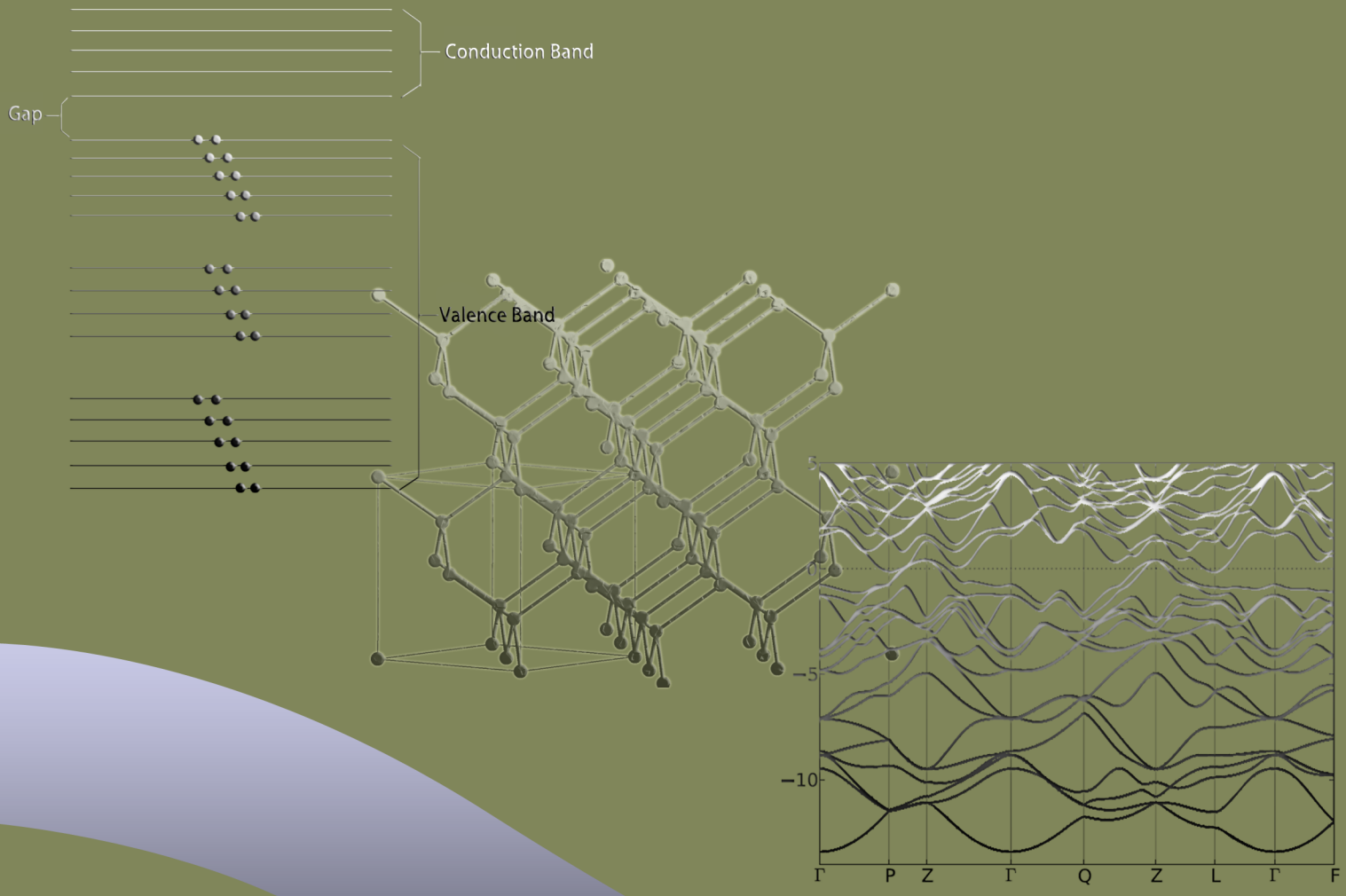
Queen Mary, University of London

School of Physics and Astronomy

XAFS实验相关问题



打孔机操作

在高压XAFS实验中，需要将样品至于两个diamond cell中间，而装载样品需要一个中间有微孔的金属片，对于金属片上的微孔有特殊要求，即微孔越均匀越好，在Diamond Light Source实验室，有专门用于打不同尺寸微孔的打孔机，更换不同的打孔指针可以实现微米量级打孔，打孔机具体的操作步骤如下：

装载好金属片后，现在金属片上打一个试探性的标记，然后将打孔指针移开，移动观察显微镜视野中的金属片，直至找到标记的中心，此时移回指针，则此时指针位于聚焦中心，打出的孔均匀性最好，之后选择金属品合适的位置进行打孔即可，具体的原理是：在不单独移动金属片位置时，打孔指针与金属片相对位置不变，因此将一开始在金属片上打的标记移动到观察显微镜视野中心时（观察显微镜的视野中心就是打孔的聚焦中心），打孔指针也跟着一起移动到了打孔的聚焦中心，之后移回打孔指针，便可以单独移动金属片至合适位置实现最佳效果的打孔。

Absorption spectrum spike caused by diamond cell

在高压XAFS实验中，需要使用diamond cell产生高压，而diamond cell晶体的某些晶面会对某些能量的入射X-ray产生衍射（Bragg Law），此时如果使用投射模式探测出射X-ray信号，由于某些特定能量值的X-ray被diamond cell衍射，因此探测到的出射信号中，这些具有特定能量值的X-ray对应的出射信号会非常弱，对应到吸收谱中，这些特定的能量值点会出现吸收的极大，而这些“吸收”显然不是真实的吸收，这些“吸收极大”被称为spike。为尽量避免spike的出现，有不同的方法，我们采用的方法是通过荧光模式（fluorescence mode）采集吸收信号，在探测荧光信号时，可以选择一个很窄的能量范围探测，则在这一能量范围内，X-ray被diamond cell衍射的几率会大大降低，因此在某种程度上可以避免spike的出现，同时，将入射X-ray一方的diamond anvil cell做成如图1. 所示的形状，这样可以在入射X-ray未到达样品前尽量减弱diamond anvil cell对X-ray的反射： 另外一种方法是通过特定的方法

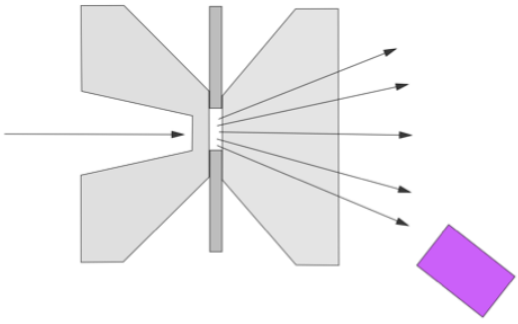


Figure 1. The diamond anvil cell used for fluorescence mode measurement to avoid diamond reflection.

使出射的X-ray 能量发散，这样即使某一个能量值对应的X-ray 被diamond cell 衍射，其余部分的X-ray 依然可以被探测到，这样也可以在某种程度上避免spike 的出现，还有一种比

较典型的方法也是目前采用比较广泛的方法，就是使用多晶——polycrystalline （或纳米晶——nano-crystalline）diamond anvil cell，这样可以大大降低diamond anvil cell对单一波长的X-ray 的反射以尽量避免吸收谱中Spike的出现。

The determination of high pressure using Ruby

When doing high pressure experiment, diamond anvil cell is always the choice to introduce experimentally needed high pressure. However, it is a problem how we can determine the high pressure of our sample within the diamond anvil cell, and this is where Ruby comes to help. The PL wavelength of Rugy is strongly correlated with pressure, thereby if we put Ruby together with our sample in the diamond anvil cell, we can then measure PL from Ruby to indirectly determine the high pressure introduced by diamond anvil cell. Of course, the exact numerical relationship between PL wavelength and pressure for Ruby should be calibrated before the experiment with our sample.

Harmonics in Fluorescence Mode Windowing

For XAS measurement, if the sample is suitable for fluorescence mode measurement (either dilute or condensed thin), then each time before scanning across the energy range (the measurement scanning), we need to set a energy window to tell the detector where our fluorescence signal is. If not, the detector (or the data collection program) will never know what real value of our fluorescence intensity is, which will make the obtained XAS spectra meaningless. What is energy window? It is just like monochromatic facility which filter what we need from what we don't. Basically, it employs the diffraction of X-ray to separate between different energy values. However, for any diffraction, we could have many ordered diffraction maximum besides the 1st order maximum. And also the first order maximum for one specific energy value could also be the 2nd or even 3rd order maximum of other specific energy values. And this is called harmonics problem. To solve this problem, Diamond Light Source employs harmonics mirror to reduce the intensity of harmonics.

Length scale of XAS local probe

The local length scale that can be detected by XAS techinique is 5∼10 Å, as is discussed in Fundamentals of XAS (Chapter 3, P_{16}).

About X-ray brilliance

For synchrotron light source, an important measure of X-ray quality when comparing between different X-ray sources is called *brilliance*. The formula is given as:

$$brilliance = \frac{photons}{second \cdot mrad^2 \cdot mm^2 \cdot 0.1\%BW} \tag{0-1}$$

The explanation:

1. '$0.1\%BW$' – Only the photons falling within 0.1% bandwidth (BW) of the central wavelength (or frequency) are taken into consideration for the measurement.
2. 'mm^2' – The cross-sectional area of the beam.
3. '$mrad^2$' – The angular divergence of the photons.
4. '$second$' – Number of photons produced per second.

This discussion is from Synchrotron Light Source Wikipage.