Far Ultraviolet Spectroscopy of Solids in the Range 6–36 eV Using Synchrotron Radiation from an Electron Storage Ring*

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The 240 MeV electron storage ring at the University of Wisconsin Physical Science Laboratory provides an intense polarized continuum light source in the far ultraviolet. Experimental equipment is described which allows the measurement of optical reflectivity and absorption of solids in the photon energy range 6–36 eV at temperatures between 90 and 500 K with this source. A discussion of the experimental procedures and the performance of the system is presented.

INTRODUCTION

Optical absorption measurements in the far ultraviolet have been hindered by the lack of a strong continuum source for wavelengths shorter than 1000 Å. In this region, conventional gas discharge lamps provide only low intensity continua over limited spectral regions, while the stronger lines in the spectra are not sufficiently close to one another to allow high resolution data to be obtained. Polarized beams are even harder to obtain because of the low reflectance of multireflection polarizers below 1000 Å. Furthermore, no windows are available for wavelengths shorter than 1050 Å, so that the relatively high pressures of the gas sources have prevented the attainment of ultrahigh vacuum conditions ($\leq 10^{-8}$ Torr) at the sample. Because cryopumping by the sample rapidly contaminates its surface in poor vacua, only two low temperature reflection measurements have been carried out below 1050 Å, none using conventional sources.

Synchrotron radiation is free of the major limitations to far uv spectroscopy imposed by conventional light sources. It provides an intense, polarized continuum spectrum which covers the entire vacuum uv range. The properties of synchrotron radiation are well known, and have been exploited in a number of laboratories. The radiation from an accelerated, relativistic electron is emitted in a narrow cone centered about the instantaneous velocity vector of the electron. Thus a continuous beam of relativistic electrons confined to a circular orbit by a magnetic field produces a flat pancake of radiation centered in the plane of the electron orbit. The small divergence of radiation perpendicular to the orbit arises from the finite width of the emission cone of each electron and increases with longer wavelengths. In the plane of the electron orbit the radiation is plane polarized with the electric field vector parallel to the electron orbit. The polarization efficiency decreases with increasing angle from the orbital plane and with increasing wavelength.

The apparatus for measuring reflectivity and transmission of solids, described below, uses as a source of synchrotron radiation the 240 MeV electron storage ring at the University of Wisconsin Physical Science Laboratory. The properties of this synchrotron radiation source have already been discussed. An electron beam of about 10 mA circulating current is injected into the storage ring from a fixed field alternating gradient synchrotron. A 32 MHz rf cavity supplies power to the beam to replace radiative losses by the electrons. The current of electrons decays slowly (typically in 2 h to one-half its initial value) due to scattering from residual gas ions in the ring.

In order to take advantage of the special features of the synchrotron radiation source a system was designed which combines the following features: (a) high vacuum (about $10^{-9}$ Torr) in the sample chamber to allow optical measurements at low as well as high temperatures; (b) a cryostated sample stage adjustable externally which carries a number of samples to avoid frequent exposure to the atmosphere; (c) a scanning reflectometer which monitors the incident and reflected light intensity by means of a rapidly rotating light pipe; and (d) focusing optics for optimizing the light accepted by the monochromator and for compensating the astigmatism of the 0.5 m spherical grating.

The experimental system is described in Secs. I–III. Procedures for making the measurements are outlined in Sec. IV, and a discussion of the performance of the system follows in Sec. V.

I. OPTICAL SYSTEM

A schematic diagram of the optical system as seen from above is shown in Fig. 1. It is composed of four major sections: (1) the storage ring source of synchrotron radiation; (2) a glancing incidence mirror to focus the beam on the monochromator entrance slit; (3) the grating monochromator for dispersion of the light; and (4) the experimental chamber in which a focused beam is produced to measure optical properties of solids.

A bending magnet normal to the electron orbit causes the emission of synchrotron radiation by the relativistic electrons in the storage ring. In order to maximize the usable light intensity, the glancing incidence focusing mirror captures a finite angle (≈ 0.3 mrad) of the light from the ring. Because each electron emits radiation in a narrow cone tangent to its orbit, the effective source is not strictly a point but is rather a short segment of the electron orbit. The length of this segment is small compared...
Fig. 1. The optical system viewed from above. For detail of experimental chamber see Figs. 3 and 4.

to the focal length of the mirror, so that a good focus is obtained at the entrance slit of the monochromator.

The focusing mirror is an off-axis section of an ellipsoid. The angle of incidence is about 80°, so that sufficient reflectance is obtained without coating the glass surface. The imaging ratio of the ellipsoidal mirror is 3:1, so that a 1 mm diam demagnified image of the orbit segment is produced at the entrance slit and the divergence of the beam is increased to fill the ruled area of the grating. It is important to spread the photon intensity over a large area of the optical elements in order to reduce radiation damage and photodissociation of residual gas molecules, which we have already observed in the form of a brown coloration on the ellipsoidal mirror and on the surface of both gratings used, one coated with Pt, the other with Al/MgF₂.

As shown in Fig. 1, a small portion of the beam passes the focusing mirror. It is used by the University of Illinois group. Both experiments can operate simultaneously without interference.

A small quartz light pipe available at the entrance slit of the monochromator (see Fig. 1) can be moved directly in front of the entrance slit. An auxiliary, external light source can then be used for alignment purposes with visible light in zero order of the grating or for calibration of the monochromator using the atomic spectral lines of a gas discharge lamp.

The 0.5 m f/11.4 McPherson monochromator is of the Seya–Namioka design, constructed with stainless steel walls. The top of the monochromator has been modified to accept three O-ring feedthroughs and a large viewing window which is covered during measurements. A pair of baffles operated by the feedthroughs permits the beam to be masked in the vertical direction. A glass viewing screen coated on the front side with sodium salicylate can be moved into the beam behind the vertical baffles. The blue fluorescence from this sodium salicylate displays the intensity distribution of the incoming beam, which is useful for alignment of the monochromator. The vertical baffles can be used to reduce stray light and to increase polarization efficiency by limiting the vertical height of the beam.

Filters of LiF, MgF₂, fused quartz, and glass can be moved by external controls into the beam behind the monochromator exit slit in order to determine stray light and higher order contributions. It is a disadvantage of the Seya–Namioka design that the spherical grating, used off-axis, produces a large astigmatism, so that a point source at the entrance slit is imaged at the exit slit as a vertical line. Focusing in the vertical plane occurs considerably behind the exit slit. The radii of curvature of the toroidal focusing mirror in the experimental chamber were chosen to compensate for the astigmatism of the grating. This toroidal, Au coated focusing mirror deflects the light beam upward at 96° to produce a focus of about 1 mm diameter on the sample surface. As sketched in Fig. 3 another fluorescent viewing screen and a shutter can be moved by external controls into place above the toroidal mirror. Light pipes for detection, as well as other alignment features of the experimental chamber, are discussed in Sec. III.

II. ULTRAHIGH VACUUM SYSTEM

The storage ring is maintained by ion pumps at pressures in the low 10⁻⁹ Torr region. The entire experimental system for far uv spectroscopy must be compatible with the very low pressures in the ring, since in this photon energy region there is no window material available which could be used to seal off the vacuum of the ring from that of the experimental system. Furthermore, oil pumps must be avoided to prevent contamination by hydrocarbons, which would dissociate under the intense radiation, producing heavy outgassing and a carbon film.

A schematic diagram of the vacuum system is shown in Fig. 2. All pumping is by ion and sublimation pumps, after roughing with oil-less mechanical and cryosorption pumps.

Fig. 2. Ultrahigh vacuum system, showing approximate pressures maintained during measurements.
The commercial vacuum monochromator outgasses rather heavily, reaching an ultimate vacuum of only $2 \times 10^{-7}$ Torr in our system, so that a buffer section between it and the storage ring is necessary. The stainless steel separation chamber which houses the ellipsoidal focusing mirror maintains a $10^{-8}$ Torr pressure between the monochromator and storage ring with the help of a low conductance baffle in front of the entrance slit.

The filaments of the sublimation pumps must be baffled to prevent contamination of optical surfaces with titanium. Sublimation pumping was found necessary to attain the low pressures needed to start the ion pump against the heavy outgassing of the monochromator. An electrically grounded stainless steel screen above the ion pump prevents gas discharge in the ion pump from entering the monochromator at higher pressures.

A low conductance baffle just outside the exit slit reduces residual gas flow from the monochromator to the experimental chamber. As a result, the vacuum in the experimental chamber during measurement is typically $2 \times 10^{-9}$ Torr. The attainment of an ultrahigh vacuum is crucial for reflectivity measurements at high photon energies, especially for low temperature measurements, where condensation of residual gases by the sample can be a severe problem. With a vacuum of $2 \times 10^{-9}$ Torr in the experimental chamber, we have observed no contamination problems with samples held at 90 K for periods of several hours.

![Fig. 3. Experimental chamber. The reflection light pipe is shown along its rotation axis. In the vertical plane depicted, the beam emerging from the exit slit is converging due to the astigmatism of the spherical grating used at oblique incidence.](image)

**III. EXPERIMENTAL CHAMBER**

The stainless steel experimental chamber is mounted above a combination ion/sublimation pump. A schematic cross-sectional view of the chamber from the side is shown in Fig. 3. A rather large number of access ports are provided for inserting mechanical, optical, and electrical parts into the vacuum to accommodate several kinds of experiments, some being carried out now and others planned for the future.

In the vertical plane shown in Fig. 3, the light beam from the monochromator is converging to a focus a considerable distance behind the exit slit because of the astigmatism of the spherical grating as discussed in Sec. II. Thus the beam has a noticeable height at the exit slit. The height shown in Fig. 3 corresponds to that allowed by the geometry of the monochromator. In the present case the light beam has a considerably smaller height at the exit slit than shown due to the small vertical dispersion of the synchrotron source. In spite of this it was found necessary to compensate for the astigmatism with the toroidal mirror in order to obtain a small image on the sample surface and to have all light fall onto the small front surface of the rotating light pipe used for reflection measurements.

The toroidal mirror produces a focus of about 1 mm diameter on the sample surface, with the beam hitting the sample at near-normal incidence ($\approx 6^\circ$). The reflection light pipe shown in Figs. 3 and 4 is made of quartz and is bent
off axis, so that it fully captures alternately the incident \( I_0 \) and the reflected \( R I_0 \) beam as it rotates. The front surface of the light pipe is coated with sodium salicylate, so that the incident far uv light is converted into blue fluorescence, which is transmitted down the light pipe by total internal reflection to the photomultiplier placed outside the window. The reflection light pipe is rotated at about 30 Hz to provide a high precision scanning reflectometer. A transmission light pipe, also coated with sodium salicylate, can be rotated into the beam transmitted through the sample for measurements of the optical density.

Two ports are available to view the optical alignment as the orientation of sample and light pipe are adjusted under vacuum. With the grating at zeroth order, the cross section of the beam can be seen on the sodium salicylate by scattered visible light and by the blue fluorescence caused by uv photons. The quality of the focus is tested by observing the visible focus on the sample holder when it, rather than the sample, intercepts the beam. Too little light is scattered by the high quality sample surface for the light spot to be seen there. A front surface mirror behind one of the viewports permits observation of the toroidal mirror.

A detailed view of the upper portion of the experimental chamber is shown in Fig. 4. The reflection light pipe is held in a frame constructed of magnesium, and the entire assembly is dynamically balanced. The orientation of the light pipe relative to the frame is adjusted so that the frame does not interfere with the light beam during measurement of \( I_0 \) and \( R I_0 \) or with the view of the alignment from the viewports.

The back end of the reflection light pipe rotates close to the inside surface of a third viewport. The light transmitted down the light pipe passes through the viewport to a photomultiplier tube outside the vacuum which registers the time dependent photocurrent as the light pipe rotates. The light pipe is driven from its opposite end by a magnetically coupled rotary feedthrough. A stainless steel bellows section connects the feedthrough flange to the chamber, so that the position of the reflection light pipe can be adjusted under vacuum for optimum alignment.

The adjustable stainless steel cryostat shown in Fig. 4 operates in the following manner. The double wall shaft supporting the liquid nitrogen reservoir passes through an O-ring seal. The bottom plate of the cryostat is connected to the liquid nitrogen tank by a bellows. This bellows allows the bottom plate to pivot around a pointed shaft fixed to the tank. Four stainless steel adjustment wires determine the tilt of the bottom plate. Two wires are adjusted against the tension of springs attached to the two other wires.

In this configuration, liquid nitrogen drains down to the bottom plate of the cryostat, so that alignment of the sample at low temperatures is possible. Vertical height adjustment and rotation about the vertical axis are achieved at the O-ring seal. Translations in the horizontal plane are accomplished by sideways bending of the upper bellows to which the O-ring connector is attached. The other two rotations, about orthogonal horizontal axes, are obtained by the pivot and adjustment wires. Since the pivot is somewhat above the sample plane these pivoting motions cause some translation of the sample, but a good alignment is easily reached.

A stainless steel spacer is mounted between the copper sample holder and the bottom plate of the cryostat. This spacer provides a thermal resistance which lengthens the cooldown time of the sample holder to about 1 h. As a consequence, the tank cools much more rapidly than the sample holder when liquid nitrogen is introduced into the cryostat, so that most of the residual gases in the chamber condense on the tank and not on the sample surface.

Four samples are mounted 45° apart in slots on the back side of the sample holder; 3.2 mm diam holes in the holder expose part of the sample surfaces to the incident light. Thus the holder acts as a thermal shield at the desired temperature surrounding most of the sample. We have found that this radiation shielding assures that the samples reach the same temperature as the holder. All four samples are measured without breaking vacuum. A heater mounted on the back of the sample holder can raise the temperature to above 200°C to outgas the sample surfaces. Thermocouple and heater leads are routed along the liquid nitrogen tank to electrical feedthroughs in the top flange of the experimental chamber.

### IV. EXPERIMENTAL PROCEDURES

Four samples are prepared and mounted in the sample holder. At present, these may be either freshly cleaved or polished bulk samples or thin film samples evaporated on substrates. Heat lamps or a dry nitrogen gas flow are used to prevent moisture adsorption during mounting. The sample holder is attached to the cryostat, the top flange is replaced, and the chamber is pumped down, reaching \( 10^{-6} \) Torr within 12 h. Baking of the sample holder by the heater at 120–200°C improves the vacuum considerably, so that \( \approx 3 \times 10^{-9} \) Torr is reached in 1–2 days with the samples hot. This baking outgasses the sample surfaces to be measured, removing contaminants, adsorbed gas, and moisture. Measurements are carried out in the temperature range 90–500 K, and when not being measured, the samples are left at high temperature to remove any residual gas condensed on the surface during the lower temperature measurements.

It was found necessary to carry out the alignment of the sample and the light pipe using first order light in the ultraviolet rather than to rely on visual alignment
using zero order light. This is because the images of different orders are displaced if the grating is slightly rotated so that the rulings deviate from the normal to the plane of incidence. A small displacement of the images is particularly critical in our case, where it is essential for absolute reflectance measurements that the total first order beam of the incident as well as the reflected light impinge on the small front surface of the reflection light pipe. The following alignment procedure was adopted. The reflection light pipe, rotated to intercept the incident light, is adjusted to maximize $I_0$ at a wavelength setting of about $\lambda = 800 \text{ Å}$. This adjustment, being independent of the sample, needs to be checked only infrequently. The sample is adjusted to maximize the $R I_0$ signal measured by the photomultiplier while the reflection light pipe is turned toward the sample and the wavelength setting corresponds to a reflection peak in first order. This alignment procedure yields reflectivity values which are consistent and reproducible within $\pm 1\%$.

Despite the presence of the fluorescent sodium salicylate on the front surface of the reflection light pipe one is unable to see the first order light. When optimal alignment in first order is reached the zero order light spot is displaced from the center of the reflection light pipe by about 1.5 mm.

As the reflection light pipe rotates, the signal from the photomultiplier tube is processed by an FET multiplexer to derive two channels $I_0$ and $R I_0$, both corrected for dark current. A servo control of the photomultiplier high voltage maintains a constant $I_0$ signal, so that the reflectance $R$ is obtained directly and continuously as the wavelength is scanned linearly in time. The wavelength resolution of our measurements ranges from 3.5 to 8.8 Å for slitwidths from 100 to 400 $\mu_\text{m}$ respectively. Reflectance spectra are recorded in analog form on a strip chart recorder as well as in digital form on magnetic tape using a digital voltmeter. A complete reflectance spectrum between 350 and 3000 Å is measured in about 15 min, producing typically 2000 digital data values on tape which are then directly accessible to the computer for smoothing, second order correction, Kramers–Kronig analysis, and plotting. At any time the beam current in the ring can also be recorded on tape so that the $I_0$ or transmission spectrum can be normalized to the beam current.

\section{V. PERFORMANCE OF THE SYSTEM}

The source spectrum emitted from the storage ring by a 1 mA beam of 240 MeV electrons is shown in Fig. 5. The intensity of the source shown in Fig. 5 is the integrated intensity over the 30 mrad capture angle of the ellipsoidal mirror. The intensity of synchrotron radiation is proportional to the beam current, which is presently in the 10 mA range, and will be increased in the future.

Figure 5 also shows the spectrum of light at the sample for a slitwidth of 400 $\mu_\text{m}$, normalized to a 1 mA beam and

\begin{figure}
\centering
\includegraphics[width=\textwidth]{fig5.png}
\caption{Spectrum of synchrotron radiation source (emitted into the 30 mrad angle intercepted by the ellipsoidal focusing mirror) and spectrum of usable light at the sample. Both curves are normalized to a 1 mA beam of 240 MeV electrons in the storage ring. The 1200 line/mm aluminized grating has a blaze angle of 3.10° and is overcoated with MgF$_2$. The dashed curve below 300 Å is stray light resulting from small angle scattering near zero order.}
\end{figure}

1 Å bandwidth. This spectrum is measured by the photomultiplier tube from the sodium salicylate fluorescence at the reflection light pipe. To determine the absolute intensity, the photomultiplier efficiency was measured at the 4200 Å fluorescence peak of sodium salicylate by comparison with a silicon photodiode calibrated for absolute intensity.

The light intensity of this synchrotron source is much higher than that of conventional sources. Condensed spark discharges in rare gases\cite{12} give a maximum intensity at the entrance slit of about $2 \times 10^8$ photons/Å sec down to 580 Å, whereas we obtain at our sample about $1.5 \times 10^9$ photons/Å sec at 600 Å for a typical beam current of 5 mA.

A 1200 line/mm aluminized grating overcoated with MgF$_2$ is normally used in our measurements. It has a blaze angle of 5°10'. The peak intensity occurs near the blaze wavelength. The drop in intensity below 1300 Å is attributed to absorption by the MgF$_2$, but a considerable intensity is maintained down to 600 Å, due to a peak near this wavelength in the reflectance of the gold film\cite{13} covering the toroidal mirror. Another reflectance peak of gold helps to extend the useful light region down to 350 Å.

At zero wavelength (zero order of the grating) the grating acts as a nondispersive spherical mirror to reflect all wavelengths out the exit slit, producing a large intensity signal. Small angle scattering near zero order enhances the width of the zero order peak far beyond the resolution width. This stray light intensity shown by the dotted curve in Fig. 5 decreases to a fairly small value at a setting of 300 Å, where real first order light starts to appear. We have measured the approximate spectral distribution and intensity of stray light in our optical system. The total stray light contribution is well described.
by an extrapolation to larger λ settings of the zero order peak, shown as a dotted line in Fig. 5. The stray light intensity drops rapidly as λ → 200 Å, then decreases slowly to an essentially constant value above 600 Å. The distribution of wavelengths in the stray light is independent of the wavelength setting of the monochromator. The stray light is composed mainly of long wavelength components above 2000 Å. We expect to have smaller stray light contributions in the vacuum ultraviolet than do other workers using conventional sources, because we have a more favorable ratio of short to long wavelength light in the synchrotron radiation spectrum. The stray light contribution in our spectrum is estimated to be less than 2% above 500 Å and completely negligible above 1200 Å.

Higher order contributions to the spectrum must be considered because of the strong light intensity at high photon energies. We have observed second order structures in our raw data, especially in the transparent region of insulators. From these measurements empirical second order intensities were determined and the measured reflectance spectra are being corrected accordingly. Below 800 Å the correction is negligible. Up to 2000 Å the second order contribution to the spectrum is about 4%, and above 2000 Å it rises to about 15%. Corrections for higher than second order light are not necessary.

Figure 6 shows as an example the reflectance spectra of CaF₂ at 90, 300, and 430 K obtained with this system. The spectra are terminated at low energy where the crystal becomes transparent (the fundamental absorption edge). Figure 6 demonstrates the importance of measuring the reflectance at different temperatures. One observes temperature dependent shifts in energy, sharpening of the peaks, and additional structure at low temperatures.

Figure 6 shows the reflectance curves for the three temperatures offset vertically for clarity. Without this shift the curves would overlap considerably. This attests to the favorable reproducibility of our alignment as well as to the cleanliness of the surface, because conservation of oscillator strength requires that the average magnitudes across the spectrum be independent of temperature. The lack of evidence of second order structures in Fig. 6 attests to the accuracy of the second order correction. The large magnitude of the doublet near 34 eV indicates that the stray light, which always reduces measured structure, cannot be larger than the estimate given above.

The error in the absolute magnitude of our reflectance curves is estimated to be ΔR = ±3%. Structure considerably smaller than this error can of course be observed because of the much higher relative accuracy. Considerably larger discrepancies in the absolute magnitudes between the results obtained by different workers are common in the literature not only because of experimental errors but largely due to the strong dependence of reflectance magnitudes on the quality and cleanliness of sample surfaces at these high photon energies. We found that the outgassing technique and the high vacuum maintained are important in this respect.

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A Computer Controlled Interferometer System for Precision Relative Angle Measurements

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An angle measuring interferometer system has been constructed for use in a high precision crystal-diffraction $\gamma$- and $\chi$-ray spectrometer. The angle measuring device has been designed to be free of most sources of systematic error (e.g., movements of the axes of rotation, thermal effects, etc.). Electronic circuits count $\frac{1}{2}$-fringe increments which correspond to a change in angle between two rotating platforms of $\Delta \phi = 0.025^\circ$. A PDP-8 computer reads the incremental fringe count and corrects drifts in angle by applying analog signals to magnetostriuctive transducers. Using the computer, the spectrometer can be made to scan over a succession of diffraction angles, remaining locked at each position under feedback control. Preliminary tests indicate that the over-all system is capable of making consistent diffraction-angle measurements within an accuracy of $\pm 0.005^\circ$; this upper limit of stability is determined by the spectrometer resolution and the limited counting statistics obtained.

INTRODUCTION

The double flat-crystal-diffraction spectrometer, shown in Fig. 1, can be used for $\gamma$- and $\chi$-ray measurements related to the fine structure constant $\alpha$. A determination of the numerical value of $\alpha$ ($\approx 1/137$) can be obtained from high precision measurement of angular intervals between certain $\gamma$- and $\chi$-rays when diffracted to first and third order. The method is comparatively free of theoretical models (e.g., quantum electrodynamics). Details of the spectrometer and the experimental method are discussed in Ref. 1. [It is to be noted that R. D. Deslattes and W. C. Sauer (U. S. Nat. Bur. Stand.) are attempting a similar measurement of $\alpha$ via a measurement of the electron Compton wavelength $\lambda_e$. They will use an optical interferometer for measurement of absolute diffraction angles, and combined optical and $\chi$-ray interferometers for measurement of crystal lattice spacings. See Refs. 5-9.] This paper will describe the diffraction angle measuring system used in the spectrometer.

Because the range of diffraction angles in this experiment is small ($\approx \pm 2^\circ$), in order to obtain $\alpha$ to a few parts in $10^6$ one must measure the angular intervals between the required diffraction lines with a precision of a few ($\approx \pm 5$) milliarcseconds. Consequently, an interferometric angle measuring system, controlled by a PDP-8 computer, has been developed.

GEOMETRIC ARRANGEMENT

Figure 1 shows the crystal spectrometer arrangement, whereby a $\gamma$- or $\chi$-ray beam is successively diffracted through angle $\theta_B$ from two crystals $X_1$ and $X_2$. Note that the angle $\phi$ between the crystals is twice the diffraction angle ($\phi = 2\theta_B$). As in Fig. 2, $X_1$ and $X_2$ are mounted on turntables $T_1$ and $T_2$ which rotate about axes $A_1$ and $A_2$, respectively. $T_1$ is rotated about $A_1$ in angular steps of $1''$ by means of a motor driven micrometer $M_1$. $T_2$ is pulled by hanging-weight devices $H_L$ and $H_R$ and is constrained by nickel wires $W_L$, $W_R$. The far ends of the wires are attached to motor driven micrometers $M_L$ and $M_R$, which can move $T_2$ about $A_2$ in steps of $1''$. 