The published version of this preprint appeared in IEEE Transactions on Applied Superconductivity 23, 6000704 (June 2013).

Effect of Thermal Cycle on the Lattice Structure in RHQ-Nb_3Al Superconducting Wire

Xinzhe Jin , Tatsushi Nakamoto, Kiyosumi Tsuchiya, Toru Ogitsu, Akira Yamamoto, Akihiro Kikuchi, Takao Takeuchi, Stefanus Harjo, Takayoshi Ito, and Yo Tomota

***Abstract*—In A15 superconducting wires, it is known that the critical current has a dependence on the strain in a high magnetic field. Therefore, RHQ-Nb_3Al wires are being studied to develop a high field magnet. Since the wire was composited by three or more materials usually, residual strain is induced by different coefficients of thermal expansion in materials in the cooling process after A15 phase transformation. In neutron diffraction measurements at room temperature, we previously reported that the residual strain of Nb_3Al filaments in the wire is tensile. We also reported that the smaller tensile residual strain at room temperature is better for mechanical performance of the superconducting wire. These results indicate that reduction of the residual strain is needed. In this study, we effectively reduced the residual strain by using a thermal cycle method after A15 phase transformation. By applying one thermal cycle process, the tensile residual strain was decreased by approximately 0.08%. The thermal cycle method will be useful to improve the mechanical strength of RHQ-Nb_3Al wire. In this paper, we report the details of the thermal cycle method, its effect on the lattice structures of Nb_3Al and Cu at room temperature, and the analysis results from the perspective of material strength in strain recovery and its hysteresis.**

***Index Terms*— neutron diffraction, A15, Nb_3Al, RHQ, residual strain, multi-peak analysis**

I. INTRODUCTION

Development of Nb_3Al superconducting wires fabricated by the rapid heating and quenching (RHQ) method has been carried out for a high field magnet [1–3]. For RHQ treatment above 2000 °C, Nb or Ta with a high melting point is chosen as the matrix material. Since the coefficients of thermal expansion (CTE) in Nb and Ta are smaller than those of Cu and Nb_3Al, the residual strain in Nb_3Al induced by the difference of the CTEs is tensile at room temperature [4].

Residual strain is induced in the cooling process after heat treatment of A15 phase transformation. With the temperature decreasing, the materials in the wire interact with each other and the thermal stress is not fully constrained. Then, residual strains are induced in the materials. To understand the strain generative mechanism, it is necessary to study the lattice structure behavior in the temperature profile.

In RHQ-Nb_3Al wires having different matrix materials, we reported that have different residual strains in the Nb_3Al filaments at room temperature in the axial direction of the wire [4]. This difference has an effect on the mechanical properties of the wire. A higher reversible strain limit of the critical current is determined in a sample having smaller tensile residual strain in the axial direction of the Nb_3Al filaments. Therefore, reducing the tensile residual strain related to improvement of the mechanical performance.

We have already reported on the residual strain generation for Nb/Ta matrix wire in heat treatment of the A15 phase transformation [5]. In this study, we found that the residual strain has a dependence on the thermal cycle after heat treatment of the A15 phase transformation.

II. EXPERIMENTAL

The Nb_3Al wire having Nb/Ta composite matrix after the A15 phase transformation was chosen for measuring neutron diffraction [5]. The material of the inter-filament is Ta. A series of neutron diffraction measurement was carried out between 23 °C (room temperature) and 800 °C in the J-PARC "TAKUMI" diffractometer with the time-of-flight (TOF) method [5, 6].The sample was placed in a non-crystalline glass tube in a vacuum and the temperature was increased by using an infrared heater, as shown in Fig. 1. The neutron beam size of the sample slit was 5 mm in width and 6 mm in height. This experiment was carried out with proton beam power of 200 kW.

The temperature profile is shown in Fig. 2. After heat treatment of A15 phase transformation consisting of an 800 °C holding process for 10 h, one thermal cycle was performed. The neutron diffraction measurements were carried out during the thermal cycle process as shown in the figure. First, the sample was measured from room temperature to 700 °C with 100 °C steps in the temperature holding status at intervals of 30 min. Next, the sample was measured in the 800 °C holding status for 1 h. Finally, the sample was measured to 200 °C with 200 °C

Manuscript received 3 February 2012. This work was supported by the LHC luminosity upgrade.

Xinzhe Jin, Tatsushi Nakamoto, Toru Ogitsu, Kiyosumi Tsuchiya and Akira Yamamoto are with the High Energy Accelerator Research Organization (KEK), Ibaraki, 305-0801 Japan.

(Corresponding author to provide phone: +81-29-864-5460; e-mail: shintetsu.kin@kek.jp).

Akihiro Kikuchi and Takao Takeuchi are with the National Institute for Materials Science, Ibaraki, 305-0047 Japan.

Stefanus Harjo is with the Japan Atomic Energy Agency, Tokai-mura, Naka-gun, Ibaraki, 319-1195 Japan.

Takayoshi Ito is with the Comprehensive Research Organization for Science and Society, Tokai-mura, Naka-gun, Ibaraki, 319-1106 Japan.

Yo Tomota is with the Department of Mechanical Engineering, Ibaraki University, Hitachi, Ibaraki, 316-8511 Japan.

The published version of this preprint appeared in IEEE Transactions on Applied Superconductivity 23, 6000704 (June 2013).

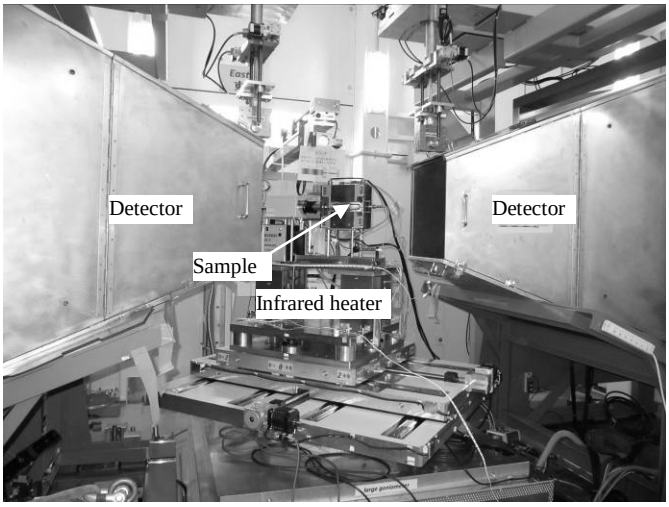

Fig. 1 Neutron diffraction measurement at J-PATC "Takumi" by using infrared heater at high temperature. The sample was measured in a vacuum.

steps in a natural cooling process.

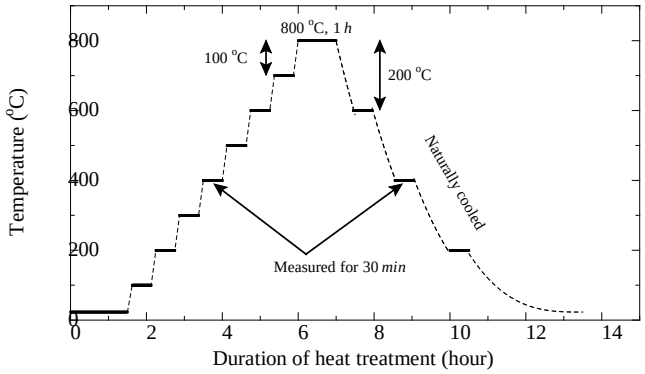

Fig. 2 Temperature profile for thermal cycle. The neutron diffraction measurements were carried out over one thermal cycle after A15 transformation.

III. Result and Discussion

A. Neutron diffraction patterns and their analysis

The neutron diffraction patterns measured at room temperature after the A15 phase transformation before the thermal cycle process are shown in Fig. 3. By comparing each peak intensity in the same Miller's index between the axial and the transverse directions, it can be seen that all materials in the sample have texture. Due to the texture of the lattice planes, it is difficult to determine the lattice structure parameters by using the peak intensities in the patterns of the material. For this reason, the values of the lattice spacing d was obtained by using the peak position in the TOF by single peak analysis in the thermal cycle process to determine the lattice structure change. To estimate the changes of the lattice spacing d, the lattice spacing expansion ratio R_d was calculated as

$$R_d = (d - d_{RT})/d_{RT}.$$

Where d_{RT} is the lattice spacing at room temperature. The lattice spacing expansion ratio R_d in single material corresponds to CTE that has independence in temperature.

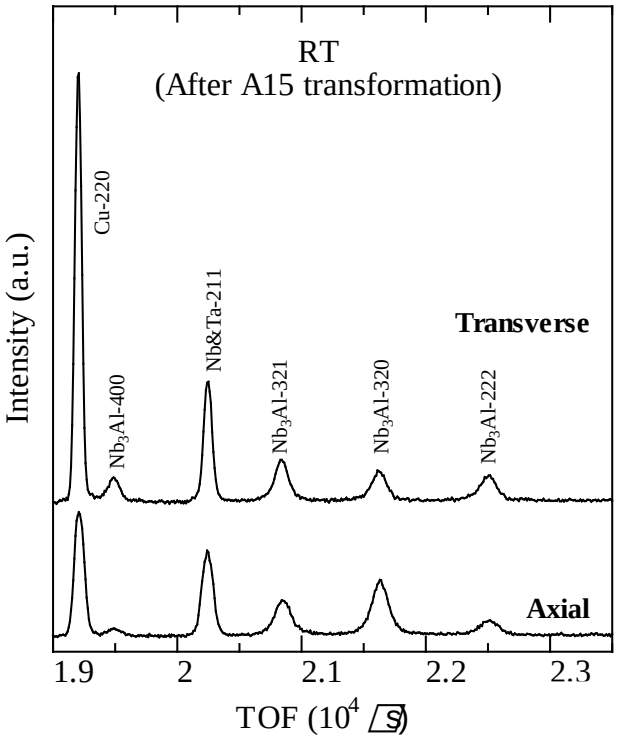

Fig. 3. Neutron diffraction patterns for transverse and axial directions of the wire. Each pattern was measured for 90 minutes at room temperature before the thermal cycle.

B. Thermal cycle effect on the Nb_3Al filaments

The temperature dependences of the lattice spacing expansion ratios R_d for the Nb_3Al filaments in the axial and the transverse directions are shown in Fig. 4 (a) and (b), respectively. Eight peaks of the large lattice spacing d are observed. All the peaks show almost the same behavior in each direction of the wire. This result indicates that the lattice strain of the Nb_3Al in the wire is isotropic with thermal change.

In the axial direction, shown in Fig. 4 (a), hysteresis behavior is shown in the thermal cycle, and that irreversibility is very strong. By this hysteresis, the lattice spacing expansion ratio R_d in the Nb_3Al filaments is decreased by approximately 0.1% at 200 °C, then the residual strain is decreased by 0.08% that obtained by using the lattice spacing d_0 of a powder sample [7]. This decrease indicating the different behaviors of residual stress in the Nb_3Al in strain generation and recovery may be induced by the phase stress change in the Nb_3Al filaments by the thermal cycle. Considering that the residual strain in Nb_3Sn filaments can be controlled by Pre-Bending treatment in Nb_3Sn wires [8], the thermal cycle method is also a control method of the residual stress in Nb_3Al filaments. Differences of the both method are that the Pre-Bending treatment and the thermal cycle are using a stress and heat to control the residual stress, respectively, and these show the increased and decreased effects in the residual strain in axial direction of wire, respectively. As a result, the mechanical performance of the Nb/Ta matrix RHQ-Nb_3Al wire can be improved by the thermal cycle method in the axial direction of the wire.

The published version of this preprint appeared in IEEE Transactions on Applied Superconductivity 23, 6000704 (June 2013).

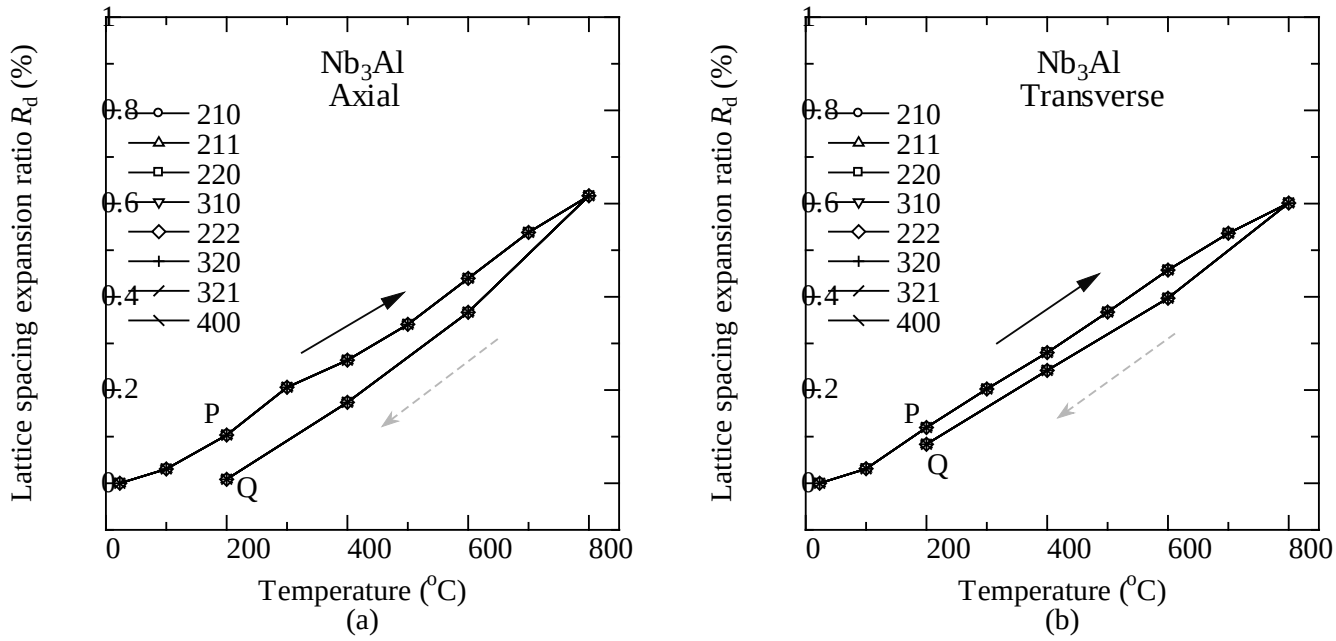

Fig. 4. Temperature dependence of the lattice spacing expansion ratio R_d in the thermal cycle process for Nb_3Al filaments in (a) axial and (b) transverse directions. All the errors of the lattice spacing expansion ratio R_d were less than 0.01%.

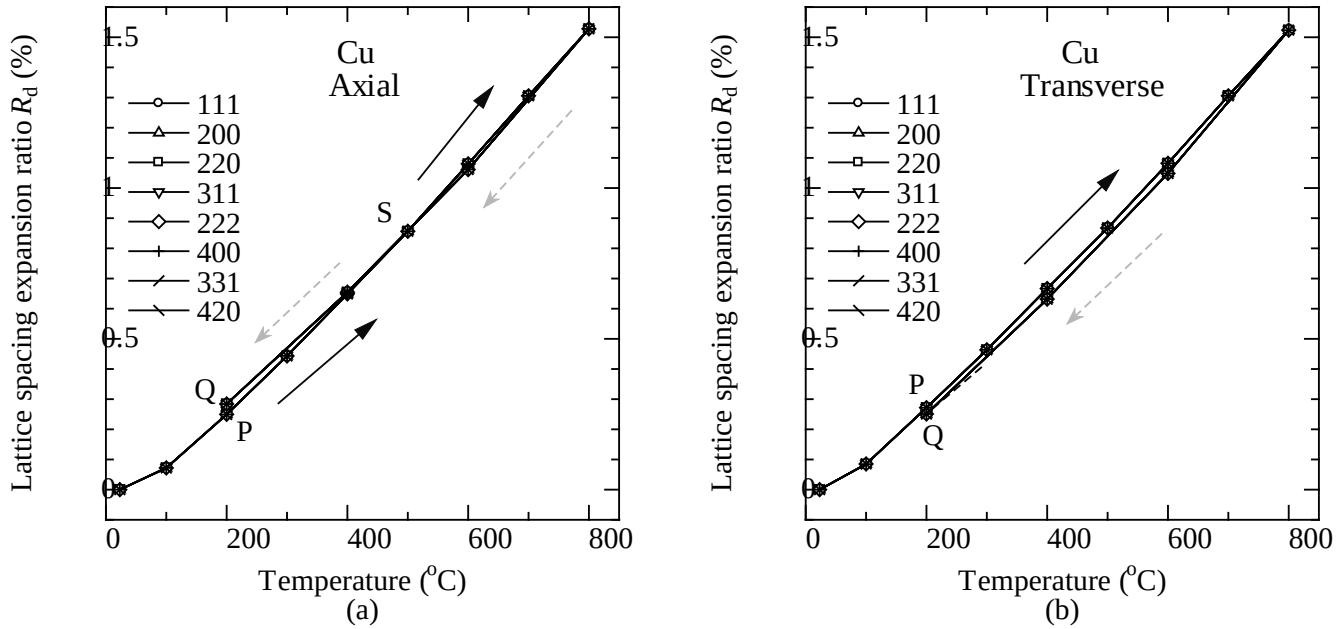

Fig. 5. Temperature dependence of the lattice spacing expansion ratio R_d in the thermal cycle process for Cu plating in (a) axial and (b) transverse directions. The point S for the axial direction in 500 °C shows the intersection in the thermal cycle process. All the errors of the lattice spacing expansion ratio R_d were less than 0.003%.

In the transverse direction shown in Fig. 4 (b), the hysteresis behavior shows smaller decreasing of the lattice spacing expansion ratio R_d in comparison with that in the axial direction. The result indicates that the delay of the interaction between the Nb_3Al filaments and the other materials is not strong in the transverse direction. This is due to the imperative stresses of action and reaction with small delay in the materials at the cross-sectional surface. By the thermal cycle, the lattice spacing expansion ratio R_d is decreased by 0.02% at 200 °C in the transverse direction. Considering the electrical magnetic force in magnets, a larger tensile residual strain in the transverse direction is better for improving the proof stress. Therefore, it is beneficial that the lattice spacing expansion ratio R_d has a small decrease.

The published version of this preprint appeared in IEEE Transactions on Applied Superconductivity 23, 6000704 (June 2013).

C. *Thermal cycle effect on the Cu plating*

Further observation was focused on the Cu plating. The temperature dependences of the lattice spacing expansion ratios R_d for the Cu plating in the axial and the transverse directions are shown in Fig. 5 (a) and (b), respectively. Again, eight peaks having large the lattice spacing *d* are observed. All the peaks show a similar behavior in each direction of the wire, as also seen in the Nb_3Al filaments.

In the axial direction shown in Fig. 5 (a), the hysteresis behavior is shown with weak irreversibility. The lattice spacing expansion R_d in the temperature increasing and decreasing processes intersect at 500 °C between the behaviors. After the intersection in the temperature decreasing process, the lattice spacing expansion ratio R_d is slightly increased. This increasing is considered to be induced by the additional burden acting on the matrix with the decreasing of the lattice spacing expansion ratio R_d in the Nb_3Al filaments.

In the transverse direction shown in Fig. 5 (b), the hysteresis also exhibits weak irreversibility. No intersection occurs in the thermal cycle process, and the lattice spacing expansion ratio R_d in the temperature decreasing process is slightly smaller than that in the temperature increasing process. This may be due to the small change of the lattice spacing expansion ratio R_d in the Nb_3Al filaments.

IV. Conclusion

Neutron diffraction measurements for RHQ-Nb_3Al wire with a Nb/Ta matrix were carried out between room-temperature and 800 °C after the A15 phase transformation. By the one thermal cycle, the lattice spacing expansion ratio R_d for Nb_3Al in the axial direction was decreased by approximately 0.1%. The mechanical performance of the wire may be improved by reducing the lattice spacing expansion ratio R_d in the Nb_3Al filaments with the thermal cycle method in the axial direction. The interaction of the residual stress between the Nb_3Al filaments and the Cu plating through the Nb/Ta matrix was verified.

Acknowledgment

This research has been partially supported by a Grant-in-Aid for Scientific Research (B) (20340065), Grant-in-Aid for Scientific Research on Innovative Areas (23104003), and CERN-KEK collaboration program for development of high field superconducting accelerator magnets.

References

[1] T. Takeuchi, K. Tsuchiya, K. Nakagawa, S. Nimori, N. Banno, Y. Iijima, A. Kikuchi, and T. Nakamoto, "A New RHQT Nb_3Al Superconducting Wire with a Ta/Cu/Ta Three-layer Filament-barrier Structure," *Supercond. Sci. Technol.*, vol. 25, pp. 065016, 2012.

[2] A. Kikuchi, R. Yamada, K. Tsuchiya, T. Nakamoto, E. Barzi, D. Turrioni, K. Sasaki, Q. Xu, A. Terashima, H. Takigawa, A. Yamamoto, K. Nakagawa, T. Takeuchi, M. Lamm, and A. V. Zlobin, "Strand and Cable Development for a High Field Nb_3Al Common Coil Magnet," *IEEE Trans. Appl. Supercond*., vol. 20, pp. 1428-1431, 2010.

[3] T. Takeuchi, K. Tsuchiya, M. Saeda, N. Banno, A. Kikuchi, and Y. Iijima, "Electron Backscatter Diffraction Analysis of Nb_3Al Multifilamentary Strands Prepared by Rapid Heating, Quenching and Transformation Annealing," *Supercond. Sci. Technol.*, vol. 23, pp. 125001, 2010.

[4] X. Jin, H. Oguro, T. Nakamoto, S. Awaji, A. Yamamoto, K. Tsuchiya, T. Ogitsu, A. Kikuchi, T. Takeuchi, S. Harjo, and T. Ito, "Tensile Strain Dependence of Critical Current of RHQ-Nb_3Al Wires," *Cryogenics*, in press.

[5] X. Jin, T. Nakamoto, S. Harjo, T. Ito, T. Ogitsu, K. Tsuchiya, A. Yamamoto, A. Kikuchi, T. Takeuchi, and T. Hemmi, "Observation of A15 Phase Transformation in RHQ-Nb_3Al Wire by Neutron Diffraction at High-temperature," *J. Alloys Compds.*, vol. 535, pp. 124-128, 2012.

[6] S. Harjo, T. Ito, K. Aizawa, H. Arima, J. Abe, A. Moriai, T. Iwahashi, and T. Kamiyama, "Current Status of Engineering Materials Diffractometer at J-PARC," *Mater. Sci. Forum*, vol. 681, pp. 443-448, 2011.

[7] X. Jin, T. Nakamoto, T. Ito, S. Harjo, A. Kikuchi, T. Takeuchi, K. Tsuchiya, T. Hemmi, T. Ogitsu, and A. Yamamoto, "Residual Strain Dependence on Matrix Structure in RHQ-Nb_3Al Wires by Neutron Diffraction Measurement," *Supercond. Sci. Technol*., vol. 25, pp. 065021, 2012.

[8] S. Awaji, H. Oguro, G. Nishijima, K. Watanabe, S. Harjo, T. Kamiyama, and K. Katagiri, "Prebending effects in bronze route Nb_3Sn wires," *Supercond. Sci. Technol*., vol. 18, pp. S313-S318, 2005.