Development of STJ for neutron detector on Si-LBO hybrid substrate by surface-activated room-temperature bonding

S Endo1,2, G Fujii2, M Ukibe2, H Takagi2, M Ohkubo2, M Naruse1, H Myoren1, C Otani3, and T Taino1
1 Graduate School of Science and Engineering Saitama University, 225 Shimo-Ohkubo, Sakura-ku, Saitama, 338-8570, Japan
2 AIST, 1-1-1 Umezono, Tsukuba-shi, Ibaraki 305-8568, Japan
3 RIKEN, 519-1399 Aramaki-Aoba, Aoba-ku, Sendai, Miyagi 980-0845, Japan

taino@mail.saitama-u.ac.jp

Abstract. We have been developing superconducting tunnel junctions (STJs) for neutron detector on a Si-Li2B4O7 (LBO) hybrid substrate. An active area of each STJ was limited by dicing in order to obtain high spatial resolution. The Si-LBO hybrid substrate was bonded by a surface-activated room-temperature bonding. In this bonding method, the Si and LBO surfaces were sputter-etched by high energy Ar ion beam and bonded in a vacuum. We fabricated STJs on the Si-LBO hybrid substrate and measured their current-voltage characteristic. Their leakage currents (I_leak) were < 10 nA, and so we succeeded in fabricating STJs on the Si-LBO hybrid substrate, which can be expected to operate as neutron detectors with high performance.

1. Introduction
Neutron has a low cross section to metals, although its cross section to light elements, such as hydrogen and lithium is high. For this reason, neutron has been expected as a probe in order to search the state of hydrogen and lithium surrounded within metal frame non-destructively. For example, neutron diffraction is well known to be a useful technique for analyzing a bulk texture of metallic materials because of a large penetration depth of the neutron beam, and utilized in many industrial fields such as an automotive industry [1]. In those research fields, it is necessary for a neutron detector to exhibit high detection efficiency, high spatial resolution, and high counting rate capability, simultaneously. Superconducting tunnel junctions (STJs) on a single crystal Li2B4O7 (LBO) substrate is one of the promising neutron detector [2]. This detector has the excellent performance that a detection efficiency for thermal neutrons (1.8 Å) is nearly 100 % when enriched 6Li10BO with a thickness of 300 µm is used as the substrate. On the other hand, it is difficult to directly identify the irradiating position because the detector utilizes the whole substrate as an absorber for neutrons and generated quasiparticles are diffused in the whole substrate [3].

In this paper, we have proposed and fabricated a new STJ with a high spatial resolution for neutrons. The STJ was fabricated on the Si-LBO hybrid substrate by surface-activated room-temperature bonding. The fabrication process and the measured results are reported.
2. Our proposed STJ for neutron detector

Figure 1 (a) shows the cross-sectional view of the STJ on the LBO substrate. Incoming neutron into the substrate can be detected by utilizing nuclear reactions of neutron with $^6$Li and $^{10}$B, which lead to the excitation of a large number of phonons in the substrate. After these reactions, phonons propagate in the substrate, and are measured by STJs. It is difficult to directly identify the irradiating position without taking the coincidence of the pulse height from neighboring STJs because the detector utilizes the whole substrate as an absorber for neutrons. In order to solve that difficulty, we have proposed a new STJ detector as follow. The new STJ detector is fabricated on the Si-LBO hybrid substrate, which is made by the bonding of Si and LBO substrate. The Si-LBO hybrid substrate is diced to limit the active area for the improvement of the spatial resolution while keeping high detection efficiency, as shown in Figure 1 (b), (c) [4]. The STJs are located on the Si substrate and trenches between STJs are formed from the LBO substrate. The diffusion of phonons generated in the LBO due to the capture of neutrons is restricted because the connected areas of the Si between each STJ are quite small. Phonons generated in the LBO are propagated to Si without reflection because LBO and Si have very good acoustic impedance matching ($Z_{\text{LBO}} = 17.7$, and $Z_{\text{Si}} = 17.4$). In addition, neutron penetrate the trenches because the nuclear reaction between neutron and Si is very low.

![Figure 1. Cross-sectional view of (a) the conventional STJ for neutron detector on LBO substrate and (b) our proposed STJ detector for neutron detector on Si-LBO hybrid substrate and (c) Top view of our proposed STJ detector for neutron detector on Si-LBO hybrid substrate.](image)

3. Si-LBO hybrid substrate by surface-activated room-temperature bonding

3.1 Surface-activated room-temperature bonding

As mentioned above, the Si-LBO hybrid substrate is key technology for our proposed detector. The Si-LBO hybrid substrate was bonded by surface-activated room-temperature bonding [5]. In this bonding method, the contact surfaces are sputter-etched by ion or neutralized atom beam of inert gas, such as Ar and are bonded in vacuum. The sputter-etching removes contaminant layers and absorbed molecules on the surface. Then, surface atoms have activity to form chemical bonds between other atoms even at room temperature. Therefore, it is expected that the phonon signal attenuation at Si-LBO bonding interface can be ignored because no intermediate layer exists at the Si-LBO bonding interface.

3.2 Forming of Si-LBO hybrid substrate by surface-activated bonding

3inch Si and LBO wafers were used to make hybrid substrates. Table 1 shows bonding conditions of Si-LBO substrate. Figure 2 shows the cross-sectional view optical micrograph of Si/LBO interface by surface-activated bonding. In this figure, it cannot see any intermediate layer in the interface. Figure 3 shows the top view photograph of the Si-LBO hybrid substrate from the LBO side. The Newton’s
rings were observed in the figure. In this region, the Si and LBO are not bonded well. The surface smoothness of the substrate is very important for the bonding. The rms roughness of the LBO has 0.7 nm using an atomic force microscope. To achieve good bonding result, the rms roughness of the substrates needs to be less than 0.3 nm [6]. Although Newton’s rings existed in small area of the substrate, we have achieved the formation of the Si-LBO hybrid substrate by surface-activated bonding.

| Table 1. Bonding conditions. |
|-----------------------------|
| Si/LBO Substrate Size       | 3 inch / 3 inch          |
| Si/LBO Substrate Thickness  | 400 µm / 250 µm          |
| Etching Gas                 | Ar fast atom beam        |
| Degree of vacuum            | $1 \times 10^{-5}$ Pa    |
| Load                        | 10000 N                  |
| Bonding Time                | 60 sec                   |

**Figure 2.** The cross-sectional view optical micrograph of Si/LBO interface by surface-activated bonding.

**Figure 3.** The top view photograph of Si-LBO hybrid substrate from the side of the transparent(LBO) substrate.

4. **Fabrication and evaluation of STJ on Si-LBO hybrid substrate**

4.1. Fabrication process

We fabricated the prototype of STJs on the Si-LBO hybrid substrate with a size of $10 \times 10$ mm square. Figure 4 (a) shows a cross-section view of prototype STJ neutron detectors and Figure 4 (b) shows the top view photograph of the fabricated STJ. Prototype STJs had a Nb/Al-AlO$_x$/Al/Nb layer structures of 100/70/70/300 nm in this study. The Nb/Al multilayer was fabricated in the Clean Room for Analog & digital superconductivity (CRAVITY) [7]. The tunnel barrier was formed by conventional O$_2$ oxidation to achieve a critical current density ($J_c$) of about 200 A/cm$^2$. SiO$_2$ layer with a thickness of about 100 nm was deposited on the LBO side of the hybrid substrate by tetraethyl orthosilicate (TEOS) chemical vapor deposition (CVD) for preventing LBO from deliquescent.

25-pixel STJ arrays fabricated on the chip and the size of all the STJs was $100 \times 100$ µm. The STJs were fabricated via conventional photolithography, DC magnetron sputtering, a lift-off technique, reactive-ion etching, and wet etching.

4.2. Current-voltage ($I-V$) characteristic

The STJ properties were measured with a cryogen-free $^3$He cryostat with a base temperature of 0.31 K. The $I-V$ characteristics were measured to evaluate leakage currents ($I_{leak}$). The $I_{leak}$ was defined by a sub gap current value around $\sim 0.4$ mV in a magnetic field of about 10 mT. Figure 5 shows an $I-V$ characteristic of the STJ on the Si-LBO hybrid substrate. The STJ shows an excellent $I-V$
characteristic having a low sub gap current of less than 10 nA. A dynamic resistance ($R_d$) was 96 kΩ. The numbers of short and open ($N_{open}$) junctions were only two and one, respectively. From these results, our proposed STJ on Si-LBO hybrid substrate can be expected to operate as a neutron detector. In the near future, it is irradiated with the neutron in order to confirm the operation as the neutron detector.

5. Conclusion

We developed new STJ neutron detector utilizing a Si-LBO hybrid substrate in order to improve its spatial resolution. The Si-LBO hybrid substrate was formed by surface-activated bonding method. We fabricated STJs on the Si-LBO hybrid substrate with a 10 × 10 mm square. The STJ showed excellent $I$-$V$ characteristics having a low sub gap current of less than 10 nA. As the results, our proposed STJs on the Si-LBO hybrid substrate can be expected to operate as neutron detectors.

Acknowledgements

This work was supported by the Takahashi Industrial and Economic Research Foundation. The devices were fabricated in the clean room for analog-digital superconductivity (CRAVITY) in National Institute of Advanced Industrial Science and Technology (AIST).

References

[1] Ikeda Y, Taketani A, Takamura M, Sunaga H, Kumagai M, Oba Y, Otake Y, Suzuki H 2016 Nucl. Instr. and Meth. A 530 61
[2] Nakamura T, Katagiri M, Ukibe M, Ikeuchi T, Ohkubo 2004 Nucl. Instr. and Meth. A 520 67
[3] Kurakado M, Kaminohara S, Kagamihara A, Hirota K, Hashimoto H, Sato H, Hotchi H, Shimizu H, Taniguchi K 2003 Nucl. Instr. and Meth. A 506 134
[4] Endo S, Naruse M, Myoren H, Otani C, Taino T 2015 76th JSAP Autumn Meeting 14p-4A-21 (in Japanese)
[5] Takagi H, Maeda R, Hosoda N, Suga T 1999 Appl. Phys. Lett. 74 2387
[6] Takagi H, Maeda R, Chung R, Hosoda N, Suga T 1998 Jpn. J. Appl. Phys. 37 4197
[7] Web site: https://unit.aist.go.jp/riif/openi/cravity/en/index.html