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Effect of different deformation and annealing procedures on non-magnetic textured $Cu_{60}Ni_{40}$ alloy substrates.

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Abstract: In this work, a series of specimens was prepared by the casting method. Sharp cube textured substrates were processed by heavy cold rolling and recrystallization annealing (RABiTS method). Both the rolling and the recrystallization texture in alloy tapes were investigated by means of X-ray diffraction and EBSD, respectively. The results showed that a strong copper-type deformation texture was obtained in the heavy cold rolled substrate. It also turns out that the recrystallization annealing process is very important to the texture transition in Cu-Ni alloy substrates. The cube texture content in $Cu_{60}Ni_{40}$ alloy substrates reached 99.7% (≤10°) by optimizing the cold rolling procedure and the recrystallizing heat treatment process, while the content of low-angle grain boundaries (from 2 to 10°) in the substrate reached 95.1%.

Key words: Non-magnetic, CuNi alloy substrates; Annealing; Cube texture; Grain boundaries

1. Introduction

The second generation of high temperature superconducting tapes based on the $Y_1Ba_2Cu_3O_{7-\sigma}$ (YBCO) compound, has a large potential for many application, e.g. electricity, transport, high magnetic field devices, military and other applications [1]. Using rolling assisted biaxially textured substrates (RABiTS) [2] is one of the primary methods for the manufacture of coated conductors. As such metallic substrates, Ni-W alloys have been widely studied and especially processing of Ni-5%W tapes [3,4] has now reached a mature level for commercialization [5]. However, the Curie temperature of this alloy is 330 K, and is ferromagnetic at 77 K, which can produce magnetic hysteresis losses [6]. Some researchers studied the magnetic and mechanical property of non/low-level magnetic alloys, e.g. Ni-Cu binary alloys [6-11] and ternary Ni-base alloys [12-14], to find appropriate solutions. The price of Cu is 1/6 of that of Ni and these two metals form a continuous solid solution. It was found that with a Cu content above 54 %, Ni-Cu tapes are non-magnetic [12]. A. Tuissi et al. [15] studied the recrystallization texture as well as mechanical and magnetic properties of Cu-Ni tapes, with 10 and 30% Ni, respectively. A. Girard *et al.* [7] studied $Cu_{55}Ni_{45}$ and $Cu_{70}Ni_{30}$ and the annealing procedures affecting grain boundary depth. It was observed that after annealing, both two alloys generate a partly twinned, strong cube texture. H.Q. Qiu *et al.* [16] produced $Cu_{60}Ni_{40}$ tapes by utilizing two annealing procedures, with a final cube texture content of 87.9%. Vannozzi *et al.* [13] manufactured Ni-Cu-Co ternary alloy tapes where the cube texture content after annealing was 95%. F. Fan et al. [17] found that in $Cu_{60}Ni_{40}$ tapes a sharp (001) texture could be obtained after annealing 1 h at 650-1000 ℃.

However, there are only a few reports systematically analyzing sub-orientation and grain characterization of Cu-Ni alloy tapes. This paper reports on non-magnetic $Cu_{60}Ni_{40}$ tapes produced by the RABiTS method, and studies both recrystallization texture and grain sub-orientation affected by cold rolling and annealing procedures in many details. Several microscopic techniques are applied to observe the differences between cold rolling and annealing.

2. Experimental

A $Cu_{60}Ni_{40}$ ingot was prepared by vacuum induction melting of 99.9% pure metals. The ingot was hot forged into a rectangular shape with dimensions 20×15×10 mm^3. After removing the oxide surface, the material was cold rolled, applying less than 5% reduction per pass until the final tape thickness (more than 95% total reduction). The samples were annealed in a protective atmosphere of 4% H_2 in Ar, following one-step or two-step annealing procedures.

The texture of the cold rolled substrates was investigated with a Bruker D8 X-ray goniometer. The microstructure, e.g. grain orientation distribution and grain boundary features of the recrystallized substrates was investigated with a SEM equipped with an Electron Back Scattering Diffraction setup (EBSD, JEOL JSM6500F), covering a region of 600 μm×400 μm, with 4 μm steps and an operating distance of 20 mm, while the grain sizes were defined by 2° misorientation.

3. Results and discussion

3.1 $Cu_{60}Ni_{40}$ tape deformation texture affected by the rolling procedure

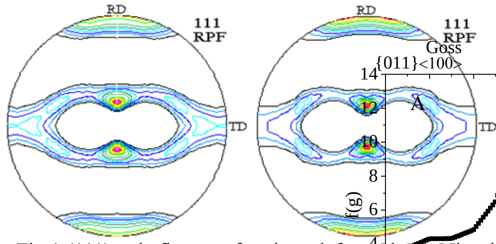

Fig.1 (111) pole figures of various deformed $Cu_{60}Ni_{40}$ alloy tapes:
a)95%; b)99%

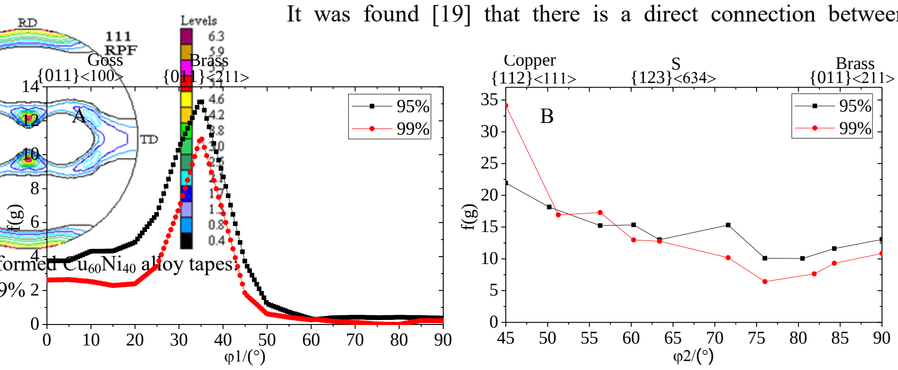

Fig.2 a) α-fiber and b) β-fiber for various deformed $Cu_{60}Ni_{40}$ substrates

It was found [19] that there is a direct connection between deformation texture and recrystallization texture in face centered cubic (fcc) metals, where a strong Cube texture is obtained after annealing a Copper-type rolling texture, whereas mixed recrystallization is obtained after annealing of a mixed rolling texture, while a Cube texture couldn't be obtained after annealing Brass-type rolling texture. It is apparent that Copper and S orientations affect the generation of cube recrystallization while the rolling procedure affects the deformation texture. A Copper-type deformation texture can be obtained from high stacking fault energy fcc metals after strong deformation [18]. Fig. 1 shows (111) pole figures of highly deformed $Cu_{60}Ni_{40}$ alloy tapes (a. 95%, b. 99%). It can be seen that the two deformed alloys have typical Copper-type rolling textures, and that with increasing deformation S and Copper orientations became more concentrated around their ideal orientation. For the effect of the degree of deformation on characterizing the rolling texture, we studied the deformed $Cu_{60}Ni_{40}$ tapes α-fiber and β-fiber (see Fig. 2). It can be seen that with increasing deformation, the amount of Goss and Brass orientation decreased slightly, whereas the Copper and S orientations became more concentrated around their ideal orientation. This shows that 99% deformation leads to a stronger Copper-type rolling texture, agreeing with Fig.1, and a stronger Cube recrystallization can be expected.

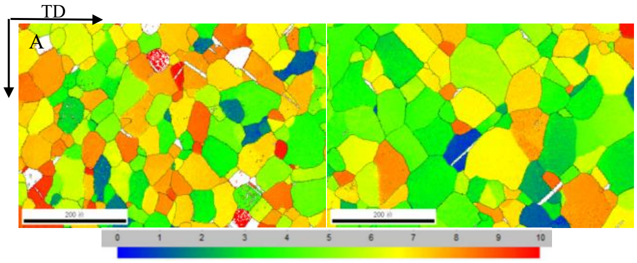

Fig. 3 EBSD maps of $Cu_{60}Ni_{40}$ alloy substrates annealed at various temperatures for 30 min: a) 1000℃; b) 1050℃

3.2 The effect of the annealing procedure on $Cu_{60}Ni_{40}$ recrystallization texture

After one-step annealing, the 99% deformed tape (5% deformation per pass) was investigated by the means of EBSD technique for sub-orientation in different recrystallized samples. Figure 3 shows the EBSD maps of $Cu_{60}Ni_{40}$ substrates after annealing during 30 min at 1000 °C (sample A) or 1050 °C (sample B). The Cube orientation contents while the amount of low-angle grain boundary (2-10°) is, respectively, 96.1%, 98%, 75%, and 86%. After annealing, twins were generated in both samples. These alloys have high Cube contents, however, their proportions of low-angle grain boundaries are slightly low. The reason is that after annealing, the Cube orientation (≤10°) deviated from the ideal cube texture, thus resulting in a misorientation between two adjacent cube oriented grains, and in the generation of some twins (see Fig. 3). It was reported [19,20] that annealing twins and large-angle grain boundaries strongly affect the superconducting critical current density (J_c). In order to obtain a higher J_c, the non-cube oriented grains and annealing twins must be avoided, thus increasing the content of low-angle grain boundaries and Cube orientation. The content of twin boundaries decreased because of the increasing of annealing temperature (see Fig. 3 which shows sample B has the lower twin boundaries than sample A). However, there are several huge grains in sample B, in agreement with Ref. 21, which states that increasing temperature or longer annealing time will lead to abnormal grain growth and secondary recrystallization, at the expense of the generation of Cube oriented grains.

For eliminating annealing twin orientations and improving the quality of $Cu_{60}Ni_{40}$ alloy tapes, we annealed them by a two-step

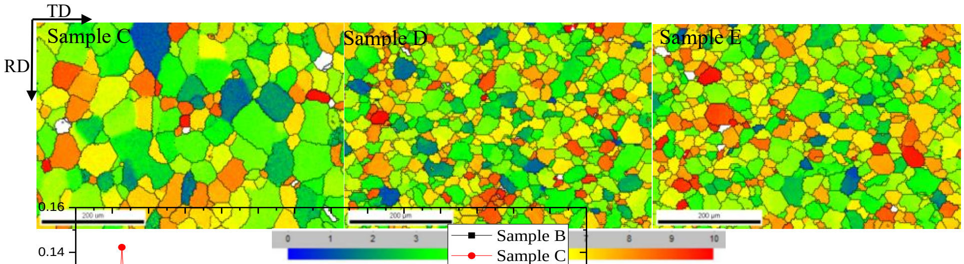

Fig. 4 EBSD maps of $Cu_{60}Ni_{40}$ alloy after two step recrystallisation

Table 1 Two-step annealing processes with various parameters

Samples	First step	Second step
C	550°C, 30min	950°C, 30min
D	550°C, 30min	1000°C, 30min
E	550°C, 30min	1050°C, 30min

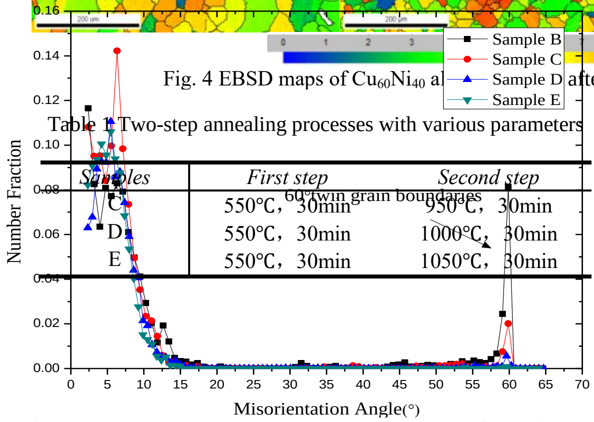

Fig. 5 Misorientation distributions in $Cu_{60}Ni_{40}$ tapes after various annealing processes

method. V. Subramanya Sarma [21] stated that two-step anneals can yield a higher Cube orientation content and better grain boundaries than one-step anneals in Ni-W/Ni-Cr complex alloy substrates. The two-step annealing procedure means that the alloy tapes are first annealed at a relatively low temperature for a given time, followed by annealing at a higher temperature anneal. The first annealing step contributes to the nucleation of Cube oriented grains . During the 2nd step, the Cube oriented grains grow at the expense of the adjacent non-cube oriented grains, thus producing a higher content of cube recrystallization and lower deviation (≤10°) from the ideal cube texture in the substrate. The parameters of the first annealing step are very important for the final result. After 50% of the recrystallization process, according to Gerber *et al.* [22], the cube oriented grains have apparently the advantage among all recrystallization grains. Since H.Q. Qiu [16] stated that recrystallization of $Cu_{60}Ni_{40}$ alloy tape reaches 50% after annealing for 30 min at 550 °C, we used these parameters for the first annealing step.

Table 1 shows three kinds of two-step annealing parameters. In these processes, the first step was always 550 °C/30 min., while the second annealing step was performed at 950 °C/ 30 min (sample C), 1000 °C/ 30 min (Sample D), or 1050 °C/ 30 min (sample E).

Figure 4 shows the sub-orientation maps of $Cu_{60}Ni_{40}$ alloy tapes after these different annealing procedures. The amount of cube orientation recrystallization texture was 98.7%, 99.4%, and 99.7%, respectively, i.e. the same level as commercial Ni-W alloy substrates. In particular, the proportion of low-angle grain boundaries (2-10°) in sample E was 95.1%, which is significantly higher than for samples C (88.6%) and D (92.0%). Figure 5 shows the misorientation distributions of $Cu_{60}Ni_{40}$ alloy tapes after different annealing steps. The lowest content of twins (60° misorientation) was found in sample E. These $Cu_{60}Ni_{40}$ cube textures after one-step or two-step annealing were compared in Fig. 6. It can be seen that most grains in sample E were concentrated around ±4.1° deviation from the ideal cube orientation, agreeing with the highest content of low-angle grain boundaries (see Fig. 5), which is also expected to contribute to generating high quality superconductors.

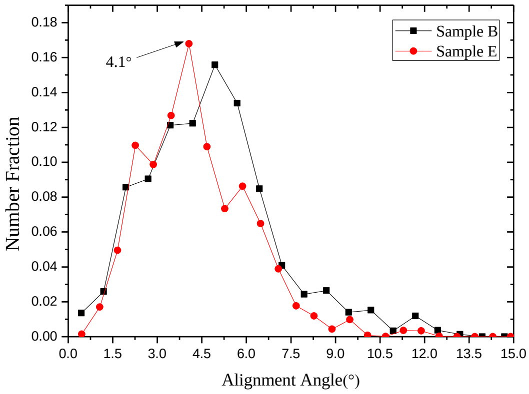

Fig. 6 Curve of cube texture as a fuction of deviation angle of $Cu_{60}Ni_{40}$ substrates with various annealing processes

4. Conclusion

(1) A strong Copper-type rolling texture, which contributes to generating cube recrystallization texture, was obtained by cold rolling (5% deformation per pass, 99% total deformation);

(2) The two-step annealing process could improve the $Cu_{60}Ni_{40}$ cube recrystallization texture. Non-magnetic $Cu_{60}Ni_{40}$ alloy tapes in which the amount of cube texture (≤10°) was 99.7% and the proportion of low-angle grain boundaries (2-10°) reaches 95.1% were produced by annealing first at 550℃ for 30 min and then at 1050℃ for 30 min, thus reaching the same level as commercial Ni-W alloy substrates. Such high quality $Cu_{60}Ni_{40}$ alloy tapes may be used for the following deposition of buffer and superconductor layers.

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