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journal or publication title	IEEE Transactions on Applied Superconductivity
volume	16
number	2
page range	1228-1231
year	2006
URL	http://hdl.handle.net/10097/47166

doi: 10.1109/TASC.2006.870005

Neutron Diffraction Study on Prebending Effects for Bronze Route $\mathrm{Nb_3Sn}$ Wires Without Reinforcement

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***Abstract*—The critical current, upper critical field and critical temperature of bronze route $\mathrm{Nb_3Sn}$ commercial wires are enhanced by applying a repeated bending strain at room temperature, i.e., “prebending strain”. In order to investigate the prebending effects from a viewpoint of a residual strain, axial and lateral residual strains were evaluated directly by neutron diffraction at room temperature. We found that the axial residual strain changes from -0.10% to 0.02 % but the lateral one is unchanged by applying a prebending strain of 0.5% for an ordinary bronze route $\mathrm{(Nb, Ti)_3Sn}$ wires without reinforcement. Hence, in the case of the ordinary $\mathrm{Nb_3Sn}$ wires without reinforcement, the prebending treatment modifies only the axial residual strain states independently to the lateral one, although it may depend on the wire structure. The critical current properties under the axial tensile strain suggest that the axial residual strain is reduced by about 0.11% but the radial residual strain unchanged by the prebending treatment of 0.5%. This is consistent with the results of the neutron diffraction.**

***Index Terms*—Critical current, $\mathrm{Nb_3Sn}$, neutron diffraction, residual strain.**

I. INTRODUCTION

$\mathrm{Nb_3Sn}$ is recognized as one of the important practical superconducting materials for a high field magnet over 10 T. However, it is well known that the strong sensitivity of its superconducting properties to stress/strain still remains as a serious problem [1]. In addition, the critical current density J_c of the practical $\mathrm{Nb_3Sn}$ wires is deteriorated due to the residual strain by the thermal contraction difference between $\mathrm{Nb_3Sn}$ filaments and the materials composing the wire, such as bronze matrix, Cu, Nb and so on. Therefore, J_c can be enhanced if one may realize a strain-free state for $\mathrm{Nb_3Sn}$ filaments. In fact, Ochiai *et al.* have already reported that the loaded and unloaded tensile strain treatment increases B_{c2} and J_c for the $\mathrm{Nb_3Sn}$ wires [2]. From this point of view, the react and wind technique is a convenient and efficient method to control the residual strain. Generally, it has been considered that the bending strain above 0.5% induced from pulleys and a coil bobbin during the winding process gives rise to a serious damage for the $\mathrm{Nb_3Sn}$ wires [3]. Recently, we found that for high strength $\mathrm{Nb_3Sn}$ wires, the repeated bending loads applied as coil-winding enhance not only I_c, but also upper critical field B_{c2} and critical temperature T_c [4], [5]. We call the repeated bending strain at room temperature “prebending strain”. It was confirmed that I_c increases for three different type of the bronze route $\mathrm{Nb_3Sn}$ practical wires, and the reinforced $\mathrm{Nb_3Sn}$ wire with Cu-24wt%Nb in outer part of the wire, $\mathrm{CuNb/Nb_3Sn}$, shows larger enhancement of I_c than the ordinary $\mathrm{Nb_3Sn}$ wire [4]. Comparison of the axial tensile strain dependence of I_c for high strength $\mathrm{Nb_3Sn}$ wires with and without prebending strain treatment indicates a reduction of the axial residual strain, and also the enhancement of the maximum value at the peak for the axial strain dependence of I_c [6]. In other words, the critical current at the axial-strain-free state, I_{cm}, is also increased by the prebending treatment for the CuNb reinforced $\mathrm{Nb_3Sn}$ wires. This is one possible reason why the I_c enhancement of $\mathrm{CuNb/Nb_3Sn}$ wire by the prebending treatment is larger than those of the other wires. It also suggested that the radial/tangential residual strain is different from zero even for the axial strain free state. Therefore, I_c of the composite $\mathrm{Nb_3Sn}$ wires can be enhanced drastically, if we optimize the strain states three-dimensionally. For this purpose, we have to understand the three dimensional residual strain state and its effect on the superconducting properties.

Flükiger *et al.* carried out a neutron diffraction study for the bronze route $\mathrm{Nb_3Sn}$ wires without reinforcement and discussed the relation between superconductivity and Martensitic tetragonal transformation [7]. They showed that neutron diffraction is a suitable tool for structure analysis and evaluation of the relative change of the lattice parameters. We tried to measure the residual strain state for the axial and lateral direction for composite $\mathrm{Nb_3Sn}$ wires without reinforcement, which does not show the I_{cm} enhancement, as a first step. In this paper, we report the direct measurement of strain by the neutron diffraction for ordinary bronze route $\mathrm{Nb_3Sn}$ wires without reinforcement, which reveal a small enhancement effect due to the prebending treatment and discuss the prebending effects from the viewpoint of the strain state.

II. EXPERIMENTAL

In order to obtain the typical behavior in the prebending treatment, ordinary nontwisted bronze route practical $\mathrm{Nb_3Sn}$ wires without reinforcement were used in this study. The specifications and the SEM images of the wire are shown in Table I and Fig. 1, respectively. This wire is one of the US-Japan reference $\mathrm{Nb_3Sn}$ wires [8]. As the prebending strain treatment, the

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Manuscript received September 19, 2005. This work was supported in part by Industrial Technology Research Grant Program in ’04 from New Energy and Industrial Technology Development Organization (NEDO) of Japan and the Ministry of Education, Science and Technology, Japan.

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Digital Object Identifier 10.1109/TASC.2006.870005

TABLE I
SPECIFICATIONS OF BRONZE ROUTE Nb_3Sn WIRE

bronze (wt%)	Cu-13.2Sn-0.3Ti
barrier	Nb
wire diameter (mm)	1.06
twist	no
filament diameter (μm)	3.8
number of filaments	361 × 31
Cu/non Cu (%)	0.859
heat treatment	645 °C × 200 h

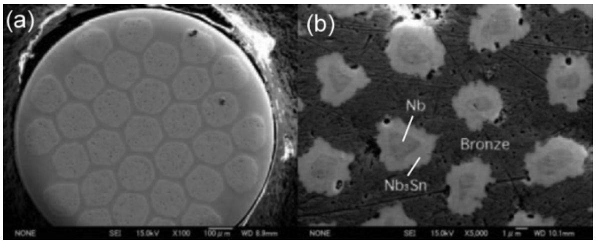

Fig. 1. SEM images of the bronze route Nb_3Sn wire used in this study.

bending strain, ϵ_{pb}, was loaded and unloaded 5 times at room temperature, where the opposite bending strains were applied alternately. For the neutron diffraction measurement, the wire was cut into 100 short pieces with 10 mm in length and stacked parallel into a cubic shape with 10 mm on the side. The neutron diffraction measurements were conducted at a time of flight (TOF) neutron diffractometer Sirius [9] installed at the pulsed spallation neutron facility KENS, High Energy Accelerator Research Organization, Japan. At high resolution scattering bank ($153° < 2\theta < 175°$), diffraction patterns with many hkl peaks for $d < 2.5$ Å can be measured simultaneously. We evaluated the diffraction for both axial and lateral directions by using backward bank with 90 degree rotation of the specimen. Because of the round shape of the wire, the observation for the lateral direction includes both the radial and tangential component of the strain. In order to improve the signal to noise ratio, we integrated the diffraction patterns for 1 h. In addition, the X-ray diffraction for the Nb_3Sn filaments was also measured as a reference without residual strain. Nb_3Sn filaments were obtained from the same wire by an acid etching. The transport I_c properties were evaluated as a function of axial tensile strain at 14.5 T and 4.2 K. A 1 $\mu V/cm$ criterion was used for the I_c definition.

III. RESULTS AND DISCUSSION

A. Evaluation of Residual Strain

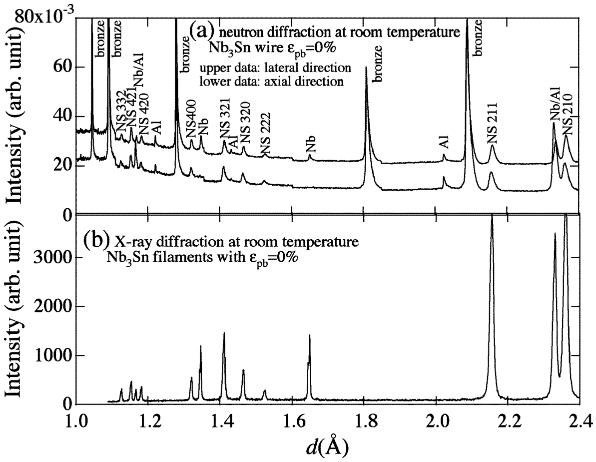

Fig. 2. Diffraction patterns of (a) bronze route Nb_3Sn wire and (b) its filaments.

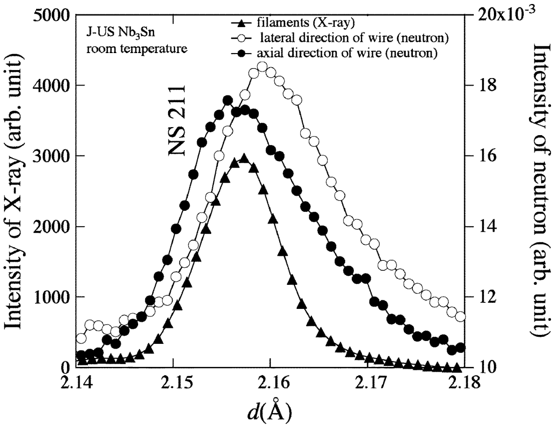

Fig. 3. Comparison of the 211 diffraction peaks of Nb_3Sn for the axial and the lateral directions.

Fig. 2(a) shows the neutron diffraction patterns of the samples without prebending treatment for the axial and lateral directions. The diffraction peaks of the constituent materials, i.e., Nb_3Sn, bronze and Nb are seen in the figure. The peaks of Aluminum appear because we use an aluminum tape to fix the specimen. The X-ray diffraction data of the filaments of the same wire are also shown in Fig. 2(b). In Fig. 2(b), the diffraction peaks of Nb are observed although all the matrix materials were etched away. These peaks come from the unreacted Nb core of the filament. The Nb core can be seen in Fig. 1(b). The volume fraction of Nb core is about 25%, which was determined from the SEM image. Thermal contraction coefficient of Nb is very close to that of the Nb_3Sn and, hence, we consider that the XRD data of the filaments in Fig. 2(b) gives the stress free lattice constant. We obtained the lattice parameter of 5.2860 Å from the XRD of the Nb_3Sn filaments by the Nelson and Relay method. This value was used as a stress free lattice parameter of Nb_3Sn in this study. In order to discuss the detail of the residual strain, we analyzed the 211 diffraction peak of Nb_3Sn as shown for example in Fig. 3. The d-value for the axial direction of the wire is about 0.0018 Å smaller than that of the filaments. However, the d-value of the peak for the lateral direction of the wire is about 0.0001 Å larger than that of the filaments. We calculated residual strains from a few diffraction peaks statistically. The obtained results are summarized in Table II. The axial and lateral residual strains of Nb_3Sn are about $-0.103 \pm 0.003\%$ and $0.03 \pm 0.015\%$, respectively. Note that we take the compressive strain negative in this study. Assuming the thermal expansion constants and elastic ones equal to $7.64 \times 10^{-6}/K$ and 110 GPa for Nb_3Sn, $17.3 \times 10^{-6}/K$ and 124 GPa for Bronze and $16.8 \times 10^{-6}/K$ and 114 GPa for Cu, respectively, we estimated a residual strain of 0.10% at room temperature for Nb_3Sn on the basis of the axial stress balance [10]. In this calculation, we also assumed that only Cu is yielded at above 20.7 MPa and Bronze

TABLE II
SUMMARY OF MEASURED LATTICE CONSTANTS AND STRAINS

	ϵ_{pb}=0 %		ϵ_{pb}=0.5 %		
	a (Å)	strain (%)	a (Å)	strain (%)	Δstrain (%)
Nb_3Sn					
axial	5.28006±0.0002	-0.103±0.003	5.2871±0.0003	0.020±0.006	0.12
lateral	5.2877±0.0008	0.03±0.015	5.2878±0.0010	0.034±0.018	0.004
Bronze					
axial	3.6196±0.0008		3.6191±0.001		-0.013
lateral	3.6173±0.0001		3.6177±0.0008		0.011

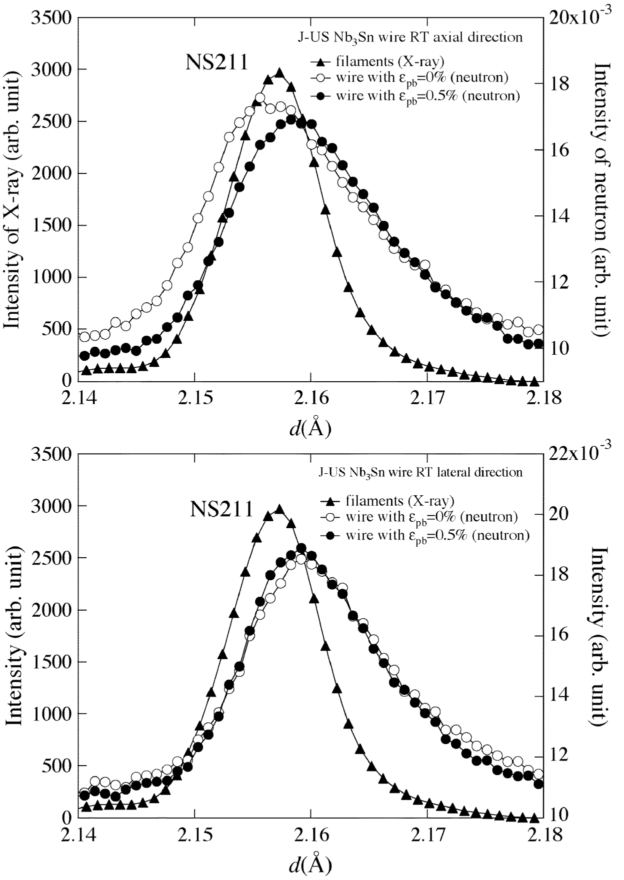

Fig. 4. Prebending effects on the 211 diffraction peaks of $\mathrm{Nb_3Sn}$ for (a) axial and (b) lateral directions.

becomes very soft at 600 K [7]. The estimated axial residual strain mentioned above is in agreement with the experimental result obtained from the neutron diffraction as shown in Table II.

Next, we compare the neutron diffraction patterns between the samples with and without prebending strain of 0.5% as shown in Fig. 4. The d-value of $\mathrm{Nb_3Sn}$ 211 reflections changes from 2.1556 Å to 2.1592 Å for axial direction, but is unchanged for lateral direction. The main characteristics of the residual strains after the prebending treatment are also listed in Table II. By applying the prebending strain of 0.5%, the compressive residual strain of 0.103% becomes tensile strain of 0.02% for the axial direction, but the tensile strain of 0.03% keeps constant for the lateral direction. Therefore, we found that the prebending strain changes only axial residual strain independently to the lateral one in the ordinary $\mathrm{Nb_3Sn}$ wire without reinforcement. On the other hand, the residual strain of the bronze can not be determined, because a lattice parameter of the bronze in the stress free state is unknown. However, the relative change of the residual strain due to the prebending strain can be discussed, if we use the d-value of the bronze before prebending treatment as a stress free value. The residual strain of the bronze shifts about 0.013% toward compressive side, which is the opposite direction with $\mathrm{Nb_3Sn}$, although it is lower than the deviation as shown in Table II. When the prebending strain is applied, it is considered that a part of the bronze deforms plastically. The plastic deformation of the bronze plays an important role for the reduction of the residual strain of $\mathrm{Nb_3Sn}$ due to the prebending treatment as described previously [11]. However, since only elastic deformation is detected by the neutron diffraction measurement, it is considered that the obtained residual strain of the bronze is very small.

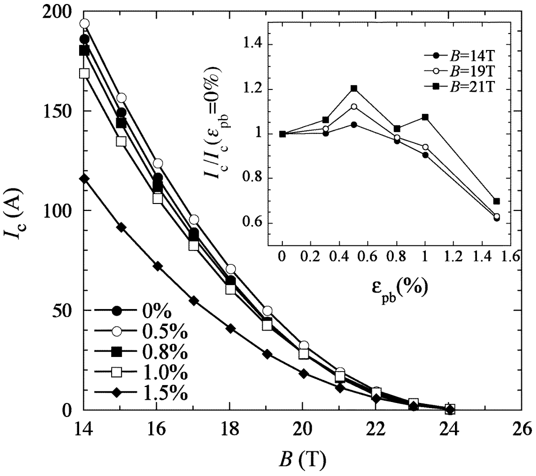

Fig. 5. Prebending effects on $I_c - B$ properties. Inset shows the normalized I_c as a function of prebending strain.

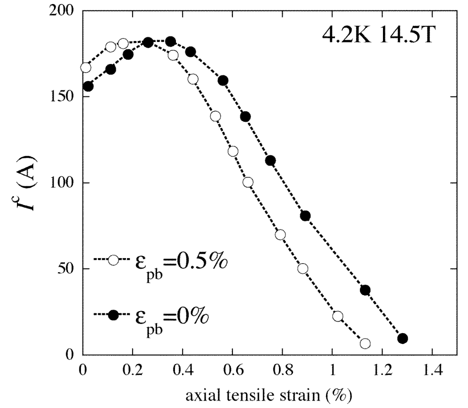

Fig. 6. Axial tensile strain dependence of I_c before and after prebending treatment.

B. Critical Current Properties

The prebending strain effects on I_c are shown in Fig. 5. The I_c value is about 186.3 A at 14 T before prebending treatment. With increasing ϵ_{pb}, I_c increases to the maximum value of 194.1 A at 14 T for $\epsilon_{pb} = 0.5\%$ and then decreases. This trend is almost similar in the whole magnetic field region. The inset of the figure exhibits the normalized I_c as a function of prebending strain at various magnetic fields. The enhancement of I_c becomes larger in higher magnetic fields and above 120% over 21 T. Fig. 6 shows the axial tensile strain dependence of I_c of the as heat-treated and prebent specimens. The I_c-strain curve shifts about 0.12% toward the compressive side by the prebending strain. This means that the axial residual strain, which was determined by the peak strain, is reduced by about 0.11%. This relative change of axial residual strain by the prebending treatment of 0.5% is in agreement with the result of the neutron diffraction for the axial direction as mentioned above, in spite of the temperature difference between the neutron diffraction experiments at room temperature and the J_c measurement at 4.2 K. Hence, the relative change of the residual strain due to the prebending treatment at room temperature is kept even at low temperature of 4.2 K.

On the other hand, the maximum critical current on the axial strain dependence of critical current, I_{cm}, is not enhanced in this sample, although the high strength Nb_3Sn wires with reinforcement reveal a large enhancement effect of the I_{cm} value [4], [6]. We consider that the prebending treatment changes not only axial but also radial/tangential residual strains, because the enhancement of the I_{cm} is often observed for the high strength Nb_3Sn wires. In this study, however, the prebending treatment affects only axial residual strain and the lateral one is unchanged. Hence, the conservation of the lateral residual strain observed by the neutron diffraction is also consistent with the unchanged I_{cm} behavior. The independent nature of the residual strain along the longitudinal and the lateral direction on the prebending effect is likely to depend on the wire structure. In the case of the internal reinforced Nb_3Sn wires, the lateral strain is probably not constant versus the prebending treatment. Because the I_{cm} value increases by the prebending treatment for the CuNb reinforced Nb_3Sn wires, in contrast with ordinary Nb_3Sn wires used in this study.

The increase of I_{cm} due to the prebending treatment is very important for both basic research and applications. The detailed evaluation of the three dimensional residual strain is necessary for various kinds of the Nb_3Sn wires. The high resolution neutron diffraction study is a very useful tool for understanding the residual strain state in practical Nb_3Sn wires.

IV. CONCLUSION

Residual strain states both axial and radial/tangential directions were directly evaluated from high-resolution neutron diffraction measurements. I_c properties under axial tensile strain for the bronze route Nb_3Sn practical wire without reinforcement are discussed, when prebending treatment is applied. We found that only the axial residual strain for ordinary Nb_3Sn wire is changed from -0.10% to 0.02% and the lateral one is unchanged, by the prebending treatment of 0.5%. This difference of the residual strain between before and after the prebending treatment can explain the enhancement of I_c and the change of the strain dependence of I_c.

ACKNOWLEDGMENT

The authors would like to thank Furukawa Electric Co. Ltd. for supplying samples. The high magnetic field measurements were performed at HFLSM, IMR, Tohoku University. The authors would also like to thank Y. Ishikawa, Y. Sasaki, and K. Sai of the HFLSM and H. Miura, S. Tanno, and K. Hosokura of the Center for Low Temperature Science for their assistance.

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