Development of On-line Instrumentation and Techniques to Detect and Measure Particulates

Quarterly Technical Progress Report

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Abstract

In the first quarter of the project, we reviewed many past references about using light scattering to characterize particulate matters. We also constructed light sources, detection systems and PM synthesizer for the project.

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EXECUTIVE SUMMARY

The first quarter of this project went smoothly ---- we finished acquisition of major equipment needed for the project in a week and got into full speed immediately; we developed laser diode drivers, CCD detectors and mono-sized particulate generators; our reference/literature investigation gave us a more detailed understanding of the light scattering process and let us pay special attention to several PM scattering cases, and we are also on the way to write computer simulation programs for the multi-wavelength scattering process.

1. The major equipment that we acquired for this project include:
 - 1GHz bandwidth oscilloscope, Model LeCroy 534 with 16M (4CH x 4M) of memory, this allows us to debug all the electronics for the experiment as well conduct data acquisition during the experiment. An existing multi-functional ADC card from National Instrument is already available for final experimental setup.
 - Hamamatsu PMT detector (R957-H08) detector with integrated high voltage assembly, this allows us to detect light scattering at very low PM concentrations
 - Optical tables, 2 pcs will give us enough room to conduct experiment
 - SRS DG345 function generator, this gives us the capability to provide various modulation to the lasers as well as the timing for the experiment
 - SRS 834 lock-in amplifier and NewFocus Chopper system, this will allow us to collect signal in modulated form
 - Diode lasers with 6 different wavelengths ranging from 635nm to 980nm were acquired
 - Diode pumped CW laser at 1064nm and 532nm were acquired
 - Laser particle scattering counter, Model 227A from Pacific Scientific Instruments, this is a hand-held device that could detect particle density at fixed sizes (0.5 μm and larger for one channel and 0.7, 1, 3 and 5 μm for the 2nd channel).
2. Electronics, optical and PM synthesis equipment we have developed
 - Laser diode drivers with external modulation bandwidth about 1MHz, including low current models (<250mA) and also high power models (>5A), have been constructed. These laser drivers will drive up to 12 different lasers at the same time.
 - Optics for construction of passively Q-switched lasers have been acquired, this will enable our construction of lasers at 266nm and 355nm.
 - Detector array system based on CMOS array detectors. The detector array we constructed could have integration time down below 1 μs and dynamic range up to 80dB
 - We have also designed and constructed a mono-sized PM generator, and we will test it with the laser particle scattering device (Model 227A from Pacific Scientific Instrument) acquired above. We are also checking the possibility of testing it with a more precise PM measurement meter from Wyatt Technology.
3. Literature review
 - We found more related literature on PM light scattering, especially the special issue in Meas. Sci. Technology, Vol 9, 141-220, 1998, which shed light into light scattering on irregular, i.e. non spherical, particulates.

- From the vast amount of data in PM scattering in water (www.wyatt.com), we also found that we should compare our multi-wavelength scattering results with results obtained with the multi-angular detection scheme.
- We also are looking into other possible information, besides physical sizes, that we could collect with multiple lasers. This include laser induced fluorescence and Raman scattering. The detector array that we build therefore should have the capability to look at these signals.

EXPERIMENTAL

1. Data acquisition system

The 1GHz bandwidth digital oscilloscope also come with 16MB of memory, thus allow us to record data over a very long period of time. Since the fluctuation of light scattering signal could be huge over the time, this capability of the scope will help our initial identification/analysis of signals. In the final experimental setup, even longer data from multiple detectors (e.g. 3) have to be acquired, we already had an ADC card from National Instrument (Model 6023E), which has a maximum data sampling rate of 200kHz with 12-bit resolution and 16 single ended/8 differential input channels. Programs based on VB and Labview are already available, only minor changes are needed to adapt for this project.

2. Laser sources

Some more details about the diode lasers we acquired, they come in the following wavelengths and power,

Wavelength	635nm	650nm	780nm	810nm	830nm	980nm	532nm	1064nm
Power (mW)	30	40	80	1000	30	30	30	200
Package	TO	Cir. TO	Bare	C-mnt	TO	FC	DP/CW	DP/CW

Note: Cir.TO: Circular output (from Blue sky) in a TO can, C-mnt: C-mount bare chip, FC: Fiber coupled, DP/CW: Diode pumped CW operation (could be modulated at low frequency up to 10kHz)

We have also acquired optics for construction of the 355nm and 266nm sources, these optics are based on passively Q-switched lasers (Cr:YAG and Nd:YAG). We hope to generate laser pulses around 1ns, pulse energy over 300 μ J at 1064nm, and at a repetition rate of over 1kHz. The table blow lists the optics set that we acquired,

	L1(mm)	L2(mm)	S1	S2
Configuration 1	0.75	0.25	HR1064, HT808	R=25% 1064
Configuration 2	1	1.5	HR1064, HT808	AR/HT 1064
Configuration 3	3	1.5	HR1064, HT808	AR/HT 1064
Configuration 4	3	3	HR1064, HT808	R=25% 1064

The schematic for the diode pumped passively Q-switched laser is given below

Figure 1. Schematic for the diode pumped passively Q-switch laser

In all configurations, S1 and S2 have parallelism within 5sec, cross sections of 3x3mm. Cr:YAG has absorption about 6cm^{-1} at 1064 nm. The 4 combinations above should give us laser pulses with pulse width ranging from 300ps or less to 4ns, and pulse energy from $10\mu\text{J}$ to $300\mu\text{J}$.

The passively Q-switched lasers are often continuously pumped, with a repetition rate ranging from 1kHz to 10s kHz, and the timing between the pulses have a jitter of $\sim 100\text{ns}$. After review literature, we found that we need to have the capability of externally trigger the laser pulse. The repetition rate should be $< 1\text{kHz}$, and most often $\sim 100\text{Hz}$. The low repetition rate is decided by the data acquisition and analysis speed. Reducing the repetition rate has several advantages. First, the lower repetition rate will lower the pump laser duty cycle, therefore reduce the thermal lensing effect and increase the gain volume. Second, when external triggered, we have a better control of the timing. As the delay between the rising edge of the pump laser current pulse and the Q-switched laser pulse is decided by the gain volume and pump laser power, we could make sure that the Q-switched laser pulses come in at the desired time with a jitter less than 10ns. This is enough since the motion of particles in the air is more than $1\mu\text{s}$. The drivers we designed is based on the WLD3343 chips from Wavelength Electronics. Each WLD3343 could pump out 2.5A, and we use the parallel link to annex two WLD3343 together to provide the 5A required.

3. Array detector

We wish to have an array detector that could allow sub microsecond integration. Our passively Q-switched laser generate intense pulses and allow us to conduct such experiments as LIF and Raman scattering, and the signal is best collected within the short time window, instead of having to integrate over a long time when the noise is also introduced.

We developed two kinds of array detectors. The first one is based on regular scanning detectors, such as a linear CCD array detector used in scanners. Such CCD chips are already widely used in ultra-compact spectrometers, e.g. Ocean Optics S2000. But, the integration time of this CCD chips is limited to more than 1ms, because the time it takes to scan through the 1024 elements is limited by the clock rate (1ms is needed when the scan clock is at 1MHz). Also, the 1024 pixels are not exposed to the same time window. Based on an array detector chip from Photon-Vision Systems (PVS), we designed a board for their LIS-1024 chip, it could have dynamic range of 80dB while the scanning clock could be as high as 1MHz. This board could also be externally triggered. Our design allows this board to be easily changed to fit other CCD detectors, including the ultra-deep well photodiode array, Hamamatsu S3094.

We also developed a board based on PVS' ELIS1024, this chip features the short integration window option, which allows us to integrated all the pixels for a very short while ($<1\mu\text{s}$) at exactly the same time. Also, this chip allows pinning of the 1024 pixels, thus reducing the total of scanning pixels when collecting data (the effective data pixels could be reduced to 64). This not only reduces the time to read the pixels (as short as $64\mu\text{s}$ is needed when the clock is at 1MHz), but also provide the on-chip integration of the pixels where their signal are originated from the same wavelength and should be collected together.

The well depth of the LIS1024 and ELIS1024 are all larger than the CCD array detectors such as the SONY ILX511 used in the OceanOptics S2000 spectrometer. The deep well depth will allow integration over a relatively longer time, while the short integration window could allow transient signal detection, such as LIF and Raman scattering.

	ILX511	LIS1024	ELIS1024	S3903
Well depth (e^-) /pixel	350,000	8,000,000	800,000	31,000,000
Integration time	$>1\text{ ms}$	$\sim 1\text{ ms}$	$<1\mu\text{s}$	$>31\text{ms}$

4. Mono-sized particulate generation

We designed and constructed a mono-sized particulate generator. We will first use mono-sized particles that are commercially available from chemical suppliers, such as Aldrich. These monosized particles are not soluble in water, and we could just first mix the particles with water. A nebulizer will vaporize the water+PM colloidal mixture, and then the flow is pass through a dryer system which gets rid of water. The PM thus have mono-size distribution and know chemical properties. A recirculation system will bring back the PM material to mix with water again and continue the generation for the experimental study.

We are also designing another PM synthesizer, which could allow the generation of mono-sized liquid form aerosols. The designed figure is given below. The chemicals could be dissolved in a certain liquid (e.g. methanol). The liquid is dragged by the high pressure gas jet and experiences a supersonic expansion in the normal pressure chamber. The free expanding beam will form aerosols of different sizes in the air, and a mono-sized aerosol could be picked up by a crossing high- pressure air beam and pushed toward the exiting aperture. After studied in a sealed window chamber, the aerosol material will be recondensed on a cooling/impacting surface and recirculated for continuous study. This design is much more challenging than the first design, because the liquid form aerosols are the tough most to measure/verify their sizes. We will verify our design by using the laser particle sizing instrument we acquired.

Figure 3. Schematic of the WLD3343 x 2 + HTC-3000 board

Figure 4. External current modulation (black) and the actual current (green) and the laser pulses (yellow)

Figure 5. Picture of the TC+LD board with the green laser on the optical table.

RESULTS AND DISCUSSION

We constructed a laser diode driver with rise/fall time less than 400ns. We tested this driver with a CW pumped 532nm Nd:YVO4+KTP laser first, and the results are presented in figure 3, 4 and 5. We could see that the rise/fall time of the pump laser and relative jitter between the 532nm laser pulse and the laser current rise time (note that this jitter will be much smaller when passively Q-switched laser is used). The successful operation of the laser diode drivers will give us the capability to externally trigger the semiconductor lasers as well as passively Q-switched lasers. In the next quarter, we will use the 5A driver to drive a 2W pump laser diode (from OSRAM SPI CG81), and the laser diode will pump the passively Q-switched laser optics that we acquired. We hope that we will get all the light sources ready by the end of the second quarter.

The detector array with sub- μ s integration window will enable us to conduct LIF and Raman scattering measurements. The figure below shows the Shutter Time is set at 50 μ s. Much shorter time has been demonstrated to below 1 μ s.

Figure 6. Figure shows the timing for the logical control of the ELIS1024, and the RSET (reset from external trigger) is independent of the camera CLK (clock) on board.

Figure 7. The array detector assembly includes the “Array Chips” board and the “Clock and Logic” board.

We successfully constructed a recirculating neubilizer that could generate mono-sized insoluble (in water) solid particles in the air. We are also in the process of constructing a PM synthesizer that could generate liquid form aerosols. We need to test the particle size distribution using the laser particle scattering meter we acquired and also the more sophisticated DAWN instrument.

Literature review

We did a more thorough literature review of light scattering techniques for particulate study. One of the most interesting literatures comes from the special issue from Meas. Sci. Tech. Vol. 9, 1998 (page 141-224). Here, 7 papers discussed the light scattering results on irregular, i.e. non-spherical, particulates. The results clearly show that special cares, e.g. time average, must be taken to treat the results from such irregular shaped particulates. Since in the real world, particulates are often generated in irregular shapes, these results convince us that we must consider this effect both in the theoretical simulation model development but also in the experiments.

We also run into the reference database of Wyatt Technology, who now focuses on particulate detection in liquid/biological systems. But their instruments, i.e. DAWN scattering sphere, are also widely used in particulate characterization in the air. We will also calibrate our instrument against systems similar to DAWN. We are in the process of writing simulation code based on the latest literature resources. We will include in the model the simulation of non-spherical shaped particles. There are 2 such multi-angular DAWN instrument available on Caltech campus, and we will be able to compare our multi-wavelength results with results from these DAWN instruments.

HYPOTHESIS AND CONCLUSIONS

This past quarter proceeded as planned and we successfully prepared the array of CW laser sources, and the laser drivers with high current output and fast rise time are ready for the construction of passively Q-switched UV laser sources. We also constructed a PM generator that

could generate solid mono-sized particles, and this generator is ready to conduct experiment. We also found some latest results on PM light scattering on irregular particulates, and the results give us new directions for our simulation modeling.

Planned schedule from the statement of work

Task	Technical Milestone	Schedule
1. Assembly of the multiwavelength light source	Ready diode & DP chip lasers, drivers	Month 1-6
	Ready beam combination system	Month 1-6
2. Construction of the PM synthesizer	Verify that monosize PM are generated	Month 1-6
3. Simulation of Ralyeigh and Mie Scattering	Literature review	Month 1-3
	reviewComputer program that could generate simulated scattering spectrum	Month 1-6
4. Laboratory demonstration of instrument	Experimental scattering spectrum database for different PM sizes	Month 7-18
	Compare with theory and conventional PM monitoring data	
5.Application of the PM analyzer to a combustion environment: engine intake area	Correlation of our instrument data with conventional PM monitoring data	Month 13-24
6.Application of the PM analyzer to a combustion environment: engine exhaust	Correlation of our instrument data with total PM mass emission, new data (PM size and chemical composition) about in-situ PM monitoring	Month 13-24
7. Applicability assessment for PM emissions from coal fired power plants	Design/modify our PM instrument for smoke stack PM monitoring	Month 24-30
8. Instrument design optimization	Optimize the instrument during different experiments	Month 13-36

Reference:

1. J.J. Zayhowski, C. Dill III, C. Cook, J.L. Daneu, OSA TOPS Vol.26,178-186, 1999
2. Special issue in Meas. Sc. Tech. Vol. 9, 141-220, 1998
3. For particulate scattering database, please go and check www.wyatt.com
4. Comparison of the array detectors, www.oceanoptics.com, www.photon-vision.com