UT APRAD SOR, Excimer Group
Progress Report 2010

Motivation and frame

In the extreme ultraviolet (EUV) spectral region (20 eV - 280 eV) laser-plasma and discharge-plasma sources emit energies per pulse and repetition rates sufficient to be considered a valuable alternative to synchrotrons and short-wavelength free-electron lasers (X-FELs) in many irradiation experiments, namely when the peak power and the brightness are more important than the average power and when it is not required a narrow spectral band. In fact, synchrotron beam-lines are often over-subscribed by factors of three or more; despite the widely-acknowledged advantages of synchrotron radiation, there is not enough to go round. Due to the cost of such sources (more than 100M€ to build, about 1M€ per beam-line per year to run) this is likely to remain the case. The same considerations apply for short-wavelength FELs that are being developed in different countries, as their size and costs are similar to synchrotrons. In this strategic frame, laser-plasma and discharge-plasma sources available in the market and in particular those described in this Report are alternative, table-top, cheaper, and more accessible sources which can offer at least some of the synchrotrons performance.

The multipurpose Laser-Plasma-source and Micro-Exposure-Tool EGERIA

The Laser-Produced-Plasma facility called EGERIA (**E**xtreme ultraviolet light **G**eneration for **E**xperimental **R**esearch and **I**ndustrial **A**pplications) is based on the use of a XeCl excimer laser whose beam is focused on a metal tape placed in a vacuum chamber, thus creating a plasma which recombines and emits pulsed radiation. The features of the emitted radiation (i.e. spectral range, peak intensity, pulse duration in the nanosecond range) can be managed by adjusting the laser beam intensity on target and by selecting the target material. The spectral range spanned by EGERIA is between 20 eV and 1800 eV. A debris mitigation system (two ENEA patents) reduces by three orders of magnitude the debris emitted by the tape target, thus allowing almost clean irradiations in the EUV. More information are detailed in the website: www.frascati.enea.it/Impianti/SorgenteRi-X%20da%20laser-plasma/SorgenteR-Xdalaser-plasma.html).

Presently, the plasma source EGERIA is an international-level facility that has been used by biologists (*in vivo* X-ray microscopy of bacteria, DNA repair, micro-radiography), hysicists (heavy ions generation, X-ray atomic spectroscopy, micro-devices for photonics, contact-lithography, imaging of sub-micrometric structures on optically active materials) and industry (writing arbitrary pattern on anti-counterfeiting tags).

The Micro-Exposure-Tool (MET) EGERIA is a complex apparatus comprising the EGERIA source, an optical collector and an accurate optical projection system able to print pattern with a spatial resolution better than 100 nm. The MET-EGERIA was awarded of the prize of Excellence ENEA 2008, and in 2010 it has been evaluated by the Italian Ministry of Education, University and Research (MIUR) as “Emerging Project” in the National Roadmap of Research Infrastructure.

The MET-EGERIA is used for projection- lithography to generate pattern with a sub-micrometer spatial resolution.

Figure 1 shows a top view of the EGERIA source chamber and of the MET chamber. Both vacuum chambers are placed in a clean room, as shown in Fig. 2.

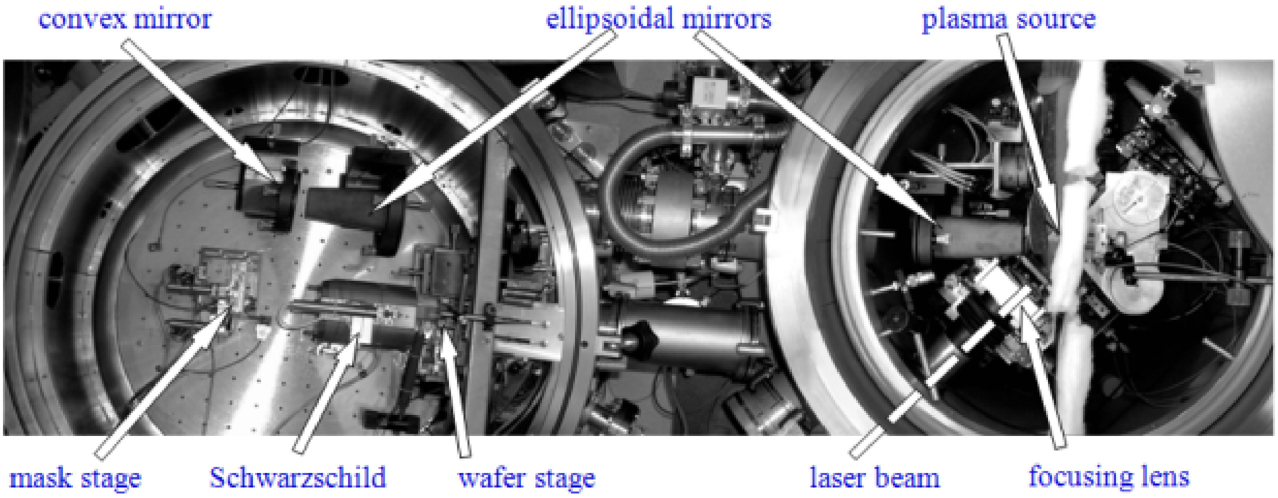

Figure 1 Top picture of the EGERIA vacuum chamber (right) where the laser-plasma is generated and of the Micro Exposure Tool high-vacuum chamber (left) where sub-micrometer pattern are written either on a photoresist or on a fluorescent material.

Figure 2 The EGERIA source (on the right) and the Micro Exposure Tool (on the left) inside the clean room ISO 7 (10.000).

During 2010, the laser plasma source EGERIA was used to irradiate several samples in the extreme ultraviolet spectral region (20 eV < hν < 280 eV) in cooperation with European Institutes and with other ENEA Laboratories. EGERIA has been recently implemented with new absolute photodiodes, in order to obtain absolute EUV dose measurements with an uncertainty of few percent.
A particularly interesting experiment has been carried out in the frame of a collaboration with the Birmingham University (UK). Different samples of a new kind of negative photoresists for extreme ultraviolet lithography (EUVL), synthesized at the Birmingham University, have been irradiated at

different EUV dose levels in order to obtain the photoresist response curve. The results (see Fig. 3) are still under analysis.

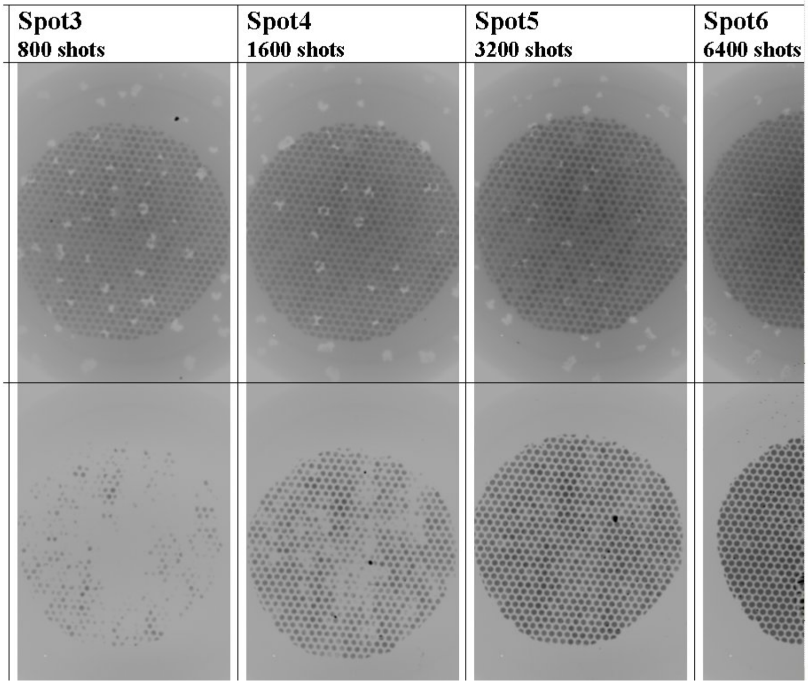

Figure 3: Negative photoresists of type "A" (upper row) and of type "B" (lower row), from Birmingham University, exposed to different EUV dose values through a mask having hexagonal holes placed at a distance of 1 mm from the resist. The photoresists are observed under an optical microscope in reflection mode (after development of the resists). The darker regions correspond to higher photoresist thickness.

From Fig. 3, the different sensitivity and contrast of the two types of photoresist are evident. Further experiments in cooperation with the Birmingham University are planned after completing the analysis of these first results.

It is worth noticing that the research on innovative photoresists for EUVL has a great relevance in the semiconductor roadmap (http://www.itrs.net/), where the issue of high-sensitivity and high-spatial resolution resists is of paramount importance.

Discharge-plasma source of EUV radiation

A xenon Discharge Produced Plasma (DPP) EUV incoherent radiation source was jointly designed by ENEA Frascati and the Physics Department of L'Aquila University in the frame of the FIRB-EUVL project (2003-2008) and was assembled and tested in L'Aquila University. In April 2010 the apparatus has been transferred to our laboratory, where it is operating since November 2010 after several improvements and optimizations, in order to obtain a stable and reliable operation, see Fig. 4. In particular, the Xe injection system has been rebuilt, and the discharge modulator and circuit diagnostics have been improved. In addition, the vacuum chamber devoted to irradiation experiments and target positioning has been modified and connected to the source to obtain a larger radiation cone available for irradiations.

The DPP is based on the generation of a xenon plasma column (10 mm in length and 0.5 mm in diameter) in an alumina capillary tube, as shown in Fig. 5.

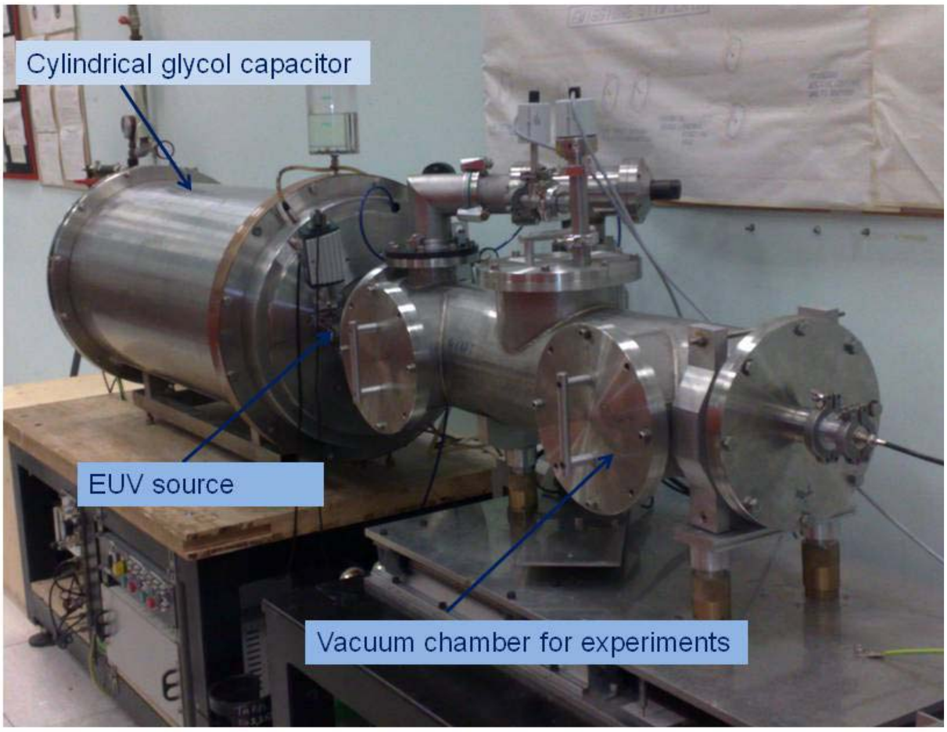

Figure 4. Photograph of the DPP operating at the ENEA Frascati Research Centre, with the main components highlighted.

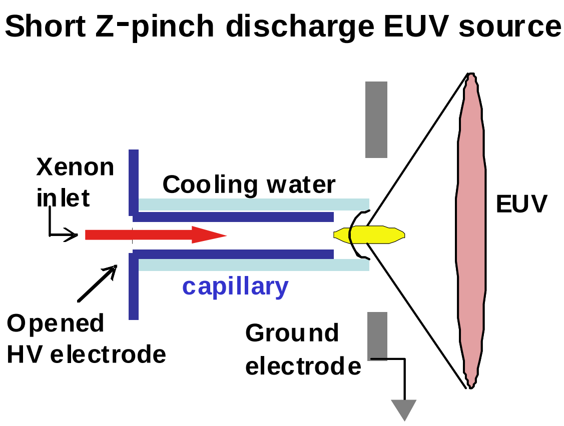

Figure 5. Schematic of the DPP shown in Fig. 4.

After evacuating the capillary down to a residual pressure of about 10^{-6} mbar, a small Xe flow (~ 5 standard cubic centimeter per minute) is injected, maintaining an equilibrium pressure of about $5{\cdot}10^{-3}$ mbar in the vacuum chamber connected to the source. The Xe plasma is generated by a short and intense current pulse (10 kA, 250 ns) flowing in the capillary filled by pre-ionized low pressure Xe. The current pulse is obtained by properly discharging in the capillary a 50-nF glycol low-inductance cylindrical capacitor, charged up to (20 - 25) kV. The plasma, pinched towards the capillary axis by the magnetic field produced by the high-current, is heated up to a temperature of (30 - 40) eV and after recombination it emits radiation in the (10 – 20) nm wavelength region, as shown in Fig. 6.

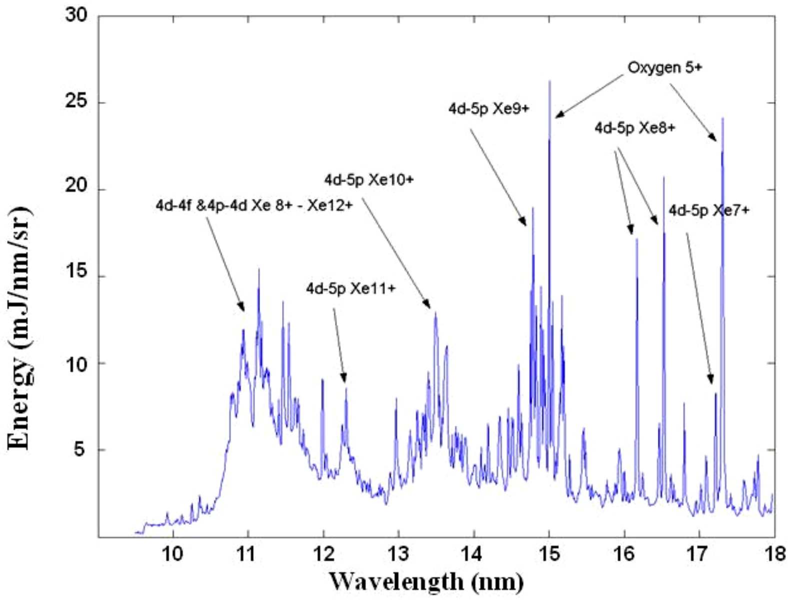

Figure 6. Typical spectrum of the radiation emitted by the DPP.

At a repetition rate of 10 Hz, the source stability is better than 5% r.m.s. The conversion efficiency of the energy stored in the capacitor into EUV radiation is 0.3% on 2π sr at λ = 13.5 nm (which is the wavelength of interest for the next generation EUV lithography) on a 2% bandwidth. The EUV radiation pulses have a 60 ns FWHM (see Fig. 7) and an energy of ~ 4 mJ/sr/shot at λ = 13.5 nm within the 2% bandwidth, whilst the energy emitted on the whole band (10 – 20) nm is about one order of magnitude larger.

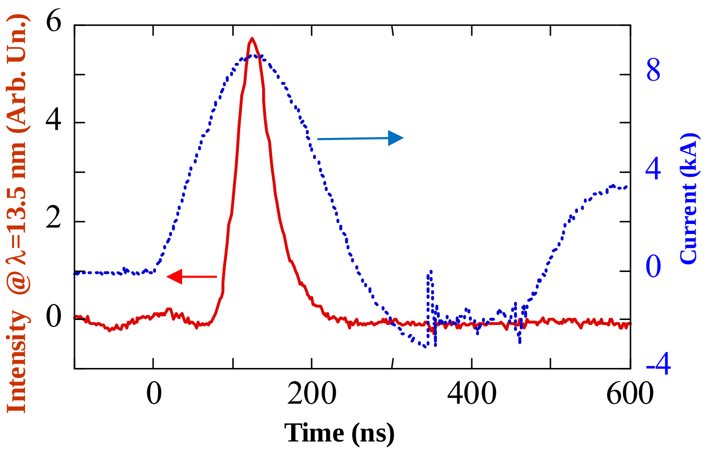

Figure 7. Time evolution of the discharge current and of the radiation emission.

The emitted radiation can be collected in a 60° aperture-cone in the vacuum chamber dedicated to the experiments. The wavelength and the shape of the emission peaks depend on both the nature and flow of the used gas and on discharge parameters, so that the source can be optimized for different specific applications like imaging, microscopy, spectroscopy, microlithography, photo resist testing as well as color centre generation, material ablation, EUV optics diagnostics, X-ray fluorescence, and so on. As a consequence, the DPP frames within the laboratory research line on the EUV radiation generation and relevant applications. It may greatly improve the range of future collaborations and R&D activities.

In addition to the work for a further characterization and optimization, it is planned to use the DPP in the framework of the anti-counterfeiting technique developed and patented by ENEA.

Invisible writing method as a novel anti-counterfeiting technique

A recent update of the Organisation for Economic Co-operation and Development (OECD) has estimated in $250 billion (in 2007) the worldwide value of international trade in counterfeit and pirated goods. This means that the available anti-counterfeiting techniques (fluorescent and thermo-chromatic inks/dyes, demetallization, radio-frequency, surface engraving, micro-texts and holograms) used so far do not offer a good protection from unauthorized copying or replication. As a consequence, up till now there is no absolute certitude that the documents, objects, currency, identity/credit/debit cards, commercial/artistic objects, forensic documents, dangerous wastes, pharmaceutic products, copyright protection systems protected by existing anti-counterfeiting techniques are genuine.

Using the short-wavelength radiation emitted by EGERIA, we have exploited the capability to write an invisible image on a fluorescent film that can be used as a tag for identification and traceability. This is a novel anti-counterfeiting system that prevents cloning and allows a security degree tailored on the customer's demand, up to a marking that is impossible to counterfeit. The reading technique comprises a small device that catches the invisible image and, if required, provides a digital decoding of the same image. The reading device has the capability to communicate with local or remote computer systems, and, if required, it can be based on a simple mobile phone digital camera.

During 2010, we made a prototype of anticounterfeiting tag applied to a plastic substrate, commonly used for credit or identity cards. The ENEA MNF Lab deposited a thin film of luminescent material on the plastic. After the deposition, we exposed the film to the EGERIA radiation trough a contact (transmission) mask, thus transferring an invisible image on it, and then the card was protected by a standard 25-μm-thick plastic coating. The final result is shown in Fig. 8. The invisible pattern can be observed only using the specific ENEA patented reading technique.

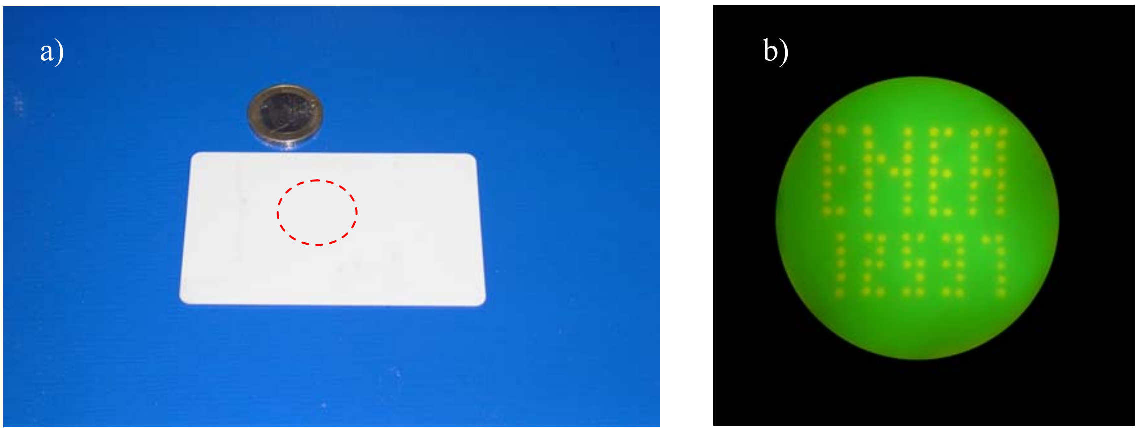

Figure 8. a) Picture taken under natural light illumination of a standard plastic card (85 mm×54 mm) with an invisible mark within the dashed area written by the radiation emitted by EGERIA. b) picture of the same dashed area shown in a), but with the pattern made visible by means of the compact and portable ENEA reading system prototype.

Linen textile coloration by nanosecond Laser pulses

The main interest in obtaining a permanent yellowish coloration of linen textiles is the search of a mechanism possibly leading to the formation of the Turin Shroud image: in fact, to date no one, not even with state-of-the-art technology has ever duplicated the Shroud image in all its chemical and physical characteristics, and the very thin (about 200 nm) coloration depth is one of the most difficult to replicate features of the Shroud image.

During 2010, in collaboration with ENEA MNF Laboratory we obtained experimental results showing that a short and intense burst of 193-nm excimer laser radiation provides a linen coloration having many peculiar features of the body image embedded into the Turin Shroud, including the hue of color, the color penetration depth and the lack of fluorescence, see Fig. 9.

Most importantly, thanks to the accurate characterization of both laser irradiation parameters and coloration results at the fiber level, we have recognized distinct physical and photo-chemical processes that account for both the experimentally achieved coloration and latent coloration. These processes may have played an important role in the generation of the body image on the Shroud of Turin.

During 2010 these results have been published in international journals, and their relevance echoed by several media (newspapers, websites, telecasts) pushed a group of Shroud's scholars (members of the international Shroud Science Group) to assign to ENEA the organization of the International Workshop on the Scientific approach to the Acheiropoietos Images (IWSAI 2010) that was held at the ENEA Frascati Center 4 through 6 May, 2010.

Figure 9 Microscope view of a single fiber of linen colored after excimer laser irradiation at λ = 193 nm. The mechanical damage in the middle shows a colorless medulla, suggesting the color penetrates only the primary cell wall of the fiber, which is about 0.2 μm thick. The fiber has an average diameter of 20 μm.