Magnetization studies of RHQT-processed Nb₃Al wires for high-field accelerator magnet applications

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Abstract. Rapid heating/quenching and transformation (RHQT)-processed Nb₃Al wires possess better strain tolerance than Nb₃Sn wires and exhibit similar high-field properties. Therefore, Nb₃Al wires might be promising candidates for use in future high-field accelerator magnets. For this reason, we have been developing RHQT-processed Nb₃Al wires for a number of years. During this development, magnetization measurements on several samples have been carried out as a function of either temperature or the magnetic field. This paper presents some of the results of the magnetization measurements performed.

1. Introduction

Rapid heating/quenching and transformation (RHQT)-processed Nb₃Al wires [1] possess better strain tolerance [2, 3] than Nb₃Sn wires and exhibit similar high-field properties. Therefore, they might be promising candidates for use in future high-field accelerator magnets. However, in order to meet the demands of future accelerator magnet designs, it is necessary to develop and enhance the performance of Nb₃Al wires. For this reason, we have been developing RHQT-processed Nb₃Al wires for a number of years [4, 5].

Magnetization is an important property of superconducting wires used in actual accelerator magnet; this is because it directly affects the stability and field quality of accelerator magnets through superconductor magnetization.

In recently developed Nb₃Sn wires, particularly internal-tin route wires, the achieved current density is extremely high: approximately 3000 A/mm² in the non-copper area at 12 T and 4.2 K. In order to achieve this high current density, the amount of copper was reduced and the areas of Nb and Sn in the cross section of the wire were increased to the highest degree possible, and eventually, the area of Nb₃Sn in the cross section of the wire was increased. Consequently, there was an increased tendency of the effective filament diameter to be large due to merging of the filaments after the reaction; this resulted in the problem of low-field instability [6].

On the other hand, RHQT-processed Nb₃Al wires are fabricated by a transformation process in which a Nb(Al) supersaturated bcc solid solution phase is converted to a A15 phase. Therefore, it is believed that the effective filament diameter of RHQT-processed Nb₃Al wire would be almost the same as that during the original fabrication. However, it has recently been found that conventional Nb-matrix wires exhibit rather strong magnetic instability at a low field [7, 8] and this is attributed to the
superconductive nature of Nb, which is used as the matrix material. In order to solve this problem, we began developing new Nb₃Al wires with a Ta matrix [9]. During the development, we measured the magnetization of the wires in addition to their critical current and critical temperature, in order to determine the nature of various superconducting phases in the wires and to study the magnetization characteristics of the wires.

2. Experimental Details

2.1. Wire description

Two multifilamentary Nb/Al precursor wires were prepared using a conventional jelly-roll (JR) process at Hitachi Cable, Ltd. The design parameters of the wires are listed in Table 1. One of the wires is a Ta-matrix wire and the other is a conventional Nb-matrix wire. These precursor wires having a diameter of 1.35 mm were then subjected to RHQ treatment. During this treatment, the precursor wire was rapidly heated up to approximately 2000 °C by ohmic heating at a constant current (I_{RHQ}) and subsequently quenched in a Ga bath at approximately 50 °C. Through this process, the Nb/Al composite filaments were converted into a Nb(Al) supersaturated bcc solid solution. After this process, the wires were drawn down to different final sizes and were finally heat treated at 800 °C for 10 h to convert the bcc phase into the superconducting A15 phase. Figure 1 shows the cross section of the heat-treated wire (ME476) having a diameter of 1.0 mm and its X-ray intensity maps obtained to observe the element distribution in a filament.

| Table 1. Precursor wire parameters. |
|-------------------------------------|
| Wire diameter (mm)                  | ME476 | ME451 |
| Matrix material and matrix ratio    | Ta (0.8) | Nb (0.7) |
| Filament diameter (µm) and spacing (µm) | 69, 8 | 63, 6.4 |
| Number of filaments                 | 222 | 294 |
| Non-Cu J_c (A/mm²) @ 15 T, 4.2 K    | 623 | 946 |
| for wire with φ1.35 mm (0% area reduction) |  |  |
| for wire with φ1.0 mm (45% area reduction) |  |  |

Figure 1. SEM images of ME476 wire: (a) overview, (b) filament structures after heat treatment, and (c) X-ray intensity distribution maps obtained to observe the element composition in a filament.
2.2. Measurement procedures
In order to carry out magnetic characterization of the wires, magnetic moments were measured as a function of either temperature or the magnetic field by using a Quantum Design SQUID magnetometer. The lengths of the samples used in this study were distributed between 2.5 to 7 mm. During the measurement of temperature dependence, which was performed in order to obtain information about the nature and size of the various superconducting phases in the wires, the samples were cooled to 2 K in a zero field. Thereafter a 10 mT field was applied and the magnetic moment was measured at intervals of 0.1 K until the transition temperature was exceeded. In order to study the applied field dependence of the magnetic moment, the measurement was carried out in a field range of -5 to 5 T and at temperatures of 4.4 K and 2.0 K.

2.3. Magnetic moment and superconductor volume
In general, the magnetic induction in materials is expressed by

$$B = \mu_0 (H_{\text{eff}} + M) = \mu_0 (H_{\text{ap}} + H_d + M) = \mu_0 (H_{\text{ap}} - NM + M)$$

(1)

where $H_{\text{eff}}$ is the effective internal field that is equal to the applied external field $H_{\text{ap}}$ corrected by the demagnetizing field $H_d$, $M$ is the magnetization of the material and $N$ is the demagnetizing factor.

The magnetic induction becomes zero if we assume that the material is in a perfect shielding state. Thus, we obtain the magnetization as follows:

$$M = -H_{\text{ap}} / (1 - N)$$

(2)

For a long cylinder in parallel and transverse fields, $N = 0$ and $N = 2$, respectively.

From equation (2), the magnetic moment obtained by the SQUID magnetometer can be expressed as follows:

$$m = M \cdot V = -H_{\text{ap}} / (1 - N) \cdot V$$

(3)

where $V$ is the superconductor volume.

3. Results and Discussion

3.1. Temperature dependence of magnetic moment
Figure 2 shows the magnetic moment as a function of temperature for the samples ME476 ($\phi 1.35 \text{ mm}$) and ME451 ($\phi 1.0 \text{ mm}$) in a field of 10 mT. The magnetic moments in the parallel and transverse fields are plotted in Figs. 2(a) and 2(b). For both wires, the first drop in the signal is observed at $T = 17.6$ K; this drop can be attributed to the superconducting transition of Nb$_3$Al. As the temperature decreases, the magnetic moment of the ME476 sample remains almost constant down to $T = 4.2$ K, where the second drop occurs; this drop can be attributed to the superconducting transition of the Ta matrix. In the case of the ME451 sample, the second drop occurs at $T = 9.1$ K, which corresponds to the superconducting transition of the Nb matrix. After this drop, the magnetic moments attain their second plateaus. These two plateaus for both the wires correspond to the condition of perfect magnetic shielding: the first is due to Nb$_3$Al, and the second is due to the matrix material (Ta or Nb). We can use these plateau values of the magnetic moment in equation (3) and estimate the wire diameters and the filament sizes [10]. In figure 3, the ratio of the estimated wire diameter to the measured wire diameter is shown as a function of length-to-diameter ratio ($\gamma$) of the sample wires. It can be observed that when $\gamma$ is below 5, the estimated wire diameters are considerably larger than the actual wire diameter. This could be attributed to the demagnetization effect of the samples. $\gamma$ of the filament is considerably higher than that of the wire diameter. Therefore, the effect of demagnetization might be negligible. We calculated the filament diameters of several samples using equation (3) and obtained reasonable values. However, we could not examine the accuracy of the estimated filament diameter because the actual filament diameter, which was the reference, was not known.

The other features that can be observed from these figures are as follows. First, the transition width of the Nb matrix is slightly broader than that of the Ta matrix. Second, there is a small gradual decrease of the magnetic moment below 8 K in the data of ME476 ($\phi 1.35 \text{ mm}$), which was measured in
the transverse field. The reason for this broader transition width of Nb matrix is not clear, but we assume it to be related to the small amount of impurities in the matrix material. For the second feature, we speculate the existence of a small amount of unreacted Nb in the filaments. The existence of the Nb and/or Nb-rich region in the RHQT filaments has been reported by P. Lee [11].

Figure 2. Magnetic moment as a function of temperature: (a) Magnetic moment of ME476 in parallel and transverse fields and (b) magnetic moment of ME451 in parallel and transverse fields.

Figure 3. Wire diameter estimated from magnetic moment as a function of sample length-to-diameter ratio.

3.2. Effect of RHQ current on m(T) curve

The RHQ current is an essential processing parameter that determines the microstructures of as-quenched and subsequently transformed wires. Therefore it greatly influences the $J_c$ and $T_c$ properties of the wires. Various studies have reported on this effect for different kinds of wires [1, 4, 5]. Here, we investigated the effect of the RHQ current on the curve of the magnetic moment $m(T)$ of the ME476 wires.

The normalized $m(T)$ plots of the samples, which were subjected to different RHQ currents and then heat-treated at $800 \degree C$ for 10 h, are shown in figure 4. The figure shows that the $m(T)$ plots of the ME476 sample in the RHQ current range of 226 A to 229 A are almost the same; further, it shows that only the $m(T)$ of the sample treated at 230.5 A shifts slightly towards the lower temperature side. In order to compare this behaviour of $m(T)$ with those of other wire properties, the plots of the critical temperature ($T_c$) defined by the resistance measurement and the non-Cu $J_c$ value of the same samples
are shown in figure 5. The following points can be deduced on comparing figures 4 and 5. (1) The shape of the $m(T)$ plot is not affected by the change in the RHQ current if the current is in the plateau region, where the $J_c$ and $T_c$ are relatively unaffected by the RHQ current. (2) The results of the $m(T)$ measurement are qualitatively consistent with those of the $T_c$ measurement.

Figure 4. Normalized magnetic moments of ME476 ($\phi 1.35$ mm) samples subjected to different RHQ currents.

Figure 5. RHQ current dependence of $T_c$ and non-Cu $J_c$ for ME476 sample with $\phi 1.35$ mm.

3.3. Effect of area reduction on $m(T)$ curve

The effect of area reduction (AR) after the RHQ treatment is another concern in the fabrication of the wires, because such reduction will usually improve the $J_c$ properties of the Nb$_3$Al wires. Therefore, we measured the $m(T)$ of the ME476 samples with different AR ratios. Figure 6 shows a typical result of this measurement. At first glance, the $m(T)$ plot does not appear to be affected by the AR treatment, however, a closer examination reveals a small effect of the treatment on the transition width of the Nb$_3$Al phase. That is, the transition width becomes narrower with the increase in the level of AR treatment. This behaviour was also observed in the transition width of $T_c$ obtained by the resistance measurement as shown in figure 7.

Figure 6. Normalized magnetic moments of ME476 samples with different AR ratios.

Figure 7. Variations in $T_c$ and the transition width for ME476 samples as a function of AR level.
3.4. Magnetization
The main objective of developing the Ta-matrix wires was to reduce the low-field instability observed in conventional RHQT-processed Nb,Al wires [7, 9]. Therefore, the magnetization of the Ta-matrix wires was examined in detail and compared to that of the Nb-matrix wires (ME451). During the measurements, an external field was applied perpendicularly to the sample axis.

Figure 8 shows the magnetization curves of the Nb-matrix sample (ME451) with diameters of 1.35 mm and 1.02 mm at 4.4 K and 2 K, respectively. The filament diameter and the filament spacing of the thinner wire are approximately 47 µm and 4.7 µm, respectively. A number of flux jumps can be observed in the magnetization curve at 4.4 K, and at 2 K, the number of jumps increases further. As the temperature decreases from 4.4 K to 2 K, the magnetic field above the region that experiences the flux jump increases from approximately 0.5 T to approximately 0.7 T. Further, by reducing the wire diameter, a considerable increase in magnetization at the low field can be observed; this effect is stronger at 4.4 K than at 2 K.

Figure 9 shows the magnetization curve of the Ta-matrix sample (ME476) with diameters of 1.35 mm and 1.02 mm at 4.4 K and 2 K, respectively. The filament diameter and the filament spacing of the thinner wire are 51 µm and 5.9 µm, respectively. The magnetization curves at 4.4 K are very smooth and no flux jump can be observed. However, small flux jumps can be observed in the magnetization curves at 2 K below 0.7 T. These jumps have also been observed in the magnetization

Figure 8. Magnetization of samples with Nb-matrix (ME451) at 4.4 K and 2 K.

Figure 9. Magnetization of samples with Ta-matrix (ME476) at 4.4 K and 2 K.
curves of other Ta-matrix wires [8]. The characteristics of these tiny flux jumps appear to be different compared to those of the flux jumps observed in the ME451 sample; that is, the flux penetration might be limited only to the surface region of the wire. Further, no flux penetration into the filament region was observed. At present it is unclear whether any problems related to these tiny flux jumps will be encountered in the application of Ta-matrix wires for high-field accelerator magnets.

As can be observed from figures 8 and 9, although the widths of the Nb-matrix wires at 2 T are almost the same, the magnetization widths of these wires below 0.5 T are roughly twice those of the Ta-matrix wire. The large magnetization in the Nb-matrix wire might be attributed to the proximity coupling between the filaments in this wire.

4. Conclusion
In this study, we measured the magnetic moment of RHQT-processed Nb₃Al wires as a function of temperature during their fabrication; the measured data were compared with results obtained from the Tc measurement. The main results of this study are summarized as follows. (1) The results of measurement of the magnetic moment are consistent with those of the Tc obtained by the resistance measurement. (2) The m(T) curve of the Nb₃Al wire is not very sensitive to the compositional homogeneity in the filaments. (3) The measurement of the magnetic moment would be effective in detecting the presence of other superconducting phases as well as the Nb₃Al phase.

In addition to measuring the magnetic moment, we measured magnetization as a function of the field. This provided useful information about the stability and/or magnetization of the wires. The following points have been elucidated in this study. (1) Magnetization of the newly developed Ta-matrix wire in a low-field region was approximately half that of the Nb-matrix wire. (2) When Ta was used as the matrix material, flux jumps at 4.4 K were completely suppressed. A small variation in the magnetization was observed at 2 K in the field below 0.7 T; however, flux penetration because of this change appeared to be limited only to the surface region of the wire. Complete flux penetration did not occur.

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