CVD Diamond Windows for Synchrotron Radiation Beamlines

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Abstract

Beryllium windows (Be) generally fulfill two functions as standard beamline components. One is the absorbance of low energy photons to reduce thermal load on other components down the beamline. The other is to separate the ultrahigh vacuum of the storage ring from the beamline or from the atmosphere, respectively [1]. On the other hand Be windows show adverse effects on coherence of synchrotron radiation in applications requiring a high beam quality. Degradation of beam quality due to the Be foil is mainly due to its roughness and thickness variation (results in phase shifts and thus in loss of coherence) as well as due to its surface imperfections and inclusions (results in diffraction and thus in beam speckles) [2]. To overcome these constraints diamond windows have been developed for the Swiss Light Source (SLS) beamlines at the Paul Scherrer Institut (PSI). Diamond foils are manufactured using chemical vapour deposition (CVD) technique and brazed on copper frames in high-vacuum. CVD diamond has excellent optical properties as compared to Be. A single CVD diamond window combines the functionalities of a thermal filter as well as of a vacuum and safety element which usually requires two separate Be windows. This is due to the excellent mechanical and thermal properties of CVD diamond. Results of numerical optimisation of the mechanical behaviour of the CVD diamond window in case of an air inrush, as well as its thermal behaviour during brazing, bake-out and synchrotron beam absorption will be given. Results of mechanical and optical tests performed on a prototype CVD window will also be presented [3]. After completion of these successful tests CVD diamond windows are installed on the last SLS beamlines (X02DA TOMCAT and X05DA Optics). Based on this design, an off-the-shelf diamond window is being brought to market through Diamond Materials GmbH [4].

1. Mechanical design

The CVD diamond window device is integrated on a double side DN63CF flange (fig.1). The free aperture for synchrotron radiation measures 18 x 6 mm. For example at a distance of about 10 m after the undulator source the resulting spot size would be 6 mm x 3.3 mm on the front end window. The diamond foil itself has an oval shape of 22 x 10 mm and a thickness of 100 µm or 200 µm. It is actively brazed (no metallization on CVD diamond required) on an OFHC (oxygen-free high conductivity) copper block in a high vacuum atmosphere. The copper block is designed to dissipate thermal power on the CVD diamond window (brazing, bake-out, synchrotron beam exposure) and to compensate for mechanical strain and stress during the brazing process. The complete design allows a baking temperature of up to 250°C. A separate U-shaped copper tube provides additional water cooling for the CVD diamond window while exposed to synchrotron radiation. Depending on the thickness (100 µm or 200 µm) the CVD diamond window can absorb a power between 100 W to 160 W at a standard SLS dipole magnet without being destroyed.
2. Mechanical properties of CVD diamond as compared to Beryllium

Mechanical properties of CVD diamond surpass those of Beryllium which makes it a preferred window material [4]. CVD diamond has a high thermal conductivity of about 2’000 W/mK which is about five times that of copper and ten times that of beryllium, respectively (fig. 2). This high conductivity allows higher surface power densities and results in reduced stress and strain as compared to a beryllium window. Both materials show a high transmittance for x-ray radiation at low energies due to their low atomic numbers (fig. 2). However, CVD diamond shows a reduced transmittance below 40 keV as compared to beryllium windows of the same thickness (fig. 3). On the other hand degradation of x-ray beam quality can be observed using beryllium windows due to defects, impurities, surface roughness and variations in thickness which results in beam speckles (diffraction) and in reduced coherence (phase shifts) [2]. Much higher quality of polishing and of purity as well as a higher degree of constant thickness is achieved with CVD diamond material as compared to beryllium. A significantly less degradation of quality of synchrotron beams is expected.
<table>
<thead>
<tr>
<th>Property</th>
<th>Diamond</th>
<th>Beryllium</th>
</tr>
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<tbody>
<tr>
<td>Atomic number</td>
<td>6</td>
<td>4</td>
</tr>
<tr>
<td>Hardness</td>
<td>12 000 – 15 000 kg/mm²</td>
<td>150 – 200 kg/mm²</td>
</tr>
<tr>
<td>Strength, tensile</td>
<td>&gt;1200 Mpa</td>
<td>310-550 Mpa</td>
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<tr>
<td>Density</td>
<td>3.52 g/cm³</td>
<td>1.85 g/cm³</td>
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<tr>
<td>Young’s modulus</td>
<td>1140 GPa</td>
<td>290 Gpa</td>
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<tr>
<td>Poisson’s ratio</td>
<td>0.069</td>
<td>0.075</td>
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<tr>
<td>Specific heat</td>
<td>0.52 J/gK</td>
<td>1.87 J/gK</td>
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<tr>
<td>Thermal expansion</td>
<td>1.1 ppm/K (at RT)</td>
<td>11.6 ppm/K (at RT)</td>
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<tr>
<td>coefficient</td>
<td>2.6 ppm/K (20-500°C)</td>
<td>15.0 ppm/K (25-500°C)</td>
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<tr>
<td>Thermal conductivity</td>
<td>2000 W/mK (at RT)</td>
<td>180 W/mK (at RT)</td>
</tr>
<tr>
<td></td>
<td>730 W/mK (at 500°C)</td>
<td>97 W/mK (at 540°C)</td>
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<td>Optical transparency</td>
<td>UV to far IR</td>
<td>Opaque</td>
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<tr>
<td>Resistivity</td>
<td>Insulator $10^{13}$ - $10^{16}$ Ωcm</td>
<td>Conductor 4.1 Ωcm</td>
</tr>
<tr>
<td>Melting temperature</td>
<td>at 1500°C diamond transforms to graphite</td>
<td>1285 °C</td>
</tr>
<tr>
<td>Toxicity</td>
<td>None</td>
<td>High (even small amounts can cause chronic beryllium disease)</td>
</tr>
</tbody>
</table>

Fig. 2: Comparison of mechanical properties of CVD diamond and beryllium

Fig. 3: Comparison of x-ray transmission of Beryllium (dashed) and of CVD diamond (solid)
3. Finite Element modelling and thermal Analysis of CVD diamond window

Thermal and mechanical analysis was performed using the finite element (FE) code ANSYS 10.0. The solid element was used for the modeling of the copper support and the shell element was used for the modeling of the CVD diamond window.

Since the CVD diamond window acts as thermal filter FE modelling (FEM) was used to determine the temperature distribution on the cooper block and on the brazing transition. The temperature of the copper block is maintained at 30°C due to the water cooling loop. A thermal load of 160 W was applied on the centre of the square surface (6 mm x 3.3 mm) of the diamond foil with a thickness of 200 µm. Figure 4 depicts the results of this simulation. The temperature distribution on the foil reaches a maximum of 333°C in the centre of the diamond window and decreases rapidly to 230°C on the transition to the cooper block. The average temperature level achieved is still acceptable for the transition to copper because it is well below the temperature range for brazing.

Fig. 4: Temperature distribution in °C resulting from a heat load of 160 W applied in the centre of the window (surface of 6 x 3.3 mm, thickness of 200 µm).

Maximum stress of 377 N/mm² due to the thermal load of 160 W is reached in the centre of the diamond window (fig. 5). Additional stress is induced by vacuum operation (pressure difference to the atmosphere of 1 bar) and by brazing. This results in a total maximum of 578 N/mm² in the centre of the window (fig.6). This number is still acceptable with respect to maximum tensile stress of diamond of 1’200 N/mm².
Fig. 5: Von Mises stress due to a heat flow of 160 W

Fig. 6: Von Mises stress due to 160 W, 1 bar pressure and brazing stress
A maximum stress of 577 N/mm² due to heat load, pressure difference of 1 bar and brazing, results in a deformation of 60 µm in the centre of the diamond window (fig. 7 and fig. 8).

Fig. 7: Deformation of the Diamond foil [mm] along horizontal axis.

Fig. 8: Deformation of the Diamond foil [mm] along vertical axis.
4. Verification of safety function of CVD diamond window against inrush of air

The CVD diamond window is also used as a vacuum safety element in case of an inrush of air coming from the experimental side of the beam line. Finite element modelling of the inrush of air case shows that a window shall withstand it. To experimentally verify this behaviour against an inrush of air we apply an over pressure of 3 bars on the beamline side of the window. No permanent deformation or leak is detected. Additional orifices of 40 mm-diameter which act as beam aperture along the beamline will further damp pressure waves due to an inrush of air. As demonstrated we consider the CVD diamond window as safe as long as the resulting pressure peak is maintained below 3 bars.

5. Measurement of surface roughness of CVD diamond window

After brazing we measure the surface roughness of the CVD diamond foil of thickness of 100 µm to control the finish of the surface and to detect any alterations due to the brazing process. For this purpose we use a Zygo Newview 5010 white light interference microscope with a resolution below 0.1 nm. A three dimensional plot of the surface roughness with a peak to valley (pv) value of 16 nm and with a root to mean square deviation (rms) value of 2.4 nm is represented in figure 9. This result is compared with a similar measurement of a high quality Beryllium window (PF60) of thickness 75 µm: peak to valley value of 7 µm and 630 nm rms. In comparison to Be the CVD diamond window shows a much better surface smoothness and much less variation in surface thickness.

![Figure 9: roughness measurement on the CVD diamond window after brazing: pv 16 nm / rms 2.4 nm](image-url)
6. Influence of CVD diamond window on coherence of synchrotron radiation

Coherence properties of x-ray beams (and of any phase-shifting devices along the beamline) may be characterized by using a shearing interferometer technique and by observing a magnified Moiré-type fringe pattern on a (large pixel-sized) standard x-ray detector [3]. The arrangement of this transmission-type interferometer consists of a beam splitting phase grating and of a slightly tilted analyzer grating which provides the spatial resolution required on a standard x-ray detector. Parts of x-ray beams of the same source interfere in the detector plane and show a “magnified” (Moiré-effect) fringe pattern caused by a small or “shear” tilt angle (on the order of 0.5°) between the gratings. From the observed fringe pattern the resulting and normalized intensity distribution $|\gamma|$ is calculated. The experiment has been performed at the European Synchrotron Radiation Facility in Grenoble, France (ESFR), on the ID 19 beamline at 17.5 keV of x-rays. The fringe pattern denotes coherence of the x-ray beam (plus any phase-shifting elements along the beamline) once with a CVD diamond window inserted (normalized intensity distribution $|\gamma_1|$ in fig. 10) and once without (normalized intensity distribution $|\gamma_2|$ in fig. 11). The ratio of the two intensity distributions $|\gamma_1|/|\gamma_2|$ is depicted in figure 12. The fringe pattern of the x-ray beam only affected by the CVD diamond window alone remains unaltered. To conclude it is demonstrated that the CVD diamond window doesn’t influence significantly x-ray beam coherence.

Fig. 10: Normalize intensity distribution $|\gamma_1|$ with CVD Window inserted

Fig. 11 Normalize intensity distribution $|\gamma_2|$ without CVD window
7. Conclusions

This new type of a CVD diamond window has been mechanically characterized both by modelling and by experiment and has been successfully tested to have no significant influence on x-ray beam coherence. It is now installed in the X02DA Tomcat-beamline and is planed to be used for several new beamlines at the SLS. New types of CVD diamond windows will be developed and optimised for other thermal power levels (below 100 W and above 160 W) and for different x-ray beam sizes. Based on the window design presented, an off-the-shelf diamond window is being brought to market through Diamond Materials GmbH [4].

8. Acknowledgments

The authors acknowledge the support of the technical staff of PSI. Especially we thank R. Schraner and M. Kleeb for their support to build the prototype and X. Wang for the last FEM calculations of the CVD diamond window.

9. References