

Making Light Matter

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Guest Editorial

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1. Field and Challenges

Lasers permeate our daily lives in many ways and specifically materials processing with lasers is key for numerous applications in science, technology, medicine, and industry. During the last years, different trends have been observed in the field of laser materials processing, i.e., (1) the pursuit of reaching extremely small scales and ultimate resolution in laser nanostructuring [1], and (2) an up-scaling of the processing rates for meeting industrial demands [2]. Both trends are driven by the rapid advancements in ultrafast laser technologies in terms of throughput, complexity, and interoperability — currently manifesting in a Moore-law-like scaling law of the average output powers with time — and, at the same time, benefiting from the latest developments in data-driven processes. Most applications of laser processing do manifest between these two extremes and are critically impacted by latest developments in material sciences.

Europe plays a major role in each of these fields, as it successfully combines an excellent scientific environment with advanced technological capabilities and, thus, provided the ground for developing a successful industry in laser technologies. Long lasting scientific conferences, such as the *Spring Meeting* of the *European Materials Research Society* (E-MRS) [3] represent an important platform to exchange the latest developments among those fields.

2. The Conference Symposium

This Special Issue represents the proceedings of the Symposium L "Making light matter: lasers in material sciences and photonics" held from May 29th to June 2nd 2023 in the Congress & Exhibition Centre in Strasbourg, France, during the 2023 Spring Meeting of the E-MRS.

This traditional symposium addressed in its 40th anniversary edition the field of laser materials processing and laser-matter interaction, including laser- and plasma-based materials synthesis, surface structuring and functionalization, 3D processing, additive manufacturing, and process analytics and materials diagnostics with the special emphasis on the micro- and nanoscale. The symposium included fundamental and applied topics and provided a bridge between science and technology for scientist promoting research in the field of physics, materials science, chemistry, biology, and life-science.

Symposium L received more than 140 abstracts submitted from 23 countries distributed over five continents. Ten abstracts were selected as invited talks, which were given along with 85 oral presentations in 18 sessions devoted to industrial laser machining, ultra-short and ultra-high power laser interaction with matter, laser additive manufacturing, biological laser surface engineering, laser-induced periodic surface structures (LIPSS) and direct laser interference patterning (DLIP), laser beam engineering for surface processing, laser surface texturing applications, laser-induced plasma and applications, laser-induced melting and crystallization, laser-induced forward transfer (LIFT), pulsed laser deposition (PLD), etc. Two of these sessions were organized jointly together with the Symposium Q "Advanced functional films grown by pulsed deposition methods — II". Additional 47 contributions were presented in a poster session.

The invited speakers of Symposium L were (listed alphabetically): Dave H.A. Blank (jointly with Symposium Q), Douglas B. Chrisey, Maria Farsari, Mario García Lechuga, Yoshio Hayasaki, Anne-Marie Kietzig, Alexandre Mermillod-Blondin, Masaaki Sakakura, Jakub Siegel (not presented due to illness), Peter Simon.

A committee selected two outstanding oral presentations of excellent graduate students (Laura Rammelt and Tommaso Serra), who received the *2023 EMRS Symposium L Young Researcher Awards*. During the symposium's poster session, additional three poster prize awardees were selected by the session chairs from the presenting students. These *Outstanding Student Poster Awards* were awarded to Christian Rainer (1st place), Valentina Iacono (2nd place), and Malen Razkin Bartolomé (3rd place).

The symposium organizers would like to thank the two *symposium assistants* Malen Razkin Bartolomé and Daiwei Zhang, and the 14 *session chairs*, who all greatly contributed to the success of this symposium (ordered alphabetically): Dave H.A. Blank, Germán Francisco de la Fuente, Thibault J.-Y. Derrien, Maria Farsari, Mario García Lechuga, Stephan Gräf, Emmanuel Haro-Poniatowski, Anne-Marie Kietzig, Alexandre Mermillod-Blondin, Johannes Pedarnig, Maria Pervolaraki, Esther Rebollar, Peter Simon, and Pol Sopeña.

The symposium organizers greatly acknowledge the support of five companies (ordered alphabetically): *Amplitude Systèmes* (France), *BeamXpert* (Germany), *Biomimetic* (Greece), *LAYERTEC* (Germany), *OPTOPRIM* (France). Their contribution made it possible to present the poster awards to outstanding students and to provide financial support for students and invited speakers.

The proceedings of the symposium L were organized as a Special Issue in the journal *Physica Status Solidi A* (PSSA) together with the Wiley in-house Editor Marc Zastrow. Out of 20 submitted manuscripts 14 were published in PSSA after regular peer-review. Three more selected papers related to invited talks of the symposium were submitted to the journal *Laser & Photonics Reviews* (LPR) and published after regular peer review.

The guest editors would like to thank all peer reviewers of the articles in this special journal issue and the editorial staff of PSSA and LPR for their support and guidance.

3. Selected Highlights of the Symposium

The vast majority of manuscripts published in this cross-journal Special Issue are devoted to fundamentals or applications of laser direct writing, laser texturing, and surface engineering. A particular focus here is on so-called *laser-induced periodic surface structures* (LIPSS) that form in a self-organized way during the materials processing with lasers and that feature numerous possibilities of surface functionalization applications.

Jürgens et al. (article number 2301114) reviewed in LPR the diagnostics of laser-induced plasmas in solid dielectrics. Such electron-hole plasmas are formed in the bulk of transparent materials (glasses, crystals) during the irradiation with intense, ultrashort laser pulses. They play a central role in diverse areas, such as high-harmonic generation, optical bulk modification and damage, and laser processing for 3D photonics applications. The authors thoroughly analyze the currently available experimental techniques as well as promising approaches for exploring the formation, dynamics, and subsequent relaxation of such a confined, microscopic plasmas in bulk transparent solids.

Zyla and Farsari (article number 2301312) presented a *Perspective* article in LPR on the current trends and future developments in the field of laser-based 3D-printing via multiphoton lithography (MPL). Along the trends, the authors highlighted recent advancements, and potential applications. The authors delved into challenges faced by MPL, providing insights on how to overcome them for improving the efficiency of MPL processing. Future

opportunities and perspectives of MPL in advanced additive manufacturing (AM) at the micro- and nanoscale were outlined across various research fields and in industry.

García Lechuga et al. (article number $\underline{2301327}$) uncovered in LPR their original research on the amorphization and ablation of crystalline silicon using ultrafast lasers with different irradiation wavelengths (UV - IR) and pulse durations (14 fs - 130 fs). Starting from a thorough literature review, the authors extended the laser processing parameter space with the aim of increasing amorphization layer thickness (typically some tens of nm) for meeting requirements in silicon photonics. The amorphization and the ablation threshold fluences both significantly increased from $\lambda \sim 260$ nm to 1030 nm irradiation wavelength, followed by a near-constant level at NIR wavelength up to $\lambda = 3000$ nm. Differences in the amorphization layer thicknesses on Si(100) and Si(111) are discussed, identifying an anomalously large fluence range for laser-induced amorphization in the UV.

Rathnayaka and Kietzig (article number 2300723) studied the adhesion of thermally cured epoxy resin on fs-laser textured (224 fs pulse duration, 1030 nm center wavelength) aluminum alloy sheets. This included the measurement of the contact angle of the epoxy resin as well as measurements of the maximum breaking stress of differently laser-textured surface topographies (tapered circular dimples, hexagonal holes, near-wavelength sized LIPSS) and with the polished flat surfaces as a reference. All laser-textured surfaces increased the epoxy resin wettability and adhesion on the aluminum alloy. This increase in adhesion was attributed to an increase in surface area and the resulting mechanical interlocking.

Kraft et al. (article number 2300710) followed a similar approach of continuous wave (cw), ns- and fs-laser surface texturing (1030 or 1070 nm wavelength) of dimples periodic ablation lines, or crossed line grids on steel surfaces for improving the adhesion of the subsequent

thermal spray-coating process of different metallic materials (tungsten, stellite 6). Their focus was on the high-speed laser processing employing polygon-scanners, allowing to manufacture up to 1300 ablation lines per second. *In-situ* heat-accumulation measurements were performed by a fast infrared thermal monitoring system. *Ex-situ* standardized tensile adhesion tests were performed to characterize the deposited coating layer, revealing tensile strength $R_{\rm H}$ between 34 and 73 MPa. Calculations of the possible area structuring rates of the laser process revealed that processing rates of hundreds of cm² per minute can be achieved, depending on the texture geometry and the laser source. Via process optimization (e.g., multi-spot parallel processing or the use of laser pulse bursts) areal processing rates in the m² per minute range might be achieved.

De Nalda et al. (article number 2300721) addressed the open question, how much a temporal chirp of fs-laser pulses affects the formation near-wavelength sized LIPSS (so-called *low spatial frequency LIPSS*, LSFL)? For that, they irradiated thin films of poly(trimethylene terephthalate) (PTT) deposited by spin coating on silicon by multiple fs-laser pulses (50 fs pulse duration, 800 nm center wavelength, 1 kHz pulse repetition rate, $5 \times 10^4 - 20 \times 10^4$ pulses) in a spot-processing geometry and characterized the surface topography by atomic fore microscopy. For stretched pulse durations between 50 and 200 fs neither the spatial periods of the LIPSS (ranging between ~650 and ~850 nm) nor their amplitudes (modulation depth, ranging between ~75 and ~300 nm) indicated a significant dependence on the temporal chirp or its sign (positive chirp: "red" spectral components are arriving first to the sample; negative chirp: "blue" spectral components are arriving first).

Dominic et al. (article number <u>2300703</u>) investigated sub-wavelength-sized *high spatial* frequency LIPSS (HSFL) formed upon fs-laser irradiation (60 fs pulse duration, 800 nm center wavelength, 1 kHz pulse repetition rate) of tungsten in ultrahigh vacuum conditions. An

analysis of the surface topography along with two-temperature model coupled molecular dynamics simulations reveal that the HSFL formation on tungsten originates from laser-induced thermal stresses that are involving both, surface tension and tensile forces. Moreover, the experimental observation of sub-surface cavitation effects points towards a hydrodynamics-based origin for these nanostructures.

Müller et al. (article number 2300719) explored in a multi-method surface analytical approach the influence of the laser pulse repetition rate and the number of passes (over-scans) on the surface topography and chemistry during the ultrashort pulsed laser processing (925 fs pulse duration, 1030 nm center wavelength) of sub-100 nm sized HSFL on Ti-6Al-4V titanium alloy in ambient air environment. The authors demonstrated that it is possible to increase the processing speed of HSFL to pulse repetition rates up to 400 kHz in an industrially suitable manner while simultaneously avoiding limiting heat accumulation effects. Hard X-ray photoelectron spectroscopy (HAXPES) and depth-profiling Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) revealed that the several tens of nanometer shallow HSFL nanostructures on Ti-6Al-4V are accompanied by a graded near-surface oxide layer here that extents a few tens of nanometers into depth and consists mainly of TiO₂ with a small admixture of Ti₂O₃.

Madapana et al. (article number 2300610) also used Ti-6Al-4V titanium alloy for scanprocessing of areas filled with parallel micrometric ablation lines that are covered with
nanometric surface features (including LSFL). The ultrafast laser processed surfaces (100 fs –
3 ps pulse duration, 800 nm center wavelength, 10 kHz pulse repetition rate) were
subsequently characterized microscopically and surface analytically and corrosion test were
performed by electrochemical impedance spectroscopy (EIS). The authors reported the
presence of strain in the grains of the irradiated material for depths up 150 μm, aligned in the

direction of some specific crystallographic planes. Additionally, a more than 100 nm thick laser-induced oxide layer consisting of TiO₂ and Ti₂O₃ was found at the surface. Moreover, the authors concluded from the EIS measurements that the laser processing resulted in a thick protective passive layer (being responsible for a higher corrosion resistance), covered by a thin outer porous layer.

Voss et al. (article number 2300920) combined electrochemical pre- and post-anodization steps, with the ultrashort pulsed laser processing (925 fs pulse duration, 1030 nm center wavelength) of hierarchical micro-spikes covered with nanoscale laser-induced periodic surface structures (LIPSS) on Ti-6Al-4V in ambient air environment Scanning electron and white light interference microscopy, X-ray diffraction analyses, micro-Raman spectroscopy and hard X-ray photoelectron spectroscopy are employed to evaluate morphological, topographical, structural, and chemical surface alterations for processing conditions previously explored for applications in medical implants.

Lorenz et al. (article number 2300482) combined the ultrashort pulsed laser surface texturing (10 ps pulse duration, 1030 nm wavelength, 100 kHz pulse repetition rate) of stainless steel (AISI 304) with additional chemical modification through a self-assembled monolayer of a fluorinated, phosphonic acid-modified alkane, resulting in the formation of a hierarchical micro-nanostructured surfaces (including LSFL) with highly hydrophobic properties. Additional exposition to mechanical wear led to the abrasive removal of parts of the topmost surface layers, causing a reduction of the water contact angle. This surface wettability change is influenced by the design pattern of the hierarchical surface, the laser processing parameters, and the strength of the abrasive wear. The authors developed a model that is capable to explain several of the observed trends.

Nedyalkov et al. (article number 2300478) studied the influence of the ambient pressure on the electrical resistance of surface tracks processed by ps-laser pulses (10 ps pulse duration, 355 or 1064 nm wavelength, 1 kHz pulse repetition rate) at the surface of AlN ceramic in different vacuum conditions (10^{-4} torr up to atmospheric pressure). It was demonstrated that under certain conditions the ps-laser processing can turn the irradiated material electrically conductive. A decrease of the resistivity by up to a few orders of magnitude, from about tens of $k\Omega$ to a few Ω was observed, depending on the processing conditions. Laser processing in the presence of nitrogen gas flow led to a reduction of the tracks' resistance. The systematically explored effects were attributed to the formation of an Al-enriched layer and its oxidation either during (at high pressures) or after (at low pressures) laser-induced material decomposition and surface cooling.

Ogor et al. (article number 2300486) studied the speed-up and parallelization of the additive manufacturing approach based on multi-photon polymerization. The authors developed a phenomenological digital model of the photochemical process for parallelized multiphoton-based fabrication of three-dimensional structures by imaging a 1920 × 1080 pixels spatial light modulator onto an ultrasensitive polymeric triplet–triplet annihilation resist. In that approach, optical propagation and chemical diffusion effects are involved. For single laser exposures, the simulation provided reasonable predictions of 2D and 2.5D microscale structures and helped to improve 3D structures' resolution upon two laser exposure treatments.

Heinke et al. (article number 2300485) combined the process of laser cleaning with atmospheric pressure plasma jet (APPJ) processing of different multi-component optical materials (N-BK7, Zerodur[®]). The laser processing step enables the removal of nonvolatile reaction products formed during the APPJ etching from metal oxides contained in the optical

glasses. The processed samples were examined by scanning electron microscopy. Sets of optimized laser processing parameters (laser fluence, pulse number ranges) were identified that allowed the removal of the APPJ residual layer without any observable damage of the substrate material.

Lorenz et al. (article number $\underline{2300481}$) studied the fabrication of plasmonic microcubes by laser ablation of gold nanoparticle (Au-NP) loaded acrylates. The samples were prepared by UV light curing of an Au-filled acrylate film and subsequent thermal treatment, leading to the precipitation and growth of 1 to 10 nm sized Au-NPs as characterized by transmission electron microscopy (TEM) and optical emission spectroscopy. Additional laser processing (sub-ps pulse duration, 343 nm wavelength, 100 kHz pulse repetition rate) of the Au-NP acrylate films in a cross-hatched line processing scheme using a moderately focused laser beam (Gaussian beam radius $\sim 16~\mu m$) then led to the formation of gold micro-cuboids with sizes down to 15 μm . This concept allows an easy and large-scale fabrication of microstructures with independently adjustable plasmonic properties.

A working safety aspect of growing relevance was tackled by Kraft et al. (article number 2300725). The ever-growing pulse repetition rates of high energy ultrashort laser pulses can drive the secondary emission of hard X-rays during the laser machining of technically materials over the (country specific) regulatory safety limits. The article reports on a set of experiments using differently laser-pretreated stainless steel (AISI 304) sheets processed with high repetition rate ultrashort laser pulses (600 fs pulse duration, 1030 nm wavelength, 35 μJ laser pulse energy, 0.5 to 2.0 MHz pulse repetition rate) focused by a 167 mm focal length f-theta objective. The measurements showed an influence of the surface roughness and the number of laser scans on the laser-induced X-ray skin dose rate. Additionally, the dependency of X-ray emission on the scan direction relatively to the laser beam polarization state was

explored. The authors concluded that in almost every of their situations the laser-induced secondary X-ray emission exceeded the German legal limitation of 1 μ Sv/h in 10 cm distance. In such circumstances protection measures must be taken to shield against this hazardous ionizing radiation, while operating the laser system.

In a guest contribution from the Symposium E "Carbon- and/or nitrogen-containing thin films and nanomaterials", Naser et al. (article number 2300750) explored the fast large-area characterization of silicone films by Coherent Raman Scattering (CRS) imaging, providing a chemically specific contrast. The authors compared their results to that of conventional (spontaneous) Raman (SR) spectroscopy, stimulated Raman scattering (SRS), and coherent anti-stokes Raman scattering (CARS).

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The Guest Editors and Symposium Organizers



Jörn Bonse

Jörn Bonse received a Diploma degree in Physics from the University of Hannover in 1996, and a Doctoral degree from the Technical University of Berlin in 2001. He held research positions at the Laser Zentrum Hannover, the Max-Born-Institute in Berlin, the Spanish National Research Council in Madrid, and was appointed as a Senior Laser Application Specialist at Newport Spectra-Physics. Currently, he is a Senior Scientist at the German Federal Institute for Materials Research and Testing (BAM). His research interests include fundamentals and applications of laser-matter interaction, laser-induced periodic surface structures, surface functionalization, time-resolved optical techniques, and laser processes in photovoltaics.

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