Application of sapphire bonding for suspension of cryogenic mirrors

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Abstract. In order to design a suspension for large cryogenic mirror, we have measured thermal conductance and shear strength of sapphire bonding in comparison with direct bonding and hydroxide-catalysis bonding. Thermal conductance per unit area of 4 [W/K/mm²] for the direct bonding and 0.3 [W/K/mm²] for the hydroxide-catalysis bonding were obtained around 20K. Shear strength of 28[MPa] for the direct bonding and 6.5 [MPa] for the hydroxide-catalysis bonding were measured at 300K. Based on those values, an estimated area that support a weight of a mirror produces a temperature step of less than 1% of a difference of temperature in between the main mirror and a mirror of Suspension Point Interferometer.

1. Introduction
Suspension of cryogenic mirror must work as not only a heat path of conduction cooling but also a vibration isolator against external vibration sources. A suspension has to be made of a material with large thermal conductivity at cryogenic temperatures in order to achieve an efficient heat transfer. Also a pendulum system that consist of a mirror and the suspension is required to have a small loss for mirror oscillations for suppressing thermal noise. From this point of view, fundamental technologies of cryogenic mirror and suspension have been developed.[1] [2] [3] Monocrystalline α-alumina (Sapphire) has been selected as substrate of the mirror and material of supporting rods. Figure 1 shows a basic configuration of cryogenic mirror and suspension. The weight of the mirror deforms thin sapphire rods. These rods contact along half of the cylindrical circumference of the mirror and make a pendulum with high quality factor. The heat caused by a finite absorption of intense laser light is transferred to sapphire rods through the elastic contacts between the mirror and rods then carried by conduction through the sapphire rods to a mirror Suspension Point Interferometer(SPI), which relays heat path to a cryocooler system.

The basic scheme of cryogenic mirror and suspension is now demonstrated by the Cryogenic Laser Interferometer Observatory (CLIO) , which is a prototype for proving a system of cryogenic mirror.[4] It is not simply applicable, however, in the case of LCGT. In the current design of LCGT, sapphire rods with a diameter of 1.8 mm support a sapphire mirror with radius of
125 mm. Table 1 lists design parameters of main mirror and suspension rods. The diameter of sapphire rods is designed to take into account the estimated heat generation in the mirror and size effect of thermal conductivity of thin rods into consideration.[5] According to the extrapolation the data shown in the Ref.[6], the minimum bending radius of sapphire rod with diameter of 1.8 mm is estimated as \( R_{\text{min}} \approx 1400 \text{ mm} \), which make the solution used in CLIO unusable. A candidates solution is to introduce a technique of sapphire bonding. For example, we can avoid the \( R_{\text{min}} \) problem by bonding four rods on the side surface of the cylindrical mirror.

In the following sections, prospects of constructing mirror-suspension of LCGT are described from the viewpoint of heat transport and mechanical support. Thermal conductance of bonded boundary was evaluated around operating temperatures of the cryogenic mirror. Also shear strength of bonding was measured at 300K. A confirmation of quality factor of the suspension with bonding is a pending issue at this moment. Two bonding techniques, which are hydroxide-catalysis bonding and direct bonding, are mainly discussed.

![Figure 1](image.png)

**Figure 1.** A basic structure of cryogenic mirror. Both the mirror and rods are made of monocrystalline \( \alpha \)-alumina (Sapphire). A weight of the mirror deforms supporting rods and make elastic contact. A heat caused by a finite absorption of intense laser light is transferred to sapphire rods through elastic contacts between the mirror and rods then carried by conduction of sapphire rods to a mirror of Suspension Point Interferometer (SPI), which relays heat path to a cryocooler system. Upper ends of supporting rods are connected to SPI.
2. Sapphire-sapphire bonding

In this section, we will briefly review some techniques of sapphire bonding.

2.1. Hydroxide-catalysis bonding

Technique of hydroxide-catalysis bonding (HCB) was developed to obtain a tight bonding between fused silica at room temperature.[7] After a fine polishing (∼ \( \lambda/10 \)) and cleaning contaminants, a small amount of an aqueous solution of potassium hydroxide appropriate to the surface area to be bonded is introduced between the fused silica surfaces and the surfaces brought together. The chemical bonding process is initiated through hydroxide-catalyzed surface hydration and dehydration at room temperature. Readjustments of the pieces being joined are possible within approximately 30-40 minutes of the start of the joining process. The maximum strength of bonding is reached after several weeks. A same process of bonding is applicable to sapphire surfaces although a tensile strength is lower than that of fused silica.[8]

A surface to be bonded does not depend on a direction of crystal surface because HCB is basically chemical gluing and process of bonding can carry out in the room temperature. It makes possible to assemble mirror suspension independent of mirror fabrication process although method of HCB remains an small amount of impurities on the bonded boundary.

2.2. Direct bonding

Direct bonding is a method of bonding two pieces of bulk without any bonding agent. It may be considered as a combined process of the optical contact and the diffusion bonding. One of the actual applications of sapphire direct bonding is making cells for treating hydrogen fluoride solution.[9] The surfaces of the pieces of sapphire to be bonded should be of optical flatness and free from contaminations. A heat treatment under appropriate contact pressure makes the surfaces to be bonded together. This method of bonding do not always guarantee a perfect optical transparency of bonded boundary at present.

Similar bonding technique for various laser crystals has been developed although details of the process have not been published.[10] This method, in principle, does not leave any impurities on the bonded boundary.

One of the important points of direct bonding is that thermal expansion of the surfaces to be bonded should be matched because of the anisotropic thermal expansion of sapphire. For example, a difference of expansion rate \( \Delta L/L \) reaches to about 0.1% between the direction of c-axis and a-axis when the temperature rise from 300K to 1700K. [11]

Therefore one should pay attention to adjust direction of c-axis in each surface to be bonded when two surfaces are parallel to c-axis of each crystal. For example, two pieces of crystal could be bonded in the case of 19 degrees of difference of c-axis but bonding failed in the case of 35 degrees of difference. However the bonding could occur, there remained a little slip of bonded boundary in the case of 19 degrees of difference of c-axis. A slip of bonded boundary affects a precision of geometries of structure when we apply sapphire bonding for constructing a suspension of mirror. It has been empirically known that bonding does not depend on three fold rotation symmetry of a plane being perpendicular to c-axis.

In general, two crystals can be bonded at the plane on which the thermal expansion and the surface density of atoms are identical. This condition may be satisfied at the plane where Bravais lattices of each crystal are related with translation or with reflection of the plane of bonding. The former case is equivalent to a single crystal and the latter corresponds to a twin crystal.

Figure 2 shows an example of connecting a rod to the mirror by direct bonding. The c-axis of the mirror is parallel to cylindrical axis of the mirror to avoid birefringence effect. The c-axis of a mirror and a rod are perpendicular to each other. We need some matching section to connect the mirror and the suspension rod by direct bonding. As shown in the right
Figure 2. Example of connecting a rod to the mirror by direct bonding. Left: The c-axis of a mirror and a rod are perpendicular to each other. The lower end of the rod can not bond to the side of the mirror because of anisotropic thermal expansion on the surfaces to be bonded. Right: By using planes with inclination of 45 degrees, directions of the rod and the mirror can be matched to bond. This picture sketches that only a single rod connects to the mirror for simplicity reason.

Figure 3. Thermal conductance per area of bonded boundary in $10 \, \text{K} \leq T \leq 30 \, \text{K}$. Solid squares $\square$ denote $Y_{th}/A$ of AFB and solid circles $\bullet$ denote $Y_{th}/A$ of HCB where $A$ denotes the area of bonded boundary. Data around the operating temperature of the cryogenic mirror and the suspension are plotted.

of Fig.2, it can be realized by a plane with inclination of 45 degrees to each c-axis of two pieces of crystals.

Direct bonding is expected not only to realize a strong bonding as a bulk crystal but also to minimize a thermal resistance on the bonded boundary. On the other hand, there may exist some complexities in assembling and maintenance.

2.3. Other method of bonding

Double heat treatment with chemical surface treatment make a tight bonding of Ti doped sapphire, however the process is complicated.[12]

Surface-activated bonding is successful for sapphire-aluminium bonding but shows some difficulties for sapphire-sapphire bonding.[13] The method of brazing or soldering have been studied in between alumina ceramics and metals, however, we have not studied those methods.

3. Thermal conductance of bonded boundaries

We measured thermal conductivity of samples at cryogenic temperatures in comparison with methods of direct bonding and hydroxide-catalysis bonding. Parameters of samples are listed in Table 2. On measuring thermal conductivity, base temperature and heat current were controlled by two heaters, and then temperature gradient was monitored in both the bulk and the bonded part of the sample.[14] Thermal conductance of bonded boundary is defined as $Y_{th} \equiv \dot{q}/\Delta T$, where $\Delta T$ denotes a difference of temperature across the boundary and $\dot{q}$ denotes a heat current flowing through the boundary. From a difference of thermal conductivities of the bulk and the bonded part, the thermal conductance of the boundary was calculated. In this calculation,
Table 2. Description of Samples. AFB: Adhesion Free Bonding, HCB: Hydroxide-Catalysis Bonding, CSI: Crystal Systems Inc., NGK: NGK Insulators, LTD. AFB is a kind of direct bonding. For both samples, two pieces of raw materials with length of 30 mm were bonded together by each bonding method. The reason why the AFB sample has 55 mm length is a 45 degrees inclination of bonded boundary.

| Bonding Method | Size (mm)  | Manufacturer and Grade |
|----------------|------------|------------------------|
| AFB            | 4×4×55     | CSI, HEMEX             |
| HCB            | φ10×60     | NGK, ...               |

temperature dependence of thermal conductivity was ignored. An evaluated error from this assumption was less than 10 %. Figure 3 shows thermal conductance per area of bonded boundary.

In the Figure 3, $Y_{th}/A$ looked almost constant around $T=20$ K, which is the operating temperature of the cryogenic mirror. We take $Y_{th}/A=4$ W/K/mm$^2$ for Adhesion Free Bonding (AFB, Onyx Inc.[10]) and $Y_{th}/A=0.3$ W/K/mm$^2$ for HCB around $T=20$ K from the Figure 3.

4. Strength of bonding
Shear strength of bonded boundary was measured. Samples are straight cylinders with a $\phi 10$ mm × 60 mm. Two of the cylinders with a $\phi 10$ mm × 30 mm were bonded together to make a sample. All the bonding surfaces are parallel to c-axis of each crystal. Aging period is about 1 month for direct bonded sample and is about 3 years for hydroxide-catalysis bonded sample in the atmosphere at room temperature. No thermal cycle between 300 K and cryogenic temperatures have been experienced for both samples. One end of the sample was fixed and a torque was applied at the other end. The maximum torque of breaking the sample was recorded. Data were taken only at room temperature at present. Breaking occurred along the bonded boundary for HCB sample. In the case of the direct bonded sample, about 1/3 of bonding area broke along the bonded boundary and another 2/3 of area cracked into the substrate with about 50 degrees inclination to the c-axis.

Using the measured shear strength $\sigma_{shear}$ of each method of bonding, we determined the required area that can support the weight of the mirror when four rods are bonded on the side surface of the mirror. In this evaluation, $\sigma_{shear}$ is assumed to be independent on temperature.

Also a difference of temperature across the bonded boundary was evaluated for each bonding method. Table 3 shows the results. In the Table 3, a bonding area was took twice of the minimum required area per rod as a safety margin. The values of $2A_{min}$ in the Table 3 are practically realizable when the $\phi 1.8$ mm rod is used. For example, consider a rectangular bonding surface with 1.8 mm width that is made by machining and polishing a half of the thickness along the rod. Length of the rectangular surface needs 2.8 mm for direct bonding and 12 mm for HCB in each rod respectively.

Each suspension rod transfers heat current of $\dot{q}=500$ mW from the mirror to the SPI. The designed temperature of the mirror is $T=20$ K and that of the SPI is 10 K. Estimated temperature step $\Delta T_B$ due to the bonded boundary is much smaller than the total difference of temperature between the mirror and the SPI for both cases. So bonding does not affect the designed efficiency of heat transfer of the suspension.

5. Summary and Conclusion
From the experimental data, a bonding area of 5.06 mm$^2$ is required by the method of direct bonding and a bonding area of 22 mm$^2$ is required by hydroxide-catalysis bonding for supporting
Table 3. Shear strength of bonding, required bonding area and estimated temperature step across the bonded boundary. $A_m$ denotes a minimum required area for supporting the mirror with four rods. Here we took the bonding area as twice of $A_m$ as a safety margin. Shear strength $\sigma_{\text{shear}}$ is assumed to be independent of temperature. $\Delta T_B$ denotes the estimated temperature step across the bonded boundary.

| Shear strength $\sigma_{\text{shear}}$ | Direct Bonding | Hydroxide-catalysis Bonding |
|--------------------------------------|---------------|-------------------------------|
| Bonding area $2A_m$                 | 28.4 MPa      | 6.53 MPa                      |
| Bonding area $2A_m$                 | 5.06 mm$^2$   | 22.0 mm$^2$                   |
| Thermal conductance on $2A_m$       | 20 W/K        | 6.6 W/K                       |
| $\Delta T_B$ for $\dot{q}=500$ mW | 25 mK         | 76 mK                         |

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