Development of Self-bonding NbTi Wires for Superconducting Magnets

Yonghoon Kim, Takumi Sato*, Hiroyuki Fukushima*, Hideki Ii*, Hirokazu Tsubouchi*, Kiyoshige Hirose**

ABSTRACT We have developed the self-bonding NbTi wires for superconducting magnets, which can eliminate an epoxy impregnation process which requires some time and cost in the superconducting coil manufacturing, and we have evaluated its bonding strength and the characteristics of the superconducting coil. Consequently, we confirmed the optimum heat treatment condition to obtain a sufficient bonding strength and found out that the properties are stable without degradation of the bonding strength after the heat cycle treatment between room temperature and ultralow temperature. As the current test result on a sample of small-sized superconducting coil made of self-bonding NbTi wires, we found that it had the characteristic equivalent of an epoxy impregnated coil with no characteristic degradations were caused by heat cycle or quench occurred and, in a 4.5 T backup magnetic field, a 100% load factor and an 8.0 T maximum experienced magnetic field could be achieved. A medium-sized superconducting coil had no characteristic degradations after heat cycle or quench and 100% load factor was achieved under the current test, therefore we could confirm the usefulness of the self-bonding superconducting wire.

1. INTRODUCTION

Major countries have declared, one after another, to become a carbon-neutral country and it generated overwhelming green actions on a global scale. On a relevant note, the numbers of Large Scale Integration (LSI) and power Integrated Circuits (IC) used in the electronic devices and electric/hybrid automobiles, which contribute to the carbon-neutral, are increasing rapidly and causing the serious shortage of semiconductor supply. In response to the above, the manufacturers for the semiconductor manufacturing equipment and materials are undertaking to increase their production capacity, and the manufacturers of silicon wafers, the semiconductors' substrate, are considering building new plants. The diameter of silicon wafers has been increasing from 200 mm to 300 mm in order to improve the efficiency of semiconductor manufacturing. In the Magnetic Field Applied Czochralski (MCZ) method, which is one of the manufacturing methods, a magnetic field is applied to the silicon melt by a superconducting coil during the pulling process of single-crystal ingots to achieve both large diameter and homogenization. The superconducting coil which plays an important role in that process is made of an insulated superconducting wire that is winded and then impregnated with an epoxy resin to restrain the movement of the superconducting wire. Therefore, the movement of the wire due to the electromagnetic force is suppressed while the coil is energized, and the occurrence of quenches, the switch from the superconducting state to the normal-conductive state, is prevented. However, the epoxy resin impregnation process requires many steps, including special vacuum impregnation to minimize voids in the resin for ensuring the necessary adhesive strength, and the epoxy resin waste generated in the manufacturing process is also an issue.

Therefore, we developed the self-bonding resin coated NbTi wires for superconducting magnets, which can eliminate an epoxy impregnation process. As the result, we concentrate our efforts to simplify the fabrication process of silicon wafer manufacturing equipment and to contribute to a smooth supply of the semiconductors. The self-bonding NbTi wires for superconducting magnets have an outer layer of adhesive resin on the insulation layer as shown in Figure 1 so that it has a special function that the wires are bonded to each other only by heat treatment after winding. That makes great advantages so that the superconducting coil manufacturing process can be simplified drastically and no epoxy resin waste is generated. This time, we have developed the self-bonding resin which has the sufficient adhesive strength with a heat treatment and made a small-sized superconducting coil using the self-bonding NbTi wires, which is coated with that resin. We confirmed that the coil has a 100% performance without superconducting characteristic degradations and consolidated the element technology applied to practical superconducting coils. Here we are reporting the results.

* Incubator Department, R&D Division

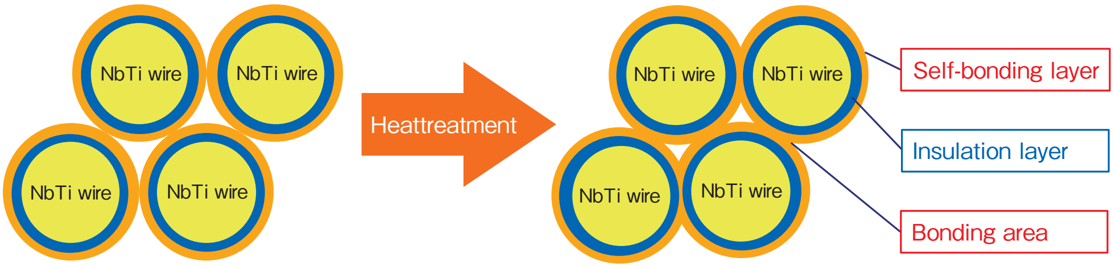


Figure 1 Schematic diagram of a self-bonding NbTi superconducting wire.

2. DEVELOPMENT OF SELF-BONDING NbTi WIRES

The coating materials and dimensional data for the self-bonding NbTi wire are shown in Table 1. We have developed two types of the electric wires which use a Polyvinyl Formal (PVF) as the general insulation coating for the superconducting wires and also the Polyamide Imide (PAI) as the high-heat resistant resin. Here we report mainly the self-bonding wire with the PAI insulation. For the bonding resin, we selected the material which can be bonded at a low temperature less than 200°C. Moreover, we investigated modification of the material to achieve a better bonding property and high mechanical performances, so that we could developed the phenoxy based resin composition which met the necessary propreties. We set the thickness of the insulation layer and the self-bonding layer, as shown in Table 1, to 0.03 mm and 0.04 mm respectively.

Table 1 Specifications for self-bonding wire products.

Item	Unit	Developed product (Self-bonding wire)
Insulation coating material	—	Polyvinyl formal (PVF) or Polyamide imide (PAI)
Self-bonding material	—	Phenoxy based resin composition
Conductor diameter	mm	0.98
Insulation layer thickness	mm	0.03
Self-bonding layer thickness	mm	0.04
Outer diameter	mm	1.12

In the manufacturing processes of the product, it has a coating process of the self-bonding resin added to the insulation resin coating process, therefore the thermal history on the superconducting wire increases. To confirm the influences on the superconducting properties caused by the addition of the coating process, critical current Ic (note: limiting current value which can be applied) at 4.1 K, 10^{-13} Ωm before/after the self-bonding coating is compared in the magnetic field region of 4 - 8 T, and it was confirmed that there is almost no variation in the value as shown in Figure 2 and sufficient superconducting property was obtained for the self-bonding superconducting wire.

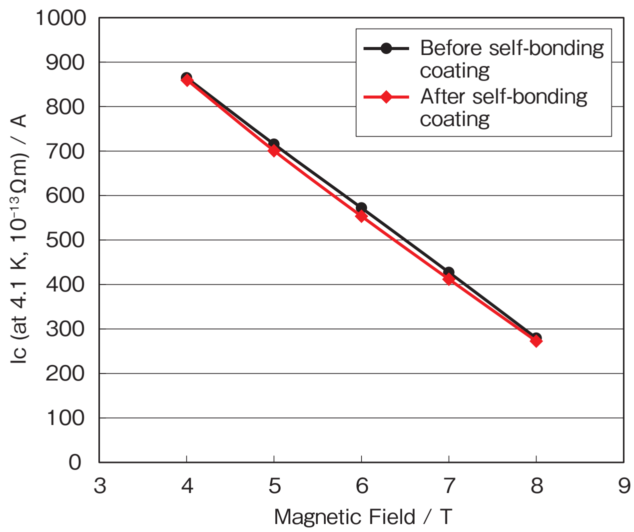


Figure 2 Critical current Ic (at 4.1 K, 10^{-13} Ωm) for superconducting NbTi wires.

3. CHARACTERISTICS OF SELF-BONDING STRENGTH

3.1 Self-Bonding Strength Evaluation by a Helical Coil

We have manufactured a sample (length = 3 cm approx.) in compliance with the helical coil method of Japanese Industrial Standards (JIS 3216-3), using the self-bonding NbTi wire. We set the sample in the forced-blasting-air-type oven and changed the setting temperature (130 - 195°C: surface temperature of the sample) and exposure time (0.5 - 16 hours) to investigate the values of the sample's bonding strength at each condition. The bonding strength was evaluated by perpendicular tensile testing (speed = 20 mm/min) of the helical coil with a tension tester. The photos of the helical coil sample and the bonding strength measurement by the tensile testing are shown in Figure 3.

Figure 4 shows the effect of the heating temperature of 130 - 195°C on the self-bonding strength. The self-bonding strengths for the specific thermal treatment times (0.5 hour, 1 hour, and 2 hours) had a tendency to increase according to an increase in the thermal treatment time. It was confirmed that the self-bonding strength of the sample exposed for 2 hours becomes 6.2 N at 130°C and up to 9.4 N at 195°C. It can be considered that the cause for this strength increase by the increase of thermal treatment temperature is related to a temperature dependency of the melt viscosity of the self-bonding material. The flow of resin happens easily caused by the decrease of its melt viscosity at the higher temperature, therefore bonding interfacial area between wires becomes wider and the bonding strength increases. It is also considered that the bonding strength increase according to lengthening of the thermal treatment from 0.5 hour to 2 hours is related to the increment of interfacial area between wires.

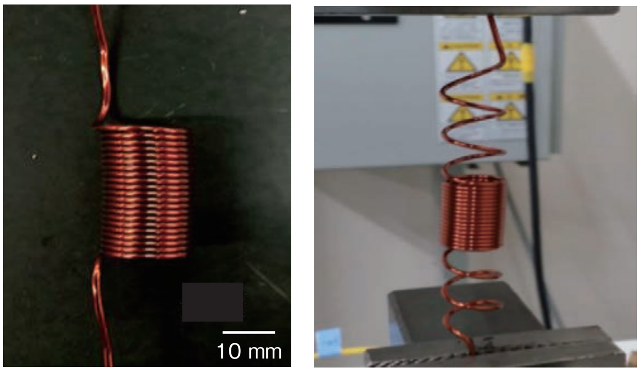


Figure 3 Photos of a helical coil sample and the bonding strength measurement by tensile testing of a coil.

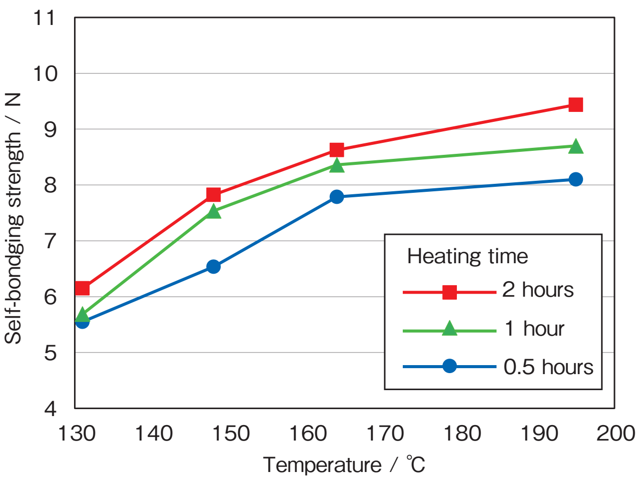


Figure 4 Effect of the heating temperature change on the self-bonding strength.

The helical coil sample for this bonding strength test can have a thermal treatment at around 200°C, however, considering an actual coil in the practical use, various materials other than a superconducting wire are used, therefore the thermal treatment condition at lower temperature is favorable. We selected 148°C as a low temperature condition and 195°C as a high temperature condition, had a long-hour thermal treatment up to 16 hours, and examined the effect of thermal treatment hours on the bonding strength. From the result, shown in Figure 5, it is confirmed that the bonding strength is higher, for up to 2 hours, with the sample at 195°C thermal treatment but for more than 4 hours, with the sample at 148°C thermal treatment. In the thermal treatment under the condition of 195°C for more than 4 hours, we assumed that thermal degradation of the resin layer by long-hour thermal treatment at the high temperature in the atmosphere is related as the cause of the self-bonding strength decrease. Meanwhile, under the thermal condition of 148°C, the strength can be stable at a high value at a treatment of more than 4hours, and the value of 11.6 N can be obtained with the sample at 16 hour-treatment. From the above result, considering the heat resistance of the material and the thermal distribution in the coil, a long-hour thermal treatment at the low temperature is considered more effective for practical coils.

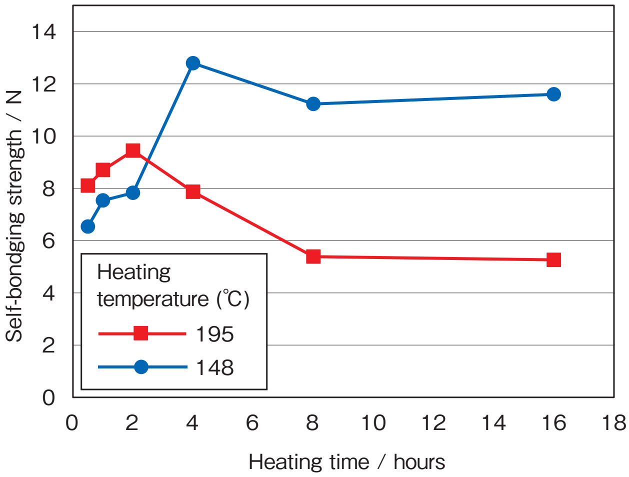


Figure 5 Effect of heating time change on the self-bonding strength.

3.2 Heat Cycle Test

As a reliability test of the phenoxy material for a self-bonding superconducting wire application, we had an external observation and a self-bonding strength test on the helical coil after the heat cycle treatment. The purpose of this test is to confirm the variation in the bonding strength which might occur because of cracks in a bonding layer during the heat cycle. We selected the low temperature condition as the bonding condition of the coil, and had a heat treatment on the coil at 150°C for 8 hours. Subsequently the sample at the room temperature is pre-cooled in liquid nitrogen (77K) for 5 min and then soaked in liquid helium (4K) for 30 min and back to room temperature. This cooling process from room temperature to 4 K in the above was repeated for 5 times, 10 times, and 15 times continuously. After that, we checked abnormalities in the external appearance and then measured the self-bonding strength by the perpendicular tensile testing (speed = 20 mm/min) same as the one mentioned in Section 3.1 above.

Figure 6 shows the test results of the external observation and the self-bonding strength on the helical coil after heat cycle. The sample presented almost no degradation in bonding strength with 15 times heat cycles and no abnormality of its external appearance was observed.

From the above results, we concluded that the self-bonding NbTi wire for a superconducting coil can have a stable self-bonding strength without any influence of the temperature variation from room temperature to 4 K.

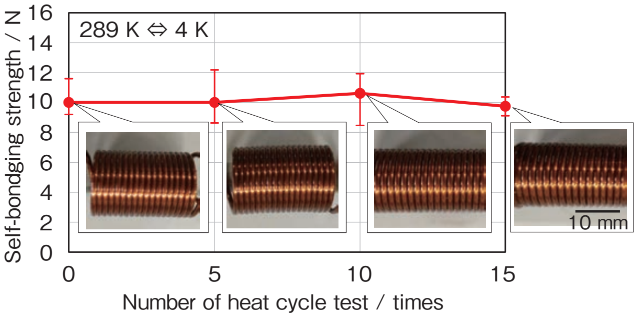


Figure 6 Photos and the self-bonding strength for the heat cycle treated coils.

4. CHARACTERISTIC EVALUATION FOR THE SELF-BONDING COIL

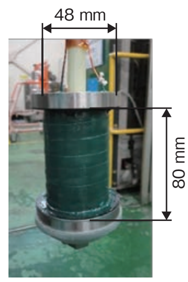
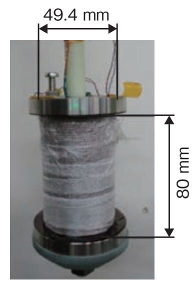
4.1 Manufacturing of a Small Coil

For verification purpose of the applicability of a self-bonding NbTi wire to the superconducting coil, we manufactured a small coil made of the self-bonding NbTi wire and examined the current-carrying properties at the coil condition. Moreover, we manufactured an epoxy impregnated coil for comparison. And current characteristics for each coil were evaluated by applying magnetic field and current. We describe the small coil manufacturing in the following.

The specifications and pictures of the small coils are shown in Table 2. The size of the coil is a 25 mm inner diameter, an approx. 50 mm outer diameter, and an approx. 80 mm height and the maximum electromagnetic force applied to this coil is 32 MPa at the inner most layer of the coil's center. We used SUS304 for the bobbin and polyimide sheet (thickness = 125 μm) for insulation between the bobbin and the superconducting wire.

We manufactured an epoxy impregnated coil for comparison. A PVF coated wire (ϕ1.05) without self-bonding coating was used, and Nitofix SK-229 was used as an impregnation epoxy resin. After coil winding, it was cured at room temperature for 72 hours.

Table 2 Specifications and photos for small coils.

	Epoxy impregnation	Self-bonding
Coil configuration	Solenoid	←
Coil inner diameter (mm)	25.6	←
Coil outer diameter (mm)	48	49.4
Coil height (mm)	80	←
Total turn frequency	816	846
Number of layers	12	←
Thermal treatment condition	25℃ × 3 days	220℃ × 90 min
Overview		

4.2 Current Test Results of the Small Coil

The current test was done by using 8 T superconducting magnet and a cryostat. The small coil was precooled in liquid nitrogen and then soaked in liquid helium and subjected to the current test. To confirm the effect of the heat cycle, the coil was set in room temperature temporarily after the current test and cooled again and reconducted the current test. DC electric current of 300 A and 500 A were used as the current and excitation speed was set at 50 A/min.

The following characteristics were confirmed at the current test on the small self-bonding coil (Figure 7).

(1) In 4.5 T outer magnetic field (8.0 T maximum exposure of the magnetic field on the superconducting wire), after quench occurred at the currents of 191 A and 219 A, the coil current reached up to 288 A and 100% load factor (note: ratio of the quench magnetic field, where the magnetic field is 100% when Ic flows, the indicator of the mechanical stability of a coil) was obtained.

(2) When the coil is set at room temperature temporarily and then cooled and tested again, it obtains a 500 A current without an outer magnetic field, and no characteristic degradation is observed.

Meanwhile, for the epoxy impregnated coil, we obtained the following results (Figure 8).

(1) After quench occurred at the first excitation, we set the coil at room temperature and tested again. The coil current and magnetic field reached 500 A and 6.3 T respectively and it is confirmed that no effect of quench and heat cycle occurred.

(2) Same as for the self-bonding coil, in 4.5 T outer magnetic field, the quench occurred when the current was 279 A, the Ic limit of the wire, and 100% load factor is achieved.

From the above results, the self-bonding coil had no characteristic degradation caused by heat cycle and quench and obtained 100% load factor, therefore we found out that it has the characteristics at same level as the epoxy impregnated coil.

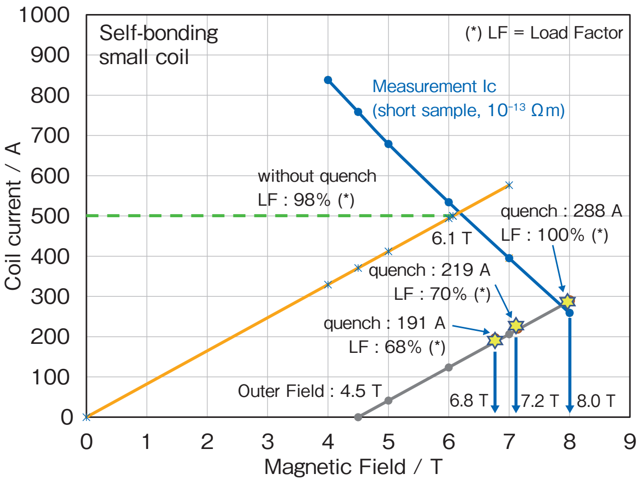


Figure 7 **Results of electrical current tests for a self-bonding small coil.**

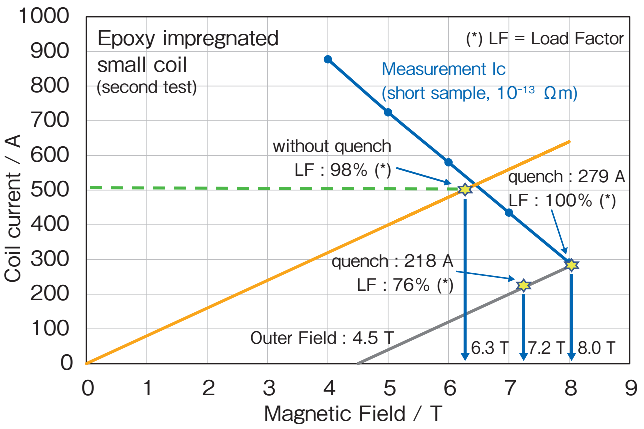
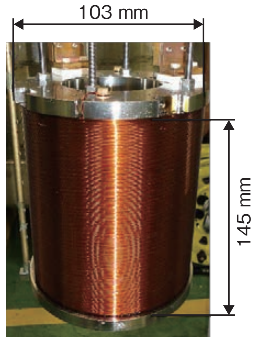


Figure 8 **Results of electrical current tests for an epoxy impregnated small coil.**

4.3 Manufacturing of a Medium Coil

Following the current-carrying property test of the small coil, we prepared a self-bonding coil which had a larger coil diameter to have a larger electromagnetic force, and had the same current test procedure. The size and picture of the manufactured coil are shown in Table 3. The maximum electromagnetic force applied to this coil is 114 MPa at the innermost layer of the coil's center. We used SUS304 for the bobbin and polyimide sheet, same as for the small coil, for insulation between the bobbin and superconducting wire.

Table 3 **Specifications and a photo for a self-bonding medium coil.**

	Sell-bonding
Coil configuration	Solenoid
Coil inner diameter (mm)	100.3
Coil outer diameter (mm)	139.7
Coil height (mm)	145.5
Total turn frequency	2580
Number of layers	20
Thermal treatment condition	220℃ × 90 min
Overview	

4.4 Current Test Results of the Medium Coil

The current test was carried out as same as for the small coil and following results were confirmed (Figure 9).

(1) The quench occurred at the current of 380 A for the first current-carrying (load factor: 96%) and the coil obtained 391 A, Ic of the wire material, and 100% load factor for the second current-carrying.

(2) After the coil was in room temperature and cooled again and carried current, no quench occurred and the coil current achieved 391 A same as in the previous test.

For the medium coil, it achieved 100% load factor after one training quench at the high load factor, and kept the previous current-carrying history even with having an experience of temperature rise, therefore we concluded that the self-bonding NbTi wire can be applicable for superconducting coils.

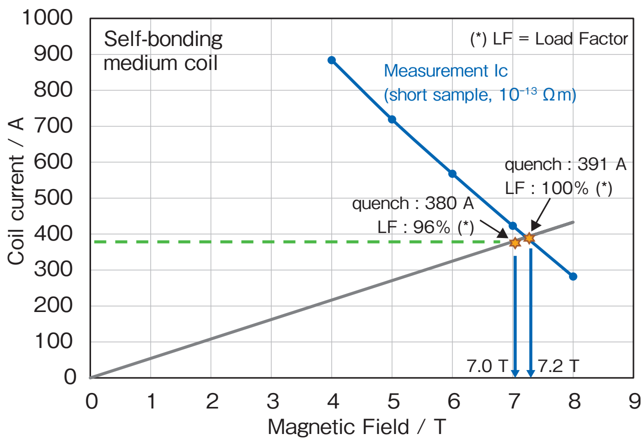


Figure 9 **Results of electrical current tests for a self-bonding medium coil.**

5. CONCLUSION

We have developed the self-bonding NbTi wires for superconducting magnets, which can eliminate an epoxy impregnation process which requires some time and cost while the superconducting coil manufacturing, and evaluated its bonding strength and the characteristics of the superconducting coil. The following results are obtained.

(1) We investigated the effect of the heat treatment temperature and time with respect to the bonding strength and confirmed the optimum conditions to obtain the sufficient bonding strength. At the heat cycle test which was conducted as the reliability test, it was found that the properties were stable without degradation of the bonding strength.

(2) From the result of current test on the small coil manufactured by using the self-bonding wire, there was no performance degradation caused by the heat cycle and quench, 100% load factor and 8.0 T maximum experienced magnetic field were achieved in 4.5 T backup magnetic field.

(3) For the test of the critical current performance of medium coil, there was no performance degradation after the heat cycle and quench and 100% load factor was achieved. For applications in the practical superconducting coil, the adequacy of the self-bonding wire was confirmed.

Since we confirmed the satisfactory performances with the small and medium coils, we will attempt the application of the self-bonding NbTi wires in lager superconducting magnets in pursue of its practical use.