Written in April 2010

Innovative Instrumentation for Synchrotron Science: XMaS leads the way

The EPSRC-funded synchrotron beamline at ESRF, Grenoble, which is operated through the Universities of Liverpool and Warwick, was named **XMaS** because of the **X**-ray **Ma**gnetic **S**cattering studies planned as its stock-in-trade when it opened to researchers in 1997. In fact, although such studies have dominated the research programme of the Collaborating Research Group (CRG) of UK users, the beamline has a broader portfolio of research embracing soft matter, surface electrochemistry and even medical physics. All CRGs at ESRF exist on the bending-magnet, radiation sources rather than the brighter insertion device lines operated by the ESRF itself. If CRG lines are to compete with ESRF's own they must compensate for this disadvantage by exploiting 'niche markets' or developing instrumentation that gives them an edge, rather than merely providing 'overspill' capacity. XMaS has done this successfully and this article focuses on instrumental developments showing how following blue skies research objectives can lead to commercial instruments with wide suitability. All the devices described below are now available commercially through licence agreements made by **XMaS** through its parent universities. The EPSRC has published an article on these licence agreements in its newsletter Connect, edition 77 in page 5. (http://www.epsrc.ac.uk/pubs/mags/connect/Pages/default.aspx)

The X Y Z motorised sample mount

The amazing speed with which synchrotron data can be accumulated is of little value if the experiment has to be frequently interrupted in order for manual adjustments to be made to the sample or the sample holder. For example many materials are most interesting at low temperatures and therefore they have to be mounted in cryostats and cooled down and of course they move when the cold finger on which they are mounted is cooled down. Lining-up is not just a matter of making the beam hit the sample at the correct angle! Crystals are rarely of uniform perfection: they contain some regions from which the Bragg peaks will be much sharper than others. Aligning these so called 'sweet-spots' with the centre of rotation of the diffractometer is labour and time intensive if manual intervention is needed, as it usually is. Closing the x-ray shutter, then opening up the hutch, then making the instrumental adjustments and finally shutting up the hutch again according to the necessary safely protocols, takes time and certainly exhausts patience. Therefore the **XMaS** team developed a motorised X, Y, Z mount for the diffractometer circle which can, for example, hold a closed cycle cryostat to allow the location and alignment on sample sweet-spots and then follow them as the sample is cooled down and the sample mount cryostat contracts. Clearly the device, which is shown in **Figure 1**, must be as compact and light as possible because it has to be carried on the diffractometer circle without degrading its operation.

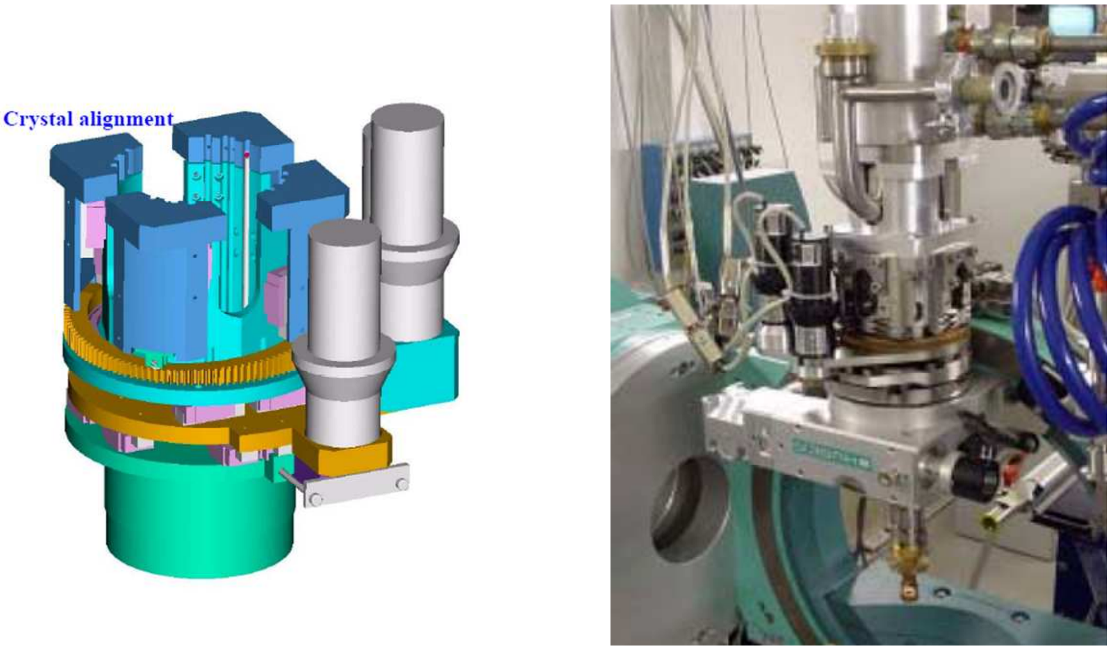

Figure 1: XYZ mount

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The XMaS device has all three linear translations guided by miniature high precision linear bearings and driven through stepper-motors with 100:1 harmonic drive gearboxes to minimise backlash. The independent X and Y movements are produced by mounting a cam onto the output shaft of the corresponding harmonic drive. The rotary motions are transformed into linear translations. The Z translation is achieved via a large toothed ring cam. The cryostat and Z stage are mounted upon cam followers, which are held in place on the cam by sixteen strong springs. Thus, as the large toothed cam turns, the Z translation is obtained. The movements have sub-micron resolution and can certainly maintain the position of the sample to better than the 30 micron "sphere of confusion" of our Huber diffractometer. Users estimate that this device can save more than half a day of frustrating manual setting up, which is very significant compared to typical beam time allocations. The device is also equally useful for positioning samples optimally at room or elevated temperatures.

The Tube Slits

Grazing incidence diffraction studies are performed at angles of less than 1° and, of course, on samples of limited size. This can lead to the incident beam "spilling over" the edge of the sample, which in turn generally increases the background scattering. Most synchrotron beam sizes are less than 1 mm × 1 mm and thus the vertical (or horizontal) extent of the beam may need to be less than 50 μm to avoid beam over-spill. As the physical size of the focal spot is largely aberration limited, the usual way of defining such a small incident beam is by placing slits very close to the sample. Also, by similarly placing slits in the exit beam very close to the sample, a well defined sample footprint is obtained with the bonus of further reductions in background scatter. Conventional motorised slit assemblies are generally bulky and, at positions close to the sample, increase the likelihood of collisions with the diffractometer, which must be avoided at all costs. The **XMaS** team has developed a novel in-vacuum slit assembly which has minimal physical dimensions close to the sample position: it is shown in **Figure 2**.

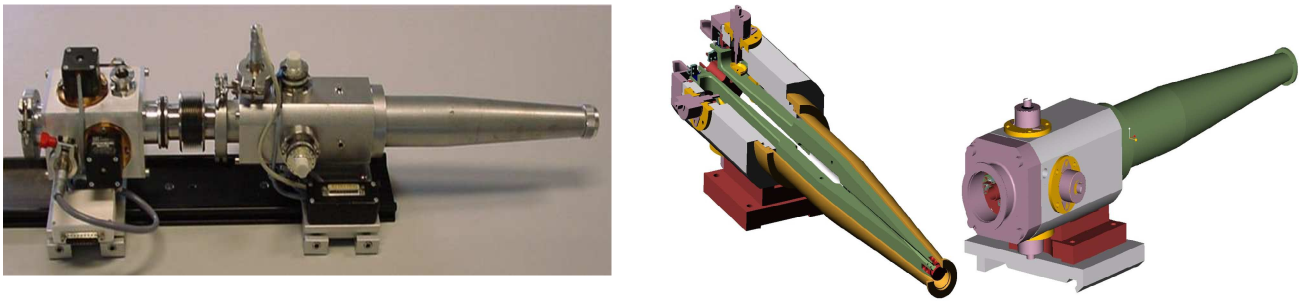

Figure 2: In vacuum tube-slits

The primary design criterion was that it should not exceed the physical dimensions of the vacuum flight tubes normally employed and for this reason a tubular design was adopted. In order to avoid 'crashes' all the actuation mechanics are remote from the sample position, with the four independent tungsten slit jaws moved by a system of levers. Actuation is achieved through use of four in-house designed miniature linear vacuum feed-throughs, which allow the motors to be mounted in air. **Figure 3** provides a striking demonstration: it shows a "single crystal" diffraction pattern obtained from a polycrystalline powder of $MnAu_2$ by using the tube slits to select a single crystallite with a 50μm x 50μm beam. A spin-off from the novel vacuum actuators is the **XMaS in-vacuum slits**, where the team have simply integrated these devices into a conventional slit screen.

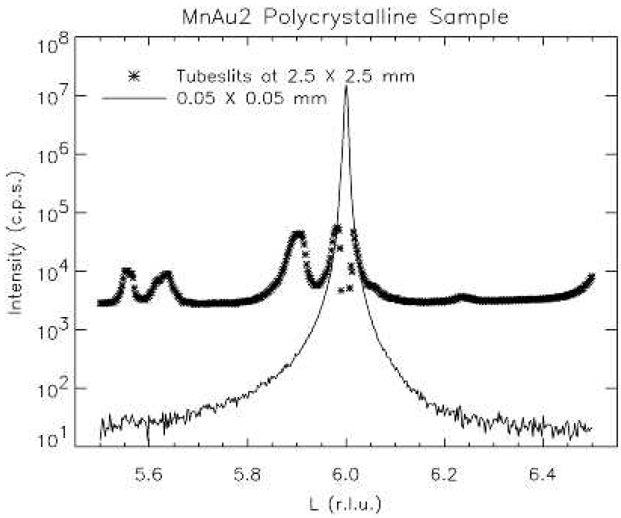

Figure 3: single crystallite with a 50µm x 50µm beam

The Polarisation analyser

Research into actinide and rare-earth magnetism, often requires the incident x-ray energy be tuned to a sample absorption edge, where the scattered magnetic intensity may be enhanced by several orders of magnitude (resonant scattering) and then polarisation analysis needs to be employed to reveal detailed information both on crystallographic magnetic structure and on the electronic states. Analysis of linear polarisation parallel and perpendicular to the diffraction plane requires the use of an analyser crystal chosen so that it has a Bragg angle close to 45° , i.e. 90º scattering angle, for the resonant energy of the experiment. With actinide compounds, which are one preoccupation of **XMaS**, it is necessary to minimize, or preferably remove, the air-paths encountered by the incident and diffracted x-rays, as the M edges lie between 3.5keV and 5keV and air absorption is significant, current exploitation of even lower energies makes vacuum operation an absolute necessity. The **XMaS** in-vacuum polarisation analyser, shown in **Figure 4** is miniaturised as far as possible in order to minimise the weight 'hung' off the detector arm. It has three rotation axes, the first is the analyser crystal θ axis; the second axis allows rotation of the diffracting plane of the analyser crystal about the beam. The third axis allows the detector to track the diffracted beam. Thus the changes in polarisation that occur in "magnetic diffraction" can be analysed.

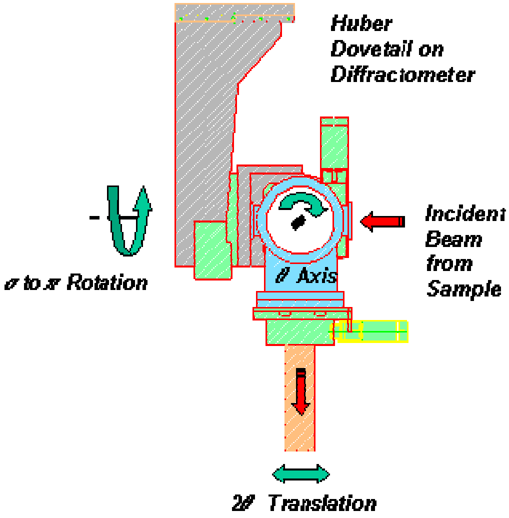

Figure 4: Polarisation Analyser

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1 Tesla and 4 Tesla Magnets

The combination of lower temperatures and higher magnetic fields opens up an ever wider range of investigations. Very high magnetic fields (above 10 Tesla, say) can be achieved with large, cryomagnets but their bulk inevitably severely restricts the geometries that can be used in diffraction studies. Other beamlines have these, so the brief for **XMaS** was to obtain increasingly large magnetic field environments that were compatible with the three dimensional functionality of the diffractometer. Fields up to 1.5 Tesla were achieved several years ago with a water-cooled iron-pole electromagnet that sits within the diffractometer's χ circle and has a gap big enough to accommodate a cryostat tail, see **Figure 5**, but that is about the limit for an electromagnet.

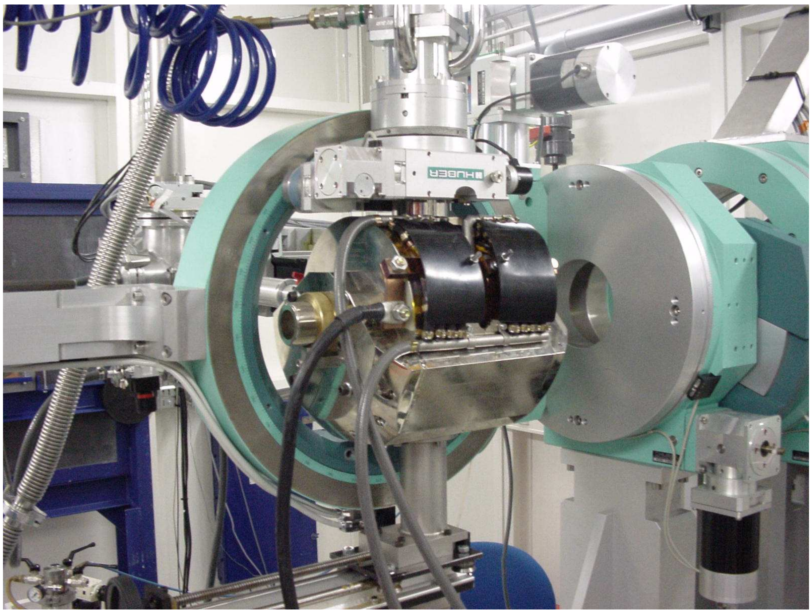

Figure 5: Electromagnet

In order to increase the field further a superconducting magnet is necessary and this was designed in collaboration with American Magnetics Inc. (AMI). It comprises a superconducting split coil wound from twisted multi-filamentary niobium-titanium embedded in a copper matrix on a former constructed of titanium to ensure rigidity (the force between the two coils is high). The cryogen-free magnet has a warm bore to accommodate environmental stages at all temperatures. Most importantly the magnet provides up to 180° radial access to the central field which is 4 Tesla, with a homogeneity of ± 1% in a 1 cm diameter spherical volume. The **XMaS/AMI** superconducting magnet has been designed to fit within the Eulerian cradle of the diffractometer and allows versatile field orientations as can be seen in **Figure 6** which shows schematics for different geometries. The magnet can be turned along the vertical axis through 90º, facilitating application of magnetic fields both along and transverse to the incident beam direction, geometries of special importance for studies of magnetic materials. An efficient yoke configuration occupies the lower half of the vertical scattering plane, leaving the other half open for the cryostat and scattered beam as shown in Figure.

The variable temperature sample environment is not without interest: it is based on a standard two-stage displex, capable of reaching 10K but with a third stage, developed by the cryogenics group at the ILL, next door, which makes it capable of operating down to an unprecedented 1.7K. **XMaS** was the first beamline to use the prototype. It can even be turned upside down without a degradation of the base temperature. At **XMaS** we have experimented with the replacement of ^{4}He with ^{3}He, which lowers the base temperature to around 1K, a very real extension to the range of magnetic phenomena that can be studied and one well suited to the bending magnet location since beam heating on brighter insertion device lines can be prohibitive.

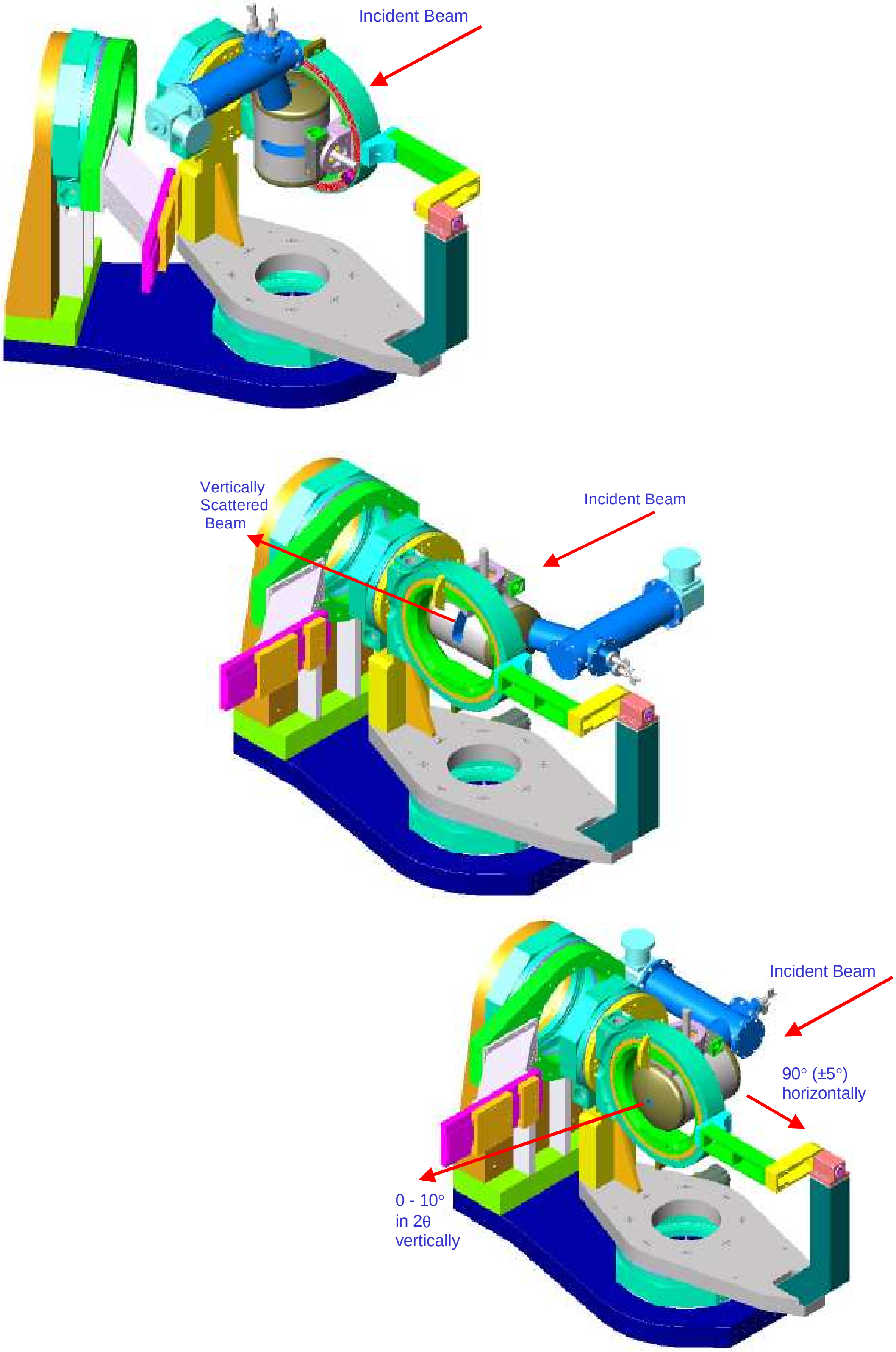

Figure 6: Geometries available with the XMaS/AMI superconducting magnet.

Novel Diffraction Oscillator

X-ray photons from a synchrotron bending magnet are linearly polarised in the horizontal plane of the electron beam orbit. However, for many magnetic investigations it is desirable to produce circular or vertical linear polarization. This can be achieved with use of a device known as a phase plate. By varying the incident angle onto this device, the polarization of the exit beam may be manipulated. Having proven the efficient operation of this device, the **XMaS** group, in collaboration with NPL, set about designing a compact oscillator to rapidly change the polarization.

This has led to a drastic reduction in system noise, through use of lock-in techniques, with a corresponding reduction in data acquisition times. The prototype design, consisting of a flexible hinge driven by four piezos, is shown in **Figure 7**. The device has now been taken to production. It is envisaged that this device will also find application as a sample oscillator or even a polarization analyzer oscillator, allowing lock-in techniques to be employed in other classes of experiment.

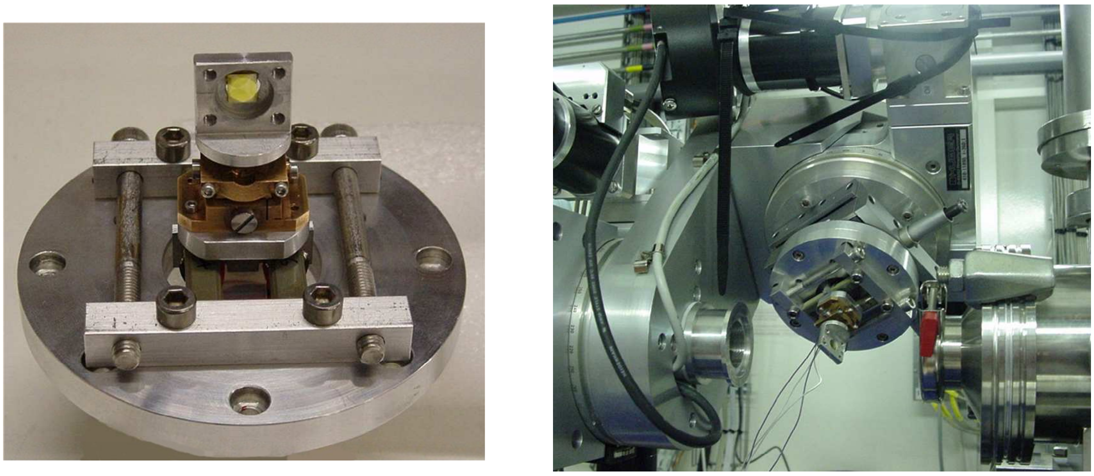

Figure 7: Compact Diffaction Oscillator

In-vacuum X-ray Attenuators

A major source of degradation in overall quality of the vacuum in the experimental hutch are the pneumatically operated beam attenuators which are prone to sticking and regularly create gas bursts when actuated. A new attenuator assembly has been built employing latching solenoids as the motive force. A typical solenoid has a stroke of only 10-15mm, not enough to pull a 15×15 mm foil in and out of an X-ray beam with a section of some 5mm^2 . This problem has been overcome by using a lever and hinge assembly to increase the overall stroke (**Figure 8**). When the foil is in the beam, the iron piston is latched within the solenoid and when the foil is out of the beam, it is latched by a spring. A strong magnet, mounted within the rotating hinge assembly, changes position when the foil is in or out of the beam, two Hall Effect switches, mounted externally of the vacuum system can detect the actual position of the foil. This simple magnetic system avoids any vacuum feed-through and is high vacuum compatible.

Overall the XMaS team has been responsible for eight instrumental developments that are now manufactured commercially under licence. We are grateful to the active collaboration of Huber GmbH, AMI and the Universities of Liverpool and Warwick in the commercialisation of these devices.

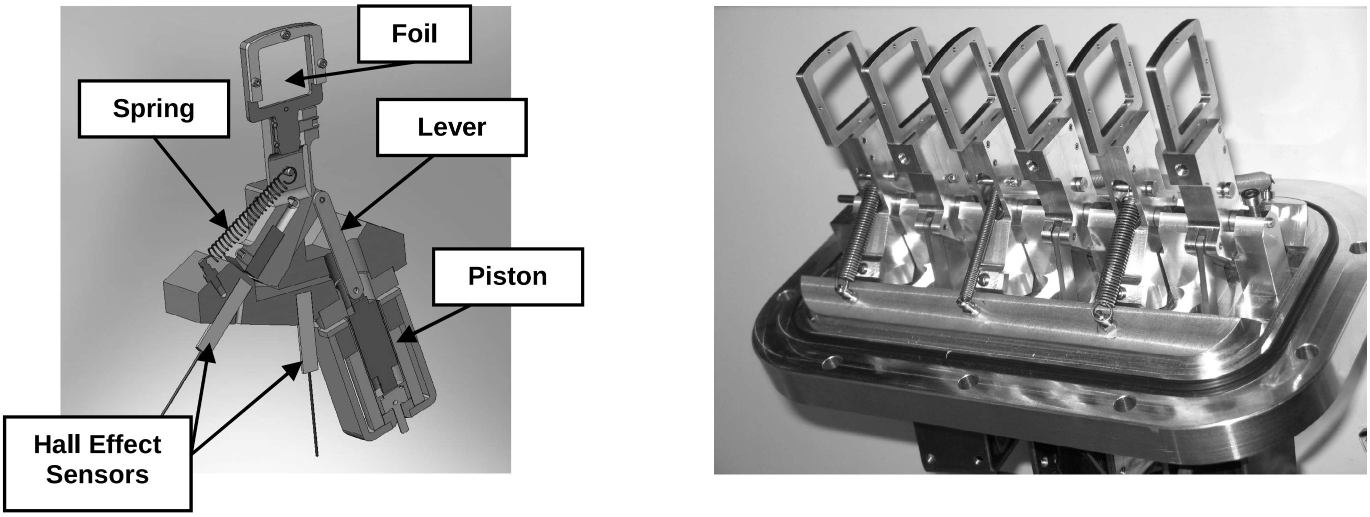

Figure 8: Attenuators