DEVELOPMENT OF A SPECTROMETRY SYSTEM USING LOCK-IN AMPLIFICATION TECHNIQUE

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Abstract. Raman spectroscopy and optical second-harmonic (SH) spectroscopy has a very important role in studies of optical properties of materials. Our goal is to build a spectrometry system to measure weak optical signals such as Raman scattering and surface SH signals. We studied to improve an old Double Grating Spectrograph GDM-1000 (Carl Zeiss, Jena, Germany) for wider range of wavelengths and transformed it successfully into a spectrometry system coupling with computer using Lock-in amplification technique. By this spectrometry system we can now obtain weak optical signals, for example, Raman Spectra of Vietnam petrol extracts excited by not only an 2W Argon laser but also by a 30mW He-Ne laser.

Key words: Raman spectroscopy, Optical- Second Harmonic Spectroscopy, Surface SH generation.

1. Introduction

Laser Raman spectroscopy including Raman Scattering, Resonance Raman Scattering, Stimulated Raman Scattering (SRS), Coherent Anti- Stokes Raman Scattering (CARS) has been used as a very powerful spectroscopic technique to study molecular structures and optical properties of condensed-phase matters. Recently, Nonlinear Optical Sum Frequency Generation (SFG) and Second Harmonic Generation (SHG) effects have become a spectroscopic tool for surface probe with high surface specificity [1]. The Second Harmonic Spectroscopy is considered as a prominent candidate for better understanding of the optical properties of nanostructures [2]. However, these optical signals are very weak and it requires a spectrometry system with high resolution and sensitivity. The Double Grating Spectrograph GDM-1000 (Carl Zeiss Jena Germany) of our laboratory has a high resolution but it operates with an old self-recorder and a head-on type photomultiplier M12FC which is sensitive to the ultraviolet region (400-550nm). It is suitable only for studying Raman Scattering excited by 5W Ion-Argon laser or high-pressure Hg lamps. Furthermore, the recorded spectra could not be treated by computer. We studied to improve this spectrograph for higher sensitivity in wider range of wavelengths and coupling with computer to treat data. We replaced the M12FC PMT by a side-on R-928 PMT (Hamamatsu) and detected PMT signals by a digital signal processor (DSP) lock-in amplifier (Model SR830, Stanford). A stepper motor with suitable design and construction was connected. A compatible control and data- acquisition program was written for the new spectrometry system. Several measurements were carried out to determine the operation regime and calibration. We succeeded in obtaining Raman spectra of petrol extracts by 30mW He-Ne laser excitation. Our new spectrometry system shows many advantages for studying Raman and Fluorescence spectra as well as weak optical signal spectra in general.

2. Construction of the improved Spectrometry system

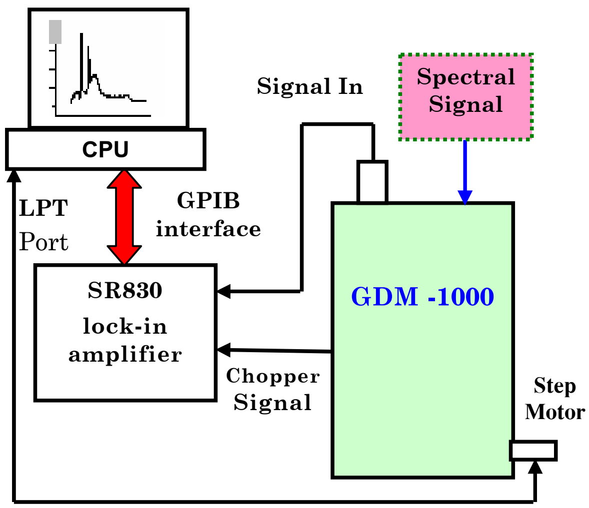

Fig.1: Experimental setup of the spectrometry system

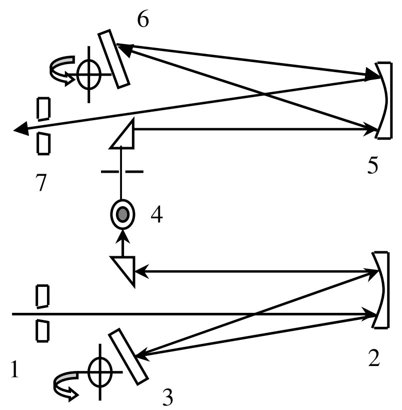

Fig. 2: Schematic of GDM-1000
1-input slit;
2,5 -mirrors; 4-chopper;
3,6- Gratings;
7- output slit

Our experimental setup of the improved spectrometry is shown in Fig.1. The monochromator GDM-1000 consisting of two 650lines/mm gratings can operate in spectrum region of 7500 -16675 cm-1 (for the first order of the grating) and in the region of 16675- 28700 cm-1 (for the second order of grating). The optical schematic of the GDM-1000 is given in Fig.2. We removed the self-recorder and studied to couple the spectrograph with computer.

A stepper motor of 1.8o /step was installed in a suitable position to rotate the grating for wavelength scanning. One step of the motor corresponds to 0.0832cm-1.

In the circuit controlling stepper motor through parallel port of computer, a sequence of digital signals is sent to LPT port: 0001;0011;0010;0110;0100;1100;1000;1001 corresponding to positions steps 1→ 2→ 3 →4 →5 →6 →7 →8 (Fig.3) [3].

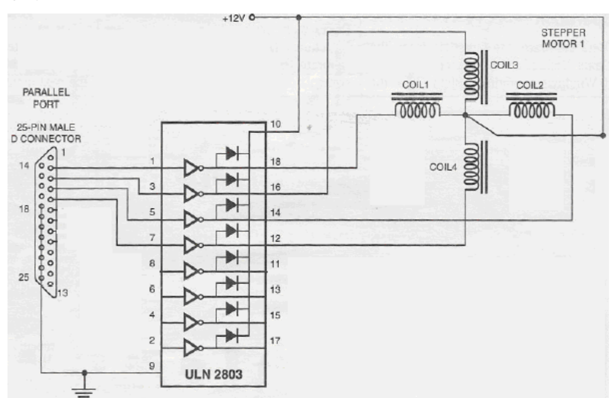

Fig.3: Diagram of stepper motor controller

The head-on type photo multiplier M12FC of the GDM-1000 spectrograph is sensitive in region of 400-550nm wavelengths (Fig.4a). For the longer wavelength response up to 900nm, we replaced the M12FC PMT by a side-on R-928 PMT (Hamamatsu). The spectral response characteristics of R-928 PMT is given in Fig.4b. This side-on R-928 PMT employs a reflection-mode photo cathode and a circular-cage structure electron multiplier which has good sensitivity and high amplification at a relatively low supply voltage. A DP-type socket (C6270-Hamamatsu) with built-in voltage divider and power supply was used to operate the R-928 PMT. The C6270 socket has input voltage of 15V 1Vdc and give output voltage of 0-1250 Vdc .

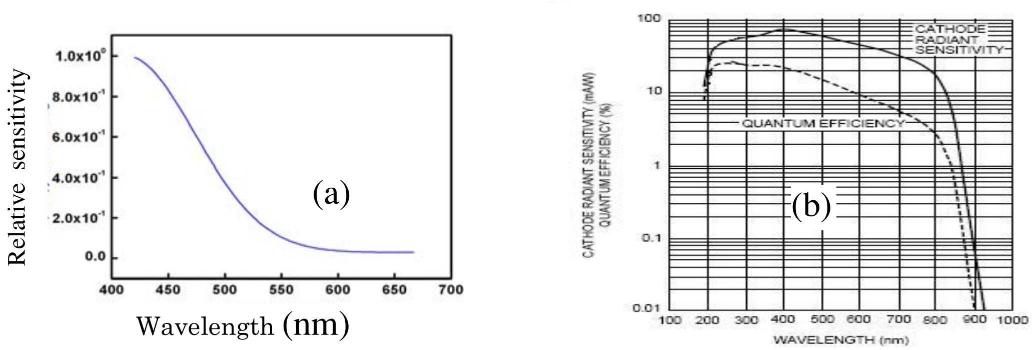

Fig.4 : Spectral sensitivity of the M12FC (a) and the R-928 PMT (b)

Spectral signals from PMT were detected by a DSP lock-in amplifier (Model SR830, Stanford). The block diagram of the SR830 lock-in amplifier was presented in Fig 5. The SR830 has five boards: Key board, display board, Analog Input board, DSP Logic board and CPU and Power supply board. The DSP logic board takes a digital input from the A/D converter on the Analog Input board and perform all of the computations related to the measurements before it is displayed on the screen. This includes generating the digital reference sine wave, demodulating the signal, low-pass filtering the results, and offset and expanding the outputs. The internal oscillator sine output and AUX D/A outputs are generated on this board. The reference phases lock loop control the clock of this board whenever the reference mode is external. These functions are implemented within a system comprised of five function blocks: the digital signal processor (DSP), the DAC outputs, the timing signal generator, the reference clock generator and the I/O interface. Through the use of highly efficient algorithms, the system is capable of real-time lock-in operation to 100kHz with 24dB/oct filtering on both X and Y as well as providing a synthesized analog sine output. The SR830 utilizes a Motorola 24-bit DSP56001 digital signal processor (U501). The DSP is configured without external memory. The lock-in algorithms run entirely within the internal program and data memory of the DSP itself. The host processor bus is connected to the main CPU board via the I/O interface on the DSP logic board. The 80C186 processor on the CPU board acts as the "host" processor to the DSP.DSP firmware and commands are downloaded from the CPU board to invoke different operating modes. The analog input board provides the very important link between the input spectral signal and the SDP processor. From the front panel BNC the spectral signal passes through a low distortion front-end amplifier, gain stages, notch filters, anti-aliasing filter and finally an A/D converter. Once converted to digital form, the input signal is ready to be processed by the Digital Signal Processor.

The SR830 DSP Lock-in Amplifier can be remotely programmed via either the RS232 or GPIB (IEEE-488) interfaces. Any computer supporting one of these interfaces may be used to program the SR830. Both interfaces are receiving at all times, however, the SR830 will send responses to only one interface. We could specify the output interface with the [Setup] key or use the OUTX command at the beginning of every program to direct the responses to the correct interface.

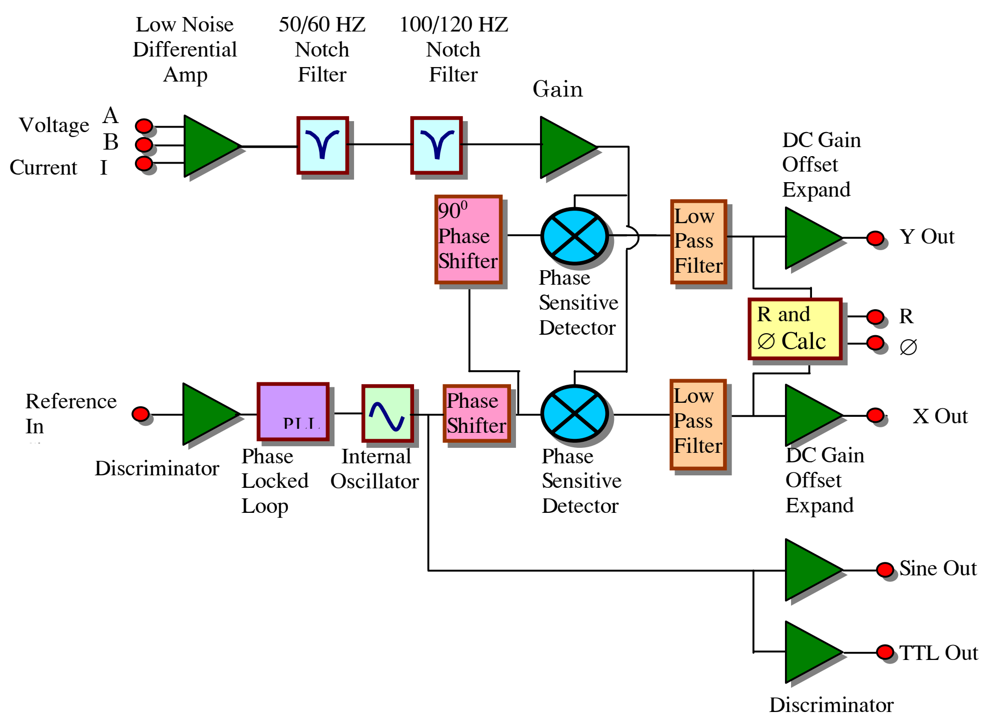

Fig 5: The block diagram of the SR830 lock-in amplifier

In this research, a GPIB card is used to communicate between computer and SR830. Before attempting to communicate with the SR830 over the GPIB interface, the SR830's device address must be set. The address is set with the [Setup] key and may be set between 1 and 30. Communications with the SR830 uses ASCII characters. Commands may be in either upper or lower case and may contain any number of embedded space characters. A command to the SR830 consists of a four characters command mnemonic, arguments if necessary, and a command terminator. The SR830 has a 256 character input buffer and processes commands in the order received. Similarly, the SR830 has a 256-character output buffer to store outputs until the host computer is ready to receive. Detailed command list can be seen in reference [4]. A computer program (named Spectra-Department of Quantum Optics-VNU) was written coding in Visual Basic to control the spectrum measurement procedure and data- acquisition. The interface of this program is presented bellow.

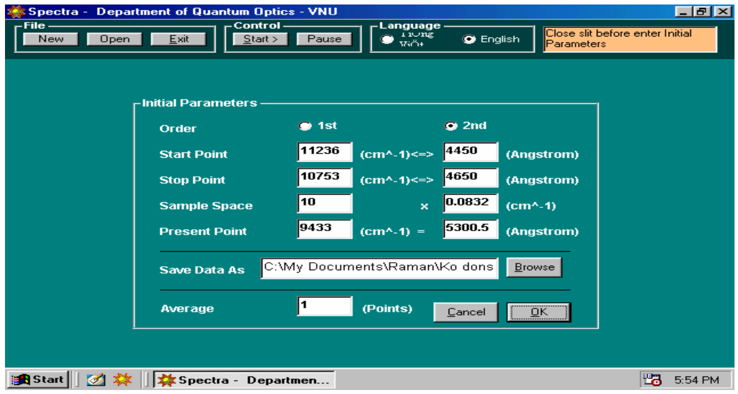

Fig.6: The interface of the spectrum measurement procedure

3. Spectrum measurements

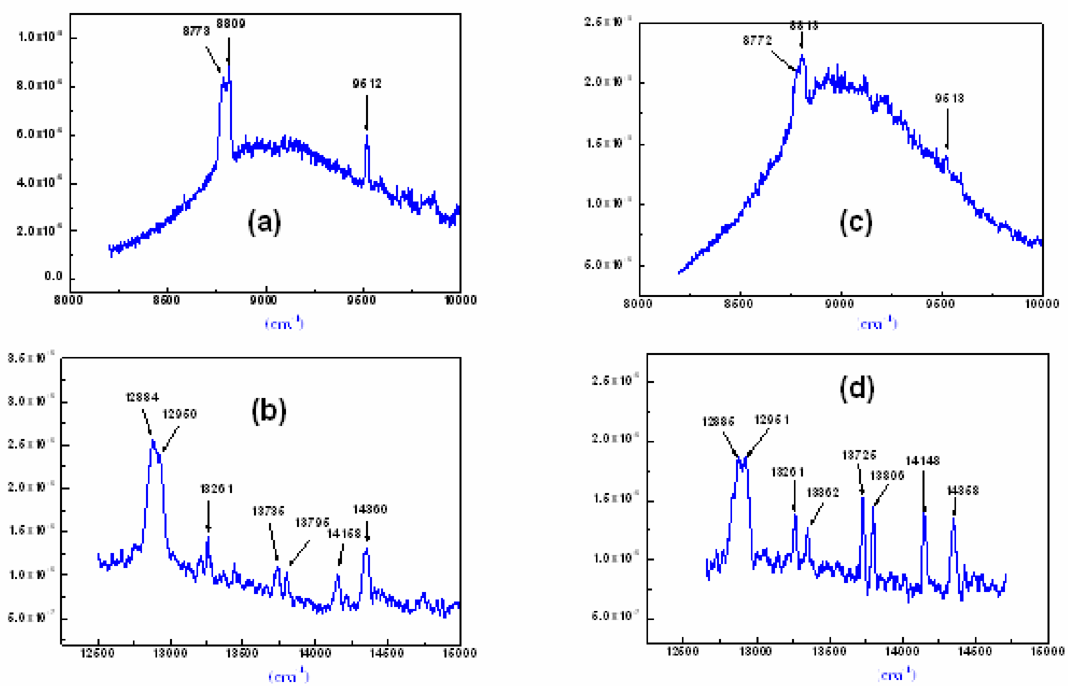

Fig 7: Raman spectra of petrol extract samples
a-Sample 5B110 excited by Argon laser; b- Sample 5B110 excited by He-Ne laser
c-Sample 5B115 excited by Argon laser; d- Sample 5B115 excited by He-Ne laser

We carried out several spectrum measurements to determine the operation regime and calibration of the improved spectrometry system, for examples, fluorescence spectra of Mn-doped ZnS, Stokes and Anti-Stokes Raman scattering spectra of some typical Hydrocarbons excited by Argon laser and He-Ne laser. Our new spectrometry system shows high sensitivity and resolution and many advantages for studying Raman and Fluorescence spectra. We succeeded in obtaining Raman spectra of petrol extracts by 30mW He-Ne laser excitation. The high sensitivity of this spectrometry system in the Red and IR -spectrum region gives the capacity to study the Raman spectra of weak intensity excited by a medium power He-Ne laser instead of CW-high power water - cooled Ion-Argon laser. Furthermore, there was not Fluorescence spectrum profile in Raman spectra excited by He-Ne laser of 632,8nm wavelength. In many cases of Raman spectrum studies, the excitation by near UV- Argon laser results in fluorescence which overlaps Raman spectrum. This was proved very clearly in the Raman spectra of the petrol extract sample 5B110 and 5B11 (petrol extracts from Bach Ho, Vietnam). The Raman spectra excited by He-Ne laser showed more Raman peaks, that means more information, in comparison with Argon laser excitation (Fig.7)

4. Conclusion

We improved an old Double Grating Spectrograph GDM-1000 (Carl Zeiss, Jena, Germany) for high sensitivity in wider range of wavelengths and transformed it successfully into a high resolution spectrometry system coupling with computer using Lock-in amplification technique. This is an economic solution and very suitable for teaching and research in Universities. By this spectrometry system we can now obtain weak optical signals, for example, Raman Spectra of Vietnam petrol extracts excited by a 30mW He-Ne laser instead of Ion Argon laser. The Raman excitation by He-Ne laser showed many advantages in comparison with Argon laser excitation.

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