Low-noise Cryocooler-cooled Compact SQUID-NDE System for Carbon-Fiber Composites

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Abstract. A low-noise and compact SQUID-NDE system cooled by a cryocooler has been developed for practical NDE of carbon-fiber composites. In order to suppress the magnetic noise due to a cryocooler, a planar high-T_c SQUID gradiometer, coaxial pulse tube cryocooler, and SQUID stage separated from the cold head of the cryocooler were introduced. There was no increase of magnetic flux noise due to the cryocooler compared to noise by liquid-nitrogen cooling with the same SQUID. The temperature at the SQUID stage was controlled at 75K±0.05K for several hours by heaters. The field generator with ferrite cores was employed for relatively lower electric conductive composites. Detections of deep-lying hidden slots in stacked carbon-fiber composite specimens were demonstrated to show the effectiveness of the system.

1. Introduction

Carbon-fiber composites, such as carbon fiber reinforced plastics (CFRPs) and carbon fiber reinforced carbon matrix composites (C/Cs), have been employed in many structures, especially in aerospace structures like noses and wings of aircrafts and space shuttles because they have lightweight and high fatigue resistance [1][2]. In addition to these advantages, C/Cs have heat resistance up to 3000K, too [1][2]. However, conventional non-destructive evaluation (NDE) techniques, using X-ray, ultrasonic and acoustic emission, have some difficulties to detect defects in the composites in many cases, due to the unique properties of the composites [3-5]. For more reliable use of these composites, development and establishment of effective NDE techniques are the urgent businesses.

On the other hand, it was clarified the NDE technique using superconducting quantum interference device (SQUID-NDE) is suitable for conductive materials by many researches, which mainly focused on aluminium materials in aircrafts [6-8], because of the incomparable high magnetic sensitivity of SQUID in the wide frequency range from DC to 1MHz [9]. In recent years, some researches of SQUID-NDE on the carbon-fiber composites have been started and revealing that SQUID-NDE has possibilities not only to detect small and deep-lying defects in the composites, but also to evaluate damage degree in the defective composites [10-12]. At the present time, SQUID-NDE is an attractive candidate for the composites and may be unique available technique for C/Cs [11].

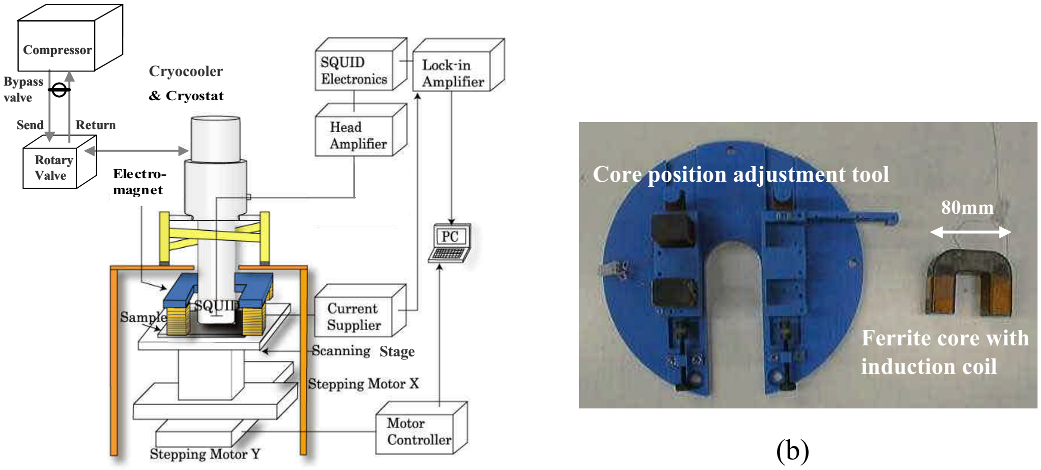
In order to realize on-site NDE measurements on the composites, an easy-handling and practical NDE system is necessary. For this, we have developed a low-noise and compact cryocooler-cooled SQUID-NDE system with field generator using ferrite cores. As for cooling a high sensitive SQUID by a cryocooler, there are three well-known problems. These are the magnetic noise from magnetic materials used in cryocooler, mechanical vibration due to cryocooler transmitted to the SQUID, and thermal fluctuation at the cold end that causes drift of I_c of SQUID [13-15]. In order to suppress the noise increase, we have employed a planar high-T_c SQUID gradiometer, coaxial pulse tube cryocooler (PTC), and the SQUID-set stage separated from the cold head of the cryocooler. Because the change of working temperature causes the change of SQUID sensitivity, two heaters have been introduced for thermal control. For much lower electric conductivity of the composites than metals, we have employed a magnetic field generator using two U-shaped ferrite cores wounded by wire coils; e.g. electromagnets, to induce enough eddy current density in the composites [16].

The magnetic flux noise of the cryocooler-cooled system was measured and compared with that of liquid-nitrogen-cooled system using the same SQUID. Thermal stability at the SQUID stage was also investigated. The NDE measurements on deep-lying slots in stacked C/Cs board specimens were carried out to demonstrate the defect detection ability for the carbon-fiber composites.

2. SQUID-NDE system cooled by a cryocooler

Schematic drawing of the developed SQUID-NDE system with cryocooler is shown in Fig.1 (a). This system is modified from a SQUID-NDE system using liquid nitrogen [11]. The system is composed of a high-T_c SQUID gradiometer and SQUID electronics, lock-in amplifier and current supplier, two-dimensional scanning stage, the field generator using electromagnets, and the coaxial PTC with the compressor and the rotary valve.

The field generator composed of a pair of U-shaped ferrite cores with permeability of $2300\mu_0$ and 400 turn coils is shown in Fig.1 (b). The field generator generates ac magnetic field of 0.5mT at a point 10mm straight away from the core end by current of 100mA. The frequency dependence of the generated field is roughly flat in the frequency range between 1Hz and 400Hz. Two electromagnets are set at both sides of the SQUID to keep symmetry of the field. Alignments of the electromagnets are carefully adjusted to minimize the magnetic flux from the electromagnets coupled to the SQUID before NDE measurements.



(a) (b)

Figure 1. SQUID-NDE system with cryocooler. (a) Schematic drawing of the system. (b) The field generator composed of core position adjustment tool and electromagnets.

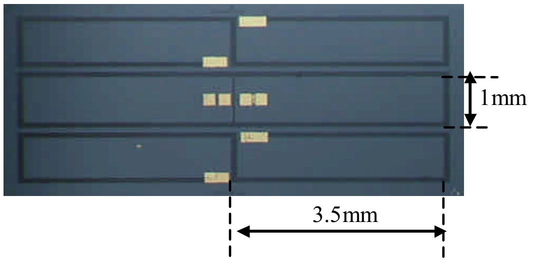


Figure 2. The planar high-T_c SQUID gradiometer with baseline of 3.5mm.

Table 1. Characteristics of the high-T_c SQUID gradiometer (at 77K by LN_2).

SQUID inductance	100pH
Pick-up coil size	3.5mm x 1mm
Baseline length	3.5mm
I_c	60μA
R_n	2.4Ω
ΔV	8μV
Magnetic flux noise	$20\mu\phi_0/\mathrm{Hz}^{1/2}$@100Hz (in MRS)

2.1. *High-T_c SQUID gradiometer*

We have employed a planar high-T_c SQUID gradiometer fabricated in AIST to reduce the environmental magnetic noise and also the magnetic noise due to the mechanical vibration of cryocooler. Use of the SQUID gradiometer also contributes to construct a compact NDE system because of no necessity of magnetic shielding. Figure 2 shows the microscopic photograph of the SQUID gradiometer. $SrTiO_3$ bicrystal substrate with mis-orientation angle of 30 degree was used. $YBa_2Cu_3O_{7-\delta}$ thin film with thickness of 200nm was deposited on the substrate using pulse laser deposition technique. The gradiometeric pickup coil, composed of two rectangular coils with size of 3.5mm x 1mm (in the middle of the photo), are connected directly to the SQUID ring located at the center of the device. The modulation coils, which shape looks like the mathematical symbol "∞", locate in upper and down regions of the pickup coil. One of them is actually used, and the other is dummy to keep symmetry. The characteristics of the SQUID are summarized in Table 1.

2.2. *Coaxial pulse tube cryocooler*

A coaxial PTC has been developed for the system. Maximum amplitude of the mechanical vibration of a coaxial PTC is generally smaller than conventional two-axial PTC. In order to suppress the cryocooler-generated magnetic noise, the cryocooler and cryostat were made of non-magnetic materials. The coaxial pulse tube and cold head were made of stainless steel and copper, respectively. The cryostat was made of aluminium, copper and acrylic resin. The photograph of the PTC with cryostat is shown in Fig.3. For easy-handling and practical use, the cryostat and PTC was designed to be compact (totally 4 kg in weight, 50mm in diameter, and 400mm in height). Because the compressor and rotary valve for the PTC generate magnetic noises, they are spatially separated from the cryostat with length of a few meters. The characteristics of the PTC are summarized in Table 2.

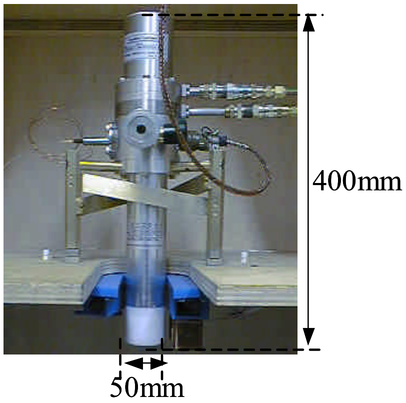


Figure 3. Coaxial pulse tube cryocooler integrated in the cryostat.

Table 2. Characteristics of the coaxial pulse tube cryocooler

Cooling capacity	8W (60Hz at 77K)
Ultimate temperature	53K (60Hz)
Cooling time (77K)	25min.
Weight	4kg
Compressor power	800W

The pressure oscillation frequency of the cryocooler, which is generated by means of the motor-driven rotary valve, is 2Hz. The temperature of the cold head is primarily controlled by amount of helium gas using the bypass valve that controls the gas volume supplied to the cryocooler (See Fig.1 (a)), and secondary controlled by heaters.

2.3. Separation of the SQUID-mounted stage

Although the vibration of the coaxial PTC is small, the vibration of the SQUID cannot still be neglected if the SQUID is directory mounted on the cold head of the PTC. For further reduction of the vibration of the SQUID, we have designed an alternative stage where the SQUID gradiometer is mounted, separated from the cold head. Figure 4 illustrates schematically the cross-section view of the bottom part of the cryostat. The SQUID-set stage, made of copper, is connected to the cold head with flexible fine copper wires. The copper wires reduce the magnitude of the vibration and conduct the heat from the cold head to the SQUID-set stage. The stage is fixed through FRP rods to the inner wall of the cryostat. The SQUID is mounted on the stage by using Apiezon grease, and cooled around 77K. The distance between the SQUID and the outer bottom of the cryostat is 4mm.

The SQUID-set stage and the copper wires should play the role of thermal capacity and thermal buffer, respectively. Thus, the SQUID on the stage should be less affected by the thermal fluctuation of the cold head than the SQUID directly mounted on the cold head. One-layer super-insulator films are glued on the inner bottom and wall of the acryl cap to reduce the heat intrusion from outside. Thermocouples are mounted on the sidewall of the cold head and the SQUID-set stage to monitor the temperatures.

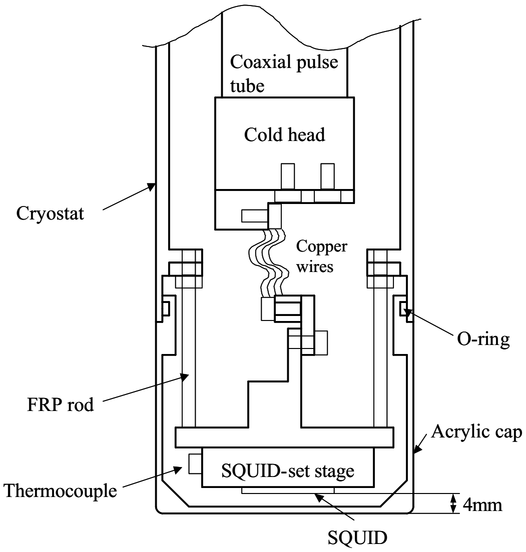


Figure 4. Schematic cross-section view of the bottom part of the cryostat.

3. Measurements and Results

3.1. System noise

We evaluated the noise performance of the cryocooler-cooled SQUID-NDE system by measuring the magnetic flux noise while cooling the SQUID at 75.3K, and comparing the noise with that by former SQUID-NDE system using liquid nitrogen and the same SQUID. Figure 5 shows the both noise spectra in the frequency range between 0.1Hz to 1000Hz. The bold line and broken line correspond to results by using cryocooler and using liquid nitrogen, respectively. Each measurement was conducted without magnetic shielding and with FLL (flux locked loop) operation of the SQUID and dc bias. There is neither increase of white noise nor peak noises due to the vibration of the cryocooler. Minimum magnetic flux noise and magnetic filed gradient noise at 100Hz is about $80\mu\phi_0/\mathrm{Hz}^{1/2}$ and 1.3 $\mathrm{nT/m/Hz}^{1/2}$, respectively. The noise by using cryocooler is a little bit lower than that by using liquid nitrogen. It should be due to the lower cooling temperature. Figure 5 indicates that the magnetic noise due to the vibration has been well suppressed by using the high-T_c SQUID gradiometer, the coaxial PTC, and the separation of the SQUID-set stage.

We preliminary estimated the allowable amplitude of the vibration for the SQUID-NDE system. For the estimation, we measured the environmental magnetic field gradient at the place where the SQUID will be set using a fluxgate magnetic sensor. The magnitude of the gradient was about 20μT/m. In the case that a SQUID magnetometer vibrates with amplitude of l in an environmental magnetic field gradient of $\mathrm{d}B_z/\mathrm{d}l$, the magnetic noise ϕ_n due to the vibration is expressed by the following equation,

$$\phi_n=(\mathrm{d}B_z/\mathrm{d}l)\cdot l\cdot S \tag{1}$$

where S is a pick-up coil area of the SQUID magnetometer. In this system, the directly coupled type high-T_c SQUID gradiometer is used. The imbalanced area between the pick-up coils of the gradiometer is about 1/1000 of the single pick-up coil area, 3.5mm x 1mm. We desired the peak noise due to the vibration was smaller than $50\mu\phi_0$. Then, the allowable amplitude of the vibration is estimated to be shorter than 1.4μm according to the equation (1). The real vibration amplitude at the SQUID-set stage was confirmed by

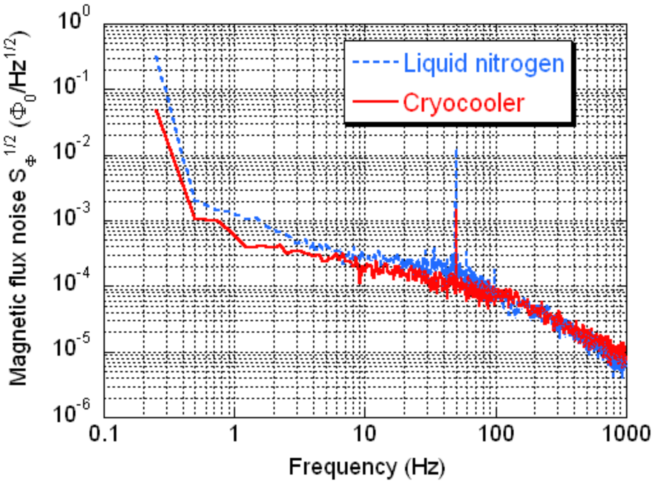


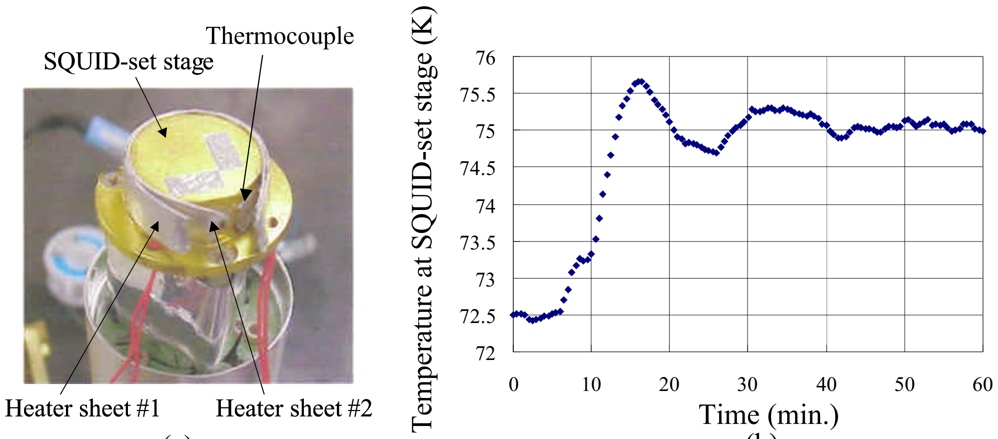
Figure 5. Magnetic flux noise spectra of the cryocooler-cooled system, and liquid-nitrogen-cooled system using the same high-T_c SQUID gradiometer.

measurements with an accelerometer. The amplitude of the vibration along *x*-axis, *y*-axis (horizontal) and *z*-axis (vertical) were all within 0.8 – 1.5μm when the gas line made of copper between the cryocooler and the rotary valve was rigidly fixed. The pick-up coil area of SQUID magnetometer is usually about several mm^2 or more. It will cause a peak noise over $10^4 \mu \phi_0$ due to the vibration with the amplitude 1.4μm. Therefore, a SQUID gradiometer is most effective factor to suppress the magnetic noise due to the vibration.

3.2. *Thermal stability*

The thermal stability of the system was investigated by measuring the temperatures at the SQUID-set stage and the cold head by the thermocouples. Firstly, the thermal drifts at both were measured. The temperatures starting from 75K (SQUID-set stage) and 68.2K(cold head) were recorded for 12 hours without temperature control. During the measurements, the temperature of the laboratory was controlled at 296K±0.5K by air conditioner. The temperatures at the SQUID-set stage and cold head increased 0.1K and 0.07K per hour, respectively. The drift at the SQUID-set stage causes the change of magnetic sensitivity and increase of magnetic noise due to the change of I_c of the SQUID. For long time measurements more than several hours, the drift is required to be suppressed less than 0.1K. Then, we introduced the temperature control. Considering that the time constant by the bypass valve control is too long, although this control makes less magnetic noise, we adopted a temperature control with heater.

The temperature control system was composed of two heater sheets (MINCO 9914) and temperature controller (Lakeshore Model331). Two heater sheets enclosed in indium sheets were wounded around the SQUID stage (see Fig.6 (a)). The current supplied to each heater is flowing in the opposite direction to each other, in order to cancel out the magnetic noises due to the currents. The thermal stability with the temperature control system was measured while stabilizing the SQUID-set stage at 75K. The result is shown in Fig.7. Starting from 72.5K at the SQUID stage, it took about forty-five minutes to stabilize the temperature at 75K. After the temperature was stabilized, the temperature fluctuation was within ±0.05K for several hours.

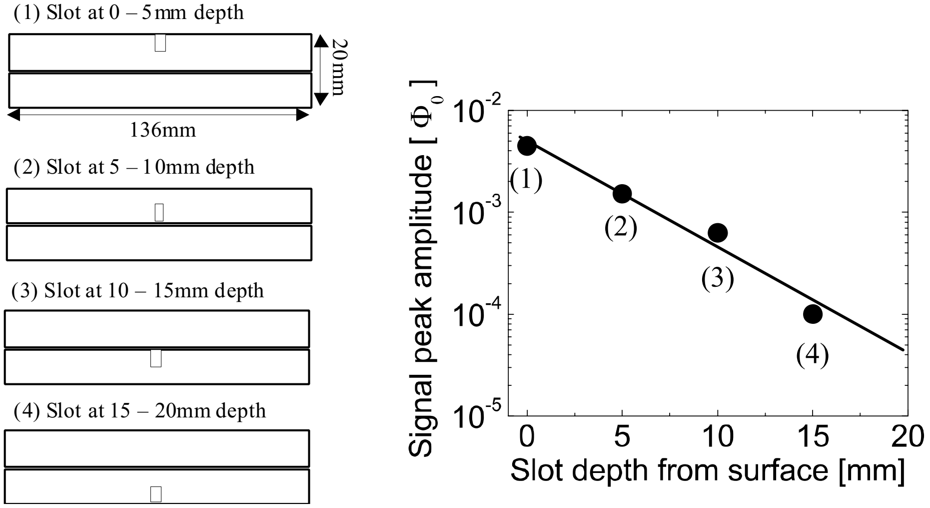


(a) (b)

Figure 6. (a) Photograph of the SQUID-set stage wounded by two heater sheets. The cryostat is up side down in the photo. (b) Time trace of the temperature at SQUID-set stage with the temperature control system. The final temperature was set at 75K.

3.3. *Application to carbon-fiber composites*

The ability of the SQUID-NDE system to detect defects in carbon-fiber composites was demonstrated by detecting deep-lying slots in C/Cs specimens. Figure 7 (a) shows the cross-section of the prepared 4 specimens. All specimens were composed of two C/C boards with thickness of 10mm, in which one board was stacked on the other. Size of the specimens was 136mm in width, 240mm in length, and 20mm in total thickness. Each specimen had a slot of 20mm in length, 2mm in width and 5mm height at various depths in the center of the specimen plane. The NDE measurements on the specimens were carried out by the SQUID-NDE system with stand-off of 7mm, current of 5mA at 310Hz supplied to the electromagnets, and sampling interval of 2mm. The quadratic-poles-like magnetic signal due to the slot was detected near above the slot by two-dimensional scanning on the



(a) (b)

Figure 7. Detection of deep-lying slots in thick carbon-fiber composites by the cryocooler-cooled SQUID-NDE system. (a) Four stacked C/Cs specimens with slots at various depths. (b) The slot depth from the surface vs. signal amplitude due to each slot.

specimens. Figure 7 (b) shows the amplitude between the positive peak and negative peak of the quadratic-poles-like signal due to each slot. The deepest slot with depth of 15mm was successfully detected. According to the relation between the slot depth and the signal amplitude and the system noise, deeper slot with depth of 30mm should be detectable by the SQUID-NDE system with current of 100mA. Because the amplitude of the current used in the measurements was limited by the residual magnetic flux coupled to the SQUID, more accurate adjustment of the electromagnets will solve this problem.

4. Conclusions

An easy-handling and low-noise SQUID-NDE system cooled by a cryocooler has been developed for practical NDE of carbon-fiber composites. A high-T_c SQUID gradiometer, coaxial PTC, and separation of the SQUID-set stage were employed to realize a low magnetic noise, low mechanical vibration and compact system without magnetic shielding. The minimum magnetic flux noise is about $80\mu\phi_0/\mathrm{Hz}^{1/2}$ at 100Hz dominated mainly by SQUID itself. We estimated that the magnetic noise due to the vibration in this system should be about $50\mu\phi_0$ with vibration amplitude of 1.4μm. This low noise owes primarily to the SQUID gradiometer, secondly to lowly suppressed vibration. The temperature control system with two heater sheets was introduced to stabilize the temperature of the SQUID stage at 75K with thermal fluctuation of ±0.05K for several hours. The deep-lying slot below 15mm from the surface in stacked C/Cs specimen was successfully detected by the SQUID-NDE system. We concluded that this system has a potential for easy-handing and on-site NDE measurements on the carbon-fiber composite structures.

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