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Bonding Techniques for the Fabrication of Internally Cooled X-ray Monochromators

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ABSTRACT

At CHESS, 2500 W total are absorbed by the first crystal of the double bounce monochromators located on the A2 and F2 wiggler beamlines. In order to dissipate this absorbed power and deliver the highest X-ray flux to an end station, we have explored the technique of internally cooling the silicon first crystals with water channels. This technique brings with it the need for reliable mechanical joints between the silicon diffracting surface and a glass or silicon water manifold. The joint must have structural strength to resist the internal water pressure and the cyclic heat load, be vacuum leak tight for operation in UHV, and not act as a source of residual strain in the crystal lattice of the diffracting surface. We have explored four bonding techniques which have been tested for their suitability to monochromator fabrication: direct silicon to silicon bonding, anodic glass to silicon bonding, a variety of ceramic and die attach adhesives (alumina, zirconia, silica /silver) and metallic diffusion bonding / brazing. In this paper, we characterize each method with respect to the requirements of structural integrity (bond tensile strength), residual strain (minimal effect on diffraction quality) and vacuum compatibility.

Keywords: x-ray monochromator, silicon bonding, direct bonding, anodic bonding, field assisted bonding

2. INTRODUCTION

The first crystal of a double bounce monochromator is subjected to extreme heat loads when used on a wiggler beamline. The two 24 pole wigglers at CHESS produce x-ray beams of roughly 2500W with a stored beam current of 100mA (a typical operating current.) This power applied to the inclined first crystal gives a surface power density of almost 30 W/mm^2. As improvements are continuously being made to CESR (Cornell Electron Storage Ring) the stored beam currents are increasing beyond the capability of the simple contact cooled monochromators currently used (1 or 2mm thick Si <111> wafers cooled through contact with a water-cooled copper block with a thin liquid layer of indium-gallium to reduce the thermal contact resistance.)

The science of high heat load wiggler monochromator design has advanced rapidly in the past decade as currents and power densities have increased significantly with the introduction of 3rd generation sources. Advances in cooling techniques including cryogenics[1], liquid metals[2], and microchannel configurations[3] have kept pace with ever increasing heat loads. Still fabrication induced strain is often a limitation to the implementation of advanced cooling schemes[4]

due to the lack of a reliable silicon bonding technique.

Proposed in section 4 is a simple 1mm wide channel, internally cooled design that is adaptable to a variety of coolants and channel geometries for future tests. Initial goals are to prove the fabrication techniques and pinpoint design challenges for optimization and hence provide a performance baseline for future work. Figure 1 schematically shows the geometry of the cooled top piece and the thick water manifold.

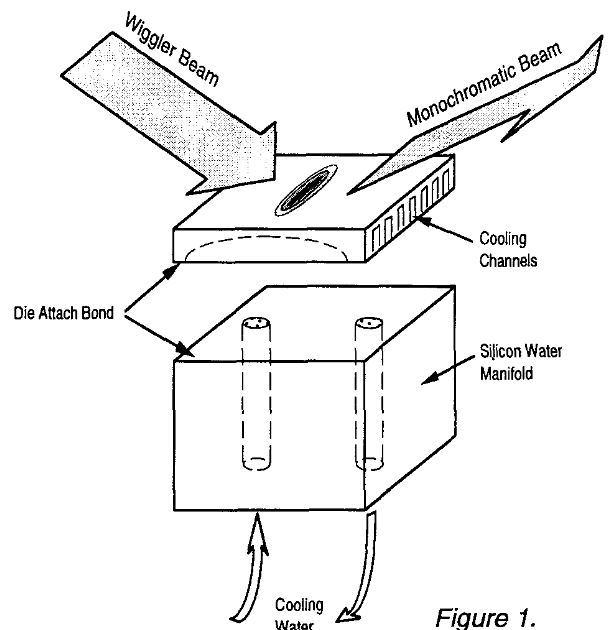


Figure 1.

3. SILICON BONDING TECHNIQUES

To increase the heat removal from the thermally distorted diffracting surface of the monochromator cooling channels should be placed directly below the surface. For optimal heat transfer, channel dimensions tend to shrink and fabrication of channels by core drilling becomes impossible. In order to fabricate small channels, wet orientation dependent etching[5,6] or diamond plunge cutting saws are required. Using this cooling channel configuration we are forced to assemble the monochromator from two halves bonded together with the channels cut into the underside of the top diffracting piece. For internally cooled monochromator applications, the bond is required to exhibit the following properties: minimal strain induced in the crystal, radiation resistant for long life, and hermetic to avoid coolant leaks to the vacuum system. Significant strains can be introduced into a monochromator crystal during its fabrication and bonding. Normally the strain from fabrication can be etched away, but the bonding strain is intrinsic to the required processing and cannot be removed. A hermetic seal is required to unsure there is no possibility of a water to vacuum leak since the monochromator is operating in a UHV chamber. In the design presented here we rely on the bond to be vacuum leak tight, but beyond the prototyping phase an air guard vented to atmosphere will be implemented in the monochromator designs to avoid direct water to vacuum joints. The bond tensile strength must be sufficient to resist the load imposed by the pressurized coolant flowing through the channels. The required bond strength including a safety margin is generally quite low (80psi) for reasonable coolant pressures. A diverse body of bonding techniques were selected for exploration in this paper for reasons of their prominence in the literature: direct bonding[7,8] and anodic bonding, the lack of results in the literature: metallic diffusion bonding, or their previous application to high heat loaded monochromators within the lab: die attach paste (DAP) adhesives. The following techniques are by no means inclusive, but representative of possible bonding solutions

3.1. Direct Silicon to Silicon Bonding

Direct bonded micromachined (wet chemical etched) silicon devices have been successfully

fabricated for laser diode heat exchanger applications by Hunt et al.[9] If this same technique is to be used in the fabrication of x-ray monochromators, some questions which were left unanswered in the laser diode application must be explored. First, the wet chemical etch which was used to machine the cooling channels can only be used with <110> oriented wafers. In many x-ray applications, the diffracting surface of the monochromator must be <111> oriented. Also, in the laser diode application, the bonded wafers were 1 mm thick whereas it may be necessary to use thicker wafers when fabricating an x-ray monochromator. And, finally, the issue of the lattice strain resulting from the bonding process has not been addressed. By way of preliminary tests, we explored whether wafers of different orientation could be bonded together and we also explored the feasibility of bonding together 5 mm thick wafers and used a synchrotron x-ray beam to measure the strain induced in the diffracting surface of the 5 mm thick wafers.

All bonding was carried out at the Cornell Nanofabrication Facility using a modified RCA cleaning procedure, omitting the final HF dip, following the technique of Hunt. In the final deionized water rinse, the resistivity is raised above 16 MΩ-cm. After blow drying with dry nitrogen, the wafers were pressed together, at room temperature, inside the class 10 laminar flow hood where the cleaning took place. After this the bonded pairs were annealed at 800°C for 1 hour in a dry N_2 ambient. We were able to bond 500µm thick by 75mm diameter Si <111> wafers together with approximately 80% coverage as viewed with an infrared transmission microscope. We were also able to bond a 500µm thick by 75mm diameter Si <100> wafer to a similar Si <111> oriented wafer with comparable results. No x-ray measurements were made of these CZ grown wafers.

With 5 mm thick by 100mm diameter Si <111> float zone wafers, the bond coverage was only approximately 10%. The lattice strain at the surface of these wafers was measured at the CHESS F3 station by taking rocking curves before and after bonding with an x-ray beam whose footprint on the wafer measured 25mm x 32mm. Before bonding, a rocking curve taken at 8.33 keV measured 9.7 arcsec FWHM. After the partial bonding, the same surface of the same wafer, when measured with 11.2 keV x-rays this time, gave a rocking curve of 7.9 arcsec FWHM.

While partially bonded 5mm thick wafers showed negligible strain induced from the bonding process, it remains to be seen whether fully bonded wafers will show the same results. The fact that direct bonding seems to work regardless of orientation opens up the possibility for the use of wet chemical etching as a method of fabricating cooling channels in an x-ray monochromator with a Si <111> diffracting surface. All of the wafers used in these tests were commercially available. No measurements of their surface roughness or flatness were made. It could be that in order to bond thicker wafers, flatness beyond what is commercially available must be obtained. We are currently exploring this area at CHESS.

3.2. Anodic Silicon to Glass Bonding

Since Wallis and Pomerantz[10] reported in 1969 on their development of a simple technique to bond silicon to glass numerous applications in the microelectronics industry have been re-

ported. The bonding process, called anodic bonding or field assisted bonding, utilizes the electrostatic attraction between the silicon and glass to bring the surfaces into close proximity allowing chemical bonding to take place. The experimental set up is almost trivial. The silicon and glass surfaces to be bonded are polished to a smooth, flat surface and placed in contact on a grounded hot plate. The assembly is heated to 400°C (for silicon to glass) and a DC voltage of -1000V is applied to the glass. The bond forms immediately, propagating visibly outward from the high voltage probe and fully seals the substrates within 1 to 5 minutes.

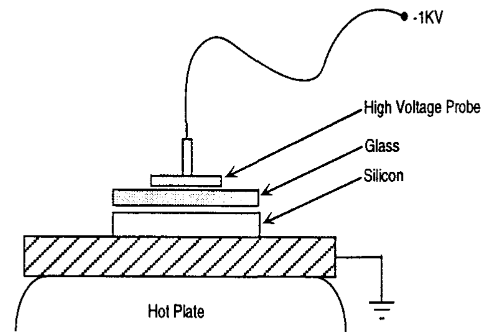


Fig. 2- Anodic bonding experimental setup

Our experimental setup is shown in figure 2 and was at first set up in a class 100 cleanroom to prevent surface contamination, but further tests proved that with careful cleaning (solvent rise and blown dry with N_2) and handling of the substrates the anodic bonding process could be carried out in still room air without adverse effects. The commercially available 50mm x 250μm silicon <111> wafers and glass disks (Corning 7740 Pyrex) used for our tests proved sufficiently well polished and bonded readily.

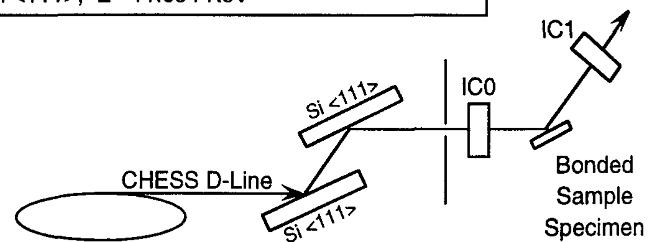


Fig. 3- Strain measurement experimental setup.

In order to characterize the lattice strain induced in the silicon after bonding with the glass, we used the double bounce monochromator setup (see figure 3) on the CHESS D-line beamline and rocked the bonded specimen. Strain was measured by comparing the rocking curve width using 11.56KeV x-ray (Pt L_{III} edge) before and after the bond took place, with the difference being the increased strain due to the bonding. The illuminated area of the crystal was 10mm x 3mm and centered on the wafer. The rocking curve widths were minimized with respect to the chi tilt and all of the samples were tested in the same manner to allow for comparisons between the bonding methods.

Table 1 - Anodic Bond Comparison

Glass Type Bond	Silicon Thickness (mm)	Glass Thickness (mm)	Rocking Curve Width (FWHM) (Arc Seconds)	Multiples of Unbonded Wafer (FWHM)
Si <111>	0.25	-	8.0	1.0
Si - 7740	0.25	1.00	49.4 ±4	6.2
Si - 7740	3.0:	1.00	19.5 ± 1	2.4

Due to the dissimilar thermal expansion coefficients of the silicon and glass used, the strain levels are quite elevated in the silicon as shown in table 1. Sample coupons tested used thick 1mm glass to bond to the thin 250μm silicon wafer, causing the silicon to be highly strained. When bonded to a more realistic 3mm thick silicon wafer the induced strain was reduced to a level reasonable for monochromator fabrication. Ideally sandwich structures with very thin or even sputtered (1-2μm) glass layers could be used to further reduce the bonding strain. Re-

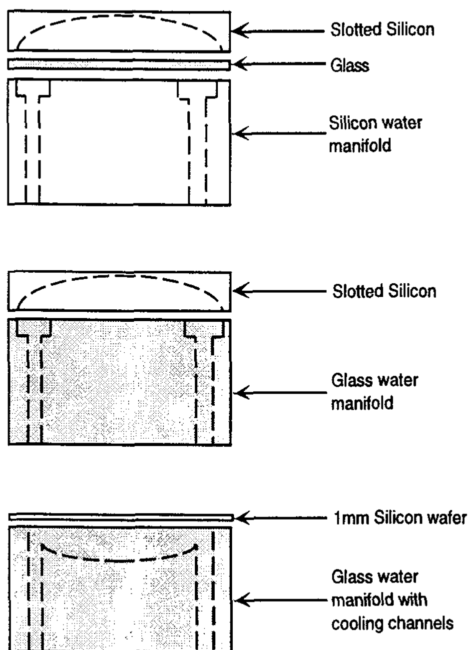


Fig 4 - Three possible anodically bonded internally-cooled monochromator configurations

ductions in the strain may also be possible through the use of more appropriate glasses: Corning 7570 a low viscosity glass that softens at lower temperatures than the Pyrex, and Corning 1737 a glass with a thermal expansion tailored to match that of silicon for use in flat panel display devices.

With reductions in bonding strain this technique holds much promise for monochromator fabrication due to its ease of processing, high bond strength, and hermetic nature. Three prototype monochromators are proposed for implementation using anodic bonding as shown in figure 4. The first, a sandwich structure, would require two bonds, but should minimize the strain due to the possibility of using very thin glass layers, the other designs require only a single bond but without a glass well matched thermally to silicon could severely strain the thin crystal.

3.3. DAP and ceramic adhesives

Perhaps the ideal bonding solution would be the use of a liquid adhesive to join the silicon pieces. The simplicity of processing an adhesive and lack of surface preparation and polishing makes it very attractive for rapid prototyping of new designs. Unfortunately the harsh radiation environment limits the selection of an adhesive to ceramic based cements of which we selected a number to test. Epoxies and polymer-based DAP can survive only short periods of time exposed to the high radiation dosages experienced in the monochromator chambers. Two commercially available die attach pastes Johnson-Matthey JM6100 and JM4613 commonly used in the microelectronics industry for packaging were tested. The Johnson-Matthey DAP is composed of finely powdered silver and glass with an organic solvent binder; the JM6100 and JM4613 differ only in glass type and composition. The solvent is completely driven out during a atmospheric bake of the assembly and the remaining glass-silver mixture wets the silicon surfaces and forms a strong solid bond. The thermal firing cycle determines the porosity of the final bond. Rapid firing causes rapid evolution of the solvent and the resulting formation of worm holes by the gases escaping through the adhesive layer resulting in water leak paths.

For comparison ceramic adhesives from Cotronics (Resbond 904 - a zirconium oxide based adhesive) and Aremco (516 - an aluminum oxide based adhesive) were also tested as low cost alternatives to the DAP.

All adhesives were fired as per the manufacturers instructions in an air atmosphere, computer controlled furnace for precise processing. The DAP samples were fired as slowly as possible within the recommendations of the manufacturer to avoid pinhole formation. When

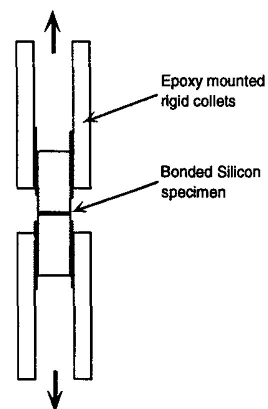


Fig 5- Tensile test experimental setup.

possible samples were run together to avoid processing variations. Due to the lack of tensile test data available for the ceramic adhesives, specimens were fabricated and loaded to failure (figure 5.) Ultimate tensile strengths were calculated from the specimen geometry and failure load and are given in table 2. All but the Aremco 516, which broke easily during routine handling, exhibited strengths beyond the required design limit. Due to the very limited number of samples tested these data should only be considered as baselines strengths.

Table 2 - Die Attach Pastes and Ceramic Adhesive Comparison

Adhesive	Supplier	Composition	Bond Strength (PSI)	Rocking Curve Width (FWHM) (Arc Seconds)	Multiples of Unbonded Wafer (FWHM)
-	Virginia Semi.	Si <111>	-	8.0	1.0
JM6100	Johnson-Matthey	Silver-Glass	111	14 ± 1	1.8
JM4613	Johnson-Matthey	Silver-Glass	322	35 ± 1	4.4
#904	Cotronics	Zirconia	126	57 ± 1	7.1
#516	Aremco	Alumina	0*	236 ± 2	29.5

Inspection of the broken bond surfaces reveal the porous nature of the bonds, with numerous voids and passages, leading us to believe that hermetic seals would be difficult with any of the four adhesives tested due to the evaporation of the liquid binder. Actual tests of the DAP with pressurized water have proven that the DAP absorbs moisture and weeps water droplets and additional coatings of DAP do not reduce the leakage.

In order to gauge the strain induced in the bonded crystals, a sample sandwich structure consisting of two Si <111> wafers 50mm x 250µm thick were bonded together and tested using the same method applied to the anodic bonding samples. An adhesive layer 75µm - 125µm thick was applied to the surface of one wafer, bonded to a second identical wafer, and processed as recommended (see appendix for processing parameters) The strain measurements, summarized in Table 2 show that the alumina and zirconia based adhesives severely strain the crystals making them unsuitable for monochromator fabrication. The strain induced by the DAP is acceptable, though higher than expected showing the strain sensitivity of the thin wafers. The prototype monochromator presented in section 4 (using JM4613) had a 10KeV rocking curve width of 15 arc seconds, substantially less than that of the coupons. The JM6100 shows particularly good results and could be promising for use in bonding applications where hermaticity is not critical, but a simple structural bond is.

3.4. Metallic interlayers

Brazing or diffusion bonding of silicon with a metallic interlayer was not initially considered due to the possible introduction of large strains into the crystal. Differences in the thermal expansions of a metallic interlayer and the Si substrates to be bonded would be expected to create large strains in the crystal lattice. Otherwise vacuum brazing has many potential benefits in terms of processing due to its simplicity

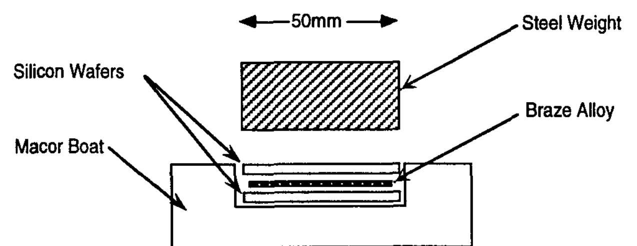


Fig. 6- Detail of the experimental braze fixture for wafer coupon tests.

and lack of stringent surface preparation. If the surfaces fully wet then very strong and hermetic seals would be expected. Inspection of the binary phase diagrams led to the decision to attempt a diffusion bond of silicon to silicon with a 90μm silver foil interlayer. Due to the high solid solubility of aluminum in silicon a 25μm Al foil was also selected for the test. Initially we were concerned that the native oxide layer on the silicon may prevent sufficient wetting by the metals and attempted to use a 25μm titanium foil as a bond material due to its active wetting nature. These are by no means the only possible braze alloys and in the future additional lower temperature, and hopefully lower strain alloys will be explored.

Table 3 - Metallic Bond Comparison

Metallic Bond	Hold Temp (°C)	Hold Time (min.)	Bond Temp (°C)	Hold Time (min.)	Wetting Qualitative	Rocking Curve Width (FWHM) (Arc Seconds)	Multiples of Unbonded Wafer (FWHM)
Si <111>						8.0	1.0
Si -Ag	800	15	850	5	Excellent	30 ± 8	3.8
Si - Al	550	15	600	5	Excellent	69.8 ± 3	8.7
Si - Ti	-	-	800	120	Poor	585	73.0

Commercial silicon wafers were used as coupons to test the bonding properties of the various combinations. Two 50mm x 250μm wafers were placed in a Macor ceramic boat (figure 6) with their polished surfaces facing each other and the metallic foil in between. A 500 gram stainless steel weight was used to insure close contact between the metal foils and the silicon wafers. The boat was heated in a quartz tube furnace evacuated to 1×10^{-6} torr and processed as listed in table 3 for each of the attempted bonds.

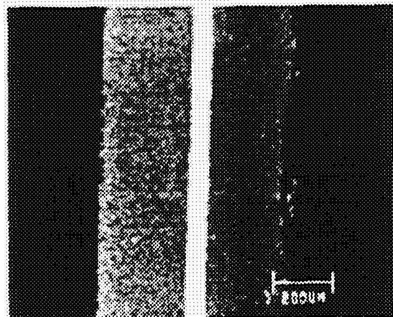


Fig 7a - Si-Ag-Si bond

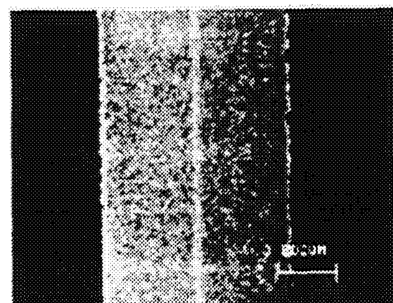


Fig 7b - Si-Al-Si bond

Sectioning of the silver and aluminum bonding coupons (figure 7a, 7b) shows a complete and uniform bond indicating excellent wetting of the silicon. Where complete wetting is visible we expect high strength hermetic bonds as is typical of vacuum brazing. Titanium wetting of the silicon was poor and non-uniform, resulting in a low bond strength which allowed the wafers to be pulled apart by hand. The rocking curve widths of the brazed coupons were measured in the same manner as before. As can be seen from the results summarized in table 3, the silver braze alone did not severely strain the crystal lattice.

4. MONOCHROMATOR DESIGN

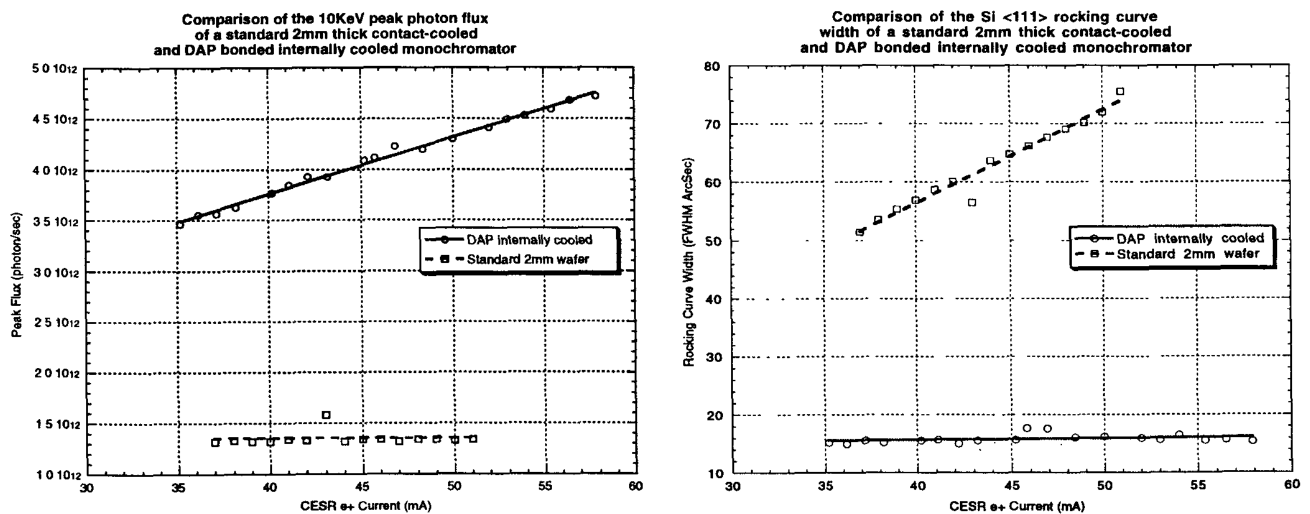
In parallel with the experimental development and comparison of the various bonding techniques a prototype monochromator was built. The crystal design was intended to be a simple first step at producing internally water-cooled monochromators in order to perfect the technologies of cutting, channeling, and bonding two separate silicon pieces. This first generation prototype was fabricated without the benefit of the results presented here to prove the effec-

tiveness of the internal cooling channels and pinpoint critical development issues. The diffracting surface, with cooling channels machined from the bottom, is connected to the manifold using the JM4613 DAP previously used by Bilderback[11]. Pipe inserts feed water into the silicon manifold using compression sleeves to force indium wire seals into contact with the inside surface of core drilled holes. The seal is made far (25mm) from the diffracting surface and a bulkhead provides for strain relief and precise positioning of the pipe inserts. Chilled, filtered water flowing at 1-2 gpm and at 30 psi is used as a coolant.

The top piece was machined with an additional 20mm of silicon above the cooling channels in an attempt to minimize residual strain from the bonding processing. This material was removed after bonding leaving a 1mm thick hot wall. Even after multiple coatings of the DAP, reliable vacuum leak-tight joints proved elusive and an epoxy coating (Hysol RE2039) was applied so that testing could continue. The coating proved to have a lifetime of roughly 2 months before it hardened and began leaking.

The prototype monochromator was tested on the F2 wiggler station and compared with the previously used contact cooled monochromator. In order to characterize the performance of the two monochromators the full F2 wiggler beam at 10keV was used without slits or filters. Under identical conditions the rocking curve widths and the peak photon throughput of the crystals were measured as a function of the beam current. If the crystal was performing ideally and without any thermal distortion the peak photon flux would be a linearly increasing function with increasing currents and the rocking curve width (FWHM) would remain unchanged. As can be seen in figure 8 the flux from the contact cooled crystal has saturated at low currents and the rocking curve width (a good measure of the thermal distortion) increases with current. The internally cooled monochromator shows significantly improved performance at these power loads, with photon fluxes a factor of 3 or more than the contact cooled design. The rocking curve width displays a flat response even though it has been broadened by the introduction of some strain (roughly twice the ideal width) during the DAP processing, but far better than expected from the sample coupons.

Fig. 8



With these promising initial results a second generation of monochromators are being built incorporating the silver metallic bond and the anodic silicon-glass bond for improved lifetime in the harsh radiation environment. While some residual bonding strain is expected, cooling improvements over the existing contact cooled design should make up for the increased manufacturing strain.

5. CONCLUSIONS

Without an effective silicon bonding technique our ability to fabricate the next generation of internally cooled wiggler optics is limited. In order to determine a suitable bonding technology a variety of bonding techniques were attempted and characterized. While none of the bonding methods proved to be without drawbacks a number were acceptable for monochromator fabrication since the wafers coupons used are overly sensitive to induced strain. The use of thin wafers for testing purposes was convenient, but not realistic due to the much thicker silicon sections used in actual monochromators. This can be seen with the anodic bonding results where the 250μm thick wafers are highly strained while the more realistic 3mm thick silicon sample was far more resistant to strain. Prototype monochromators built with the these bonding methods strain levels should be well within the acceptable range of 5 - 10 arc seconds.

The direct silicon to silicon bonding technique shows much promise and additional work to produce a prototype full scale monochromator is underway. All of the bonding requirements are met with the caveat that the processing is difficult with the proposed monochromator configuration. In order to successfully bond thick wafers together super-polished wafers are required and further processing to cut channels and drill holes appears to introduce unacceptable levels of surface contamination, preventing successful direct bonds. A simple modification to the design to encapsulate cooling channels with a thin cover wafer may be more appropriate (fig. 9) and allows the thin wafer to deflect and bond regardless of irregularities and particulate on the grooved manifold surface.

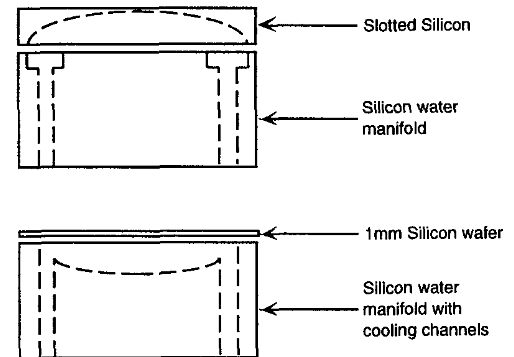


Fig. 9- Two possible direct bonded monochromator configurations.

The anodic bonding is a fascinating development and will be further developed using glasses better matched to the thermal expansion of silicon. Already we have reduced the processing temperatures required to form a successful bond from 400°C to 250°C and this is expected to reduce the strain level in the crystals. Recent reports[12] have indicated that bonding at room temperature is possible using sputtered glass thin films further reducing the thermal mismatch strain.

Die attach paste has been shown to be acceptable in terms of bond strength and radiation resistance, but because of its porous nature it is not UHV compatible and the epoxy coatings

applied for leak tightness are unacceptable for long term use. Crystal strain measurements again show the sensitivity of the thin wafer samples and the reduced strain of the full scale prototype. The ease of application and processing of the DAP has been a benefit during the prototyping phase allowing us to quickly build monochromators for testing. The ceramic adhesives tested are also attractive in terms of their simple processing and application, but their strain characteristics make them far less suitable than the DAP without solving the hermaticity issue.

Metallic bonding was not initially pursued due to the anticipated high strain levels as seen with the silicon-titanium bond, but the silver bond appears to have potential applications where bond strength and hermeticity are vital due to its relatively low induced strain. Further thick silver bond specimens will be used to test its usefulness with full size prototypes.

The bonding technologies can also be used in situations beyond the internal cooling channel application. A sagittal focusing monochromator (used as the second crystal of a double bounce monochromator) capable of focusing at higher energies than previously possible was designed and constructed. As shown in figure 10 and 11, it consists of a highly flexible base with a series of bonded diffracting teeth. The original design shown above it is compromised by the fact that increased flexibility obtained by increasing the groove width between the teeth also reduces the flux throughput. By constructing the monochromator from two separate crystal and bonding them together we were able to increase the flexibility allowing the base to be bent to a tight radius for high energies while maximizing the diffracting surface area. The bond was made by Johnson-Matthey DAP JM4613 since the requirement of hermeticity did not apply to this design. The monochromator has been successfully tested and a publication dealing with its design and performance will follow.

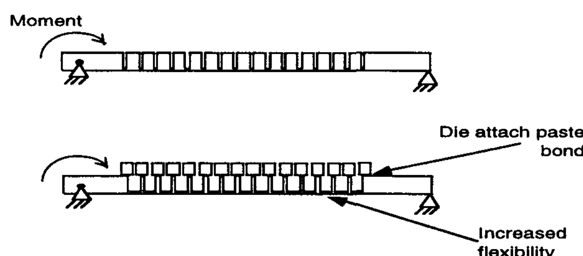
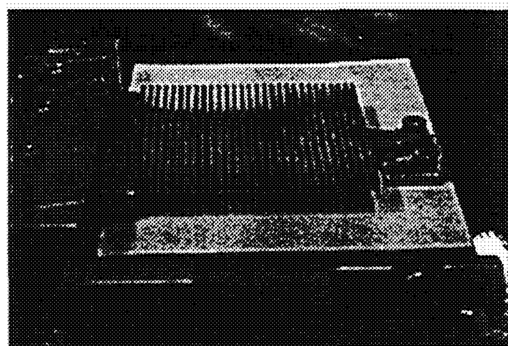


Fig. 10 - Traditional and high energy sagittal focusing monochromators

Fig. 11 - Completed monochromator



6. ACKNOWLEDGMENTS

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7. APPENDIX

Suppliers:

1) Cotronics Corp., 3379 Shore Parkway, Brooklyn, NY 11235
2) Aremco Products Inc., 23 Snowden Ave., Ossining, NY 10562
3) Johnson-Matthey Electronic Materials, 10080 Willow Creek Road, San Diego, CA 92131

Ceramic Adhesive Furnace Processing:

Cotronics 904
1°C/minute to 70°C, hold for 240 minutes

Aremco 560
5°C/minute to 93°C, hold for 120 minutes
5°C/minute to 204°C, hold for 120 minutes
5°C/minute to 371°C, hold for 120 minutes

DAP JM 4613 / JM6100
1°C/minute to 50°C, hold for 120 minutes
1°C/minute to 100°C, hold for 180 minutes
1°C/minute to 430°C, hold for 60 minutes

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