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Pump Probe Experiment for Optical Diagnosis of Very Thick Scattering Media

Scattering media with very high optical density are present in a great variety of application: clouds, paints, spray and biological cells. New optical diagnosis tools developments are very important in order to get in situ characterization without disturbing the sample. The goal of this paper is to describe a new method for determining the Particle Size Distribution (PSD) of a very thick scattering medium. This method is based on Optical Density (OD) measurements for different wavelengths, and a numerical inversion algorithm. The experimental challenge is to operate a light source that is both bright enough and tuneable in order to carry out such measurements. In addition, the scattered light has to be filtered. We will demonstrate that femtosecond spectroscopy overcomes all of these difficulties. A numerical inversion algorithm has been implemented in order to determine the PSD from an OD spectrum.

Introduction

The Optical Density spectrum (*OD*) can be obtained using very basic measurements. The idea is to measure the transmission of a given sample for different wavelengths λ. Optical density is defined by:

$$OD(\lambda) = \log_{10}\left(\frac{I_0}{I_t}\right) \quad (1)$$

where I_0 denotes the incident flux, and I_t the transmitted flux after the sample. Many commercial spectrophotometers can be readily found, and almost every optical laboratory possesses one. *OD* measurements can give a huge variety of information regarding the sample of interest: optical, physical and chemical properties. The goal of this paper is to focus on the Particle Size Distribution *PSD* determination of very thick scattering media. The size of the different particles of a scattering medium is a key factor [14,15]. When studying the optical behaviour of a cloud, it is the size of water droplets that governs the behaviour of the cloud. The size of the particles in an exhaust pipe just at the outlet of a combustion chamber will give relevant information about the efficiency of combustion. The composition of paints can be optimized if the size of the particles of a paint spread and dried in a coat is known. The list of all possible applications of (*PSD*) determination is quite long. In this paper, we study the model case of a suspension of homogenous spherical particles in a solvent. We define r as the radius of the particle and m the ratio between the refractive index of the particle and the refractive index of the solvent. This index ratio can be a complex number if we consider absorption processes. At the interface between the particle and the solvent, the change of refractive index will create reflection or refraction of the light. As a result, the interaction between light and particles will change the direction of the incoming beam light. This is the so called scattering process. The extinction cross section σ_{ext}, *i.e.* the probability that a photon will be scattered by a particle (or be absorbed) is calculated by Lorenz-Mie theory [10], and strongly depends on m, r and the wavelength λ. For a suspension of particles with an uniform radius r, the Beer-Lambert law can be written as:

$$I_t = I_0 \exp\left[-NL\sigma_{ext}(m,r,\lambda)\right]$$

where N is the number of particles per m^3 and L is the path length of the sample. In this simple case, $OD=NL\sigma/ln(10)$. Under real conditions, a distribution of particles with different radii has to be considered. The optical density can be expressed as a function of the normalized particle size distribution *PSD* or $f(r)$ in the following way:

$$OD(\lambda) = \frac{NL}{\ln 10}\int f(r).\sigma_{ext}(m,r,\lambda)dr \quad (2)$$

Equation (2) simply expresses the fact that the optical density is a linear combination of the different optical densities that would be obtained with a single radius r. **Our goal is to invert equation (2), in other words to determine *f(r)* as a function of the experimental data points *OD(λ)* assuming that we know σ_{ext}.** For many interesting applications, a typical *OD* can be greater than 5. In other words, the transmitted flux will be at least 5 orders of magnitude smaller than the incident flux. A photon will undergo a large number of scattering events while it travels through the sample. As a result, the scattered flux will be stronger (5 orders of magnitude stronger) than the transmitted flux.

Under such conditions, a measurement of angular distribution [4,8] of scattered light $I(\theta)$ could be more efficient. Equation (2) can be transposed for angular distribution as:

$$I(\theta) \propto \int f(r).\sigma_{scat}(m,r,\lambda)dr \qquad (3)$$

where σ_{scat} is the intensity scattered for various angles θ. Note, however, that this equation is limited by the assumption of single scattering event [10]. For thick media, we have to consider multiple convolution integrals, where the order of convolution is equal to the number of scattering events that one photon has undergone on its way through the sample. As a result, for a typical *OD* greater than 0.5, $f(r)$ cannot be numerically determined from a measurement of $I(\theta)$. Angular methods based on equation 3 are consequently limited to dilute media[1]. On the contrary, *OD* is based on the measurement of transmitted light, *i.e.* non-scattered light. As a result, it is possible to carry out measurements even if the assumption of a single scattering event is not true, and to characterize very dense scattering media. However, the scattered light is still an issue as it might be collected by the detector, and as mentioned earlier, the scattered flux can be greater than the transmitted flux. To get a proper determination of the *PSD*, three major difficulties must be overcome. First the light source has to be bright enough and tuneable. Then, we have to be able to get rid of the scattered light in the collection angle of the detector. Finally, we have to carry out the inversion of equation (2). We will show how femtosecond laser technology can solve the first two points. We used a numerical code developed in our laboratory in order to carry out the numerical inversion.

Light source requirements

We will first briefly discuss here how bright and spectrally large the light source should be in order to makeover accurate measurements. We have represented (Figure1) the evolution of σ_{ext} as a function of the radius of the particle for a given m and λ. For small particles $(mr/\lambda<<1)$, $\sigma_{ext}(m,r,\lambda)$ follows the Rayleigh regime and equation 3 can be rewritten as:

$$\sigma(m,r,\lambda) \propto \frac{r^6}{\lambda^4} \quad , \quad OD(\lambda) \propto \frac{\int f(r)r^6 dr}{\lambda^4}$$

This exhibits a clear redundancy of the spectral measurement: a single measurement for a given wavelength determines $\int f(r)r^6 dr$ *i.e.* all of the information we can obtain about $f(r)$, and this is not enough to determine the full *PSD*. For big particles $(mr/\lambda>>1)$, the expression of $\sigma(m,r,\lambda)$ falls into the geometric regime: $\sigma(m,r,\lambda)=2\pi r^2$. We can draw analogous conclusions as there is no more dependence with λ.Therefore, if we want to determine $f(r)$ accurately, the wavelength range has to match the radius range so that mr/λ is in the intermediate regime, also called the *Mie* regime.

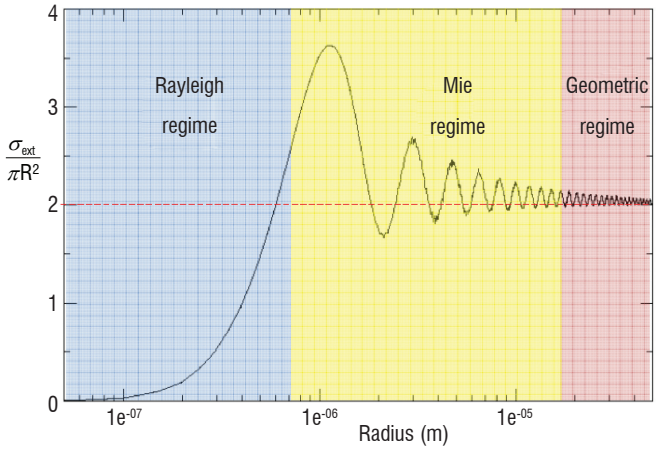

Figure 1 – Evolution of σ_{ext} with the radius of the particle. λ=400 *nm* and m=1.18. Note the different regimes: Rayleigh, Mie Geometric.

Note here that this condition not only depends on the ratio r/λ, but on the contrast of index: for a larger m, the limit between the Rayleigh regime and the Mie regime is shifted toward smaller radii. We used the inversion scheme [2] (described at the end of this article) to check the feasibility of the inversion for m=1.18 (particles of SiO_2 in water), and for a spectral measurement of 12 points between 400 nm and 700 nm. We were able to carry out an accurate inversion for *PSD* with a central radius between 100 nm and 5 μm (The *OD* spectra were calculated using the Mie theory). This shows that a huge spectral width is required, (400 nm - 700 nm typically), in order to carry out accurate inversion on a relevant radius range (100 nm - 5 μm). For larger or smaller radii, we have to consider broader light sources. Amplified femtosecond laser sources, coupled with Optical Parametric Amplifier technology (*OPA*) can deliver pulses from 200 nm up to 20 μm. Furthermore, the typical energy delivered per pulse is 100 μJ. With a detection limit of 30 photons, and a wavelength of 500 nm, such a system could measure an *OD* up to 13. For such level of *OD*, the amount of scattered light is huge so we have to implement a filtering device.

Filtering out the scattered light

We now have to study carefully the effect of the parasitic scattered light that might reach the detector. In order to accurately measure the *OD,* this light must be filtered.This is feasible by placing a lens after the scattering sample and a small pinhole at the focal point (Figure 2). The transmitted light maintains its initial propagation direction after the sample. As a result, it will go through the pinhole and reach the detector. Most of the scattered light will be stopped by the pinhole.

1 We mention here one possible way to carry out a *PSD* determination based on an angular method in the multiple scattering regime. A succession of scattering events will strongly affect the Degree Of Polarization (*DOP*) of the light. We could measure $I(\theta)$ and $DOP(\theta)$ simultaneously. However, the relationship between these two observables and the *PSD* is not direct as there is no simple analytic expression. A numerical optimization scheme is required. This method was implemented successfully in our laboratory and *PSD* with *OD* up to 5 were determined [18].

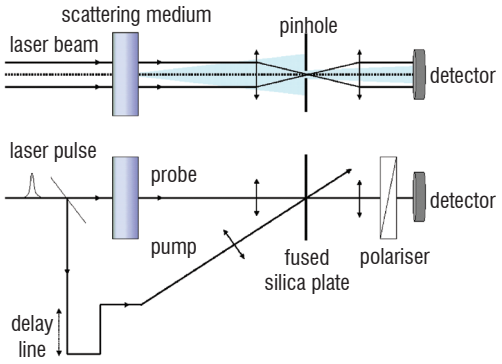

Figure 2 – Comparison of classical spatial filtering (top part), and femtosecond spatial filtering (bottom part). Most of the scattered light is stopped by the pinhole. The pump beam drills a virtual pinhole in the fused silica plate.

The principle of "femtosecond spatial filtering" is to use femtosecond laser pulses, and to generate optical Kerr gating in a fused silica plate [12]. Each laser pulse is split in two parts: the probe pulse and the pump pulse. Thanks to a delay line, we can fine-tune the time delay between the 2 pulses. The probe pulse goes through the sample in order to measure the *OD*, and then, both pump pulse and probe pulse are focused into the fused silica plate. The pump pulse instantaneously induces a birefringence in the plate (which will only last during the pump pulse), which in turn induces a rotation of the polarization of the probe beam. With a crossed polarizer placed in front of the detector, **the light can reach the detector only if the two beams perfectly overlap spatially and temporally.** Let's consider first the spatial overlap. This is strictly speaking the equivalent of a virtual pinhole: all of the probe light that is not overlapping the pump light will be stopped by the analyzer. The advantages of such of virtual pinhole compared to a classical one are huge. First, the size of such a pinhole can be fine tuned by changing the focus of the pump beam. Then, the overlap can be optimized at any space time by optimizing the Kerr effect signal. Let's now consider the temporal overlap. The ballistic light (transmitted light) will go straight through the sample, it will arrive first (Figure 3). The scattered light will undergo a more complex pathway, it will arrive later. As a result, when we fine tune the delay between the pump pulse and the probe pulse, the "temporal gate" will first sample the ballistic light, and then the scattered light. The experiment is repeated for several delays between the two pulses in order to obtain the temporal profile of the scattered light. Then we can integrate temporally the light around the ballistic "zero delay" and realize a temporal filtering of the scattered light.

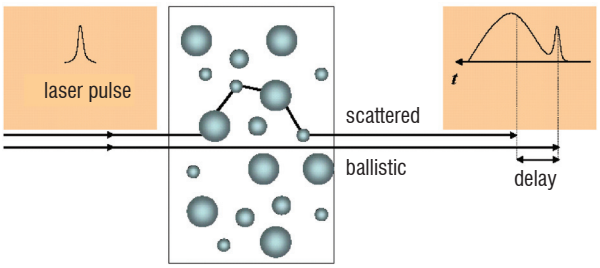

Figure 3 – Temporal filtering: different path way for the ballistic light and the scattered light.

In order to evaluate the relative efficiency of the temporal and spatial filtering **we developed a temporal Monte Carlo scheme [1,17]**. We sent particles of light (that we will call photons) randomly through the sample by computer simulation. Knowing the spatial size of the laser beam and the duration of the pulse, we randomly picked an initial position and an initial time for the photon. Then we calculated the different probability laws (distance between two scattering events, angle, time spent in the particle) in order to randomly build a pathway for the photon through the sample, toward the detector, keeping track of the time of flight of the photon. We carefully reproduced the geometry of figure 2, defining the position of the iris and the lens, in order to evaluate precisely the relative weight of the transmitted light and scattered light that goes through the iris and reaches the detector. We used a pinhole of 30 μm diameter and reasonable values for the other geometrical parameters. We considered that the spatial filtering was efficient if the scattered flux was smaller than or equal to the transmitted flux (error of 2 on I_t *i.e.* 0.3 on the *OD*). We observed that **the spatial filtering is efficient for a typical *OD* up to 7**. We also simulated the efficiency of the temporal filtering integrating the weight of the photon around the ballistic "zero delay" and outside of this temporal window. This additional **temporal filtering is efficient for a typical *OD* up to 11.** We observed that both spatial and temporal filtering are much more efficient for small particles than for big particles (typical values given earlier were estimated for 1 μm radius SiO_2 particle in water, L=1 cm, λ =500 nm). This is mainly due to the fact that big particles mainly scatter light in the forward direction. This will add almost no delay and almost no deviation to the photon.

Experimental illustration

We carried out a preliminary experiment using a 250 μJ, 1 kHz, 800 nm amplified laser source (Amplitude Technology, *LCAR/UPS*). The pump pulse was generated using a small part of the laser energy (10 μJ). The remainder was used to seed a Non collinear Optical Parametric Amplifier *(NOPA)* [3,6,11,13] tuneable from 490 nm to 700 nm. The spectral bandwidth of the *NOPA* output was carefully reduced and controlled for every wavelength. An additional measurement at 400 nm was obtained with the frequency doubled output of the laser. We carried out measurements on a calibrated suspension of SiO_2 particles in water. An average *OD* of 2 was small enough to be filtered spatially. Nevertheless, we carefully checked the temporal profile of the Kerr signal obtained after the sample and after a reference sample of water. No delayed scattered light was observed. In both cases, we measured a cross correlation of 200 fs. Typical results are shown (Figure 4). The black squares represent the *OD* obtained at different wavelengths. Experimental results are the averaging of ten measurements. The error bars are given by the root mean square of the distribution, mainly due to the laser instability. Error bars and average values were input into the inversion scheme outlined at the end of this article. The result of the inversion is represented (Figure 5) with red squares. The *PSD* result was then used to calculate an optimized *OD* spectrum using equation 2, plotted (Figure 4) in red. The match between the measured and optimized *OD* spectrum is excellent. The *PSD* result is compared with other results based on Polarization Induced Differential Scattering measurements (*PIDS* black circles (Figure 5)). As far as the radial average is concerned, both measurements are in good agreement with the specification of the SiO_2 particles we used to realize the experiment: 0.1 μm. The discrepancy regarding the distribution width is probably due to the fact that we are at the border of the Mie regime which is a problem for both methods (*OD* and *PIDS*). These are the first preliminary experimental results.

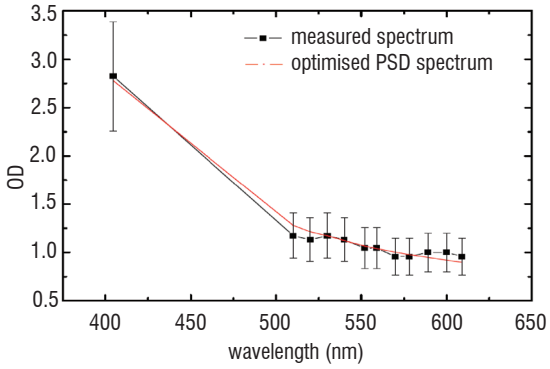

Figure 4 – *OD* spectra of a suspension of SiO_2 particles in water (0.1 μm central radius).

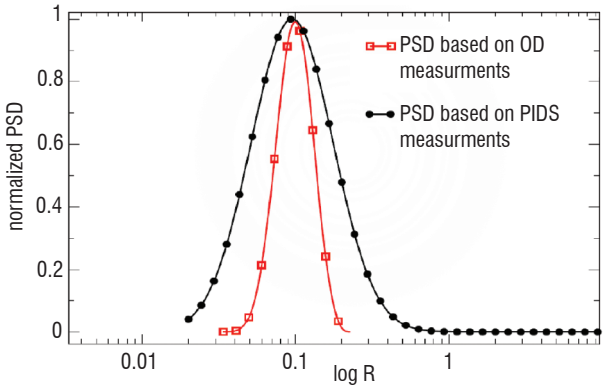

Figure 5 – Comparison of the inverted spectra with results based on a Polarisation Induced Differential Scattering measurement (*PIDS*). The two measurements were taken on the same sample.

Conclusion

We have demonstrated that femtosecond laser sources are very efficient for taking *OD* measurements of very dense scattering media. Such sources are bright and tuneable. Furthermore, an accurate filtering of scattered light can be implemented. We have carried out a preliminary experiment and have successfully retrieved the *PSD* of a model sample. Further experiments will be implemented using broader and brighter femtosecond laser sources and different samples ■

Box 1 - Instability of the Fredholm integral equation of the first kind. One possible resolution

Numerical instability

Equation (2) and equation (3) are called Fredholm integral equations of the first kind [9]:

$$g(y) = \int_{x_{\min}}^{x_{\max}} f(x).K(x,y)dx$$

Our goal is to invert this equation, in other words to determine $f(x)$ as a function of experimental data points $g(y)$ assuming that we know $K(x,y)$ (equation 2 and 3 are technically written with R_{min} and R_{max}, the physical limit for the determination of the particle size). This problem is ill conditioned. Let's draw some comparisons between this mathematical problem and the non linear differential equation. We assume that $x(t)$ is a solution of a non linear differential equation, with the initial condition $x(0)=x_0$ and $y(t)$ solution for $y(0)=y_0$.y_0 and x_0 can be very close and $x(t)$ and $y(t)$ can be very different. This is a very famous effect, known as the "butterfly effect". We will demonstrate here that a Fredholm equation leads to the same effect: a tiny little difference on the experimental data point g (initial condition) can have dramatic consequences on the solution f. This is due to the nature of the integral operator of the right hand side of this equation: given the Lesbegue theorem,

$$\forall y, \lim_{n\to\infty} \int_{x_{\min}}^{x_{\max}} \sin(nx).K(x,y)dx = 0$$

(as far as K is a continuous function, which is true for our application). As a result, $f(x)$ and $f(x)+A\sin(nx)$ will both be good solutions for the experimental data set $g(y)$ since the integration of the oscillating term can be arbitrarily small, as long as n is big enough. We have shown that there are two similar data sets, corresponding to two very distinct solutions. Fast oscillations of $f(x)$ will have very little impact on $g(y)$. **The only way to correctly solve this equation is to find a way to constrain the solution not to oscillate too fast.** Furthermore, some experimental data $g(y)$ may have no solution $f(x)$. We can differentiate the Fredholm equation with respect to y. The right hand side will be very regular as $K(x,y)$ is regular. As a result, $g(y)$ can have a solution only if it is regular. This might not be true as we consider experimental data with potential discontinuities and angular points. As a result, **we have to consider the experimental data $g(y)$, and a confidence interval** and assume that within this interval, we can find a regular curve $g(y)$ that will admit a solution. There is one more issue to consider: for our particular application, $f(x)$ is strictly positive (Particle size distribution $f(r)$). **The solution has to be constrained so that it always remains positive.** We have developed a numerical process in order to address all of these issues [5]. This method was initially developed to invert the data of angular single scattering experiments [4] (equation 3). The transposition to the spectral domain (equation 2) was straightforward [2]. This method is one inversion method among many others [16]. Let's describe briefly the general idea of our method.

One possible solution

The first step is to enter the result of the N measurements $g_i=g(y_i)$. For each y_i, $(i=1,N)$, the experiment has been done several times so we have a distribution of experimental values. For each parameter y_i, we enter the average value g_i and a confidence interval Δg_i. N measurements could in the ideal case lead to the determination of N independent parameters. We logically try to find a first solution $f_0(r)$ as a linear combination of N base functions $S_j(r)$:

$$f_0(x) = \sum_{j=1}^{N} C_j S_j(x)$$

S_j can be a step function, defined on knot points x_j: inside the interval $[x_j , x_{j+\mu}]$ S_j is equal to 1 and is 0 outside. In order to get a more accurate inversion, we use *cubic splines* for the base functions S_j as it will lead to a more regular solution, but the resolution is strictly equivalent to step functions. The choice of the grid x_j is critical as it directly governs how fast the solution can oscillate. In order to determine the first solution f_0, the decomposition is input in the Fredholm equation:

$$\forall i g_i = \sum_{j=1}^{N} K_{ij} C_j \quad \text{with} \quad K_{ij} = \int_{x_j}^{x_{j+1}} S_j(x) K(x,y) dx$$

This is a square linear system, the inversion is straightforward, and we determine the vector (C_j) and the solution f_0. This basic result is not accurate for three reasons. First, the grid (x_j) is not optimized. If it is too fine, the solution is allowed to oscillate too fast and the numerical system is not stable. Then, we have tried to extract N independent parameters (C_j) from N distinct measurements. This is not appropriate because the N measurements might be redundant (for example, if the *PSD* $f(r)$ is larger than the Mie regime). Finally, f_0 can be negative. To tackle these points, the second step of our method consists of a numerical regularization based on the Mellin transform formalism [9]. We are able to define a set of eigen functions of the operator $f \rightarrow \int fK$:

$$f_\omega(x) = \frac{\cos(\omega \ln(x))}{\sqrt{x}}$$

Using the Lesbegue theorem, these eigen values will decrease very strongly with ω. The idea is to decompose the coarse solution f_0 we obtained previously on the f_ω bases. Then, we only consider the lower frequencies ($\omega<\omega_{cut}$) to calculate the correspondent $g_i(\omega_{cut})$. When we realize:

$$\forall i, \left| g_i(\omega_\infty) - g_i(\omega_{cut}) \right| < \Delta g_i$$

we obtain the maximum frequency ω_{cut} to consider. In other words, if we allow the solution f to oscillate faster than ω_{cut}, this will correspond to a precision requirement on g_i greater than Δg_i. To avoid this situation, the step of the grid $ln(x_j)$-$ln(x_{j-1})$ will be equal to π/ω_{cut}. Decreasing ω_{cut} will increase the step of the grid. As x_{min} and x_{max} are fixed, this will in turn decrease the number of base functions S_j in the linear decomposition. The linear system is now a rectangular system and the algorithm will perform the inversion using a standard *Non Negative Least Squared Method* proposed by Lawson and Hanson [7]. This will constrain the solution to remain positive. Every time we find a solution, we calculate the corresponding g_i, and check that it is within the confidence interval. This method has been implemented, checked and used successfully to solve many different problems, in the angular situation (equation 3) [5], and in the spectral situation (equation 2) [2].

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Acronyms
DOP (Degree Of Polarization)
NOPA (Non collinear Optical Parametric Amplifier)
OP (Optical Density)
OPA (Optical Parametric Amplifier)
PIDS (Polarization Induced Differential Scattering)
PSD (Particle size Distribution)

AUTHORS

Marie Barthelemy graduated in fundamental physics from the Université Paul Sabatier, Toulouse, France. She's particularly interested in laser matter interaction and potential applications on biological systems. For this reason, she chose to study for a Ph.D. at Onera on high scattering media using ultrafast optical diagnosis. She received her PhD degree on February 2009. She has joined the Femto team in the Laboratoire Collisions Agregats Réactivité to work for a year on UV ultrashort pulse shaping.

Laurent Hespel received his Ph.D. degree in physics from the Ecole Centrale de Paris, Châtenay-Malabry, France in 1999. The subject was "Theoretical and experimental studies of radiative transfer in scattering media. Experimental determination of radiative properties under high charge rate conditions". He studied for it in the group led by J. Greffet. He then joined the Onera (the French Aerospace Lab), first as a post doctoral fellow for a year and then as a research scientist. He is currently at the head of a research group named "Light interaction with Matter, Imaging and detection Laser Systems unit." His research group works on a large variety of areas of application: development of new laser imaging systems, Lidar, radiative transfer modelling, simulation of speckle, optical diagnosis of scattering media…

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Thibault Dartigalongue graduated from Ecole Polytechnique in 2001. He received his Ph.D. degree in physics from Ecole Polytechnique Palaiseau, France in 2005. The subject was "Conformational dynamic of myoglobin followed by time resolved circular dichroism". He studied for it in the group led by F. Hache in Laboratoire d'Optique et Bioscience. He then joined Dwayne Miller's group at the University of Toronto as a post doctoral fellow for two years. He worked on ultrafast electron diffraction, studying the thermal dynamics of gold and silicon. He is currently a Research scientist at Onera (French aerospace lab). He is involved in the development of a femtosecond laser laboratory devoted to optical diagnosis of very thick scattering media.