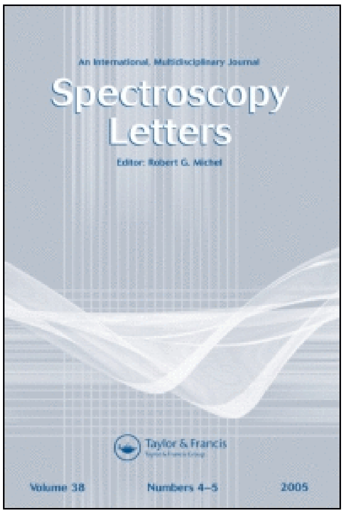

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A Polariton Experimental Arrangement

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A POLARITON EXPERIMENTAL ARRANGEMENT+

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(ABSTRACT)

A system is described for polariton investigation in solids through Raman scattering. Restrictive conditions concerning acceptance angle and angular precision measurements, including line shape and intensity of polariton Raman lines, are met by a simple mechanical and optical concept. Results are optimized by a photon counting arrangement that allowed a very practical data acquisition system.

INTRODUCTION

The angular dependence of light scattering in solids, and in particular polariton curves, have been treated by numerous authors; see for example the review by Barker and Loudon[1], and Scott[2]. The experimental aspects of the measurement of polariton dispersion curves, including variations in line positions, intensities and widths, require considerable attention to detail if accurate results are to be obtained. Nicola and Leite[3], have recently shown new features in the dispersion, intensity and line widths of a phonon polariton in zinc selenide at room temperature, and more recent work[4] has shown similar results in cadmium sulfide at low temperature. The upper branch made of E_1 polariton in ZnO (5) was first observed with the aid of the present experimental arrangement. It is the purpose of this paper to describe the experimental arrangement used in the measurements on zinc selenide, cadmium sulfide and zinc oxide, which attempts to minimize all sources of error.

MECHANICAL AND OPTICAL SYSTEM

Requirements

Several requirements must be met by the mechanical and optical system. Firstly, the laser beam incident on the crystal must be well collimated, highly polarized, preferably at a constant angle of incidence, and of constant (or accurately known) power. Secondly, the scattering angle must be accurately measurable and easily adjustable over an adequate range, and the solid angle collected by the system must be well defined. Thirdly, it is desirable that the area of the grating illuminated by scattered light should not vary as a function of the scattering angle, since the efficiency of the grating may vary across its surface. This is a major problem with the "conventional" polariton set-up such as that shown in figure 1. Finally the system must be mechanically stable and free of vibration, these last points being particularly important since measurements may be spread over a considerable period of time and since the system is required to support a cryostat. To our knowledge no polariton measurement experimental system so far published, allows for variable temperature measurements over a wide range.

Optical Arrangement

Figure 2 shows the general principles of the arrangement, and figure 3 the mechanical system.

The central shaft of the rotating table (a high precision Karl Lambrecht divided circle reading to 15 seconds of arc) and the arm which supports the two reflecting prisms are fixed to the sample chamber. The prisms were clamped against rubber "O" rings, the clamping screws providing fine adjustment of the prism angle. The first prism in the series and the sample lie on the rotation axis, and the laser beam enters the system vertically along this axis.

An air spaced calcite polarizer of high extinction ratio (10^{-6}) assures the correct linear polarization for any position of the rotating table. The beam splitter placed immediately before the sample sends a small fraction of the laser beam to a monitor photodiode, which is used to

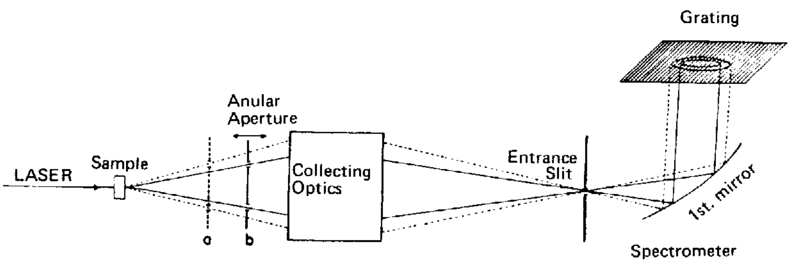

Fig. 1 Conventional Polariton Measurement System.

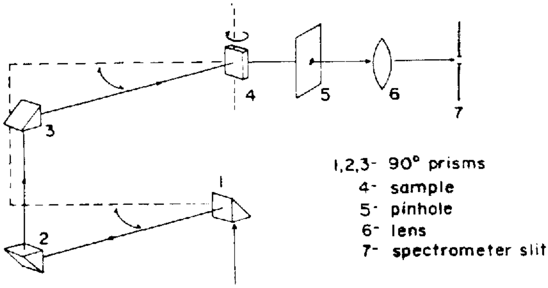

Fig. 2 Optical System.

maintain the laser output constant via a feedback loop. At the small angles typical of polariton dispersion measurements the various reflections do not produce appreciable beam depolarization, and thus in such cases the beam power monitor is not essential.

The sample position with respect to the spectrometer was adjusted by powerfully illuminating the spectrometer exit slit, and placing the sample on the image of this slit. A pinhole was placed 10 cm in front of the sample to define the solid angle collected, which in general was less than 2 x 10^{-6} steradians (5 minutes of arc diameter). The zero (forward) scattering angle was determined by measuring all dispersion curves on both sides of zero, since the curves are known to be symmetric. This removes any small alignment errors.

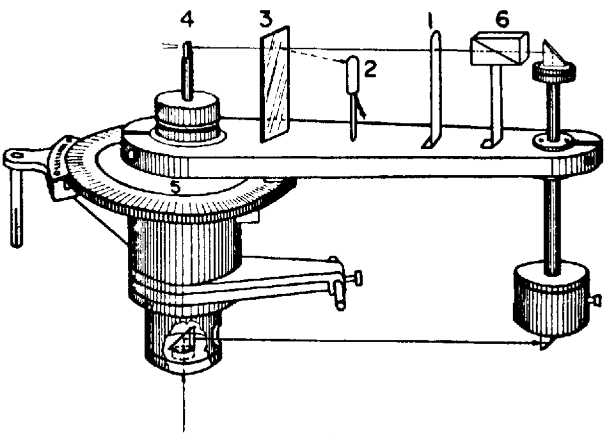

Fig. 3 Mechanical System.

Sample Chamber and Cryostat

Details of the sample chamber are shown in figs. 4 and 5. It consists of a vacuum vessel with detachable upper and lower parts. The lower part contains the optical windows and can be easily removed to obtain access to the sample holder and heating device. Through the upper aperture it is possible to dismantle the unit completely. The inner part consists of an integrated assembly which performs the functions of height and rotational adjustments, cryostat, thermal insulation, sorption vacuum pump, heater and sample holder. The whole assembly is sustained by the height adjusting nut in the upper neck. A triangular "pressure ring" is placed near the sample in order to reduce vibrations. Its design minimizes heat losses. The

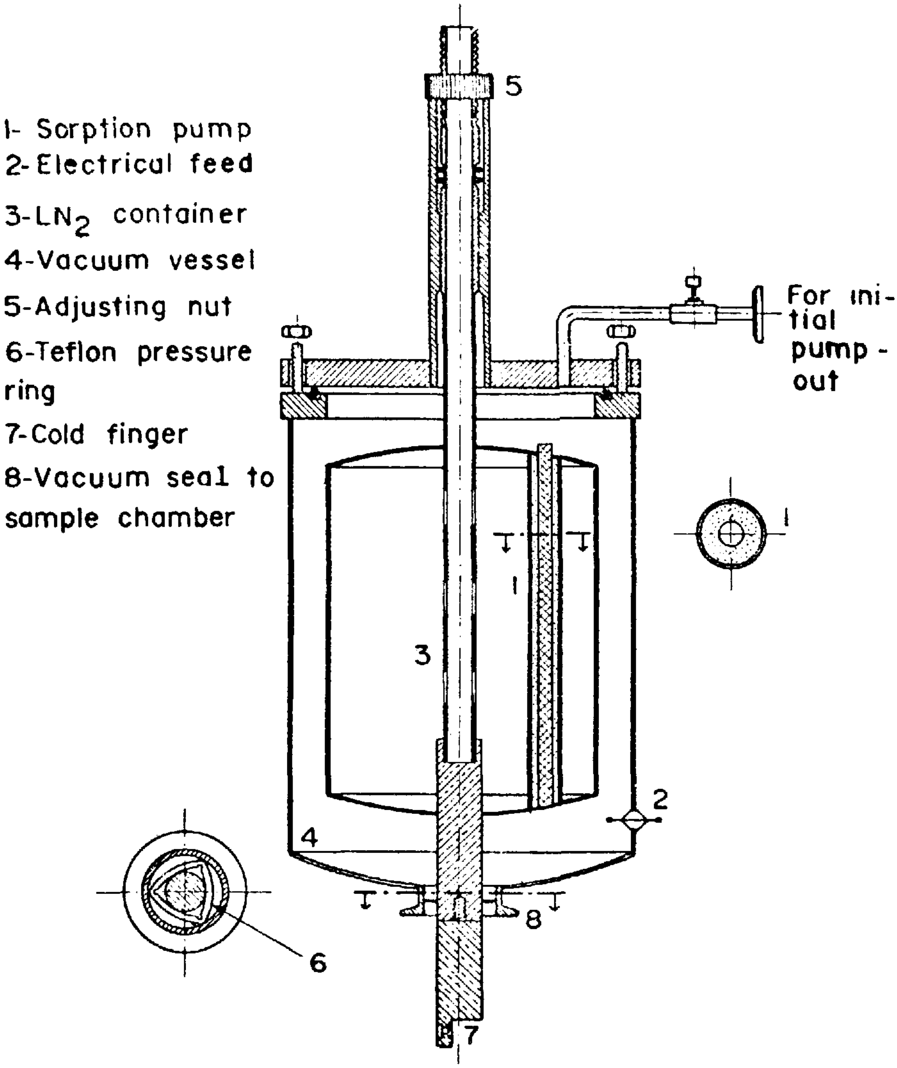

Fig. 4 Cryostat.

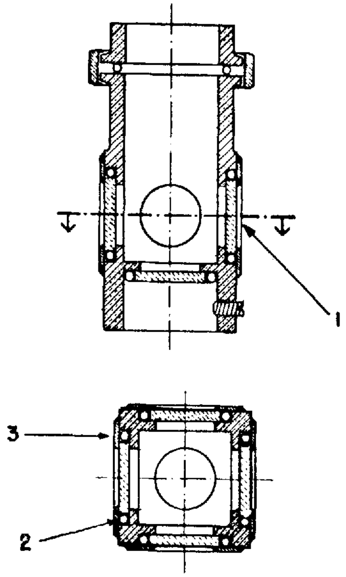

Fig. 5 Sample Chamber

inbuilt sorption pump permits operation without external pumps and provides a clean vacuum. The "hot head" is conventional, and with proper choice of thermal resistor, it is possible to operate over a very wide range of temperature control conditions. Depending on the materials employed, temperatures from 77°K up to 700°K are feasible. Both heater and thermocouple wires pass through the pressure ring holes to connections in the side of the external vessel. The details of the vacuum seals for the optical windows are shown in fig. 5.

This method of sealing the windows has the advantage of providing unusually good optical access to the sample, but requires windows with well-finished, accurately circular edges.

The liquid nitrogen consumption in normal operation at 77°K was 4 litres per day.

DATA COLLECTION SYSTEM

Requirements

A prime requirement of the data collection system is a high signal to noise ratio. Particular attention must be paid to this, since the very small collection angles for the scattered light inevitably greatly reduce the signal. For accurate measurement of intensity variations the overall gain and zero level of the system must be stable, and the system must be accurately linear. Measurements of small shifts in line positions and line widths require a data collection system which ensures accurate synchronization between the recorder intensity and the grating position.

These requirements are best met by using a photon counting detection system and digital control of the spectrometer. Photon counting provides a small improvement in signal to noise over "conventional" detection[6] but is particularly useful in this case since the gain and zero stability are greatly improved. In order to obtain high linearity the photomultiplier is operated at low gain, and thus the anode current remains small and fatigue effects are avoided.

Monochromator Controller

The controller was designed for use with a Jarrell Ash model 25-100 monochromator, which has a stepper-motor drive requiring 20 pulses per cm^{-1}. The system could be readily modified for any stepper-motor driven instrument.

A block diagram of the controller is shown in fig. 6. The spectrum is broken up into channels, the purpose of the controller being to determine, independently, the time taken for a given channel (the integration time) and the frequency interval represented by that channel (resolution). A channel advance pulse is provided at the end of each channel. The stepper motor pulses are spaced uniformly throughout the chosen channel pe-iod, so that the output represents a time integral over the chosen frequency interval.

In fig. 6 all counters are shown as 4-bit binaries for clarity, whereas in the actual unit counters 1, 2 and 3 are 8-bit binaries and counter 4, 12 bit. The clock frequency is 2560 Hz, obtained by dividing down a 1.28 MHz crystal oscillator by a factor of 500. The time per channel and frequency range swept per channel are variable over a range of 0.5 to 128 seconds and 0.25 to 64 cm^{-1} respectively, in binary steps. This range and the binary sequence are very convenient in practice. The output of counter 3 provides the channel advance pulse,

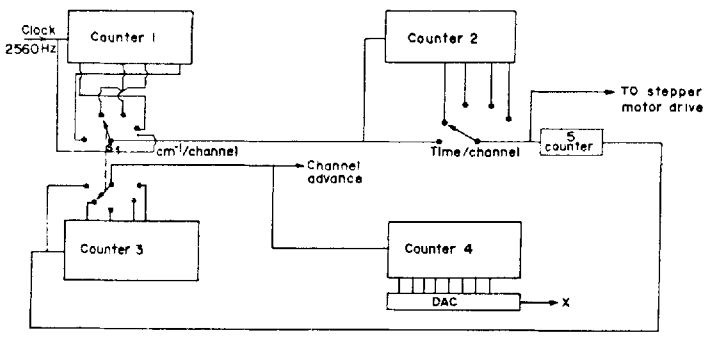

Fig. 6 Controller (Diagrammatic)

and as the number of pulses per channel selected is varied with S1, so the frequency of the pulses at the output of counter 1 is varied in an inverse manner. Thus the time per channel is independent of the setting of S1. Counter 4 records the number of channels already swept, and its outputs are connected to a digital-to-analogue converter. The output of the converter is used to drive the X axis of an X, Y recorder, assuring synchronism between the recorded data and the monochromator movement. An automatic stop facility is provided by halting the clock input when a given channel count is reached. In a similar manner a pause is obtained by halting the clock.

Pulse scaler system

The channel advance pulse generated by the monochromator system is used to control the print-out of the accumulated count by a NIM-bin counter and print controller. This provides a typed and paper-tape record from a teleprinter. A real-time graphical display is also useful.

The system used to provide the graphical display is shown in fig. 7. The vertical scale of the display is selected by the "base range switch" which determines, via the range scaler and data selector, which output of the 12 bit prescaler is connected to the 8 bit data scaler. Full scaling of 2^8 up to 2^{20} (256 to $\sim 10^6$) is thus available. At the beginning of each channel the pre-scale selected by the base range switch is set into the range scaler. If the data scales does not overflow during a channel then the channel advance pulse causes the loading of the data scaler into the memory and the re-setting of the data scaler and pre-scaler. The memory is directly connected to an 8-bit DAC, which drives the Y axis of an XY recorder. The data are thus presented as a histogram.

If overflow occurs, the range scaler is incremented, the last bit of the data scaler is re-set, and the counting is then continued. This results in the displayed range being halved for any channel which overflows the selected range. If necessary the process is repeated. This provides an effective auto-ranging capability easily interpreted visual results, an example of

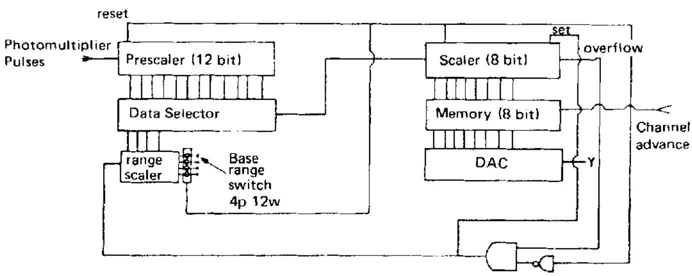

Fig. 7 Data Collection System (Diagrammatic)

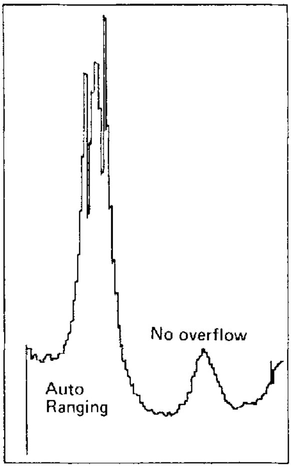

Fig. 8 Typical Spectrum Showing Operation of the Auto-Range Facility.

which is shown in fig. 8. The range scaler reloads the switch selected base-range at the start of each channel, so that when overflows cease to occur the scale returns to its pre-selected value.

The spectrometer controller and pulse scaler system were built from medium-scale-integration TTL units, and the detailed logical design presents no difficulties.

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