Superconducting Pickup Coils Fabricated On A Glass Epoxy Polyimide Resin Substrate For SQUID Magnetometers

J Kawai, T Shimozu, M Kawabata, G Uehara and H Kado

Applied Electronics Laboratory, Kanazawa Institute of Technology, 3 Amaike, Kanazawa, Ishikawa, 920-1331 Japan

E-mail: j-kawai@neptune.kanazawa-it.ac.jp

Abstract. We developed a new fabrication technique of superconducting pickup coils on a glass epoxy polyimide resin (GEPR) substrate, which is robust against mechanical shock compared to those fabricated on a silicon substrate. Niobium pickup coils were fabricated on a 3-inch-shape GEPR wafer using thin-film process. The wafer has copper terminals embedded on the surface in advance. Therefore, an additional substrate is not needed to assemble a magnetometer/gradiometer with a SQUID chip. Connecting these pickup coils with a SQUID chip, a magnetometer and a gradiometer were fabricated and were characterized. Both the magnetometer and the gradiometer have no damage against 50 thermal cycles between room temperature and 77 K. The field resolution of these magnetometer and gradiometer with the detection area of 10 mm × 10 mm are 2.7 fT/√Hz and 4.1 fT/√Hz at 1 kHz respectively, which are good enough for biomagnetic measurements.

1. Introduction

SQUID magnetometers and gradiometers are used as ultra-sensitive magnetic sensors for various applications such as magnetoencephalograph (MEG) [1], magnetocardiograph (MCG) [2], non-destructive evaluation [3] and so on. In combining a pickup coil with a SQUID with a flux transformer, various types of magnetometers and gradiometers are realized. A co-axial pickup coil to detect z-component of a magnetic field is usually fabricated by using superconducting wire wound around a cylindrical bobbin [4], while a planar pickup coil to detect a tangential component is fabricated on a silicon (Si) substrate by using thin-film technology. In this case, the Si-pickup coil is mounted on an additional substrate such as a printed board together with a SQUID chip [5] [6]. This assembling method, however, has a frequent risk that the Si-pickup coil is easily broken due to mechanical shock and thermal stress attributed to the difference of thermal expansion coefficient between Si and the additional substrate and a glue to fix the pickup coil with the substrate when it is cooled down at extremely low temperature.

We have developed a new fabrication technique of robust planar pickup coils using glass epoxy polyimide resin (GEPR) substrate having copper terminals. The advantage of this method is also that an additional substrate such as a glass epoxy printed board is not needed. The GEPR pickup coil with copper terminals doubles as the substrate for the SQUID chip. Therefore, a SQUID chip is just mounted on the pickup coil and is connected to the terminals with wirebonds.

We fabricated and characterized single-turn pickup coils for a SQUID magnetometer and gradiometric pickup coils for a SQUID gradiometer.
2. Fabrication Process

2.1. Glass epoxy polyimide resin (GEPR) wafer

Glass epoxy polyimide resin (GEPR) has heat-resistance up to 250 °C, which means it can resist heat in plasma processing compared to glass epoxy resin used as printed boards. A GEPR substrate was formed into 3-inch-wafer shape to adapt to thin-film fabrication process, as shown in Fig. 1. The thickness of the substrate is 0.8 mm and the roughness of the surface was 1µm-pp on average. On the surface of the substrate, two kinds of copper terminals were embedded in advance. One is to be connected to a SQUID chip with wirebonds and to cables with solder when a pickup coil is completed, and the other is located at the corresponding position to the bonding pads of the pickup coil, which functions as the foundation for wirebonds. If there is no foundation under the bonding pads, superconducting wirebond connections between the pickup coil and the input coil in the SQUID chip cannot be performed because the GERP substrate is so soft that ultrasonic disperse.

2.2. Fabrication process of pickup coils

Niobium (Nb) pickup coils were fabricated on the GERP wafer using thin-film process, in which we adopted liquid polyimide (TORAY, SP-811) as planarization layer of the surface of the wafer and insulators for multi-layer process. Glass transition temperature (Tg) of this polyimide is 300 °C. The polyimide layer was formed with a spin coater and then was baked in an oven to harden. The baked polyimide layer on the copper terminals is etched in an alkaline solution (2.38%-TMAH). Figure 1 shows the dependence of the etching rate of the polyimide layer on the baking temperature when the baking time was 20 minutes. The polyimide layer was not etched when it was baked at more than 220°C. We confirmed that the etching rate decreased as the baking time increased.

The shape of the etched pattern was also evaluated against the baking temperature. We measured the width of a rectangular pattern with the width of 50 µm for different baking temperature of the polyimide layer of 1µm in thickness. In Fig. 2, the width of the etched pattern measured with a stylus surface profiler was depicted for different baking temperature. The baking time was 20 minutes. At temperature lower than 170 °C, the pattern became broader while the pattern was almost stable at more than 170 °C. From these results, we set the baking temperature and the time to be 170 °C and 20 minutes, respectively. This condition does not deteriorate niobium film very much.

The spin-coated polyimide layer planarized the surface of GEPR wafer well. Figure 3 (a) shows an SEM image of a bare surface of the GEPR wafer. The observed area is about 90 µm x 120 µm. The line in the middle is the edge of the embedded copper terminal. The mean roughness of the GEPR...
surface was about 1µm-pp and the difference of the level between the surface and the copper terminal is also about 1µm. Figure 3 (b) shows the same area with the spin-coated polyimide layer baked at 170 °C for 20 minutes. The surface and the edge line were completely planarized and the roughness of the GEPR surface was improved to be less than 200 nm. This surface is smooth enough for deposition of a niobium film with the thickness of 1 µm.

On this planarized wafer, an 800-nm-thick Nb layer was deposited with a dc-magnetoron sputtering with the power density of 7.4 W/cm² in Ar atmosphere. The flow and the pressure of Ar were 20 sccm and 1.3 Pa, respectively. The deposited Nb film was then patterned with a reactive ion etching (RIE) with the power density of 0.1 W/cm² in CF₄ mixed with O₂ of 7% in flow. Here, a single-turn pickup coil for a magnetometer was completed. To fabricate gradiometric coils, 1-µm liquid polyimide layer as an insulator was continuously formed and the via-hole on the first Nb pattern was etched in the same way as the first planarization layer was formed and etched. Then, the second Nb layer of 1 µm in thickness was sputtered and patterned. Lastly, a polyimide layer as a passivation layer was formed on the surface.

Figure 4 shows an SEM image of the cross section of the multilayer in the gradiometric pickup coil.

![Figure 3](image1.png)

**Figure 3.** SEM images of the surface of a GERP wafer and a copper terminal. (a): bare surface. (b): spin-coated and baked polyimide layer. Observation area of both images are the same.

![Figure 4](image2.png)

**Figure 4.** An SEM image of the cross section of the multilayer consisting of the planarization polyimide layer, first Nb layer, polyimide insulation layer and the second Nb layer.

![Figure 5](image3.png)

**Figure 5.** A completed GEPR wafer on which 4 rectangular coils for SQUID magnetometers and 2 gradiometric coils for a SQUID gradiometers are fabricated. The detection area of each coil is 10 mm × 10 mm.
It is clear that the second Nb layer has a good coverage thanks to the planarization effect of the polyimide insulation layer over the first Nb layer. Therefore, larger critical current of the pickup coil is expected. The passivation layer is not seen in this image.

Figure 5 shows a completed wafer integrated with single-turn pickup coils for a SQUID magnetometer and gradiometric pickup coils for a SQUID gradiometer. The detection area of both coils is 10 mm × 10 mm and the line width of the coil is 100 µm. The base line of the gradiometric coil is 4 cm. The copper terminals seen at both sides of the wafer are for bonding and soldering terminals. These pickup coils were easily cut into pieces with a rotary cutter commonly used.

The residual resistive ratio (RRR), which is the ratio of the resistance between at room temperature and at the temperature just before superconductive transition, was evaluated to be 3.5 – 4.0. These values are a little smaller than those obtained from a Nb film of the same thickness deposited on a Si wafer. It is probably because that the outgas from the GEPR wafer during Nb sputtering affected the quality of the Nb film. We think, however, this deterioration in Nb film does not influence the performance of the pickup coil very much. The critical current of these pickup coils in liquid helium was measured to be larger than 70 mA, which is as large as that of the input coil in a SQUID we are fabricating.

2.3. Assembly method

In combination with a SQUID having a flux transformer, the fabricated GEPR pickup coils were assembled into a magnetometer and a gradiometer as shown in Fig. 6, where (a) is the magnetometer and (b) is the gradiometer. As the SQUIDs used here were designed for an axial gradiometer we have developed for an MEG system, the matching between these pickup coils and the input coil of the SQUID is not optimized. The inductance of the input coil of the SQUID is larger than that of these pickup coils.

The assembling method is as follows: A 2.5 mm × 2.5 mm SQUID chip was glued on the pickup coil. Electrical connections between the bias, the output and the feedback coil of the SQUID and the soldering terminals were done with 25-mm aluminum wirebonds. Between the input coil in the SQUID and the pickup coil, superconducting connections were done with annealed 25-µm Nb wirebonds. A small resistor was placed adjacent to the SQUID chip as a heater to remove trapped flux when needed. The wirebonded SQUID chip and the heater resistor were covered with an epoxy resin for protection [6]. The two holes in the gradiometer are for screws to fix it with a measurement probe when it is inserted into the cryostat.

Figure 6. Assembled SQUID magnetometer (upper) and gradiometer (lower) combining SQUIDs and the GEPR pickup coils. The hole on the gradiometer is for screws to fix it to a measurement probe. The SQUID chip is covered with an epoxy resin.
Table 1. Typical parameters

| parameter                        | magnetometer | gradiometer |
|---------------------------------|--------------|-------------|
| Maximum critical current (µA)   | 30           | 31          |
| Maximum voltage modulation (µV) | 66           | 76          |
| Shunt resistance (Ω)            | 2.9          | 3.3         |
| Field resolution (fT/√Hz)       | 2.7          | 4.1         |

3. Characterization

The assembled magnetometer and gradiometer, which were set in a glass fiber reinforced plastics (GFRP) cryostat and were connected with a flux locked loop (FLL) with a direct voltage readout, were characterized in a liquid helium. The measurement was done in a magnetically shielded chamber with the shielding factor of 1/1000 at 100 Hz.

The typical parameters are summarized in Table 1. Calibrating the sensitivity with our calibration system [7], the field resolution was measured to be 2.7 fT/√Hz for the magnetometer and 4.1 fT/√Hz for the gradiometer at 1 kHz, respectively. These resolutions are good enough for biomagnetic measurements and other high-sensitive magnetic measurements.

We also evaluated durability of the pickup coils against thermal cycles. These magnetometers and gradiometer were cooled down in liquid nitrogen for 1 minute. Then they are taken out and were dried with a drier. After this process was repeated 10 times, the magnetometer and gradiometer were checked in liquid helium focusing on the voltage-to-flux (V-Φ) characteristics. In this experiment, an external current to provide the SQUID with a magnetic flux was applied through the feedback coil, which is not coupled to the SQUID loop but coupled to the input coil magnetically. Since the input coil is connected to the pickup coil, if there is disconnection in the pickup coil due to the thermal shock, the period of V-Φ curve is clearly changed. We repeated the above process 5 times, i.e. 50 times in liquid nitrogen and 5 times in liquid helium, for the magnetometer and for the gradiometer in each. It was confirmed that there was no disconnection in both, and the V-Φ characteristics was not changed. From this result, we conclude that these pickup coils are robust enough against thermal cycles and the assembling method was also reliable.
4. Conclusion

Using a GEPR substrate, a new technique to fabricate robust and easy-handling superconductive pickup coils for planar SQUID magnetometers and gradiometers has been developed. With a multi-layer process adopting liquid polyimide as an insulator, gradiometric pickup coils having a crossover in its pattern were successfully fabricated. Assembled SQUID magnetometer and gradiometers combining the pickup coils with SQUIDs have a good performance and durability against thermal cycles. We think it is possible that SQUIDs are fabricated on a GEPR substrate together with pickup coils.

References
[1] Pizzela V, Penna D, Gratta D, and Romani L 2001 Supercond. Sci. Technol. 14 79-114
[2] Kandori A, Shimizu W, Yokosawa M, Noda T, Kamakura S, Kiyatake K, Murakami M, Miyashita T, Ogata K, and Tsukada K 2004 Biomag2004 355-356
[3] Isawa K, Nakayama S, Morooka T, Ikeda M, Takagi S, Chinone K, and Tosaka S 2005 Physica C 418 1-8
[4] Kado H, Higuch M, Shimogawara M, Haruta Y, Adachi Y, Kawai J, Ogata H, and Uehara G, 1999 IEEE Trans. Appl. Supercond. 9 4057-4062
[5] Adachi Y, Uehara G, Kawai J, Kawabata H, Ogata H, Komori H, and Kado H 2001 Supercond. Sci. Technol. 14 1075-1080
[6] Cantor R, Hall J, Matlabov N, and Volegov P 2007 IEEE Trans. Appl. Supercond. 17 672-675
[7] Kawai J, Sakamoto Y, Kawabata M, Shimizu T, Uehara G, and Ogata H 2005 IEEE Trans. Appl. Supercond. 15 3901-3905
[8] Uehara G, Adachi Y, Kawai J, Shimogawara M, Higuchi M, Haruta Y, Ogata H, and Kado H 2003 IEICE Trans. Electron. E86-C 43-53