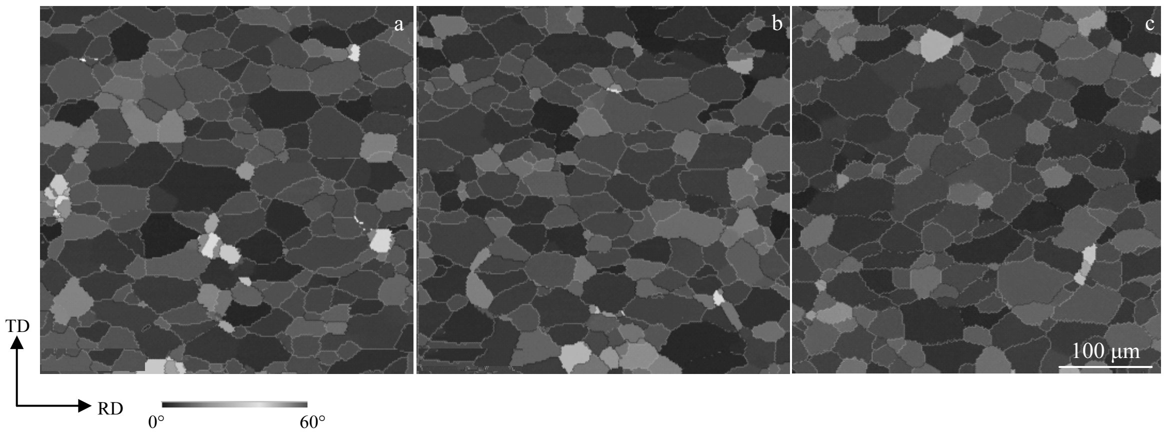

Fig.6 Crystal orientation maps of substrates annealed at 1200 °C/60 min: (a) sample #1, (b) sample #2, and (c) sample #3

3 Conclusions

1) Ni7W/Ni12W/Ni7W composite substrates optimized with intermediate annealing temperature obtain a strong cube texture at 1200 °C/120 min. The highest cube area fraction of 95% (<10°) is obtained in sample intermediately annealed at 600 °C/60min.

2) Intermediate annealing could well optimize the deformation microstructure, recovery samples present a typical morphology of lamellar microstructure, and the deformation texture of Ni7W/Ni12W/Ni7W composite substrates is mixed rolling texture between copper-type and brass-type.

3) Samples intermediately annealed at 550 °C/60 min and 575 °C/60 min exhibit a fine rolling texture; however, a larger amount of cube orientation is found in recovery and initial recrystallization specimen intermediately annealed at 600 °C/60 min, also its cube oriented grains demonstrate a larger size advantage, which strongly favors the cube texture formation during subsequent recrystallization.

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轧制中间热处理温度对 Ni7W/Ni12W/Ni7W 复合基带组织及织构的影响

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摘　要：采用压延辅助双轴织构技术制备了涂层导体用 Ni7W/Ni12W/Ni7W 复合基带。复合基带轧制形变过程加入 3 次不同温度的轧制中间热处理优化其形变组织。采用 X 射线衍射及背散射电子衍射技术分析了复合基带轧制过程中表面及截面的形变、回复组织，以此探索轧制中间热处理温度对 Ni7W/Ni12W/Ni7W 复合基带形变及立方织构形成过程的影响机制。结果显示，经 600 ℃/60 min 轧制中间热处理后的复合基带在较低的再结晶温度下获得了 95% (<10°)的高立方织构。晶体取向分布图表明该中间热处理温度下复合基带回复及初始再结晶组织中立方取向的晶粒含量都更高；不同取向间点对点的晶体取向差分析证实立方取向晶粒有较大的晶界角，这使立方取向在回复及初始再结晶过程具有高的界面迁移率，从而促进了后续再结晶过程强立方织构的形成。

关键词：高温超导材料；NiW 基带；轧制中间退火；形变组织；立方取向

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