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Trial Manufacturing of Jelly-Rolled Nb/Al Monofilamentary Wire with Very Small Diameter below 50 microns

Akihiro Kikuchi[1], Yasuo Iijima[1], Ataru Ichinose[2], Masatoshi Kawano[3], Masaru Yamamoto[3], Motoyoshi Kimura[3], Jun Nagamatsu[4], Masato Otsubo[4], Kazuto Hirata[1], Shigeki Nimori[1] and Kiyosumi Tsuchiya[5]

[1] National Institute for Materials Science, Tsukuba, Ibaraki 305-0047, Japan.

[2] Central Research Institute of Electric Power Industry, Yokosuka, Kanagawa 240-0196, Japan.

[3] Meiko Futaba Co., Ltd., Chuo, Yamanashi 409-3842, Japan.

[4] Yuki Precision Co., Ltd., Chigasaki, Kanagawa 253-0084, Japan.

[5] High Energy Accelerator Research Organization, Tsukuba, Ibaraki 305-0801, Japan.

E-mail: KIKUCHI.Akihiro@nims.go.jp

Abstract. Jelly-rolled Nb/Al composite monofilamentary wires having an outer diameter of 50 microns and more than 100 meters in length were successfully fabricated. Moreover, some of these thin wires were additional drawn to 30 microns in outer diameter in this study. We recognized again that jelly-rolled Nb/Al composite monofilamentary wires have excellent cold workability and drawability. The transmission electron microscopic observation revealed that the niobium and aluminum laminations retained a nano-scaled fiber composite structure in the longitudinal direction like the microarchitecture of Japanese bamboo, even in the very thin wire with an outer diameter of 30 μm. The thickness of the aluminum sheet is approximately 70 nm, which is almost comparable to the value calculated from the reduction ratio. The critical temperature of 16.2 K (on set) and 15.7 K (off set) were obtained after the diffusion reaction at 850 ^{o}C for 10 h. The transport critical currents in liquid helium (4.2 K) under external magnetic fields of 1 T and 8 T are 7.2 A and 0.7 A, corresponding to non-Cu critical current densities of 15,000 and 1,500 A/mm^2, respectively. These values are greater than those of industrial Nb-Ti and MgB_2 commercial multifilamentary wires.

Introduction

In general, practical metallic superconducting wires, such as Nb-Ti, Nb_3Sn and others, have a fine multifilamentary configuration. The predominant reasons are: a) a reduction of electromagnetic losses, b) improvement of intrinsic and thermal stabilities, and c) reduction of mechanical strain [1]. In particular for the A15 compounds, the promotion of chemical reaction through shortening of the diffusion distance should be added to the above list. According to these understandings, we have been developing the Nb_3Al monolith wires having multifilament configuration. However, several R&D issues, such as the wire breakage of multifilamentary precursors, costly Cu stabilizer fabrication and low mechanical irreversible strain, still remain [2]. On the other hand, we are considering a different approach to fabricate Nb_3Al superconducting wires which would have low electromagnetic losses, good intrinsic and thermal stabilities, and excellent flexibility for winding through the React & Wind method. The anticipated fabrication cost would be lower. The conductor would consist of a multiple very fine single strand wires braided into a cylindrical pattern. An example of such a conductor braid geometry is known as the "BNL Braid" [3]. In this paper, as the first step for our new approach for Nb_3Al conductors, we manufactured Jelly-Rolled Nb/Al monofilamentary precursor wires of diameter below 50 μm. The drawing workability, microstructure changes, and superconducting properties after heat treatment of very fine Nb_3Al monofilamentary wires were investigated.

1. Jelly-rolled Nb/Al laminated billet assembly and hydrostatic extrusion

In 2017, some jelly-roll (JR) production facilities, such as special metal fixtures for manual jelly rolling, a precision sheet cutting machine, and an automatic tightening and spot welding machine, have been moved from SH Copper Products (now Hitachi Metals Neomaterial, Ltd.) to NIMS. Therefore, at present, jelly-rolled Nb/Al laminated billets is being fabricated in-house at NIMS. Figure 1 is a picture of the jelly-rolled billet assembly room established in 2017 at NIMS. In addition, Figures 2, 3 and 4 are pictures for precision sheet cutting, manual jelly rolling and automatic JR tightening and spot welding, respectively.

Figure 1. Photograph of the jelly-rolled billet assembly room established in 2017 at NIMS.

Figure 2. Photograph of precision sheet cutting machine.

Figure 3. Photograph of manual jelly rolling.

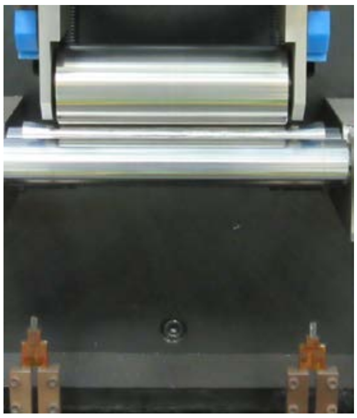

Figure 4. Photograph of automatic JR tightening and spot welding machine.

A pure niobium sheet of nominal dimensions 0.1 mm in thickness and 150 mm wide, and a pure aluminum sheet of 0.3 mm thickness and 150 mm width, were used as starting materials for the jelly-rolled Nb/Al laminated billet. Those sheets were tightly overlapped several tens of turn together around a pure tantalum rod with a diameter of 2.0 mm and 150 mm in length. Note that the final five turns were overlapped by only the niobium sheet, which would act as a diffusion barrier. Then, this Nb/Al laminated composite was inserted into an annealed OFC tube with tip OFC and rear stainless steel (SUS) plugs. The outer and inner diameters of the OFC tube are 12.3 mm and 10.1 mm, respectively. These jelly-rolled Nb/Al laminated billets were extruded at room temperature by using a vertical hydrostatic extruder having 100 tons of maximum load. The hydrostatic extrusion was performed at Hitachi Metals Neomaterial, Ltd. The liquid medium used for applying hydrostatic pressure at room temperature was castor oil. The diameter of the billet decreased from 12.3 mm to 5.0 mm, which corresponds to an extrusion ratio of 6.05. Figure 5 are transverse cross-section images of the jelly-rolled Nb/Al laminated composites taken by an optical microscope (Nikon: ECLIPSE-LV150), before and after the hydrostatic extrusion. These cross-sections keep similar configurations, and thus the hydrostatic extrusion at room temperature resulted in a normal smooth metal flow. The Cu/non-Cu ratio of the present billet is 0.5.

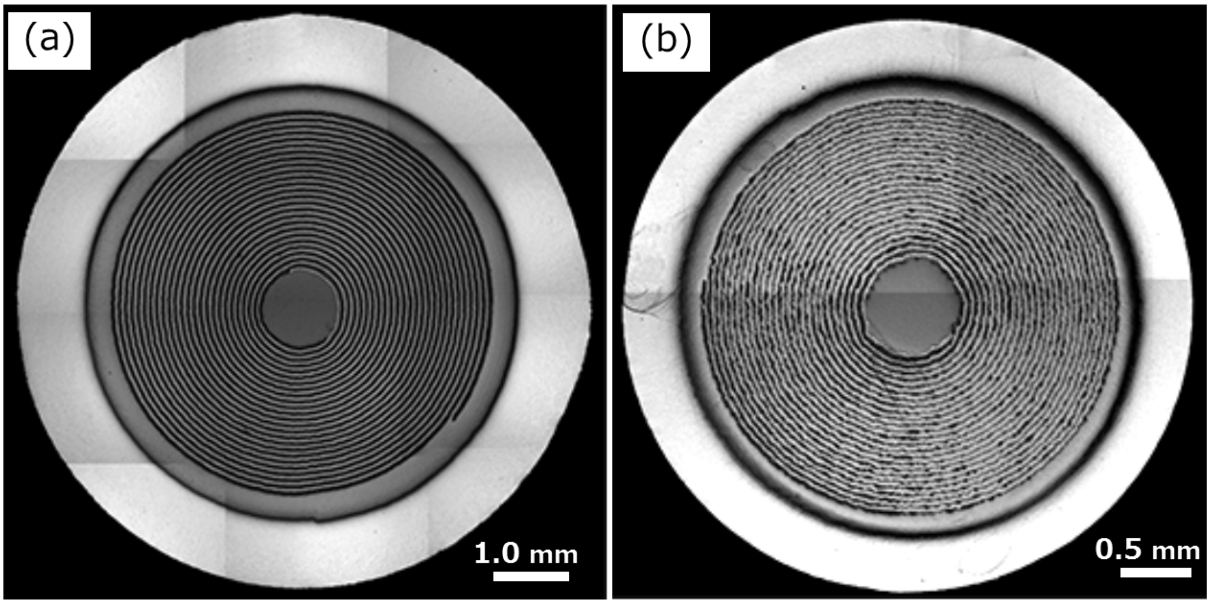

Figure 5. Comparison of transverse cross-sections of the jelly-rolled Nb/Al laminated composites (a) before and (b) after the hydrostatic extrusion at room temperature. The outer diameters of (a) and (b) are 12.3 mm and 5.0 mm, respectively.

2. Cold die drawing of jelly-rolled Nb/Al laminated precursors

After the hydrostatic extrusion at room temperature, we have tried an area reduction of the jelly-rolled Nb/Al laminated monofilamentary precursor only using cold die drawing without multifilament bundling. Intermediate annealing was not performed. In NIMS, the outer diameter was drawn to 0.56 mm from 5.0 mm by using a single-head drawing machine with general hard steel metal dies and a lubricant of a rapeseed oil. An area reduction ratio between dies was consistently about 11 % and the drawing speed was approximately 5.0 m/min. No wire breakages happened at this stage. Then, 0.56 mm wires were taken to Meiko Futaba Co., Ltd. for drawing down the wire diameter from 0.56 mm to 50 microns using a wet-type continuous drawing machine with diamond dies. These are mass-production facilities for pure electro-Cu wires. The area reduction ratio between dies was 10-20 %

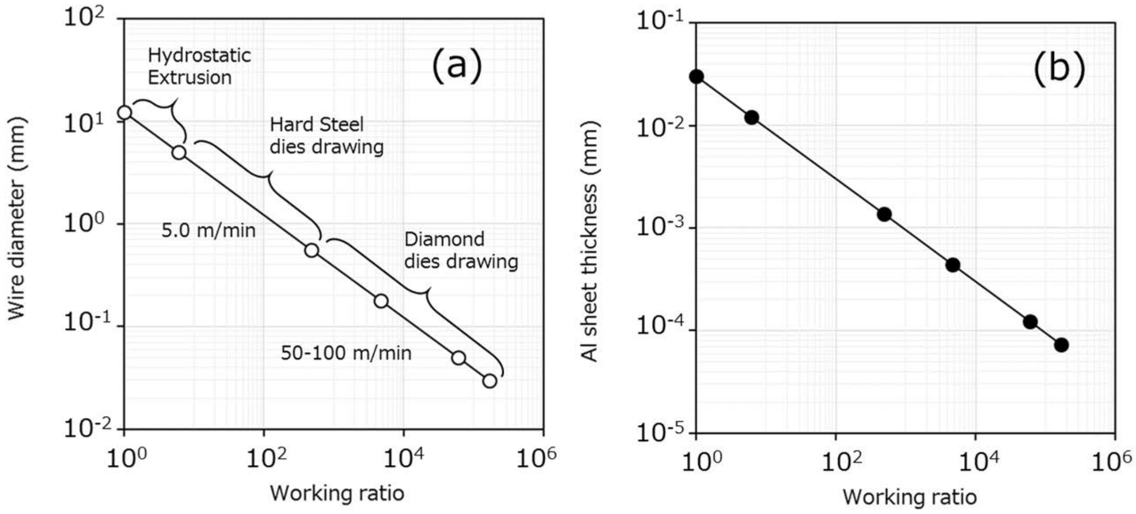

Figure 6. (a) Jelly-rolled wire diameter versus working ratio, and (b) The aluminum sheet thickness in the jelly-rolled Nb/Al composite versus working ratio.

and the drawing speed was 50-100 m/min. No wire breakages happened down to a diameter of 0.18 mm, but several wire breakages happened between 0.18 mm and 0.05 mm. Eventually a jelly-rolled Nb/Al laminated monofiamentary wire with a diameter of 50 μm and 128 meters in length was successfully fabricated. The superconducting filament diameter without Cu is below 50 microns. A part of the 50 micron wire could be demonstrated additionally to draw down to 30 μm in outer diameter. Figure 6 is a calculated scheme of the cold work in this study: (a) shows the relationship between the wire diameter and the working ratio and (b) shows the relationship between the aluminum sheet thickness and the working ratio.

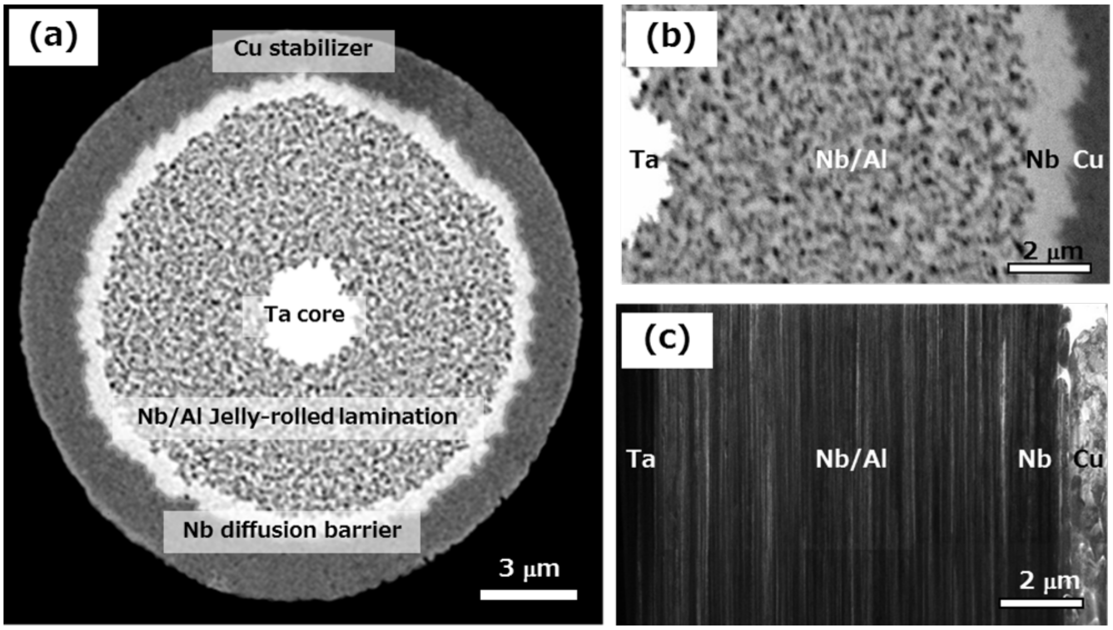

Figure 7. (a) Whole transverse cross-section image taken by SEM on the jelly-rolled Nb/Al laminated composites wire with a diameter of 30 μm. (b) Enlarged transverse cross-section image of (a). (c) Longitudinal cross section image taken by STEM of (a).

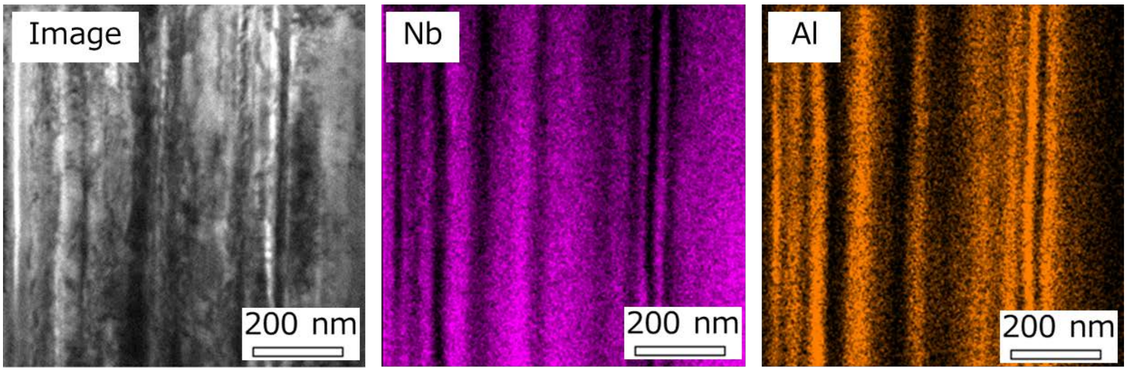

Figure 8. (a) Enlarged longitudinal cross-section image taken by TEM on the jelly-rolled Nb/Al laminated composite wire 30 μm in diameter. (b) EDX element mapping of niobium and (c) EDX element mapping of aluminum.

Figure 7 are microstructure images of the cross-sections of the jelly-rolled Nb/Al laminated composites wire with 30 μm in diameter. Figure 7 (a) and (b) show back-scattered images of a whole and an enlarged vertical cross-sections taken by a scanning electron microscope (Hitachi; TM5050). Figure 7 (c) is a scanning transmission electron microscope (JEOL; JEM-2100F) image of the longitudinal cross-section. Focused ion beam milling (FIB) was used for the scanning transmission electron microscopy (STEM) sample preparation. According to the vertical cross-section image of Figure 7 (a) and (b), the Nb/Al laminated structure seems to be almost broken and to mechanically mix together well comparison to the cross-section just after extrusion. But, Nb/Al lamination retains a nano-scaled fiber composite structure in the longitudinal direction as shown in Figure 7 (c). These fiber architectures are expected to maintain a flexibility and tenacity like Japanese bamboo, thus the very thin wire could be drawn even to below 30 μm in diameter. Figure 8 show an enlarged TEM image in the longitudinal direction of the 30 micron wire and the element mappings of niobium and aluminum by energy dispersive X-ray Spectroscopy (EDX). The thickness of the aluminum fibers is approximately 50-70 nm, which is comparable to the value calculated from the reduction ratio as shown in Figure 6 (b).

3. Critical temperature, critical current and non-Cu critical current density

Figure 9 shows the resistivity versus temperature curves for the 30 micron and 50 micron wires. This measurement was performed by a DC four probe method with 1 mA of constant current. The distance between the voltage taps is 10 mm. Measurement samples were set at an appropriate stable temperature position from the surface of liquid helium and the temperature was controlled by small coil heater with a ramping rate of 4.0 K/h. Both wire samples were diffusion-reacted at 850 ^{o}C for 10 h in an electric tube furnace with a high dynamic vacuum atmosphere of 10^{-4} Pa. The critical temperature, T_c of the 30 micron wire was slightly higher than that of the 50 micron wire. Its onset value is 16.2 K and offset value is 15.7 K. These value are lower than that (17.6 K) of the rapid heating/quenching and transforming (RHQT) processed Nb_3Al multifilamentary wires [2]. The normal state resistivity, ρ_n just before a superconducting transition of the 30 micron wire was larger than that of the 50 micron wire.

Figure 10 shows the transport critical current, I_c as a function of external magnetic field in liquid helium (4.2 K). The closed circles and the open circles are results for the 50 micron and 30 micron wires, respectively. The heat treatment conditions of the wire samples are the same as for Figure 8, i. e. 800 ^{o}C for 10 h. The transport I_c (4.2 K) of the 50 micron wire was about two times larger than that of the 30 micron wires, and it exceeded 10 A at 4.2 K and 1 T. However, the vertical cross-sectional area of the 50 micron wire is 2.8 times larger than that of the 30 micron wire, therefore, the non-Cu critical current density of the 30 micron wire becomes larger than that of the 50 micron wire.

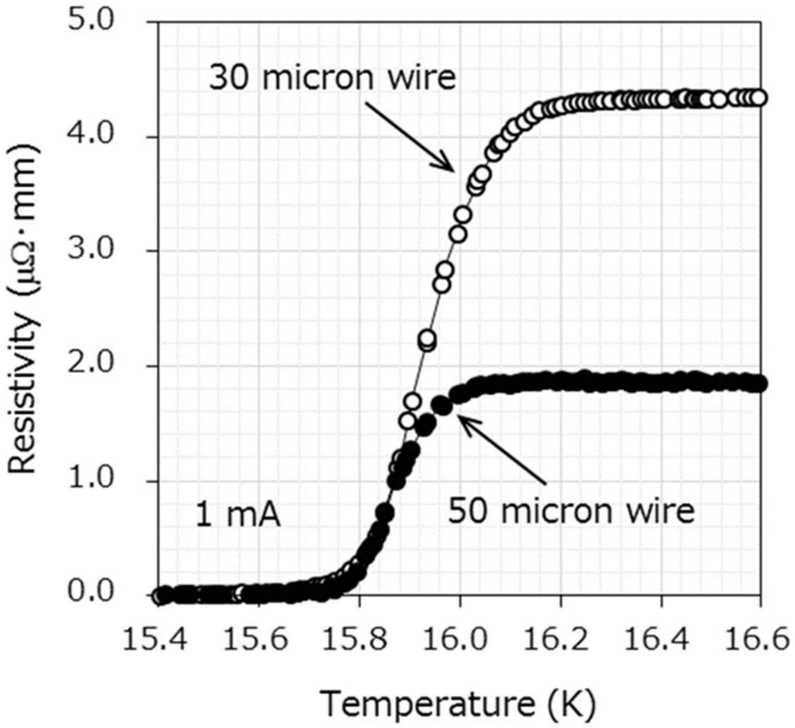

Figure 9. The resistivity versus temperature curves for 30 micron wire (open circles) and 50 micron wire (closed circles), respectively.

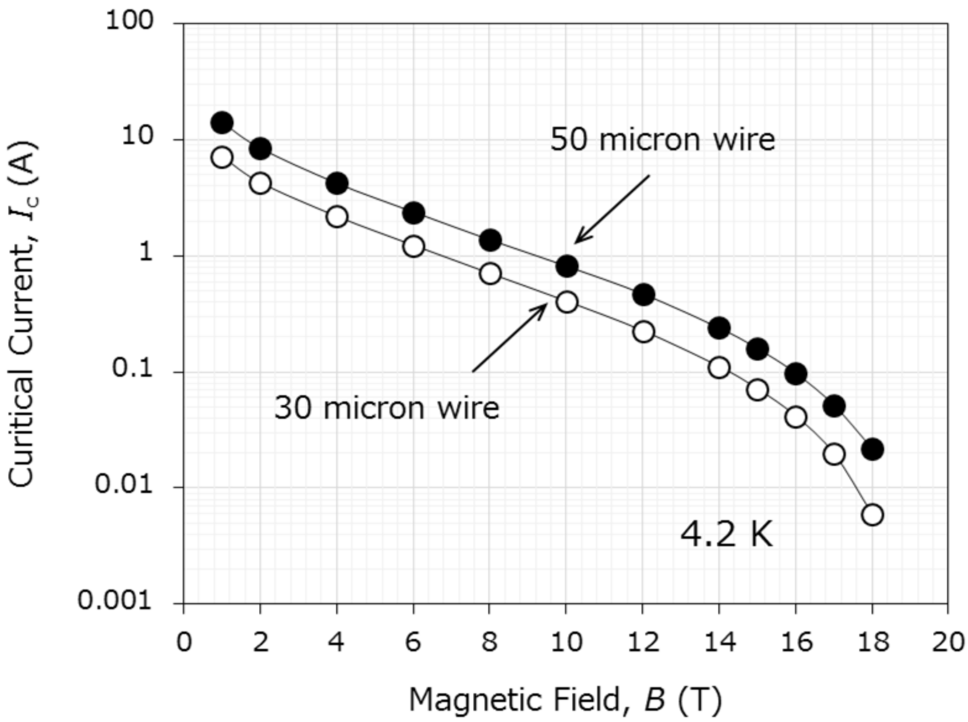

Figure 10. The transport critical current versus external magnetic field curves for 30 micron Nb_3Al wire (open circles) and 50 micron Nb_3Al wire (closed circles), respectively.

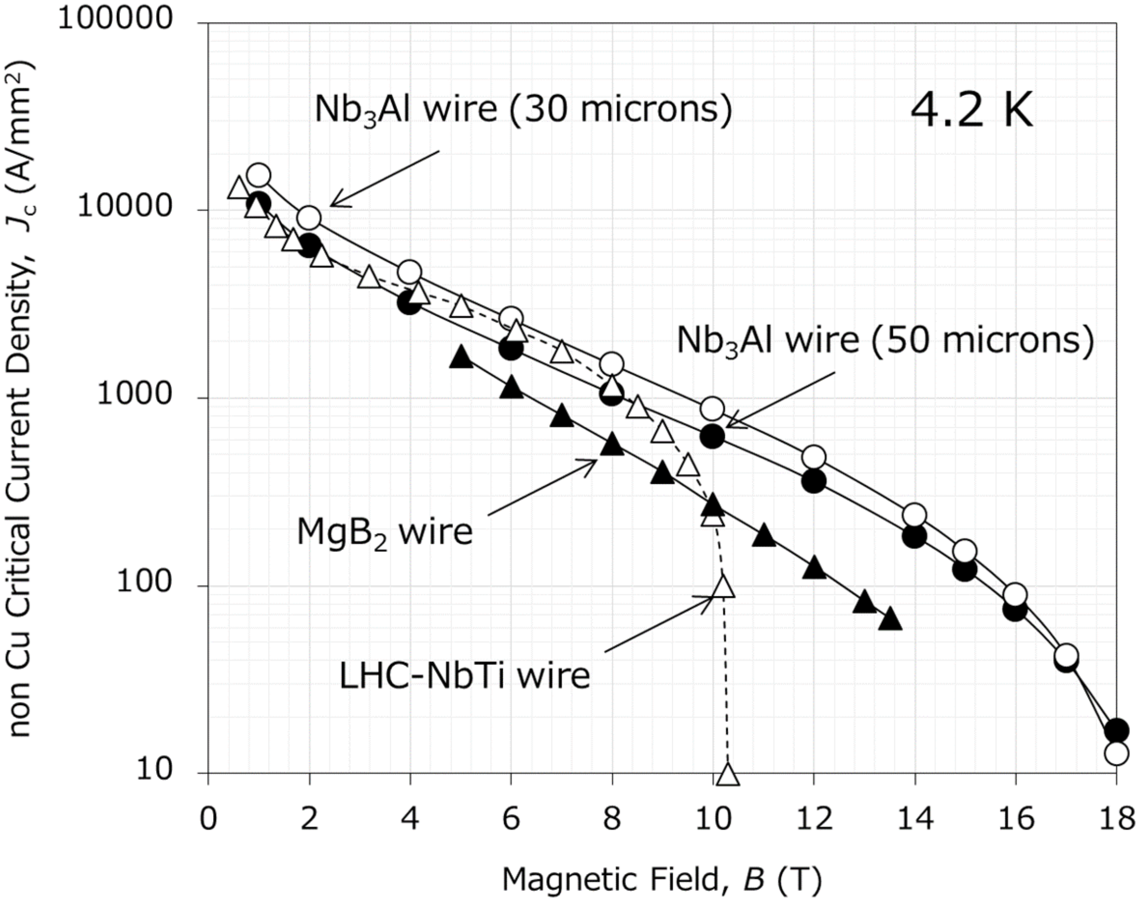

Figure 11. Comparison of the non-Cu critical current density as a function of external magnetic field for (a) 30 micron Nb_3Al wire, (b) 50 micron Nb_3Al wire, (c) industrial Nb-Ti wire used for the LHC [4] and (d) MgB_2 wires produced by Hyper Tech Research in the USA [5].

The diffusion distance between niobium and aluminum becomes smaller on decreasing the wire diameter, and thus the synthesis of A15-Nb_3Al phases may improve in the 30 micron wire. Figure 11 is the comparison of the non-Cu critical current density as a function of external magnet field for the present Nb_3Al wires in comparison to commercial Nb-Ti and MgB_2 multifilamentary wires. The non-Cu J_c of the present 30 micron Nb_3Al wire is exceeding that of the Nb-Ti wire and is much larger than that of MgB_2 wire. The heat treatment condition has not been optimized yet, and it can be expected to much improve the J_c in near future. Besides, the RHQT process [2] should be of interest for obtaining good superconducting properties as well.

4. Conclusions

In this study, we have recognized that jelly-rolled Nb/Al composite monofiamentary wires have excellent cold workability and drawability, and very thin Nb_3Al wires have been demonstrated. The reduction of the wire diameter is very effective to reduce the bending moment. In addition, it is well known that Nb_3Al wires show excellent strain tolerance, therefore, very thin Nb_3Al wires may keep the flexibility without performance degradations after heat treatment. This is promising for wire braiding and cabling, as well as for a coil application through the React & Wind method. Because the heat treatment condition for Nb_3Al thin wires has not been optimized yet, the present critical current density is not so large in comparison to industrial Nb_3Sn wires. In addition, in order to increase the transport current capacity, a braid conductor using several hundreds of Nb_3Al fine monofiamentary wires will be demonstrated in near future. Furthermore, it will be needed to study the relationship between a braiding compaction factor and the critical current density, in order to achieve comparable the engineering J_c (J_e) to conventional monolith wire with multifilamentary architectures.

5. References

[1] M. N. Wilson 1983 *Superconducting Magnets* (Oxford: Oxford University Press) p. 289
[2] A. Kikuchi 2012 *J. Cryo. Super. Soc. Jpn.* **47** 503
[3] A. D. Mcinturff 1979 *Treatise on Materials Science and Technology* vol 14, ed. Thomas Luhman and David Dew-Hughes (London: Academic Press) p. 132
[4] T. Boutboul, S. Le Naour, D. Leroy, L. Oberli and V. Previtali 2006 *IEEE Transactions on Applied Supercond.* **16** 1184
[5] G. Z. Li, M. D. Sumption, J. B. Zwayer, M. A. Susner, M. A. Rindfleisch, C. J. Thong, M. J. Tomsic, and E. W. Collings 2013 *Supercond. Sci. Technol.* **26** 095007

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