

Single crystal fibers growth

Lasers Development
Lasers Applications
High Intensity Lasers

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### Introduction

The Lasers Technology Program of IPEN is committed to the development of new lasers based on the research of optical materials and new technologies, as well to laser applications in several areas: Nuclear, Medicine, Dentistry, Industry, Environment and Advanced Research. Additional goals of the Program are human resource development and innovation, in association with Brazilian Universities and commercial partners. The Program is basically divided into two main areas. "Material and Laser Development", includes crystal growth of optical materials (laser crystals); characterization, modeling and optical spectroscopy of solids, plasmas and biological materials; development of compact diode pumped-solid state lasers and the development of a high power (TW) laser system, one of the main projects of the Center for Lasers and Applications. High power ultrashort pulses lasers based on Chirped Pulse Amplification technologies and Ti:Sapphire gain media, allowed the generation of TW peak power on top of conventional optical tables and the realization of relativistic intensities ( $10^{18}$  W/cm<sup>2</sup>) at modest costs.

The main area, "Laser Applications", is concerned with technological laser uses such as laser processing, laser remote sensing, development of new diagnostic and therapeutic methods such as optical coherence tomography (OCT), laser Doppler flowmetry, photosensitization, prevention of dental caries, plus other advanced applications of high intensity lasers.

Recent activities are highlighted bellow:

- Development of single crystal fibers for compact laser systems;
- Growth of a solid solution  $LiGd_{0.232}Lu_{0.75}Nd_{0.018}F_4$ :Nd crystal suitable to obtain a laser medium for mode-locking purposes;
- Characterization, modeling and optical spectroscopy of rare-earth doped crystals and glasses for the development of solid laser medium;
- First single crystal Nd:YLF fiber laser;
- Evaluation of the performance of fs laser-induced Breakdown Spectroscopy (fs-LIBS) for the determination of elements in animal tissues.
- New method for the evaluation of microvascular functionality using low-frequency fluctuations in the laser Doppler flow signal;
- Construction of an automatized workstation with ultrashort laser pulses (femtoseconds) for the study of thermal and non-thermal processes in dielectrics, semiconductors and metals;
- Study of a therapeutic method combining Nd:YAG laser and topical fluoride treatment for effective reduction of caries incidence in patient.
- Development of studies showing that photodynamic antimicrobial therapy is able to reduce 99% of multi-resistant bacteria in burn wounds.
- Analysis of Optical Coherent Tomography applied to dermatology (research work winner of the Natura Campus 2010 Premium for Technological Innovation),
- New LIDAR system for Industrial Emission and Detection installed in Cubatão/SP (collaboration in The National Institutes of Science and Technology Program /INCT).
- Studies for isotope enrichment by ultrashort laser pulses.

### **Lasers Development**

#### **Crystal growth**

The Crystal Growth Laboratory works on bulk, micro and nanocrystals research for materials properties studies and development of new lasers including lamp and diode pumped systems from fluoride single crystals, as  $LiREF_4$  and  $KRE_3F_{10}$  (where RE = rare earth ions), and double tungstates single crystal fibers, as  $AB(WO_4)_2$  (where A = Na, Li and B = La, Gd, Nd, Yb, Eu). The research on crystal growth performed in this period was supported by CNPq, Fapesp and CAPES including collaboration projects with

period was supported by CNPq, Fapesp and CAPES including collaboration projects with Federal University of Sergipe - UFS (CNPq "casadinho" project) and Federal University of Pernambuco - UFPE (CNPq-INCT program in National Institute for Science and Technology in Photonics).

#### **Bulk single crystals growth**

Thulium ions (Tm<sup>3+</sup>) are suitable for frequency upconversion of infrared to visible light, because these ions have long-lived high excited 4f<sup>n</sup> states that give rise to strong blue luminescence. In this period the effect of Nd and Yb used as sensitizers to pump Tm ions has been studied. Two matrices have been utilized as host for these ions: LiYF<sub>4</sub> (YLF) and KY<sub>3</sub>F<sub>10</sub> (KY3F), which have the scheelite and fluorite structures, respectively. YLF:Yb:Nd:Tm crystals were grown by the Czochralski method and doped with 0.5 mol% Tm and/or 20 mol% Yb, and/or 1.3 mol% Nd. KY3F samples of good quality were obtained using a simple synthesis method consisted by the slow cooling of liquid charges under a HF+Ar atmosphere. This method reduced the time to produce samples with sufficient transparency for spectroscopic studies. KY3F:Yb:Nd:Tm crystals were doped with 0.5 mol% Tm, and/or 1.3 mol% Nd and different concentrations of Yb (5, 10, 20 and 30 mol%). Spectroscopic studies determined the mechanisms of energy transfer that lead to the thulium upconversion emissions in the blue and ultraviolet regions.

YLF:Er (15 mol%), YLF:Nd (1.3 mol%) and YLF:Yb (10 mol%):Tm(1 mol%) crystals grown by the Czochralski method were also utilized to obtain lasers with new features.

A solid solution LiGd<sub>0.232</sub>Lu<sub>0.75</sub>Nd<sub>0.018</sub>F<sub>4</sub>:Nd crystal was successfully grown and the spectroscopic studies showed that Nd presents larger bandwidth at 792 and 797 nm, which can be suitable to obtain a laser medium for mode-locking purposes. Inspired in the results obtained in the growth of solid solutions of LiGd<sub>1.x</sub>Lu<sub>x</sub>F<sub>4</sub> by zone melting and by crystal growth from the melt, it is in course the study of the LiF - GdF<sub>3</sub> - LuF<sub>3</sub> phase diagram by computational methods, using the commercial simulation program FactSage.

#### Single crystal fibers growth

Despite new developments for compact laser devices, the Nd3+ ion continues to be the most widely used active laser ion. In 2004 we started a program on single fiber growth by the micropulling down (µ-PD) method aiming the development of laser materials. The objective was to obtain single crystal fibers with constant diameter and good optical quality for laser tests. This project that included the installation of two μ-PD furnaces started with fluorides fibers growth, resulted in 2009/2010 in the demonstration, for the first time, of laser action from Nd:LiYF<sub>4</sub> (Nd:YLF) single crystal fibers. At the present other materials are under investigation as fluoride fibers of Nd:BaY<sub>2</sub>F<sub>8</sub> and rare earth double tungstates fibers also for laser tests. We have prepared Nd:NaLa(WO<sub>4</sub>)<sub>2</sub>, Nd:NaGd(WO<sub>4</sub>)<sub>2</sub> and Eu, Yb and Nd doped LiLa(WO<sub>4</sub>)<sub>2</sub>. The structural and optical characterizations of these materials are under investigation to improve their quality for future laser tests.



Figure 1. Crystal fiber growing in the micro-pulling down system

# **Development of compact diode-pumped solid-state laser sources**

This activity comprises the development of new laser sources based on diode pumped solid state lasers (DPSSL) for applications in research, industry, medical and pollution control. Our investigations are focused mainly on controlling the temporal, spectral and spatial features of the laser beam. In some cases it also includes the production design of such systems including the reliability tests and application experiments. We have built several DPSSL systems emitting from the blue up to the far infrared. Some highlights of the last period are shown below:

- A high power 2.3  $\mu m$  Yb:Tm:YLF laser diodepumped simultaneously at 685 and 960 nm. The achieved output power of 620 mW is the highest reported so far.

### **Lasers Development**

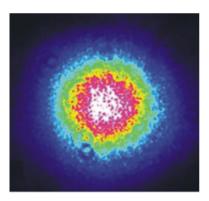


Figure 2. Gaussain beam quality obtained from the Yb:Tm:YLF laser

- Compact, diode-side-pumped Nd<sup>3+</sup>:YLF laser at 1053 nm with 45% efficiency and diffraction-limited quality by mode controlling. The highest efficiency obtained to date and 9.5 W of output power in a transversely diode-pumped Nd<sup>3+</sup>:YLF slab laser operating at 1053 nm were obtained.
- 620 mW Single-Frequency Nd:YVO $_4$ /BiB $_3$ O $_6$  Red Laser. Using a type-I critically phase-matched bismuth borate crystal, a record 620 mW single-frequency red laser at 671 nm is achieved from intra-cavity second harmonic generation (SHG) of a  $\pi$ -polarized single-end pumped Nd:YVO $_4$  ring laser oscillating on the lambda similar to 1342 nm transition.
- Record 1.3 W single-frequency red laser at 661 nm. Achieved from intra-cavity second harmonic generation of a Nd:YLF ring laser oscillating on the  $\pi\text{-polarized}$  transition (lambda similar to 1321 nm).



Figure 3. 1.3 Watt single-frequency Nd:YLF/ppKTP red laser

- High pulse power Er:YLF laser operating at 2.8 μm wavelength (laser cavity shown in Fig. 4).

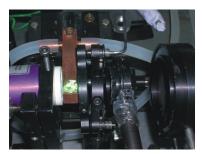


Figure 4. We reported 60% higher average output power for short pulse duration

- First single crystal Nd:YLF fiber laser. Fiber orientation (Fig. 5) and the optical arrangement used in diode laser pumping (Fig. 6) are shown.



Figure 5. 700 µm diameter Nd:YLF fiber

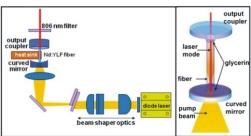


Figure 6. 300 mW of output power and 37% slope efficiency were achieved

#### Production and optical characterization of active laser media based on nanopowders and metamaterials

This activity comprises the development of new laser sources based on diode pumped dispersive media. Lasing action in Nd: YVO<sub>4</sub> nanopowder has been analyzed by investigating the  ${}^4F_{32} \rightarrow {}^4I_{11/2}$  transition. A method to quantitatively determine the upconversion rate and the contribution of the spontaneous emission in the samples backscattering emission that includes the random laser emission as a function of pump power has been created.

# Characterization, modeling and optical spectroscopy of rare-earth doped solid laser media

A luminescence spectroscopic system with spectral and temporal discrimination that uses a Box-car technique and tunable laser excitations of 4 ns (10 Hz) in the range of 420 to 2000 nm (10mJ), was used for lifetime measurements of rare-earth ions in glasses and fluorides crystals. These measurements allowed determining the rate constant of the nonradiative energy transfer that happens due to dipole-dipole interactions between donor and acceptor ions in solids. Energy transfer mechanism involving two interacting erbium (and holmium) ions in the first (and second) excited state, energytransfer up-conversion has been observed and the rate constant determined. The aim of this study is the development of solid laser medium emitting in the mid-infrared (~2750 nm) and to improve light signal amplifiers based on thulium-doped materials that operate in S-band of telecommunication (1470-1500 nm). A detailed investigation of the energy transfer process from the first excited state of holmium caused by Pr3+ ions in fluorozirconate (ZBLAN) glass was carried out. Solving the rate

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equations one can verify the potential gain of the laser medium and know how it can be affected by dopant concentration (activator and sensitizer ions) and pumping intensity. ZBLAN glasses doped with 4 mol% of Ho<sup>3+</sup> and co-doped with 0.1, 0.2 and 0.3 mol% of Pr<sup>3+</sup>were used to evaluate the 2850 nm emission gain under 650 nm continuous pumping regime. Only the ZBLAN glass doped with 4 mol% of Ho<sup>3+</sup> and co-doped with 0.3 mol% of Pr<sup>3+</sup> showed a positive small signal gain, which indicates that Ho<sup>3+</sup>, Pr<sup>3+</sup>-codoped ZBLAN glass is a promasing candidate for high power laser operation at 2850 nm using diode laser pumping at 650 nm. ZBLAN glasses doped with 0.5, 1, 2, 3 and 4 mol% of Dy3+ were investigated using the time-resolved luminescence spectroscopy. Selective laser excitation at 1125 and 1358 nm established that the energy levels above the second excited state of Dy<sup>3</sup> are entirely quenched by non-radiative multiphonon emission in ZBLAN glass. Only two emissions are present with peaks at 1700 nm and 2885 nm with low quantum efficiencies. Excited state absorption (ESA) was detected by monitoring the rise time of the 1700 nm luminescence after tuning the probe wavelength across the spectral range from 1100 to 1400 nm. As a result of nonradiative decay of the higher excited states, ESA contributes to the heating of 3 microns fiber laser based on Dy<sup>3+</sup>-doped fluoride glass. It was establihed that pump ESA not the short decay times of the metastable energy levels has the biggest impact on the performance of Dy3+-doped ZBLAN fiber laser operation at the previously demonstrated pump wavelengths of 1100 and 1300 nm. These results suggest that fiber laser operation is temperature dependent, which may account for large difference in performance of Dy3+- doped ZBLAN fiber lasers pumped at either 1100 nm or 1300 nm because 40% more heat is generated at the former pump wavelength.



Figure 7. The decay characteristics of the excited states of rare earth ions were measured using an pulsed (4 ns) laser excitation from a tunable double optical parametric oscillator (OPO) pumped by the third (or second) harmonic of a Q-switched Nd: YAG laser working in the visible from 420 to  $685\,\mathrm{nm}$  and in the infrared from (1) 670 to 990 nm and (2) 1150 to 2050 nm

# Characterization, modeling and optical spectroscopy of plasmas

Laser plasma spectroscopy technique was applied to ultrashort pulses generated plasmas to evaluate the performance of femtosecond laser-induced Breakdown Spectroscopy (fs-LIBS) for the determination of elements in animal tissues. Sample pellets were prepared from certified reference materials, such as liver, kidney, muscle, hepatopancreas, and oyster, after cryogenic grinding assisted homogenization. Individual samples were placed in two-axis computercontrolled translation stage that moved in the plane orthogonal to a beam originating from a Ti:Saphire chirped-pulse amplification (CPA) laser system operating at 800 nm and producing a train of 840 μJ, 40 fs pulses at 90 Hz. The plasma emission was coupled into the optical fiber of a high-resolution intensified charge-coupled device (ICCD)-echelle spectrometer. Time resolved characteristics of the laser-produced plasmas showed that the best results were obtained with delay times between 80 and 120 ns. Data obtained indicate that both are matrixindependent sampling process and that fs-LIBS can be used for the determination of Ca, Cu, Fe, K, Mg, Na, and P elements. The same technique and apparatus were used to verify the applicability of the technique to monitor the migration of amalgam elements (mercury, tin, silver, copper and zinc) to the neighbor dental tissue in human samples with a spatial resolution of 100 µm. It was possible to determine the penetration depth of mercury, silver, cooper and tin. We also observed a deeper migration of these elements on permanent teeth when compared with deciduous ones. Although the presence of zinc could not be detected under the experimental conditions, the emission lines corresponding to many other elements such as calcium, sodium, phospor, among others, were observed, so configuring the femtosecond LIBS technique as a valuable tool to map the distribution of endogenous and exogenous tooth elements.

### **Lasers Applications**

#### Microvascular function evaluation by lowfrequency fluctuations in the laser Doppler flow signal

The laser Doppler flowmetry has been used to study microvascular dysfunctions, common in diabetics and chronic smokers. Low-frequency fluctuations in the laser Doppler flow signal (LDFS) from the skin are related to microvascular mechanisms of flow control. Wavelet spectral analysis has been used to correlate fluctuations in the LDFS with the endothelial, neurogenic and myogenic mechanisms of control in the frequency intervals 0.0095-0.02 Hz, 0.02-0.06 Hz and 0.06-0.16 Hz, respectively. Generally the signal power, in each frequency interval, derived from the respective wavelet coefficients, is used as a measure of the activity of the related mechanism of microvascular control. However, the time-domain characteristics of the fluctuations in the LDFS in each frequency interval are poorly known. As a consequence, there is a lack of objective criteria to properly measure, in each frequency interval, the related hemodynamic parameters. A time-domain method was developed to analyze and quantify fluctuations in the LDFS in each frequency band. Baseline and thermally stimulated LDFS of forearms from healthy volunteers were collected and analyzed. The data obtained indicate that inappropriate time windows, frequently used for measurements, increase the variability of the measured signal power, diminishing the capability of the method when assessing microvascular dynamics and dysfunctions. Objective criteria were proposed to diminish the measured hemodynamic parameters variability, improving the method sensitivity. Potential applications are assessing endothelial, neurogenic and myogenic dysfunctions.

# Laser processing of special materials: thermal and non-thermal process

Modern technological advances have demanded the development of new materials like high mechanical strength steels, superalloys, ceramics and composites, besides very small pieces with complex geometrical forms. Consequently, traditional milling and welding processes can no longer fulfill the requirements demanded by modern applications. Hence, laser processing comes as very useful and versatile alternative method, and has been used here for cutting welding, heat treating and ablating of some materials for important technological applications. In welding, a pulsed Nd:YAG laser has been used to join very thin foils of alloys highly resistant to corrosion with the purpose of using then as protective shields for sensors against harsh media. Pressure, flow and temperature sensors used in many industrial and nuclear plants must withstand extreme conditions of pressure, temperature and corrosive environments. This is done by covering these devices with foils of special alloys with 100

μm of thickness. To accomplish this task, welding of AISI 316L stainless steel, Hastellov C-276, tantalum, Ti6Al4V and Monel 400 superalloy has been developed in pure material and dissimilar combinations. The experiments lead to hermetic and sound laser welds in several geometries between these thin foils and also between thin and thick base metal. Dissimilar welding of AISI 304 stainless steel and Inconel 600 Ni alloy was developed and the study of the morphology of solidification was used to explain its microstructure development. Also the effects of gas protection during Ti6Al4V welding was studied and optimized. Laser heat treatment is another area of study and it was used in hardening of gray cast iron and aluminum-silicon alloy used in internal parts of combustion engines for the automobile industry. Besides thermal processes accomplished by traditional lasers, another program has been developed where thermal effects can be absent in the region of interaction between laser focus and processed piece. This occurs when laser pulses of very short temporal length, in the order of femtoseconds  $(10^{-14} \text{ to } 10^{-13} \text{ s})$  are employed. To use such pulses a workstation was built and automatized and process parameters for thermal and non-thermal regions were obtained for many dielectrics, semiconductors and metals. These data are essential for machining in micron or submicron scale and have been used to obtain such structures in optical glasses and ceramics. Microfluidic devices were directly milled in BK7 optical glass with excellent precision and geometrical form. Texturing in ceramics surface was also done with femtosecond pulses and used to improve cellular growth and adhesion in medical implants.

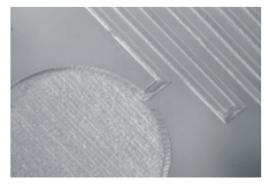


Figure 8. Microfluidic device machined with femtosecond laser pulses. Each channel has a 100  $\mu m$  square profile

# Physical characterization of biological tissues for the development of new diagnostic and therapeutic methods

The physical characterization of biological tissues, mainly the study of their optical properties and thermal responses, allows the development of new processes using high intensity lasers, which can be useful as new diagnostic or therapeutic methods in Dentistry and Medicine. Protocol were developed and tested for several diseases. The caries

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prevention with laser was also studied *in vitro* and a clinical trial was carried out to prove the safety and effectiveness of the method. A cream for topical use for the clinical photodynamic therapy of skin cancer was developed, tested and patented in Veterinary Medicine. We are also studying the potential treatment of burned skin with high intensity femtosecond laser as well as the potential use of erbium laser to cut bone during surgery.

# Prevention of dental caries with lasers "in vitro"

The decline in dental caries over the last few decades has been attributed to the extensive use of fluoride. Although fluoride is the most powerful treatment to prevent tooth decay, the development of new methods to completely control this disease is still necessary, mainly in developing countries. In this way, lasers combined or not with fluoride, have been tested on teeth to improve dental enamel properties in order to enhance its resistance to demineralization. We investigated the compositional and crystallographic changes in vitro on enamel when irradiated by Er, Cr: YSGG or Nd:YAG associated with black coating, its resistance to demineralization when irradiation is associated with fluoride, and CaF2-like material formation and retention. Sample surfaces were analyzed by ATR-FTIR. Irradiation with Er, Cr: YSGG laser promoted a significant decrease on carbonate content of enamel. After Nd:YAG irradiation, it was observed a significant decrease of carbonate and amides I and II. X-ray diffraction measured at Synchrotron facility showed that both laser irradiations promoted formation of αtricalcium phosphate and tetracalcium phosphate, and a significant increase on the crystal growth of the enamel apatite. These changes can explain the improved resistance of enamel to demineralization observed in another study, in which enamel slices received professional fluoride gel applied before or after irradiation. Both lasers significantly reduced enamel demineralization, and the previous APF-gel application followed by laser showed the higher reduction of enamel demineralization. CaF, formed before pH-cycling was significantly higher in groups were APF was associated with laser irradiation. After demineralization in vitro, these groups also presented higher CaF, retention in respect to isolated treatments (only APF or only laser). The combined treatment of laser irradiation with fluoride propitiates an expressive fluoride uptake, reducing the progression of carieslike lesions, and this treatment is more effective than laser or fluoride alone.

# Prevention of dental caries with lasers clinical trial

After all *in vitro* results described above, a doubleblind crossover clinical trial was developed, in which 121 teeth of 33 volunteers were selected. In all volunteers, the right side teeth were selected for Nd:YAG laser + APF application (lased group) and the left side teeth were kept as control group (only APF application). Nd:YAG laser irradiated teeth painted with a black organic ink; after that, topical APF was applied. Recalls were made after 1 year in order to evaluate the formation of white-spot lesions or caries cavities. After 1 year, this clinical experiment showed an overall reduction of 60.2% in caries incidence in (lased group + APF) when compared with control (no treatment) and 39,2% of reduction when compared with only fluoride group. As a conclusion, combined Nd:YAG laser and topical fluoride treatment was effective for reducing the incidence of caries *in vivo*.

#### Lasers in Periodontology - clinical trial

The Nd: YAG laser efficacy associated with conventional treatment for bacterial reduction has been evaluate after Nd:YAG laser irradiation was associated with scaling and root planning in class II furcation defects in patients with chronic periodontitis. Thirty-four furcation lesions were selected from 17 subjects. The control group received conventional treatment, and the experimental group received the same treatment followed by Nd:YAG laser irradiation. Both treatments resulted in improvements of most clinical parameters. A significant reduction of colony forming unit (CFU) of total bacteria number was observed in both groups. The highest reduction was noted in the experimental group immediately after the treatment. The number of dark pigmented bacteria and the percentage of patients with Porphyromonas gingivalis, Prevotella intermedia, and Actinobacillus actinomycetemcomitans reduced immediately after the treatment and returned to values close to the initial ones 6 weeks after the baseline for both groups. The Nd:YAG laser associated with conventional treatment promoted significant bacterial reduction in class II furcation immediately after irradiation, although this reduction was not observed 6 weeks after the baseline

#### **Development of therapeutic processes of photosensitization and photobiomodulation**

The aim of this activity is to develop new therapeutic processes using innovative technology through low-power lasers and light-emitting diodes to provide a nonthermal, noninvasive, environmental safe treatment that can be useful in the Health Sciences. The overall mission of this activity is to develop phototherapeutic processes for human health through of the physical, chemical and biological knowledge of the low intensity light tissue interaction. Our major interests are to investigate the effects of low intensity laser therapy (LILT) and photodynamic antimicrobial therapy (PAT) on biosystems.

### **Lasers Applications**

#### **Photobiomodulation**

LILT is a treatment modality that is becoming more useful in Health Sciences, although mechanisms underlying light effects are still not completely understood. Our group develops researches on light dosimetry and tissue optics to provide to the health professionals scientific background for using this technology to benefit patients. Studies were carried out in vitro and in vivo to investigate the influence of LILT on cell cultures, wound healing, and edema. Our results show that LILT promotes a faster differentiation of human dental pulp stem cells into osteoblast cells compared to nonirradiated control cells opening new possibilities mostly on tissue engineering techniques; preventive laser application is effective in the prevention and treatment of oral mucositis and its daily use contributed to the relief of painful symptoms collaborating to improve the life quality of oncologic patients; near-infrared LILT showed to be efficient on edema prevention and treatment when lymph nodes were irradiated being a potential alternative to anti-inflammatory drugs; light attenuation coefficient changes during wound healing and inflammatory process suggesting that this parameter is suitable to optimize low intensity laser therapy.

#### **Photosensitization**

PAT is based on the application of a photosensitizer and a light source to kill microorganisms. PAT can be useful in life sciences, like odontology, where along with conventional treatment provides a higher efficiency to care for local infections. Alone, neither photosensitizer nor light has the capacity to produce deleterious effect on the biological system. However, when combined, they can eliminate bacteria and fungi, including those resistant to conventional antibiotics and antifungals. Our results show that PAT was able to reduce 99% of multi-resistant bacteria in burn wounds, delayed bacteremia, kept the bacterial levels lower compared to control group and increased the animal survival for 24 h; light parameters play an important role on yeast inactivation and the same energy density under different irradiation parameters presents dissimilar quantity of cell death; methylene blue transport thought yeast membrane can change PAT effects, as well as the photosensitizer (PS) preferential bind site inside the cell; microbial strain characteristics can directly interfere on PAT results and cells appear to be killed by an apoptotic-like effect; PAT can kill fungal cells in vivo and reduce its recovery from infected site; it is possible to induce dental caries in a rat model since demineralization areas were identified by optical coherence tomography, and PAT significantly reduced the number of total bacteria compared to control without treatment and bacterial cells level remained lower than control group until 10 days post-treatment; treatment of herpes labialis with PAT is effective, has no side effects, and when associated with LILT accelerates

the healing process.

# Optical coherence tomography applications

Optical coherence tomography (OCT) is a diagnostic imaging technology based on low length coherence interferometry in which the coherence features of photons are explored, leading to an imaging technology that is capable of producing non-contact, non-destructive, high-resolution crosssectional images of internal microstructures of living tissues. We implemented several OCT systems. There are available OCT's in several wavelengths (830, 903 and 1300 nm) with polarization and Doppler flow capabilities. With a Polarization Sensitive OCT (PS-OCT) birefringence images can be performed, in this way the differences between the refraction indices can be analyzed as an image, making diagnoses simple to be performed. Besides PS-OCT images, a more complex, but also more complete, way to study the polarization properties of light can be done using the Mueller Matrix theory. OCT with micron resolution allows tiny and delicate structures precise study such as hair stream. Hair streams have about 70 microns in diameter, in such a manner that the cosmetic field can also take advantage of this technology by evaluating physical and optical effects caused in the hair streams by the chemical treatment. We have used OCT to determine the medium refraction index of different hair groups, blond and Caucasian before and after a chemical treatment. In collaboration with a cosmetic industry we developed non-invasive methods, for in vivo visualization and access the skin topography changes during a cosmetic treatment of periorbital region with an anti-aging product. We found that this method was able to quantify small variations in skin surface roughness (1%). After 28 days, significant reductions in roughness (6.2%) were observed in treated areas whereas non-treated areas showed no changes in skin roughness. Frequency depth distribution of OCT values (5 to 600 µm) showed a reduction in the frequency of 170 - 300 µm events and an increase in the frequency of 5 - 170 µm events. These results are consistent with the wrinkle reduction profile expected from anti-aging treatments. OCT proved to be a highly sensitive method to detect skin topography variation. We also have used the OCT technique to follow in real time optical changes in yeasts organized in biofilm simultaneous to PDT.

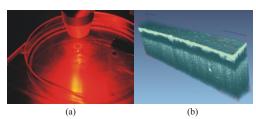


Figure 9. (a) Biofilm in SE disks during PAT, (b) 3D OCT image after PAT with 1 mM MB and red laser. Image generated by VGStudioMax 1.2®

### **Lasers Applications**

#### Laser remote sensing of the atmosphere

LIDAR is the acronym for Light Detection and Ranging and like RADAR operates on the same principles of sending electromagnetic radiation into the atmosphere and detecting the returned light signal. This technique is powerful dues its high temporal (seconds to minutes) and spatial (below 10 m) resolutions. With a few laser shots the system is able to characterize the atmospheric dynamics, the presence of pollutants and study dispersion patterns important features in pollution monitoring. Also this system is capable of tracing mid- and longrange transport of biomass burning activities in rural areas of the country. Also the LIDAR is an important climatological tool for studying cloud-base altitudes, water vapor atmospheric content and aerosol optical properties, which have been investigated since 2001, and the Laboratory of Environmental Laser Application was created. The LIDAR system at IPEN operated on physical principles of elastic scattering, namely Rayleigh and Mie scattering, and inelastic scattering, Raman scattering. The former is used to monitor aerosols in the atmosphere, and besides their optical properties, aerosols act as tracers for atmospheric dynamics and cloud formation. The Raman channels in the system are used to perform water vapor, nitrogen and oxygen concentration profiling. Besides in a collaborative effort with Howard University and Goddard Space Flight Center NASA we have developed an independent calibration methodology to obtain a vertical distribution of water vapor mixing ratio in the atmosphere based on physical principles. The Laboratory of Environmental Laser Applications has started in 2008 a joint project with Instituto Politécnico at University of São Paulo SP to develop a LIDAR like system to evaluate oil refining processes based on the particle distribution exhaust from refineries and the system is part of a The National Institutes of Science and Technology Program (INCT). The latest project involves the understanding of the so-called aerosol indirect effects based on the particle higroscopicity an important process in climate change.



Figure 10. LIDAR system in operation at IPEN, which shows the second harmonic laser beam from Q-switched Nd:YAG laser (left) and the telescope (right)

### **High Intensity Lasers**

# Operation and optimization of the TW peakpower laser and applications

High power ultrashort pulses lasers based on CPA (Chirped Pulse Amplification) technologies that allow the study, in conventional laboratory scales, of phenomena that only 10 years ago were restricted to national laboratories with annual budgets of billions of dollars. In the Center for Lasers and Applications at IPEN, a hybrid Ti:Sapphire/Cr:LiSAF TW peak power laser system is under continuous development. A flashlamp pumping cavity for a Cr:LiSAF gain medium in the shape of a rod was built. The pumping cavity was developed aiming to minimize the thermal load on the Cr:LiSAF crystal by the use of absorption filters between the filters and the gain medium, allowing the amplification of ultrashort pulses to the terawatt peak power region at high repetition rates. The pumping cavity was initially used in a laser configuration, and generated 60 µs pulses with energy up to 2.8 J, with an average power of 30 W at 15 Hz repetition rate, the highest reported to date. The utilization of the pumping cavity in a hybrid Ti:Sapphire/Cr:LiSAF CPA system produced pulses with 30 mJ of energy and 60 fs of duration at 5 Hz repetition rate, reaching 0.5 TW of peak power, the highest in the southern hemisphere. Our laboratory also has another amplified laser system capable of generating up to  $800 \mu J$ , 25 fs pulses or 300  $\mu J$ , 5 fs pulses. Even at lower peak powers, the ultrashort character of the pulses generates nonlinear phenomena, particularly those initiated by multiphotonic processes that generate free electrons. Ultrashort pulses with up to 30 GW peak power were utilized to crest defects (color centers, vacancies) in crystals, glasses and polymers, to measure nonlinear effects in solid and liquid samples, and to ablate and machine technological materials with precision on the micrometer scales without heat affected zones. This nonthermal ablation is being studied for the removal of necrosed material from burned animals and its effects on the tissue regeneration. Also, the generation of High Harmonics by ultrashort lasers pulses is under study to produce in the VUV and soft X-ray region with durations into the attosecond regime.

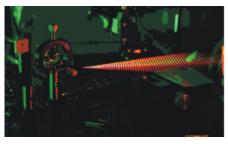


Figure 11. Harmonics generated by 1 Hz trains of pulses focused in air

#### Color center production by high intensity ultra short laser pulses

Ultrashort pulses with peak powers up to 30 GW were used to create color centers in fluoride crystals grown in our facilities, in glasses and polymers. It was possible to determine that the centers generation begins with multiphotonic absorption, favored by the high intensity due to the short pulse duration that neutralizes negative ions, which are kicked out of their sites. Once the centers are formed by electrons trapped in the vacancies, they act as a probe to study the material structure. The confined characteristic of the laser beam creates a high density of basic color centers that results in its aggregation in secondary, more complex, centers. These secondary centers are generated with a higher concentration than attainable with traditional ways of creating centers, such as exposure to ionizing radiation. Also, the laser excites the centers during its formation process, allowing emission spectroscopy to be performed during its creation. Centers were created in LiF (Lithium Fluoride), pure, Tm and O doped YLF (Yttrium Lithium Fluoride) crystals, and also in BaLiF crystals grown by different methods. Color centers were similarly created in ZBLAN glasses and PMMA polymer samples, and its properties investigated.

# Pump and probe studies of ultrafast magnetization dynamics in the femtosecond timescale

Since the advent of reliable solid state lasers in the 90's producing ultrashort light pulses routinely, the field of time-resolved optical spectroscopy has experienced a boom. The availability of new sources, wavelengths and increased time resolution has allowed better comprehension of different phenomena occurring in chemistry, condensed matter physics and biology. In this context one of the most widespread used techniques to study ultrafast phenomena is the pump and probe optical technique. In this kind of experiment one stronger ultrashort light pulse interacts with the medium under investigation, disturbing the system. The return of the system to equilibrium is then monitored by probing the optical properties of the system with a weaker delayed ultrashort probe pulse. The arrival of the light probe is controlled in a stroboscopically way, following the return of the system to equilibrium. After the pump pulse excites the system, the gained energy will be redistributed in the medium in different channels, and the internal forces governing this redistribution of the energy can be investigated. A pump and probe setup was bult in our ultrafast laser laboratory, to study the ultrafast dynamics of the magnetization in technological materials by the simultaneous measurement of the electron and spin dynamics with time resolution limited by the duration of the pump pulse. Amplitude and the direction of the magnetization vector M were measured to retrieve its trajectory from tens of femtoseconds up to a few

### **High Intensity Lasers**

nanoseconds in Co-rare earth doped films. Our focus is the investigation of the role of the rare earth doping in the ultrafast demagnetization and damping of the precessation motion in transiction metals.

# **Determination of the ultrashort pulses** ablation threshold of solid samples

Ultrashort pulse laser ablation of solids is due to an electron avalanche induced breakdown process that occurs when seed electrons are accelerated in the laser field, exponentially generating free electrons by collisions. The breakdown takes place when the plasma originated by the avalanche electrons reaches a critical density and transfers energy to lattice ions, which expand away from the surface after the pulse has passed. In metals, the seed electrons are always present (conduction band free electrons), and in dielectrics and semiconductors they are excited from the valence band to the conduction band by the pulse leading edge. Although the seed electrons have dissimilar origins in different material types, a metallization occurs in dielectrics and semiconductor after they are produced, and the avalanche evolves deterministically in time in the same way in all solid materials that now behave like metals. These mechanisms confer a nonselective characteristic to the ultrashort pulse ablation, and the intensity ablation threshold of a material is the only parameter relevant to the etching process. In our labs we created a new method to determine the ablation threshold by ultrashort laser pulses, based on scanning the sample diagonally across the beamwaist of a focused beam. The scan etches a profile in the material surface, and the measurement of this profile maximum transversal dimension and the knowledge of the laser pulses power immediately determines the material ablation threshold. We have applied this technique to a variety of materials, ranging from crystals to metals, composite polymers and even biological samples (animal tooth and bone), obtaining results agreeing with the ones reported in the literature.

# Generation of high harmonics into the VUV and soft X-ray spectral regions

When an ultrashort pulse impinges on a gas at low pressure, electrons can be freed from its parent atoms by the leading edge of high intensity ultrashort pulses, and then be accelerated by the pulse carrier wave into a quivering motion. When these electrons collide with the atom, its energy is released in the form of harmonics of the exciting field, and if the kinetic energy is sufficiently high, the harmonics can reach the UV and soft X-ray region, generating photons up to a few keV. In the High intensity ultrashort pulses laser laboratory, we are pursuing the generation of these harmonics into the region of the water window, around 2-4 nm, which are proposed to be used in high-resolution radiographies of living tissues. For this goal, pulses

with durations of 25 fs and 5 fs will be focused on noble gases at pressures ranging from around 50 mbar up to 500 mbar, inside a vacuum chamber instrumented for the generation, detection and characterization of the harmonics produced.

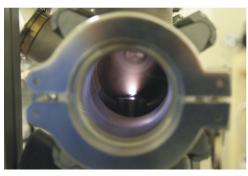


Figure 12. Ablation of a metallic sample under vacuum, to study the isotope enrichment due to the separation that occurs in the ablation plume

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#### **Honor Mention and Awards**

Claudia Rodrigues Emilio and Denise Maria Zezell - "Comparison between two different photodymanic therapy of tumor protocols applied to the treatment of skin cancer in feline", doctorate project was selected to participate at the Campus of Excellence 2008, Spain - veterinarian medical area. In this event, more than 10 Nobel Prizes discussed and choosed the best 100 graduate projects from Latin America and Africa.

Anderson Zanardi de Freitas, Nilson Dias Vieira Junior, André Rolim Baby, Telma Mary Kaneko, Maria Valéria Robel Velasco, Monica Beatriz Mathor and Valcinir Bedin - "Optical coherence tomography applied to cosmetology: preliminary structural characterization of hair fibers", Best Work Award (Third Place) - XXII Congresso Brasileiro de Cosmetologia, ABC - Associação Brasileira de Cosmetologia, 2008.

Anderson Zanardi de Freitas, Marcus Paulo Raele, Hindra Colodetti and Luciana Sanglard - "Measurement by optical coherence tomography of wear promoted in the enamel of deciduous teeth by abrasion technique", Prof. Guedes-Pinto Award (Third Place) - IV Congresso de Odontopediatria - Associação Paulista de Cirurgiões-Dentistas, 2008.

### **Program Team**

#### **Honor Mention and Awards**

Fábio Juliano da Silva Lopes and Eduardo Landulfo - "Measurements of tropospheric aerosol in São Paulo - Brazil using a Lidar system from Centro de Lasers e Aplicações do IPEN and CALIPSO satellite", LAMP Poster Award SPIE; Best Poster Award (Third Place) - Winter College on Optics in Environmental Science - International Centre for Theoretical Physics - Trieste, Italy, 2009.

Marcus Paulo Raele, Marcello Magri Amaral, Nilson Dias Vieira Junior and Anderson Zanardi de Freitas - "PS-OCT Birrefringent Meassurements", Best Poster Award (Optics) - XXXII Encontro Nacional de Física da Matéria Condensada, Águas de Lindóia - São Paulo, 2009.

IPEN was engaged in the Projeto Cenpe-Cana financed by Petrobrás to measure the level of atmospherical pollution in the interior of São Paulo state using a LIDAR movel station. The physicist Eduardo Landulfo gave an interview to EPTV, subsidiary of TV Globo (Rio Claro), to explain the project objectives that uses a LIDAR mobile station for analising the pollution caused by cane burning, 2009.

Luciana Faria Sanglard, H. Colodetti, Célia R. M. D. Rodrigues, Nilson Dias Vieira Junior, Marcus Paulo Raele and Anderson Zanardi de Freitas - "Optical coherence tomography and scanning electron microscopy analysis of micro abrasion effects in deciduous teeth enamel", Best Oral Presentation (Second Place) - I Encontro da Divisão Sul Americana da Federação Mundial de Laser em Odontologia e do V Congresso da Associação Brasileira de Laser em Odontologia - São Paulo, SP, 2009.

The physicist Ricardo Elgul Samad won the 4th edition of the After Image Photo Contest sponsored by the Optics and Photonics News (OPN), an international magazine. The magazine, which publishes the latest developments in the field of optics, is a monthly publication of the Optical Society of America and is directed at researchers, engineers, entrepreneurs and students. In 2009 the magazine received 81 entries to compete with images produced in laboratories, a record number of participants. In the winning shot, the effect produced by the motion of a sheet of paper across the pulses from a high power laser installed at IPEN is observed.

Anderson Zanardi de Freitas - "Avaliação do uso da tomografia de coerência óptica em dermatologia", Inovação Tecnológica Natura Campus 2010 Award (First Place) - ceremony realized in the Natura company centre. Freitas won also a total free course in the Massachusetts Institute of Technology (USA) and a MAC Book Pro13 microcomputer.