IV. OPTICAL AND INFRARED SPECTROSCOPY

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RESEARCH OBJECTIVES AND SUMMARY OF RESEARCH

The activities of this group have been concentrated on the study of the optical properties of solids, and our present projects fall conveniently into six classes. A number of experiments are also being planned in cooperation with members of the Materials Center for Science and Engineering, the Department of Electrical Engineering, the Laser Group, and the Department of Physical Chemistry of the Massachusetts Institute of Technology.

1. Techniques of Far Infrared Spectroscopy

The techniques of grating spectroscopy in the far infrared[1] have now been extended to interferometry. The Michelson interferometer[2] has been modified to suit the needs of our group[3] and has been used successfully in the range 25-1000 microns. Liquid-helium and liquid-nitrogen cryostats, together with a sample holder for studying the reflectance and transmittance of solids up to 700°K, have almost been completed.

Modifications of the computer program for more flexible use are in progress, and the present program[4] has been made available to a number of institutions in this country and abroad.

Both the spectrometer and interferometer are being used continuously for the study of the optical properties of solids in the infrared, together with the Perkin-Elmer Model 521 spectrophotometer (in the Spectroscopy Laboratory, M. I. T.), so that the range 2.5-1000 microns can now be covered. The detector optics in the grating instrument has been redesigned to accomodate an off-axis ellipsoidal mirror[5] that will increase the use of the slit height. Progress is also being made in the use of metal meshes[6] as reflection filters to obtain higher spectral purity in the 100-1000 micron region.

The study of low-temperature detectors for the 50-100 micron region has been curtailed somewhat, owing to lack of manpower for this particular problem which is more of the nature of a technical one, at present. The advent of the commerical Texas Instrument Company doped germanium bolometer[7] and the Mullard indium-antimonide detector[8] has also been a contributing factor in this decision.

Considerable interest has been created by our measurements of detectivity, D^*, and response of Golay detectors[9]; we plan to continue these measurements as the need arises.

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(Mr. H. D. Wactlar is now at the Computation Center, University of Maryland, College Park, Maryland)

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2. Dielectric Dispersion in Ferroelectric Materials

The lowest mode of vibration in $SrTiO_3$ has been shown by a number of authors[1-4] to exhibit a temperature dependence above its Curie temperature which has been predicted by Cochran[5] from the temperature dependence of the dielectric constant. We have studied a number of titanates[6] and zirconates,[7] and have unambiguously assigned the presumed normal modes. We propose to do a temperature-dependent study of $CaTiO_3$ and also to repeat $BaTiO_3$, as there are conflicting reports on this material.[3,8] The study of $PbZrO_3$ and $PbHfO_3$ as a function of temperature above and below their Curie temperatures are also of considerable importance, as both of these materials are antiferroelectric.[9,10] Preliminary work had been started,[4] and we propose to continue this research with further measurements, using both the grating instrument and the interferometer in the longwave region.

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3. Magnetic Resonances in Solids

Preliminary measurements of a few of the potassium transition metal fluorides having the perovskite structure have been started this year with a limited amount of success.[1] This may have been due to the sample preparation, and new flame-fusion-grown materials have now been obtained.

Below the Curie point, there is a strong effective field leading to a zero-field splitting of the resonance line. In the simplest situation, at absolute zero, the effective field apart from the applied magnetic field[2] is given by

$$H_{eff} = [H_A(2\ H_E + H_A)]^{1/2},$$

where H_A is the effective anisotropy field of one sublattice, and H_E is the exchange field.

The resonance frequency for uniaxial symmetry about the sublattice polarization axis is given by

$$\omega = g\{[H_A(2\ H_E + H_A)]^{1/2} \pm H\}$$

$$= g\ [(2K/\chi_\perp)^{1/2} \pm H],$$

where H is the applied field, K is the anisotropy constant, and $\chi_\perp$ is the static susceptibility perpendicular to the easy axis.[3]

The degree of antiferromagnetism changes with temperature up to its Néel temperature, thereby causing the resonant frequency to be strongly temperature-dependent.[4,5] The externally applied field in the preferred direction leads to two resonant conditions.[6]

A liquid-helium cryostat (designed and constructed in collaboration with Dr. S. Zwerdling of the Material Center for Science and Engineering, M.I.T., and the Janis Research Company, Waltham, Massachusetts) will be used for the temperature-dependent studies. The sample is surrounded by a helium exchange gas and the temperature of the sample can be held from 1.5°K to 77°K by careful balancing of the pressure of the exchange gas and the current through a small heater.

A second cryostat is about to be constructed to house a superconducting magnet with a field up to 45 kilogauss so that the field dependence of the resonance can be investigated. The use of a superconducting magnet necessitates employment of light pipe techniques, and possibly a low-temperature detector, but the design has been made sufficiently flexible to cover a number of variations.

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4. Electronic Transitions

Rare-earth ions in various host lattices can have their ground levels split at far infrared frequencies by the crystal field. It has been shown by Hadni[1,2] that there are other splitting possibilities such as coupling between two nearest-neighbor ions. Electronic Raman lines have also been observed in pure praseodynium chloride.[3] Magnetic dipole transitions in the ground state at liquid-helium temperatures are allowed in a number of materials, but these are probably very sharp and weak and, at the present time, beyond the resolution of our instruments; this work will have to await the advent of powerful far infrared lasers in the 100-1000 micron region.

We have looked at a number of rare-earth doped materials in the near infrared and are about to study them when the host crystal becomes transparent again on the low-frequency side of the lattice absorption. A temperature-dependent study of any resonance observed with the helium cryostat described above should provide useful information that cannot be obtained from fluorescent studies.

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5. Lattice Vibrations

The frequencies of the presumed normal modes of a number of perovskites have been completed[1-4] and the work has now been extended to mixed crystals of $KMgF_3$-$KNiF_3$ in various ratios. Preliminary reflectance measurements have been made, and the results are to be repeated on the interferometer before the final Kramers-Kronig analyses are made.

Completion of the work on several alkali-earth platinum halide complexes has led to a successful interpretation of the frequencies of vibration.[5] Other noble-metal halide complexes are now under investigation at room temperature, and are to be undertaken at liquid-nitrogen temperatures.

The reflectivity of cadium selenide in the region 2-100 microns is to be repeated at longer wavelengths and as a function of temperature to observe the influence of the free carriers on the reflectivity.[6] The work will be done on different thickness in the two-phonon and three-phonon regions and other related II-VI and III-V materials.

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6. Raman Spectra of Solids

The Spectroscopy Laboratory, M. I. T., has now acquired a Cary 81 Raman Spectrophotometer. Consequently, an obvious research project appears to be the Raman study of the solids that are now under investigation in the infrared. Measurements have been made on $SrTiO_3$, $BaTiO_3$, and $KTaO_3$, all three materials giving good second-order Raman spectra. $KMgF_3$ should not give a Raman spectrum and none was found. The $CaTiO_3$ and $PbTiO_3$ ceramics gave no Raman spectra because of excessive Rayleigh scattering.

A heated sample holder has been constructed and preliminary measurements on $BaTiO_3$ as a function of temperature have shown bands disappearing as the sample is raised above the Curie temperature. The Raman spectra may also be fruitful in the interpretation of the ferroelectric nature of these materials.

There is some conflict between our results on $SrTiO_3$[1] and $BaTiO_3$[2,3] and those published by others. Further investigation of these interesting materials (including $CaTiO_3$) is being undertaken. The construction of a low-temperature sample holder is also contemplated.

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A. TECHNIQUES OF FAR INFRARED INTERFEROMETRY

1. Introduction

Several authors have demonstrated that improved performance can be obtained in the far infrared by using interferometric techniques, and the theories of the different methods have been adequately presented.[1-5]

Briefly, if two equal beams of monochromatic radiation of wavenumber ν are superimposed, with a path difference Δ, then the aggregate illumination E will be

$$E(\Delta) = E_1(1 + \cos 2\ \nu\Delta), \tag{1}$$

where E_1 is proportional to the illumination of either one of the equal beams. For heterochromatic radiation this becomes

$$E(\Delta) = \int_{-\infty}^{\infty} E_1(\nu)\,[1 + \cos 2\pi\nu\Delta]\,d\nu \tag{2}$$

which can be written as

$$E(\Delta) = \int_{-\infty}^{\infty} E_1(\nu)\,d\nu + \int_{-\infty}^{\infty} E_1(\nu)\cos 2\pi\nu\Delta d\nu. \tag{3}$$

The value of the illumination when $\Delta = 0$ is

$$E(0) = 2\int_{-\infty}^{\infty} E_1(\nu)\,d\nu \tag{4}$$

and at arbitrary values of path difference

$$E(\Delta) = \frac{1}{2}E(0) + \int_{-\infty}^{\infty} E_1(\nu)\cos 2\pi\nu\Delta d\nu. \tag{5}$$

If we now consider a new function $F(\Delta)$ defined by the difference between $E(\Delta)$ and $\frac{1}{2}E(0)$ and call it the interferogram function, we have

$$F(\Delta) = E(\Delta) - \frac{1}{2}E(0) = \int_{-\infty}^{\infty} E_1(\nu)\cos 2\pi\nu\Delta d\nu. \tag{6}$$

The function $E(\Delta)$ is the one that can be recorded by the detector in the interferometer. $E_1(\nu)$ has to be determined from a Fourier transform,

$$E_1(\nu) = \int_{-\infty}^{\infty} F(\Delta)\cos 2\pi\nu\Delta d\Delta. \tag{7}$$

In order to perform this computation it is necessary to choose discrete values of sampling interval, Δ_s.

The equation transforming the interferogram function $F(\Delta)$ into its spectral function $E_1(\nu)$ can now be written as a sum over the actual data points.

$$E_1(\nu) = \Delta_s\left[F(0) + 2\sum_{n=1}^{n=\Delta_{max}/\Delta_s} F(n\Delta_s)\cos 2\pi n\Delta_s\nu\right],$$

where Δ_s is the sampling interval, $F(0) = \frac{1}{2}E(0)$, and n is the total number of sampling points. By limiting the sum to a finite number of data points, it is truncated and an error is introduced which yields spurious oscillations in the spectrum estimate from the interferogram; these can be suppressed by smoothing the interferogram through use of an apodization function multiplier. Triangular apodization is employed here (multiplication of $F(\Delta)$ by $1 - \Delta/\Delta_{max}$) and this generates a scanning function similar to an ideal grating instrumental line shape.[6]

$$\frac{\sin^2 (\nu-\nu')\Delta_{max}}{((\nu-\nu')\Delta_{max})^2}.$$

This apodization function reduces the effective resolution by $1/\sqrt{2}$ but yields a more accurate spectrum. Various types of apodization functions and their effect have been well described by Llewellyn-Jones.[5]

Communication and sampling theory given by Goldman[7] tells that the optimum distance between samples of data of $E(\Delta)$ is $\Delta_s \leq \frac{1}{2\nu_{max}}$, where ν_{max} is the highest wavenumber radiation incident on the detector to obtain all of the information in the spectrum from $0 < \nu < \nu_{max}$. (For example, $\nu_{max} \sim 400\ cm^{-1}$ $\Delta_s \leq 12.5$ microns. With a choice of 4 microns or 8 microns we could conceivably obtain all information from 0-12.5 cm^{-1} or 0-625 cm^{-1}, respectively. These options are incorporated in our computer program.)

Richards has pointed out that the computed spectrum at frequency ν will contain false energy of frequencies $2n\nu_{max} - \nu$ and $2(n-1)\nu_{max} + \nu$, where n = 1, 2, 3 . . . . In order to obtain an unambiguous spectrum, it is necessary to cut off the spectrum to zero at ν_{max} and keep it zero at all higher frequencies. The time constant τ of the system should be chosen so that its path difference equivalent $\Delta_\tau = \frac{d\Delta}{dt}\tau_o$ is approximately one quarter of the sampling interval, Δ_s (i.e. $\Delta_s \approx 4\Delta_\tau$). Very high frequencies will be suppressed by the time constant and this factor contributes in the filtering process of removing false energy. So the filtering is not so difficult in practice as the removal of unwanted orders from a grating (for example, crystal choppers, rest-strahlen filters, etc.), since ν_{max} can be chosen in a reasonably arbitrary manner. A filter of SrF_2 + LiF + black polyethylene[8] cuts off ~25 μ (400 cm^{-1}), whereas the ν_{max} for $\Delta_s = 8$ μ is 625 cm^{-1}.

To obtain the resolution capability, it is convenient to compute the spectrum in steps of half the resolution width. The smallest spectral interval that can be resolved is $\delta\nu = \left(\frac{1}{\Delta_{max}}\right) cm^{-1}$, where Δ_{max} is the maximum path difference to which the interferogram is measured up to the fundamental limit as given by Jacquinot.[9]

In order to double the resolution in an interferometer, it is necessary to double Δ_{max} and to take twice as long to retain the same S/N. In a grating instrument the

entrance and exit slits must be halved, and to retain a comparable spectrum the scanning time must be increased by a factor of 16 as the S/N is decreased fourfold.

Research in the far infrared is limited by detector noise and not by background noise; interference spectroscopy has the major advantage (known as the Fellgett advantage[10]) that the whole spectral region of interest is incident on the detector at any one time, whereas in a monochromator only a single resolution width passes through the exit slit.

The disadvantage of needing a computer to process the data (and its built-in delay — usually one day for a 1-minute run, two days for a 5-minute run) is outweighed somewhat by the ability to compute directly and plot ratios of spectra, attenuation constants, strengths of absorption lines, and to perform Kramers-Kronig analyses of reflectance data to obtain dielectric constant data. All computation work is done on the IBM 7094 computer at the M. I. T. Computation Center.

2. The Michelson Interferometer

The FS-520 vacuum interferometer[11] manufactured by Research and Industrial Instrument Company, London, England, has been modified slightly to suit the needs of our research. Figure IV-1 shows the instrument with a helium cryostat installed in the sample chamber. The basic layout and block diagram are shown in Figs. IV-2 and IV-3.

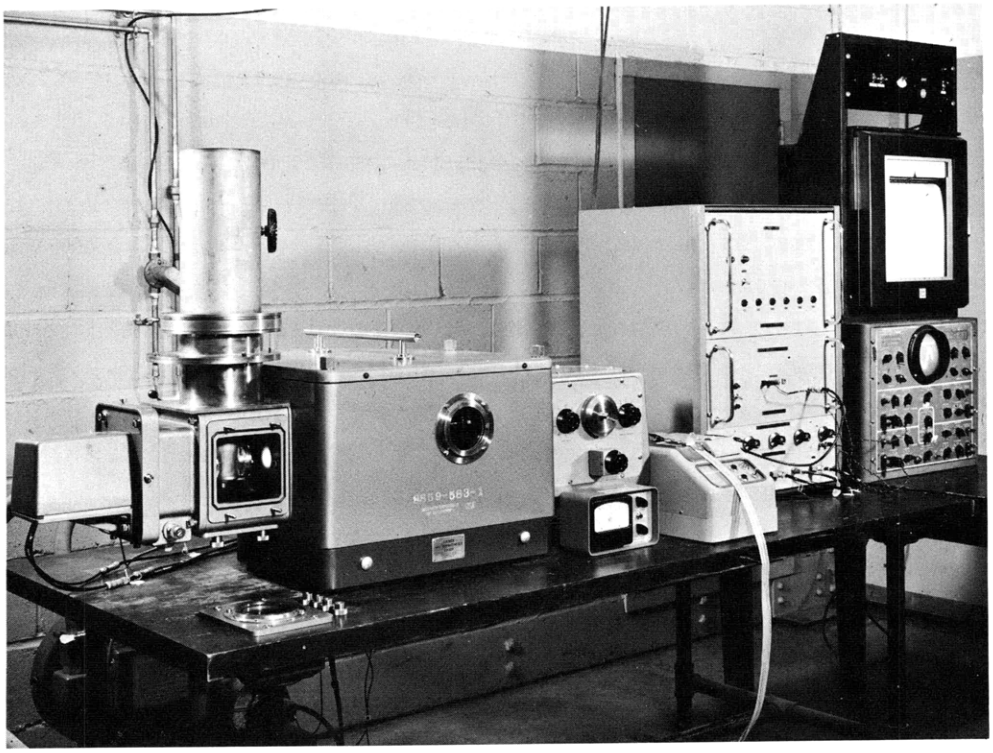

Fig. IV-1. Far Infrared Interferometer.

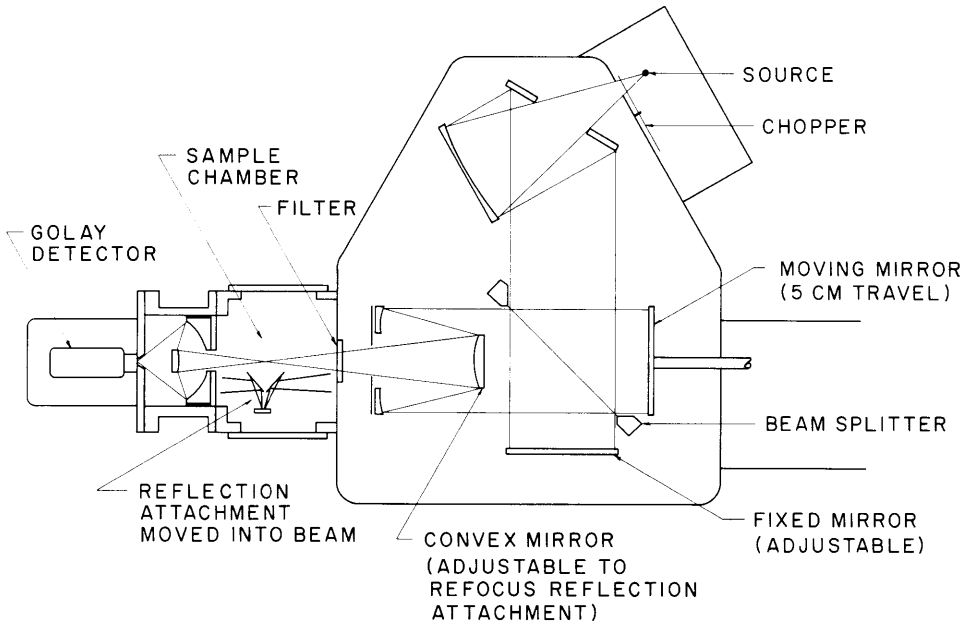

Fig. IV-2. Optical layout.

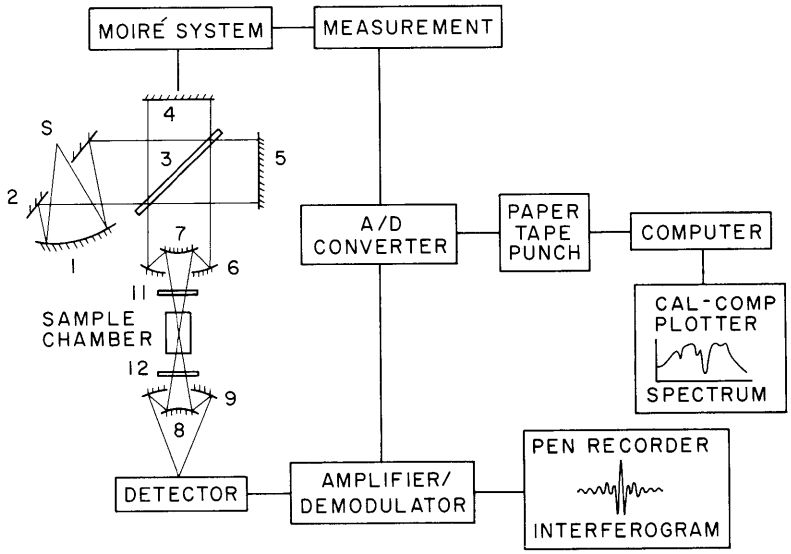

Fig. IV-3. Block diagram of the system.

Two beam splitters are now in use so that the regions 50-400 cm^{-1} (0.00025-inch mylar beam splitter) and 20-150 cm^{-1} (0.0010-inch mylar beam splitter) can be covered. Examples are shown in Figs. IV-4 and IV-5. BeO + ZnO + black polyethylene are employed in Fig. IV-4, and wedged crystal quartz, ~0.5 mm thick + black polyethylene in Fig. IV-5. The second hoop can be eliminated by using an additional filter of BeO + ZnO.[8] No trouble in going to longer wavelengths is anticipated when using thicker beam splitters, except for loss if signal to noise; here, it may be a decided advantage to use cooled detectors, for example, the indium antimonide detector used by Llewellyn-Jones,[5] the germanium bolometer described by Richards[4] or one of our own carbon or germanium bolometers.[12]

A reflection attachment for heating samples up to 700°K is shown in Fig. IV-6 and this can easily be introduced into the instrument (see Fig. IV-2).

A nitrogen cryostat for cold reflectance and transmittance measurements of a number of samples is shown in Fig. IV-7. All of these attachments can also be employed in the vacuum grating instrument.[12]

The interferogram for the reflectance of NaCl is shown in Fig. IV-8 and the computed reflectivity is shown in Fig. IV-9. This can be compared with the results obtained by Yamada, Mitsuishi, and Yoshinaga[13] and by Geick.[14] The frequency dependence is in good agreement, but the intensity discrepancy between the two reflectance curves probably arises from the error in the different gain settings of the background interferogram and sample interferogram. The gain of the Golay amplifier has now been accurately calibrated and this parameter is incorporated in the computer program so that accurate reflectivity measurements can now be made. An example of an interferogram (Fig. IV-10) computed first on one side and then on the other is shown in Fig. IV-11 for the reflectance of CdSe. The agreement between the two individual curves is quite good but considerable trouble was taken in setting up the interferometer to give an output point at zero path and also to get reasonably symmetric output data punchings of the interferogram. In order to reduce this task, the instrument has been changed slightly so that interferograms can be made from $-2 < \Delta < 8$ cm, with the result that double-sided interferograms can be computed with up to 0.5 cm^{-1} resolution. This should improve the accuracy of the reflectance measurements,[14] as any slight asymmetry in the interferograms will be "averaged out" and it will no longer be necessary to accurately obtain a point at zero path.[15] For high resolution studies, however, single-sided interferograms will have to be used.

We would like to thank Dr. S. S. Mitra of the Research Institute, Illinois Institute of Technology, for the CdSe sample.

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(Mr. H. D. Wactlar is now at the Computation Center, University of Maryland, College Park, Maryland)

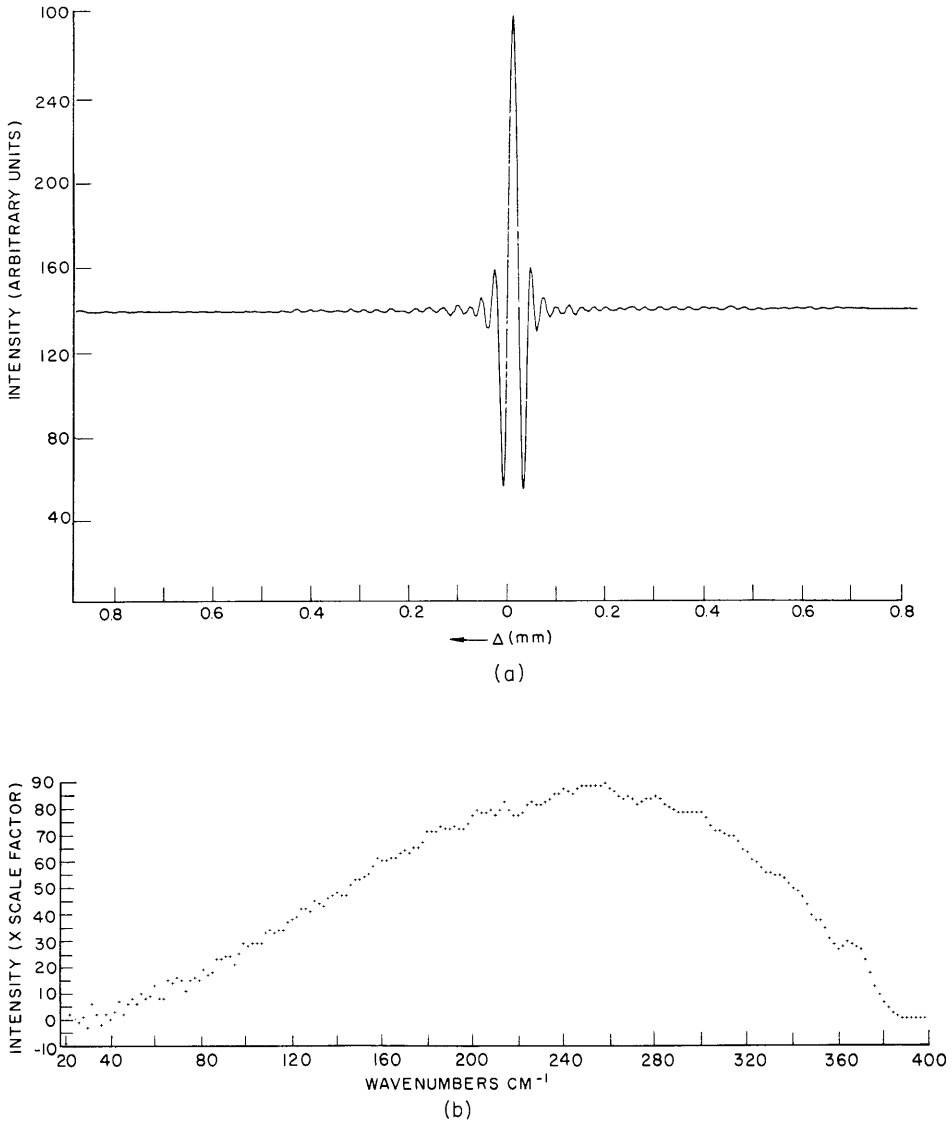
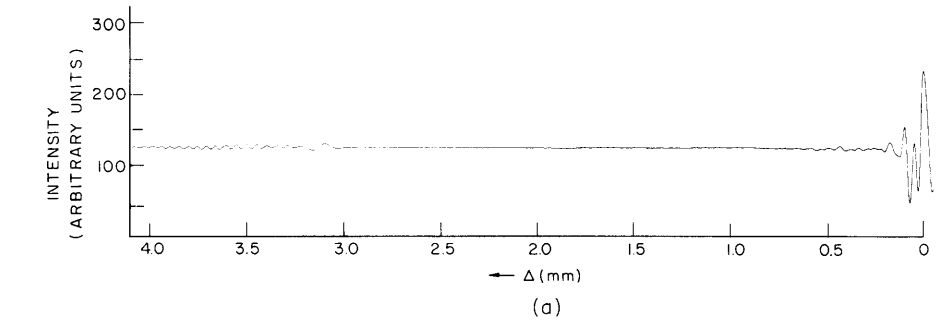


Fig. IV-4. (a) Background interferogram (0.25-mil mylar beam splitter; BeO + ZnO + black polyethylene filters).

(b) Background spectrum (20-400 cm^{-1}). The small ripples are due to interference in the filters; the band at ~360 cm^{-1} is due to the mylar film.



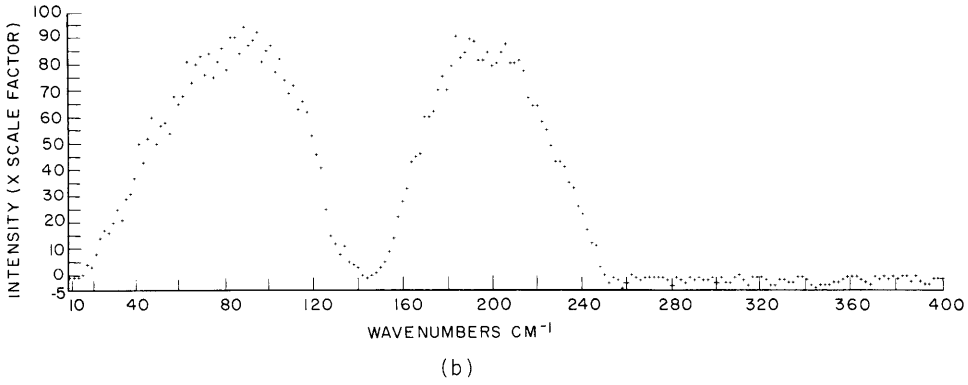


Fig. IV-5. (a) Background interferogram (1.0-mil mylar beam splitter; BeO + ZnO + black polyethylene + 0.75 mm crystal quartz (not wedged).

(b) Background spectrum (10-400 cm^{-1}). Note the two hoops caused by the beam splitter, the efficient cutoff of the crystal-quartz filter at 250 cm^{-1}, and the channel spectra superimposed on the background because of interference in the quartz plate. The interference in the quartz can easily be removed by wedging the quartz; this is observed in the interferogram (a) when the ripples beyond 3-mm path difference completely disappear.

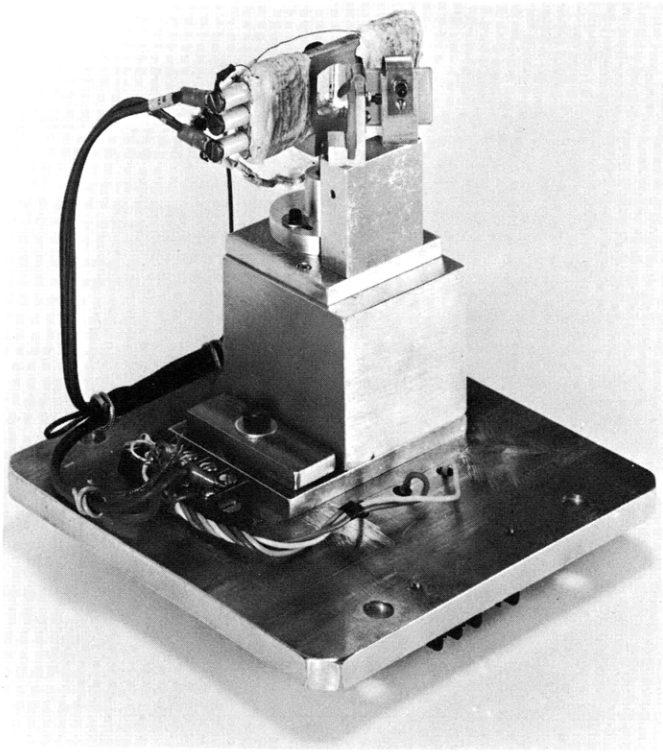


Fig. IV-6. Reflectance attachment for heated samples.

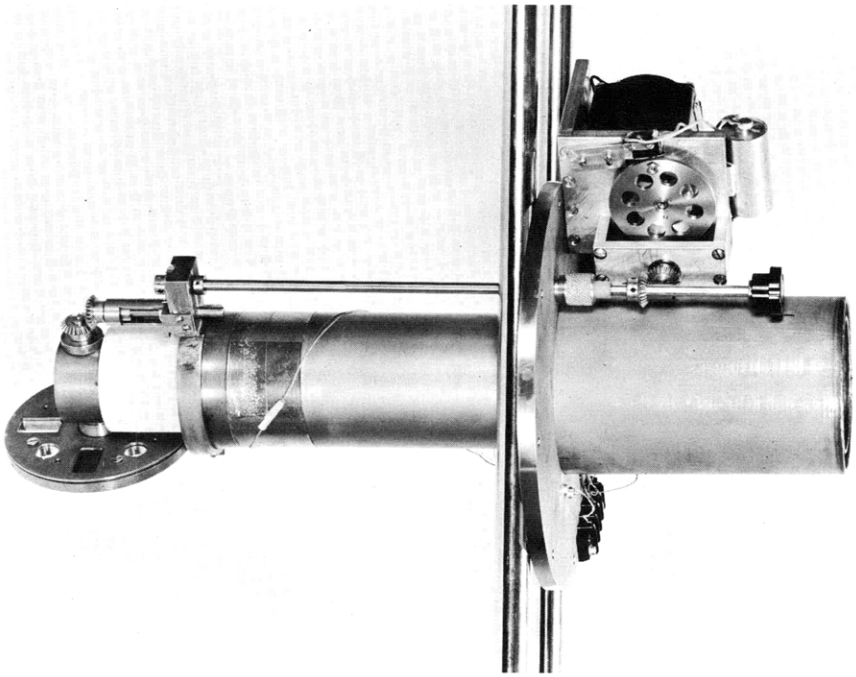


Fig. IV-7. Liquid-nitrogen cryostat for reflectance and transmittance samples.

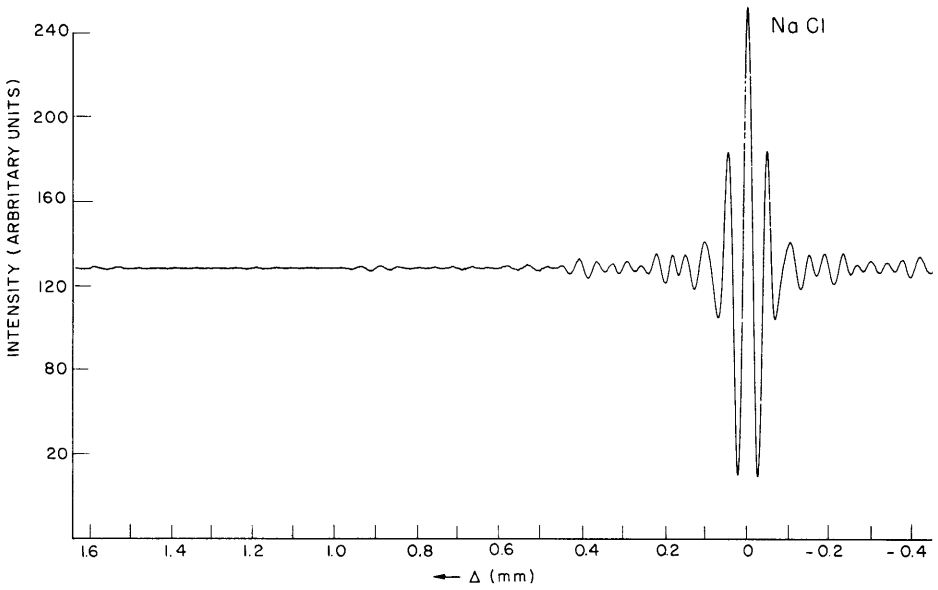


Fig. IV-8. NaCl reflectance interferogram.

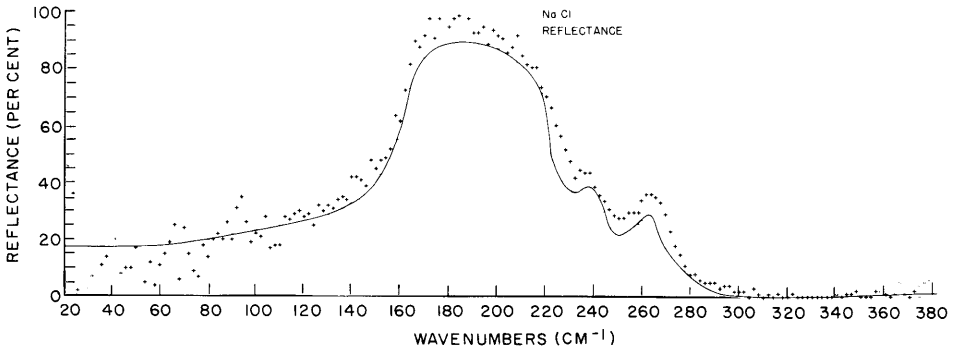


Fig. IV-9. NaCl reflectance spectrum. (The solid curve shows the results obtained by Yamada, Mitsuishi, and Yoshinaga and by Geick.)

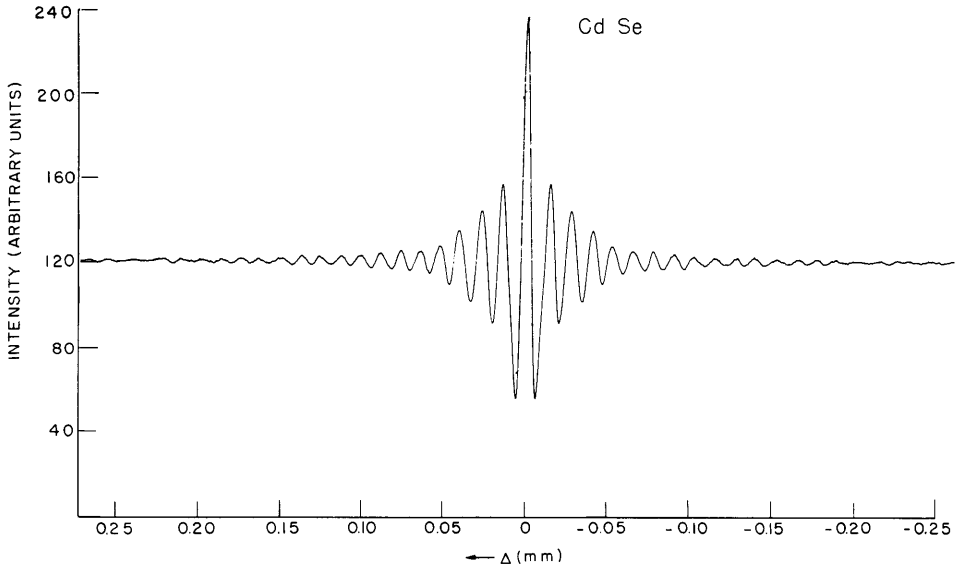


Fig. IV-10. CdSe reflectance interferogram.

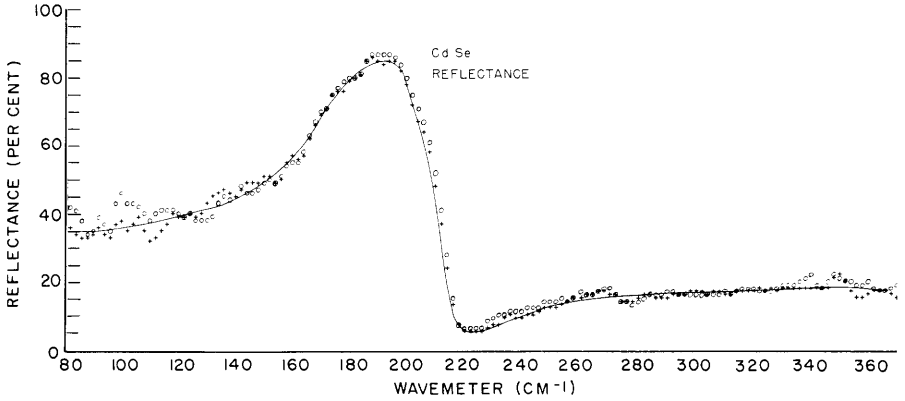


Fig. IV-11. CdSe reflectance spectrum. The circles were computed from the data of Fig. IV-10 with positive Δ, and the crosses with negative Δ.

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