

NSG-212

UNPUBLISHED PRELIMINARY DATA

RECENT INSTRUMENTATION FOR THE OPTICAL STUDIES
OF SOLIDS IN THE VACUUM ULTRA-VIOLET

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July 1964

OTS PRICE

XEROX	\$	<i>2.00 KS</i>
MICROFILM	\$	<i>0.50 mf</i>

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and Department of Physics
Cornell University
Ithaca, New York

N 65 10684
(ACCESSION NUMBER)

28
(PAGES)

CR 59470
(NASA CR OR TMX OR AD NUMBER)

(THRU)

1
(CODE)

26
(CATEGORY)

REVISION CONTROL No. _____

Recent Instrumentation for the Optical Studies
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Synopsis

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Recent progress in vacuum ultra-violet instrumentation and techniques useful to solid state physics is described, although much of the instrumentation advances are also of use to other V-uv studies. Improvements in sources, detectors, and dispersive systems (particularly in grating reflectivities) are noted. While commercial instrumentation is not described, it is perhaps in this area that the most striking instrumental progress has come. Besides optical studies, work on internal and external photoemission continues to augment the optical work. Interest in luminescent systems is also still useful as indicating something about the energy band scheme in insulating

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solids. An important advance is noted wherein use is made of laser radiation in conjunction with ultra-violet radiation in a double photon absorption process in KI.

Author

Introduction

It seems fair to say that, previous to 1945, research in the spectral region of the vacuum ultra-violet, now some eighty years old, went somewhat unnoticed and was mostly confined to atomic spectroscopy. After that year, under the great impetus given it by interest in solid state physics and later by rocket instrumentation and the advent of what Tousey in his valuable review¹ calls "space astronomy," the field greatly expanded and became of importance to other areas. It was not until 1962, however, that the first International Conference on Ultra-Violet Radiation Physics was held, in Los Angeles under the organization of Professor Weissler. The papers given there form a sizable compendium² on the status of the field as of that time. About a month later, in Leningrad, our Soviet co-workers held a similar conference.³

It is the purpose of this paper to review recent progress in instrumentation for solid state physics studies in this spectral region. It will be somewhat arbitrary, both as to what is taken as recent and how far down toward the X-uv⁴ is considered. It is abundantly clear also that much optical instrumentation for solid state studies will be useful elsewhere, and vice versa. In presenting this material, then, no sharp line of demarcation will be drawn, but instrumentation generally of the past few years will be emphasized. Since little of this is work of our own laboratory, and that work

not necessarily the writer's, it seems somewhat presumptuous of him to undertake doing this. He apologizes for the presumption and only hopes that the principle advances are noted and the purposes of this Conference served.

Techniques and Instrumentation

In reviewing the advances of the past few years, both in the techniques and tools of the V-uv spectroscopist, as well as what has been learned with their use, one is rather impressed with the fact that, for the most part, the advances have been in refinement. We are still measuring optical transmissions in thin films, or reflectivities in bulk to infer the energy band scheme in solids. We are using luminescent excitation and photoelectric effects, both internal and external, to tell us the same story. Basically, our instruments cannot change very much for doing these.

Conspicuous in the instrumentation progress in the field during the past few years, is the growing list of beautiful and excellent instruments and accessories commercially available, both in our own country and abroad. Largely the instrumentation is in the dispersing system and vacuum system, but at the same time equipment may be obtained complete with source, motor drive, filters, detector, and recording system, as one's taste and purse dictate. In many ways this in itself becomes really the outstanding contribution to the instrumentation for advance in the field. For information on such, however, one had best refer to advertising in trade journals and literature available from manufacturers. Our comments beyond this will not touch on specific commercial instruments.

Sources: Interest in new sources continues. While spark

sources are used, and particularly so as the X-uv region is approached, the low voltage d.c. discharge in hydrogen is probably the most widely used V-uv source. It is useful to 900A and can be extended to about 500A with the use of argon and helium, the region below 1000A being obtained principally in line spectra. Probably the most versatile intense source in use at the present time (and available commercially) is the capillary discharge source of Newburgh, Heroux and Hinteregger,⁵ which can be used either in a d.c. mode or at higher pressures and with helium in a condensed discharge mode to produce the He₂ continuum above 600A studied by Tanaka.⁶ This has the disadvantage that it is a rather higher voltage source and necessitates large differential pumping at the entrance slit of the monochromator because of its high operating pressure. In the same paper, these authors describe another source producing lines of ionized gases. This is a rugged version of the well known Schueler lamp. Lines down to 256A were observed. With fairly wide slits the photon flux in any one line is not high (on the order of 10⁷ photons/sec.) but, depending on the detection system employed, ample for useful measurements.

Other hydrogen discharges excited by microwave power generators have been described recently.^{7,8,9} While these appear to be of comparable intensity to the low voltage, hot cathode sources, their principle advantage would seem to be

in a simpler structure. Maintaining a microwave unit, on the other hand, would seem to represent more of a complication than that of maintaining a d.c. supply.

One of the more interesting and promising sources described in the past two years is one¹⁰ based on the Penning discharge. This is a d.c. discharge run at moderate pressures in a strong magnetic field between two cathodes at the ends of a cylindrical interaction region which is surrounded by the anode. The discharge is run with argon and operates at relatively high voltage. Lines down to a few hundred angstroms are readily photographed in grazing incidence instruments. In a version of this source constructed for one of our normal incidence instruments, lines were observed only to about as far as were observable with the hot cathode capillary argon discharge and with about the same intensity. In our version of the lamp, however, the life was very short before sputtering of the molybdenum cathodes shorted them to the anode. Further development of this source may yet make it a useful tool.

Radiation from high energy electrons is the basis for two other sources. Of prime importance here is the synchrotron radiation from radially accelerated electrons. Besides being an intense source of continuum from the visible down to the x-ray region (depending on electron energy and orbit radius), it is a source of intensity calculable from first principles

and the geometry of the beam. To date this has not yet been properly exploited but a group at the U. S. National Bureau of Standards is undertaking work making use of this radiation.¹¹ Cerenkov radiation from high energy electrons injected into a gas has been used^{12,13} to determine the gas index over a wide range. While application to solids is not clear, it forms an interesting method. Filters only were used to isolate spectral regions, the gas pressure being varied and the threshold noted at which radiation of the various spectral regions appeared. To apply this technique to solids would require varying the energy of the incident electron beam instead to determine the various wavelength thresholds.

Dispersive Systems: The concave grating reigns supreme for the dispersion of vacuum ultraviolet radiation. Among gratings, the replica is by far the most important (except in the X-uv region), both because of its high quality and reasonable cost. In the X-uv region the grating still is glass and is used at grazing incidence for best efficiency¹⁴ but in the 500-2000A region the grating mounting and coating vary. Workers in the field divide in their use of the normal incidence instrument and the use of the Seya-Namioka focusing configuration. Commercial instruments are available in both forms. Since precise focus is usually not of prime importance in solid state investigations, the instruments may or may not make adjustment in focus as the grating is turned. One recent

monochromator couples the position of the exit slit to the grating angle to maintain it at the focus.¹⁵

One important aspect of the dispersing system is the efficiency of the grating, which is enhanced by both blazing and by raising the reflectivity. Much work has been done in achieving high reflectivities down to 1000Å. Speed of evaporation, degree of vacuum during and immediately after evaporation, and interference effects are all matters that influence the reflectivity. If air could be kept from the reflecting surface, the evidence is that the reflectivity of aluminum could be as high as 85% at a wavelength as low as 1100Å.¹⁶ Unfortunately this seems an impracticality but from the work of Haas and Tousey it has been known for a number of years that a layer of MgF_2 retards the formation of oxide and, through interference, further increases the reflectivity.¹⁷ Gratings so coated are now commercially available. In the 2000Å region a uv-irradiated SiO film over the aluminum holds the reflectivity to 90%.¹⁸ Aluminum covered with a layer of LiF will hold the reflectivity to 60% at 1000Å and a very light overcoat of MgF_2 on this retards deterioration of the LiF.¹⁹ Below 1000Å rhodium appears quite good but generally speaking platinum here appears to still be the best material to use until one gets into the grazing incidence type of instrument.^{20,21} Other work²² indicates gold might be useful but demonstrates in both platinum and gold wide variability in the reflectivity

and, indeed, on the efficacy of blaze, even on gratings appearing identical under microscopic examination. Very recent work shows refractory metals to be good reflectors below 1000Å.^{22a} It is quite clear that the last word has not been heard on gratings.

Concurrent with grating reflectivity progress has been the exploration of other reflectance coatings making use of interference. To date, modest V-uv reflection filters have been made²³ and multiple beam interferometry has been extended down to 1825Å.²⁴ While no significant application to solid state physics can be claimed for either, the trend is clear.

Detectors: As with other aspects of V-uv instrumentation, the progress in the means of detecting the radiation is again primarily in refinement and understanding of our present detectors. For the most part, sodium salicylate is still used as a wavelength converter, the blue wavelength radiation of which is detected by conventional low noise photomultipliers. Tuchscheerer²⁵ has investigated in the near-uv the role of its thickness, its emission spectrum, and relative efficiency but probably the definitive work for our purposes will be that of Allison, Burns and Tuzzolino.²⁶ The quantum efficiency is found again to be near unity, a standard method of application is given,²⁷ the effect of thickness and the angular distribution determined. A feature of this work was the use as an absolute fluorescence photon detector of a silicon surface-barrier photodiode, which had earlier been found to be an effective V-uv detector with rising efficiency over the range

of 10 to 21 ev.²⁸ Other new phosphors have recently been looked at²⁹ for use in the vacuum ultraviolet but only one other now seems to be a serious contender to the sodium salicylate. This is the phosphor "Liumogen," which is quite flat in spectral response³⁰ and goes down in evaporation in a rather glassy state. As such it has been used to measure the absorption of thin absorbing films which may be evaporated over it.³¹ It is rather more temperature sensitive than is sodium salicylate but this is of concern only in absolute measurements.

One interesting phosphor application makes use of $\text{CaSO}_4:\text{Mn}$. This is irradiated by the ultraviolet radiation being measured and its luminescence then measured upon thermal stimulation, the phosphor storing the information sought. A coarse but simple reflectometer has been constructed using this detector.³²

Metallic photoemitters make good detectors below 1000A. They have the advantage that scattered visible light is not so serious, it being of not enough energy to eject many electrons. The currents while small, can be detected conveniently with sensitive electrometers or otherwise made to produce large effects, either through electron acceleration and subsequent excitation of an auxiliary phosphor³³ or by sending the electrons into a photomultiplier structure of the conventional type³⁴ or into the Bendix channel or strip multiplier.³⁵ The latter is a particularly good device, being very insensitive to atmos-

pheric exposure and small enough to make very effective use of the primary photoelectrons, which may be generated from a cathode of one's choosing. This can also be the case in the open multiplier. Since the pioneering work of Weissler, gold still seems to be preferred as a photocathode. Recent work³⁶ on absolute measurements of intensity making use of a type of double ionization chamber has remeasured the photoelectric yield of gold as well as of low reflectivity gold black. (Further information on sodium salicylate was also obtained.)

Sealed off photodiodes and photomultipliers with uv-transmitting windows are being developed but, while useful to 1100Å, are not yet in wide laboratory use.³⁷

In the X-uv region photon counters are used,^{38,39,40} but unfortunately in the V-uv essentially no use has yet been made of them. Nor is photography a much used technique in the region, but it can still be used on occasion to advantage. The emulsion must be either free of gelatin or it must be sensitized with a phosphor. Sodium salicylate itself can be used for this as was recently done in a determination of the number of electrons in the conduction band of aluminum by photographically observing the transmission cutoff of thin aluminum films.⁴¹

Miscellaneous: Interest has for some time been shown in the development of polarizers for this region of the spectrum. Particularly in the study of impurity centers in solids, where symmetry in the crystal plays an important role, studies in

polarized excitation can be illuminating. Until recently such measurements had to be restricted to the visible or near-uv region of the spectrum. Walker has now described a polarizer made up of a stack of LiF plates for use down to 1200Å.⁴² The polarization is quite high but the transmission is fairly low. Nevertheless it will be a useful adjunct and applications for it will not be long in coming.

Vacuum technology is not usually considered part of optics technology but with more work needing to be done on surfaces of pure materials which are now studied in a poor vacuum, the need is arising for better vacua. The importance of this to the reflectivity of aluminum was cited earlier. We are investigating a similar situation in LiF, where an impurity, long-wavelength photoemission tail is observed. This is being studied down to about 1150Å in an ultra-high vacuum region sealed off from the rest of the vacuum system with a thin LiF window. The phenomenon is quite likely associated with a decrease in transmission we and others⁴³ have observed in LiF after cleaving. Lower wavelengths can be reached at ultra-high vacuum by sealing off the region with only a very thin transmitting film after the rough vacuum is obtained both in the sample chamber and the surrounding region.^{43a}

Scattered light has in past work become serious. A few years ago we investigated a position-modulated exit slit used in conjunction with a line spectrum as a means to eliminate

the problem.⁴⁴ Phase sensitive synchronous detection was used with a sensitized photomultiplier. With the detection frequency the same as that of the drive, maximum signal is observed on either side of the line, the phase reversing between them. When the scattered light modulation itself becomes comparable to the line modulation, at weak lines near the central image, it becomes profitable to change detector frequency to the second harmonic of the slit drive, maximum signal then coming with the line central on the slit. While the system is encouraging enough to warrant further work, we still use photoemissive detectors as being more convenient and reasonably adequate.

A somewhat analogous system utilizing a spectrally sensitive radiation chopper which effectively modulates the V-uv radiation without much being done to scattered radiation has also been used to advantage. For example, Madden and Canfield describe an elegant, near-normal incidence reflectometer providing AC discrimination between radiation passed by either high silica glass and LiF or between LiF and an open window.⁴⁵

Measurements: Since optical studies generally involve measurement of both an incident and emergent beam of radiation, considerable time would be saved and convenience gained if a double beam arrangement could be contrived.⁴⁶ Such instruments in the visible and near-uv are commonplace by now. In the V-uv region such an instrument has been devised^{46a} which achieves

the double beam configuration by converting the uv-radiation directly to visible light with sodium salicylate both on the chopper and immediately behind the sample, imaging both phosphors on a single photomultiplier which is followed by amplifiers and a servo system balancing the two signals by moving an optical wedge in the path of the "incident radiation phosphor" light. A commercial instrument providing a two beam arrangement has also just been made available.

Techniques have not changed greatly, however, during the past few years. Measurements, rather, are simply more reliable and extended. Examples of good measurement are well exemplified by the work of Eby, Teegarden and Dutton on the absorption of the alkali halides.⁴⁷ While the work is now nearly five years old, it seems safe to say that the techniques or value of the data have not been greatly improved on since. A modified Hanovia, low voltage, hot cathode hydrogen discharge was the source; the dispersing system a small normal incidence grating monochromator; absorption was determined by evaporating two different thicknesses of salt on thin LiF substrates, the relative absorption being given by the difference in transmitted radiation in the two samples; the radiation was detected with an end-on photomultiplier coated with sodium salicylate, the spectrum being scanned in 100Å intervals to eliminate question concerning source stability.

Much this same technique was used last year by Baldini

in an investigation on some of the solid rare gases.³¹ In this instance a short sapphire rod is evaporated on each end with the phosphor "liumogen". At helium temperature, films of the rare gas are deposited over this, of different thickness on the two ends. Light incident on the film is partially absorbed and the remainder excites the phosphor immediately beneath it, which light is seen by the photomultiplier looking in at the other end of the rod. The cryostat is then rotated to repeat the operation with the film on the other end. The difference gives the relative absorption of the solid gas, free of reflectivity and scattered light error. The technique has been extended to a study of hydrogen and deuterium impurities in rare gas films.⁴⁸

Many people are now using reflectivity data and Kramers-Kronig analysis in determining optical constants. Probably the most complete analyses of this sort are those of the General Electric group in the United States. In their work on the optical properties of a few semiconductors⁴⁹ a normal incidence monochromator (semi-focusing) is again used with a hot cathode, capillary, low voltage hydrogen and argon discharge source. Two sensitized photomultipliers were used -- one for the incident radiation with the sample removed from the beam and the other for the radiation reflected from the sample in place. Combining their measurements in the V-uv region with the best results of other workers on silver and copper⁵⁰ and on aluminum,⁵¹ a

very detailed picture of the band structure in these metals has been obtained. It is this type of progress which appears to be the most striking of all during the past few years.

The use of two photomultipliers in absolute reflectivity measurements may lead to difficulty unless the two are inter-compared. However, for much work this is not necessary. Smith's light pipe⁵² in principle allows of absolute measurement but is rather inefficient in our experience. We catch the incident or reflected radiation on an aluminum mirror which can be swung around the sample to be reflected upwards at about 45° to a downward looking, phosphor-coated photomultiplier mounted axially over the sample, the incident beam being observed by removing the sample. Signals on the order of ten higher than with the light pipe are obtained with this. Phillip's two detector system, avoiding the extra reflection, probably gives signals still higher by nearly the same order of magnitude. Below about 1000Å, our reflector we simply replace by a gold photoemitter, recording the emission.

Another recent two photomultiplier reflectometer⁵³ is based on a method of Tousey.⁵⁴ Measurement of reflectivity is made at only two angles and the optical constants are then read from predetermined sets of characteristic curves, correction being made through other nomograms for grating polarization effects. One of the two photomultipliers is arranged to move through twice the angle of incidence and the other, fixed,

catches part of the very astigmatic image of the entrance slit at the exit slit to monitor the incident light.

Hunter has recently applied a different and new technique for obtaining optical constants in the V-uv region.^{55,56} He studies the cut-off slope in the reflectivity vs. angle curve as functions of wavelength, in the region of the critical angle for the materials, together with the fringes observed on the normal incidence side of the critical angle. This must be done at wavelengths below where (for aluminum -- about 830A) the index of the material goes to less than unity and the transmission becomes quite high. The interference part of this technique is somewhat similar to another, rather more complicated method, where reflectivity behavior in thin films is studied as a function of the film thickness.⁵⁷

Another approach to the optical properties of solids is through a study of impurity or defect absorption and luminescent properties of the material. Many of the alkali halides, as an important class of materials, involve V-uv studies of such excitations. Alderson and Dimond have recently studied the excitation of KCl:Tl over a region of from 4 to 10 ev.⁵⁸ Effects of impurity, exciton, and band-to-band excitations were observed. Mahr, in our laboratory, has carried on investigations on mixed systems,⁵⁹ notably KI in KCl, measurements on which just get into the V-uv. Lifetime studies on the localized excitations involve short uv-light flashes. Use

was made of sparks and conventional flash lamps, which in quartz give radiation down to near 1850A and with special windows could go well beyond this. Many of the usual sources could also be pulsed.

An incentive for pulsed sources may come from the desire to employ a new technique and the one seemingly exciting development in recent years for this field. Many materials, such as the alkali halides, have such high absorption constants in their intrinsic regions, that study of bulk absorption is impossible. Photoconductivity in them has been observed only in recent years,^{60,61,62} and deductions of the electron "schubweg" lead to a value so small as to make one wonder whether it should be called conductivity. The concensus is that all excitations take place immediately adjacent to the surface and the interior of the crystal plays no role. It has become a possibility that, with the advent of the laser, one might carry out in a two photon process interior excitations with energetic enough photons well removed from the absorption edge. Last year, Hopfield, Worlock and Park did this, mixing light from a ruby laser with that of a conventional source near the absorption edge of KI, observing the expected increase in absorption of the uv-light when the laser beam was also incident on the KI crystal. In spite of the application thus far being at the long wavelength edge of the V-uv, this seems to represent the one striking experimental development for the field. Tenta-

tively, we have observed the same absorption with the technique and are attempting to ascertain whether electrical signals observed represent true photoconductivity or are merely the effect any crystal would show when effectively struck with a hammer.

Other solids studies relating to their optical properties, while using the V-uv are perhaps more electrical in nature. Such are studies in external photoelectric emission, internal photoconductivity, conductivity "glow" curves, and the like. These have been and likely will remain popular. The niceties of work in this general field, however, are rather in the electrical instrumentation than in the optical and so they will not be dealt with extensively. As a representative example, the extension of earlier work on LiF and KCl can be cited. Newburgh⁶⁴ has carried measurements on external electron yield and energy distribution out to nearly 50 ev, using much the same instrumentation as used by Heroux and Hinteregger⁶⁵ in a similar study on some metals. The energy analysis was carried out in planar geometry rather than in semispherical geometry or by magnetic analyzer as others have used. Such yield and energy distribution measurements nicely augment optical measurements and can themselves lead to a rather detailed picture of the band structure in a number of materials.^{66,67}

A final technique relating to optical properties of solids is entirely electrical. This is in study of the discreet

energy losses suffered by electrons in passing through thin films. Principally revealed in such studies are the effects of plasma oscillations.⁶⁸ Simpson proposes, as a result of progress in obtaining very monoenergetic electron beams, doing V-uv and X-uv spectroscopy of atomic states in the range of 1 to 100 ev, with a resolution of about 1000 at "wavelengths" below 600A.^{69,70} The analogue in solids is the discrete energy loss mechanism. Such precision in the latter field, however, seems very remote.

Conclusion

While no pretense is made here that all the recent developments in V-uv optics technology as related to solid state physics have been cited, particularly those from abroad, it is hoped that the major developments have been given and the general trends in the field indicated.

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- * Work supported by the following United States Government agencies: Office of Naval Research, Advanced Research Projects Agency through the Materials Science Center of Cornell University, and the National Aeronautics and Space Administration. The author is further greatly indebted to the National Science Foundation for making his attendance at this ICO Conference possible.
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