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Recent Advances in Soft X-ray Scattering Instrumentation at the National Synchrotron Light Source

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ABSTRACT:
For the study of condensed matter systems x-ray scattering experiments are often the best choice as they have several desirable features including complete conservation of momentum in the incident and detected particles, well characterized initial and final electronic states, and insensitivity of photon transport to external electric and magnetic fields (as compared to photoelectrons for example). To extend these techniques to the soft x-ray region ($\hbar\nu < 1$keV) the lack of suitable detectors, and the difficulties associated with performing scattering experiments in vacuum must be overcome. In this paper we provide details of our instrumental development program, and show some representative examples of experiments we have performed to date.

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INTRODUCTION:

As part of our program in soft x-ray scattering, we have recently made the first studies of resonant magnetic scattering at the Iron $L_{2,3}$ absorption edges [1,2]. This was accomplished by measuring the specular reflectivity of p-polarized light from single crystal Fe films, and Fe/Gd multilayers subjected to an external magnetic field. Large changes were observed near the absorption edges in reflectivity, and at Bragg reflections.

Figure 1 shows the specular reflectivity and asymmetry ratio obtained from a 35Å Fe(110) single crystal film in the presence of an external magnetic field (400 Gauss) utilizing a scattering angle (2 Θ) of 35 degrees. I^+ and I^- are the two reflectivity curves measured for opposite field directions. R, the asymmetry ratio, is defined as the difference between the two curves divided by the sum. Large changes in the reflectivity near the Fe $L_{2,3}$ absorption edges were observed upon reversal of the direction of the magnetic field. The results agree well with an atomic-like model calculation based on the resonant magnetic scattering process. This technique is related to the Faraday effect and magnetic circular dichroism through Kramers-Kronig relations. Measurements of this type provide important information on the spin polarized local density of states in the conduction band.

The data in figure 2 are specular reflectivity measurements from a (32.5Å Fe/32.5Å $Gd)_{22}$ multilayer as functions of both photon energy and wave vector transfer. For photon energies much lower than the Fe L_3 absorption edge (707 eV), the Bragg reflection corresponding to the multilayer periodicity can be clearly observed. Subsidiary maxima corresponding to the finite number of layers (22) were also observed. At 700 eV, the intensity of the Bragg reflection reaches a maximum as the real part of the scattering factor increases rapidly and the imaginary part is still small. The large changes in the real part of the scattering factor also introduce significant index of refraction corrections to the position of the Bragg reflection. Further increase of the photon energy increases the absorption dramatically while the intensity of the Bragg reflection reduces correspondingly. At 702 eV, the Bragg reflection is no longer well defined, and by 707 eV and above, only the top most layer is being sampled by the photons. This experiment clearly demonstrates how one might take advantage of the enhancement provided by the resonant scattering process to tune the penetration depth of the probe photon beam in a reflectivity measurement. Further, these examples show that it is possible to make direct measurements of optical constants in the soft x-ray range, and in particular at absorption edges, where only a few such experimental studies have previously been performed [3].

The exciting results of these demonstration experiments provide us with ample motivation for the further development of soft x-ray scattering instrumentation. To make these measurements and extend them to additional systems, however, some instrumental difficulties must be addressed. Previously, other groups [4,5] have faced these problems and developed instruments to suit their experimental requirements. For our applications the measurements must be performed in vacuum since the $L_{2,3}$ and $M_{4,5}$ edges of the 3d and 4f transition metals all lie in the 500 to 1500 eV range. In addition, while several detectors might function in this photon energy range, we are constrained by the need to scan the detector in angle and have the further requirement that the detector be able to reject the longer wavelength radiation which is often

present in beamlines utilizing grazing incidence optics in their monochromators.

Since the scattering in our experiments is typically measured far above the critical angle, the reflected intensity can be relatively weak so it is important to configure the spectrometer to maximize the sensitivity of the detector. Finally, our ultimate aim is the development of the capability to study thin film, interface, and surface scattering from samples prepared in-situ which requires a true ultra-high vacuum apparatus. The prototype experimental results described above are from samples transferred to the apparatus in air with no special attention to surface preparation, and the data were obtained at pressures of 10^{-7} Torr or higher. In this paper we show instrumentation developments under way which we feel will provide us with an instrument which will satisfy all our requirements.

APPARATUS:

In our first experiments, we attempted to utilize channel plates as our photon detector, which met our requirements for ease of mounting in a scanned 2-Θ configuration, but had some significant limitations from the standpoint of sensitivity. The crux of the problem is that the plates are far more sensitive to 10-100 eV radiation (soft contamination) than to the 700 eV radiation we need to detect. The situation is further aggravated by the experiment geometry where the resonant radiation is reflected at 4 or more times the critical angle, while the soft radiation is at or below its critical angle. For our measurements, this meant that the beamline had to be operated in a mode which reduced the soft radiation to 10^{-4} or less than that of the 700 eV photons. While we actually succeeded in this endeavor, it was not without considerable difficulty. This early experience lead us to the choice of our present detector, a low pressure parallel plate proportional avalanche counter developed by Smith et al. [6].

This detector was operated at 27 Torr of Ar 50%/Ethane 50% in a 40 mm long chamber. The detector pressure is measured with a differential capacitance manometer which provides the feedback signal for a commercial pressure controller and proportional valve. In this way the pressure is maintained to better than 0.1 Torr which stabilizes the detector gain, and provides some protection against accidental implosion of the detector window. The window for the detector is a 2.5 μm aluminized mylar film supported on a electroformed nickel mesh. The overall efficiency of the detector is about 10% with an energy resolution of 40%. The signal from the detector cathode is fed to a charge preamplifier, followed by a delay line amplifier to provide pulse shaping and further amplification. The conditioned signal is monitored with a pulse height analyzer which supplies TTL pulses for a selected region of the pulse height spectrum. In this way, we can easily reject signal from the long wavelength radiation, and watch for saturation of the detector which occurs when the input charge exceeds about 0.05 pC. Saturation effects are generally not a problem for all but the smallest 2-Θ measurements. In this case a filter [7] placed just before the sample to reduce the soft scattered radiation has been an effective means to avoid detector saturation problems.

For our experiments the angular resolution requirements are quite modest, typically .5 to 1° full width at half maximum is more than adequate. The signal levels are, however, fairly low so we have chosen our scattering geometry to take advantage of the relatively large active area of the detector. In practice this means moving the detector as far away from the sample

as possible and using the widest slits possible. This strategy takes advantage of the width of the illuminated spot on the sample which is often 5 to 10 mm in the scattering plane for our experiments. To keep the vacuum chamber dimensions to a manageable size, we confine the scanning range to 90° in 2-Θ, and make provision for several possible input ports for the incident radiation. In this way a nearly full coverage of 2-theta angles is possible, although the configuration shown in figure 3 has suited all of our experiments to date. At maximum extension, the first and second slits are 25mm and 270 mm respectively from the spectrometer axis. Both slit openings are set to 2.3 mm in figure 4 providing an angular resolution of 0.6°.

To provide the angular drive for the sample and detector we utilized a refurbished Picker two circle scattering spectrometer and developed a coaxial differentially pumped spindle to couple the motions into the vacuum chamber. The spindle is comprised of two concentric tubes which are bolted to the motions of the Picker. The outer drive (2-Θ axis) has 8 mini conflat flanges through its wall to provide the gas and electrical feedthroughs for the detector. It makes a differentially pumped seal with a 200mm (8") conflat flange bolted to the stationary vacuum chamber, and also provides the seat for the differentially pumped seal against the outside wall of the center drive (ω axis). The center spindle carries the sample on a 115mm (4.5") conflat flange which can be seen clearly in figure 4. The 57*mm* clear bore of this shaft lends itself to the mounting of more complex sample holders than the electromagnet shown in figure 4. These might include for example heating stages, cryostats, or more powerful magnets as required by a particular experiment. Overall this spectrometer has enough flexibility to accommodate a variety of sample and detector mounting arrangements.

SUMMARY:

With our present instrument, we have performed demonstration experiments utilizing resonant scattering in the soft x-ray range by which the study of buried interfaces should be possible. The combination of magnetization sensitivity, resonant enhancement, and tunability of penetration depth make this a promising technique for the study of magnetism in thin film materials. The apparatus has also clearly demonstrated the feasibility of direct measurement of optical constants in the soft x-ray range, and in particular near absorption edges where such measurements are notoriously difficult to make.

To pursue these techniques further, planned improvements in the instrumentation include upgrade of the vacuum to UHV standards by the addition of ion pumping, and the substitution of low permeability windows for the detector. Sample temperature control is also under development to allow the study of temperature dependant systems.

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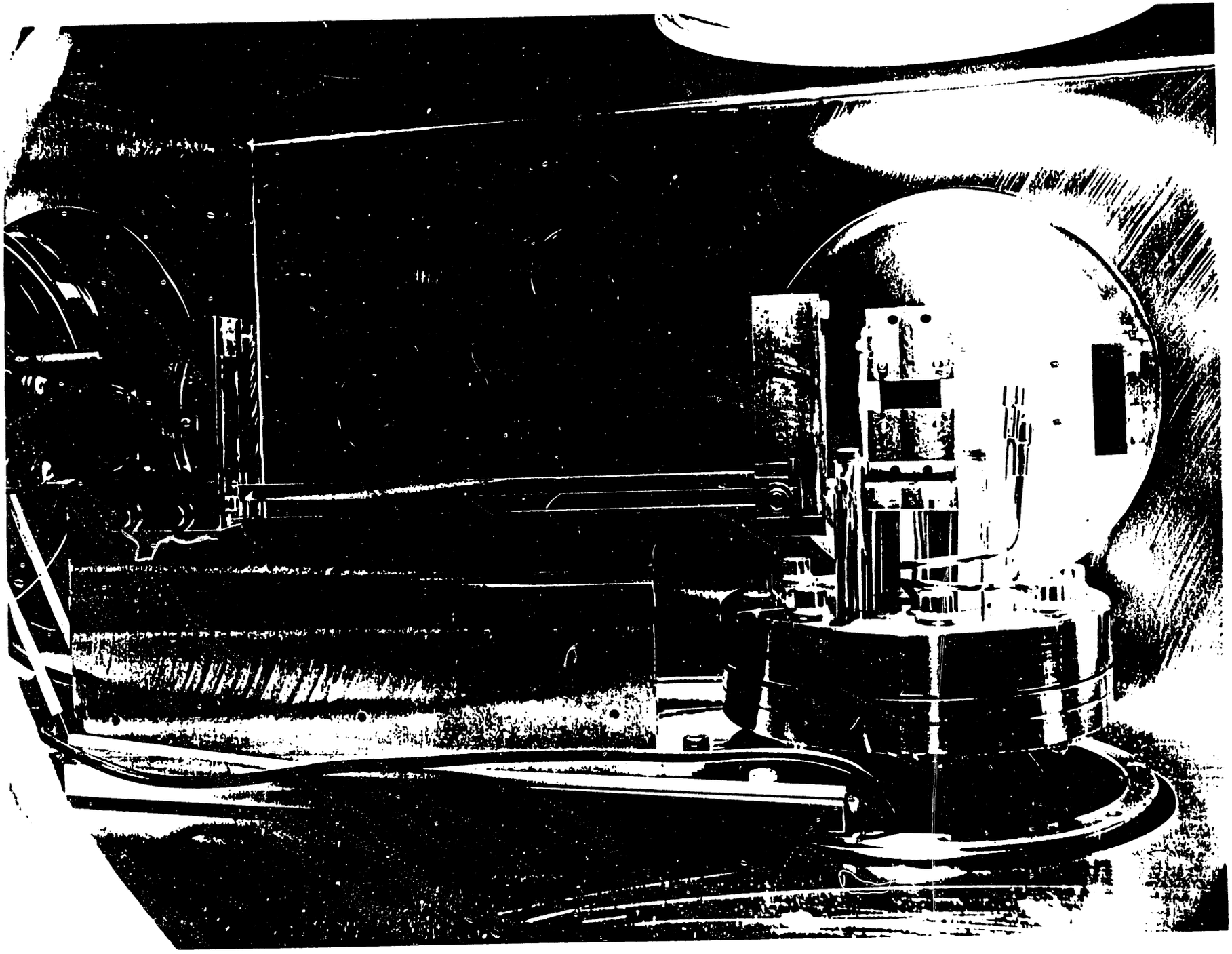
FIGURE CAPTIONS:

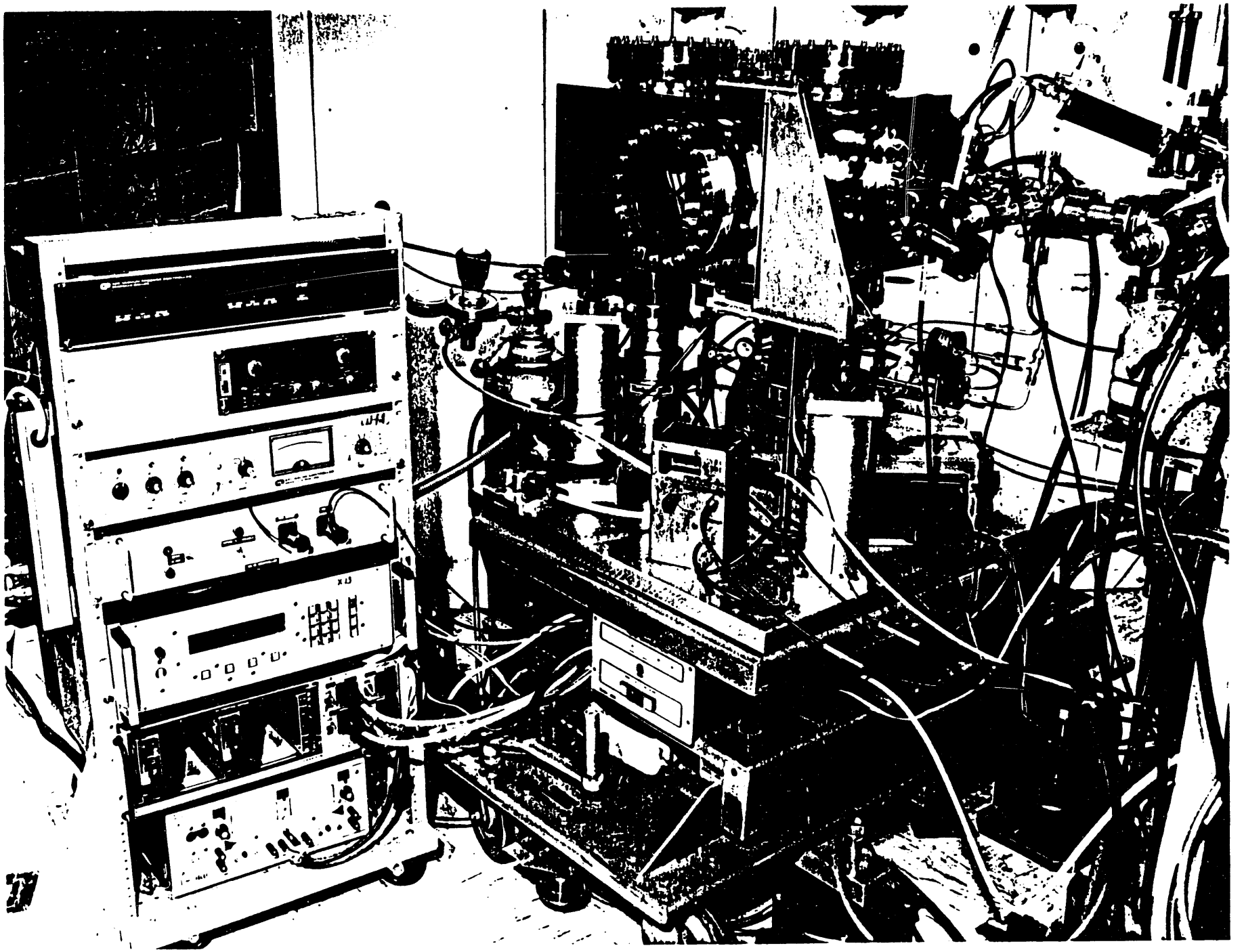
Figure 1 Specular reflectivity and asymmetry ratio measured from a 35Å Fe(110) single crystal as described in text. Note the magnitude of the effect (nearly 15% in R)!

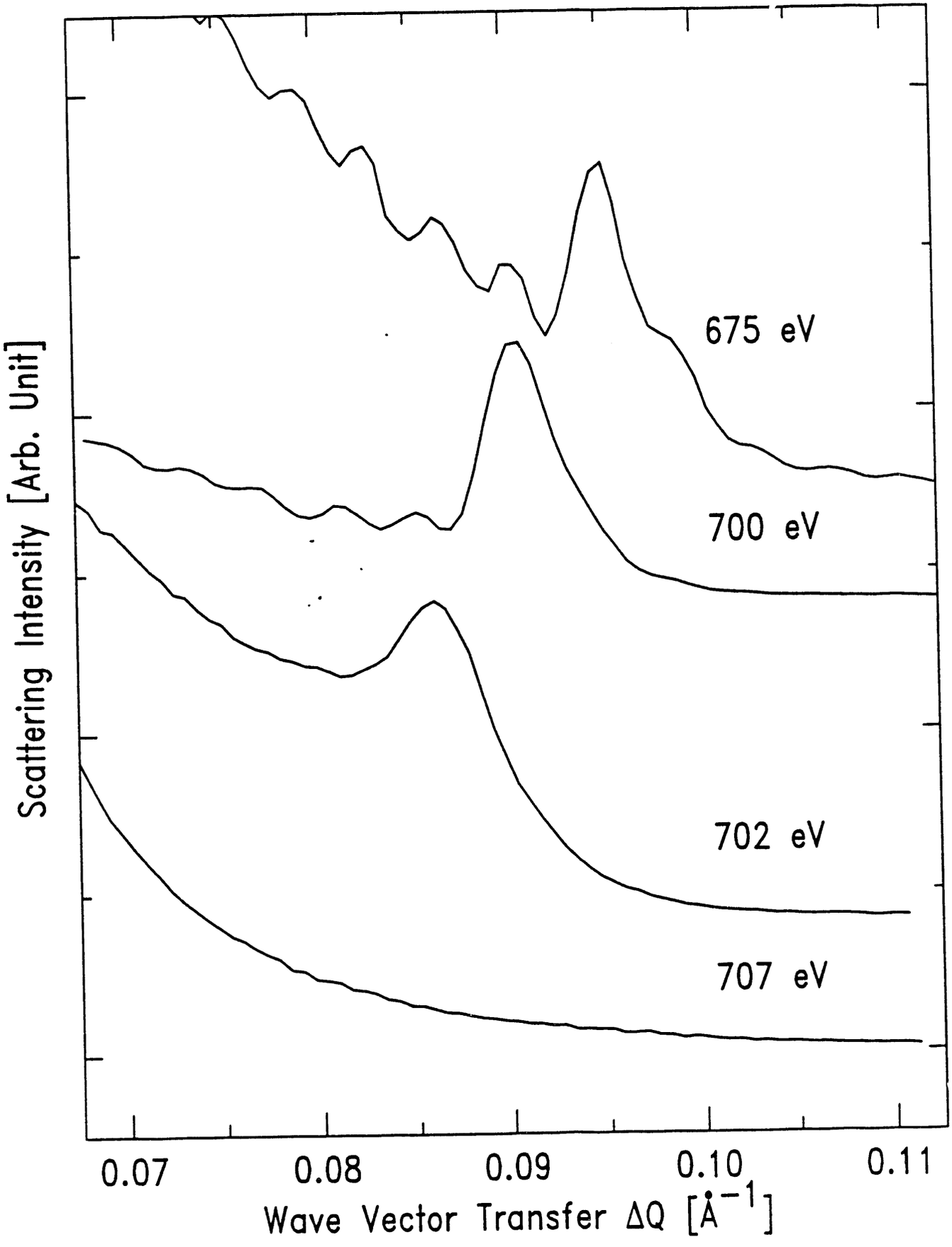
Figure 2 Specular reflectivity for first order Bragg peak from Fe/Gd multilayer as described in text. Note the index of refraction shift and extinction of the Bragg peak as the photon energy is increased to the Fe L_3 absorption edge (707 eV).

Figure 3 Photograph of the scattering spectrometer operating at NSLS beamline U13UA. Motor, detector, and vacuum interlock electronics are in the rack to the left of the spectrometer. Below the vacuum chamber can be seen the turbo molecular pump, the differentially pumped spindle (mounted atop the $\omega/2\text{-}\Theta$ drive), and the detector pressure control hardware (mounted on the spindle). The spectrometer is mounted on a lift table to facilitate its use at other beamlines.

Figure 4 View of the inside of the vacuum chamber through the viewport visible in figure 3. The detector is at the left (mounted on the slit bench) with the first slit at the other end of the bench near the sample which is mounted in the electromagnet. Just to the right of the sample is a 1500Å Al filter for stray light reduction.






675 eV
700 eV
702 eV
707 eV
Scattering Intensity [Arb. Unit]
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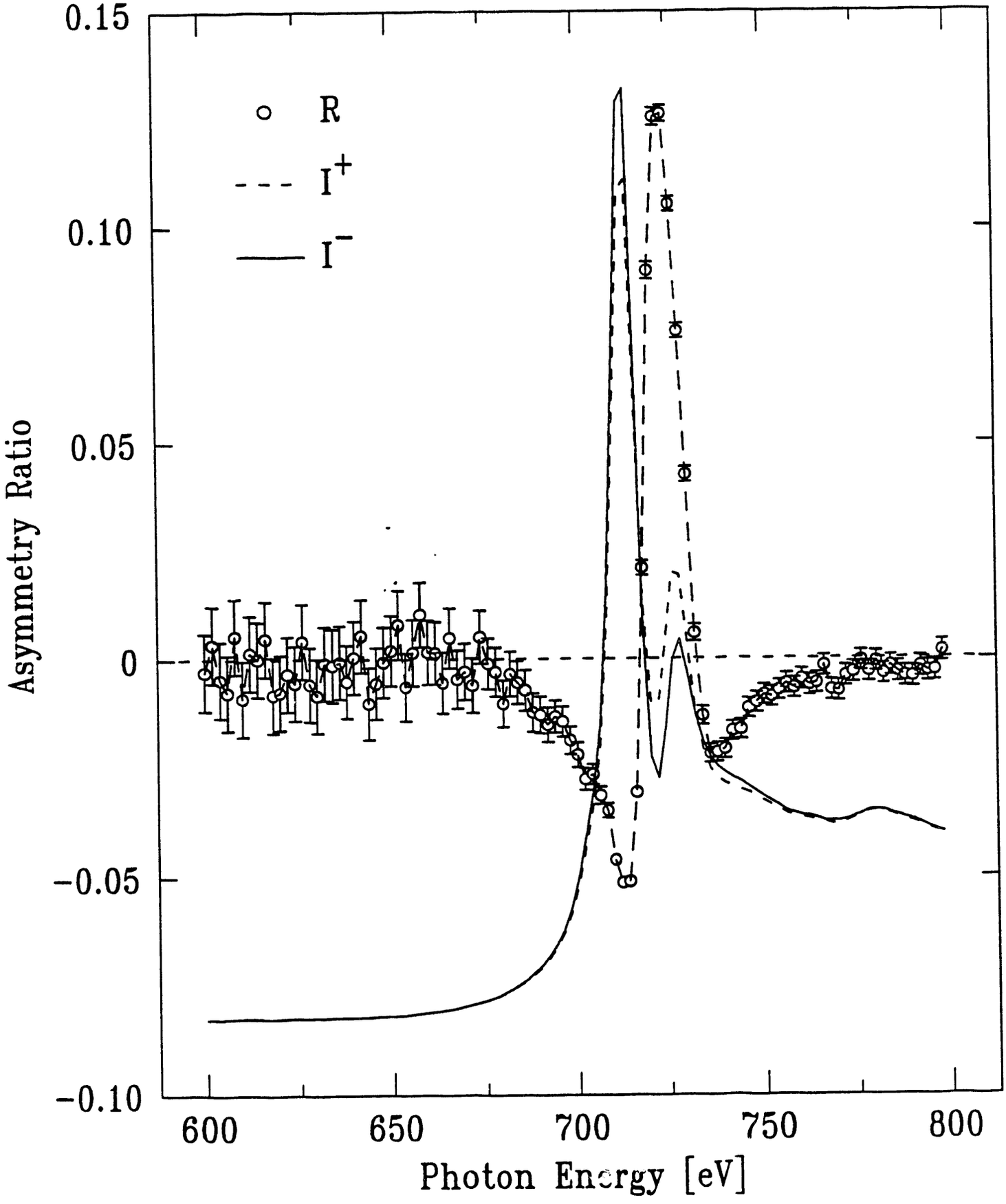
0.08
0.09
0.10
0.11
Wave Vector Transfer ΔQ [Å^{-1}]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0.10
0.05
0
−0.05
−0.10
Asymmetry Ratio
R
I+
I−
600
650
700
750
800
Photon Energy [eV]

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