Stable cube texture in an advanced Ni alloy composite substrate prepared by powder metallurgic method

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Abstract

Thin, reinforced and biaxially textured Ni5W/Ni12W/Ni5W composite substrate for coated conductor applications has been fabricated by traditional powder metallurgy method using the sparking plasma sintering (SPS) technology, followed by cold rolling and annealing. In-situ EBSD strain-stress analysis shows that the yield stress (δ0.2) can reach 240MPa. The high quality of cubic texture and boundaries of low misorientation angle were stable until elongations as high as 2%. Meanwhile, the cubic grain fractions on surfaces of top Ni5W layer of the composite tapes are 98.3%, 99.5% and 99.8%, respectively, corresponding to be annealed at 1250°C for 60min, 120min and 180min, indicating the cube texture can successfully sustain after severe treatment condition.

1. Introduction

In recent years, the second generation of coated conductors (CCs) based on both the YBa2Cu3O7−x (Y123) and GdBa2Cu3O7−x (Gd123) superconductors have been paid broad attentions all over the world due to their potential advantages of high irreversibility field and cost-effective production [1-3]. One of the essential requirements for the Rolling-Assisted Biaxially Textured Substrate employed in CCs is to develop a sharp cube texture in materials having high strength and low magnetism. Among various metallic combinations, Ni-W alloys have been systematically investigated and widely used as a substrate material for coated conductors during the past decade [4-7]. Until now, although hundred meter lengths of Ni-5at.%W tape with cube texture and high surface quality can be produced by several groups or companies [8-11], the mechanical and magnetic behaviors of the substrates can still

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be improved by increasing the tungsten content in the NiW alloy. The sharp cube recrystallization texture could not be easily formed in high tungsten NiW alloys by means of cold rolling and recrystallization annealing, due to the reduction of Stacking Fault Energy (SFE) originating from the increased strength and possibly from the presence of short-range order in these alloys [12, 13]. This problem has been solved by the design of so-called composite substrates [14-15]. The composite substrate has a sandwich architecture, where the outer layers are based on low W Nickel-Tungsten alloy facilitating the formation of a sharp cubic texture, the high W Nickel alloy at the core layer ensuring both mechanical reinforcement and lower magnetization of the whole substrate.

Previously, we have successfully fabricated the new composite substrates with a sharp cubic texture and reduced saturation magnetization from a multilayer ingot which was synthesized by the sparking plasma sintering (SPS) technique and powder metallurgy (PM). The techniques for fabricating a series of composite tapes with both sharp cubic texture and enhanced strength like Ni5W/Ni9W/Ni5W and Ni5W/Ni12W/Ni5W have been reported [16-18]. Practically, the substrates are submitted to a certain strain during further coated conductor processing: growth of a buffer layer and the YBCO layer on its surface in a reel-to-reel process, under temperatures ranging from 800°C to 1100°C. It is thus necessary to investigate the stability of the micro-texture of the outer Ni5W layer in the composite tape under tensile stress and at high temperature to avoid the degradation of the cubic texture, which may subsequently affect the quality of the epitaxial layers. In this paper, a Ni5W/Ni12W/Ni5W composite tape has been prepared using SPS technique followed by traditional cold rolling and recrystallization. The stability of cube texture and grain boundary evolutions of the composite tapes under tensile processing and high temperature annealing are discussed.

2. Experimental Details

2.1. Preparation of Composite Substrate
The SPS powder metallurgy route was used to produce the Ni5W/Ni5W/Ni5W starting ingots. The Ni powders (99.9%) and W powders (99.8%) were mixed by ball milling (FRITSCH P6) in Argon atmosphere with alloy compositions of Ni–5at.%W(Ni5W) and Ni–12at.%W (Ni12W), respectively. The milling treatment is preformed at 150rpm for 5h at room temperature. The mixed powders were first packed into a graphite mould in the sequence of Ni5W-Ni12W-Ni5W and then cold pressed under an axial pressure of 15MPa. The SPS technique under a pressure of 30MPa was employed to synthesize the starting composite ingot at a temperature of 800°C. The sintered composite ingot was then cold rolled to 80µm thickness, with reduction ratios of below 5% per pass, the total reduction being larger than 99%. The rolled composite metallic tapes were heat treated by a two-step annealing process, holding the sample at a lower temperature (about the recrystallization temperature) for half an hour before reaching the annealing temperature of 1250°C dwelling for different time in a flowing Ar-4%H2 atmosphere in order to get the desired orientation at the top surface of the composite substrate.

2.2. Texture Analysis
The micro-texture and misorientation distribution information were acquired on the composite tapes from collecting data by means of electron backscattering diffraction (EBSD) (on a 600×400µm²
The cubic texture component was evaluated using an orientation imaging microscopy (OIM) software. For an in-situ EBSD characteristic, the microstructure analysis with in-situ tension was performed on the surface of the composite substrate using scanning electron microscopy (SEM: JEOL JSM 6500F) comprising EBSD analysis and an in-situ tensile system. The sample size was 15mm×3mm×75µm, which was annealed at 1250°C for 60min taking the two-step annealing process. The data were collected at various in-situ tensile stages. For texture stability analysis at high temperature conditions, the as-obtained composite tapes were recrystallized at 1250°C for different dwell time, i.e., 60min, 120min and 180min, respectively. The as-annealed specimens were then investigated by means of EBSD analysis system to evaluate the texture stability of the top Ni5W layer of the composite substrates under the severe heat treatment conditions.

3. Results and Discussion

3.1 In-situ EBSD analysis of the micro-texture of a Ni5W surface layer of the composite tape during tensile tests

It was reported [19] that the substrate does not withstand strains above 0.5% in compression and 0.2% in tension, and the stress at low strain (e.g. 0.2% yield strength) is more critical for the substrate material in many applications. In this work, we have investigated the in-situ micro-texture evaluation of the Ni5W layer in the composite tape under tensile conditions.

![Figure 1](image-url)

**Figure 1.** The stress-strain curve of the Ni5W/Ni12W/Ni5W composite tape measured by tensile tester coupled with in-situ EBSD analysis.

The stress-strain data of the Ni5W/Ni12W/Ni5W composite tape are plotted in Figure 1. The sample was fixed in the tensile tester that was attached to the EBSD system, giving the possibility to analyze the evolution of the surface misorientation of the outer Ni5W layer in-situ during processing under tensile conditions. The sample was pulled along the rolling direction at a speed of 0.4mm/min. Meanwhile the EBSD data were recorded at several strain stages (the original stage, the elongation percentages corresponding to 0.5% and 2% stages) in order to evaluate the micro-texture of the outer Ni5W layer in the composite substrate under tensile processing conditions. From the strain-stress curve, it was found that the yield strength ($\delta_{0.2}$) of the composite substrate exceeds 240MPa. This value is satisfied to the required value of 200~250MPa for practical applications of coated tapes.
predicted by Goyal et al. [20].

As shown in Figure 2, the percentage of the cube grains is almost unchanged after elongation, and the amount of cube texture within the misorientation angle of 10° are 99.8%, 99% and 98.7% at the original stage, at 0.5% and 2% elongations, respectively. These results indicate that the sharp cube texture on the Ni5W layer surface of the as-obtained composite substrate has been obtained. The good results can also be proved by the (111) pole figures related to various tensile stages, i.e., 0%, 0.5% and 2%, respectively, shown as in Figure 2. It is observed that the micro-texture can sustain a certain strain. Especially, when the elongation percentage reaches 0.5%, which corresponds to the application limit of the coated tapes, the cube texture is still stable. Furthermore, the sharp cube texture is still stable on the Ni5W outer layer when the elongation rate is as high as 2%, even being markedly higher than that of the practical application condition for coated conductors. This result shows that Ni5W/Ni12W/Ni5W composite substrates, having a stable and high quality of cube texture, can be used for epitaxial growth of the buffer layer and YBCO film using a Reel-to-Reel technology, where a tension is applied to the substrate during processing.

**Figure 2.** EBSD images (upper pictures) and (111) pole figures of the surface of the Ni5W layer submitted to various tensile stresses.
Since the critical current density ($J_c$ of the YBCO conductor layer) is strongly dependent on the grain boundary angle, it is also necessary to characterize the grain boundaries of the composite tapes at different strain stages. Figure 3 shows the grain boundary distribution maps of the outer Ni5W layers for various elongations. The length of grain boundaries with low misorientation angles (<10°) reaches 87.2%, 88.3% and 88.4% respectively at different elongation stages, indicating that the grain boundaries are very stable. Furthermore, it was seen that there is no obvious increasing with the twin boundary among the three tension stages. This result is also in good agreement with the grain boundary stability in the tensile processing, which is supposed to be favorable for the epitaxial deposition of further buffer layers and YBCO films on this composite substrate.

3.2 Stability of the cube texture for the composite tapes at high temperature condition

![Figure 3. Grain boundary misorientation distribution of the top Ni5W layer at various elongation stages](image)

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3.2 Stability of the cube texture for the composite tapes at high temperature condition

![Figure 4. EBSD maps of top Ni5W layers of composite tapes annealed at 1250°C for 60min (a); 120min (b); 180min (c)](image)

In order to investigate the texture stability of the composite tape under a practical use such as
epitaxial deposition of both buffers and YBCO layers, the as-obtained composite tapes have been studied at high temperature up to 1250°C for different dwell time, which is higher than the practical temperature for superconducting tape processing.

Figure 4 shows the EBSD maps of the composite tapes, which has been submitted to a high temperature treatment at 1250°C for 60min, 120min and 180min, respectively. It was found that the cubic grain proportions with a misorientation angle of 10° are as high as 98.3%, 99.5% and 99.8%, respectively, indicating that the sharp cubic texture is still maintained at the Ni5W surface layer of the as-produced composite tape after carrying out an severe annealing. Especially, in spite of the longer annealing time, the abnormal growth of grains was not observed in the specimen with annealing at 1250°C for 180min, which is possibly due to the diffusion of the W towards the outer layer to suppress the abnormal growth tendency [18]. Consequently, the sharp cube texture at the Ni5W surface of the composite tape was successfully retained after submitting the tape to a tensile test and processing it again at high temperature, demonstrating the high stability of the composite substrate under a practical stress and high temperature condition.

Table 1. Boundary length fraction for different misorientation angle ranges of Ni5W layer with various annealing processes

|                | 2°~4° | 4°~6° | 6°~8° | 8°~10° | >10° |
|----------------|-------|-------|-------|--------|------|
| 1250°C for 60min | 0.158 | 0.313 | 0.221 | 0.159 | 0.118 |
| 1250°C for 120min | 0.195 | 0.364 | 0.244 | 0.117 | 0.08  |
| 1250°C for 180min | 0.227 | 0.335 | 0.189 | 0.13  | 0.119 |

Table 1 shows the length fractions for the misorientation angle ranges from 2° to 4°, 4° to 6°, 6° to 8°, 8° to 10°, >10°, respectively for three annealing processes. It was seen that the highest fraction value of low misorientation angle boundary appears at the process of 1250°C for 120min. At the same time, there is no obvious difference observed between length fractions for the three annealing processes. This result demonstrates that the grain boundary is stable during the high temperature annealing process until at 1250°C for 180min, which is supposed to be favorable for the epitaxial deposition of further buffer layers and YBCO films on this composite substrate.

4. Conclusions

A Ni5W/Ni12W/Ni5W composite tape with the required properties for the functional use as a substrate for coated conductor tapes has been produced. The results show that the top Ni5W layer in the composite tape presents a sharp and pure (001) <100> cube texture, i.e. the most appropriate texture for high quality YBCO coated conductor tapes. The EBSD analysis performed in-situ on both micro-texture and grain boundaries of the Ni5W layer during tensile test of the composite tape shows that the volume fraction of the cubic grains and the relative length of grain boundaries with low misorientation angles are as high as 98% and 87%, respectively, for elongations of 2%, thus indicating that the cube texture and grain boundaries have a good stability under tensile conditions. Moreover, when applying an additional annealing treatment at 1250°C for 180min, the Ni5W surface layer of the composite tape still maintains a sharp cube texture. These results are significant and
demonstrate the stability of cube texture in the composite substrate under practical stress and high temperature conditions. It is strongly believed that this composite tape is a promising and competitive candidate as a low-cost substrate with high and stable quality of cube texture and mechanical properties for large-scale coated conductor production in the near future.

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**Reference**

[1] Holesinger TG, Civale L, Maiorov B, et al 2008 *Advanced Materials* **20**:391.
[2] Teranishi R, Izumi T, Shiohara Y 2006 *Superconductor Science and technology* **19**: S4.
[3] Obradors X, Puig T, Pomar A, et al 2006 *Superconductor Science and technology* **19**: S13.
[4] Goyal A, Norton DP, Kroeger DM, Christen DK, Paranthaman M, Specht ED, et al 1997 *J Mater Res* **12**: 2924-40.
[5] Matsumoto K, Kim SB, Wen J G, Hirabayashi I, Watanabe T, Uno N, et al 1999 *IEEE Trans Appl Supercond* **9**(2): 1539–42.
[6] Cantoni C, Christen DK, Feenstra R, Goyal A, Ownby GW, Zehner DM 2000 *Appl Phys Lett* **79**:3077–79.
[7] Goyal A, et al 2002 *Physica C* **382**:251.
[8] Eickemeyer J, Selbmann D, Opitz R, De Boer B, Holzapfel B, Schultz L, et al 2001 *Supercond Sci Technol* **14**:152–9.
[9] Zhang W, et al 2007 *Physica C* **505**:463–65.
[10] http://www.evico.de
[11] http://www.kisware.gr
[12] Subramanya Sarma V, Eickemeyer J, Schultz L and Holzapfel B 2004 *Scr. Mater.* **50**:953.
[13] Subramanya Sarma V, Eickemeyer J, Mickel C, Schultz L, Holzapfel B 2004 *Material Science and Engineering A* **380**:30-33
[14] Goyal A 2001 *US Patent* No.6 180, 570,
[15] Goyal A 2002 *US Patent* No.6, 375, 768,
[16] Suo H L, Zhao Y, Liu M, He D, Zhang Y X, Zhou M L 2007 *IEEE Transactions on Applied Superconductivity* **17**(2): 3420-23
[17] Zhao Y, Suo H L, Liu M, He D, Zhang Y X, Ma L and Zhou M L 2007 *Acta Materialia* **55**:2609-14
[18] Suo H L, Zhao Y, Liu M, Zhang Y X, He D, Ma L and Zhou M L 2008 *Acta Materialia* **56**:23-30
[19] Goyal A, Norton D P, Kroeger D M, Christen D K, Paranthaman M, Specht E D 1997 *J Mater Res* **12**:2924
[20] Goyal A, Paranthaman M, Schoop U 2004 *MRS* **29**:552–561.