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# Fabrication of a Textured Non-Magnetic Ni-12at.%V Alloy Substrate for Coated Conductors

M. M. Gao, J.-C. Grivel, H. L. Suo, Y. Zhao, D. He, and N. H. Andersen

Abstract—Ni-12at.%V alloy is a promising candidate for non-magnetic cube textured metallic substrates used for high temperature coated conductors. In this work, a textured Ni-12at.%V substrate has been fabricated by powder metallurgy route. After cold rolling and recrystallization annealing, a cube texture content as high as 99% ( $< 10^{\circ}$ ) was obtained, and the low angle grain boundary fraction is 89%. In addition, the FWHM values of phi-scan and rocking curve are 7.77° and 7.37°, respectively. According to micro-hardness measurement, the HV value of Ni-12at.%V tape is three times higher than that of pure Ni and to a certain extent higher than that of Ni-5at.%W. Furthermore, the Curie temperature of the Ni-12at.%V substrate is decreased to less than 77 K due to the addition of vanadium element in Ni.

*Index Terms*—Coated conductor, cube texture, magnetization loss, Ni alloy.

### I. INTRODUCTION

**I** N RECENT years the Ni-W alloy substrates have been investigated extensively worldwide due to their suitability for coated conductor applications. The substrate must provide a sharp cube texture, high yield strength and oxidation resistance to satisfy the practical requirements during epitaxial growth of buffer layers and superconducting layers [1], [2]. In particular, the Ni-5at.%W(Ni5W) alloy substrate is a attractive candidate alloy and could be produced in hundred-meter scale long tapes by several companies or institutes [3]–[5]. However, the Curie temperature of the Ni5W alloy is 335 K, which means that the ferromagnetism might be a problem and limit the practical applications of the coated conductor even at 77 K (viewed to be the highest working temperature of the coated conductors).

Within the development of non-magnetic textured substrates, several Ni based binary alloys [6]–[8] and ternary alloys [9], [10] have been prepared and characterized on the basis of their mechanical strength, magnetic property and cube texture. Due

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to the obvious decrease of Curie temperature with increasing V content, the Ni-V alloy (V > 9at.%) has turned out to be a promising non-magnetic metallic substrate which can be manufactured with an adequate cube texture using the RABiTS (Rolling Assisted Biaxially textured substrate) method. S. Ceresara, et al. [6] and B. de Boer, et al. [11] have successfully fabricated the textured Ni-11at.%V (Ni11V) and Ni-9at.%V (Ni9V) substrates, respectively, using the melting route. In order to evaluate the suitability of such a non-magnetic Ni-V substrates for epitaxial growth, the YBCO/CeO<sub>2</sub>/NiO [12] and YBCO/CeO<sub>2</sub>/YSZ/CeO<sub>2</sub>/NiO [13] buffer layer and superconducting layer architectures have been selected and deposited on the Ni11V substrate by pulsed laser deposition technique. Using these two architectures, the critical current density value at 77 K and zero magnetic field can reach 0.6  $MA/cm^2$ , which is slightly lower compared to that deposited on Ni5W substrates using the same architecture. This might be caused by intrinsic defects such as broader misorientation and twin misorientation in the Ni-V substrate that have been transferred to the YBCO layer during the epitaxial deposition process [13], [14]. Thus, in order to increase the critical current of the superconducting film grown on non-magnetic Ni-V substrates, further investigations should be oriented towards the manufacturing of Ni-V substrates free of twins and misoriented grains.

The purpose of the present work was to fabricate a non-magnetic textured Ni-12at.%V (Ni12V) substrate by means of the powder metallurgy route which is considered to have advantages compared to the melting route. The macro- and micro- texture have been characterized by both the X-ray diffraction method and EBSD method. At the same time, the micro-hardness and the mass magnetization-temperature dependence have also been investigated to estimate the mechanical and magnetic properties of the Ni12V substrate.

#### **II. EXPERIMENT**

The powder metallurgy route was employed to prepare a Ni<sub>88</sub>V<sub>12</sub> ingot. The nickel powder (purity of 99.9%) and vanadium powder (purity of 99.5%) were weighted in stoichiometric proportions of Ni-12at.%V followed by mixing homogeneously and ball milling for 72 h. After cold isostatic pressing, the pressed bulk was annealed at 1300°C for 10 h as sintering and homogeneity treatment. The thickness of the starting ingot was 8 mm. The cube texture was formed following the cold rolling and recrystallization annealing. The final thickness of the tape is 130  $\mu$ m, which corresponds to a total rolling reduction of 98.4% in thickness. The tape was annealed at various temperatures ranging from 900°C to 1400°C





Fig. 1. (a) XRD profiles; (b) ratio of  $I_{(111)}/I_{(111)} + I_{(200)}$  and cube texture content of Ni12V tapes annealed at different temperatures.

for 1 h to evaluate the cube texture formation in a  $N_2 - 4\%H_2$  gas mixture using the two-step annealing method [15].

Texture of the recrystallized tapes was investigated with an X-ray texture goniometer as well as with a SEM, equipped with an electron back scattering diffraction (EBSD) detector. For X-ray analysis, the  $\theta - 2\theta$  scanning and pole figure (PF) measurements were carried out.

To estimate the strength of the tapes made from different alloys, the Vickers micro-hardness HV-0.2 was measured. A VSM system was used to characterize the magnetic properties of the Ni-12at.%V substrate.

#### **III. RESULTS AND DISCUSSION**

Fig. 1(a) shows the XRD patterns of the Ni12 V tapes annealed at temperatures between 1000°C and 1400°C for 1 h. It is obvious that the (200) reflection peak at 50.7° is the strongest reflection peak in the scanning region, which indicates that a good c-axis orientation has been obtained in all annealing conditions. At the same time, a low intensity (111) peak can be observed at 44.4°. In order to characterize the out-plane orientation of the annealed substrates, the value of the intensity ratio  $I_{(111)}$  to  $I_{(111)} + I_{(200)}$  which (hereafter is named R) has been used to quantitatively evaluate the evolution of the out of plane orientation with increasing annealing

Fig. 2. The orientation distribution map a) and the GBs distribution b) of the Ni12V tape annealed at  $1400^{\circ}$ C for 3 h.

temperature (Fig. 1(b)). The value of R decrease monotonically with increasing annealing temperature, which indicates that the out-plane orientation become sharper with increasing annealing temperature up to 1300°C. In addition, the cube texture content (within a misorientation angle of 10°) also has been plotted in Fig. 1(b) as a function of annealing temperature. The result of continuous increase of cube texture content is in good agreement with the XRD result of decrease in the intensity of (111) orientation. The cube texture content can reach 92.9% (< 10°) for the Ni12 V substrate annealed at 1400°C for 1 h.

In metallic textured substrates used for epitaxial deposition applications, the twins and misoriented grains have been suggested to be significantly responsible for the limitation of the critical current density in the superconducting films [13], [16]. In order to eliminate the twin and broadened cube grains, consequently, increasing the fraction of cube grains, the annealing parameters have been optimized by prolonging the dwell time to 3 h annealing at 1400°C. Fig. 2(a) shows the orientation distribution of the Ni12 V substrate after full recrystallization. The cube grains within a misorientation angle of 10° were colored. The cube texture content is as high as 99% as calculated from EBSD data, whereas the average grain size is 36  $\mu$ m. Furthermore, according to the grain boundary distribution curve (Fig. 2(b)), the fraction of low angle grain boundary (< 10°) can



Fig. 3. The phi-scan a) and rocking curve b) of the Ni12V substrate annealed at  $1400^\circ\mathrm{C}$  for 3 h.

be calculated as 89%. This relatively high fraction of low angle GBs is expected to be favorable for the critical current density since it could be transmitted to superconducting film by means of epitaxy. However, a small amount of twin boundaries (2.9%) can still be detected, although it is much lower in present work than in a previous reported work with about 13.4% [17]. XRD measurements also have been carried out to evaluate the cube texture of the annealed Ni12 V substrate. From the phi-scan and rocking curve, it is found that the FWHM value is 7.77° and 7.37° respectively (Fig. 3). Consequently, it can be concluded that an extremely sharp cube texture has been obtained in the present Ni12V substrate, which is almost equal to the specifications of commercial Ni5W substrate.

Besides the cube texture, mechanical properties are also an important aspect for the advanced metallic textured substrate because long tapes will be subjected to strain inside the deposition chamber during continuous deposition of the buffer layers and superconducting layer. In the present work, the attention has been limited to micro-hardness at room temperature to characterize the mechanical strength of the Ni12V tape. Fig. 4 shows the micro-hardness of Ni-V alloys with different V contents after recrystallization as well as the micro-hardness value of the Ni5W alloy (red star) prepared by ourselves using the similar preparation solution as comparison. It is clearly shown that the micro hardness significantly increases with increasing the V content due to the effect of solid solution strengthening. For the Ni12V substrate, the micro-hardness value is 204, which is three times higher than that of a pure Ni (74) tape as well as to a certain



Fig. 4. The micro-hardness of a Ni5W tape and a series of Ni-V alloy tapes.



Fig. 5. Mass magnetization as a function of temperature in Ni5W and Ni12V tapes.

extent higher than that of the Ni5W alloy (187). This indicates that the mechanical strength of Ni12V substrate could satisfy the requirements for continuous deposition and may thus allow obtaining higher engineering current densities in the coated conductors.

With respect to the practical use of coated conductors, the magnetization loss should be minimized in order to diminish energy losses in alternating current (ac) applications. The temperature dependence of the mass magnetization M (T) of the Ni12V tape, together with the data of a Ni5W substrate measured in a magnetic field of 100 Oe applied parallel to the plane are shown in Fig. 5. It is found that for the Ni12V substrate the value of M is zero down to 77 K. This indicates that the Curie temperature has been suppressed to below 77 K for the Ni12V substrate, while magnetization losses can be expected in a Ni5W substrate at 77 K.

According to the above analysis, it is easy to conclude that the cube texture has been developed and improved successfully in the Ni12V substrate by the powder metallurgy route. Thus the Ni12V substrate which will avoid magnetization losses at 77 K and has a higher mechanical strength than the Ni5W substrate could be employed to deposit the buffer layers and superconducting films. As an alternative, to further improve the mechanical strength of the substrate, the Ni12V alloy could be employed as the outer layer material of a composite substrate to fabricate strengthened fully non-magnetic substrate using the multilayer fabrication technique of composite substrates that have been developed recently [18]–[20]. Architectures like Ni12V/Ni9W or Ni12V/Ni12W is considered for producing such kind of substrate. Work in this direction is now in progress.

## **IV. CONCLUSIONS**

A non-magnetic textured Ni12V substrate has been fabricated successfully by powder metallurgy route. Firstly, the influence of annealing temperature on the cube texture formation has been investigated and optimized annealing parameters have been determined. The cube texture content of fully recrystallized Ni12V substrate is 99% within misorientation angle of  $10^{\circ}$ , at the same time, the fraction of low angle GBs is 89% while the twin boundary content is decreased down to 2.9%. Furthermore, the FWHM value of the phi-scan and rocking curve are 7.77° and 7.37° respectively. These results are similar to the texture quality of commercial Ni5W substrates. Then the micro-hardness of a series of Ni-V alloys together with Ni5W has been investigated, it was found that the micro-hardness value of the Ni12V alloy is three times higher than that of pure Ni and to a certain extent higher than that of the Ni5W substrate. Magnetization measurements indicate that magnetization losses in the Ni12V substrate can be avoided at 77 K. Therefore, it is strongly believed that the Ni12V substrate is a promising and competitive candidate as a low-cost, non-magnetic substrate with high quality of cube texture and good mechanical strength for large-scale coated conductor applications in the near future.

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