

## Absolute photoabsorption measurements of Mg, Al, and Si in the soft-x-ray region below the $L_{2,3}$ edges

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Absolute measurements of the photoabsorption coefficient of Mg, Al, and Si from 25 eV up to the  $L_3$  absorption edge are presented. Transmission measurements were performed on free-standing thin films using a laser-produced plasma source. The surface oxide is corrected for by taking the ratio of the absorption for different film thicknesses. The values so obtained are, in general, lower than have been reported in the literature. Structure below the edge is observed for Al and Si. Despite the fact that the absorption below the  $L_3$  edge is due to the valence or conduction electrons, the magnitude of the absorption coefficient for the solid is much higher than is predicted by a simple Drude model and is close to that expected from calculations for the free atom.

### I. INTRODUCTION

There is a continuing need for improved measurements of the optical constants of materials in the soft x-ray and ultraviolet (XUV) regions. This need is driven by the current activity in synchrotron based research, extreme ultraviolet and x-ray lithography, x-ray laser research, x-ray astronomy, and plasma diagnostics. Optical constants are essential to the design of optical elements such as mirrors, multilayers, and filters, for calculating the response of radiation detectors as well as for the interpretation of experimental measurements. In the spectral range 25–300 eV, which includes the region of interest for extreme ultraviolet lithography, the optical constants are poorly known because of the difficulties involved in their measurement. These difficulties arise because of the strong absorption of most materials in this spectral region. As a result of the high absorption thin samples are required and measurements are sensitive to any overlayers which are present, for example, due to carbon contamination or oxides.

This paper presents measurements of the absorption coefficient of freestanding films Mg, Al, and Si for photon energies below the  $L_3$  edge ( $2p_{3/2}$  ionization threshold). Due to the relatively low absorption below the  $L_3$  edge, these materials are useful as filters or as the spacer material in multilayer mirrors. There have been many studies of the absorption spectra of these materials in the past; however, most of these studies have focused on the region above the edge.

For photon energies above 50 eV the material optical constants may be determined using the tabulated atomic scattering factors<sup>1</sup> of the constituent atoms. The assumption is made that the atoms within the material interact with radiation in the same way as if they were isolated atoms. This independent atom approximation is valid for photon energies sufficiently far from absorption edges. Fine structure in the absorption coefficient near absorption edges yields information on the local environ-

ment of an atom<sup>2</sup> and it is clear that the absorption is not simply a sum of cross sections of the individual atoms. It is also clear that the independent atom approximation is not valid at sufficiently low energies where the band structure in a solid is important in determining the optical properties. One may ask how low in energy the independent atom approach may be applied to give a reasonable approximation to material optical properties.

For photon energies below the  $L_3$  edge, absorption is due to the conduction electrons in Mg and Al or the valence electrons in Si and, therefore, may not be completely atomiclike. Indeed, the measurements reported here show structure in the absorption coefficient below the  $L_3$  edges of all 3 materials and particularly for Al and Si. At the same time the overall magnitude of the absorption coefficient is close to the calculated *atomic* photoabsorption cross section.<sup>3</sup>

The measurements of the absorption coefficient were performed on an instrument based on a laser-produced plasma x-ray source. While the flux and spectral resolution are less than that available at a synchrotron, they were more than adequate for the measurements being reported here. The laser-produced plasma source also has the advantage that it is a dedicated instrument which permits the careful systematic checks (e.g., higher order or stray light) that are required for precision absolute measurements. The instrument is described in Sec. III and in detail in Ref. 4. Also, in Sec. III the sample preparation and measurements of film thickness are discussed. A noteworthy result is that while Mg films completely oxidize in air we were able to make stable Mg films by overcoating with Si and these films are very useful as filters in the 25–50 eV range.

### II. OPTICAL CONSTANTS

Ultimately one is interested not only in the absorption coefficient but in both the real and imaginary parts

of the index of refraction (or equivalently in the complex dielectric constant). Both the real (dispersive) and imaginary (absorptive) parts of the index of refraction may be obtained from the angle dependent reflectivity as measured on a smooth and clean surface. Indeed such measurements have been successfully performed on materials which do not have an oxidation problem<sup>5</sup> and on samples which were deposited in UHV.<sup>6,7</sup> Alternatively, the optical constants may be obtained using the Kramers-Kronig relations knowing only the absorption coefficient over a broad energy range. This has been done recently for all the elements  $Z = 1 - 92$  in the energy region 50 to 30 000 eV.<sup>1</sup> Since we currently do not have the capability of making reflectance measurements on UHV prepared surfaces we have instead performed transmission measurements and corrected for the oxide absorption.

The dielectric constant of a material may be related to the atomic scattering factors of the constituent atoms by

$$\epsilon = (1 - \delta - i\beta)^2 = 1 - \frac{r_0 \lambda^2}{\pi} \sum_{\alpha} N_{\alpha} (f_{1\alpha} + i f_{2\alpha}), \quad (2.1)$$

where  $r_0 = e^2/mc^2$  is the classical electron radius,  $N_{\alpha}$  is the number of atoms per unit volume of type  $\alpha$ , and  $f_{\alpha} = f_{1\alpha} + i f_{2\alpha}$  is the atomic scattering factor for atom  $\alpha$ . The linear absorption coefficient is then given by  $\mu_l = 4\pi\beta/\lambda$ , where  $\beta$  is the imaginary part of the refractive index. The results presented here are given as the mass absorption coefficient,  $\mu$  which is related to the linear absorption coefficient,  $\mu_l$  by  $\mu = \mu_l/\rho$ . The advantage of using the atomic scattering factor is that the optical constants of any material may be obtained assuming that response is atomiclike, i.e., that due to a collection of noninteracting atoms. The atomiclike assumption is clearly a poor approximation in the vicinity of absorption edges where there is structure at energies both below (excitonic) and above (extended x-ray absorption fine structure) the edge which are dependent on the chemical environment of the absorbing atom. At low energies where the absorption is due to the valence electrons the optical constants also exhibit condensed matter/chemical structure. The measurements which are reported here do exhibit nonatomiclike structure below the  $L_3$  absorption edges but agree in magnitude with the calculated atomic photoabsorption cross section.<sup>3</sup> Thus to a first approximation the atomiclike assumption appears to be reasonable for these materials and for photon energies at least down to 25 eV.

In the visible and ultraviolet regions the optical properties of a simple metal, such as Al, are reasonably well described by the Drude model for a free electron gas:

$$\epsilon = (1 - \delta)^2 - \beta^2 = 1 - \frac{\omega_p^2 \tau^2}{1 + \omega^2 \tau^2}, \quad (2.2)$$

$$\epsilon_2 = 2(1 - \delta)\beta = \frac{\omega_p^2 \tau^2}{\omega \tau (1 + \omega^2 \tau^2)}, \quad (2.3)$$

where  $\omega_p = \sqrt{4\pi N_e r_0 c^2}$  is the plasma frequency,  $N_e$  is the density of free electrons, and  $\tau$  is a relaxation time. Above the plasma frequency the Drude model predicts that the imaginary part of the dielectric constant  $\epsilon_2 \sim \omega^{-3}$  and thus the absorption coefficient  $\mu \sim \omega^{-2}$ .

Smith *et al.*<sup>8</sup> noted that the existing data for the absorption coefficient decreased more slowly with energy above the plasma frequency ( $\hbar\omega_p \simeq 15$  eV for Al) than the  $1/E^2$  dependence predicted by a Drude fit to the data below the plasma frequency. They suggested that the discrepancy might be due to surface contamination of the samples and called for improved measurements in the region between the plasma frequency and the  $L_{2,3}$  edges. Our measurements confirm that the absorption is indeed higher than predicted by the Drude model (with a fixed relaxation time) in this region and that it is close to recent atomic calculations.

### III. EXPERIMENT

#### A. The instrument

The measurements were performed with a reflectometer using the XUV radiation from a laser-produced plasma. The source/monochromator produces continuously tunable radiation over the range 25 eV–300 eV. A schematic diagram of the instrument is shown in Fig. 1 and it is described in detail in Ref. 4. The laser light is focused on a solid rod to form the plasma, a monochromator selects the desired wavelength from the broadband emission of the plasma and finally the monochromatic light interacts with the sample and is detected.

The plasma x-ray source is produced by a Nd:YAG laser (Continuum, Inc. Model YG661-10). When the 1.064  $\mu\text{m}$  output is frequency doubled the laser delivers 330 mJ/pulse of 532 nm light in 8 nsec at a maximum repetition rate of 10 Hz. The light is focused onto the target with a resulting intensity in the range of  $10^{12}$  W/cm<sup>2</sup>. The target is a cylinder, 31.75 mm in diameter and 305 mm long, which is rotated after each laser shot by a stepper motor actuated by the laser firing signal. The target rod has a useful life of about 96 000 shots which is quite long considering that measurements are typically obtained using one shot per data point. For these measurements a gold target was used since it produces a smooth continuous spectrum.

The monochromator<sup>9</sup> was designed to collect light from a large solid angle for use with sources of high divergence such as a laser produced plasma source. A three meter radius spherical grating disperses the light and focuses on a fixed exit slit. The wavelength is scanned by

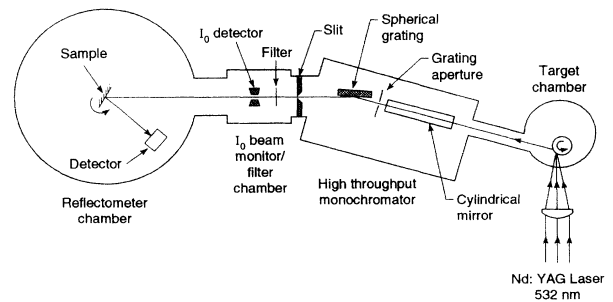


FIG. 1. Schematic plan view of the instrument showing the target chamber where the laser is focused on a rotating target rod, the monochromator, and the reflectometer chamber (not to scale).

rotating the grating about an axis parallel to the ruling using a sine bar drive. A cylindrical bent glass premirror is placed orthogonally to the grating to achieve a high collection solid angle and to protect the grating from debris from the source. With the variable radius mirror focused at the sample the vertical beam size is 0.3 mm full width at half maximum. The horizontal beam size at the sample is easily variable from a maximum of 30 mm down to less than 1 mm. With the full horizontal beam accepted the system produces  $2 \times 10^8$  photons/pulse at the output of the monochromator with a 1% bandwidth near 100 eV. The monochromator is capable of providing a spectral resolution of up to 1/500. The higher orders of the monochromator have been carefully measured using a second grating<sup>4</sup> and are effectively suppressed by using filters which are placed after the monochromator exit slit.

In order to correct for the shot-to-shot variations in the source intensity an  $I_0$  detector is used. Silicon photodiodes were used for both the  $I$  and  $I_0$  detectors. Photon "shot noise" limited measurements are obtained with these detectors down to about  $10^3$  photons per pulse where the detector noise begins to be important.<sup>4</sup> All the measurements reported here were obtained with one shot per data point.

### B. Sample preparation

The Mg and Al films were fabricated by dc magnetron sputtering in a vacuum system with 2 m torr of Ar gas and a base pressure of  $1 \times 10^{-6}$  torr. The films were deposited onto Si wafers which had been coated with photoresist to facilitate the removal of the film. The deposited films were removed by soaking in acetone. The Al films were allowed to form a natural oxide layer, which was determined to be about 40 Å on each side (see Fig. 3). However, since Mg is known to continue to oxidize in air, the Mg films were coated on both sides with a thin ( $\approx 50$  Å) layer of Si which later formed a stable layer of  $\text{SiO}_2$ . Since the deposition system had been designed for

growing multilayer films it was a simple matter to deposit a thin Si layer first and then on top of the Mg layers without breaking vacuum. The Si/Mg/Si free standing filters were found to be stable for at least a year when stored in air. The samples were freestanding with an area of 1.25  $\text{cm}^2$  and thicknesses which ranged from 0.3–2  $\mu\text{m}$ .

In order to obtain the absolute absorption coefficient an accurate measure of the film thickness is required. Two different methods were used to obtain the film thicknesses. First, the reflectance of each sample was measured prior to removing the film from its substrate. An example of a reflectance measurement is shown in Fig. 2 for an Al film. The data were fitted to the Fresnel reflectivity taking into account the reflections from each interface and using optical constants obtained from Ref. 1. The Al thickness was obtained from the fit and is mainly determined by the frequency of the Kiessig fringes which are produced by the interference of radiation reflected from the top and bottom of the film. The accuracy of the thickness determined in this way is estimated to be  $\pm 40$  Å. No attempt was made to extract optical constants from this data because of the oxide.

The second method for measuring thickness was performed on the freestanding film after removal from the substrate. The thickness was determined from the energy loss of  $\alpha$  particles passing through the films. The film was placed between an  $\text{Am}^{241}$   $\alpha$  particle source and a Si surface barrier detector in a small vacuum chamber. The energy loss of the 5.486 MeV  $\alpha$  particles was converted into thickness using the tabulated stopping powers of  $\alpha$  particles.<sup>10</sup> The  $\alpha$  particle energy loss method determines the mass per unit area as opposed to the reflectance method which provides a measure of the thickness. In principal, the two methods combined yield the density of the film. With our setup we were able to determine thicknesses to an accuracy of  $\pm 400$  Å for Mg and  $\pm 300$  Å for Al.

The film thicknesses measured by the two methods in general agreed to within the accuracy of the energy loss measurement assuming the bulk densities of 2.70  $\text{gm}/\text{cm}^3$

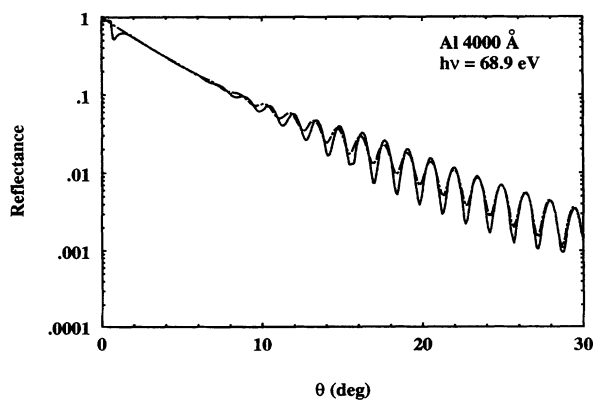


FIG. 2. The reflectance versus incident angle,  $\theta$ , measured at  $h\nu = 68.9$  eV for a 4000 Å Al film deposited onto a resist coated silicon wafer. The dashed curve is a fit to the data which was used to determine the film thickness.

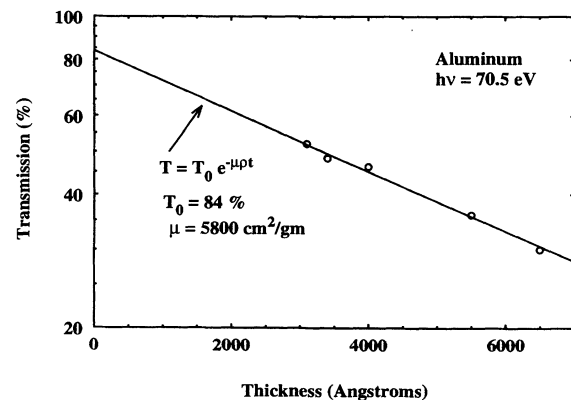


FIG. 3. Transmission versus thickness for 5 Al foil samples measured at  $h\nu = 70.5$  eV. The absorption coefficient is given by the slope and is 5800  $\text{cm}^2/\text{gm}$ . The transmission extrapolated to zero thickness corresponds to approximately 85 Å of  $\text{Al}_2\text{O}_3$ .

for Al and  $1.74 \text{ gm/cm}^3$  for Mg. In the end the thickness as determined by reflectance was used since the accuracy was approximately ten times that of the  $\alpha$  particle method.

Since the films were handled in air, and therefore oxidized, and because oxygen is very absorbing at these photon energies (about 15 times that of Al at 70 eV) it is important that the absorption be corrected for this effect. In order to correct for the oxide layers, the transmission of films with different thicknesses were measured. Assuming that the oxide layers are the same for the different samples the absorption coefficient of the unoxidized material is obtained from the change in transmission with thickness. In Fig. 3 the transmission at 70.5 eV is shown for five different Al samples. The absorption coefficient is given by the slope of the fit and the intercept corresponds to the transmission of  $85 \text{ \AA}$  of  $\text{Al}_2\text{O}_3$ . The absorption coefficients for Mg and Al were obtained in this way. The measurements for Si were obtained on a thick ( $1.95 \mu\text{m}$ ) sample so the error produced by the oxide layer is small.

## IV. RESULTS

### A. Aluminum

Aluminum is one of the most well studied elements in terms of its optical properties. The optical constants have been determined over a very broad energy range using a Kramers-Kronig analysis of reflectance and absorption data in the pioneering work of Philipp and Ehrenreich<sup>11</sup> and more recently by Shiles *et al.*<sup>12</sup> and Smith *et al.*<sup>8</sup> However, in the spectral range from above the plasma frequency (15 eV) to the  $L_3$  edge (72.7 eV) where absorption is relatively low, there have been only a few experimental measurements many of which were performed on partially oxidized samples.<sup>13-15</sup> Also many of the previous measurements in this energy range were focused on the region above the absorption edge.<sup>16</sup> The need for better measurements in this spectral region has been pointed out by Smith *et al.*<sup>8</sup> in their review of the optical constants of Al.

Our measurements of the absorption coefficient of Al are shown as a solid line in Fig. 4. The best estimate based on previously available data is represented by the compilation of Henke, Gullikson, and Davis<sup>1</sup> (dotted line) and is as much as a factor of two larger than the measurements presented here. Our results are also in very good agreement with the recent reflectance and photoelectric yield measurements of Birken *et al.*<sup>6</sup> which were performed on samples evaporated *in situ* in UHV.

In the region from 1.5 eV to the plasma frequency (15 eV) the optical constants are well described by the Drude model for free electrons. A fit of the Drude model to the low energy data is shown in Fig. 4 (using the parameters  $\hbar\omega_p = 14.8 \text{ eV}$ ,  $\hbar/\tau = 0.6 \text{ eV}$ ) and greatly underestimates the absorption coefficient above the plasma frequency. Rather, the absorption coefficient is closer to the calculated photoabsorption cross section for atomic Al.<sup>3</sup>

A minimum in the absorption coefficient is observed at

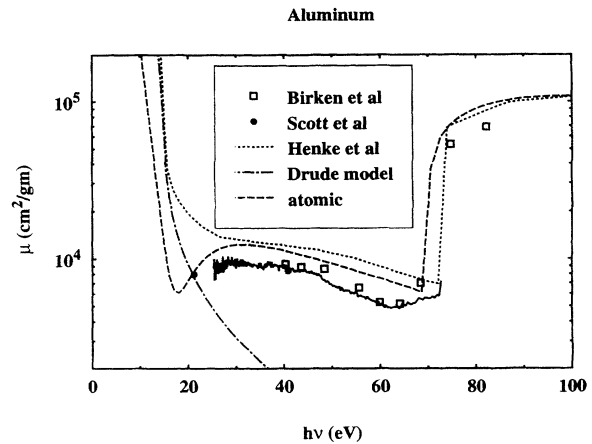


FIG. 4. The mass absorption coefficient of aluminum versus photon energy. The measurements reported here (solid line) are in good agreement with measurements on samples prepared in UHV by Scott *et al.* and by Birken *et al.* The measurements are in general agreement with the atomic calculations of Doolen and Liberman, but are much larger than an extrapolation of the low energy data using a simple Drude model.

around 62.5 eV, about 10 eV below the  $L_3$  edge. This minimum shows up dramatically as a peak in the transmittance of a thick ( $\sim 4 \mu\text{m}$ ) Al foil. (see Fig. 5) The minimum is clearly not an interference effect since it occurs at the same photon energy independent of film thickness. It does not seem likely that the minimum is related to impurities (e.g., oxide) since it is observed in many samples which include films sputter deposited in our laboratory and from Luxel Corp. (Friday Harbor, WA) and also in rolled foils from two different sources. Also, the absorption measurements are consistent with measurements on UHV prepared films.<sup>6,7</sup>

Although our measurements do not extend that low in

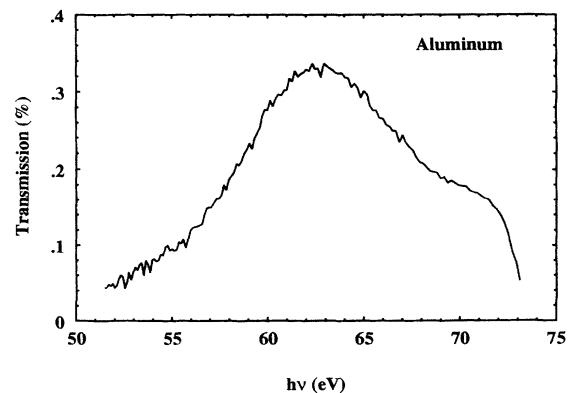


FIG. 5. The measured transmission versus photon energy for a thick ( $\sim 4 \mu\text{m}$ ) aluminum foil. The minimum in the absorption coefficient shows up dramatically as a peak at 62.5 eV.

energy there appears to be a minimum in the absorption coefficient near 20 eV (note the measurement by Scott *et al.*<sup>7</sup> at 21.2 eV). This minimum is close to the minimum in the calculated atomic cross section at 18 eV. This is apparently a Cooper minimum which evolves into the well known Cooper minimum near 50 eV for the 3*p* electrons of Ar.<sup>17</sup>

### B. Magnesium

In the case of Mg there are few measurements of the absorption coefficient below the  $L_3$  edge (49.5 eV). This situation is at least partially due to the difficulties caused by the rapid oxidation of Mg in air. The best measurements available are those of Kröger and Tombouljian,<sup>18</sup> shown in Fig. 6. It is likely that the samples used in Ref. 18 were oxidized since the films were evaporated in a relatively poor vacuum and were handled in air prior to measurement. The oxidation problem was overcome for the present work by sandwiching the Mg between Si layers. The free standing films made in this way are very stable and were also used as filters in the energy range 25–49.5 eV.

Our measurements are shown as the solid line in Fig. 6 and are in good agreement with the theoretical atomic photoabsorption cross section calculated by Doolen and Liberman.<sup>3</sup> The tabulated absorption coefficients from Henke, Gullikson, and Davis<sup>1</sup> (dotted line) reflect the best empirical estimate based on previous measurements and theoretical calculations. In contrast with aluminum, there does not appear to be a pronounced minimum in the absorption coefficient. Although not indicated in Fig. 6, the absorption coefficient increases at low energies as the plasma frequency is approached,  $\hbar\omega_p = 10.6$  eV.

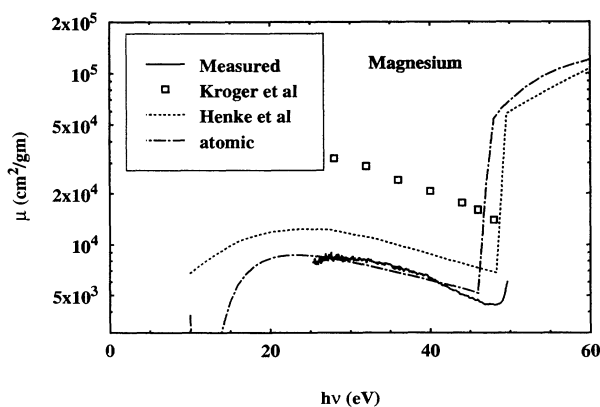


FIG. 6. The mass absorption coefficient of Mg versus photon energy. The new measurements being reported here are shown as a solid line and are much lower than previous measurements but are in good agreement with the theoretical atomic calculations of Doolen and Liberman. The compilation of Henke, Gullikson, and Davis is shown for comparison.

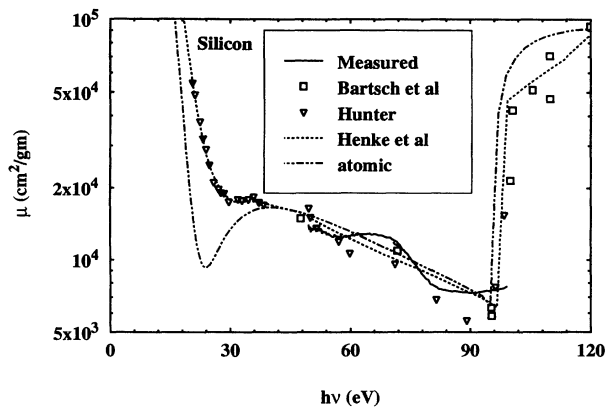


FIG. 7. The mass absorption coefficient of Si versus photon energy. Our measurements, shown as a solid line, are in good agreement with those of Bartsch *et al.*, and with the calculations of Doolen and Liberman and the compilation of Henke *et al.* However, the new measurements exhibit an anomalous energy dependence below the  $L_3$  edge.

### C. Silicon

The absorption coefficient of silicon is shown in Fig. 7. The Si  $L_3$  edge occurs at 99.8 eV (Ref. 19), and at low energies the absorption coefficient rises rapidly near the plasma frequency,  $\hbar\omega_p = 16.8$  eV.<sup>20</sup> Our measurements are shown as a solid line and are in generally good agreement with the absorption measurements of Hunter<sup>21</sup> and the reflectance measurements of Bartsch *et al.*<sup>22</sup> Our measurements were obtained using a 1.95  $\mu\text{m}$  thick single crystal obtained from Virginia Semiconductor. The thickness was determined using only the  $\alpha$  particle method. There are many other measurements particularly in the region just above the  $L$  edge [e.g., (Ref. 19)]. The best estimate of the absorption coefficient from previous data is represented by the compilation of Henke, Gullikson, and Davis<sup>1</sup> (dotted line). Also shown in Fig. 7 are the atomic calculations of Doolen and Liberman.<sup>3</sup> Below the edge the absorption coefficient is almost constant and exhibits a broad minimum at about 90 eV. This structure was apparently not observed in any of the previous measurements.

## V. CONCLUSIONS

Measurements of the photoabsorption cross sections of Mg, Al, and Si from about 25 eV to the  $L_3$  absorption edge are presented. The effect of oxide layers is canceled by taking the ratio of the absorption for different film thicknesses. The values so obtained are in general lower than have been reported in the literature. Despite the fact that the absorption below the  $L_3$  edge is due to the valence/conduction electrons, the magnitude of the absorption coefficient for the solid is close to that expected from calculations for the free atom. This result suggests that the atomic scattering factors<sup>1</sup> may provide a reasonable approximation to the optical constants of bulk

materials for photon energies down to at least 25 eV. The new measurements reported here have been used in the continued updating of the atomic scattering factor tables.<sup>23</sup>

It seems likely that the structure observed below the  $L_{2,3}$  edges in Al and Si may be due to interband absorption. However, another interesting possibility is that it is some sort of a "pre-edge" effect such as occurs for resonant Raman scattering. If the structure is a pre-edge effect then it is surprising that it would occur for Al but not for Mg. Excitonic effects are observed for silicon very close to the  $L$  edge.<sup>24</sup> However, it seems surprising that excitonic effects could extend as far as 10 eV below the edge. In closing, it is interesting that even for elements as well studied as Al and Si there are still new features to be found in the optical absorption spectra.

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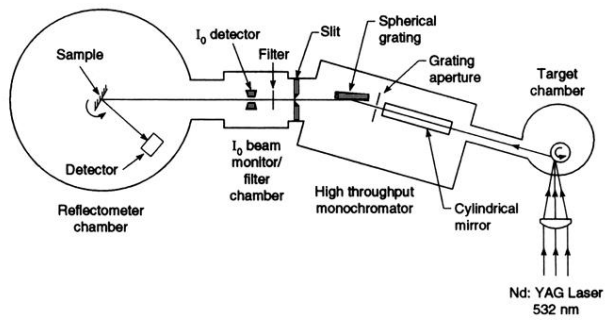


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