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## AN ELLIPSOMETER WITH VARIABLE ANGLE OF INCIDENCE FOR STUDIES IN ULTRAHIGH VACUUM

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16. ABSTRACT

   An ellipsometer that is designed for studies in ultrahigh vacuum is described. The windows of the vacuum chamber are incorporated into the optical bench system by means of flexible bellows. This allows measurements to be made over a large range of angle of incidence. The angle of incidence can be chosen so that maximum sensitivity is obtained. The principal angle of incidence can be determined, and straightforward corrections for strain birefringence of vacuum chamber windows can be made.

   Atomically clean surfaces of sodium chloride and lithium fluoride were investigated to verify the performance of the system. Submonolayer and monolayer coverage of water on these surfaces could be detected. On cleavage planes of NaCl, a first monolayer of adsorbed water is complete at about 1 torr only.

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# AN ELLIPSOMETER WITH VARIABLE ANGLE OF INCIDENCE FOR STUDIES IN ULTRAHIGH VACUUM

# I. INTRODUCTION

Ellipsometry has been widely used for the study of optical constants of surfaces and thin films. Film thickness up to tens of thousands of angstroms can be determined by ellipsometry within about 1 percent [1]; the instrument is sensitive enough to detect submonolayer coverage; and an accuracy of <1Å can be obtained [2-4].

For investigation of uncontaminated surfaces, film deposition, physisorption and chemisorption, ultrahigh vacuum or an extremely well controlled gaseous environment is required. This necessitates making optical measurements through vacuum chamber windows. Ellipsometers adapted to vacuum systems have been described by several authors [5-8]. Because fixed windows were used in these systems, measurements could be made only at a single angle of incidence.

Sensitivity considerations indicate that maximum experimental sensitivity is obtained at a specific angle of incidence, usually within ±10 deg of the principal angle [9-11]. Determination of the Brewster angle or the principal angle of incidence for substrate or film is a simple means of obtaining refractive indices [12-16]. For nonabsorbing isotropic media, the difference of phase shifts, $\Delta$, for the components of the electric vector in the plane of incidence and normal to it is $\pi/2$ at the Brewster angle, which can easily be determined by ellipsometry if the angle of incidence can be varied.

Since a single set of ellipsometer readings determines two quantities only, readings at various values of an independent variable such as wavelength, film thickness, substrate optical constants, refractive index of surrounding media, or angle of incidence have been considered by various authors [17-20] as a means of obtaining real and imaginary parts of the refractive index and film thickness by ellipsometry only. Of the several possibilities, variation of angle of incidence is experimentally and physically most appropriate. Shewchun and Rowe [9] have shown the viability of this approach; substrate optical constants, surface film thickness, and surface film optical constants were determined for metal/metal oxide systems.

Systematic errors in ellipsometry are generally due to (1) incorrect settings of polarizer, analyzer, and quarter wave plate; (2) an imperfect compensator; (3) multiple reflections between the various optical components; and (4) strain birefringence of cell windows. In the case of variable angle of incidence vacuum ellipsometry, strain birefringence of cell windows can only be eliminated if the light beam reproducibly probes a defined area of the windows for each measurement.

After consideration of the various advantages and possibilities of variable angle of incidence ellipsometry for surface studies in a controlled gaseous environment, an ellipsometer adapted to an ultrahigh vacuum system was designed and constructed. The apparatus allows for variation of angle of incidence and for corrections of window effects.

# II. APPARATUS

## A. Mechanical and Optical Arrangement

The basic experimental arrangement is illustrated in Figures 1 and 2. Figure 1 shows the ellipsometer frame (1)[1] plus optical components (2-7) attached to one of the two 15.24 cm (6 in. ) top flanges of an ultrahigh vacuum chamber (8). The frame consists of two optical benches (9) welded[2] to a cylindrical adapter (10). Both ellipsometer arms can be rotated concentrically and independently by means of large [28 cm (11 in. ) diameter] precision ball bearings. The adapter is mounted to the top of the vacuum chamber by a central socket ball joint (11); three set screws (12) allow tilting of the apparatus with respect to the chamber. The sample to be studied is positioned in the chamber with its surface coincident with the axis of rotation of the ellipsometer arms; misalignment of up to 5 deg can be compensated by tilting the ellipsometer.

Figure 2 shows a schematic of the optical arrangement; a helium neon laser (1) ($\lambda$ = 6328 Å ) was used as the light source, and a photomultiplier (2) and picoammeter were used as the detector. Ellipsometer-grade Glan-Thompson prisms (3) were used as the polarizer and analyzer; a mica quarter-wave plate (4) was used as the compensator. The polarizer could be set to a ±45 deg position relative to the plane of incidence; the compensator and analyzer

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1. The numbers in parentheses correspond to the same designations on the figures.
2. 6061-T6 aluminum alloy construction; dimensions determined by load testing and stress analysis to provide negligible deflection and mechanical instabilities.

LEGEND

(1) ELLIPSOMETER FRAME
(2) He Ne LASER
(3) POLARIZER
(4) BELLOWS-WINDOWS
(5) COMPENSATOR
(6) ANALYZER
(7) PHOTOMULTIPLIER TUBE
(8) ULTRAHIGH VACUUM CHAMBER
(9) OPTICAL BENCHES
(10) ADAPTER
(11) SOCKET BALL JOINT
(12) SETSCREWS
(13) ION PUMP
(14) MASS SPECTROMETER HEAD
(15) GAS SUPPLY
(16) PRESSURE GAUGE
(17) DIVIDED CIRCLES
(18) VERNIERS
(19) SAMPLE MANIPULATOR
(20) ION BOMBARDMENT GUN
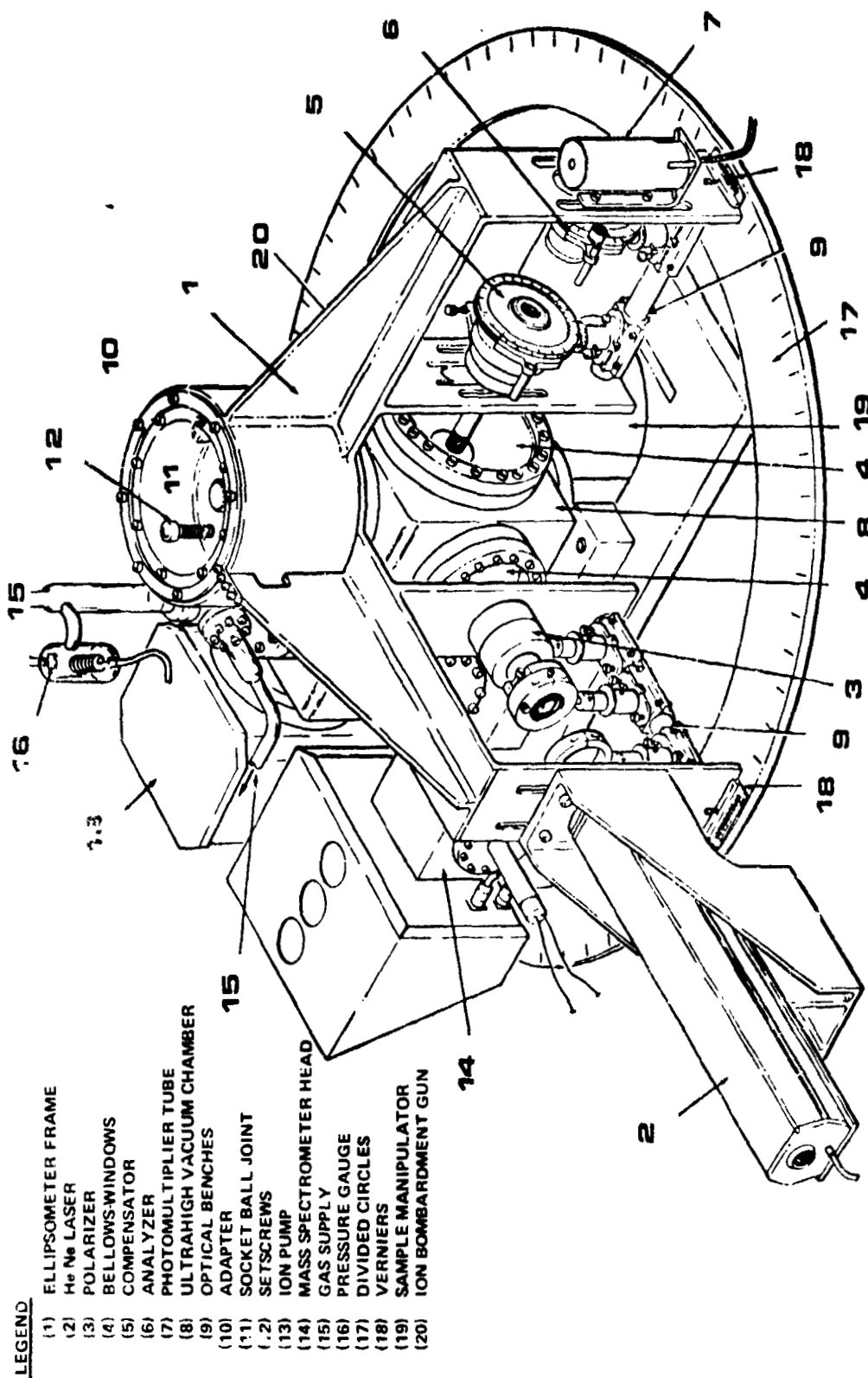
Figure 1. Variable angle of incidence ellipsometer with ultrahigh vacuum chamber and accessories.

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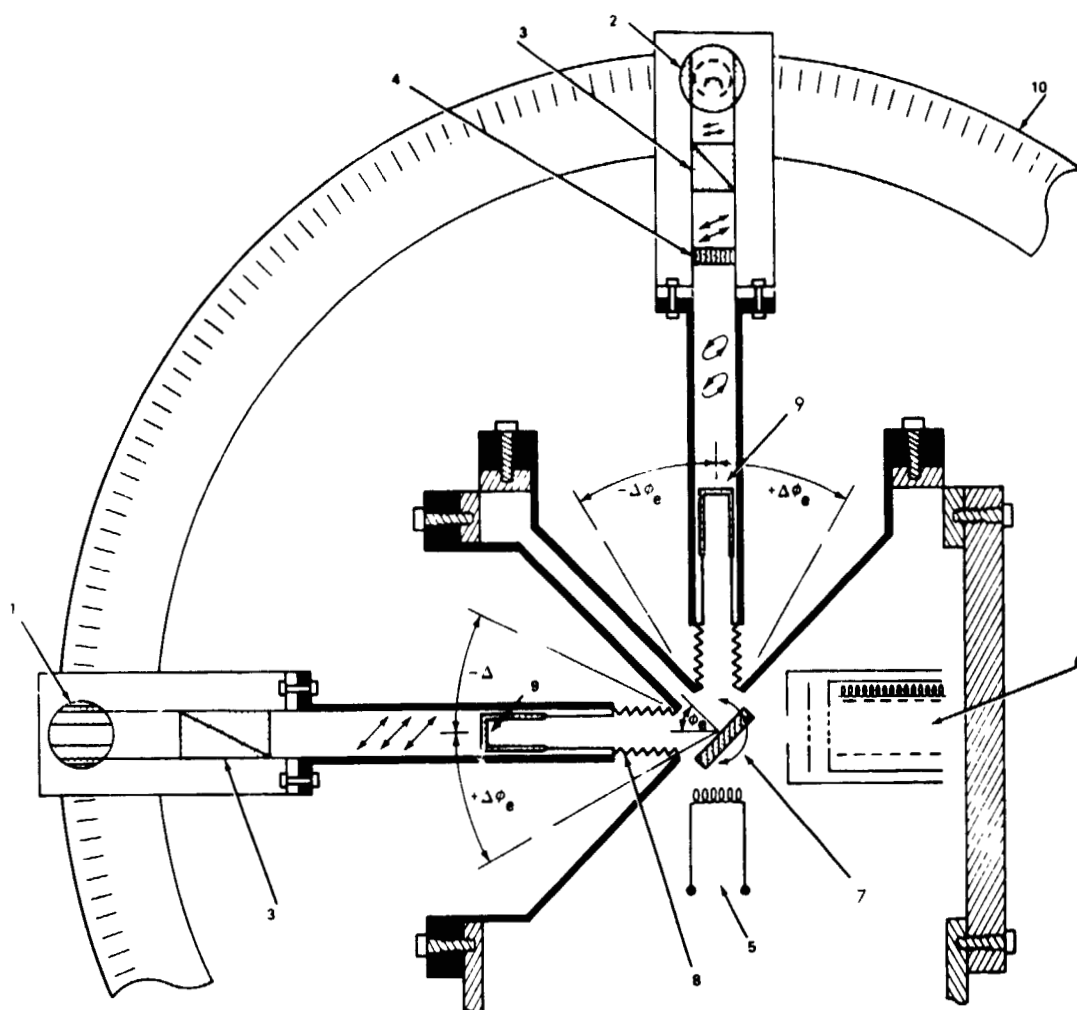
LEGEND

(1) He-Ne LASER
(2) PHOTOMULTPLIER TUBE
(3) GLAN-THOMPSON PRISMS
(4) QUARTER-WAVE PLATE
(5) FILAMENT FOR ELECTRON BOMBARDMENT HEATING
(6) ION GUN
(7) SAMPLE
(8) FLEXIBLE BELLOWS
(9) PYREX WINDOW
(10) DIVIDED CIRCLE

Figure 2. Optical arrangement and variation of angle of incidence.

were mounted in divided circles readable to 10 sec of arc. As indicated in Figure 2, a PSCA (polarizer-sample-compensator-analyzer) configuration was chosen.

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Auxiliary equipment, such as an ion pump (13), a mass spectrometer (14), a gas supply system (15), and an ionization and thermocouple pressure gauge (16), is indicated in Figure 1. Figure 2 shows the tungsten filament (5) for electron bombardment heating and the ion bombardment gun (6) for cleaning samples. A turbo molecular pump with an associated mechanical roughing pump was used to reach $10^{-6}$ torr or better. Final pressures of the order of $10^{-10}$ torr were obtained by means of a conventional ion pump.

## B. Window Arrangement for Variation of Angle of Incidence

To reduce systematic errors, variable angle of incidence ellipsometry requires correction for strain birefringence of chamber windows. Since strains are known to vary across a window, simple and accurate corrections can only be made if all measurements are taken through the same region. For measurements at various angles of incidence, this condition can only be met if the windows are incorporated in the optical bench system. This was achieved by mounting the windows to the ellipsometer arms and sealing the windows to the vacuum chamber by means of flexible bellows as illustrated in Figures 1 and 2.

Figure 3 shows details of the window assembly. Optically flat ($\lambda/20$) and plane parallel ( <1 second of arc) Pyrex discs (1) were sealed to 0.63 cm (0.25 in.) diameter Pyrex tubing (2) with Kovar seals (3) After annealing, the windows were examined between crossed polarizers for strain. The two best window assemblies (of six constructed) were vacuum-brazed to stainless steel bellows (4). The bellows were heliarc-welded to conical stainless steel adapters (5) with Varian type ultrahigh vacuum flanges (6). The windows were rigidly attached to the optical benches (8). With a rectangular chamber geometry, the angle of incidence could thus be varied from 27.5 to 65 deg. Figure 1 shows the 1 m diameter divided circle (17) and vernier scales (18) that allowed measurement of the angle of incidence to 0.0125 deg.

## III. CORRECTIONS FOR SYSTEMATIC ERRORS AND COMPUTATION

An alignment procedure as suggested by McCrackin et al. [21] was used to obtain polarizer, compensator, and analyzer zero-settings. The vacuum chamber windows were inserted after zero-settings were obtained and treated as optical components of the system.

HELI-ARC WELD

VACUUM BRAZE

HELI-ARC WELD

HELI-ARC WELD

OPTICAL BENCH

**LEGEND**

(1)  PYREX WINDOW
(2)  PYREX TUBING
(3)  GLASS TO KOVAR SEAL
(4)  STAINLESS STEEL BELLOWS
(5)  STAINLESS STEEL ADAPTER
(6)  20.32 cm (8 in.) ULTRAHIGH VACUUM FLANGE
(7)  ADAPTER TO ELLIPSOMETER FRAME
(8)  ELLIPSOMETER FRAME
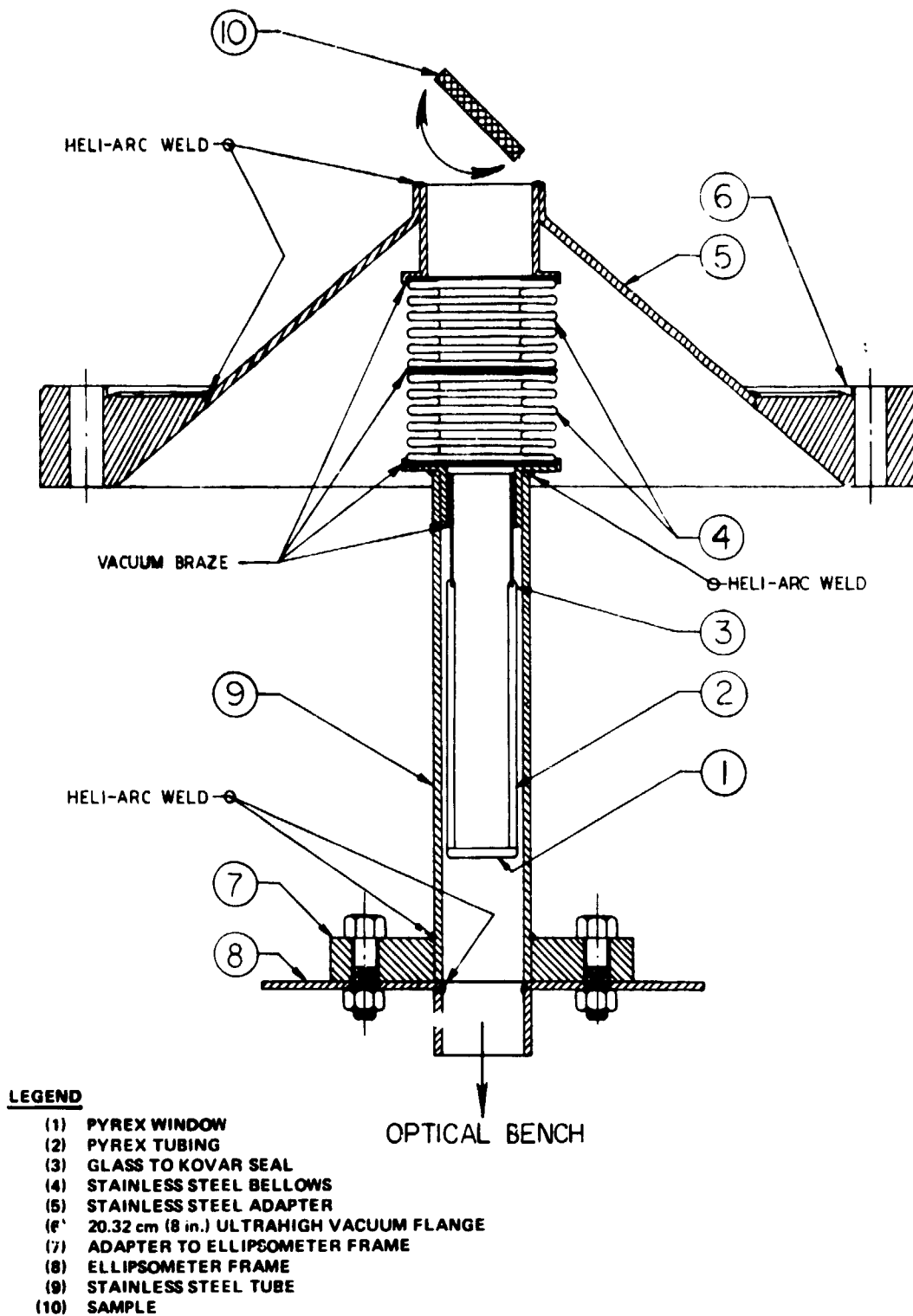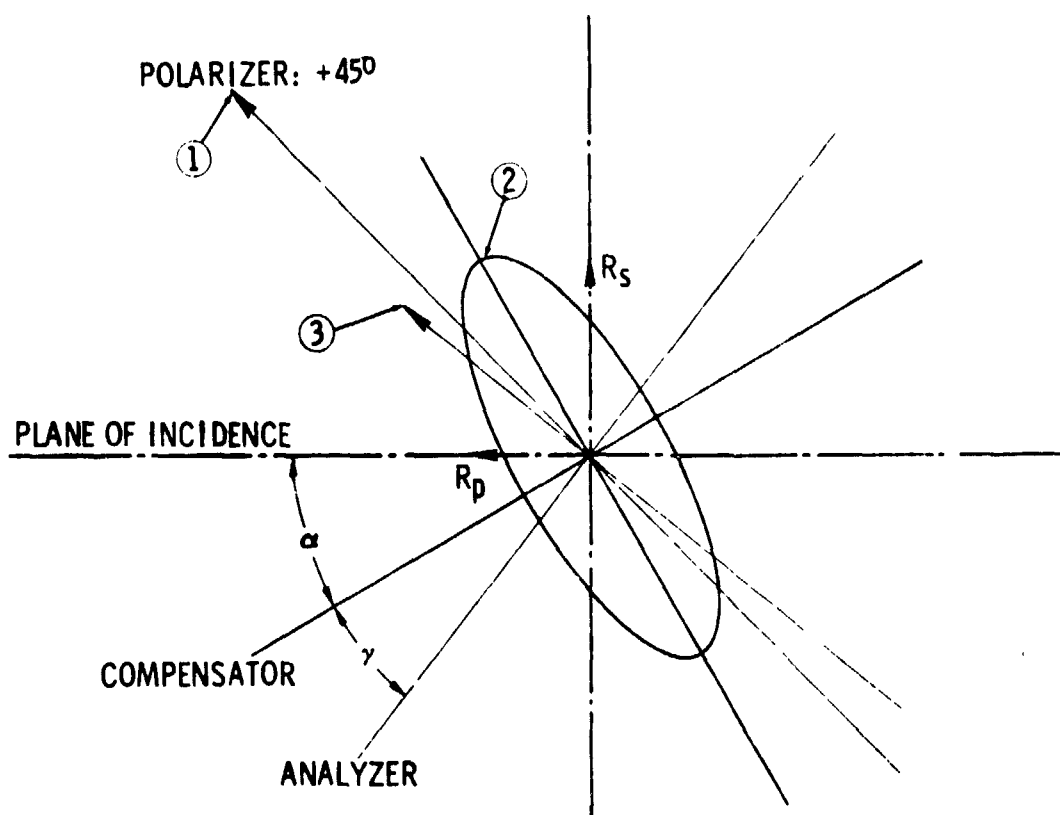(9)  STAINLESS STEEL TUBE
(10)  SAMPLE

Figure 3.  Window assembly.

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The relative attenuation, tan $\psi$, and the difference of phase shifts, $\Delta$, for the components of the electric vector in the plane of incidence and normal to it were obtained by determing $\alpha$ and $\gamma$ (Fig. 4) and using the following equations [22]:

$$\cos 2\psi = \cos 2\alpha \cdot \cos 2\gamma \tag{1}$$

$$\tan \Delta = \frac{\tan 2\gamma}{\sin 2\alpha} \quad . \tag{2}$$

POLARIZER: +45°

① 

② 

③ 

$R_s$

PLANE OF INCIDENCE

$R_p$

$\alpha$

$\gamma$

COMPENSATOR

ANALYZER

CONVENTIONS USED: R.H. MULLER, SURFACE SCIENCE, VOl. 16, 1969, P. 14:
α: COMPENSATOR AZIMUTH
γ: DIFFERENCE BETWEEN ANALYZER AND COMPENSATOR AZIMUTH.

LEGEND
(1)   INCIDENT, LINEARLY POLARIZED UNDER 45 deg
(2)   REFLECTED, ELLIPTICALLY POLARIZED
(3)   AFTER PHASE COMPENSATION

Figure 4.   Parameters of polarized light determined by ellipsometry.

The compensator (mica quarter-wave plate) was calibrated by Jerraid's method [23]; phase compensation was determined to be 91.215 + 0.003 deg.[3] The data, therefore, had to be corrected by applying the following equation:

$$\tan 2q = \tan 2(\alpha + \gamma)\cos\delta \quad , \tag{3}$$

where q is the correction required for compensator setting and $\delta$ is the compensation of the imperfect quarter-wave plate.

Incorporation of the vacuum chamber windows as optical components into the optical bench system allows determination of and correction for birefringence effects caused by localized strain in the windows. The first window was checked in the straight-through position; it was found to contribute 0.00581 to the relative amplitude attenuation, $\tan\psi$. Incorporation of the second window resulted in a change of 0.00029 at atmospheric pressure and of 0.00034 after the chamber was evacuated to ultrahigh vacuum. Respective phase shifts were -0.5970 deg and +0.4718 deg for the two windows and +0.1252 deg for the combined effect after evacuation of the chamber. Total values of $\psi_t$ and $\Delta_t$ with the sample are then described as follows:

$$\tan\psi_t\exp(i\Delta_t) = \tan\psi_1\exp(i\Delta_1)\tan\psi_2\exp(i\Delta_2)\tan\psi_3\exp(i\Delta_3)\tan\psi_s\exp(i\Delta_s) \quad . \tag{4}$$

where $\psi_1$, $\Delta_1$ and $\psi_2$, $\Delta_2$ are ellipticities produced by the two windows at atmospheric pressure and $\psi_3$, $\Delta_3$ are contributions from both windows with the chamber evacuated. $\psi_s$, $\Delta_s$ describe the sample under investigation.

Solving equation (4) for the reflectivity of the sample gives

$$\tan\psi_s\exp(i\Delta_s) = \frac{\tan\psi_t\exp(i\Delta_t)}{\tan\psi_n\exp(i\Delta_n)} \quad , \tag{5}$$

where $\psi_n$, $\Delta_n$ represent window contributions. Within error, no further contributions of window strains could be detected below 10 torr. Changes from 1 atmosphere to 10 torr were found to be reversible; therefore, evidently only elastic effects are involved.

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3. Commercially available $\lambda/4$ plates are accurate only within 3 percent.

If no corrections were made, the errors due to strain birefringence of the windows would be of the order of ±0.01 for refractive indices and absorption coefficient, and up to 5 Å for film thickness.

After correction for inaccurate compensator and window birefringence, major sources of error are caused by analyzer (A), quarter-wave plate (Q), and angle of incidence ($\varphi$) settings [24]. An uncertainty of angle of incidence of ±0.0125 deg results in an uncertainty in $\psi$ of ±0.01 deg and in $\Delta$ of ±0.0015 deg. The accuracy of azimuth readings for the quarter-wave plate and analyzer (±10 sec of arc) was sufficient to determine $\psi$ to ±0.003 deg and $\Delta$ to ±0.002 deg. The maximum total error in $\Delta$ and $\psi$ would, hence, be of the order of 0.005 deg and 0.02 deg, respectively.

Data analysis was done by several computer programs: (1) $\psi$ and $\Delta$ were calculated from experimental data and corrected for window birefringence as described in equation (5), (2) a second program was used to determine real and imaginary components of the refractive index of a film-free substrate, and (3) with n and k of the substrate known, two of the three parameters of a thin film (n, k, and film thickness d) could be computed. The approach was the usual directed trial-and-error procedure for a single value of $\psi$ and $\Delta$.

Computation of substrate optical constants, surface film thickness, and surface film optical constants of a film-covered substrate from a single set of ellipsometer readings at various angles of incidence as suggested by Shewchun and Rowe [9] could not be done. The required reproducible angular readings to 0.001 deg were not possible with the conventional photomultiplier detection system used. To obtain systematic readings of greater accuracy, a Faraday modulator and lock-in amplifier would have to be added, and measurements of angle of incidence would have to be improved to ±0.001 deg.

## IV. RESULTS AND DISCUSSION

For NaCl and LiF, values for $\psi$ and $\Delta$ were computed using bulk values for the substrate (NaCl: n = 1.5415, k = 0 [25]; LiF: n = 1.3910, k = 0 [26]; both at 6328 Å and 20°C) and assuming films with refractive index from 1.3 to 1.6. A film thickness of 3.0 Å was assumed for these calculations.

The properties of such films would be comparable to those of a monolayer of adsorbed water. Variation of $\varphi$ from 27.5 deg to 87.5 deg in increments of 2.5 deg indicates that changes in $\Delta$ and $\psi$ caused by the films are greatest

at reflection near the Brewster angle (57.02 deg for NaCl and 54.29 deg for LiF). Computed values for a 3 Å thick, nonabsorbing film with n - 1.3 on a NaCl substrate are given in Table 1 for 50 deg $< \varphi <$ 60 deg. With a transparent film, $\psi$ is not sensitive to the presence of a film; changes in $\Delta$ are more exaggerated, particularly around the Brewster angle. With an absorbing film, this behavior is reversed; again, best experimental sensitivity is obtained near the principal angle.

Film-free (100) surfaces of NaCl and LiF were obtained by cleaving single crystals in ultrahigh vacuum and bakeout at 350°C for 12 hours. Measurements were taken from 40 to 65 deg angles of incidence. Surface optical constants were computed as follows: $n_{NaCl} = 1.555 + 0.007$, $k_{NaCl} = 0.014 + 0.003$; $n_{LiF} = 1.402 \pm 0.007$, $k_{LiF} = 0.008 + 0.003$.

Since the difference between measured and bulk values could not be explained by systematic errors, it may be concluded that the lattice of alkalihalides is distorted in the surface region. Calculations of ion displacements of alkalihalides bounded by (100) faces by Benson et al. [27, 28] and successful correlation between such surface distortions and surface reactivities by Takaishi et al. [29] support this assumption. Similar effects are present with polished surfaces, polish layers, and leached-out regions with layer thicknesses of several hundred angstroms, and optical properties different from bulk properties have been detected by Yokota et al. [14]. Optically flat surfaces of clear fused quartz investigated in this study also indicate the presence of a surface layer with different optical properties. It seems that this situation is the common one rather than the exception; the frequently used practice of applying bulk values of substrates for computation of film properties may, therefore, lead to the misinterpretation of experimental data.

Admission of water vapor into the system did not alter ellipsometer readings in the pressure range $10^{-9}$ to $10^{-4}$ torr. The first monolayer of water is only completed at about 1 torr, which is in agreement with data obtained by volumetric measurements [30]. Since no variation in $\Delta$ and $\psi$ could be detected over six orders of magnitude of pressure, a film-free surface when investigated by ellipsometry can be assumed within this range. On the other hand, a large number of investigations prove that adsorption of water on cleavage planes of alkalihalides alters the surface properties with respect to heterogeneous nucleation, etc., even at pressures below $10^{-4}$ torr. These effects can be explained by a submonolayer coverage of water and masking of potential nucleation sites that are associated with points of higher free surface energy on energetically heterogeneous surfaces. More detailed results and interpretations on this subject will be presented separately.

TABLE 1. COMPUTED VARIATION OF ELLIPSOMETRIC PARAMETERS
$\Delta$ AND $\psi$ WITH ANGLE OF INCIDENCE $\varphi$ FOR A SODIUM
CHLORIDE SUBSTRATE (BULK CONSTANTS: $n_3 = 1.5415$,
$k_3 = 0$) WITH THIN SURFACE FILM ($n_2 = 1.30$, $k_2 = 0$,
$t_2 = 3$ Å). CHANGES $\partial \psi$ and $\partial \Delta$ CAUSED BY FILM
AS COMPARED TO $\Delta$ AND $\psi$ FOR FILM-FREE
SUBSTRATE ARE ALSO INCLUDED.

| Angle of Incidence $\varphi$ (deg) | $\psi$ (deg) | $\Delta$ (deg) | $\partial \psi$ (deg) | $\partial \Delta$ (deg) |
|---|---|---|---|---|
| 50.0 | 10.687 | 179.842 | 0.000 | −0.158 |
| 50.5 | 9.953 | 179.829 | 0.000 | −0.171 |
| 51.0 | 9.213 | 179.814 | 0.000 | −0.186 |
| 51.5 | 8.468 | 179.796 | 0.000 | −0.2·· |
| 52.0 | 7.718 | 179.774 | 0.000 | −0.·26 |
| 52.5 | 6.965 | 179.748 | 0.000 | −0.252 |
| 53.0 | 6.207 | 179.716 | 0.000 | −0.284 |
| 53.5 | 5.446 | 179.674 | 0.000 | −0.326 |
| 54.0 | 4.681 | 179.618 | 0.000 | −0.382 |
| 54.5 | 3.914 | 179.541 | 0.000 | −0.459 |
| 55.0 | 3.144 | 179.426 | 0.000 | −0.574 |
| 55.5 | 2.371 | 179.236 | 0.000 | −0.764 |
| 56.0 | 1.597 | 178.861 | 0.000 | −1.139 |
| 56.5 | 0.821 | 177.778 | 0.001 | −2.222 |
| 57.0 | 0.054 | 143.518 | 0.010 | −36.482 |
| 57.5 | 0.736 | 2.491 | 0.001 | +2.491 |
| 58.0 | 1.515 | 1.212 | 0.000 | +1.212 |
| 58.5 | 2.294 | 0.801 | 0.000 | +0.801 |
| 59.0 | 3.073 | 0.598 | 0.000 | +0.598 |
| 59.5 | 3.852 | 0.477 | 0.000 | +0.477 |
| 60.0 | 4.630 | 0.397 | 0.000 | +0.397 |

# V. CONCLUSION

A variable angle of incidence ellipsometer that is adapted to an ultrahigh vacuum chamber has been described. Incorporation of the vacuum chamber windows into the optical bench system allows for straightforward correction of window effects. Brewster angle or principal angle can be measured directly; reflectivity measurements can be taken at angles where maximum sensitivity is obtained — usually around Brewster or principal angle. Computed values for $\Delta$ and $\psi$ as a function of angle of incidence for thin films on alkalihalides show that for thin films, sufficient sensitivity to detect the presence of a film can only be obtained in the proximity of the Brewster angle. The system described allows investigation of uncontaminated surfaces in ultrahigh vacuum. Adsorbed or deposited films can be studied under controlled gaseous conditions.

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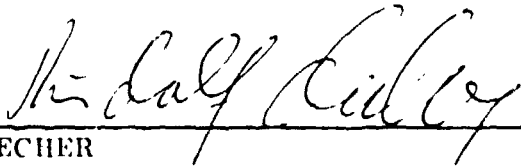30. Walter, H. U.: Z. Phys. Chem. N.F., vol. 75, 1971, p. 287.

# APPROVAL

# AN ELLIPSOMETER WITH VARIABLE ANGLE OF INCIDENCE FOR STUDIES IN ULTRAHIGH VACUUM

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and P. N. Peters

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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