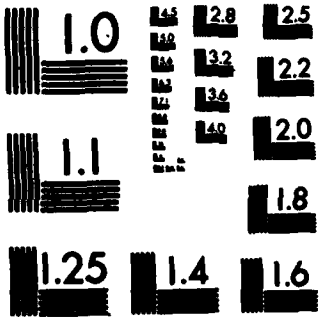
AD-A136 591 PHOTOACOUSTIC ABSORPTION SPECTROSCOPY (PAS) MEASUREMENTS(U) AIR FORCE WEAPONS LAB KIRTLAND AFB NM W WASSON NOV 83 AFWL-TR-83-101 1/1

UNCLASSIFIED F/G 20/6 NL

END
DATE FILMED
12-84
DTIC



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

AFWL-TR-83-101

AFWL-TR-
83-101

PHOTOACOUSTIC ABSORPTION SPECTROSCOPY (PAS) MEASUREMENTS

Wayne Wasson

November 1983

Final Report

Approved for public release; distribution unlimited.

AIR FORCE WEAPONS LABORATORY
Air Force Systems Command
Kirtland Air Force Base, NM 87117

This final report was prepared by the Air Force Weapons Laboratory, Kirtland Air Force Base, New Mexico, under Job Order ILIR8101. Mr. Wayne Wasson (SUE) was the Laboratory Project Officer-in-Charge.

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely Government-related procurement, the United States Government incurs no responsibility or any obligation whatsoever. The fact that the Government may have formulated or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication, or otherwise in any manner construed, as licensing the holder, or any other person or corporation; or as conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report has been authored by an employee of the United States Government. Accordingly, the United States Government retains a nonexclusive, royalty-free license to publish or reproduce the material contained herein, or allow others to do so, for the United States Government purposes.

This report has been reviewed by the Public Affairs Office and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

If your address has changed, if you wish to be removed from our mailing list, or if your organization no longer employs the addressee, please notify AFWL/SUE, Kirtland AFB, NM 87117 to help us maintain a current mailing list.

This technical report has been reviewed and is approved for publication.

WAYNE WASSON
Project Officer

FOR THE COMMANDER

JOHN C. SCOTT
Chief, Engineering Branch

WILLIAM O. MOULDS
Chief, Technical Services Division

DO NOT RETURN COPIES OF THIS REPORT UNLESS CONTRACTUAL OBLIGATIONS OR NOTICE ON A SPECIFIC DOCUMENT REQUIRES THAT IT BE RETURNED.

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE

AD-A136 591

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION: Unclassified	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT: Approved for public release; distribution unlimited.
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER(S): AFWL-TR-83-101	5. MONITORING ORGANIZATION REPORT NUMBER(S)
6a. NAME OF PERFORMING ORGANIZATION: Air Force Weapons Laboratory; 6b. OFFICE SYMBOL (If applicable): SUE	7a. NAME OF MONITORING ORGANIZATION
6c. ADDRESS (City, State and ZIP Code): Kirtland Air Force Base, NM 87117	7b. ADDRESS (City, State and ZIP Code)
8a. NAME OF FUNDING/SPONSORING ORGANIZATION; 8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
8c. ADDRESS (City, State and ZIP Code)	10. SOURCE OF FUNDING NOS.

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT NO.
61101F	ILIR	81	01

11. TITLE (Include Security Classification): PHOTOACOUSTIC ABSORPTION SPECTROSCOPY (PAS) MEASUREMENTS

12. PERSONAL AUTHOR(S): Wayne Wasson

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Yr., Mo., Day)	15. PAGE COUNT
Final	FROM 81 Oct 1 TO 83 Oct 1	1983, November	16

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB. GR.	
20	05		Photoacoustic Absorption; Thin-Film Absorption; Mirror Absorption; Window Absorption
20	06		

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

A functioning optical component absorption measurement facility has been assembled to (1) develop technical competency in evaluating the effectiveness of the technique, and (2) implement the technique as a routine measurement procedure for optical component evaluation. Both objectives have been partially successful. The conclusions of this research are that (1) measurement accuracy is highly dependent on component selection and assembly; (2) many variables must be considered when using the technique; and (3) the technique has the capability to measure one of the many variables involved in photoacoustic absorption. The research and measurements are being transitioned into mission and support project work.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION
UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS ☐	

22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE NUMBER (Include Area Code)	22c. OFFICE SYMBOL
Wayne Wasson	(505) 844-1297	SUE

DD FORM 1473, 83 APR — EDITION OF 1 JAN 73 IS OBSOLETE.

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE

CONTENTS

I. INTRODUCTION

BACKGROUND

Photoacoustic Absorption Spectroscopy (PAS) has an interesting history as a research technique; however, it seems to have eluded general use for laboratory measurement. Within the last year, many reports have been published involving the use of PAS for material identification by infrared absorption utilizing Fourier Transform Spectroscopy. The technique is particularly useful for analyzing nonoptical materials such as bituminous coal, gels, creams, powders, emulsions, and pharmaceutical products.

The utilization of PAS for identification requires only the production of spectral absorptions. From the wavelength of the absorptions, a material identification can be made. Many commercial suppliers produce PAS accessories for infrared spectrometers.

Although the variables involved in PAS for optical materials are the same as for material identification, the goal is completely different. The most useful measurement for optical materials is the amount of absorption. However, thin-film optical properties such as thermal conductivity may be measurable by PAS. This parameter would be useful for thin-film design and performance evaluation, even if known only comparatively. PAS offers the possibility of measuring several variables, but the measurement of absorption is emphasized. Other variables may be more important than absorption, since absorption can be measured by calorimetry, a technique traceable to primary standards. The problem of calibration and traceability to primary standards for PAS is difficult because more than achieving thermal equilibrium is involved. However, this problem allows determination of more variables than does calorimetry.

II. APPROACH AND OBJECTIVES

Three methods were initially envisioned to evaluate PAS. The first used a modulated laser and a closed chamber containing a window, microphone, and optical component. The modulated pressure wave detected by the microphone was coupled to a preamplifier and lock-in amplifier. The second method involved direct transducer attachment to an optical component being irradiated by a modulated or pulsed laser source. The third method would introduce a PAS accessory into a Fourier Transform Infrared Spectrometer. The latter two methods could not be used because of time restraints and the unavailability of instrumentation.

The purpose of this research was to evaluate the effectiveness of using commercially available, relatively low-cost equipment to design and assemble a PAS enclosure for determining detectability, noise levels, and measurement accuracy tolerances. After the design and assembly was completed, the flexibility of the measurement ability was determined, and an evaluation made as to applicability for routine measurement capabilities.

III. TEST EQUIPMENT AND CONDITIONS

A tunable, radio frequency waveguide carbon dioxide laser was used as the source. A mechanical chopper of variable frequency and duty cycle was used as the modulator. Two enclosures were designed to hold 3.7-cm optical components. This size was chosen because of the availability of components for mirrors and windows.

The first enclosure was fabricated from bar stock, with the entrance window and microphone on the front face, and the sample mounted on the back face (Fig. 1). The second enclosure was fabricated from bar stock, with a square tubing extension on the front face, and the entrance window on the end of the square tubing (Fig. 2). The microphone was mounted at a right angle to the window on the square tubing. The sample was mounted on the back side of the cell, as in the first. A flange was fabricated to seal the back face of the cell, making the entire configuration airtight.

The microphones used were 1.27-cm electrets with integral preamplifiers. The signal from preamplifiers was the input for a lock-in amplifier referenced to the mechanical chopper frequency. The laser, chopper, and cell were mounted on an aluminum triangular rail on a cast iron table (Fig. 3). The laser was capable of 1.5 W output on the p20 line, as determined by a power meter and spectral analyzer. Since the chopper was 50-percent duty cycle, the average power on the cell window was 0.75 W. Both cells had zinc selenide entrance windows with an anti-reflective coating on both sides, giving 94-percent transmission at a wavelength of 10.6 μm.

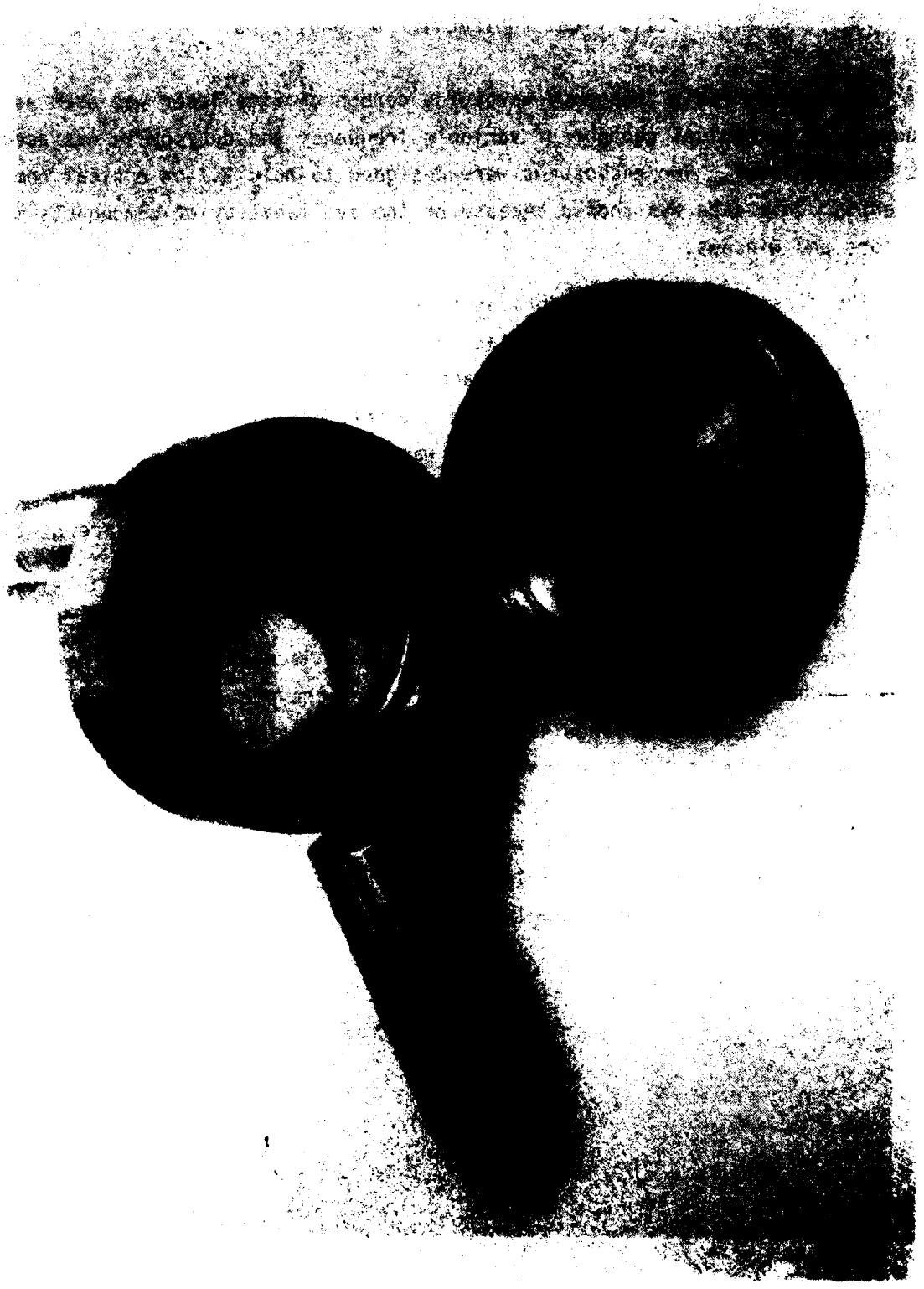


Figure 1. PAS cell No. 1.

Figure 2. PAS cell No. 2.

Figure 3. System configuration.

IV. SYSTEM OPERATION

The system requires a stable source of irradiation, meaning that approximately one-half hour of warm-up time is required for the laser to stabilize in power and mode. The electronics are turned on at the same time as the laser. The sample is then inserted into the flange of the PAS cell, and the cover flange installed. The chop rate of the laser is arbitrary, but consideration must be given to signal strength, microphone preamplifier, and lock-in frequency response for the overall measurement conditions. Also, some choppers are not frequency stable at their lower frequencies. Typical operating conditions are 100-Hz chop rate, 1.5 W laser power, and a full-scale setting of 1 mV on the lock-in amplifier.

The gain on the lock-in amplifier is then adjusted to an approximately full-scale reading by adjusting the gain control. This allows two orders of magnitude gain in sensitivity on the lock-in amplifier used for the measurements. The lock-in amplifier has an LED readout and an analog meter. The initial consideration was to maximize the signal with a strong absorber, and a glass wafer with an absorbent lacquer was used. The wafer has a small amount of specular and diffuse reflectance, however, and is therefore not optimum. The best total absorber would probably be a black gold coating on any type of substrate, because this type of coating has an absorption of greater than 99 percent (Refs. 1 and 2). In the measurement of absorption, one of the variables that must be considered is accuracy, and an uncertainty of 1 percent is quite reasonable.

Next, consideration was given to a sample of reasonably low absorption, a reference molybdenum mirror determined calorimetrically to have an absorption of 0.0159. Measurement of this sample gave two types of information: An indication of linearity, and a measure of cell noise from window and cell absorption. The reference mirror showed an absorption of 0.0182, a value dependent on (1) the laser power variation, which is typically ±5 percent over the measurement time, and (2) the uncertainty in the value of the high-absorption glass. Additionally, this measurement would indicate that cell noise is not a limiting feature at this level.

The last consideration was given to cell noise, which is a function of entrance window absorption, cell body absorption caused by optical misalignment, preamplifier noise, and false signals introduced by transmission of vibration from the mechanical chopper and laser cooling fan. The noise level appears to be approximately 2.5 orders of magnitude below total absorption. To accomplish this, the PAS cell was mounted loosely on the triangular rail with foam rubber pads.

For comparison, a polished molybdenum mirror was measured and showed an absorption of 0.0147. A gold-coated molybdenum mirror showed an absorption of 0.0133; electroless nickel-coated molybdenum mirrors showed 0.0306 to 0.0588; a chemically vapor deposited (CVD) tungsten molybdenum mirror showed 0.0980; and a zinc sulfide-thorium fluoride coating 100 nm thick on molybdenum showed 0.0118. When a potassium bromide window was used as a sample, no indication above the noise level was measured.

V. DEVELOPMENT OF PAS FOR ROUTINE MEASUREMENT

The technique of PAS has been evaluated and offers promise as a useful technique for optical measurement. The development of the technique will be continued through mission projects.

One primary consideration is that the measurement can be made independent of wavelength. Any wavelength source with proper entrance windows can be utilized. With the present apparatus, the tolerance on measurements is quite large and unpredictable due to fluctuations in laser power. Although the power is monitored with a pellicle beamsplitter and a mercury-cadmium-telluride detector, these are not sensitive indicators of laser power in the present configuration. However, improvements in configuration readout can be made to improve this monitoring capability. Vibration isolation of the PAS cell can also be improved, and improvements can be made in microphone and preamplifier noise levels.

Although the microphone and preamplifier were purchased specifically for this project, the laser and chopper were transferred from other devices and are not optimum. Both amplitude and mode stability are needed in a laser source, and the chopper should be well balanced. The problem of vibration transmission from chopper to PAS cell has not been solved; however, mounting the PAS cell on a separate rail with vibration isolation should reduce vibration to a negligible level. Cell design offers many possibilities for signal-to-noise improvements; but of the two designs fabricated and tested, no significant differences in performance were observed.

Hardware problems in PAS are the easiest to solve. Interpretation of the interaction processes are much more difficult to understand. Ideally, what is needed is a windowless cell with a perfectly stable source and a 100-percent absorber (or one with the percentage known precisely). The lack of such a cell and absorber contributes to many interactions inside the cell.

VI. CONCLUSION AND RECOMMENDATIONS

Subsequent work will focus on gathering a data base for project officers use. Data will be collected on substrate absorption of windows and mirrors. Then a series of measurements will be performed on thin-film materials in a sequence of increasing thickness to determine when substrate-thin-film interaction is no longer measurable, and to check repeatability. This function, although only comparative at this point, could be very useful as correlative data for optical performance evaluation. Data from this method will be used with calorimetric, surface analytical, ellipsometric, laser damage, and theoretical predictions to follow optical performance. Furthermore, plans are to measure thin-film thermal conductivities comparatively, for different thicknesses of material, to see whether conductivity is a constant for a given material when applied by high vacuum evaporation techniques (such as resistive, electron-beam, and laser heating); and as a function of the commercial supplier of the coating materials. The thrust of this research will be toward thin-film optical coating performance evaluation, since those mission projects will be supporting the effort.

Improvements in the sophistication of the hardware and cell design are necessary for the ultimate refinement of the technique of PAS. Improved data reduction facilities will probably also be required; however, a balance is necessary in sophistication, cost, and ease of operation so that the technique remains easily and reliably useful. One of the most attractive features of PAS is that it is a simple and quick technique. The theoretical development of PAS has been amply pursued (Refs. 3-7). Therefore, we must now try to deal with experiments and extract meaningful data to correlate with other measurements to provide a full interpretation of the interactions within PAS cells.

REFERENCES

1. Doyle, W. M., McIntosh, B. C. and Geist, Jon, Proc. SPIE 62, 166, 1975.

2. Blevin, W. R. and Brown, W. J., J. Sci. Instr. 42, 388, 1965.

3. Rosencwaig, A., Advances in Electronics and Electron Physics, Vol. 46, (L. Marton, ed.), pp. 207-311, Academic Press, New York, 1978.

4. Rosencwaig, A. and Gersho, A., "Theory of the Photoacoustic Effect with Solids," J. Appl. Phys. 47 (1), 64, 1976.

5. Bennett, H. and Forman, R., "Frequency Dependence of Photoacoustic Spectroscopy: Surface and Bulk Absorption Coefficients," J. Appl. Phys. 48 (4), 1432, 1977.

6. Hordvik, A. and Schlossberg, H., "Photoacoustic Technique for Determining Optical Absorption Coefficients in Solids," Appl. Opt. 16 (1), 101, 1977.

7. Dewey, C., "Opto-acoustic Spectroscopy," Opt. Eng. 13 (6), 483, 1974.