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OPTICAL BEHAVIOR OF FILMS FOR
RADIATION IN THE EXTREME ULTRAVIOLET

by

Thomas T. Cole, F. Oppenheimer, W. A. Rense

UNIVERSITY OF COLORADO
Space, Plasma and Radiation Laboratory
Department of Physics
Boulder, Colorado

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Contract No. AF 19(604)-5533
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GEOPHYSICS RESEARCH DIRECTORATE
AIR FORCE CAMBRIDGE RESEARCH LABORATORIES
OFFICE OF AEROSPACE RESEARCH
UNITED STATES AIR FORCE
BEDFORD, MASSACHUSETTS
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FOREWORD

The material presented in this report was written by T. Cole and is essentially that gathered by him under the supervision of F. Oppenheimer over a period of about two years.

The reflectivity and film evaporation apparatus was designed by W. A. Rense, F. Oppenheimer and Paul Johnson. It was built in the University Machine Shop. The monochromator, employing a grazing-incidence, Rowland circle type, grating mounting, was built by Ball Brothers Research Corporation.

The optical constants of films were computed from the observational data on reflectivities of films for various $\lambda$'s, thicknesses, and angles of incidence on the assumption that the generalized Fresnel's formulas hold in the spectral region of the far ultraviolet ($\lambda < 1300$ A).

The report emphasizes for far ultraviolet radiation: 1) the possibilities of developing films with high reflectivity, 2) the presence of polarization, 3) the variation of reflectivity with angle of incidence, and 4) the change in reflectivity with time after deposition of film.
ABSTRACT

The optical properties of many substances in the region of radiation wavelengths less than 2000 Å are unknown because experimental difficulties are encountered due to the opacity of most solids and gases in the extreme ultraviolet.

The theory of optical constants of absorbing substances from measurements of reflectivity is discussed. The generalized Fresnel equations were used to construct graphs of reflectivity as a function of refractive index and extinction coefficient for four angles of incidence and five values of polarization of the incident light.

The specular reflectivity of aluminum, silver, gold, magnesium fluoride, silicon monoxide, and zinc sulfide films was measured for wavelengths of 1216 Å, 1048 Å, 920 Å, 584 Å, and 304 Å using an ultraviolet monochromator and a vacuum reflectometer. Samples were vacuum evaporated onto glass and measured in situ. The transmission of an aluminum film at 584 Å and 304 Å was also measured. The optical constants of these substances were obtained.
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CHAPTER I.

INTRODUCTION

The properties of metals and dielectrics with respect to their interaction with electromagnetic radiation is of interest, not only in broadening the range of knowledge of the intrinsic properties of matter, but in the field of solid state theory. The effects of light on matter are at the foundation of many current problems concerning energy relations, atomic structure, and photon-electron interactions. From the changes in light reflected from, or originating from matter, much of the present knowledge of the nature of the universe has been obtained. Only the broad field of spectroscopy need be mentioned in this connection.

Quantities often observed in the ultraviolet, visible, and infrared portions of the electromagnetic spectrum are the frequency of emitted light; the reflectivity, or ratio of reflected light intensity to incident intensity; the intensity and directional characteristics of the transmitted light; and changes of the phase and polarization in the reflected and transmitted light. From either of the latter three groups of quantities, the optical constants of the substance may be
obtained from the equations of electromagnetic theory. The optical character of the substance is then characterized by the optical constants. They are known as the refractive index and the extinction coefficient. These "constants" are in fact functions of the frequency of the light and therefore exhibit considerable variation throughout the full range of spectral frequency. The characteristics of the light transmitted by transparent substances and the phase and polarization changes in the light reflected by opaque substances are most commonly employed for obtaining optical constants in the infrared, visible, and near ultraviolet regions. This spectral range includes radiation wavelengths of from several microns to about 2000 A. For wavelengths from 2000 A to about 100 A the most conveniently observed quantity is the reflectivity, since most substances are strong absorbers in that region. In the X-ray region with wavelengths less than a few hundred angstroms, most substances again become relatively transparent and the transmitted radiation may be used to determine optical constants, though reflectivity measurements have also been used for this purpose.

The optical constants are related to the atomic properties of the substance, and therefore to its electrical properties. Both classical and quantum mechanical dispersion theories have been developed which relate optical, electrical, and atomic properties. The relation of the dielectric constant and the electrical conductivity to the

\[1 \text{ angstrom (A)} = 10^{-8} \text{ cm.} \quad 1 \text{ micron equals } 10^{-4} \text{ cm.}\]
optical constants by means of Maxwell's equations is derived in many books on electromagnetic theory or optics. The classical theory for the optical properties of insulators and the free electron theory of metals are discussed by Seitz in The Modern Theory of Solids, as is the quantum mechanical formulation of optical properties, and appropriate experimental results are compared with theoretical predictions. The validity of all theories is restricted to certain spectral ranges, and no all-inclusive description of these properties is available at present. The classical bound electron theory of the optical properties of insulators originated by H. A. Lorentz required modification to account for absorption effects in the X-ray region, and has been superseded by the quantum formulation. The original Drude free electron theory of metals was unable to explain certain optical effects in the infrared and microwave region, known as the "anomalous skin effect." Here also quantum mechanical methods have yielded results more nearly in agreement with experiment.

Observations of the properties of transparent non-conducting substances have been made with visible light since ancient times. The formulation of the Drude free electron theory of metals in the late

---


4L. G. Schulz, Advances in Phys. 6, 102 (1957).
nineteenth century promoted a considerable amount of experimental work in the field of metal optics, most of which has been conducted in the visible and infrared portions of the spectrum. In the ultraviolet some investigations have been made, but much less has been done than in the other optical regions. This is due to certain experimental difficulties peculiar to the ultraviolet region which arise from the strong absorption of light of this frequency range by most substances, including air.

An early summary of the known properties of materials in the ultraviolet region has been given by Luckiesh. From the pioneering work of Schumann and Lyman, the short wavelength limit of transparency of common glass was known to be about 3400 Å, that of quartz 1850 Å, of fluorite about 1200 Å, and of air about 2000 Å. The reflecting power of many metals and some dielectrics had been investigated as far as 1800 Å by Hagen and Rubens, Lyman, Minor, Meier, Frehafer, Hurlburt, and others. Most of the data had been obtained by photographic methods. These methods lacked accuracy, even when used by a very experienced investigator. In addition, the nature of the surface of the sample, as obtained by polishing for example, apparently has considerable effect on the reflecting power,

---


so that much of this earlier work did not furnish consistent information. An approach from the X-ray region was adopted by Holweck, who investigated absorption and reflection of long wavelength X-rays by solids and gases. In an interesting account of his work, the absorption of a 2700 Å thick celluloid film for wavelengths from 1657 Å to 10 Å is given. The type of absorption characteristic of the region is indicated. Substances transparent to the near ultraviolet light and to moderately energetic X-rays of less than a few Angstroms in wavelength are opaque in the extreme ultraviolet region.

The strong absorption by gelatin of wavelengths less than 1850 Å necessitated the development by Schumann of special photographic plates and techniques for spectroscopy in the extreme ultraviolet. The extreme ultraviolet, sometimes called the vacuum ultraviolet, or the far ultraviolet, denotes the band of wavelengths from about 150 Å to 2000 Å. It contains the regions originally investigated by, and named for, Schumann, Lyman, and Millikan, in order of decreasing wavelength. It is bounded toward longer wavelengths by the near ultraviolet and visible spectrum, and toward shorter wavelengths by the X-ray and Gamma-ray regions.


9 Ibid., Chap. I.
In the course of his studies of the spectrum of Hydrogen in 1926, Pfund measured the average reflecting power of glass, quartz, and six metals for light of the Lyman spectrum in the range from 1216 Å to 921 Å by thermocouple and galvanometer. He found quartz to have the highest reflectance, and glass next in order in the group of substances observed. Later work by Tousey indicated an area of strong selective reflection for quartz at 1190 Å.

The optical constants of celluloid film for the 300-1000 Å region were obtained by O'Bryan in 1932 using photographic methods. This seems to be the first use in the extreme ultraviolet region of equations obtained by generalizing the Fresnel equations for reflectivity of transparent substances for polarized components of light to equations for absorbing substances. Wilsey had previously made use of them in the visible region to compare polarimetric and photometric methods of obtaining optical constants of metals. Wilsey regards them as belonging to a group of relations known generally as Ketteler's equations, though he gives no explicit reference to their source. These equations were published in Handbuch der Physik in 1928 by König.

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He attributes them to Chr. Pfeiffer, who presented them in a dissertation in 1912. Similar generalizations of Fresnel's equations have been found to apply to the reflection of X-rays incident at grazing angles.

A striking confirmation of certain aspects of the theory of metals came in 1933 with the discovery by Wood of the sudden onset of transparency of the alkali metals as the wavelength of light was reduced. Potassium was the metal most thoroughly examined. The shortest wavelength used was 1860 A. No numerical values of absorption coefficients were given, but the photographic evidence obtained made the determination of the presence of low absorption and the upper limits of transparency possible. The optical properties seemed strongly dependent on conditions of sample formation. Liquid nitrogen temperatures were required to produce films lasting only a few days in a vacuum. This behavior of the alkali metals can be explained fairly well by the classical electron theory, which predicts a critical frequency above which absorption should decrease.


No marked transmission to wavelengths as short as 1300 \( \text{A} \) was observed in ten metals investigated by Hantsler in 1934, who evaporated the metals onto celluloid. Contributions of Kronig and Zener to the theory of the optical properties of metals prompted further experimental work on the alkali metals by Wood and Lukens, who determined that the transparency of the alkali metals probably extended to at least 500 \( \text{A} \). This was implied by a lack of reflected light of this wavelength.

A comprehensive study of metallic properties in the near and extreme ultraviolet was conducted by Sabine, who evaporated films of twenty-two metals onto glass substrates and measured the reflecting power of each from the visible region to 450 \( \text{A} \) by photographic means. The films were exposed to air before being measured. As is evident from many later studies, the optical properties of metals are changed to a considerable degree by oxidation and adsorption of gases, as well as by surface contamination during handling and pumping operations. Sabine estimated his error in measurement to be as high as fifty percent of the measured values for reflecting powers less than ten percent. Since it is uncommon to find materials with a reflecting

\[ \text{19 R. de L. Kronig, Nature 132, 601 (1932); Nature 133, 211 (1934); C. Zener, Nature 132, 968 (1932); R. W. Wood and C. Lukens, Phys. Rev. 54, 332 (1938).} \]
\[ \text{20 G. B. Sabine, Phys. Rev. 55, 1064 (1939).} \]
power greater than ten percent in the extreme ultraviolet for wavelengths less than 1000 Å, the data given for that region by Sabine are generally unreliable.

Development of the photo-electric cell and the photomultiplier vacuum tube provided means of determining light intensities which were more accurate than the photographic method, and more sensitive than the thermocouple technique. Since the photomultiplier required very low and well controlled operating pressures, its extensive application for the measurement of intensities in the extreme ultraviolet region was retarded until a suitable fluorescent phosphor was found which would provide a transition in wavelength from the short-wave radiation, which will not penetrate the required tube envelope, to light of longer wavelength. Sodium salicylate was recommended by Johnson, Watanabe, and Tousey in 1951 on the basis of its relatively high and constant quantum efficiency, and it has been utilized in most recent investigations. 21 It has been shown that a zinc oxide phosphor has a greater quantum efficiency than sodium salicylate for wavelengths less than 750 Å. 22 Photocells have been developed which will


operate at rather moderate vacuum pressures, and therefore require no envelope for use in a good vacuum chamber. Reliable photomultipliers of this type are now available, but their use is not yet widespread.

The Drude theory of metals was extended by the theoretical work of Bohm and Pines, who considered collective oscillations of a gas of free electrons. A frequency of "plasma oscillations" was obtained which is identical to the critical frequency of the classical theory beyond which absorption of such a system should decrease. Confirmation of the existence of regions of transparency in the extreme ultraviolet for some metals has been obtained. Evidence that aluminum becomes transparent at wavelengths shorter than about 830 Å was given by Hass and Tousey, who observed multiple beam interference effects in the reflected light from an aluminum film at 585 Å and 735 Å. Unsupported films of aluminum, tin, indium, and bismuth were observed to be transparent to some wavelengths in the extreme ultraviolet by Walker, Rustgi, and Weissler.

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24 D. Pines, Phys. Rev. 92, 609 (1953), and preceding papers by D. Bohm and D. Pines.


The problem considered here is that of obtaining the optical constants of several common materials in the extreme ultraviolet region. Measurements of the absolute specular reflectivity have been made and optical constants have been calculated. The method of calculation used here is similar to that proposed by Tousey, and has been employed previously in the infrared region. It consists of determining the reflectivity for several angles of incidence and solving the generalized Fresnel equations with these values for the refractive index and the extinction coefficient by graphical methods. The optical properties of thin films are known to differ from those for the bulk substance. For practical applications such as film coatings, it is often the former that are desired. The properties of moderately thick films do approach those of the bulk substance.

The substances chosen for investigation have been widely used for technical purposes due to their useful optical properties and ease of vacuum evaporation. They are aluminum, silver, gold, magnesium fluoride, silicon monoxide, and zinc sulfide. The optical constants of aluminum, silver, and gold have been investigated in the near ultraviolet, visible, and infrared region, in some cases by several different observers. Although the results have not always been the same, the


general trend of values is known. The dispersion curves for magnesium fluoride and zinc sulfide in the visible and infrared are available. The properties of silicon monoxide are not well known. At the present time, there are no available values of the optical constants of most substances in the extreme ultraviolet. Those of aluminum have been estimated, some for zinc sulfide have been determined and the absorption of silicon monoxide has been studied. The general purpose of this investigation is to extend observations of the optical properties of these substances in the extreme ultraviolet region.

Optical Constants

The refractive index, \( n \), of a substance in vacuum is defined as the velocity, \( c \), of light in vacuum to the phase velocity, \( v \), of light in the substance

\[
n = \frac{c}{v}
\]

(1)

It may be shown that the refractive index of an absorbing medium is a function of the angle of incidence. An inhomogeneous wave exists in the absorbing substance for which the planes of equal phase are not, in general, coincident with the planes of equal amplitude. For light incident normally on a plane surface of an absorbing substance, no inhomogeneity exists in the wave. We will denote the refractive index of an absorbing substance for normal incidence by \( n \).

---

A monochromatic solution of Maxwell's equations for a homogeneous, isotropic, transparent medium may be expressed as a plane, time-harmonic wave in the form

$$\vec{E}(t, t) = \overrightarrow{E}_0(\vec{r}) \, e^{i \omega(t - \frac{\vec{r} \cdot \vec{u}}{c})}$$  \hspace{1cm} (4)

where $\overrightarrow{E}_0(\vec{r})$ is the reference amplitude, $\vec{r}$ is the position vector of a point in space with respect to an arbitrary origin of coordinates, and $\vec{u}$ is a unit vector in the fixed direction of propagation. Let the complex number $N$ be defined as

$$N = n - ik$$  \hspace{1cm} (5)

where $k$ is a constant. Substitution of Eqs. (1) and (5) in Eq. (4) gives

$$\vec{E}(t, t) = \overrightarrow{E}_0(\vec{r}) \, e^{-\frac{k \omega}{c} \vec{r} \cdot \vec{u}} \, e^{i \omega(t - \frac{n}{c} \vec{r} \cdot \vec{u})} \, e^{\frac{-2\pi k}{\lambda} \vec{r} \cdot \vec{u}}$$  \hspace{1cm} (6)

where

$$\overrightarrow{E}_0'(\vec{r}) = \overrightarrow{E}_0(\vec{r}) \, e^{\frac{-2\pi k}{\lambda} \vec{r} \cdot \vec{u}}$$  \hspace{1cm} (7)

and $\lambda$ is the vacuum wavelength corresponding to frequency $\omega$.

By defining a complex index of refraction, $N$, by Eq. (5) a representation is obtained which includes an exponential attenuation factor.

The intensity of an electromagnetic wave is proportional to the time average of the square of the electric vector. Therefore, the intensity variation is given by

$$I = I_0 e^{-\frac{4\pi k}{\lambda} \vec{r} \cdot \vec{u}} = I_0 e^{-\alpha(\vec{r} \cdot \vec{u})}$$  \hspace{1cm} (8)
where

\[ \alpha = \frac{4\pi k}{\lambda} \]  

and \( \alpha \) is known as the absorption coefficient. This expression relates the absorption coefficient, which may be determined by transmission measurements, to the extinction coefficient. If the light is not incident normally at a plane boundary, the extinction coefficient becomes a function of the angle of incidence due to the inhomogeneous character of the wave in an absorbing medium. \(^{31}\) We will denote the extinction coefficient for normal incidence by \( k \).

When light is incident normally at the boundary between two different media, there will be an additional loss of transmitted intensity at the boundary. The transmission, \( T \), of a uniform lamina of thickness \( d \), which is characterized by an absorption coefficient \( \alpha \), may be defined by

\[ T = (1-Q)^2 \ e^{-\alpha d} \]  

where \( Q \) is the fraction of intensity lost at the bounding surfaces by any means, such as reflection or absorption in surface layers. The squared quantity arises from the presence of two boundaries.

The Generalized Fresnel Equations

The Fresnel equations for components of the incident and reflected electric vectors as obtained from boundary conditions for

\(^{31}\)Schuster and Nicholson, op. cit., p. 255.
homogeneous, isotropic, semi-infinite media are

\[
\frac{E''_s}{E_s} = \frac{n \cos \Theta - \cos \phi}{n \cos \Theta + \cos \phi} = -\frac{\sin (\phi - \Theta)}{\sin (\phi + \Theta)} \quad (11)
\]

\[
\frac{E''_p}{E_p} = \frac{n \cos \phi - \cos \Theta}{n \cos \Theta + \cos \phi} = \frac{\tan (\phi - \Theta)}{\tan (\phi + \Theta)} \quad (12)
\]

where \( s \) denotes the component perpendicular to the plane of incidence, \( p \) denotes the component parallel to the plane of incidence, \( \phi \) is the angle of incidence, and \( \Theta \) the angle of refraction, both angles measured with respect to the normal to the surface. The double primed quantities indicate the reflected electric vector components. For such a medium in vacuum, Snell's law is

\[
n \sin \Theta = \sin \phi \quad (13)
\]

It can be shown that these equations are valid for conductors as well as for insulators if \( n \) is replaced by \( N \). These equations are generalized for homogeneous, isotropic, semi-infinite absorbing media by the substitution of the complex index defined by Eq. (5). Eq. (13) becomes

\[
\sin \Theta = \frac{1}{N} \sin \phi \quad (14)
\]

which indicates that \( \sin \Theta \) is complex, and that does not have its former simple significance. The ratios of Eqs. (11) and (12) also become complex quantities. We now define

\[
\frac{E''_s}{E_s} = \rho e^{i \delta} \quad (15)
\]

---

\[32\] Born and Wolf, op. cit., p. 612.
and

\[ \frac{E''}{E'_p} = \rho_s e^{i\delta_s} \quad (16) \]

where \( \rho_s \) and \( \rho_p \) are the amplitude reflection factors, and \( \delta_s \) and \( \delta_p \) give the phase changes on reflection. Also let

\[ N \cos \Theta = (N^2 - \sin^2 \phi)^{1/2} = a - ib \quad (17) \]

Since the intensity is proportional to the square of the amplitude, the reflectivity of light polarized perpendicular to the plane of incidence is given by

\[ R_s = \rho_s^2 \quad (18) \]

and the reflectivity of light polarized parallel to the plane of incidence is given by

\[ R_p = \rho_p^2 \quad (19) \]

Eq. (11) becomes

\[ \rho_s e^{i\delta_s} = \frac{-N \cos \phi - \cos \phi}{N \cos \phi + \cos \phi} \]

\[ = \frac{(a - \cos \phi) - ib}{(a + \cos \phi) - ib} \quad (20) \]

Eq. (12) becomes

\[ \rho_p e^{i\delta_p} = \rho_s \frac{\cos (\phi + \epsilon)}{\cos (\phi - \epsilon)} e^{i\delta_s} \]

\[ = \rho_s \frac{N \cos \phi \cos \phi - N \sin \phi \sin \phi}{N \cos \phi + N \sin \phi \sin \phi} e^{i\delta_s} \]

\[ = \rho_s \frac{(a \cos \phi - \sin^2 \phi)}{(a \cos \phi + \sin^2 \phi)} - ib \cos \phi e^{i\delta_s} \quad (21) \]
Therefore,

\[ R_s = \frac{(a - \cos \phi)^2 + b^2}{(a + \cos \phi)^2 + b^2} \]

\[ = \frac{a^2 + b^2 - 2a \cos \phi + \cos^2 \phi}{a^2 + b^2 + 2a \cos \phi + \cos^2 \phi} \quad (22) \]

and

\[ R_p = \frac{1}{\beta} \frac{(a \cos \phi - \sin^2 \phi)^2 + b^2 \cos^2 \phi}{(a \cos \phi + \sin^2 \phi)^2 + b^2 \cos^2 \phi} \]

\[ = R_s \frac{a^2 + b^2 - 2a \sin \phi \tan \phi + \sin \phi \tan \phi}{a^2 + b^2 + 2a \sin \phi \tan \phi + \sin \phi \tan \phi} \quad (23) \]

From Eq. (17)

\[ N^2 \cos^2 \theta = (a - ib)^2 \]

\[ = a^2 - 2ab - b^2 \]

\[ = N^2 - \sin^2 \phi \]

\[ = n^2 - k^2 - \sin^2 \phi - 2i nk \]

By equating real and imaginary parts, we find

\[ a^2 - b^2 = n^2 - k^2 - \sin^2 \phi \quad , \quad ab = nk \quad (24) \]

Now

\[ a^2 + b^2 = \left[ (a^2 - b^2)^2 + 4a^2 b^2 \right]^{\frac{1}{4}} \]

\[ = \left[ (n^2 - k^2 - \sin^2 \phi)^2 + 4n^2 k^2 \right]^{\frac{1}{4}} \]

The solution of Eqs. (24) is, therefore

\[ a = \left\{ \frac{1}{2} \left[ (n^2 - k^2 - \sin^2 \phi)^2 + 4n^2 k^2 \right]^{\frac{1}{4}} + \frac{1}{2} [n^2 - k^2 - \sin^2 \phi] \right\} \]

\[ b = \left\{ \frac{1}{2} \left[ (n^2 - k^2 - \sin^2 \phi)^2 + 4n^2 k^2 \right]^{\frac{1}{4}} - \frac{1}{2} [n^2 - k^2 - \sin^2 \phi] \right\} \quad (25) \]

For normal incidence Eqs. (22) and (23) reduce to
\[ R_s = R_p = \frac{(n - 1)^2 + k^2}{(n + 1)^2 + k^2} \]  

\[ (26) \]

Any arbitrary plane vibration form may be resolved into components in two mutually perpendicular directions. If we denote the corresponding intensity components of an incident light beam in the planes perpendicular and parallel to the plane of incidence as \( I_s \) and \( I_p \), then the proportion of polarization, \( P \), is defined by

\[ P = \frac{I_p - I_s}{I_p + I_s} \]  

\[ (27) \]

The degree of polarization is defined as the absolute value of the proportion of polarization. If \( I_0 \) is the unresolved intensity of the incident beam, \( I'' \) the unresolved intensity of the reflected beam, \( R_p \) the reflectivity corresponding to incident light with proportion of polarization \( P \), then evidently

\[ I'' = R_p I_0 = R_s I_s + R_p I_p \]  

\[ (28) \]

Using Eq. (27) and solving Eq. (28) for \( R_p \) gives

\[ R_p = R_s (1 - P) + R_p (1 + P) \]  

\[ (29) \]

The reflectivity is then given as a function of four variables: 

\( n, k, P, \text{ and } \phi \).
CHAPTER III

THE EXPERIMENTAL APPARATUS

The Ultraviolet Monochromator

As the source of monochromatic light an ultraviolet monochromator constructed by Ball Brothers Research Corporation of Boulder, Colorado was used. An external view of the monochromator is shown in Fig. 1. The unit was mounted on aluminum tracks supported by oak rails and fitted with a series of leveling screws as shown in Fig. 2. This arrangement facilitates cleaning and adjustment with subsequent rapid re-attachment to the reflectometer. An internal view is shown in Fig. 3. Since electromagnetic radiation in the extreme ultraviolet is strongly absorbed by air, it is necessary to evacuate the chamber containing the optical components (A, Fig. 3). This is accomplished by a water-cooled oil diffusion pump (B, Fig. 3) in series with a mechanical vacuum pump (C, Fig. 3). The internal arrangement of the optical components is shown in Fig. 4. The source of light is a normal glow discharge contained by an air-cooled Pyrex glass tube. A hollow aluminum cathode and slit anode are used. This assembly is enclosed by an aluminum housing (A, Fig. 4). The
gases used in the source are conducted by flexible tubing from storage tanks to the discharge tube, and then through the source exit slit into the monochromator vacuum chamber to be exhausted by the pumping system. Argon, helium, and hydrogen were used. Hydrogen and helium were passed through a liquid nitrogen cold trap to eliminate water vapor and other condensable impurities (A, Fig. 2). Argon was passed through a cotton filter. The wavelengths of the strongest spectrum lines obtained were 1215.7 Å, 1048.2 Å, 919.8 Å, 584.3 Å and 303.8 Å.

Light from the source is directed to a concave diffraction grating at a distance of 9.5 cm to 17.0 cm with an angle of incidence of 82° (B, Fig. 4). The grating contains 600 lines per mm. The radius of curvature of the grating is 998.5 cm. Grating and source are movable, and are mounted in a manner described elsewhere. 33 The spectrum of the grating is focused on the fixed monochromator exit slit (C, Fig. 4). The slit width is adjustable in the range from 0 to .020 in. A lead screw and gear train connected to an external counter and wheel is utilized to manually select the desired wavelength in the spectrum (D, Fig. 4) The resolution of the monochromator is 1-2Å.

Figure 1.
An External View of the Ultraviolet Monochromator
Figure 2.

The Ultraviolet Monochromator Mounting
Figure 3.

An Internal View of the Ultraviolet Monochromator
The Vacuum Reflectometer

The vacuum reflectometer is shown in Fig. 5. It is connected to the monochromator through a 2½ in. inside diameter tube (A, Fig. 5). An internal view of the reflectometer is shown in Fig. 6. The internal dimensions of the steel vacuum chamber are 32 in. x 18½ in. x 7 7/8 in. From the exit slit of the monochromator the light beam is deflected by a concave collimating mirror 1¼ in. in diameter with a radius of curvature of 460 cm (A, Fig. 6). The collimating mirror renders the divergent beam from the monochromator horizontally parallel. The beam remains somewhat divergent in the vertical plane, but it can be seen that this will have no effect on the measurement of specular reflectivity of a plane surface. After reflection from the sample mirror B, Fig. 6, the beam falls upon the phosphor coated photomultiplier tube contained in an aluminum housing (C, Fig. 6) which is mounted on the detector arm. The detector arm is supported by metal wheels and travels on a semi-circular track. Zinc oxide phosphor (Sylvania #137) was used to coat the 931A photomultiplier tube. It was found that a very thin uniform coat of this phosphor applied by mixing with ethyl alcohol was most effective. The sample mirror mounting and the detector arm are geared in a 2:1 ratio and driven about a common center of rotation by a motor (A, Fig. 7) through angles of incidence from 5° to 90° in 1½ min. This arrangement insures that the reflected beam remains on the detector during a complete cycle.
The sample mirror may be raised and lowered by the mounting shaft which is extended below the vacuum chamber through "O ring" seals. The mirror shaft is equipped externally with a tapered pin (B, Fig. 7). For vacuum evaporation in situ, and for measuring the direct beam intensity, the mirror is lowered below the light beam and the detector arm placed at the direct beam position. For measurement of reflectivity, the mirror is raised and locked into adjustment with the detector arm using the tapered pin. Both detector arm drive shaft and mirror mount shaft are fitted with position scales marked in degrees (C, Fig. 7). In addition, a series of small micro-switches mounted at ten degree intervals around the detector track provides a recorded indication of detector position through an appropriate electrical circuit. (D, Fig. 6). The reflectometer is equipped with a semi-circular, metal, 35 mm film holder (E, Fig. 6) which is raised to beam height or lowered behind a mask by means of a reversible motor (F, Fig. 6). This motor was carefully cleaned to avoid excessive outgassing. A vacuum door (G, Fig. 6) is provided which may be closed by means of a relay so that the reflectometer may be opened to atmospheric pressure while a vacuum is maintained in the monochromator, thus decreasing the time required to reach operating pressures after opening the reflectometer. The reflectometer is equipped with a water cooled oil diffusion pump (D, Fig. 7) in series with a mechanical vacuum pump (E, Fig. 7). A cold trap (H, Fig. 6) filled with liquid nitrogen, serves to pump condensable vapors and to
trap pump oil vapors entering the vacuum chamber. The chamber is equipped with a Phillips pressure gauge and a Pirani gauge, both mounted at J, Fig. 6.

Evaporation of the sample mirror was accomplished in situ, and reflectivity measurements were made without breaking the vacuum to avoid surface contamination. In Fig. 8 is shown the arrangement of the evaporation apparatus. Power is supplied from an ac transformer through a ceramic lead in the front chamber wall (B, Fig. 5) to the evaporation source filament (A, Fig. 8). An aluminum container (B, Fig. 8) protects surfaces in the reflectometer from deposits and serves as a platform for a mirror (C, Fig. 8) which is used to observe film thickness. A glass window (C, Fig. 5) is provided in the chamber top through which the filament and substrate may be observed directly from above, or by reflection in the mirror. A metal mask (D, Fig. 8) protects the substrate from heat and deposits during the time required to bring the material to be evaporated to the proper temperature. The substrate is revolved to face the evaporation source for formation of the film. The distance from filament to substrate is approximately ten centimeters. With this separation of filament and substrate the thickness of the film varies over the area of the substrate, being thickest at the center and thinner toward the edges. Since moderately thick films were produced, the thickness of the absorbing films was such as to be opaque over the entire area used for measurement. Photographs of the reflected beams showed that this variation
was not sufficient to adversely effect the width of the reflected beam.

The Amplifier-Recorder Unit

The 931A photomultiplier tube is powered by a 900 volt dc power supply (A, Fig. 9). The output from the photomultiplier is connected to a Leeds and Northrup microvolt amplifier (B, Fig. 9). The electrical connections to the vacuum chamber are made by shielded cables. The amplified signal is displayed on a Type G Speedomax Leeds and Northrup chart recorder (C, Fig. 9). The detector arm position recording circuit is connected directly to the recorder input and causes marks of varying height to appear at ten degree intervals as the detector arm travels through changing angles of incidence. Representative traces obtained on the recorder are shown in Fig. 10.
Figure 6.

An Internal View of the Vacuum Reflectometer
Figure 7.

The Vacuum Reflectometer Controls and Pumping System
Figure 9.

The Amplifier-Recorder Unit
Figure 10.

Typical Recorder Traces
CHAPTER IV.

THE EXPERIMENTAL PROCEDURE

General Discussion

Several difficulties must be overcome in obtaining an accurate measure of reflectivity for varying angles of incidence. The decreasing effective aperture of the reflecting surface of the sample for higher angles of incidence may cause it to become the limiting aperture of the optical system, which will lead to incorrect measures of reflectivity. The same portion of the photosensitive surface of the detector should be used for all measurements to avoid errors due to variation in photosensitivity. The source intensity and the sensitivity of the detection equipment must remain constant during the time required to measure the incident and reflected beam, or some method must be used to correct for variations.

The first of these difficulties is avoided in this apparatus by choosing the sample mirror of sufficient size. Glass microscope slides two inches wide were used as substrates. The horizontal width of the parallel incident beam was 1/8 inch. With these values, the angle of

\[ 34 \]

incidence at which the sample becomes the limiting aperture is eighty-six degrees twenty-five minutes.

The errors arising from variations in photosensitivity were more difficult to evaluate. The photomultiplier housing was provided with an adjustable slit 2.6 mm in height. It was found during the procedure of alignment, using an artificial white light source in the monochromator source housing, that the width of the slit required to permit the 1/8 in. wide reflected beam to remain on the photomultiplier tube during operation was 3/16 in. This was necessary due to slight vibration of the detector arm from actuation of the position micro-switches, friction in the bearings, and torque effects in the shafts. It was possible to align the components so that no difference in the indicated intensity of the reflected light during operation toward normal incidence and toward grazing incidence could be detected. The detector and mirror could also be rotated manually and the reflected intensity maximized at each angle. The values of reflectivity obtained in this manner were found to be equal to those obtained during automatic operation within the range of experimental error.

The intensity of the source was found to remain quite constant after a sufficient operating time of from ten to thirty minutes had elapsed. No sudden changes in the sensitivity of the detection circuit were observed during normal operation. The main amplifier was powered continuously to avoid any changes in sensitivity due to thermal variations or other factors during warm-up. The recorder was
powered only while in use, but its sensitivity would not be expected to show appreciable change during the four to five minutes required for a run. It was assumed that the intensity response of the detection system was linear.

Preparation of the Samples

Smooth, uncontaminated mirror surfaces were obtained by vacuum evaporation of films of the materials onto well cleaned substrates mounted in the reflectometer, and by measuring them almost immediately without increasing the chamber pressure above that required for operation. (about $10^{-4}$ mm Hg.). The entire measurement process using five wavelengths required approximately two hours. The substrate was prepared by the same procedure for all evaporations. The glass microscope slide was thoroughly scrubbed in a detergent solution of warm water and Dreft. It was then immersed in a weak sodium hydroxide solution for fifteen minutes or more, rinsed in distilled water, immersed in seventy percent nitric acid for fifteen minutes or more, rinsed again in distilled water, and placed in a thirty percent solution of hydrogen peroxide to await mounting in the reflectometer. Prior to removal from the hydrogen peroxide, the surface of the slide was swabbed with a cotton swab. It was then rinsed in distilled water, followed by 95% pure ethyl alcohol, and held until dry. Rubber gloves were worn during all handling operations, including drying. No moisture condensation occurred after rinsing.
in the alcohol, and drying time was only a few minutes. The slide was then clamped in the mirror mount and the reflectometer was immediately evacuated. The liquid nitrogen cold trap was filled as soon as a pressure of 0.05 mm Hg. was indicated, and was kept filled during the entire period of evaporation and measurement. All evaporation was conducted at a pressure of $1-2 \times 10^{-5}$ mm Hg. as indicated by the Phillips gauge. Evaporation of metal was continued until the filament was not visible through the film and substrate, so as to obtain an effectively semi-infinite film. The lack of any multiple beam interference effects in the trace of the reflected beam was considered to be a further indication that the film was sufficiently thick for application of the generalized Fresnel equations. In the case of the visibly transparent dielectric materials, this was the only direct criterion of optical thickness immediately available since interference colors could not be observed through the chamber window. The thickness of these films was later obtained by multiple beam interferometry, using a method given by Scott. 35

Reflectivity Measurements

The following procedure was used in making reflectivity measurements. With the sample mirror in the down position, the

monochromator beam was adjusted for a maximum of intensity. A check on the amount of scattered light from the monochromator was made by adjusting the grating spectrum slightly away from the line setting and noting the photomultiplier response (A, Fig. 10). The source was then turned off momentarily and the dark current of the detector was recorded. The desired wavelength was then re-adjusted for a maximum of intensity, and the direct beam intensity was recorded (B, Fig. 10). The sample mirror shaft was then raised and locked into adjustment, and a complete reflectivity measurement made from grazing incidence to normal incidence and back to grazing incidence recording the reflected beam intensity and the marked increments of angle (C, Fig. 10). The direct beam intensity was re-measured to obtain any changes. Manual measurements to check alignment and beam width were made (D, Fig. 10). The grating was set to the position at which the scattered background intensity was determined and a reflectivity measurement made of the background reflected at all angles (E, Fig. 10). The source intensity was then re-checked (F, Fig. 10). Changes in source intensity or any irregularities in the trace obtained were considered in evaluating the run and other runs made if necessary. Absolute values of reflectivity were calculated by subtracting background from beam intensities, and taking the ratio of the resulting quantities. The data thus obtained is presented in Appendix A.

The noise level as observed in the recorded signal increased with increasing amplifier sensitivity settings. The amplifier range
settings are 1X, 2X, 4X, 10X, 20X, and 40X, in order of increasing attenuation. Typical amplifier settings ranged from 2X for weak spectrum lines to 20X for strong lines. The indicated intensity of the 304 A and 920 A lines was approximately one tenth that of the 1216 A and 584 A lines, while the indicated intensity of the 1048 A line was observed to be about half that of the strong lines. Absolute intensities were not obtained due to variation of photosensitivity with wavelength. Fig. 10 indicates the noise to signal ratio for 1216 A and 304 A.

A test of reproducibility of results with changing source intensity was made by making successive reflectivity measurements as the source current was decreased. It was found that the results were the same within the probable error of measurement with signal variations differing by a factor of five. For the lowest intensities the noise level made comparison difficult. The observed reflectivity for 920 A was comparable in magnitude to that at 1048 A, however. The noise to signal ratio seldom exceeded 1:20 for the weak lines and 1:100 for the strong lines.

Analysis of data obtained from successive measurements of a gold mirror is given in order of measurement in Table I for 1216 A. The data were obtained without breaking the vacuum. Various amplifier settings were used on each wavelength. Although the measures of precision given refer to the reliability of the average reflectivity, the spread of experimental values is indicated. The variation of values is
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seen to increase with the magnitude of the reflectivity and the angle of incidence.

**Transmission Measurements**

Some transmission measurements on an unbacked film of aluminum were made. An unbacked, visibly opaque film of aluminum was obtained by evaporating the metal onto a glass slide coated with a solution of sugar, water, and aerosol. The film and substrate were then placed, film up, in a tray and water was poured in until the water level reached the film edge. The film floated away from the substrate as the sugar was dissolved. Part of the slide which was uncoated by sugar retained a portion of the film. This was used to measure the approximate thickness of the film by multiple beam interferometry. The floating aluminum film was picked up on a brass strip in which two holes had been drilled, with the film covering one of these holes. This strip was mounted on an arm clamped in the sample mirror mount as shown in Fig. 11. It was placed in the direct beam for transmission measurement. The intensity, as measured through the open hole, was compared with that through the film at appropriate wavelengths. The sample was mounted as close to the detector as possible to minimize errors due to scattering or diffusion of the light on passing through the film.
Figure 11.

The Transmission Measurement Mounting
CHAPTER V.

THE METHOD OF DETERMINING THE OPTICAL CONSTANTS

The usual polarimetric methods of measuring optical constants of absorbing substances is not suitable in the extreme ultraviolet due to the experimental arrangement in a vacuum chamber and the strong absorption by all polarizing materials. The generalized Fresnel equations, Eqs. (22) and (23), may be applied to obtain the optical constants from reflectivity measurements. In order to determine the refractive index and extinction coefficient from these equations and Eq. (29), the reflectivity is measured for two or more angles of incidence. If the polarization of the incident light were known, the result would be at least two equations in the two variables n and k, which could then be solved. The form of the equations prevents solution by algebraic methods, but a simple graphical method, which was suggested by Tousey, can be used to solve them.\(^{36}\) Values of n and k in the expected range may be used with the appropriate values of

φ and P to calculate values of $R_p$. Graphs showing the functional relation between $R_p$ and n for constant k may be constructed and used to give values of n and k corresponding to the measured reflectivity. Plotting these pairs of values in an n-k plane for two or more angles of incidence should reveal one unique pair which satisfies the equations for these angles.

The proportion of polarization of the incident light as a function of wavelength was unknown in this case. The polarization investigations described below indicated that polarization was present, but information was not available for all wavelengths. It was necessary to adopt a trial and error method by assuming several values of P and following the graphical procedures outlined above for each value. Eq. (23) indicates that $R_p$ is less than $R_s$ for $n$ or φ not equal to zero. Consequently $P$, as defined by Eq. (27), might be expected to be negative due to reflections of the source beam from the diffraction grating or collimating mirror. The values of $P$ chosen for trial were 0, -0.25, -0.50, -0.75, and -1.00. Values of φ chosen for calculation were $10^\circ$, $30^\circ$, $50^\circ$, and $70^\circ$. The range of $n$ and $k$ was chosen as 0.0 to 2.0 in tenths.

The calculations of $R_s$, $R_p$, and $R_o$ were performed on the Control Data Corporation 160 digital computer at the National Bureau of Standards, Boulder, Colorado. The calculations of $R_p$ for values of $P$ other than zero were performed by machine calculator to four decimal places using the computer results. The resulting values
were carefully plotted to three decimal places on large twenty inch square graph sheets. These graphs are reproduced in Appendix B.

The reflectivity data were used to plot n-k graphs for each substance. Fig. 12 shows two such graphs for a silicon monoxide mirror at 584 A and indicates the effect of varying the assumed polarization value. The most consistent multiple intersection was chosen to indicate the optical constants. The "most probable" values were indicated in most cases, since intersections at a single point were not obtained due to experimental errors and graphical interpolation inaccuracies.
Figure 12.

Two Optical Constant Graphs
CHAPTER VI

RESULTS

Metals

The optical constants of aluminum, silver, and gold were obtained and are listed in Table II. The absorption coefficients calculated using Eq. (9) are also included. The values of P which were required to obtain the best intersection of the four curves on the n-k graphs is indicated for each wavelength. They were the same for all metals. The optical constants were indicated by the graphs to within about 0.05.

Aluminum

The aluminum used was in the form of a fifty mil wire. The purity was estimated to be at least 99%. It was evaporated from a hairpin-shaped filament consisting of three fifteen mil tungsten wires twisted together and formed into a loop. It has been shown that although the aluminum dissolves tungsten, no tungsten appears
in the resulting film. Only a few seconds were required to produce a completely opaque film. No pinholes or surface irregularities in the film were observed. The reflectivity as a function of the angle of incidence is shown in Fig. 13. Aluminum becomes transparent for wavelengths less than about 830 Å, therefore multiple-beam interference patterns are present at 584 Å and 304 Å. Since the aluminum film could not be considered a semi-infinite medium at these wavelengths, and the application of the generalized Fresnel equations was not appropriate, estimations of the optical constants were made. From the appearance of a sharp rise in reflectivity at 584 Å and 304 Å, it appears that the refractive index is less than one. Approximate angles of "total" reflection of 50° at 584 Å and 70° at 304 Å were used in Eq. (13) with Θ equal to 90°. Total reflection does not occur due to a finite value of the extinction coefficient. This is apparent from examination of the reflectivity graphs in Appendix B. The usual simple multiple-beam interference formulas were not used, since the magnitude of phase changes at the surfaces of the film and the variation of the refractive index with the angle of incidence is unknown. The extinction coefficient was obtained from transmission measurements on an unbacked, 1000 Å thick aluminum film. Eq. (10) was used with Q assumed equal to the measured value of reflectivity of

Figure 13.
The Reflectivity of an Aluminum Mirror
aluminum at normal incidence. The values obtained compare favorably with those given by Hass and Tousey. 38

Oxidation or the adsorption of gas molecules by the surface of aluminum is apparently rapid, even at the reduced pressures in the vacuum chamber. The data given in Appendix A were obtained within one hour after evaporation. The reflectivity at normal incidence was observed to have decreased by 10% after fifteen hours in the vacuum chamber at pressures of $10^{-4}$ mm Hg. to $10^{-5}$ mm Hg. Berning, Hass, and Madden have obtained higher values of reflectivity and a lower value of the extinction coefficient. 39 This may be due to a different purity of the metal, different evaporation conditions, errors in thickness measurements, or varying surface conditions. In order to obtain the true reflectivity of aluminum, it may be necessary to conduct measurements at very low pressures, and even during the process of evaporation.

**Silver**

The silver used was in the form of a fifty mil wire and was obtained from Goldsmith Brothers Smelting and Refining Company, Chicago, Illinois. It had a minimum assay of 99.95%. It was

38 Hass and Tousey, *op. cit.*

TABLE II.

The Optical Constants and Absorption Coefficients of Aluminum, Silver, and Gold.

<table>
<thead>
<tr>
<th>Wavelength</th>
<th>Aluminum</th>
<th>Silver</th>
<th>Gold</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>n  k  (\alpha)</td>
<td>n  k  (\alpha)</td>
<td>n  k  (\alpha)</td>
</tr>
<tr>
<td>1216 A</td>
<td>0.60  1.20  1.24</td>
<td>1.03  0.43  0.44</td>
<td>1.15  0.89  0.92</td>
</tr>
<tr>
<td>1048 A</td>
<td>0.70  1.00  1.20</td>
<td>1.00  0.45  0.54</td>
<td>0.98  0.75  0.90</td>
</tr>
<tr>
<td>920 A</td>
<td>0.53  0.64  0.87</td>
<td>0.90  0.45  0.61</td>
<td>0.81  0.62  0.85</td>
</tr>
<tr>
<td>584 A</td>
<td>0.77  0.09  0.19</td>
<td>0.92  0.33  0.71</td>
<td>1.07  0.71  1.53</td>
</tr>
<tr>
<td>304 A</td>
<td>0.93  0.03  0.12</td>
<td>0.93  0.10  0.41</td>
<td>0.86  0.27  1.12</td>
</tr>
</tbody>
</table>

Note: Units of \(\alpha\) are \(10^6/\text{cm}\).
evaporated from a 1/8 in. diameter helical coil of nineteen turns of fifteen mil molybdenum wire. Fifteen to twenty minutes were required to produce an opaque film. No defects in the film could be observed. It was apparently opaque throughout the wavelength range considered here.

**Gold**

Gold wire, obtained from Goldsmith Brothers Smelting and Refining Company, with a minimum assay of 99.95% was evaporated from a conical basket fashioned from fifteen mil molybdenum wire. Ten to fifteen minutes were required to produce an opaque film. No defects in the film were observed. The reflectivity of gold at normal incidence is higher than that of aluminum and silver at 584 A and 304 A.

**Dielectrics**

The optical constants of magnesium fluoride, silicon monoxide, and zinc sulfide were obtained and are listed in Table III. The absorption coefficients are included. The values of P which were required to obtain the best intersection of the four curves on the n-k graphs is indicated for each wavelength. They were the same for all dielectrics and were equal to those used for metals. The optical constants were indicated to within about 0.05.
Magnesium Fluoride

Magnesium Fluoride in massive crystalline form was obtained from the American Fluoride Corporation, New York. Spectrographic analysis had indicated a purity of at least 99.98% except for the magnesium content which was estimated to be more than 5%. It was evaporated from a molybdenum conical basket at a rate of about 500 Å/min. The resulting film was clear and transparent with interference colors present. The thickness as measured by multiple beam interferometry was 7500 Å.

Silicon Monoxide

The substance was obtained as amorphous, black, solid particles from A. D. Mackay, Inc., New York. It sublimes slowly at high temperature. A molybdenum conical basket was used. A brown transparent film was obtained with an evaporation rate of about 200 Å/min. The measured thickness was 5150 Å. Transmission data for silicon monoxide given by Astoin and Vodar for 100 Å to 1000 Å indicate low and constant absorption, a film approximately 1500 Å thick transmitting about one third of the incident beam. This was not indicated in this case. The optical properties of silicon monoxide films seem to depend strongly on evaporation

---

40 Astoin and Vodar, op. cit.
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\[^{40}\text{Astoin and Vodar, op. cit.}\]
conditions, which apparently vary the chemical constitution of the substance. 41

- **Zinc Sulfide**

Standard reagent grade neutral zinc sulfide powder was used. It was mixed with distilled water to produce a white paste. This was placed in a helical coil 1/8 in. in diameter wound from twenty turns of fifteen mil molybdenum wire. Prior to evaporation, it was baked for two hours at red heat in the vacuum chamber to eliminate water vapor and prevent decrepitation. It sublimes slowly and produced a clear film which showed weak interference colors. A thickness of 7000 A was measured. The values of optical constants shown in Table III compare favorably with those given by Cox, Waylonis, and Hunter. 42

A check on possible fluorescence of the sample was made by investigating the reflected beam width. This was done using photographs of the reflected beam, and by sweeping the detector slowly through the reflected beam. No fluorescence was detected.

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42 Cox, Waylonis, and Hunter, op. cit.
TABLE III.

The Optical Constants and Absorption Coefficients of
Magnesium Fluoride, Silicon Monoxide, and Zinc Sulfide

<table>
<thead>
<tr>
<th></th>
<th>Magnesium Fluoride</th>
<th>Silicon Monoxide</th>
<th>Zinc Sulfide</th>
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</thead>
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<td>$\alpha$</td>
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<td>0.47</td>
</tr>
<tr>
<td>1048 A</td>
<td>1.20</td>
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<td>0.82</td>
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<tr>
<td>920 A</td>
<td>1.03</td>
<td>0.42</td>
<td>0.57</td>
</tr>
<tr>
<td>584 A</td>
<td>0.93</td>
<td>0.33</td>
<td>0.71</td>
</tr>
<tr>
<td>304 A</td>
<td>0.93</td>
<td>0.07</td>
<td>0.29</td>
</tr>
</tbody>
</table>

Note: Units of $\alpha$ are $10^6$/cm.
Polarization Investigations

Polarization of the monochromator beam should be caused by a layer of aluminum oxide which would have formed on the aluminum coated diffraction grating. Using the optical constants of aluminum oxide as given by Hass, Hunter, and Tousey for 1216 A and 900 A, it is found from the generalized Fresnel equations that at an angle of incidence of 80° an unpolarized beam should become polarized with a proportion of polarization of -0.47 at 1216 A and -0.35 at 900 A. The angle of incidence with respect to the normal to the grating surface was 82°. A grating blaze angle of about 2° gave an actual angle of incidence on the groove surfaces of 80°. Further reflections, such as from the collimating mirror, would only increase the degree of polarization.

The polarization of the incident light at 1216 A was investigated by measuring the reflectivity and transmissivity of several cleaved crystals of lithium fluoride. The data are included in Appendix A. The crystals were disks 2 mm thick and 25 mm in diameter. They were obtained from The Harshaw Chemical Company, Cleveland, Ohio. A refractive index of 1.62 for lithium fluoride at 1216 A has been given by Schneider. Transmission measurements

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43 Hass, Hunter, and Tousey, *op. cit.*

gave an extinction coefficient of $4 \times 10^{-6}$ at 1216 A. Using these values in the generalized Fresnel equations, a value of -0.5 was obtained for $P$. The results were not conclusive due to the lack of a uniform reflected beam from the irregular surface of the crystals, but were sufficiently consistent to indicate that polarization was present.

The polarization values for which the most consistent intersections were obtained on the optical constant graphs are the same for each wavelength. A strongly polarized beam is indicated at 1216 A, and the polarization decreases with decreasing wavelength.

Discussion of Results

General features of the optical properties of these substances in the extreme ultraviolet are low reflectivity and values of optical constants which are frequently less than one.

The results indicate that for shorter wavelengths in the extreme ultraviolet, reflectivity may be expected to decrease to a very low value for both metals and dielectrics. Eq. (26) indicates that a high reflectivity corresponds to comparatively large values of $k$ or $n$. Values of $n$ and $k$ in the same magnitude range are apparently common in the extreme ultraviolet, which corresponds to low values of reflectivity. It may be seen from Eq. (9) that the extinction coefficient is proportional to the wavelength of the incident light. For this reason, a lower extinction coefficient does not necessarily
indicate less absorption. Low reflectivity is to be expected near the plasma frequency due to the possible proximity of a region of relative transparency. This frequency, as calculated assuming an effective electron mass equal to the rest mass and an electron density corresponding to the valence corresponds to a wavelength of 830 A for aluminum and to 415 A for gold and silver. A sudden change occurs in the optical properties of aluminum at about 800 A, while gold and silver experience a more gradual decrease in reflectivity as the wavelength is decreased. Gold has the highest reflectivity and the highest absorption coefficient at 584 A and 304 A of the substances measured.

Many metals have refractive indices less than one in the visible and near ultraviolet regions. It is well known that this involves no contradiction of relativity principles, since \( v \) in Eq. (1) represents the phase velocity, rather than the energy or signal velocity. Values of \( n \) for the substances measured are less than one at 304 A. This is in agreement with the behavior commonly observed in the X-ray region, where values slightly less than one are obtained.
CHAPTER VII.

CONCLUSION

Techniques and Applications

The accuracy of the method by which the optical constants were obtained is dependent upon the precision of the graphical curves, the accuracy with which the polarization of the incident light is known, and the precision of the measurement of reflectivity. The reliability of the method is dependent upon the underlying theory.

The theoretical reflectivity graphs and the optical constant graphs may be constructed to almost any desired degree of precision, so that inaccuracies arising from this factor may be overcome. The reflectivity graphs given in Appendix B should be useful for obtaining the optical properties or the optical constants of substances for any wavelength at which the theory is valid.

Fig. (12) indicates that the results are sensitive to changes in the assumed polarization values. Trial and error methods are frequently more time consuming and less accurate than direct measurements. Much time is required in preparation of graphs for arbitrary values of the polarization. It is possible to eliminate the
need to determine the polarization with suitable arrangements to rotate the sample about the line of direction of the incident light beam. It would require more elaborate experimental apparatus to provide this additional motion. The polarization of reflected light might be used to construct polarizers for use in the extreme ultraviolet.

The precision in specular reflectivity measurement determines the limits of the most probable values of the optical constants. Departures from perfect smoothness of the sample surface may lead to poor results from nonspecular reflection, or from nonconformity with the conditions assumed in obtaining the Fresnel equations. Surface contamination may lead to values of optical constants which are not representative of the substance under consideration.

Since it has been found necessary to replace classical electromagnetic theory with a quantum mechanical description of absorption and scattering processes in the X-ray region, there would seem to be some justification for doubting the validity of the classical theory of reflection in the extreme ultraviolet. It has been indicated that Fresnel's equations were found to apply fairly well to reflection of X-rays near grazing incidence. The extreme ultraviolet region is one of soft X-rays with energies from about 6 ev to 30 ev. Walker, Rustgi, and Weissler have determined photoelectric yields for

aluminum, gold, and silver in this range. \textsuperscript{46} X-ray absorption limits exist for many elements in the extreme ultraviolet range, so that quantum absorption phenomena may predominate over the type of absorption mechanism prevalent in the visible spectrum.

These factors result in a method of determining optical constants which is more difficult, more time consuming, and less accurate than is desirable. However, it appeared to be the only procedure which was experimentally appropriate for application to absorbing substances in the extreme ultraviolet. The results were found to be consistent with the known properties of substances in this spectral region.

In considering practical applications of the results, it must be kept in mind that the optical properties given here correspond to surfaces which have not been exposed to the atmosphere. The effects of oxidation and other surface contamination can severely effect the optical properties, as is indicated in the case of aluminum. A practical motivation is that of obtaining a high reflectivity for diffraction gratings and mirrors at short wavelengths. For larger reflectivity at low angles of incidence the use of a metal of high atomic weight, such as gold, is indicated. At high angles of incidence it may be possible to increase reflection by using a substance with a refractive index less than one, in order to approximate total reflection.

\textsuperscript{46}Walker, Rustgi, and Weissler, \textit{op. cit.}
For the substances measured, relatively small but finite values of extinction coefficients prevent this. It can be seen from the reflectivity graphs in Appendix B that for values of \( n \) less than that value corresponding to total reflection by a transparent substance, the reflectivity should decrease rapidly with increasing \( k \) up to a value for \( k \) of approximately 0.5, after which a slow increase in reflectivity should occur. A thin coat of lithium fluoride may serve to protect aluminum from oxidation and to preserve its higher reflecting power. Magnesium fluoride is now frequently used for this purpose, but becomes opaque at longer wavelengths than does lithium fluoride. The polarization of light reflected by absorbing substances should be considered in the construction of optical systems. Intensity of reflected beams may be increased or decreased by the orientation of reflecting surfaces.

Recommendations

In addition to the need for more accurate measurement of reflectivity and a simpler method of determining optical constants, several related subjects may be worthy of further study.

Optical properties seem to be particularly dependent on surface condition. It may be possible to obtain high reflectivity with special surface treatment, such as ion bombardment, improved evaporation techniques, or specialized polishing techniques.
The characteristic absorption of different substances as a function of wavelength in the extreme ultraviolet is quite different. This was shown in the cases of aluminum, which becomes transparent; lithium fluoride, which becomes opaque; and gold, with slowly varying properties. The causes of these differences should be worthy of investigation. More accurate absorption coefficients may be determined for transparent substances by measuring films of different thicknesses. Thin unbacked films of many substances are difficult to obtain, but evaporation onto fluorescent material is possible. Preliminary studies of bismuth and beryllium have indicated transmission properties in the extreme ultraviolet similar to those of aluminum.

Further investigations of phosphors most suitable for use in the extreme ultraviolet should be conducted. It may be possible to decrease the variation of photosensitivity over the surface of a phosphor by vacuum evaporation onto the tube envelope, or open tubes may eliminate the need for such a phosphor.

The theory of optical properties in the extreme ultraviolet should be extended. Consideration of the energy band structures of metals may lead to modifications in the free electron theories.
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APPENDIX A

DATA
# TABLE IV

List of Symbols for Data in Appendix A

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>DBm</td>
<td>Incident direct beam intensity in recorder units including background</td>
</tr>
<tr>
<td>DBk</td>
<td>Incident direct background intensity in recorder units</td>
</tr>
<tr>
<td>Dk</td>
<td>Detector dark current in recorder units</td>
</tr>
<tr>
<td>Amp</td>
<td>Amplifier sensitivity selector setting</td>
</tr>
<tr>
<td>Angle</td>
<td>Angle of incidence in degrees</td>
</tr>
<tr>
<td>RBm</td>
<td>Reflected direct beam intensity in recorder units including background</td>
</tr>
<tr>
<td>RBk</td>
<td>Reflected background intensity in recorder units</td>
</tr>
<tr>
<td>R</td>
<td>Calculated reflectivity in percent</td>
</tr>
<tr>
<td>T</td>
<td>Calculated transmission in percent</td>
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</tbody>
</table>

Note: Range of recorder units is 0 to 100.
TABLE V.

ALUMINUM DATA

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<th>DBm</th>
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Note: Interference pattern present  T: 15 (1000 A thick film)

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<th>Amp</th>
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Note: Interference pattern present  T: 42 (1000 A thick film)
| TABLE VI. |
| SILVER DATA |

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### TABLE X.

**ZINC SULFIDE DATA**

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### TABLE XI

**LITHIUM FLUORIDE DATA**

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Figure 14.

**Reflectivity of an absorbing medium as a function of refractive index for unpolarized light**
Figure 15.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.20.
Figure 16.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.50.
Figure 17.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.75.
Figure 18.

REFLECTIVITY OF AN ABSORBING MEDIUM AS A FUNCTION OF REFRACTIVE INDEX FOR LIGHT POLARIZED PERPENDICULAR TO THE PLANE OF INCIDENCE
Figure 19.

REFLECTIVITY OF AN ABSORBING MEDIUM AS A FUNCTION OF REFRACTIVE INDEX FOR UNPOLARIZED LIGHT
Figure 20.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of 30°.
Figure 21.

REFLECTIVITY OF AN ABSORBING MEDIUM AS A FUNCTION OF REFRACTIVE INDEX FOR LIGHT WITH A PROPORTION OF POLARIZATION OF -0.50
Figure 22.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.75
Figure 23.

Reflectivity of an absorbing medium as a function of refractive index for light polarized perpendicular to the plane of incidence.
Figure 24.

Reflectivity of an absorbing medium as a function of refractive index for unpolarized light.
Figure 25.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.25.
Figure 26.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.50.
$\phi = 50^\circ$

Figure 27.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.75
Figure 28.

Reflectivity of an absorbing medium as a function of refractive index for light polarized perpendicular to the plane of incidence.
$\theta = 70^\circ$

**Figure 29.**

Reflectivity of an absorbing medium as a function of refractive index for unpolarized light.
Figure 30.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.28.
Figure 31.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of \(-0.50\).
Figure 32.

Reflectivity of an absorbing medium as a function of refractive index for light with a proportion of polarization of -0.75
Figure 33.

Reflectivity of an absorbing medium as a function of refractive index for light polarized perpendicular to the plane of incidence.