# **Lasers Technology** Lasers Development 133 Lasers Applications 136 141 High Intensity Lasers

## Introduction

The Lasers Technology Program of IPEN is developed at the Lasers and Applications Center. In recent years the Program was dedicated to the research and development of optical materials, as laser media and scintillation crystals; characterization, modeling and optical spectroscopy of solids, plasmas and biological materials; development of a compact diode pumped-solid state laser and the development of a high power (TW) laser system, one of the main projects. High power ultrashort pulses lasers based on CPA technologies and Ti:Sapphire gain media, allowed the generation of high peak powers in conventional optical tables, and the attainment of relativistic intensities (10<sup>18</sup> W/cm²) at modest costs.

The Program is strongly committed to the study of Laser Applications on several areas: Nuclear, Medicine and Dentistry, Industry, Environment and Advanced research, aiming not only research but diffusion and innovation, in association with Brazilian Universities and commercial partners. Some highlights of the Program:

- Development of new fluoride materials for laser systems in cooperation with Institut für Kristallzüchtung, Germany.
- Characterization, modeling and optical spectroscopy of rare-earth doped crystals and glasses for the development of solid laser medium;
- Laser induced plasma spectroscopy technique for plasma properties investigation;
- A 600mW Yb:Tm:YLF diode pumping laser emitting at 2.3 $\mu$ m best result of the literature;
- A hybrid Ti:Sapphire/Cr:LiSAF TW peak power laser system 0.5 TW ultrashort already generated;
- New pulp vitality tests, done by Laser Doppler Flowmetry increased the test performance;
- Implementation of an Optical Coherence Tomography (OCT) resulted in collaboration with a cosmetic company allowing the development of diagnose tests and tissue characterization;
- Therapeutic processes of photosensitization and photobiomodulation were studied allowing the advance of new ones using innovative technology through low-power lasers;
- Physical characterization of biological tissues optical properties and thermal responses was performed for the development of new processes using high intensity lasers;
- Collaboration among IPEN, CTMSP and INB: laser processing of special materials: welding of Zircaloy-4 applied in assembling the fuel elements used in Angra dos Reis reactor;
- A ground-based elastic backscatter LIDAR system has been upgraded and two additional channels were added in order to achieve both Nitrogen and Water Vapor Raman signals in the atmosphere up to 4 km in altitude.

These achievements were supported by national and international funding agencies (FAPESP, CNPQ, FINEP, CAPES, DAAD), as well as, cooperation with partners such as Natura, Lasertools, Smar, and others. In the period about 2 millions dollars were invested in the program.

## Lasers Development

## **Crystal growth**

The research on fluoride crystal growth performed in this period was focused in the investigation of the YLF:Yb:Tm, LiGd<sub>1-x</sub>LuxF<sub>4</sub>( $0.5 \le x \le 0.75$ ), and BaY<sub>2</sub>F<sub>8</sub> single crystals doped with Tb, Er, Pr and YLF single crystal fibers for optical applications. These projects were supported by CNPq (Edital/2004) for the investigation of Czochraslki single crystals for laser application; CNPq/PADCT project in collaboration with Federal University of Sergipe (UFS), in the investigation of new scintillator materials; CNPq-Instituto do Milênio program in collaboration with Federal University of Pernambuco, in single crystal fibers growth investigation and CAPES/PROBRALDAAD project with the Institut für Kristallzüchtung (IKZ), Germany, which common research in the investigation of the phase relation of the binary subsystem YF<sub>3</sub>-GdF<sub>3</sub>; YF<sub>3</sub>-LuF<sub>3</sub> and YF<sub>3</sub>-BaF<sub>2</sub>.

#### Fluoride single crystals growth

YLF crystals were grown by the Czochralski method; they were doped with 0.05 mol% Tm and/or 20 mol% Yb, and/or 1.3 mol% Nd. The objective was the identification of the most important processes that lead to an enhancement of thulium blue up conversion emission, under excitation around 792 nm. An enhancement in 475 nm-Tm3+emission of almost 20 times was observed in the samples YLF:Yb:Tm:Nd compared to an YLF:Yb:Tm sample. The melting behavior of LiGd<sub>1.x</sub>LuxF<sub>4</sub>  $(0.5 \le x \le 0.75)$  solid solutions was studied utilizing the melting zone technique, under a HF reactive atmosphere, to obtain high purity compounds. It was concluded that the LiGd<sub>1-x</sub>LuxF<sub>4</sub> compounds melt congruently by x values around 0.75. Crystals were successfully grown from the melt, under an Ar+CF<sub>4</sub> atmosphere, with the stoichiometric compositions, LiGd<sub>0.25</sub>Lu<sub>0.75</sub>F<sub>4</sub> and  $LiGd_{0.232}Lu_{0.75}Nd_{0.018}F_{4}$ .

#### **Phase diagram studies**

The binary phase diagram YF<sub>3</sub>GdF<sub>3</sub> was studied by differential scanning calorimetry (DSC). Yttrium fluoride and gadolinium fluoride show complete miscibility. The transformations at high temperature are of first order and occur at 1338.6 K (YF<sub>3</sub>) or 1174.8 K (GdF<sub>3</sub>). A second order shaped local maximum at 1333 K was observed for GdF<sub>3</sub>. The phase diagram GdF<sub>3</sub>LuF<sub>3</sub> was determined by DSC and X-ray powder diffraction analysis. The mutual solubility of both components is unlimited in the orthorhombic room temperature phase. The maximum solubility of Lu in the high temperature phase of GdF<sub>3</sub> (tysonite type) is about 20% and the maximum solubility of Gd in  $LuF_3$  ( $\alpha$ -YF<sub>3</sub> type) is about 40%. Intermediate compositions of the low

temperature phase decompose upon heating in a peritectoid reaction to a mixture of both high temperature phases. The BaF<sub>2</sub> - YF<sub>3</sub> system was also investigated, with focus given to the BaY<sub>2</sub>F<sub>8</sub> compound and its neighboring phases. Various difficulties hinder the thermal analysis investigation of this binary fluoride system however thermal results compared with data from the literature showed discrepancies. Thermal analysis curves show no variation in the polymorphic transition temperature of BaY<sub>2</sub>F<sub>8</sub> in each side of the compound. Conclusive evidence of the presence of the solid solution range BaY<sub>2</sub>F<sub>8</sub> mentioned in previous literature has not been found. All thermal peaks were observed to occur at temperatures much higher than described by literature phase diagram. The observed discrepancies are under further investigation.

#### Single crystal fibers growth

Regular and transparent BaLiF<sub>3</sub> and LiYF<sub>4</sub>:Nd and Er-doped single-crystal fibers were successfully grown. Several improvements were performed on the micro-pulling down system used for fiber pulling process. A new technique for welding of micro-crucibles was developed. The atmosphere control was determined as the most important factor to avoid impurity contamination from moisture and oxygen, preserving chemical composition and optical quality of the growth fibers. It is well know that optical properties of laser hosts are very important in determining laser performance.



Figure 1. BaLiF<sub>3</sub> single crystal fiber growth

## Lasers Development

## Development of compact diodepumped solid state laser sources

This activity comprises the investigation of laser systems with respect to their temporal and spectral characteristics with the purpose of improving their usage in future applications in industry, dentistry, medicine and pollution control. It also includes the production design of such systems, tests and experiments in order to make these lasers reliable and application ready. Our group has the capacity to generate know how in the area of optical resonators which allows us to achieve specific desirable characteristics of diverse nature such as temporal, thermal, spectral, beam quality and efficiency. Our diode pumped solid state lasers (DPSS) have output powers in the range of milliwatts to 50 watts and in most cases, close to Gaussians beam quality. We have build laser that operate frequency tunable in the NIR and IR.

Some highlights of this last period are:

- Single frequency Nd:LiGdF laser, continuously tunable during 150 GHz.
- TEM00 Tm:Ho:YLF emitting at 2  $\mu m$  with 2.3 watts of output power.
- Compact, diode-pumped LiF:F2 laser emitting at  $1.14~\mu m$ .
- 600mW Yb:Tm:YLF laser emitting at 2,3 μm (best result of the literature).
- Spectral conformation of laser diodes: we developed a proprietary technology to narrow the emission frequency (Figure 2) and generate dual frequency emission. This technique is useful for solid state laser pumping and telecommunications (wavelength division multiplexing).

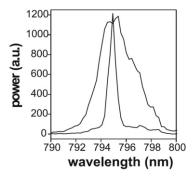


Figure 2. Frequency narrowing of a 40 watt diode

- Nd:YVO laser with 63% optical to optical efficiency and 20 watts of output power int

TEM00 mode (Figure 3).

- Nd:YLF laser operating at 1053 nm with 45% efficiency.



Figure 3. Very compact Nd:YVO laser with 20 watt of output power and 63% efficiency

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

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 (10 mJ), was used for lifetime and luminescence spectrum measurements of rate earth ions in glasses and fluoride crystals. These measurements allowed determining the rate constant of the non-radiative 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 excited state, energy-transfer 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 medium infrared and to improve light signal amplifiers based on thulium-doped materials that operate in the S-band of telecommunication (1470-1500 nm). A detailed investigation of the energy transfer process from first excited state of erbium (and holmium) caused by Nd3+, Tb3+ and Eu3+ ions in LiYF<sub>4</sub> (YLF) crystal was carried out. By solving the rate 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. Fluorozirconate (ZBLAN) and germanate glasses based on GeO<sub>2</sub> doped with 0.1 mol % of Tm<sup>3+</sup> and codoped with (0.1-4 mol %) of Tb<sup>3+</sup>, Eu<sup>3+</sup> and Ho<sup>3+</sup> were used to evaluate the 1.5 m

## Lasers Development

emission gain (or amplification gain) under 797 nm pumping. The ZBLAN glass doped with 0.5 mol% of thulium and codoped with 1 mol % of holmium showed a gain 10 times higher than one verified for Tm(0.1%):Tb(0.2%) in germanate glass. The rate equations solutions using the fourth order Runge-Kutta numeric method allowed us to get the population distribution of the three lowest levels of Er<sup>3+</sup> (and Ho<sup>3+</sup>) in YLF involved in the optical cycle of laser emission at ~3 m. This investigation was applied to Er single doped, Er-Nd, Er-Tb and Er-Eu doped YLF crystals. It was observed that 20 mol % of erbium is the best concentration to maximizes the laser gain at 3m emission in Er-single doped YLF crystal. However, the best concentrations of Er:Nd:YLF is 4 mol % of Er<sup>3+</sup> and 1.5 mol % of Nd<sup>3+</sup> that has a frequency limit of operation increased to 315 Hz. The frequency limit of operation in Er (20 mo 1%):YLF was observed to be much lower, ~15 Hz for a pumping at 980 nm.

## Characterization, modeling and optical spectroscopy of plasmas

Laser induced plasma spectroscopy technique was implemented in three setups: Nitrogen laser (UV, 10 ns), Ti:Sapphire laser (NIR, 45 fs) and Nd:YAG laser (NIR and green, 4 ns). Some experiments were carried out in order to characterize the plasmas generated by nanosecond and femtosecond lasers in several materials. The plasma can be completely characterized by its temperature and electronic density using the Boltzmann plot and Stark broadening methods. The temperature of a plasma generated in air by femtosecond pulses was determined as 130(74) 103K for a ms integrated gate time and 392(9) 103K for a ns gate time. The value for electronic density was 1.1(1) 1018 cm<sup>-3</sup> for both gates. Some stainless steel standards were irradiated by ns and fs regime, allowing us to observe that the femtosecond irradiated steel formed a plasma almost twice hotter but less dense than the nanosecond one (Figure 4). It was possible to determine the plasma evolution in soil samples irradiated by femtosecond laser, discriminating the white continuum from atomic and ionic emissions. The temperature of the plasma evolved from 114(12) 103 K in 30 ns to 12(1) 103 K in 130 ns and the electronic density diminished from 9(1) 1018cm<sup>-3</sup> to 1.5(2) 1018 cm<sup>-3</sup>. Recently, the Saha-Boltzmann method was implemented. The calibration free technique was applied to cultural heritage coins and the analytical determination of elemental composition were employed. For an "one thousand réis" coin from 1939 (Tobias Barreto) the plasma temperature was 10300(150)K, and the plasma density was 4.3(1) 1016cm<sup>-3</sup> using Saha-Boltzmann plot and 2.5(2) 1016 cm<sup>-3</sup>. The evaluated Cu, Al and Zn concentration agreed to the Edict Law for coin forgery.

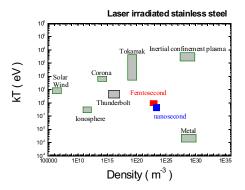


Figure 4. Comparison between the temperature (in eV) and densities (in m-3) for some natural and human created plasmas and the values evaluated for stainless steel irradiated by nanosecond and femtosecond lasers

## **Teeth vitality testing via Laser Doppler Flowmetry**

Pulp vitality tests based on assessment of pulp blood flow, measured by the laser Doppler Flowmetry (LDF), have been suggested in the literature. The relevance of the technique is justified by the limitations of the current methods for evaluation of pulpal vitality, based on sensitive stimulus, with fail after a trauma and in immature teeth. For non-invasive pulp blood flow measurements via LDF, an optical fiber delivers the laser radiation to the tooth enamel. A fraction of the transmitted radiation, by the enamel and dentin, reaches the pulp and via an inverse pathway is collected by another optical fiber. It is recognized that a fraction of the scattered radiation also reaches the periodontal and gingival tissues, and a fraction of the measured flow via LDF originates from these regions. As a consequence, generally the measured flow (F) from devitalized teeth is not zero. In addition, generally the measured flow from both vitalized and devitalized teeth varies largely among individuals, as well as among teeth of the same individual. Thus measured fluxes from both vitalized and devitalized teeth occupy a continue range of values, and generally the ranges are superimposed. In such conditions it is not possible to find a cut-off value of flow below which all devitalized teeth are correctly classified and above which all vitalized teeth are also correctly classified by the test. As a consequence, the discriminatory capability of the current vitality test based on LDF may be significantly decreased, depending on the amount of measured flow from other regions than the pulp. To improve the test accuracy, we developed two new discriminators, derived from the measured flow (F), aiming the minimization of intra and inter-subjects variability. We used the ROC curve analysis as an objective method to quantify and to compare the performance of three discriminators: I) the currently used, the measured flow F(UA); ii) the percentage of flow variation, F(%) and: iii) the difference of flow, F(Diff), being (ii) and (iii) between the interrogated tooth and its homologue vitalized. ROC analysis confirmed our expectation that the proposed discriminators allowed accuracy higher than 96% (here accuracy is the percentage of correct classification of any arbitrary pair of teeth one vital and one non-vital). The measured accuracy of current test was 22% lower. Accuracy of the current sensitive methods for vitality testing (Electric and Cold tests). The accuracies of two new, F(%) and F(Diff), non-sensitive methods (laser Doppler), are higher than sensitive methods,

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and higher than the current laser Doppler flow-based discriminatory method.

# 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 in cutting and welding of some materials for important technological applications. One of the developed processes is the welding of Zircaloy-4, a zirconium alloy, which has begun in collaboration among IPEN, CTMSP and INB. This alloy is largely used in nuclear applications and, in this specific case, these welds have been accomplished in assembling of fuel elements used in Angra dos Reis reactor. Because this material is extremely reactive with oxygen, welding must be done in controlled atmosphere, where oxygen content must be kept very low (less than 20 ppm). Due to the complexity of this spot weld with non traditional geometry, process parameters must be carefully controlled and balanced, and the studies carried out with a Nd:YAG pulsed laser has brought significant improvements in the quality of these joins. Dissimilar stainless steel welding is another activity where research has brought significant results in applications of great importance in nuclear and aero spatial fields. Following remarkable advances in this kind of welding, joining austenitic, martensitic and nickel alloys, a study was realized to understand the process of nucleation occurring in the melting zone during cooling and solidification of the welding pool. The aim was to achieve more resistant and confident welds, avoiding common defects like cracks porosity and excessive melt. Titanium and its alloys, due to their good mechanical strengths when used in high temperature, also have had many applications in medical, nuclear and aero spatial areas. Studies were carried out to determine the gas shielding influence in the properties of laser welds of these materials and resulted in optimized joints with good cosmetic and mechanical quality. Laser cutting of these materials was also optimized aiming the production of improved cut edges free of nitrates, recast material and dross. Besides thermal processes accomplished byraditional lasers, another program has been

developed where thermal effects are absent in the region of interaction between laser focus and processed piece. This occurs when very short laser pulse length are employed, in the order of femtoseconds (10-14 a 10-13 s). To use such pulses a workstation was built and the first results were obtained allowing the achievement of ablation threshold for some metals and semiconductors. These data are essential to machining in micron or sub-micron scale and have been used to obtain such structures.

## Main products and services - core business

A joint collaboration with LaserTools enterprise was concluded and resulted in transference of technology of many laser cutting and welding methods. Theses manufacturing processes has been used regularly to attend several costumers like: Zillo Lorenzetti, GM Reis, Fibra Forte / INPE and Alacer. Laser welding studies were done in copper electrical connectors used in the automobile industry to Delphi Automotive Systems. To Smar Comercial Ltda a program has been initiated to develop welds in nickel alloys foils used in chemical seals.

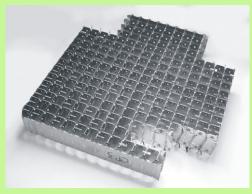


Figure 5. Spacing grating of fuel element used in Angra dos Reis nuclear reactor. Junctions are spot laser welded in thin sheets of Zircalov-4

# 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. Dental caries is considered the most prevalent disease during childhood and adolescence even though its decline in the last decades, nevertheless its incidence in the development countries is still high. For this reason, more conservative restorative methods, more efficient preventive procedures and more accurate diagnostic methods are necessary. Besides caries, periodontal and

endodontical microorganism infections are a matter of concern in Dentistry, as they are closely related to other diseases such as cardiopathologies. In this sense, an extensive study of optical and thermal properties of the dental hard tissue was conducted within a scientific cooperation among IPEN CNEN / SP and the School of Dentistry - USP.

## Spectroscopic and thermal characterization of enamel and dentine

Laser irradiation of hard dental tissues can promotes changes in their physical and chemical characteristics due to the temperature reached during irradiation. Temperature rise during diode, Er:YAG and Er:Cr:YSGG laser irradiation of enamel and dentin was determined using thermocouple and an infrared thermographic camera. Electron spin ressonance (ESR) and Fourier transform infrared spectroscopy (FTIR), showed that after heating the tissue to temperatures between 100 and 300°C, some water is eliminated and the hydrogen bonds, which determine collagen alpha-helix structure stabilization, are lost. After elimination, the collagen matrix is changed and electrons are probably trapped, giving rise to ESR signals and absorption bands in the ultravioletvisible spectral

#### **Prevention of dental caries with lasers**

Lasers can reduce the rate of subsurface demineralization in enamel. The percentage of inhibition of dental caries varies from 30 to 97.2%, and the association with fluoride enhances the inhibition of caries. Several laser wavelengths were used for this purpose. The use of TEA CO<sub>2</sub> laser (10.6 m) was investigated by measuring the calcium and phosphorous content after demineralization. Although CO, laser irradiation does not promote any surface photomodification, it was able to significantly reduce enamel demineralization. Tetracalciumphosphate was identified as a new crystalline phase formed during Ho:YLF irradiation of enamel, detected by X-ray diffraction using a synchrotron source. Er:YAG and Er,Cr:YSGG lasers changes mainly the organic matrix (collagen) and water and little the mineral matrix (OH radical). 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

## Early detection of dental caries

An optical early detection caries method studied was the fluorescence spectroscopy, which enabled to differentiate carious from natural tissue, indicating that the blue laser at 405 nm can be used clinically to carious diagnosis. The presence of three narrow fluorescence bands at 455, 582, and 622 nm indicates that the origin is from mineral matrix, while the broadband near 500 nm can be assigned to organic matrix. The optical coherence tomography (OCT) has showed to be an accurate image method that allows to detect early caries in its structure before it could be detected clinically or radiographically.

## Caries removal and cavity preparation with lasers

Enamel and dentin removal can be achieved using lasers with wavelengths that are strongly absorbed by these tissues. In the infrared, the strongest absorption bands occur in the 2-3 µm and 9-11 µm regions, that are readily accessible by holmium, erbium and CO<sub>2</sub> lasers. Usually the morphological pattern of Er:YAG laser irradiated enamel and dentin has a rough aspect with a clear exposition of the prisms and dentinal tubules. The melted surfaces covering the CO<sub>2</sub> laser irradiated enamel and dentin, occlude the dentinal tubules and the enamel prisms. The rough pattern after Er:YAG laser irradiation, which originates from the micro-explosion of water, does not occlude the dentinal tubules, whereas the surface morphology after CO2 laser irradiation, which originated from the temperature rise above hydroxyapatite melting point, shows dentinal tubules occlusion and tissue melting. These changes influence the tissue properties such as increase of acid resistance and may benefit the bond strength between the tissue and composite resin used for restorations. An optical coherence tomographic system verified the quality of restorations by measuring the distance of restorative material and normal tissue.

## Lasers in Periodontology and Endodontics

Thermal and photodisruptive laser effects result in the elimination of pathogenic microorganism in periodontology and endodontics. Although the Er:YAG laser did not interfere in the adhesion of blood components, it caused more changes on the root surface, whereas the diode laser inhibited the adhesion. The Nd:YAG laser

associated with scaling and root planning in class II furcation defects in patients with chronic periodontitis treatment promoted significant bacterial reduction immediately after irradiation. The thermal effects at the external root surface and the morphological changes on the root canal walls after continuous (CW) and pulsed diode laser root canal irradiation at 810 nm was investigated. The temperature will not increase above the safety limit (10°C) for the periodontal tissues; however, after each treatment a 20second resting time should be considered to prevent an excessive temperature rise in the tissue when the laser is operating in either continuous or pulsed mode.

# 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 (LED) to provide a nonthermal, noninvasive, environmental safe treatment that can be useful in the Health Sciences

## Photobiomodulation

Low-power laser therapy (LPLT) is a treatment modality that is becoming more useful in medicine and dentistry although mechanisms underlying light effects are still not completely understood. Studies were carried out in vitro and in vivo to investigate the influence of LPLT on cell cultures, wound healing, temporomandibular disorders, thyroid gland and caries prevention.

- Cell cultures: LPLT increases epithelial cell growth rate in stressed cells by growth under nutritionally deficient states. This cell growth improvement is directly proportional to the number of irradiations; however, it was not enough to reach the full cell growth potential rate of Vero epithelial cell line observed when growing under nutritional regular condition.
- Wound healing: In vivo researches show that LPLT may accelerate cutaneous wound healing and the recovery of dental structures involved in cavity preparation at the predentine region even if a single laser treatment is performed. Besides, polarized red laser radiation affects skin repair by reepithelization stimulation, fibroblast differentiation, protein synthesis regulation, inflammatory process reduction and collagen bundle organization. In a clinical trial, LILT was an effective adjunctive treatment that appeared to promote healing following

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gingival surgery.

- Temporomandibular disorders: LPLT was more effective than Transcutaneous Electrical Neural Stimulation (TENS) in improving the amplitude of mandibular movement in patients with temporomandibular disorders.
- Thyroid gland: LPLT of the thyroid gland may affect the level of thyroidal hormones. A statistically significant hormonal level alteration between the first day and 7 days after the last irradiation was found.
- Caries prevention: Although LPLT before acidulated phosphate fluoride application appeared to diminish the caries progression, LPLT did not present any additional benefit compared with fluoride on the prevention of induced-dental caries in rats.
- Photosensitization: Photodynamic Antimicrobial Therapy (PAT), which involves the use of light sources with appropriate wavelength to kill cells previously treated with a photosensitizer drug, is an athermal, selective and new alternative to replace antibiotics, antimicrobials and chemical agents against microorganisms. PAT has been studied as a promising approach to eradicate oral pathogenic bacteria that cause endodontic diseases, periodontitis, and peri-implantitis.
- Peri-implantitis: In vivo studies indicate PAT as a more efficient, noninvasive method that could be used to reduce periodontal bacteria in peri-implantitis when compared to conventional therapy, with flaps elevated of mucoperiosteal mucosa for scaling the implant surface. It is a potential application since PAT in this study was as effective as a surgery, therefore avoiding this invasive and painful treatment.
- Periodontitis: Aggregatibacter actinomycetemcomitans is an important periodontal pathogen that can be photosensitized in vitro by red laser combined with malachite green, a dental plaque disclosing agent. Besides, the dye is photodegraded following irradiation and new non toxic photoproducts were formed in suspension.
- Endodontic diseases: PAT was effective for reducing Enterococcus faecalis in root canals and can be an adjunct to endodontic treatment. Our results suggest that the use of PAT as an adjuvant to the conventional endodontic treatment leads to a statistically significant further reduction of bacterial load and in particular reduces the amount of bacterial regrowth after 24 h compared to PAT or endodontic treatment alone. This result indicates that the combined therapies may reduce the need of antibiotic therapy on root canal treatment, overcoming microbial resistance, a major problem nowadays.

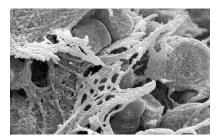


Figure 6. Electron-micrograph of the implant surface showing bacterial cells

## 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, highresolution cross-sectional images of internal microstructures of living tissues. We implemented an OCT system, based in an open air Michelson interferometer, using a femtosecond laser running at 830 nm as central wavelength and with 40 nm of bandwidth. This configuration provides 10 μm of longitudinal and lateral resolution and 3,000 µm of maximum scan depth in air. This system can be improved to be polarization sensitive, allowing measuring birefringent samples. As examples of possible applications, (where was possible to measure) simulated caries disease process (demineralization process by acid etching, and by bacteria process, Figure 7) were measured, making it possible the study of the caries dynamics.

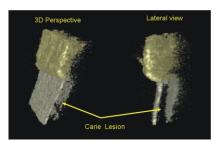


Figure 7. 3D reconstruction of human teeth for 11 days of caries disease process

This development resulted in a collaboration with a cosmetic company (NATURA) that has been supported this research. This collaboration allow us to have a commercial system working at 930 nm with longitudinal and lateral resolution of 6 µm, maximum penetration depth of 1,600 mm in air, and real time data acquisition (3 frames/sec) in maximum resolution (2000 x 512 pixels), up to 8 frames/sec are possible with lower resolution.

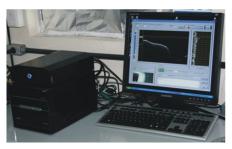


Figure 8. OCT system from Thorlabs Inc

## Laser Remote Sensing of the Atmosphere

The LIDAR technique is based on the emission of a collimated laser beam in the atmosphere and on the detection of the backscattered laser light by the suspended atmospheric aerosols and atmospheric molecules. The LIDAR technique, through its high temporal (from seconds to minutes) and spatial (less than 30 m) resolutions is a powerful tool to visualize in real time, the structure of the Planetary Boundary Layer (PBL) using the aerosols as passive tracers of the atmospheric dynamic processes. A backscattering LIDAR can thus provide information on the PBL's mixed layer depth, entrainment zones and convective cells structure, aerosol distribution, clear air layering, cloud-base altitudes, cloud statistics, atmospheric transport processes and other inferences of air motion. A groundbased elastic backscatter Lidar system was setup in the Laboratory of Environmental Laser Applications at the Centre for Laser and Applications (CLA). This system is operational since 2001 and has since then routinely collected data to monitor aerosols in the atmosphere as well the planetary boundary temporal evolution as an indication of pollutant dispersion conditions. Besides, beginning in 2007 the LIDAR system has been upgraded and two additional channels were added in order to achieve both Nitrogen and Water Vapor Raman signals in the atmosphere up to 4 km in altitude, this new feature provides this system an unique capability of not only qualitatively assessing these gaseous species but also quantitatively. Currently our group has collaborations with Lidar and atmospheric sciences groups in Greece (National Technical University of Athens), United States (Goddard Space Flight Center - NASA) and in Brazil (Insituto Nacional de Pesquisas Espacial INPE, Instituto de Pesquisas Meteorológicas IPMET/UNESP and University of São Paulo.

## **High Intens**ity Lasers

## Operation and optimization of the TW peakpower laser

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° 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. Even at lower peak powers, the ultrashort character of the pulses generates nonlinear phenomena, particularly those initiated by multiphotonic processes that generates free electrons. Ultrashort pulses with 10° GW peak power were utilized to generate color centers in fluoride crystals, allowing the study of their generation phenomena. The density of the centers created is higher than what is attainable with conventional ionizing radiation, resulting in the formation of aggregates that are stable at room temperature. The multiphotonic generation of free electrons also confers a nonselective character to the ablation of solid targets, which together with the almost nonexistent thermal effects, led us to develop a new method to determine the ultrashort ablation threshold of materials, and fully describe its formalism. The reduced thermal effects were also observed in Raman studies of ablated technological materials.



Figure 9: Hybrid Ti:Sapphire/Cr:LiSAF CPA system in operation, generating  $0.2^\circ$  TW peak power pulses

## Color center production by high intensity ultra short laser pulses in fluoride crystals

Ultrashort pulses with peak powers in the vicinity of 10 GW were used to create color centers in fluoride crystals grown in our facilities. It was possible to determine that the centers generation begins with a multiphotonic absorption, favored by the high intensity due to the short pulse duration, that neutralizes negative ions, that are kicked out of their sites in the crystalline matrix. Once the center are formed by electrons trapped in the crystalline field, they act as a probe to study the crystal structure. Once this vacation is formed, an electron is trapped in it, originating the basic color center. 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. The probe characteristic of the centers allowed the observation of different properties in each matrix, by the measurement of the centers absorption and emission spectra.

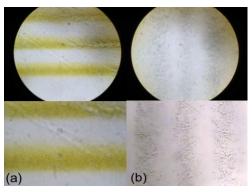


Figure 10: Optical microscopy of irradiated BLF samples with lower intensity: (a) BaLiF, grown by zone melting under HF with CCs, (b) BaLiF, grown by Czochralski under  $\mathrm{Cf_4}$ 

## **High Intensity Lasers**

# Implantation of the pump and probe optical technique to study ultrafast phenomena 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 system is excited by the pump pulse, 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. In 2007 this kind of pump and probe experiment was assembled in the Laboratory for High Intensity Lasers. To accomplish this goal it were acquired a set of Lock-in amplifiers, an ultra precise step motor, with driver, for building a delay line and a variety of first-class optomechanical components that together with the excellent infrastructure of the laboratory, will allows us to put in operation, in the Center for Lasers and Applications at IPEN-CNEN/SP a facility to do state of the art investigations of ultrafast phenomena in different systems (Figure 11). The initial tests in the setup are in progress.



Figure 11. Setup for studying ultrafast dynamic process with the pump and probe technique under development in Center of Lasers of IPEN

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Another important push to this activity are in progress conjunctly by CNRS and Fapesp, allowing the interchange of researchers between the Center for Lasers and Aplications and the Institut de Physique et Chimie des Materiaux de Strasbourg, CNRS/Strasbourg, França, for the study of ultrafast phenomena in different systems, metallic and magnetic materials, nanostructures amediumnd biological. This will be a pioneering setup to study the ultrafast magnetization dynamics in Brasil, a topic of growing interest in the international scientific community, due to the constant progress of materials science and the driving economical forces that comes from different applications that arise in the manipulation of ever more information in the ultrafast and ultrasmall scales.

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## Lasers Technology Program Team

#### Honor Mention and Awards

C.G. Farina; A. Garcez; M. Mori; Duarte, M.; M.S. Ribeiro; D.M. Zezell - "Effects of Low Intensity Laser Therapy (780nm) in Temporomandibular Disorders: Electromiographic, pain and bite force analysis" Golden Poster Award - International Conference on Lasers in Medicine - Timisoara-Romania, 2005

G.E.C. Nogueira; A.L.F. Nogueira; J.L. Lage-Marques; D.M. Zezell; F.T.O.P. Eduardo - "Determinação do Valor de Corte em Testes da Vitalidade Pulpar Dental via Fluxometria Laser Doppler", Prêmio - 1o. Lugar em Instrumentação, II Simpósio de Instrumentação e Imagens Médicas (II SIIM'2005) - São Pedro-SP, 2005

Ribeiro, A.C.; Nogueira, G.E.C.; Antoniazzi, J.H; Moritz, A.; Zezell, D.M. - "Thermal Effects of Laser Irradiation During Endodontics Treatment: Diode and Er:Cr; YSGG Lasers" - 3o. Encontro da Associação Brasileira de Lasers em Odontologia - Rio de Janeiro-RJ, 2005. Primeiro lugar pesquisa básica - oral

Soares, B.M; Prates, R.A.; Sousa, G.R.; Amorin, J.C.F.; Ribeiro, M.S.; Barbosa, M.P.; Hamdan, J.H. "Susceptibilidade de Cryptococcus neoformans var. gattii à terapia fotodinâmica"- 3o. Encontro da Associação Brasileira de Lasers em Odontologia - Rio de Janeiro-RJ, 2005. Menção Honrosa - oral

Ribeiro, A.C.; Mayer, M.P.A.; Nogueira, G.E.C.; Moritz, A., Zezell, D.M. - "Thermal Effects of Diode Laser during Endodontic Treatments" - Silver Young Researcher Award, 10th International Congress on Lasers in Dentistry - Berlin-Germany, 2006

Eduardo, F.P.; Mehnert, D.U.; Monezi, T.A.; Schubert, M.; Zezell, D.;Eduardo, C.P.; Marques, M.M. - "In Vitro Effect of Phototherapy with Low Intensity Laser (660 and 780 Nm) on Monkey Epithelial Cells (Vero) and HSV-1 in Culture"- Silver Basic Research Award, 10th International Congress on Lasers in Dentistry Berlin-Germany, 2006

Yamada Jr., A.M.; Hashimoto, M.C.E.; Cai, S.; Tanji, E.Y.; Ribeiro, M.S. - "Lethal photosensitization following blue light emitting diode and acid rhodamine B. In vitro study on Streptococcus mutants"- Silver Researcher Award, 10th International Congress on Lasers in Dentistry - Berlin-Germany, 2006

Emilio, C.R.; Dagli, M.L.Z.; Dutra, F.; Bechara, E.J.H.; Gioso, M.A.; Larsson, C.E.; Pinotti, M.; Zezell "Novo protocolo de terapia fotodinâmica utilizando-se o metil aminolevulinato e dtpa no tratamento de carcinoma espinocelular em felinos"- Prêmio "José Luiz Guerra em Oncologia Veterinária"- Faculdade de Medicina Veterinária e Zootecnia da Universidade de São Paulo, 2006

Vladimir Dourado Poli; Denise Maria Zezell, Anderson Zanardi de Freitas, Ricardo Elgul Samad, Nilson Dias Vieira Jr. - Prêmio Painel "Estudo da Interação de Laser de Femtossegundos com o Tecido Ósseo" Sociedade Brasileira de Pesquisa Odontológica (SBPqO), 2006

Garcez, A.S.; Núñez, S.C.; Ribeiro, M.S. - Prêmio Francisco Degni - 20. lugar na categoria Fórum Clínico

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com o trabalho "Terapia fotodinâmica como complemento ao tratamento endodôntico convencional", 24o. Congresso Internacional de Odontologia de São Paulo - APCD, 2006

Zanardi, P.; Aldrigui, J.M.; Cadioli, I.C.; Rodrigues, C.R.M.D.; Nogueira, G.E.C.; Wanderley, M.T. "Avaliação da Fluxometria Laser Doppler com freqüências de 3kHz e 15kHz em incisivos decíduos vitalizados, Painel IC Premiado na área clínico e social, XIV Reunião de Pesquisa e XI Seminário de Iniciação Científica da FOUSP, 2006

Garcez , A.S.; Nunez, S.C.; Zezell, D.M.; Hamblin, M.R.; Ribeiro, M.S., "Antimicrobial effects of photodynamic therapy on patients with necrotic pulps and periapical lesion" - International Conference on Lasers in Medicine - World Federation for Lasers in Dentistry, Timisoara-Romania, Golden Poster Award, 2007

Homenagem à Profa. Denise M. Zezell pelo trabalho pioneiro em Terapia Fotodinâmica de Tumores no Brasil. Workshop em "Terapia Fotodinâmica: Integração dos Aspectos Moleculares, Tecnológicos e Aplicações na Área de Saúde", São Pedro-SP, 2007

Zanardi, P.; Aldrigui, J.M.; Cadioli, I.C.; Rodrigues, C.R.M.D.; Nogueira, G.E.C.; Wanderley, M.T. "Avaliação da Fluxometria Laser Doppler com freqüências de 3 e 15kHz em incisivos decíduos vit. e desvit.", Menção Honrosa, 15 SIICUSP - Símpósio de Iniciação Científica da USP, 2007