NASA CR-54336

2

DEVELOPMENT OF OPTICAL COATINGS
FOR
CdS THIN FILM SOLAR CELLS

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By

J. C. Schaefer, E. R. Hill

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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FIRST QUARTERLY REPORT

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Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Space Power Systems Division
Clifford Swartz

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Crystal-Solid State Division
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FOREWORD

This report was prepared by The Harshaw Chemcial Company, Crystal-Solid State Division under Contract NAS 3-6464 for the NASA Lewis Research Center. Dr. A. E. Potter and Mr. C. Swartz have served as Technical Advisor and Project Manager respectively.

The Harshaw Solid State Laboratory is directed by Dr. J. M. McKenzie, Technical Director. Project direction is provided by Mr. E. R. Hill with Mr. J. C. Schaefer serving as Project Supervisor. The following Harshaw Chemical Company personnel have contributed to this program: R. W. Olmsted, R. J. Humrick and A. Hartzler.

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SUMMARY

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Coatings of SiO and MgF have been deposited by thermal vacuum evaporation. Films over 1 micron thick were mechanically unstable and thus were not suitable for use on the solar cell. A sputtering unit has been assembled to deposit glass coatings. This unit uses a triode-plasma technique with RF oscillatory potential applied to the target to allow sputtering of dielectrics. Measurements of reflectance of materials of known reflectance on surfaces similar to that of the solar cell were made. This is to evaluate the effect of Rayleigh scattering from the cell surface so that the reflectance data on the cell can be interpreted.

Author

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INTRODUCTION

The purpose of this program is to develop and apply optical coatings to CdS solar cells for the purpose of increasing the power conversion efficiency. It is desired to maximize the amount of light entering the cell in the region of the spectrum where the cell is sensitive, reject all others, and reduce the operating temperature of the cell in outer space. These conditions are not all independent since a coating which reflects strongly in the infrared will also reduce the emissivity in the region of maximum cell reradiation. The objectives stated can be reached by developing a coating with a high transmission to wave lengths less than about 1 micron, has a refractive index of about 1.2 to 1.3 in this same range and absorbs relatively strongly (about 10^{-3} to $10^{-4} cm^{-1}$) in the range of 5 to 30 microns. These properties are satisfied by ordinary glass and consequently shall be the first material to be investigated.

Other obvious properties to be desired are compatibility with the device, mechanical and chemical stability, and resistance to environmental effects. Additionally, it is desirable to have the coating seal the cell from atmospheric water vapor.

PREPARATION OF COATINGS

Description of Methods

1. Evaporation

Deposition of silica coatings by thermal evaporation of SiO in vacuum ambient is a standard process in the production of coated mirrors and multilayer optical coatings. Many other materials, such as CaF_2, and MgF_2 are also deposited in this fashion. Films of these materials have been deposited on the CdS cells in the past very successfully provided the deposited layer was less than about 1 micron thick. For the present purposes, however, the layer must be in the range of 3 to 10 microns thick. To produce mechanically stable films of this thickness requires heating of the deposition substrate to reduce the normally expected mechanical stresses. The temperature and time required are unfortunately out of the range of tolerance of the device and consequently, thick evaporated films have not been prepared to date.

2. Sputtering

A widely used, relatively old, but not widely understood process for film deposition is by cathodic sputtering in low pressure gaseous ambient. In this, high energy ions are caused to bombard a target, causing ejection of atoms and molecules from the target. These particles are then collected on the nearby substrate.

Almost any material which can be prepared in a suitable form can be deposited by sputtering, and in particular, dielectrics and refractory metals are generally amenable to the process. In this case the rate of deposition is proportional to the power absorbed by the target, and so the rate is limited by the temperature rise of the target and its surroundings. Typical rates for metals are in the range of 200Å per second and for dielectrics in the range of 100Å per second. A further advantage is that a large area target can be used to deposit onto large area substrates, and both the target and substrate can be water cooled.

Films Produced with Vacuum Evaporated Coatings

A number of vacuum evaporated SiO and MgF_2 coatings have been formed on CdS thin film cells. The SiO coating thicknesses were $.5\mu$, 1μ, and 3μ, whereas the MgF_2 coatings thicknesses were .5 micron and 3 microns. These films were prepared by vacuum evaporation at rates of 30 to 100Å per second onto room temperature substrates in an ambient pressure of 10^{-5} Torr.

Sputtering Equipment

A Consolidated Vacuum Corporation "AST-100" sputtering unit has been acquired and is in the process of being tooled for deposition of dielectric films. A brief explanation of the mode of operation of this equipment is pertinent at this time.

1. Plasma Techniques

The conventional, or at least original method for sputtering is commonly referred to as the "diode" technique wherein a cathode of the material to be sputtered and an anode of the substrate onto which the material deposits are placed in suitable relative positions in a low gas pressure ambient. The gas is usually argon or neon. A discharge is initiated by applying a high potential between anode and cathode, ionizing the gas between the electrodes. The positive ions strike the cathode causing ejection of atoms or molecules which deposit on the first cool surface contacted. For this process to function, the spacing of the cathode and anode must be more than 1 mean free path of a molecule in the gas. Because of this, a number of undesirable features result. First, the sputtered material is scattered by the ambient gas and losses due to scatter are high in practical configurations. Secondly, the gas molecules have a high probability of being incorporated in this layer. Lastly, the cathode temperature rises in spite of water cooling and since the source and target must be close, undesirable temperature rises are experienced on the substrate.

To overcome these problems, the triode or plasma technique was devised. Here the electrons are supplied in copious quantities by a heated filament. The electrons are then accelerated toward an anode causing ionization of the ambient gas on the way. The positive gas ions are then accelerated toward a third electrode held at a negative potential relative to the hot cathode. These ions eject material from the target, which material is then condensed on a nearby substrate which is held at cathode or ground potential. In this case, the cathode and anode can be rather widely spaced, and the gas pressure made correspondingly lower (about 1 to 0.1 micron). The plasma discharge is confined to the space between the cathode and anode and by means of a magnet can be concentrated to a density of about 1 ampere per square inch. The target and substrate are placed on opposite sides of the plasma column, a distance less than one mean free path. The obvious advantages of the system are the low gas pressure, essentially rectilinear propagation of the sputtered material, and confining of sputtering to only those elements to which high negative potentials are applied. Again the rate is limited by the temperature rises which are tolerable.

2. Films Produced by Sputtering

DC sputtering experiments have been concerned with the deposition of tantalum, aluminum, gold, silver and copper on various substrate materials. The substrates consisted of glass, CdS cells, aluminum oxide sheets, ammonium dihydrogen phosphate and potassium dihydrogen phosphate single crystals and "plexiglass." The purpose behind these experiments was twofold. They served as a means of checking-out the equipment while providing samples for evaluation. Discussion of sample films that were evaluated are in the Measurements section.

3. R. F. Sputtering

The D. C. Triode sputtering method is satisfactory for metals and material with resistivities above this range, the surface of the target acquires a layer of residual positive charge, reducing the field accelerating the ions, and sputtering ceases. This makes the sputtering of dielectrics impossible by this technique. To overcome this, an R-F oscillator has been connected from the target to the cathode. When the target is negative with respect to the cathode, normal sputtering occurs and the positive charge layer begins to accumulate on the target. When the target goes positive, electrons from the cathode discharge this layer, allowing sputtering to occur on the next half cycle. The frequency of the oscillator is determined by the system geometry and the target conductivity, with tens of megacycles being a convenient range of operation.

The target in this case takes the form of a condenser consisting of a layer of the dielectric material surrounding a metal plate electrode. Since it is desirable that maximum power be dissipated in the target, the conventional impedance matching networks between the target and oscillator are required. By this technique, then, any dielectric which can be prepared in the proper shape can be sputtered. The maximum rate then is determined by the temperature rise of the dielectric. This temperature rise can be large enough to cause materials such as glass to melt readily.

4. Progress and Present State

The CVC sputtering unit has been completely set-up with the exception of the RF special tooling attachments. The RF probe for dielectric sputtering has not been received. Figures 1 and 2 are photographs of the total equipment and the electrode arrangements in the bell jar, respectively. A probe has been constructed in the laboratory and has been installed in the system. It is in the process of being matched to the "driver" transmitter. Tests of capacitance, inductance and overall resonance were made to aid in the coupling of antenna (RF probe) to the transmitter. Initial ratios of about 100 to 1 of input power to useful power have been reduced to about 3.5 to 1.

MEASUREMENTS

Description of Measurements to be Made

1. Thickness

The thickness of the deposited films can be measured in several ways, 1) A Sloan Inst. multiple beam interferometer, 2) a conventional shallow field microscope capable of being focused on the top and bottom of the deposited layer, and 3) measurement of the gain in weight of the substrate after deposition. Thickness measurements made on films

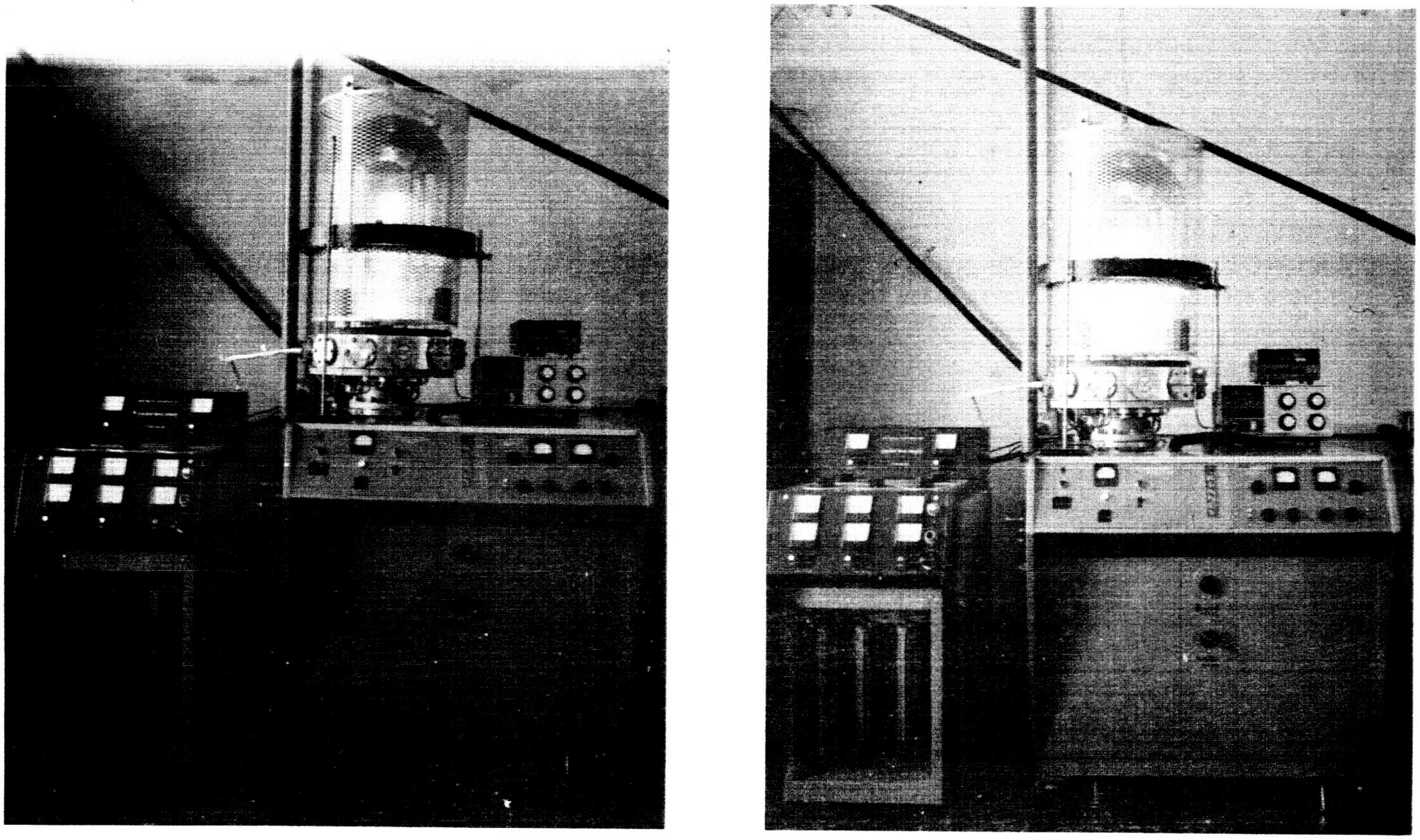


Figure 1. Sputtering Equipment and Pumping Stand

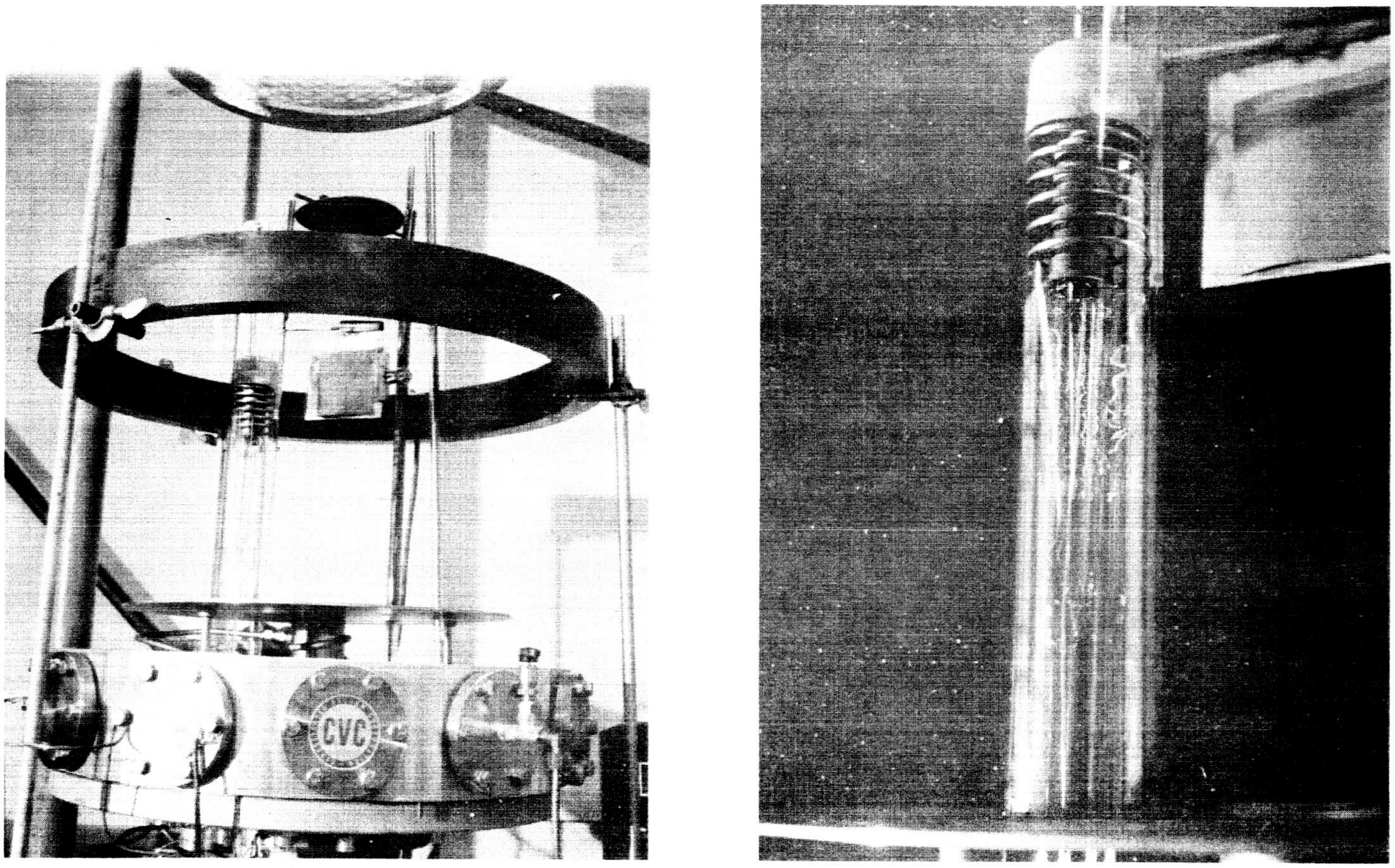


Figure 2. Target and Probe

produced by vacuum evaporation and sputtering techniques have been measured with the interferometer and microscope.

2. Qualitative Analysis

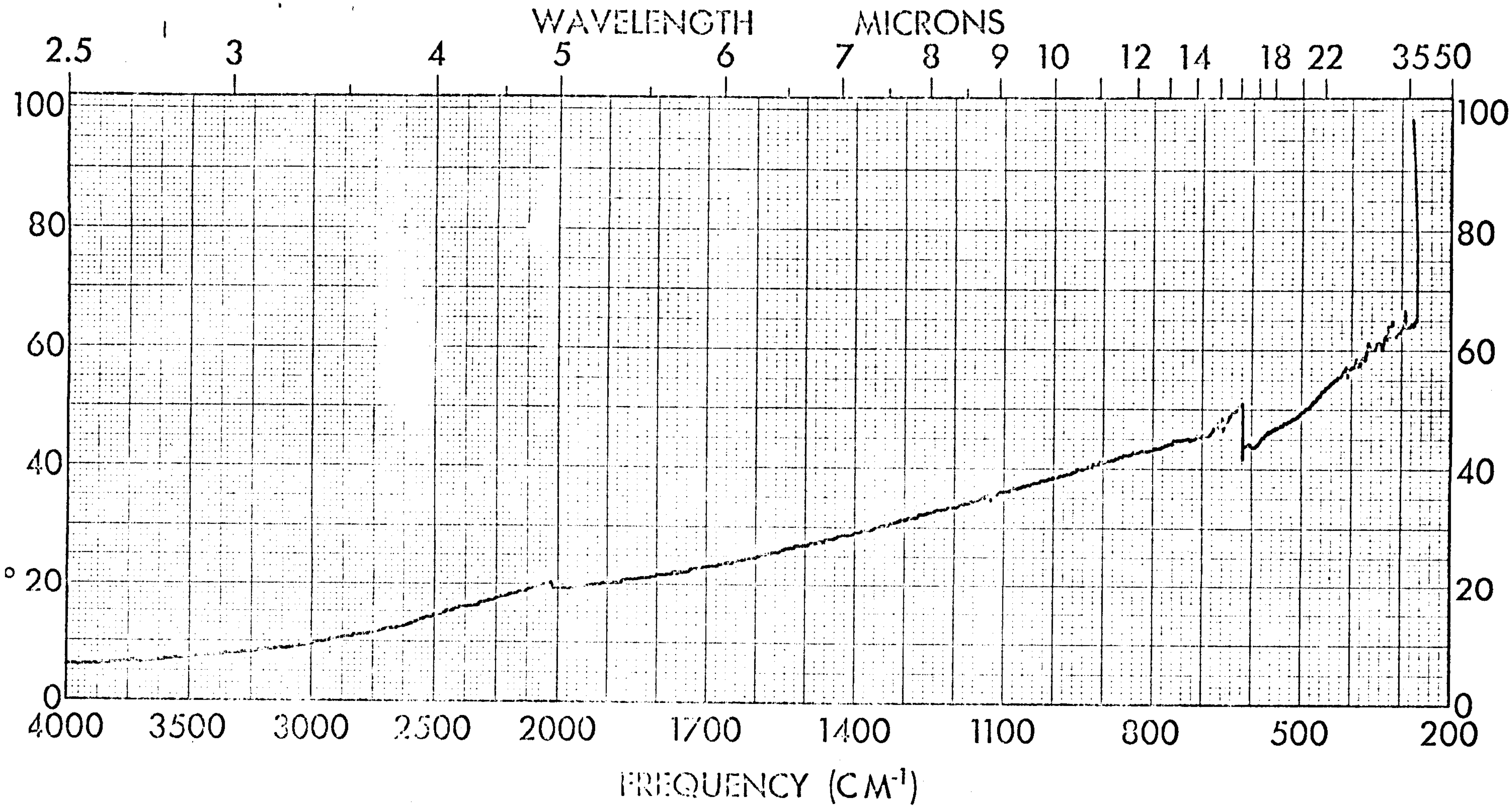
The surface of the deposited films are being examined microscopically for grain size, uniformity, adherence and conductivity. The sputtered films have exhibited grain sizes of 0.1 micron or less with uniformity and excellent adherence. Conductivity of the sputtered metals approach that of the bulk material.

3. Reflectance

The properties of the deposited layers of primary interest are the total visible and infra red reflectances. In order to evaluate the coatings it was necessary to employ a G E spectro photometer equipped with an integrating sphere capable of measuring total reflectance in the range from 0.3 to 1.2 microns in conjunction with a Perkin-Elmer spectophotometer capable of measuring specular reflectance in the range from 2.5 to 30 microns. Since the samples to be measured will not have specular surfaces, and in fact have a roughness with dimensinns in the range of 10 microns, Rayleigh scattering becomes a major factor in determining the functional dependence of specular reflectance on wavelength. As will be recalled from Rayleigh's work, the intensity of scattering from small particles is proportional to the reciprocal fourth power of the wavelength and proportional to the square of the particle volume.

Data

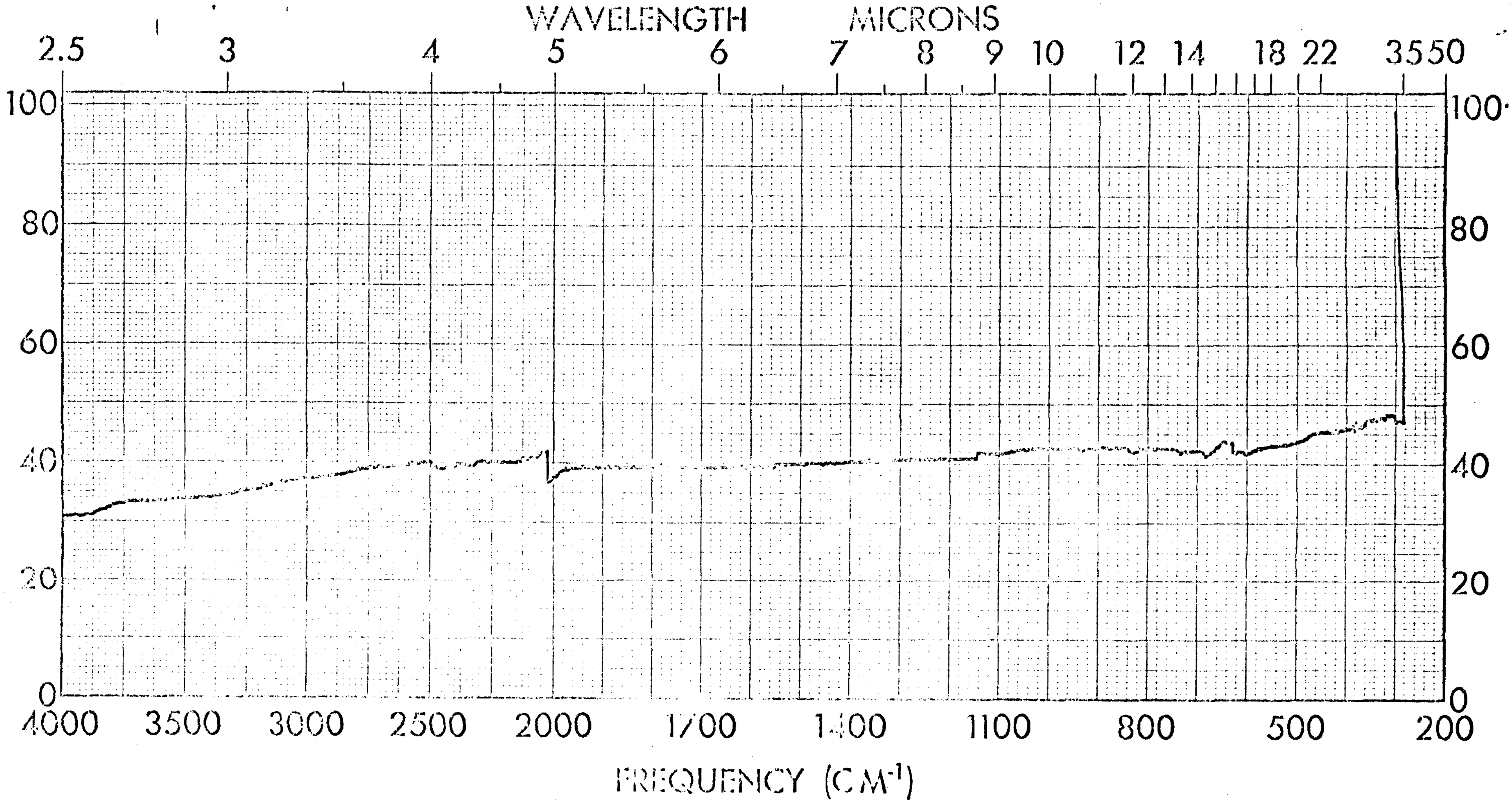
Several reflectance curves have been made of various coatings on substrates of CdS as well as on plain glass and metal. The reflectance curves that have been obtained for the SiO and MgF_2 coatings will not be shown as they are considered non-conclusive and uninformative. Only specular reflectance measurements were made on these coatings and are not considered satisfactory for our purposes. To correlate the reflectance curves that were obtained, aluminum was deposited on surfaces of a known roughness. The reflectance spectra was measured for aluminum deposited on etched molybdenum Figure 3, polished molybdenum Figure 4 and on aluminum oxide wafers Figure 5. A very good indication of Rayleigh scattering can be seen from the slope of the curves of the etched molybdenum and aluminum oxide substrates which were coated by sputtered aluminum. The curves again give only the specular reflectance and do not reveal any correlation to the previous reflectance curves. To determine total reflectance, the samples will be coated with the dielectric, then one half will be coated with about 1000Å of aluminum. The specular reflectances of the two surfaces will be determined and total reflectance of the dielectric found by taking the ratio of the specular reflectance of the dielectric to the specular reflectance of the metal coated dielectric.



Spectrum No. G-1022
Sample:Al on etched Mo
AN 62699

Phase :Solid
Date: 2-23-65
Remarks - Specular Reflectance Spectrum

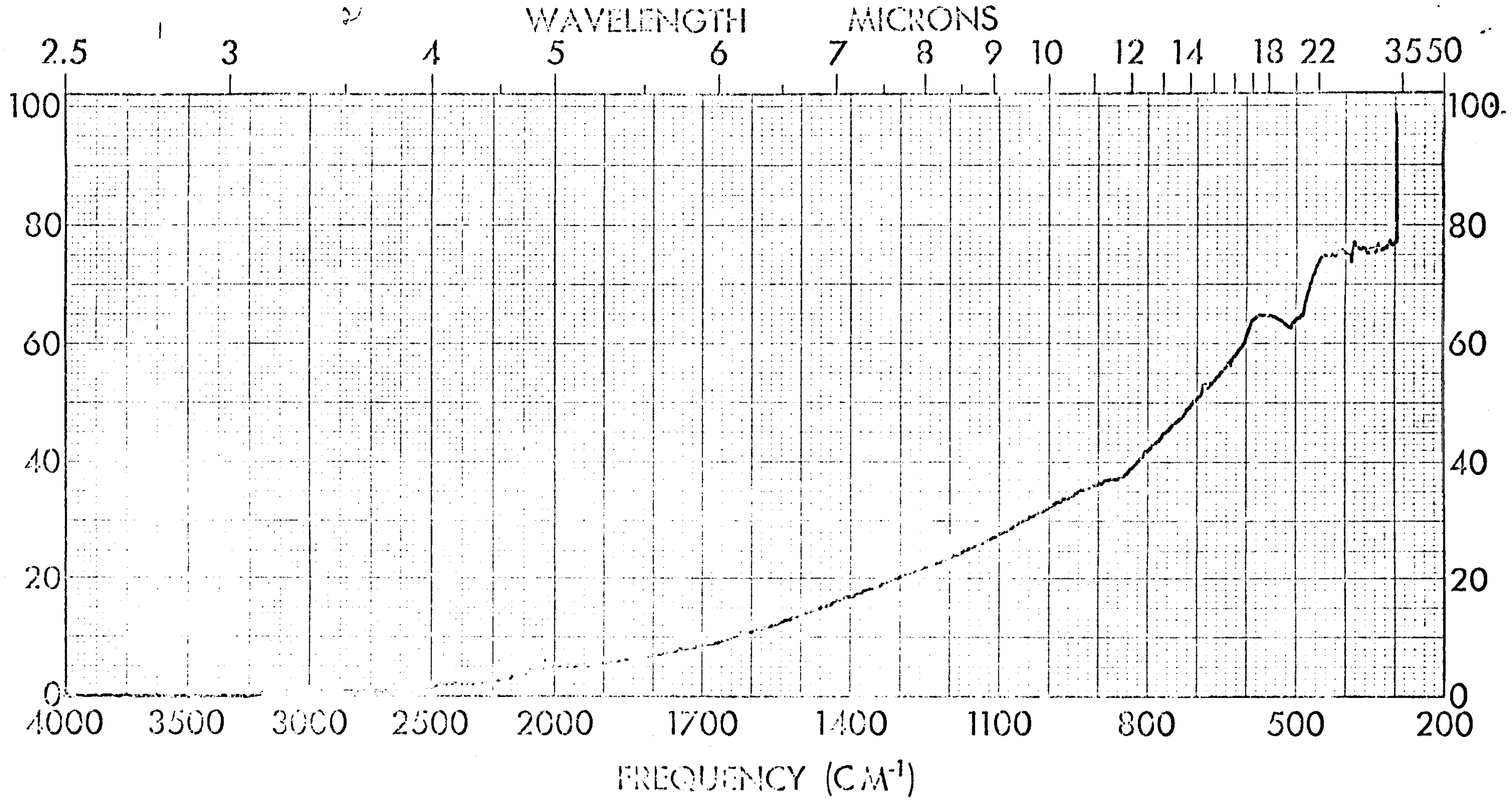
Figure 3 - Reflectance of Aluminum on Etched Molybdenum



Spectrum No. G-1020
Sample:Al on polished Mo
AN 62699

Phase:Solid
Date:2-23-65
Remarks - Specular Reflectance Spectrum

Figure 4 - Reflectance of Aluminum on Polished Molybdenum



Spectrum No. G-1021
Sample: Al on Alumina
AN 62699

Phase: Solid
Date: 2-23-65
Remarks - Specular Reflectance Spectrum

Figure 5 - Reflectance of Aluminum on Aluminum Oxide Wafer

WORK PLANNED FOR NEXT QUARTER

The complete "RF" sputtering unit will be in operation. This will include the coupling system to the probe and the probe termination. In addition a temperature sensing monitor will be installed to check on target and substrate temperatures. A water-cooled substrate holder will also be fabricated for use in the sputtering system.

Reflectance measurements will be made on substrates coated with aluminum and the desired optical coating to determine the total reflectance.

A number of coated cells will be exposed to controlled humidity conditions desiccated air and vacuum to determine whether the coating retards degradation effectively.

FINANCIAL SECTION

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Professional	Manhours
J. C. Schaefer	112
E. R. Hill	78.50
R. W. Olmsted	339
R. J. Humrick	237
A. Hartzler	331
Total	1,097.50

Non Professional	
Total	

Total Contract	$75,003.00
Funds for Cost	$70,853.00
Funds Expended	$19,134.00
Funds Remaining	$51,719.00

Harshaw Chemical Company
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