4.1 Short-wavelength Sources and Applications

In the extreme ultraviolet (EUV) spectral region (20 eV – 280 eV) laser-plasma and discharge-plasma sources can produce 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 applications, namely when the peak power and the brightness are more important than the average power and when a narrow spectral band is not required. In past years the SOR Laboratory has developed the laser-driven plasma source EGERIA, a facility that has been used by biologists (in vivo X-ray microscopy of bacteria, DNA repair, micro-radiography), physicists (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). A Micro-Exposure-Tool (MET) for micro- and nano-lithography has been developed to be used with the plasma source EGERIA. The MET is a complex apparatus comprising a debris mitigation system, an optical collector and an optical projection system able to print a pattern with a spatial resolution better than 100 nm. The MET-EGERIA was awarded the prize of Excellence ENEA 2008 and was included as “Emerging Project” in the National Roadmap of Research Infrastructures in 2010.

Fig. 4.2.1 shows a top view of the EGERIA source chamber and of the MET chamber.

Since November 2010 a xenon Discharge Produced Plasma (DPP) soft X-ray/EUV source, jointly designed by ENEA Frascati and the Physics Department of L'Aquila University, is operating at the SOR Laboratory. A photo of the DPP is shown in Figure 4.2.2.

In the following we describe the experimental activities carried out in 2014, concerning, on one side, the EUV radiation generation and its applications, and, on the other side, the development of devices for the positioning of concentrating solar systems, exploiting the laboratory expertise in lasers and optics.
4.2.1. Discharge-plasma source of EUV radiation

During 2014 the project entitled “New materials for direct nanopatterning and nanofabrication by EUV and soft X-rays exposures”, funded by CARIPLO Foundation, has been carried on. According with the project program, different innovative photoresists, synthesized by the project partners and characterized by particular optical and mechanical properties, have been exposed to the Extreme Ultraviolet (EUV) radiation emitted by the ENEA Discharge Produced Plasma (DPP) source. In order to select the more sensitive and suitable photoresists to be patterned at high spatial resolution, following the project plan, twelve different types of photoresists have been exposed to several EUV doses in the range 15-500 mJ/cm$^2$ after having been deposited as a thin layer on a silicon wafer.

In order to perform multiple sample exposures at different EUV doses without venting the vacuum chamber at each exposure, a motorized sample holder (with remote control) has been realized. The holder can host up to six 13×13 mm$^2$ sample couples which can be exposed to six different EUV doses. A couple of 7 mm diameter holes on a fixed metallic screen placed at 12 cm from the source allows each of the sample couples to be exposed on a 7 mm disk only when located in front of the holes at 8 mm from the screen, i.e. at 12.8 cm from the source. A 150-nm-thickness Ni-mesh-supported zirconium filter on the screen holes limits the exposure EUV band to the 10-20 nm range, thus cutting both the visible and the ultraviolet radiations.

Figures 4.2.3 and 4.2.4 show a schematic of the exposure setup and a photo of the sample holder, respectively.
As shown in Fig. 4.2.3, the screen is properly shaped to screen the sample holder plate from scattered UV or visible radiation.

![Image](image1.png)

*Figure 4.2.4: Photos of the sample holder: a) Full structure, b) Holder plate with six pairs of wafer samples mounted on it.*

As in the case of the photoresist samples, also the EUV detectors (PIN diodes) have been filtered by a 150-nm Zr filter in order to select the 10-20 nm wavelength range. To achieve a high accuracy on the values of the released EUV doses, an accurate calibration of the PIN diodes is needed. This is not a trivial task since some spurious effects, like a non-uniformity of the residual Xe gas pressure in the vacuum chamber or EUV grazing reflections on the chamber walls, can easily generate artefacts. Such spurious effects have been studied performing different analyses:

a) the Xe pressure has been measured in different points of the vacuum chamber;
b) the PIN diode signal has been measured at different Xe residual pressures and at different distances from the source;
c) a pin-hole camera equipped with an EUV dosimetric film has been used to take images of the radiation impinging on the PIN diode.

From a), the Xe pressure results to be rather uniform, in spite of the fact that the chamber is in a dynamic equilibrium state. The analyses b) and c) revealed the presence of undesired reflections, which have been properly screened.

The EUV fluence in the 10-20 nm range vs. distance from the EUV source at different residual pressures of Xe, as detected by the PIN diode, is reported in Fig. 4.2.5 together with its best-fit according with the theoretical behaviour expected in the case of a point-like source (as in our conditions):

\[
F(d, p) = \frac{E_0}{d^2} e^{-\alpha p/d}
\]  

(1)

where \(E_0\) is the energy/shot/sr emitted by the source, \(d\) is the distance from the source, \(p\) is the residual Xe gas pressure, and \(\alpha\) is the Xe effective absorption coefficient, i.e. the absorption coefficient averaged on the convolution of the plasma emission and the 150-nm Zr filter transmission spectra.
The very good fit of the experimental fluence values with Equation (1) demonstrates that spurious effects have been eliminated. Finally, a check has been performed on a possible sensitivity depletion of the PIN diode generally used for the fluence measurements (placed at about 50 cm from the source), due to pump-oil or other impurities deposition on the sensitive area. The exercised PIN diode has been calibrated by comparison with a new PIN diode used as absolute reference (generally kept out of the chamber in a clean box). After this calibration, the fluence on the samples is known with a relative accuracy (sample to sample) of approximately 1% and with an absolute error of about 10%.

In addition to samples exposures and detectors calibration, other actions have been carried out for improving the source reliability: current and voltage probes have been substituted; the Xe injection nozzle has been modified to enlarge the range of the Xe possible injection pressures and, consequently, of the source operating conditions; finally, the source cleanliness has been improved by substituting the brass ground electrode with an elkonite (Cu/W) one and by deflecting ionic debris through a permanent magnet dipole placed in front of the source, as shown in Fig. 4.2.6.

Once the exposures of resists for sensitivity measurements are completed, we will proceed with the development of a proper experimental set-up for high resolution patterning of photoresists.
4.2.4 Optical systems for solar technologies

The Radiation Sources Laboratory has recently applied its expertise in optics to the positioning of concentrating solar systems with respect to the sun and to innovative linear concentration schemes. To this aim, the Laboratory has developed a simplified analytical algorithm that allows the astronomical calculation of the sun position with an accuracy of about 1 minute of arc. Starting from this algorithm, a new high precision solar compass was patented (application number RM2012A000664) and two prototypes were developed during 2012-13.

In 2014 the second prototype of the ENEA electronic sun compass has been further improved and fully set operative. More specifically, the compass has been integrated in an theodolite (a Tecnix model FET402K-L) in order to exploit the high accuracy of both the goniometers and the level of the theodolite. A variable optical density filter, based on a couple of rotating polarizers, has been integrated on the compass in order to easily adjust the light intensity on the compass detector. An ultraviolet band-pass filter has been inserted between the variable filter and the compass optics (a slit realized on a glass substrate by electron beam lithography) in order to minimize the diffraction effects. This filter works by absorbing the sunlight so that possible multiple reflections are drastically cut down. A picture of this second prototype is shown in Fig. 4.2.7.

![Picture of the second prototype of the ENEA sun compass](image)

*Figure 4.2.7: The second prototype of the ENEA sun compass. The variable attenuation filter is visible on the 45° tilted entrance side of the compass detector.*

Differently from the case of the first prototype (see Annual Report 2013), the calibration of this second prototype has been obtained by means of a Calibration Optical Bench (COB) which has been properly developed. The COB is based on three collimated laser beams which simulate the solar radiation for three different values of the sun elevation angle above the horizon. By placing the compass (mounted on the theodolite) on the COB and acquiring the sun (i.e. the laser beam) image position on the compass detector as a function of the theodolite horizontal rotation angle, all the five compass calibration parameters (see Annual Report 2013) can be determined. A schematic drawing and a picture of the COB are shown in Fig. 4.2.8.

The COB allows an almost automatic calibration of sun compasses, which is a suitable feature for an industrial compass production. Furthermore, by repeating the calibration at different environment temperatures, it allows very easily to determine a potential thermal drift of the compass under calibration. For example, the second prototype showed a linear thermal drift of +0.05 arcminutes/°C in the range 0-30 °C. This thermal effect can be easily compensated while using the compass.
The good performance of both the sun compass and the COB used for the compass calibration were confirmed by tests where the experimental azimuth values, of an observed object, given by the compass have been compared with the theoretical values extracted from satellites maps or from the sun-transit technique, as shown in Figs. 4.2.9 and 4.2.10: the experimental values of the azimuth differ from the averaged theoretical ones by less than 1 arc-minute. The comparison of our compass performance with a Leica total station equipped with a multiple GPS system for orientation measurements (see Fig. 4.2.11) has been particularly significant.
Figure 4.2.10: Comparison between the North direction determined by the sun-transit technique in 2012 (dashed line) and the experimental azimuth values (for the same direction) given by the sun compass second prototype after being calibrated by COB (circles).

Figure 4.2.11: Arc-minutes of the azimuth of an electricity tower of the ENEA Centre observed from a point approximately 100 m far away: comparison between the experimental values given by the sun compass (blue circles) and that given by a Leica Total Station equipped with a multiple GPS system (green dashed line); the theoretical value, extracted by Google Maps (GM), or by Google Earth (GE) or by Faureragani (FA) at the web site http://www.faureragani.it/mygps/getlatlonita.html (red dotted lines), exploiting a reference point (a telecommunications antenna 15 km far away from the ENEA Centre), are also reported.

The orientation measurements done by the sun compass have demonstrated again an accuracy better than 1 arc-minute, much better than the total-station ones, in spite of a cost more than two orders of magnitude lower. This result demonstrates that in regions where the satellites signals for GPS can be perturbed by reflections on buildings or trees (as for the ENEA Centre case) the sun compass can be extremely competitive with respect to such orientation device. Of course, in such comparisons, the observation targets or reference points have been chosen sufficiently far away from the compass (> 15 km), so that the spatial positioning errors in the satellite maps (in the order of 1 m) lead to a negligible error (<<1 arcminute) in the calculated azimuth angle.

This second prototype of sun compass has been already used in applications: in September 2014 it has been carried at the D.D. S.r.l. site in Mereto di Tomba for measuring the orientation of the main axis of the parabolic trough of a Concentrating Solar Power (CSP) plant on the company area (just a prototype), which was nominally oriented along the South/North direction by using conventional topographic techniques. The measurements with the sun compass revealed a true axis orientation of -38.2° ± 0.2°. Such a mistake, if not included in the sun tracking calculation, could lead to a significant power
loss of the CSP plant. Figure 4.2.12 shows the parabolic trough of the D.D. CSP plant and the compass during such measurements.

As planned, the collaboration agreement between ENEA and D.D. S.r.l. for the development of three new sun compass prototypes and their integration in both the CSP plants at D.D. s.r.l. and at ENEA Casaccia Centre (UTRINN Unit) started in spring 2014. These new compasses are designed to be integrated in one of the supporting towers of each parabolic trough mirror (generally a working sun power installation is composed by more than 100 troughs) and to perform a dual function:

a) periodic measurement of the orientation of the parabolic mirror rotation axis;
b) driving the mirror to continuously face the sun, taking into account the measured orientation of its axis.

So, these compass prototypes differ from the previous mainly for four aspects:

- all the components (sun detector, GPS, electronics, etc.) are included in a single box;
- the electronics includes an interface to be connected to the PLC which receives the sun tracking angle by the compass software and drives the motors of the parabolic mirror;
- a high-precision digital inclinometer is included in the compass (rather than a spirit level) so that no operators are required for its working;
- the CCD has an automatic exposure time in such a way that no variable attenuation filters are needed to adjust the light impinging on the sensor. This fact also contributes to the automatic working of the compass.

The company decided to pursue a high level of industrial engineering for such prototypes with a dedicated electronics board now under development, so that at the end of the project the compass will be ready for a massive industrial production.
4.2.5 Multidisciplinary study of the Shroud of Arquata

In the frame of an agreement between the City of Arquata, the UTAPRAD of the ENEA Centre of Frascati and the Institute of Complex Systems of CNR, we used sophisticated optical and spectroscopic non-invasive technologies, suitable to the study of Cultural Heritage, to perform the first in-depth measurement of the Shroud of Arquata, a 1:1 copy of the Shroud of Turin which dates back to 1653. The most peculiar feature of the Shroud of Arquata is the front and back human footprint which was not produced by apparent drawings or painting as in the other copies of the Shroud, see Fig. 4.2.13.

Figure 4.2.13: Photograph of the Shroud of Arquata, balanced on the reference white. The linen cloth is 4.4 m long and 1.1 m wide, and it is sewn on a red silk fabric. The life-size frontal and dorsal images of a human body are not produced by apparent drawings or painting as in the other copies of the Shroud of Turin.

The measurements were performed in three consecutive days, from 18 to 20 June 2014. The elaboration of experimental results allowed to obtain scientific data useful to suggest the possible origins of the double image, of the stains simulating blood and of the false patches embedded on the Shroud of Arquata. In addition, the experimental data allowed to develop a plan for the proper long-term conservation of this Shroud.

Figure 4.2.14: Left: photo of the frontal legs and feet. Right: the same image subtracting the blue and the red components.
More in detail, the analysis of the high-resolution photographs separated in the three RGB channels corresponding to the three primary colours of red, green and blue allowed to:

- **a)** discover almost invisible pigments that mimic the signs of flogging on the legs, see Fig. 4.2.14.
- **b)** Identify the invisible contours of anatomical parts, such as the feet, see Fig. 4.2.14.
- **c)** Highlight invisible traces within the frontal and dorsal marks of the body, ascribed to abrasion and to a passage of liquid along the direction of the warp yarns, see Fig. 4.2.15.

To reinforce this last observation, the macro photography showed that the warp threads within the body marks have a mean diameter significantly smaller than outside the body. Accordingly, the fabric density (warp + weft) within the body marks is on average smaller (and therefore more transparent) than outside the same mark. This can facilitate the perception of the red cloth sewn below the Shroud in area, which makes the body appear on average darker than the surrounding area.

![Figure 4.2.15: Left: photograph of the face of the body on the Shroud of Arquata, balanced on the reference white. Right: the same image subtracting the blue and the red components](image)

The topological radar RGB-ITR (UTAPRAD DIM) allowed to realize a colorimetric 3-D map of the Shroud, creating a first database that will allow to monitor the actual state of preservation of the fabric and the visibility of stains, from time to time, during future analysis. This colorimetric map can also be used for museum exhibits, and educational purposes. The numerical processing of images ITR on different colour channels and multiple colour spaces have provided additional qualitative and quantitative information on the Shroud.

The infrared image obtained by the topological radar shows the silhouette of the body mark, the blood stains, the pattern of false burns and the letters of the inscription. This means all these details are due to substances penetrated inside the threads of the linen cloth of the Shroud, suggesting their probable origin of painting with a very diluted pigment. Conversely, the same infrared image shows that the internal area of the body marks appears free from foreign material (pigments).

The spatial- and spectral-resolved analysis of Laser-Induced Fluorescence (LIF) (UTAPRAD DIM) shows that:
a) The spectrum both inside and outside of the body marks is typical of the cellulose. The comparison with the LIF spectrum of a Renaissance “papier mache” confirms that the characteristics of the fluorescence of the cellulose of the Shroud of Arquata are the same as a cellulose aged 400 years.
b) Some spectral peaks relative fluorescence stain on cost, stains and central to the letters of the inscription are different than the other spectra, suggesting that these were made by a different pigment from that used for the outline of the body marks, blood, etc.
c) The fluorescence spectrum of red spots that simulate blood does not correspond to any of the spectra of these red pigments commonly used in the Renaissance: clay, lacquer of madder, red ochra, Pozzuoli red, English red, Herculaneum red, cinnabar.

The reflectance spectra and absolute absorbance (CNR ISC):
a) Validate the LIF results about the dominant presence of aged, oxidized cellulose both inside and outside of the body marks, confirming that the imprint was not created by pigments.
b) Pigments are present in the letters and in the stains that simulate blood.
c) The spectrum of the blood stain on the side suggests the presence of a pigment mixed with a substance characterized by a sharp drop of absorption around 550 nm. In the literature we have found that the methemoglobin, a form of hemoglobin oxidized by aging, has a peculiar spectral behaviour, compatible with the change at 550 nm of the spectrum of the blood stain. Therefore, it is possible that the artist who created the Shroud of Arquata in the seventeenth century has painted the blood stains by mixing pigments with blood.
d) The high reflectance at 800 nm confirms that the less reflective parts of the outline of the body, hair, blood stains and patches simulating burns are composed by added materials (pigments).
e) The set of reflectance spectra/absolute absorbance suggests that the body marks on the Shroud of Arquata were achieved by a process of degradation of the linen able to increase the rate of oxidation (aging) of the cellulose fibres, indirectly confirming the results of the photos decomposed into RGB channels. Some details (patches, blood, letters) were instead made.
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