Diamond for High-Heat-Load Synchrotron X-Ray Applications

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ABSTRACT

Synchrotron facilities worldwide provide scientists with useful radiation in the ultraviolet to the x-ray regime. Third-generation synchrotron sources will deliver photon fluxes in the 10^{15} photons/s/0.1%BW range, with brilliance on the order of 10^{18} photons/s/0.1%BW/mrad²/mm². Along with the increase in flux and brilliance is an increase in the power and power densities of the x-ray beam. Depending on the particular insertion device, the x-ray beam can have total power in excess of 10 kW and peak power density of more than 400 W/mm². Such high heat loads are a major challenge in the design and fabrication of x-ray beamline components. The superior thermal and mechanical properties of diamond make it a good candidate as material in these components. Single crystal diamonds can be used as x-ray monochromators, while polycrystalline or CVD diamonds can be used in a variety of ways on the front-end beamline components. This paper will discuss the issues regarding the feasibility of using diamond in third-generation synchrotron beamline components.

Keywords: synchrotron optics, high-heat load optics, x-ray diamond optics

INTRODUCTION

Synchrotron sources worldwide provide scientists with useful radiation in the ultraviolet to the x-ray range. Synchrotron radiation is produced by charged particles moving at relativistic speeds around a storage ring. Depending on the energy of the charged particles, the radiation produced can range between tens of eV to hundreds of keV. This paper will focus solely on the use of diamonds at hard x-ray (>1 keV) synchrotrons.

Historically, early synchrotron radiation was obtained parasitically from machines designed for high energy physics experiments. For example, the Cornell High Energy Synchrotron Source (CHESS), runs parasitically off of the Cornell Electron Storage Ring (CESR) where high energy physics experiments are performed. Later, so-called second-generation machines such as the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory in New York, were built. These machines are dedicated to the production of synchrotron radiation.

There are currently three third-generation synchrotron facilities worldwide, that are in various stages of construction: the European Synchrotron Radiation Facility (ESRF) in Grenoble, France (construction completed, in operation), the Advanced Photon Source (APS) in Argonne, Illinois, USA, (under construction), and the Super Photon Ring (Spring-8) in Harima, Japan, (under construction). The common features of these third-generation synchrotron facilities are a low particle beam emittance and the use of insertion devices (IDs). Insertion devices are periodic magnetic structures that are inserted into the path of the particle-beam. The insertion devices cause the particle beam to wiggle or undulate in the transverse direction. Depending on the amount of deviation (measured by the deflection parameter $K = 0.934 l B$, where l is the period of the magnetic device and B is the peak magnetic field) caused by the ID, the ID is called a wiggler (large deviations) or an undulator (smaller deviations). By adjusting the

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properties of the magnetic field (strength and period), the subsequent radiation can be tailored to the needs of the researcher. Third-generation synchrotron facilities worldwide will provide the researcher with x-ray radiation of unprecedented brilliance ($\sim 10^{18}$ photons/s/0.1% BW/mm²/mrad²).

In order for the researcher to fully utilize the radiation, it must be properly and safely transported to the research station and some of the x-ray beam parameters, such as beam size, energy, and intensity, must be easily manipulated according to the needs of the particular experiment. This is usually done by the "front-end" and the x-ray optics. A typical synchrotron front-end is shown in figure 1. The front-end usually includes shielded, evacuated beampipes, collimators, slits, shutters, filters, windows, and photon beam position monitors (PBPMs). Collimators and slits are used to properly aperture the beam to the desired size and to reduce stray radiation. Windows are needed to isolate the storage-ring vacuum (usually in the 10^{-9} - 10^{-11} torr range) from the experimental station (which can be at atmosphere). Filters are sometimes used to reduce the power and power density of the beam or to cut off the lower energy spectrum. Beam position monitors are used to track the motion of the photon beam due to the particle beam motion. Figures 2 and 3 show the energy spectrum of wiggler A and undulator A (at $K=2.17$) for the APS. As can be seen, the radiation spectrum is very broad. Most experiments require a much narrower band of radiation, usually in the eV or meV range. X-ray optics are used to accomplish this. The two major x-ray optical components are the monochromator and the mirror. X-ray monochromators allow the user to select a particular energy from the incoming white radiation. X-ray mirrors allow the user to cut off the high energy photons. Both can be configured to focus the beam.

Along with the increased x-ray beam brilliance and flux are major heat load problems associated with the high power and power densities of the beam. Table I [1-4] summarizes the photon beam power and power densities at some major synchrotron facilities. It is clear that the front-end components and x-ray optics will have to be carefully designed and properly cooled. This paper will acquaint the reader with some of the heat load issues facing third-generation synchrotron beamline designers and will discuss some of the design and function of the front-end and x-ray optical components. The use of diamond in these components at third generation synchrotron sources will be described, and comparisons will be made to conventional materials. In particular, the properties of diamond that are required for each application will be mentioned.

FRONT-END COMPONENTS

The design of front-end components for third-generation synchrotrons is a major challenge. Photon and safety shutters have to be able to properly block the incoming radiation without any leaks. This means that they have to absorb the total power of the beam. Beamline windows are directly in the path of the beam, and, even though they may not absorb a significant amount of power, they have to maintain their vacuum-seal integrity. Although in normal operation, slits, collimators, and beam position monitors should not see the hottest part of the beam, these components have to be designed to handle the full beam. This is due to the possibility of a particle beam mis-steer, thereby causing the photon beam to deviate from its normal course.

Table II [5-9] compares the thermal and mechanical properties of diamond with some materials commonly used for synchrotron-beamline components. Figure 4 compares the x-ray linear absorption coefficient, μ , of different materials. Figures 5 and 6 show the absorbed power as a function of thickness of several materials for the APS undulator and wiggler sources, respectively. This information will be useful in the discussion of the

feasibility of using diamond on the synchrotron beamlines. The following paragraphs describe some of the front-end components and how diamond has been or is being considered for use.

Windows

As mentioned above, windows serve to isolate the storage-ring vacuum from the user experimental station. Windows must provide good transmission of the x-rays. A typical window consists of the window itself, brazed or welded to a cooled copper structure on a Conflat flange. Traditionally, windows are made of beryllium. The low absorption coefficient and good mechanical properties make beryllium a good choice for window material. Usually, a thin graphite filter is placed upstream of the window to reduce the thermal load on the window. For example, the current design of the APS wiggler beamline window assembly consists of a 0.6 mm thick graphite filter followed by two 0.25 mm thick beryllium windows.

Table II compares some of the properties of beryllium, silicon, and copper to those of diamond. In terms of transmission, figure 4 shows that beryllium is clearly better, especially at energies below 10 keV. However, diamond has superior thermal properties. Its thermal conductivity is almost 10 times better than that of beryllium. Figures 5 and 6 show that at the APS insertion device beamlines (even for a very thin window (~0.1 mm)), the absorbed power is still several hundred watts. The dissipation of this absorbed power in the window is nontrivial because the only thermal paths are through the brazed perimeter of the window material.

Preliminary estimates suggest that, without taking safety factors into account, a 7 mm x 7 mm x 0.15 mm diamond window can handle the APS undulator source. If beryllium is used, a 0.3 mm graphite - 0.25 mm beryllium window assembly might work [10,11]. The diamond will absorb about 480 W of power, while the graphite-beryllium assembly will absorb a total of 577 W (530 W in the graphite, and 47 W in the beryllium). At 5 keV, the diamond window transmission is about 38% while that of the graphite-beryllium combination is about 23%.

It is as yet unclear if a diamond window would be better than the conventional graphite-beryllium window assembly. More reliable and accurate calculations are difficult due to the large range of thermal conductivity of CVD diamond and also due to the lack of good data regarding the heat transfer through the diamond-metal interface. Also, many questions pertaining to reliability and failure modes of these windows remain to be answered. For example, due to the ductility of beryllium, its initial failure would most likely be buckling, whereas diamond would simply fracture. While a fractured diamond window would obviously not hold vacuum, a buckled beryllium window might. Continuing studies regarding the feasibility of using diamond windows in undulator beamlines are underway at the APS.

Table I: Power and power densities at several synchrotron facilities worldwide assuming 100 mA particle current (except for NSLS).

Source	Ring particle and energy	Total power ^a (W)	Peak power density (W/mm ²) at typical front-end location ^b	Peak power density (W/mm ²) at typical x-ray optic location
NSLS X25 focused wig ^c	e ⁻ , 2.5 GeV	1600 (75)		200 (at focal point)
CHESS F-2 wiggler	e ⁺ , 5.4 GeV	2000	34 (at 12 m)	15 (at 18 m)
ESRF undulator ^d	e ⁻ , 6 GeV	855	92 (at 15 m)	23 (at 25 m)
ESRF wiggler ^e		5600	36 (at 15 m)	20 (at 25 m)
ESRF focused wig. ^f		2000 (280)	200 (at 15 m)	3500 (at focal point)
APS undulator A	e ⁺ , 7 GeV	3400	460 (at 17 m)	150 (at 30 m)
APS wiggler A		7400	250 (at 17 m)	80 (at 30 m)
Spring-8 undulator	e ⁺ , 8 GeV	5600	836 (at 20 m)	273 (at 35 m)
Spring-8 wiggler		17700	404 (at 20 m)	132 (at 35 m)

Notes:

- a: Total power produced by ID. The value in parenthesis is total power after windows, at the focal point.
- b: Peak power density. Distance from source (meters) is in parenthesis.
- c: Assuming 200 mA ring current.
- d: TROIKA beamline.
- e: Materials Science beamline.
- f: ID 9, white-beam beamline.

Sources: References 1 through 4.

Table II: Properties of beryllium, diamond, and silicon at room temperature.

Property	Beryllium	Diamond	Silicon	Copper
Atomic number, Z	4	6	14	29
Density (g/cm ³)	1.82	3.516	2.330	8.93
Thermal conductivity (W/cm-K)	2.18	21	1.25	3.98
Thermal expansion coefficient (1/K)	12x10 ⁻⁶	1.2x10 ⁻⁶	2.33x10 ⁻⁶	16.6x10 ⁻⁶
Melting point (°C)	1278	4300	1420	1084
Specific heat, C _p (J/Kg-K)	1831	520	705	386
Tensile strength (MPa)	227-621	>3000	NA	310
Yield strength (MPa)	186-413	NA	1240-2060	276

Source: References 5 through 9.

Slits, Collimators, and Shutters

Photon shutters allow the researcher to turn the beam on and off at their particular beamline. They are of primary importance in personnel safety systems. Slits and collimators are used to define the photon beam and to help reduce stray radiation. Shutters, slits and collimators are designed to absorb/stop the beam (or parts of the beam) rather than to transmit it. These components have to be built to the worst-case scenario, which is for the component to absorb the full power of the beam. Slits, collimators, and photon shutters are usually made from copper (for heat dissipation). A tungsten or Hevi-Met "safety shutter" is usually placed downstream of the photon shutter to absorb any high energy photons that may come through the photon shutter. Due to the intense heat loads at third-generation synchrotrons, many of the slit and collimator assemblies are designed to intercept the beam at grazing incidence angles so as to reduce the surface power density. The result is that the components quite large. If the thermal conductivity of the target materials can be enhanced, for example, with a CVD diamond coating, it may then be possible to make these components more compact. This is important due to the space constraints in the front ends.

Photon Beam Position Monitors (PBPM)

A typical PBPM consists of two or four electrically conducting blades positioned at the edges of the photon beam. The beam position is measured by the relative amounts of photocurrent induced in each blade by the photoelectric effect. The blades are usually oriented with their edges (smallest cross-sectional area) facing the beam. (The length of the blade is parallel to the beam to minimize absorption.) In addition, an inclined edge is usually used to reduce the heat power density on the blade edge surface. Due to the possibility of particle beam missteering, these blades may in fact see the hottest part of the x-ray beam. Preliminary studies show that a CVD diamond blade will survive direct impingement by the APS undulator A x-ray beam, while conventional metallic blades would suffer physical damage [12].

Two prototype PBPMs with CVD diamond blades have been built by the APS [12,13]. One had an aluminum coating, while another had tungsten coating (for electrical conductivity and photoemission). Successful tests have been performed at NSLS and CHESS. In this design, the diamond blades were clamped to a cooled copper block. The superior thermal conductivity of diamond keeps the blades at reasonable temperatures. In addition, diamond's low thermal expansion coefficient may improve the PBPMs stability. The possibility of an integral window and PBPM has also been raised [13]. In this case, a series of individually isolated electrically conductive patterns would be deposited/coated on a diamond window. These would act as the photoelectric sensors in lieu of the metal blades.

Front-end issues

The major issue that has yet to be resolved regarding the use of diamonds in front-end components is that of the diamond-metal interface. For windows, a vacuum tight diamond-metal bond/braze is necessary. For PBPMs a metal coating on the diamond blades is necessary for electrical conduction and photoemission. For slits, collimators, and shutters, a diamond coating on metal may be used to improve thermal conductivity. The major problem here is the difference in thermal expansion coefficients between the diamond and the metal. These components are subjected to severe heat loads and high on/off duty cycles. The difference in thermal expansion coefficients would certainly affect the integrity of the diamond-metal contact. Although the use of intermediate coatings of other materials may alleviate this problem, care must be taken so that the thermal contact between the diamond and the base substrate is not affected by the additional coatings. The use of intermediate layers to better match the thermal expansion coefficients between diamond and metals is currently being studied at the APS.

X-RAY OPTICS

Perfect Single-Crystal Monochromators

Single-crystal monochromators are probably the most common optical element used to monochromatize the synchrotron radiation. Single-crystal monochromators work by diffracting the radiation from the crystalline planes according to Bragg's law: $2d \sin q = \lambda$ where d is the interatomic plane spacing, q is the incidence angle, and λ is the wavelength of the reflected radiation. In the hard x-ray (4-20 keV) regime, single crystals are most often used because the lattice spacing of many crystals (10^{-10} m) are comparable to the x-ray wavelengths (10^{-10} to 10^{-11} m). Depending on the size of the incoming beam and the incidence angle, the beam footprint on the crystal surface can be a few square millimeters to a few square centimeters. The number of crystalline planes that are responsible for the reflection is usually around 10^4 to 10^5 , which corresponds to a depth of a few microns. In order to obtain maximum efficiency and to maintain the incoming beam brilliance, the crystalline planes must remain perfect, flat, and parallel to one another over the whole diffraction volume ($\sim$ footprint on surface $\times$ penetration depth). The crystalline interplanar distance must also remain constant. Because the Darwin width or acceptance angle (Dq for which reflection occurs at a given wavelength) and the opening angles of the synchrotron radiation are usually on the order of a few arc seconds, the crystal must remain flat to the same level. Most monochromators operate in vacuum (10^{-6} - 10^{-9} torr) in order to reduce air scattering and ozone production by the x-rays. Thus, the monochromator material must be vacuum compatible. Obviously, the material must also be stable under the harsh radiation conditions.

Silicon is the most commonly used material in x-ray monochromators. Due to the success of the semiconductor industry, perfect, dislocation-free ingots of silicon are easily available in the required sizes. Furthermore, different growth directions of the silicon ingot (1-1-1, 1-0-0, or 1-1-0) can be obtained. Silicon is UHV compatible and does not deteriorate under radiation. Silicon is also machinable, which means that intricate cooling channels can be machined directly into the silicon. Until the development of third-generation synchrotron sources, the use of properly cooled, either directly or indirectly, silicon at room temperature was sufficient to tackle the heat load problem. However, with the advent of the third-generation sources, such schemes may not be sufficient.

This is where perfect single-crystal diamonds may make a significant impact in x-ray optics. Table II compares some thermal and mechanical properties of diamond and silicon. Clearly, diamond is the material of choice. (Beryllium is also a good choice; however, it is difficult to obtain good quality single crystal beryllium). Furthermore, due to the lower atomic number, for a fixed thickness diamond absorbs less power than silicon (see figures 4-6). A commonly used figure of merit is k/ma where k is the thermal conductivity, m is the linear absorption coefficient and a is the thermal expansion coefficient. At room temperature and with 8 keV x-rays, the figure of merit for diamond is 1430000 while that of silicon is 4000.

Several successful tests of diamond as a high-heat-load x-ray monochromator have been performed [14-17]. Figure 7 shows the mounting scheme for a water jet-cooled diamond monochromator [14]. This has been tested at the NSLS focussed wiggler beamline at normal incidence power densities of up to 260 W/mm^2 , with a total power of 63 W. The absorbed power was approximately 40 W. No thermally induced distortions were observed. Recently, at the ESRF, an indirectly edge-cooled diamond monochromator has been successfully tested at incoming beam normal incidence power densities of up to 3500 W/mm^2 with a total power of 280 W [17]. The power absorbed by the diamond in this case was about 9 W. Figure 8 shows the ESRF diamond mounting scheme. These early single-crystal tests of diamond are very encouraging. However, many important issues remain and will be discussed below.

Diamond Coatings on Thin Silicon Crystals

Because x-rays are usually completely reflected within several tens of microns of depth, thin silicon crystals can be used as monochromators. The advantage here is that thin crystals will transmit most of the incoming beam power. Thus, there is less heat to be dissipated by the crystal. However, the thin crystal geometry generally also means a reduced thermal dissipation path, which is usually the crystal edges. It has been suggested that diamond coatings can be used here to enhance the thermal conductivity [18]. The major challenge in this case is that of a strain-free coating of the diamond on a piece of silicon, which may be about a hundred microns or less thick. Strains may occur during the coating process or from the difference in thermal expansion coefficients under a thermal load.

X-ray Optics Issues

The early tests of single-crystal diamond in x-ray optics are very encouraging. However, several important issues remain:

1. Availability

In order to be of use as x-ray monochromators, the size of the perfect single-crystal diamonds must be at least several millimeters square and with a thickness of at least several tens of microns. An ideal size for the APS undulator beams would be about 10 mm x 10 mm x 0.5 mm. At the time of this article, the synchrotron community as a whole has only managed to secure a small handful (less than 10) of diamond pieces (natural or synthesized) of high crystalline perfection. The sources range from South Africa [15], to General Electric in the U.S. [16] to Sumitomo in Japan [19]. These are usually about 5 mm x 5 mm square and about 0.3 to 0.5 mm thick. Most suppliers are not familiar with the crystalline perfection requirements of the synchrotron community and usually do not have any method of checking the quality. As such, it has mostly been hit or miss when it comes to the crystallographic quality of the diamonds. Furthermore, topography of these "good" diamond pieces has revealed that most of them are not perfect over the whole piece. However, depending on the size and shape of the "good" areas, this may not be a problem.

2. Growth direction

A related issue is that of growth direction. Depending on the geometry used in the beamline, diamond (111) or diamond (110) may be more suitable. Plates of diamond (111) where the (111) planes are parallel to the surface are difficult to come by. Most synthetic diamonds tend to be grown with the (100) direction normal to the large surface. Although it is possible to use diamond (100), the throughput of the monochromator will be severely reduced due to the higher (hkl) reflection needed. For the diamond structure, the (100) reflection is forbidden, and if the (100) planes are to be used, it has to be a (400) reflection. This is disadvantageous because, in the 8-20 keV range, the Darwin width of the (400) reflection is about two times smaller than that of the (111) reflection. The smaller Darwin width leads directly to a smaller photon throughput.

3. Strain-free bonding

Even though diamond has superior thermal and mechanical properties, there is still the issue of removing the power that is absorbed by the diamond. Although this power may be only a few hundred watts (for the APS insertion devices), due to the size of the diamond, proper removal of this heat is essential. In the case of direct cooling, a diamond-metal seal is required to prevent a coolant leak. In the Hart-Berman design [14], the seal is achieved by carefully tensioning the Bellevue washers. It is tricky to provide the correct amount of force sufficient to achieve a coolant seal and at the same time not strain the diamond. For the case of indirect cooling, the diamond must be in good thermal contact with the cooling block. In the ESRF design [17], thermal contact is achieved via an indium-gallium eutectic, which also holds the diamond in

place by surface tension. The heat transfer between the diamond and the cooled copper block will be enhanced if a more intimate thermal contact can be established. Both designs, direct and indirect cooling, would clearly benefit if a good diamond-substrate (substrate can be metal, polycrystalline diamond, or CVD-coated metal) bond can be achieved. As mentioned above, it is essential that such a bond/braze be strain free.

4. Fabrication issues

Due to the deviation in the growth direction or the desire for an asymmetry in the monochromator (where the crystalline planes are not parallel to the surface), it is sometimes necessary to grind or lap the surface of the monochromator. Sometimes, it may also be necessary to polish the monochromator surface. This is usually needed in cases where the incoming beam impinges on the monochromator at grazing incidence angles. With silicon, these processes have been well characterized and proven. Furthermore, the strains induced in the silicon during cutting, grinding, lapping, or polishing can be easily etched away using a variety of procedures. However, much less appears to be known about the machinability of diamond, its resulting strains in the crystal, and the possible methods of removing these strains. Diamond does have an advantage over silicon in that, due to its hardness, there is probably less strain induced by cutting, grinding, or lapping.

SUMMARY

In summary, there are many possible applications of diamonds in synchrotrons worldwide, particularly at the third generation synchrotron facilities. These applications include the use of polycrystalline, CVD, and single-crystal diamond. While it is clear that it is the semiconductor industry that is the main driving force in the rapid development of the diamond growth industry, it is hoped that there will be sufficient interest by the diamond industry to develop or adapt the technology to the needs of the synchrotron community. In particular, it is hoped that large, perfect, single crystals of diamond will be readily available in the near future. Techniques for fabrication, machining, and bonding of diamond are also needed. Prototype tests (and planned tests) of front-end devices and x-ray optics incorporating single crystal, polycrystalline, or CVD diamonds at the ESRF, APS, and Spring-8 should provide some answers regarding the reliability and feasibility of these diamond devices in the synchrotron environment.

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Figure 1: Taken from reference 20. Elevation view of a typical APS ID front end: (1) All-metal ring isolation valve (2) Photon beam position monitor (3) Fixed mask (4) Photon shutter (5) Collimator (6) All metal slow valve (7) All metal fast valve (8) Photon beam position monitor (9) Fixed mask (10) Photon shutter (11) Filter assembly (12) Safety shutter (13) Collimator (14) Window. Dual photon shutters are required for safety purposes.

Figure 2: Spectral flux expected from the APS wiggler A (B = 1.0 T), with 100 mA ring current.

Figure 3: Spectral flux expected from the APS undulator A ($K = 2.17$), with 100 mA ring current.

Figure 4: Plot of linear absorption coefficient vs. energy. The relationship between the transmitted intensity and the incident intensity is: $I_{\text{trans}} = I_{\text{inc}} e^{-\mu t}$, where t is the thickness of the filter.

Figure 5: Absorbed power vs. thickness of material for the APS undulator ($K = 2.17$) beam.

Figure 6: Absorbed power vs. thickness of material for the APS wiggler ($B = 1.0$ T) beam.

Size of diamond is 8mm x 4mm x 0.1 mm

Figure 8: ESRF edge-cooled diamond mounting scheme. The diamond is held in thermal contact to the cooled copper substrate via an indium-gallium eutectic. The diamond stays in place by surface tension. The diamond is used in the Laue geometry - where the entrance and exit beams are on different sides of the crystal surface.