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Cube texture formation of Ni9.3W alloy substrates: preparation by optimized deformation sequence and anneal process

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Abstract The Ni9.3W alloy with no ferromagnetism and high yield strength is one of the most promising textured substrate materials for coated conductors, but its low stacking fault energy makes it difficult to obtain a strong cube texture by traditional rolling methods and recrystallization anneals. This paper introduces four-time static recoveries during the rolling process. Rolled tapes with 80 μm in thickness were obtained by applying various deformation sequences between static recoveries to study their effects on the cube texture formation in Ni9.3W alloy substrates. The results show that rising gradient deformation sequence is an advantageous way to obtain a higher amount of cube texture, its content increases by 29.2% compared to that of traditional deformation sequence. The effect of the new recrystallization annealing process on the cube texture formation was analyzed. It is shown that the cube texture content increases with anneal temperature increasing in one-step anneal, but decreases again at higher anneal temperature. Two-step anneal could effectively improve the cube texture content, which could be further enhanced by extending holding time during the first-step anneal. However, too long holding time leads to the decrease in cube texture content. Finally, Ni9.3W alloy substrates with a cube texture content of ~90.0 vol% (<15°) are obtained by optimized two-step anneal.

Keywords Ni9.3W substrates; Deformation sequence; Anneal process; Cube texture

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1 Introduction

The second generation of coated conductor based on $YBa_2Cu_3O_{7-x}$ (YBCO) is considered to be a promising superconducting material with wide application prospects and a high potential of industrial application, owing to its high critical current density and low irreversible field [1–3]. The rolling assisted biaxially textured substrates (RABiTS) technology with low cost and high industrialization efficiency becomes a hot topic in the study of coated superconductors. In the RABiTS technology route [1, 2], textured metal substrates for coated conductors must present strong cube texture, high yield strength and low or no ferromagnetism for meeting more extensive applications. Many investigations on NiW alloy substrates fulfilling these requirements have been performed [4–7]. At present, Ni5W alloy substrates are commercially available [5], but they are still magnetic, which constitutes a limitation to a wide application. Ni9.3W alloy substrates with a higher yield strength and no ferromagnetism are today among the most promising textured substrate materials [7], but their low stacking fault energy (SFE) makes it difficult to obtain strong cube texture by traditional rolling methods and anneal processes [8]. Researchers introduced repeatedly static recoveries in the rolling process to reduce strain hardening, and the content of cube texture was effectively improved [9, 10]. However, as a result of its low SFE, the Ni9.3W alloy is easily twinned during the rolling deformation, and it has been shown that the presence of twins has a negative effect on cube texture formation [9–13]. Thus, the deformation sequence has a significant effect on the cube texture formation, which has so far rarely been reported. In addition, the optimizing of the recrystallization anneal process was also rarely reported. This paper introduced four-time static recoveries during the rolling process, and rolled tapes with 80 μm in thickness were obtained by adopting various deformation sequences for studying their effects on the cube texture formation in Ni9.3W substrates. One-step and two-step anneals were carried out to analyze the effect of recrystallization anneal process on the cube texture formation. Finally, Ni9.3W alloy substrates with high cube texture contents were produced by optimized two-step anneal.

2 Experimental

The Ni9.3W alloy casting ingot was prepared by vacuum induction melting from pure nickel (99.9%) and tungsten (99.9%). After melting, the casting ingot was hot-forged at 1200 °C to obtain a square rod of 15 mm × 20 mm. Subsequently, an ingot of 15 mm × 20 mm × 25 mm was cut from the Ni9.3W alloy rod as-obtained for subsequent experiments. The ingot was homogenized at 1200 °C for 24 h, rolled to 8 mm in thickness and submitted to recrystallization anneals at 900 °C for 1 h. Then, the ingot was subdivided into four samples with 8 mm in thickness (named as Samples 1, 2, 3 and 4, respectively). Sample 1 was submitted to the traditional deformation way: The sample was cold-rolled directly to 80 μm without any static recovery during the rolling process. Four-time static recoveries (550 °C, 2 h) were introduced in the subsequent rolling process for Samples 2, 3 and 4. The deformation sequences between static recoveries of four samples are not same. The deformation reduction mentioned in this study is relative reduction, e.g., the sample thickness before every static recovery is related to that of the last static recovery. More details of the four samples' deformation sequences are shown in Table 1.

The four samples were cold-rolled from 8 mm to 80 μm, with reduction ratios of ~5% per pass and total deformation reduction of ~99%. Then, the cube texture was formed in the four substrates at 1100 °C for 2 h. The effect of the deformation sequence on the cube texture formation was analyzed by measuring the cube texture performance to obtain optimized rolling deformation sequence. On this basis, the effect of recrystallization anneal process on the cube texture formation was taken into consideration. First, the anneal temperature was raised by a series of annealing processes (1150 °C, 2 h; 1200 °C, 2 h; 1250 °C, 2 h). Two-step anneal was then carried out to improve the cube texture content. The effect of different holding time (0.5, 1.0, 1.5 and 2.0 h) during the first-step anneal on the cube texture formation was studied, when the temperature was 700 °C. The second-step anneal was introduced at 1150 °C for 2 h. Optimized parameters of the first anneal step were obtained by measuring the cube texture performance. Then, the temperature of the second-step anneal was increased to obtain a higher cube texture content. Finally, Ni9.3W alloy substrates with high cube texture content were obtained by optimized two-step anneal.

The rolling texture of the rolled substrates was calculated using orientation distribution functions (ODFs) measured in step of 5° from 0° to 75° by X-ray diffractometer (XRD, Bruker D8 Advance, CuKα) [14]. Microstructure and texture of the annealed substrates were measured in a region of 400 μm × 400 μm with a step size of 2 μm by electron back-scattered diffractometer (EBSD, FEI QUANTA FEG650).

Table 1 Experimental details of relative reduction/%

Samples	1st	2nd	3rd	4th
1	–	–	–	–
2	50	50	50	50
3	35	45	55	65
4	65	55	45	35

3 Results and discussion

3.1 Deformation sequences

Figure 1 shows the contents of the rolling texture components in the four samples of Ni9.3W alloy substrates rolled to 80 μm. It can be easily seen that Sample 1 has the most Brass orientation content, reaching 37.18 vol% (<15°, the same below), while the contents of S orientation and copper orientation are the lowest among all samples, being 29.41 vol% and 6.18 vol%, respectively, which is disadvantageous for the cube texture formation [15]. On the contrary, the highest contents of S orientation and copper orientation are found in Sample 3, being 32.55 vol% and 9.02 vol%, respectively, while the contents of Brass

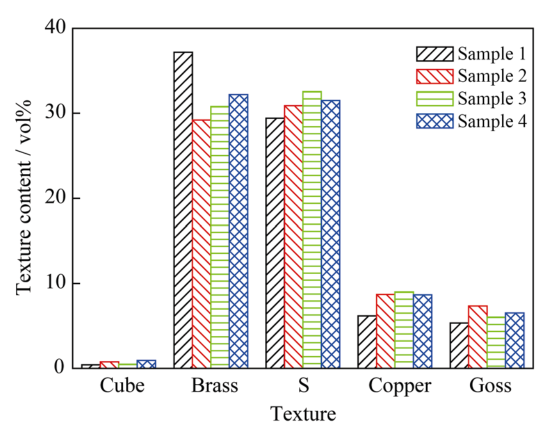

Fig. 1 Contents of main rolling textures for Ni9.3W tapes rolled to 80 μm

orientation and Goss orientation are rather low, being 30.80 vol% and 6.04 vol%, respectively, which is favorable for the cube texture formation [15, 16].

Subsequent recrystallization anneals were performed at 1100 °C for 2 h for the four kinds of substrates. EBSD maps and comprehensive properties of annealed substrates are shown in Figs. 2 and 3. It can be easily seen that the cube texture content of Sample 3 is higher with respect to other samples. In addition, the amount of low-angle grain boundaries (LAGB) and twin boundaries ($\sum^3$) of Sample 3 is superior to that of other samples. It must be noted that the cube texture content of Sample 3 increases by 29.2 vol%, 11.1 vol% and 14.1 vol%, respectively, compared to those of Samples 1 and 4. In order to confirm the result, the four samples rolled to 80 µm were annealed under higher temperature further (1150 °C, 2 h; 1200 °C, 2 h). The results show that higher cube texture content is still obtained by Sample 3 with respect to other samples. Thus, the rising gradient deformation sequence (Sample 3) is a favorable way to the formation of cube texture of Ni9.3W alloy substrates.

For Sample 2, high cube texture content is not obtained. Possibly, the reason may be that static recovery is only a

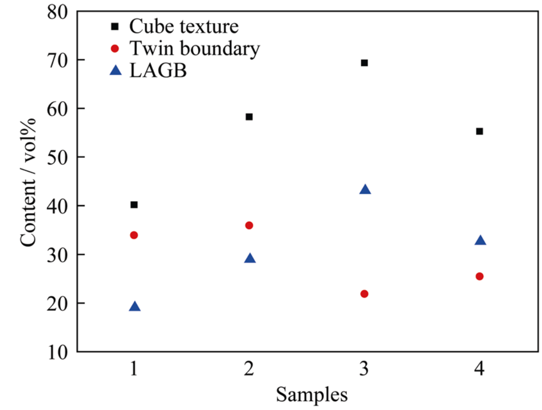

Fig. 3 Properties of Ni9.3W substrates after annealing at 1100 °C for 2 h

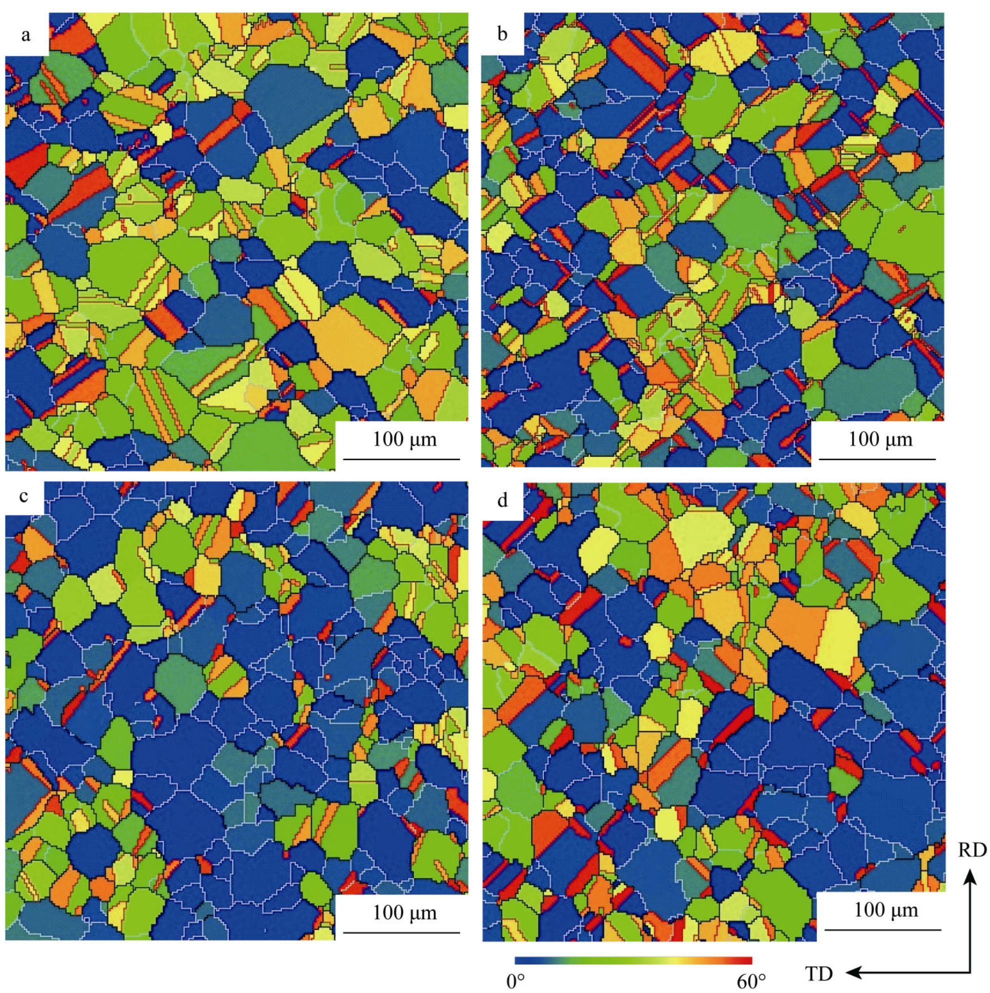

Fig. 2 EBSD maps of annealed Ni9.3W substrates (1100 °C, 2 h): **a** Sample 1, **b** Sample 2, **c** Sample 3 and **d** Sample 4 (TD, transverse direction; RD, rolling direction)

process of stress release and it is not a good choice that Sample 2 was rolled to 80 μm by applying the same deformation sequence due to too low SFE [17, 18]. For Sample 4, due to a too large reduction during the initial deformation stage, a larger stress concentration with twin deformation occurs at the early stage. It is extremely difficult to eliminate the twin of the initial stage by subsequent static recoveries, which is the reason for the higher cube texture content of Sample 3. This sample is given a small rolling deformation reduction at the initial deformation stage, while timely the static recovery eases strain hardening [17]. After subsequent static recoveries, the plastic deformation ability is further enhanced, which avoids most of the stress concentration of the material to optimize the rolling texture. Finally, Sample 3 with higher cube texture content is obtained after anneal. Thus, the time point of static recovery during the rolling process of Ni9.3W alloy is particularly important.

3.2 Optimizing of anneal process

Since Ni9.3W alloy has a lower SFE, Ni9.3W alloy substrates of a high cube texture content are not easy to be obtained after traditional anneal. From the above studies, optimized rolling deformation processing (Sample 3: the rising gradient deformation sequence) is obtained. However, higher cube texture content is not obtained after the recrystallization anneal at 1100 °C for 2 h. Therefore, to further improve the cube texture content of Ni9.3W alloy substrates, the recrystallization anneal process must be carefully studied for Sample 3. This paper describes the optimization study of the recrystallization anneal process. The cube texture content is optimized by improving the temperature of one-step recrystallization anneal process to increase the cube texture content of the Ni9.3W alloy substrates. Figure 4 shows the cube texture content of Ni9.3W alloy substrates after one-step recrystallization anneal at various temperatures. It can be easily seen that the cube texture content first increases and then decreases at higher recrystallization anneal temperature, and the cube texture content reaches its highest value of 79.3% (<15°) at 1200 °C, being optimized. Since recrystallization anneal is a process of thermal activation, it is related to the thermal activation energy outside [17, 18]. More thermal activation energy will be promoted, which could enhance the formation of the cube texture. However, too high anneal temperature leads to the decrease in the cube texture content due to the coarsening of grain size of Ni9.3W substrates under the condition.

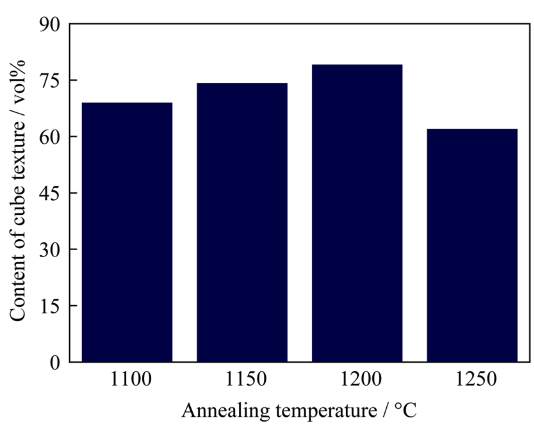

Fig. 4 Content of cube texture for Ni9.3W substrates after annealing at different temperatures

Many studies have shown that two-step anneal processes are favorable to the formation of cube texture [19]. During two-step anneal processes, the first-step anneal is very important. The temperature of the first-step anneal is 700 °C for Sample 3 [12]. The holding time of the first-step anneal is varied (0.5, 1.0, 1.5 and 2.0 h). The second-step anneal was introduced at 1150 °C for 2.0 h. Table 2 shows properties of the substrates after various holding time during the first-step anneal in the two-step anneal process. From Table 2, it can be easily seen that the content of cube texture and LAGB first increases and then decreases. On the other hand, the content of $\sum^3$ first decreases and then increases. When the holding time is 1.5 h, the content of cube texture reaches 82.6 vol% (<15°), the LAGB content is the highest (62.1 vol%), and the $\sum^3$ content is the lowest (16.7 vol%). Thus, the sharper cube texture is obtained for a holding time of 1.5 h. With the extension of holding time of the first-step anneal, the number of cube nuclei gradually increases and grows. When the holding time of the first-step anneal reaches 1.5 h, the number and size of cube nucleus is most favorable to the formation of a final cube texture, but too long holding time yields larger cube grains, which is not favorable to the formation of the cube texture during the second-step anneal. It follows that the best properties are obtained for a holding time of 1.5 h during the first-step anneal.

In order to continually improve the cube texture content of Ni9.3W alloy substrates, the temperature of the second-step anneal was further increased to obtain a higher amount of cube texture (1200, 1250 and 1300 °C). Figures 5 and 6 show EBSD maps and properties of Ni9.3W alloy

Table 2 Properties of annealed Ni9.3W substrates

Time/h	Cube texture content/vol%	$\sum^3$ content/vol%	LAGB content/vol%
0.5	74.2	25.0	44.8
1.0	81.7	21.6	54.6
1.5	82.6	16.7	62.1
2.0	72.7	25.1	47.6

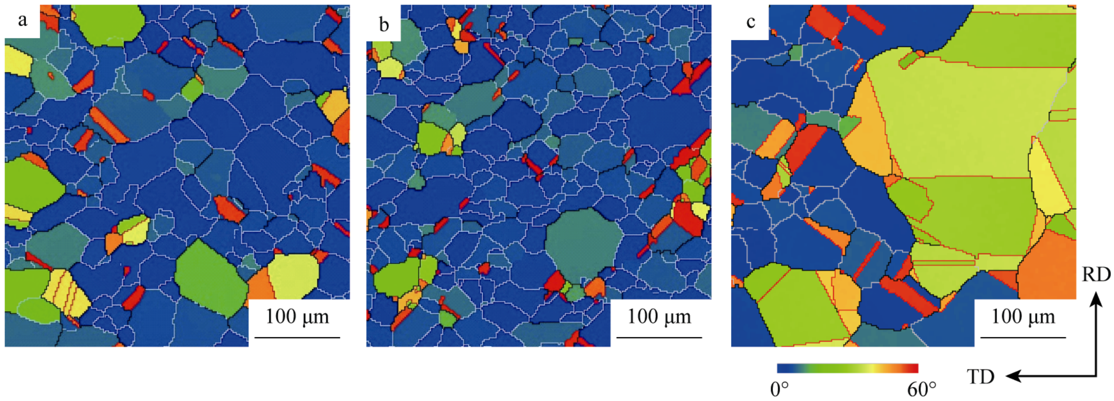

Fig. 5 EBSD maps of Ni9.3W substrates after annealing at **a** 1200 °C, **b** 1250 °C and **c** 1300 °C

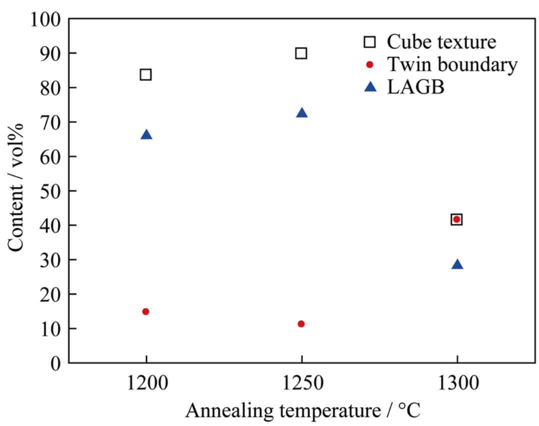

Fig. 6 Properties of Ni9.3W substrates after annealing at different temperatures

substrates after two-step anneal, respectively. It can be easily seen that the cube texture content of Ni9.3W alloy substrate reaches ~90.0 vol% (<15°) when the temperature of second-step anneal is 1250 °C, and its LAGB and $\sum^3$ contents are superior, being 72.3 vol% and 11.1 vol%, respectively. When the anneal temperature is enhanced to 1300 °C, the content of cube texture declines due to the occurrence of the secondary recrystallization. At this temperature, individual grains grow abnormally [20, 21], which could seriously inhibit the cube texture formation, yielding a low content of the cube texture of 40.4 vol%. At the same time, the performances of LAGB and $\sum^3$ contents become very poor. From the above studies, Ni9.3W alloy substrates with a high cube texture content (~90.0 vol%) are obtained by optimized two-step anneal.

4 Conclusion

For Ni9.3W alloy substrates, the rising gradient deformation sequence could effectively improve its cube texture content which increases by 29.2% compared to that of traditional deformation sequence. The cube texture content increases with anneal temperature increasing in one-step anneal, but decreases again at higher anneal temperature. Two-step anneals effectively improve the cube texture content of Ni9.3W alloy substrates, which could further be enhanced by extending the holding time during the first-step anneal. However, too long holding time would also lead to the decrease in cube texture content. Finally, Ni9.3W alloy substrates with a cube texture content of ~90.0 vol% (<15°) are obtained by optimized two-step anneal processes.

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