Evaluation of NbTi Superconducting Joints for 400MHz NMR Magnet

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***Abstract*—Persistent current NbTi superconducting joint were fabricated based on the cold-pressing welding method for 400MHz NMR magnet. The electrical properties of the joints were tested using the current decay measurement method. To simulate the actual coil assembly, a nine-joint series loop was made and tested. Test results show that the total resistance of the nine persistent joints made based on cold-pressing welding method is 3×10^{-14} Ω at 120 A under 1T background magnetic field, which meets the requirements of the 400 MHz NMR magnet. We have found that the induced current in the joint loop decays obeying three decay patterns, which is relevant to the flux creep and can be explained with *n*-value losses in the joint loop. Relaxation of magnetization in persistent current joint loops was also observed under 1T background magnetic field.**

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I. INTRODUCTION

A superconducting magnet with the center field of 9.4 T required for a 400 MHz superconducting nuclear magnetic resonance (NMR) spectrometer has been designed and assembled in IEE, CAS [1]. The main parameters for the 400 MHz NMR magnet are shown in Table I. The magnet is immersed in 4K liquid helium, and a two-stage pulse tube refrigerator (PTR) is employed to cool the magnet system to reduce the helium refill.

The magnet system consists of 17 superconducting coils and 9 superconducting joints in the circuit loop, and adjacent coils are connected by superconducting joints, as shown in

Figure 1. The 400 MHz NMR superconducting magnet works in persistent current mode, which is of key importance for the NMR magnet system to keep the stability of magnetic field with respect to time [2, 3]. The persistence of a high magnetic field NMR mainly depends on the resistive loss within the circuit. The resistance within the circuit composes of two parts, the resistance of the superconducting wires and the resistance of the superconducting joints. The superconducting wire resistance is generated due to the flux-creep in the superconducting wire, the resistive loss of which, also called the *n*-value losses, can be calculated with $V=V_c(I/I_c)^n$. The resistance of the superconducting wire can be expressed as

$$R_{wire}=\frac{V_c}{I_c}\cdot\left(\frac{I}{I_c}\right)^{n-1} \qquad (1)$$

where V_c is the voltage drop on the entire superconducting wire in a coil, $V_c = v_c l$, typically, v_c is 0.1 μV/cm for low temperature superconductor, l is the length of the superconducting wire used in the coil. I_c is the critical current of the superconducting wire, I is the transport current in the superconducting wire, and n is the *n*-value of the superconducting wire, usually larger than 50 for low temperature superconductors. NbTi wires with different specifications are employed to wind the coils. The NbTi wires are representative 400 MHz NMR conductors supplied by Oxford Superconductivity Technology (OST). The load curve for each coil and superconducting wire is shown in Figure 2. The total wire resistance of the magnet system calculated based on (1) is 1.66×10^{-16} ohm.

TABLE I

PARAMETERS FOR 400 MHz NMR MAGNET

Magnetic field	9.4 T
Operating current	67.18 A
Inductance	92.2 H
Stability of magnetic field	1 Hz/hr
Stored energy	0.21 MJ
Available bore	54 mm
5 G line in axial	1.5 m
5 G line in radius	0.85 m
R_{DSV}	10 mm
Homogeneity	0.2 ppm in DSV
Operating temperature	4.2 K
NbTi/Cu-NbTi/Cu joints	7
NbTi/CuNi-NbTi/Cu joints	2

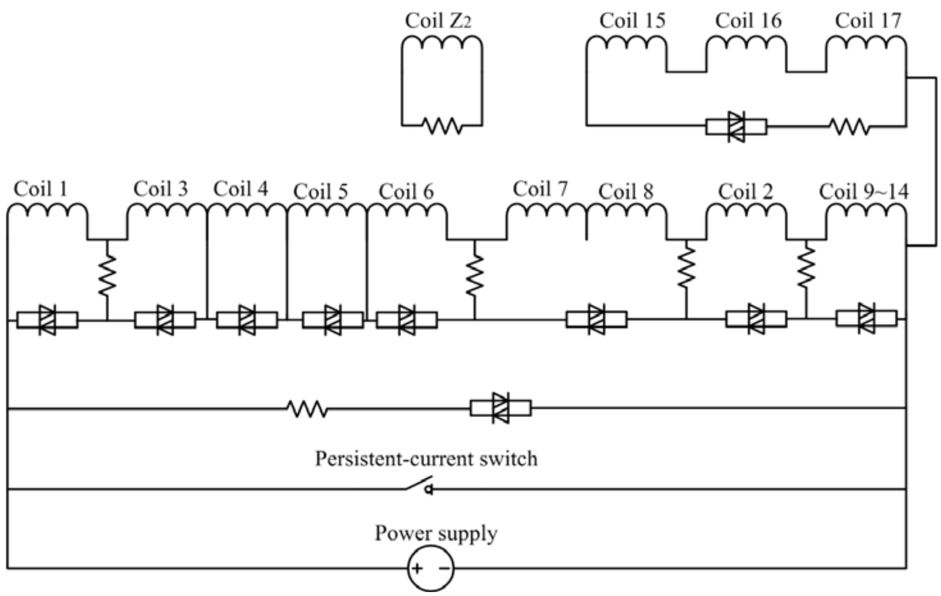

Fig.1. Circuit of the magnet system for the 400 MHz NMR spectrometer.

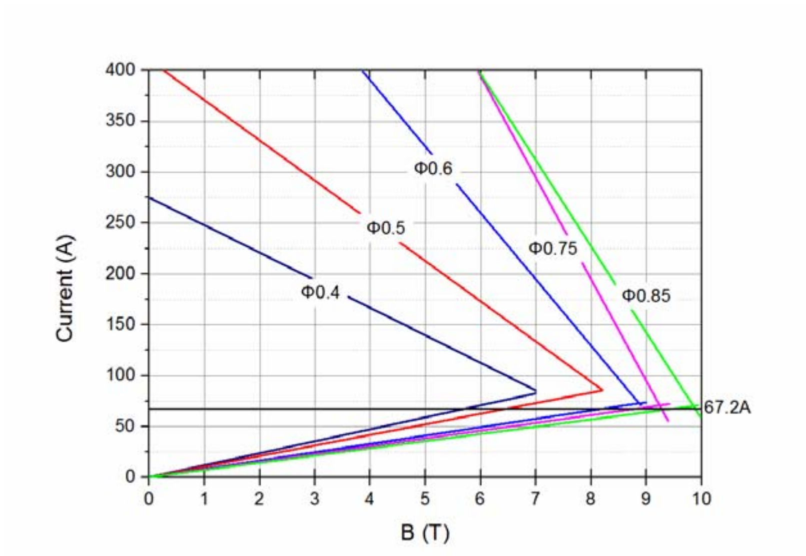

Fig.2. Load curve for each coil and superconducting wire.

The resistance of the superconducting joint is a sum of all joint resistance, *i.e.*,

$$R_{joint} = \sum_{i=1}^{N_{joint}} R_{joint}^{(i)} \tag{2}$$

For NMR or MRI (Magnetic Resonance Imaging) applications, the resonant frequency of a nucleus under a background magnetic field is given by

$$f = \frac{\gamma}{2\pi} \cdot B \tag{3}$$

where B is the magnetic induction intensity of the background field, γ is the gyromagnetic ratio of the nucleus. Then we have $f \sim B \sim I$, where I is the operating current of the magnet which generates the background magnetic field.

For a circuit composing of only resistor and inductor, the current in the circuit decays as follows:

$$I(t) = I_0 e^{-\frac{t}{\tau}} \tag{4}$$

where I_0 is the initial current in the circuit, τ is the time constant of this circuit, and is given by L/R, and where L is the inductance of the inductor, R is the resistance of the resistor. From (4) we can obtain

$$R = L \cdot \frac{\ln\left[I_0/I(t)\right]}{t} = L \cdot \frac{\ln\left[f_0/f(t)\right]}{t} \tag{5}$$

The requirement of the magnetic field stability is 1 Hz/hr, the tolerable maximum resistance of this magnet calculated based on (5) is 6.4×10^{-11} ohm. The wire resistance can be neglected compared to the joint resistance, which means that the tolerable maximum resistance for each joint is 7.11×10^{-12} ohm. Hence the requirements for each superconducting joints is to have a resistance of less than 7.11×10^{-12} ohm at a transport current larger than 67.2 amperes

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II. EXPERIMENTAL APPARATUS

The resistance of a superconducting joint in a coil assembly can be measured by the four-probe measurement method, which gives a measurement limit of 1×10^{-11} ohm [4]. The current decay measurement method can give higher measurement sensitivity if the measuring time is longer, but this method can only be used for single joint in persistent mode. If two or more joints are in series, this measurement method cannot give the detailed resistance of each joint but the total resistance of all the joints.

A measurement device is fabricated to measure the resistance of superconducting joints based on the current decay method [5], shown in Figure 3. A non-persistent 3 T solenoid magnet provides the background field during the test. The magnet power supply is a group of 120 A CRYOGENIC master-slave magnet power supply system. A current transformer for exciting the joint loop is also fabricated. An 80 A CRYOGENIC power supply is employed to charge the current transformer. A Lakeshore low temperature Hall sensor HGCA-3020 is used to detect the magnetic field generated by the joint loop. Voltage taps across the Hall sensor is connected to a Keithley model 2000 Multimeter, which then transfers the voltage data to a PC based system using LabView software through an IEEE-488 bus.

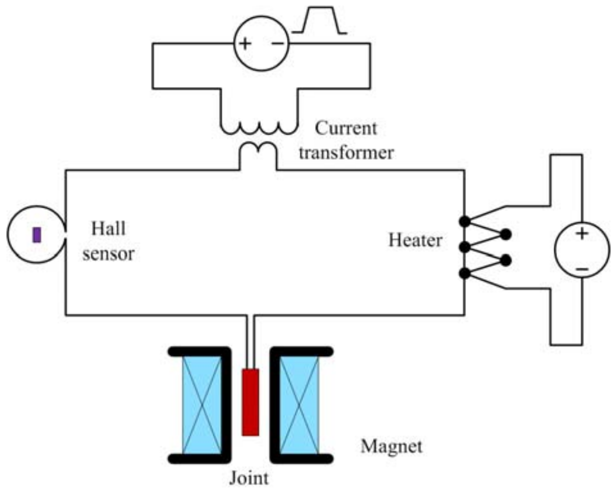

Fig.3. Joint resistance measurement system.

The test process is as follows:

(1) Excite the joint loop through the current transformer to a high current level;
(2) Quench the joint loop with a heater;
(3) Reduce the current of the excitation power supply to zero and then turn off, an induced current will be in the joint loop;
(4) Energize the background magnet to 1T;
(5) Collect the voltage data of the Hall sensor for further analysis.

It will take almost half an hour for the measurement device to release the thermal stress after bathing in liquid helium. Then the thermal drift will be stable and easy to offset. The test process should start after the thermal effect is over. To obtain a higher accuracy, the obtained voltage data from the Keithley 2000 Multimeter will be compensated according to the error plot of Hall generator provided by Lake Shore Cryotronics, Inc.

III. MEASUREMENT UNCERTAINTY

The joint loop is at first mounted on the measurement device. Then the measurement device is slowly inserted the liquid helium cryostat. After stabilizing for thirty minutes, the joint loop is energized. The excitation current increases at 0.2 A/s till the joint loop quenches. This step may repeat for many times to find the critical current of this joint. This principle here is that the induced current in the joint loop after excitation should be as high as possible. The observed scatter after stabilization is about 1 μV, which will result in an uncertainty of 2.3 A current for the measured current in the joint loop. Then we estimate the uncertainty in the measured resistance. For the current decay method, the resistance can be obtained from equation (6) [5],

$$R_j = L \cdot \frac{\ln\left[I_2(t_2)/I_1(t_1)\right]}{t_2 - t_1} \tag{6}$$

where L is the inductance of the joint loop, $I_1(t_1)$ and $I_2(t_2)$ are the currents in the joint loop at t_1 and t_2, respectively.

Assume the current in the joint loop decay very slowly, which is true in the thermal assisted flux flow process, and then the conservative estimate of joint resistance uncertainty from equation (6) is

$$\Delta R_j = L \cdot \frac{\ln\left[(1+\Delta I/I)/(1-\Delta I/I)\right]}{t_2 - t_1} \tag{7}$$

where ΔI is the current uncertainty due to the voltage scatter of the Keithley 2000 Multimeter, I is the induced current in the joint loop.

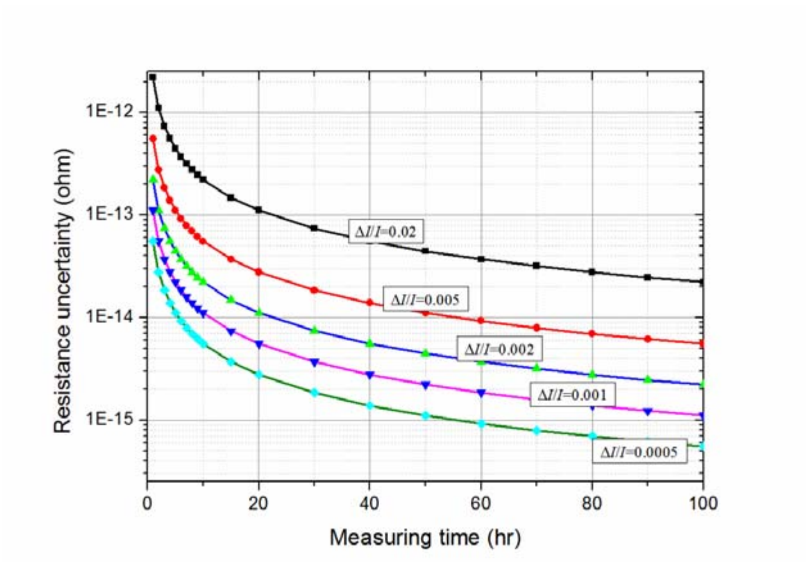

Fig.4. Resistance uncertainty *vs*. measuring time.

Figure 4 gives the relationship between resistance uncertainty and measuring time. The figure shows that the resistance uncertainty can be greatly decreased by increasing the measuring time, or/and by reducing the value of $\Delta I/I$, i.e. increasing the ampere turns of the coil where the Hall sensor is placed. To obtain a higher accuracy, reducing the value of $\Delta I/I$ is more rational than increasing the measuring time. In practical application, combining these two methods is an appropriate choice. The reasonable measuring sensitivity of the current decay measurement technique is 10^{-13} ohm~10^{-15} ohm.

The stability level of the background field is mainly dependent on the stability level of the background magnet power supply, the stability of which is 0.0015%. Besides, the background magnetic field is perpendicular to the joint loop, hence the measurement error due to the uncertainty of the background magnetic field is much less than the uncertainty of the Keithley Multimeter, thus can be neglected. The excitation power supply will be turned off when testing the joints, so there will be no EMF for the joint loop. Furthermore, the joint loop had been immersed in the liquid helium for at least half an hour to release the thermoelectric potential, the measurement error due to thermal effects can thus be neglected.

IV. FABRICATION OF PERSISTENT JOINTS

Persistent current superconducting joints between multifilamentary NbTi conductors can be fabricated based on many methods, such as diffusion bonding [4], soldering [6], spot welding [7], ultrasonic welding [8], solder matrix replacement [4, 9] and cold-pressing welding [10]. The cold-pressing welding technique is employed to fabricate the persistent current superconducting joints for 400 MHz NMR magnet for its convenience and reliability. The NbTi wires used to make these joints was the same with the superconducting wires used in the 400 MHz NMR magnet. The cold-pressing welding technique includes the flowing process:

(1) Remove stabilizer by nitric acid solution,
(2) Rinsing the filaments by fluoric acid and pure water,
(3) Cleaning the filaments by pure water and ethanol,
(4) Drying the filaments in open air with air blower,
(5) Braiding both filaments and installing them in CuNb tube,
(6) Pressing the CuNb tube with mechanical pressure in open air.

The 400 MHz NMR magnet contains nine superconducting joints, the specifications of which are shown in Table II.

TABLE II

SPECIFICATION ON THE JOINTS

Type	Specification	Quantity
NbTi/CTu-NbTi/Cu	Φ0.60-Φ0.40	1
	Φ0.40-Φ0.40	2
	Φ0.40-Φ0.50	1
	Φ0.50-Φ0.60	1
	Φ0.60-Φ0.75	1
	Φ0.75-Φ0.85	1
NbTi/CuNi-NbTi/Cu	Φ0.50CuNi-Φ0.85	1
	Φ0.50CuNi-Φ0.60	1
Total		9

V. MEASUREMENT RESULTS AND DISCUSSION

A. Joint Resistance

We measured the electrical properties of superconducting joint loops with different specifications, two specimens for each specification, and Table III presents the measurement results for these joints. For $\Delta I/I$ is 6×10^{-4}~1×10^{-4} and the measuring time is about ten hours, the measurement uncertainty can be obtained from formula (7), around 6.67×10^{-15}ohm~1.11×10^{-14}ohm

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TABLE III

MEASUREMENT RESULTS

Joint	R(ohm)@1T	I(A)
Φ0.40-Φ0.40	$<1\times10^{-13}$	196
Φ0.40-Φ0.50	$<1\times10^{-13}$	165
Φ0.50-Φ0.60	$<1\times10^{-13}$	258
Φ0.60-Φ0.60	$<1\times10^{-13}$	240
Φ0.75-Φ0.60	$<1\times10^{-13}$	340
Φ0.75-Φ0.85	$<1\times10^{-13}$	468
Φ0.50CuNi-Φ0.85	$<1\times10^{-13}$	248
Φ0.50CuNi-Φ0.60	$<1\times10^{-13}$	175

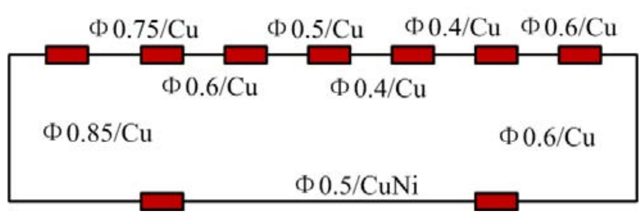

Fig.5. The joint series loop for 400MHz NMR magnet.

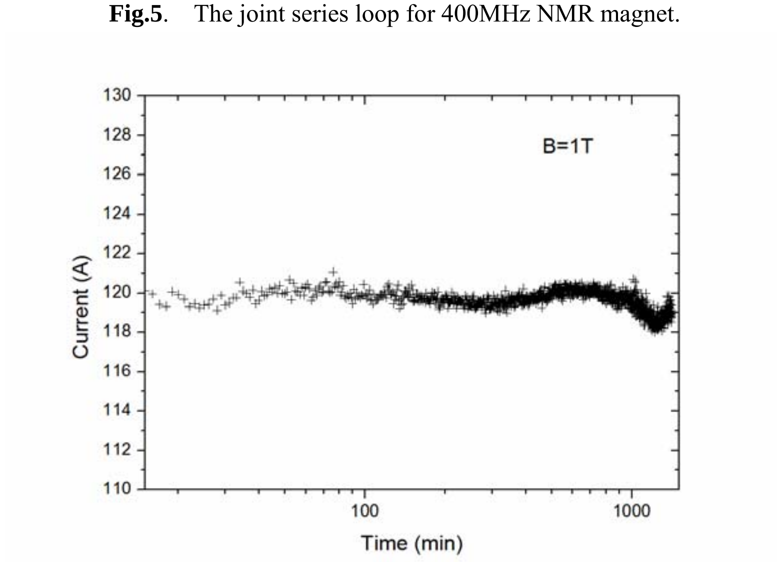

Fig.6. Current variation in the joint series loop against time.

To obtain the accurate resistance of all these superconducting joints used in the 400 MHz magnet system, all the joints with the same specification as in the 400 MHz were made in series to form a joint loop, as shown in Figure 5. The joint series loop was tested for more than twenty-four hour to obtain a high accuracy. Figure 6 shows the decay curve of the induced current in the joint series loop. The total resistance of the nine joints is 3×10^{-14} ohm at 120 A under 1T background magnetic field. The resistance of these entire joints is much less than the design requirement, which is 6.4×10^{-11} ohm, and the stable current in the joint series loop is also much higher than the operating current of the 400 MHz NMR magnet, which is 67.2 A under background magnetic field in 0.3 T.

B. Current Decay Pattern

We found a phenomenon that there is a sharp decrease of the induced current in the joint loop under 0 T background magnetic field just after the excitation process, as shown in Fig.7. This phenomenon was considered as flux creep process in the superconducting joint loop [11, 12]. This flux creep happened in the persistent joint rather than in the superconducting wire, since the induced current is far smaller than the critical current of the superconducting wire, which can be seen in Figure 2. The time-dependent current would decay as the following expression [12],

$$I\left(t\right)=\alpha-\beta\ln t \tag{8}$$

where α and β are constants.

Actually, the above equation is valid for a specific time range, such as $0< t\leq t_l$, $t_l <t\leq t_m$ or $t>t_m$, α and β are not constants for the entire measuring time, as shown in Figure 7.

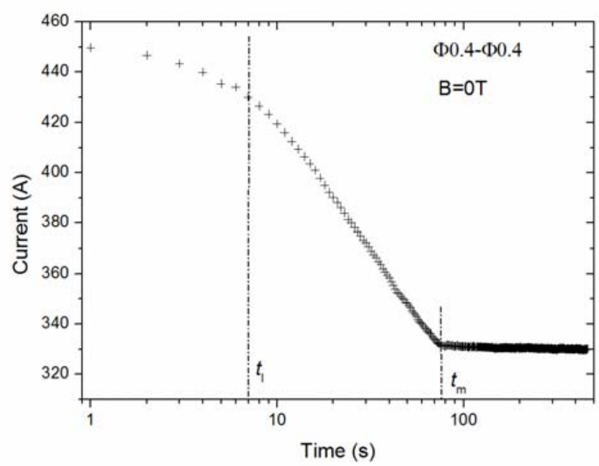

Fig.7. Logarithmic plot of current variation in the joint loop against time.

The obvious different current decay patterns at $t<t_m$ and at $t>t_m$ shows that the induced current in the joint loop decays due to different reasons. When $t<t_m$, the current decays due to the flux creep, also called thermally activated flux creep, since the current in the joint loop is

very close to its critical current, the current decreases sharply. When t=t_m, the flux creep process ends, then the current decays very slowly, which is caused due to the thermally assisted flux flow [13]. During the thermally assisted flux flow process, the resistivity of the persistent joint is constant, the current in the joint loop decays logarithmically. Fig.7 shows that the induced current in the joint loop decays following three current decay patterns,

$$I(t)=\begin{cases}\alpha_1-\beta_1\ln t & ,t<t_l\\ \alpha_2-\beta_2\ln t & ,t_l<t<t_m\\ \alpha_3-\beta_3\ln t & ,t>t_m\end{cases} \quad (9)$$

where α_1, β_1, α_2, β_2, α_3 and β_3 are constant dependent on specific persistent joints.

The transition of the induced current at t=t_m can also be explained by the n-value losses applied to superconducting wire. The voltage-current transition curve of the superconducting wire from the superconducting state into the normal state can be expressed by a power law:

$$E=E_c\left(\frac{I}{I_c}\right)^n \quad (10)$$

From formula (10) we can get the following formula,

$$\rho=\rho_c\left(\frac{I}{I_c}\right)^{n-1} \quad (11)$$

Formula (11) shows that the resistivity of the superconducting wire is not a constant, but dependent upon the operating current in the persistent joint loop. For NbTi superconducting wire, the index n>50, which means the resistivity of the superconducting wire will increase sharply when the induced current I is close to I_c. This phenomenon has been observed when testing most the joint loops.

The resistance of the joint can be calculated with parameters α_3 and β_3 in equation (9), which are determined by the data collected when t>t_m. That is to say, the resistance of the joint should be tested when the joint is in the state of thermally assisted flux flow.

C. Relaxation of Magnetization in Joint Loops

When the induced current in the joint loop under 0 T background magnetic field was stable, a 1 T background magnetic field was exerted on the persistent joint. Relaxation of magnetization in the persistent joint due to flux creep under 1 T background magnetic field was observed when testing the persistent joint loops.

The previously-mentioned thermally assisted flux flow, thermally activated flux creep and magnetization relaxation can all be classified into flux creep, and the driving force density for all these movement of vertexes is F_d=JB. The movement of vertexes can be called thermally assisted flux flow when J<<J_c and the resistivity of the superconductor is constant. When J is close to J_c and the resistivity of the superconductor is not constant any more, the movement of vertexes can be called thermally activated flux creep. Magnetization relaxation in this article is the slow flux creep under background magnetic field compared to the fast thermally activated flux creep under 0T background magnetic field.

Figure 8 shows two kinds of magnetization relaxation measured during the experiments. Current decrease in the joint loop due to flux creep is easy to understand, as sample 1# in Figure 8. However, the current increase in the joint loop is hard to understand, as sample 2# in Figure 8, since there was no varying magnetic field to excite the joint loop at that time. One possible explanation is that, the induced current in the joint loop was stable, the measured current increase or decrease happened because the flux creep of trapped flux in the persistent joint influenced the Hall sensor. Since the trapped flux may be in different directions, which may increase or decrease the measured current in the joint loop.

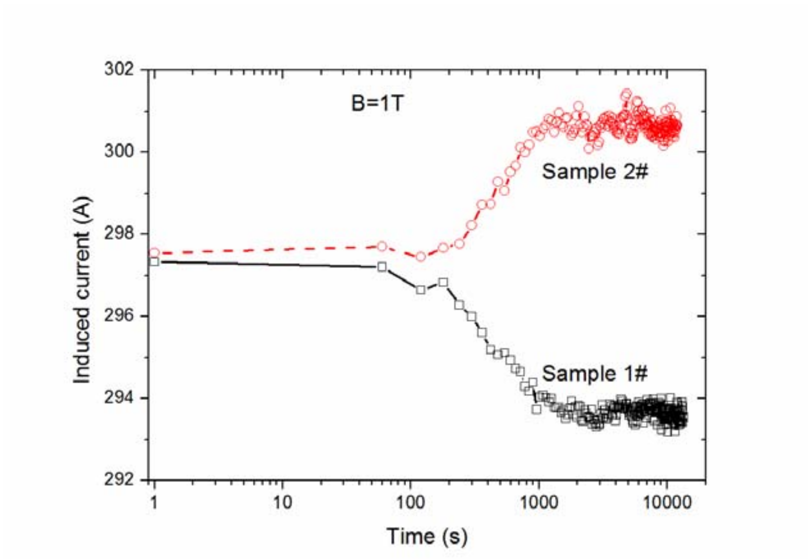

Fig.8. Current decay due to relaxation of magnetization.

Magnetic flux was trapped in the joint loop during the excitation process, since the persistent joint may quench many times in this process, the final trapped magnetic fluxes in the joint loop may be in opposite directions. When the joint loop with stable induced current was under 1 T background magnetic field, the increase of driving force on the trapped flux caused the trapped flux in the persistent joint to creep, since the microstructures of pinning centers in the persistent joint was damaged during the fabrication process. If the direction of the trapped magnetic flux in the persistent joint was the same with the direction of the magnetic field generated by the induced current in the persistent joint loop, the flux creep in the persistent joint would reduce the magnetic field measured by the Hall sensor, which will result in a reduction of the measured current in the joint loop. Otherwise, the flux creep in the persistent joint would increase the magnetic field measured by the Hall sensor, which would result in an increase of the measured current in the joint loop. This kind of magnetization relaxation process is quite slow compared with the flux creep during the excitation process under 0T background magnetic field, when testing the resistances of the persistent joints, the flux creep under background magnetic field should be considered so as to obtain a more accurate measurement result.

VI. CONCLUSION

Persistent joints were fabricated based on the cold-pressing welding method, and the electrical properties of which were studied using the current decay measurement technique. The test results show that the joints fabricated for 400 MHz NMR magnet have resistance as low as $<10^{-13}$ ohm at a transport current much higher than the operating current. The nine-joint series fabricated to simulate the coil assembly were also tested, test results shows that the total resistance is 3×10^{-14} ohm at 120 A under 1 T background magnetic field. The current in the joint loop has a sharp decrease under 0T background magnetic field due to the flux creep, after that the current will decay very slowly due to thermal assisted flux flow. The phenomenon of magnetization relaxation in persistent joint loops under 1 T background magnetic field was observed, and the resistance of the joint should be calculated based on the data collected during the process of thermal assisted flux flow after the magnetization relaxation process was over.

Test results show that the cold-pressing welding method is very reliable and can be used to fabricate NbTi-NbTi multifilamentary persistent joints for small NMR magnets.

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