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Interaction of ultrashort pulses with molecules and solids: Physics and applications

S VENUGOPAL RAO

Advanced Centre of Research in High Energy Materials (ACRHEM), University of Hyderabad, Gachibowli, Hyderabad 500 046, India E-mail: svrsp@uohyd.ernet.in; soma_venu@yahoo.com

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Abstract. The interaction of ultrashort laser pulses with molecules and solids is an extremely complex area of science research encompassing the fields of physics, chemistry, and materials science. The physics of interaction has been fairly understood over the last couple of decades and, consequently, several applications have been envisaged from these interactions in the fields of photonics, lithography, biomedicine, sensing, telecommunications etc. In the present article we describe three different components of interaction of ultrashort pulses with matter: (1) with liquid molecules/thin films wherein we present the results from our studies of optical nonlinearities predominantly using picosecond and femtosecond pulses, (2) with molecules/solids wherein plasma generated from the surface was studied for applications in understanding the molecular dynamics and towards identifying high-energy molecules and (3) within the bulk and on the surface of solids (e.g. glasses, bulk polymers and metals) resulting in micro- and nanostructures. Different applications resulting from such interactions in photonics and microfluidics are presented and discussed.

Keywords. Femtosecond; nonlinear optical; ablation; laser direct writing; high-energy materials.

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1. Introduction

State-of-the-art ultrafast laser oscillators [1] and amplifiers [2,3] with extremely high peak powers and compact table-top foot prints have enabled several opportunities in the research field of laser-matter interaction. The various major factors influencing laser-matter interaction [4–8] are: (a) pulse duration [picosecond (ps), femtosecond (fs)]: the energy coupling to the lattice is governed by this, (b) input energy/peak intensity: the kinds of nonlinear optical processes occurring depend on this and (c) wavelength of excitation, while some minor factors include pulse repetition rate, pulse shape, pulse chirp

etc. Some of the intense field (peak intensities $\gg 10^{16}$ W/cm²) phenomena [5] cannot be accomplished with the present generation ns laser pulses. The implications of such interactions are unprecedentedly wide-ranging including (a) high harmonic generation, (b) laboratory astrophysics, (c) creation of hard X-ray sources, (d) acceleration of elementary particles, (e) cancer therapy using high-energy proton, neutron beams etc. while with laser pulses possessing intensities in the range of 10^{12} – 10^{16} W/cm² the applications envisioned include (a) novel nanostructures in a variety of materials, (b) control of chemical reactions, (c) surgeries using fs pulses, (d) high-power terahertz generation, (e) biomedical imaging, (f) guided fs pulses in the atmosphere for environmental applications etc. amongst many others. Several recent review articles have discussed and highlighted the physics and applications of such interactions [4–12]. Our research group has been working on laser–matter interaction using ps and fs pulses for the last six years and herein we present some of our recent results from various experimental studies.

Based on our recent works, the interaction has been classified into three different categories. First, the interaction of short laser pulses [nanosecond (ns), ps, fs] with novel molecules (mostly in liquid form) enabled us to evaluate and quantify their third-order nonlinear optical (NLO) properties, figures-of-merit and, consequently, identify their potential for various photonic applications. NLO studies at non-resonant wavelengths and with ps/fs pulses enable us to recognize the pure electronic contribution to the nonlinearity for identifying prospective molecules for device applications. The usage of longer pulses invokes rotational, thermal and other slower nonlinearities. Novel moieties with large non-resonant nonlinearities, strong two-photon (2PA)/three-photon absorption (3PA) cross-sections/coefficients are attractive for potential applications in the fields of photonics, all-optical signal processing, lithography and biomedicine. A newly designed molecule could be useful for optical limiting applications when studied with ns/ps pulses while its effectiveness for all-optical signal processing or all-optical switching is decided by the fs nonlinear refractive index (n_2) . A molecule, depending on the structure and composition, will possess interesting NLO properties at different wavelengths and input intensities. Therefore, for identifying the complete potential of any of the molecule, studies at different input conditions (wavelength, pulse duration, input energy/peak intensity, surrounding matrix etc.) are necessary. Through our recent efforts we could also evaluate the ultrafast time-response of the optical nonlinearity. Molecules such as phthalocyanines, corroles and porphycenes were studied in solution form while some of them were investigated in thin film form [13-25]. Different techniques of ps/fs Z-scan, fs degenerate four-wave mixing etc. were used.

Secondly, we utilized short pulses to create optical breakdown on materials surface, especially solid targets (pellets made from powders), to study light emission using the technique of laser-induced breakdown spectroscopy (LIBS) to devise mechanisms towards identifying/classifying high-energy materials (e.g. RDX and HMX) [26–31] and to understand the molecular dynamics formation. In this case the pulses were focussed on the surface and the sample was scanned so as to expose a new region of the same each time the pulse hits the surface. Several reviews have identified the mechanisms and resulting applications in ns LIBS [32–34] and fs LIBS experiments [35,36]. We believe that LIBS studies with ultrafast pulses open up new avenues for understanding the materials in an improved approach.

242

Thirdly, ps/fs laser pulses were used to:

- (a) Examine the modifications in transparent dielectrics (e.g. glasses and polymers), inside and on the surface of bulk, when exposed to tightly focussed fs pulses. Laser direct writing (LDW) technique [37-43] is ubiquitous due to the relative ease in fabricating microstructures and nanostructures along with the possibility of integration for realizing complex 3D photonic/microfluidic/optofluidic devices. Our group has recently been successful in utilizing this technique for creating structures in different materials which find applications in a variety of fields [44-51]. Peak intensity of the focussed fs pulses determines the type of modification in any material: (i) an increase in refractive index *n* (type I), (ii) a decrease of *n* (type II) and (iii) formation of microvoids. There is also the possibility of creating truly three-dimensional and cm-long microchannels on the surface of any material and inside the bulk (possible in glasses too with selective etching)
- (b) Create micro-/nanostructures [52–55] on the surfaces of metals (Al, Cu, Ag) and nanoparticles with ablation being performed by placing targets in various liquids. The optical nonlinearities of the nanoparticles were evaluated using ~2 ps pulses. The resulting nanostructures find imminent applications in the field of trace explosives detection using surface enhanced Raman scattering (SERS) technique. The nanocolloids of metals (Ag, Au) also find antibacterial applications.

2. Experimental details

For NLO studies we utilized $\sim 2 \text{ ps}/\sim 40$ fs pulses from two separate Ti:sapphire amplifiers (Coherent) delivering nearly transform limited pulses at 800 nm with 1 kHz repetition rate. The maximum energy was $\sim 2/2.5$ mJ in the case of ps/fs pulses. For some of the NLO studies we utilized ~ 100 fs pulses from a Ti:sapphire amplifier (Spectra Physics) delivering ~ 1 mJ pulses at 800 nm with 1 kHz repetition rate. We also used a nanosecond (ns) laser at 532 nm delivering ~ 6 ns pulses with a few hundred mJ of energy for some specific NLO studies.

For LIBS studies we utilized both ns and fs pulses. The source for ns pulses was second harmonic of a Nd:YAG laser at 532 nm delivering \sim 7 ns pulses at 10 Hz repetition rate. An input beam with \sim 6.5 mm spot size and typical energies of 10–50 mJ/pulse was focussed using an 80 mm lens on the sample. The samples were placed on a computer controlled translation stage to ensure that a fresh portion was exposed to the laser pulses each time. The light was collected using an ICCD and a spectrometer (ANDOR) combination. All the initial experiments were performed in ambient atmosphere.

For LDW studies we utilized a Ti:sapphire amplifier (Spectra Physics) delivering 1 mJ pulses at 800 nm with 1 kHz repetition rate delivering near-transform limited ~100 fs pulses. Typically, $40 \times$ and $20 \times$ microscopic objectives were used for focussing the laser pulses and the samples were placed on an X–Y translation stage (Newport) to control the number of pulses incident on the sample. A combination of half-wave plate and polarizer was used to control the input energy from a few hundreds of nJ to a few tens of μ J. For ablation studies we again used ~2 ps/~40 fs pulses from two separate Ti:sapphire amplifiers as discussed earlier in this section. The targets were placed in liquids (placed in a petri dish) and the whole set-up was placed on X–Y translation stage. The movement

of the stage was controlled such that several cases of ablation were achieved: (a) single line ablation, (b) double line ablation and (c) multiple line ablation. In each case a large number of pulses were interacting with the same area on target.

3. Results and discussion

Typical results obtained in our group from each of these three interactions are summarized. Details of the NLO experiments can be found in our earlier publications [9–24]. Typical data for two phthalocyanines CuPc [2(3), 9(10), 16(17), 23(24)-sodium salt of tetrasulphonic acid copper phthalocyanine] and NiPc [2(3), 9(10), 16(17), 23(24)-sodium salt of tetrasulphonicacid nickel phthalocyanine] are presented here [23]. The samples were purchased from Aldrich and used as is. Neat solutions of CuPc and NiPc were dissolved in de-ionized water as solvent and typical concentrations of $(0.5-1)\times10^{-4}$ M were used. Figures 1a and 1b show representative, normalized open-aperture scans of CuPc and NIPc recorded with 532 nm, 6 ns pulses. We observed reverse saturation absorption (RSA) in these molecules for ~150–600 MW/cm² input intensities. For intensities >10⁸ W/cm², the normalized transmittance in CuPc, especially, dropped to



Figure 1. Ns open-aperture Z-scan data (open symbols) along with the fits (solid lines) for (a) CuPc with a peak intensity of 2×10^8 W/cm² and (b) NiPc with a peak intensity of 30×10^8 W/cm². Ps open-aperture Z-scan data (open symbols) along with the fits (solid lines) for (c) CuPc and (d) NiPc. Insets depict the closed-aperture data.

<0.6 indicating a strong nonlinear absorption behaviour. It is well understood and established that nonlinear absorption in such materials due to ns pulses has contribution from both excited singlet and/or triplet states due to two-photon absorption (2PA) depending on the excitation wavelength. However, for 532 nm excitation, we can approximate the nonlinear absorption to an effective process and evaluate the nonlinear absorption coefficient, β . The values of nonlinear absorption coefficient β_{eff} estimated from the theoretical fits were ~ 25 cm/GW for CuPc and ~ 1.1 cm/GW for NiPc. Figures 1c and 1d illustrate open-aperture data for NiPc and CuPc, respectively, recorded at 800 nm using ~ 2 ps pulses with peak intensities of $\sim 160 \text{ GW/cm}^2$. We again observed strong reverse saturation (RSA) kind of behaviour in this intensity range. The obtained experimental data were fitted using standard equations and the best fit was obtained with the transmission equation for three-photon absorption (3PA). The values of α_3 were estimated to be $\sim 15 \times 10^{-5}$ cm³/GW² for CuPc and $\sim 14 \times 10^{-5}$ cm³/GW² for NiPc. Insets of figure 1 show the closed-aperture scans. Depending on the pulse duration, one could obtain strong 2PA [19,21] or 3PA [20] as was observed by our group in different phthalocyanines. Different phthalocyanines were investigated and most of them had impressive figures-ofmerit. Porphycenes exhibited both 2PA and 3PA at the same wavelength of 800 nm due to their unique energy level structure [13,14]. The NLO coefficients and cross-sections evaluated in the ps (wavelengths other than 800 nm were also studied) and fs regime were large compared to some of the recently reported successful molecules with similar structure. The lifetimes of the excited state of porphycenes studied using fs and ps pumpprobe techniques were estimated to be in the sub-ps regime [24]. Four different Corrole molecules (TPC, TTC, GeTTC, PTTC) were also studied using Z-scan technique using ps and fs pulses. Once more, the NLO coefficients were evaluated along with the time response of these molecules using the pump-probe technique [25]. Fs nonlinear absorption data of TPC and TTC demonstrated the behaviour of saturable absorption (SA). Ps Z-scan data suggested a switching of SA to RSA at higher peak intensities for both TPC and TTC. Similar behaviour was observed for GeTTC and PTTC. The lifetimes of excited states estimated from pump-probe data were in the sub-picosecond regime. The summary of our detailed studies on phthalocyanines is: (a) unsymmetrical phthalocyanines (with Zn metal in the core) had better NLO properties compared to symmetrical counterparts, (b) alkoxyphthalocyanines had enhanced NLO coefficients compared to alkyl phthalocyanines studied with ns and fs pulses, (c) thin films exhibited superior NLO coefficients which was concluded from our ps studies, (d) nanoparticles demonstrated improved NLO properties compared to bulk phthalocyanines in the ns and fs domains, (e) the NLO performance of metallic phthalocyanines was superior to free-base phthalocyanines. The immediate requirement for translating the research success (at the molecular level) of designing molecules with strong NLO coefficients, to devices is in exploring the detailed NLO properties with (a) ns, ps, fs pulses simultaneously along with (b) dispersion studies (studies performed over a wide range of wavelengths in the visible and near-IR spectral region, where the actual devices might be employed if successfully created) and (c) molecules in practical/processable forms (doped in films, LB films, sol-gels, glasses etc.).

Complete details of the ns and fs LIBS experiments are reported elsewhere [27–31]. LIBS studies with fs pulses were proposed in recent years by many research groups for identification purposes such as analysis of animal tissues, explosives, biological materials/bacteria, pharmaceuticals etc. [30,31]. Some of the advantages envisioned



Figure 2. (a) LIBS spectra of HMX using fs pulses with ICCD gate delay of 100 ns and gate width of 300 ns, (b) LIBS spectra of bare Al substrate using fs pulses with ICCD gate delay of 200 ns and gate width of 50 ns.

with fs LIBS include (a) low ablation threshold, (b) less thermal damage to the sample, (c) nearly background-free spectra, devoid of continuum and (d) possibility of creating filamentation over a period of few kilometres for implementing remote LIBS [35,36]. Furthermore, numerous advances in the field of ultrashort lasers enable the exploitation of several distinctive features such as (a) compactness, portability along with (b) cost-effectiveness, (c) tunability, high-energy outputs, (d) possibility of shaping the spectral content, (e) remote experiments etc. [35,36]. Initial studies were performed using non-gated and gated mechanisms on simple high-energy materials such as ammonium perchlorate (AP), ammonium nitrate (AN) and boron potassium nitrate (BPN) to evaluate the efficacy in each case [26]. Figure 2a shows a typical LIBS spectrum of RDX (1,3,5trinitroperhydro-1,3,5-triazine). Figure 2b shows the fs LIBS spectrum of bare aluminum substrate. Our ns/fs LIBS studies on RDX, HMX and NTO obtained at similar fluences (despite ns studies being performed at 532 nm and fs studies at 800 nm) demonstrated that certain features in the fs LIBS spectra were different from the ns LIBS spectra, especially the CN/C peak ratio [27]. The persistence of emission in the case of CN and C was also evaluated in both the cases and the values obtained were different. Our efforts are also on to use simple ratiometric techniques to identify possible methodologies for the identification/discrimination of explosive and related molecules. Our future works will focus on evaluating the effects of (a) the surrounding atmosphere, (b) the number of nitro-groups in molecules of interest and (c) pulse shaping effects on the LIBS data. We strongly believe that the laser-matter interaction will be different in ns and fs cases and one need to identify correct pointers for utilizing them in appropriate applications. An important and immediate requisite is the development of a complete chemical/physical kinetic model describing the complex plasma due to the interaction of ultrafast laser pulses with matter. It will also be fruitful in understanding the persistence of emission of various atomic (C, N, O, H) and molecular (CN, C₂) species in both ns and fs LIBS experiments.

Fs laser direct writing (LDW) technique was successfully employed for creating micro-/ nanostructures (both on the surface and within the bulk and in thin films too) in several transparent polymers such as polymethyl methacrylate (PMMA), polydimethylsiloxane (PDMS), polystyrene (PS) and polyvinyl alcohol (PVA) [44–49]. Figure 3a depicts FESEM image of typical holes fabricated in PDMS with $40 \times$ microscopic objective while figure 3b shows a fabricated large-area grating within the bulk of GE124 glass.



Figure 3. (a) Surface holes on PDMS achieved with $40 \times$ objective and different input energies, (b) large area grating in a GE124 glass and (c) surface structures in PMMA with different input energies.

Figure 3c illustrates several microstructures fabricated on the surface of the bulk PMMA (1 mm/s speed) with energies varying from $\sim 10 \ \mu$ J to $\sim 50 \ \mu$ J. Depending on (a) the amount input energy used, (b) focussing conditions $(20 \times \text{ or } 40 \times)$ and (c) scanning speeds we could achieve different microstructures and nanostructures. Such surface and subsurface structures find straight applications in microfluidics and for those fabricated within the bulk have potential in the fields of photonics [34]. Using ~ 100 fs pulses and $40 \times /20 \times$ focal conditions we have been successful in fabricating (a) surface microstructures (e.g. holes, lines, channels, gratings, splitters) and buried microstructures (channels, gratings, Y channels etc.) in bulk of PMMA [45], (b) surface and buried microstructures in other polymers such as PS, PDMS, PVA etc. [46–48], (c) gratings, waveguides, buried sub-micron structures in high quantum efficiency Baccarat glass [51], (d) twodimensional gratings and other structures in FOTURAN glass, GE 124 glass etc. [50]. Fs LDW has evolved a great deal in the last decade with several incredible applications [56], for instance (a) welding of two dissimilar glasses [57], (b) creation of Bragg gratings and other photonic components in fibres, (c) creation of nanoaquarium for dynamic observation of living cells [58], (d) fabrication of lab-on-a-chip devices [38,39], (f) manufacturing superhydrophobic or superhydrophilic surfaces, (g) fabrication of splitters, couplers, photonic lattices, Mach–Zehnder interferometers, arbitrary shaped structures,



Figure 4. FESEM images of laser-exposed portions of Ag substrate in (a) acetone, [(b) is the magnified view], (c) DCM, [(d) is the magnified view] depicting different morphologies. The energy used was $\sim 200 \ \mu$ J. The scale bar is 2 μ m in (a) and (c) and 200 nm in (b) and (d).

(h) non-reciprocal ultrafast laser writing [59] to name a few. Very recently, integrated multimode interferometers with arbitrary designs were successfully fabricated by means of fs LDW for photonic boson sampling [60]. Crespi *et al* implemented boson sampling problem by studying 3-photon interference in a five-mode integrated interferometer [60] and their studies have confirmed the quantum mechanical predictions. Another exotic example of such interactions that has recently been used to predict new forms of matter successfully was reported by Vailionis *et al* [61]. They reported the synthesis of a superdense stable phase of body-centred-cubic aluminum using ultrashort laser pulses and creating extreme conditions within the sample (pressures >100 GPa and temperatures >10⁴ K).

Laser ablation in liquids using ultrashort (ps/fs) laser pulses is a promising technique for generating nanoparticles (NPs) and nanostructures (NSs) in a single experiment in plasmonic and other metals of interest [52–54]. Figures 4a, 4c present the FESEM images depicting the morphology of silver (Ag) substrate ablated in acetone and dichloromethane (DCM), respectively. Figures 4b and 4d illustrate the magnified view of the images in 4a and 4c, respectively. It is evident that the nanostructures formed on the Ag target could possibly be used for surface enhanced Raman scattering (SERS) studies towards the detection of trace quantities of any analytes. When the Ag target was ablated in liquids of chloroform (CHCl₃) and carbon tetrachloride (CCl₄), different morphologies were obtained. The FESEM images of ablated Ag targets are presented in figures 5a



Figure 5. FESEM images of laser-exposed portions of Ag substrate in (a) chloroform, [(b) is the magnified view], (c) carbon tetrachloride, [(d) is the magnified view] depicting different morphologies. The energy used was $\sim 200 \ \mu$ J. The scale bar is 2 μ m in (a) and (c) and 200 nm in (b) and (d).

and 5c for CHCl₃ and CCL₄, respectively. Figures 5b and 5d demonstrate the magnified view of the images in figures 5a and 5c, respectively. We had, indeed, utilized these nanostructures on Ag targets for SERS studies of an explosive molecule, CL-20 (hexanitrohexaazaisowurtzitane, $C_6H_6N_{12}O_{12}$). Figures 6a–6d depict the SERS spectra of CL-20 molecules (mM/ μ M concentration) adsorbed on these targets. Typical enhancement factors obtained from these studies were $\sim 10^3 - 10^4$. There is scope for further enhancements through the fine tuning of the nanostructures obtained. Our aim is to utilize the plasmonic nanostructures of Ag, Cu, and alloys prepared by ultrafast laser pulses for detection of all the common explosives using the technique of SERS. The NPs obtained from such studies can be utilized for various applications including photonics, optical limiting, bioimaging, antibacterial agents etc. This technique is capable of achieving NPs and NSs in a variety of potential semiconductor materials such as silicon, germanium, GaAs, etc. and non-plasmonic metals such as titanium, platinum etc. The formation dynamics of NPs and NSs using fs and ps pulses is still being investigated thoroughly. However, several experimental reports have demonstrated achievements of fair control over the size and morphology of NPs and NSs created and their potential applications.



Figure 6. Surface enhanced Raman spectra of CL-20 from the Ag substrates fabricated in (a) acetone, (b) DCM, (c) CHCl₃ and (d) CCl₄. Excitation wavelength was 532 nm and time of integration was 5 s.

Pramana – J. Phys., Vol. 83, No. 2, August 2014

250

4. Conclusions

Three ultrashort laser–matter interaction mechanisms have been discussed and some results are presented. Fs and ps pulses allowed us to evaluate the fast nonlinearities of several molecules. Strong 3PA along with fast response times of the nonlinearity was observed in some of the molecules and they find interesting applications in photonics. Using LDW technique we were able to accomplish several photonic/microfluidic structures in polymers and glasses. LIBS studies using ns and fs pulses enabled us to understand some of the plasma dynamics and our initial ratiometric data suggested the expediency of materials (explosives) classification. Ps and fs ablation studies facilitated us to create NPs in liquids and NSs on substrate. The obtained nanoparticles demonstrated strong nonlinear optical properties and NSs were utilized for the detection of explosives using SERS methods.

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